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<td>$H_{OG} = \frac{Z}{\int_{y_1}^{y_2} \frac{dy}{y_e - y}}$</td>
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<td>$H_G = \frac{Z}{\int_{y_1}^{y_2} \frac{dy}{y_1 - y}}$</td>
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$H_{OG} = H_G + \frac{mG_m}{L_m} \cdot H_L$

$\frac{Z}{\int_{y_1}^{y_2} \frac{dy}{y_i - y}} = \frac{Z}{N_G} = H_G = \frac{G_m}{K_G a}$

$k_G = \frac{Sc_G}{Sc_G} \frac{h}{C_pg\mu}$

$\frac{1}{H_G} = \frac{Sc_G A}{Sc_G} = \frac{f}{f_2} = \frac{C Re}{g}$

$\Delta p = p_1 - p_2 = h_r \rho u^2 = 4f Z \rho u^2$
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\[ f = \frac{h_f f_L D e g_c}{2 \rho_g u^2 z} = c^1 \text{Re}_G^n \]

\[ H_G = c^{11} \text{Sc}_G^{2/3} \text{Re}_G^n \]

\[ H_{OL} = H_L + \frac{L}{M G} H_G = \frac{z}{\int_{x_1}^{x_2} \frac{dx}{x-x_e}} \]

\[ M = \frac{L}{G M} \cdot \frac{H_G}{H_{OL}} = \frac{L}{G M} \int_{x_1}^{x_2} \frac{dx}{x-x_e} \]

\[ \text{H}_L = \frac{L}{k_L a} \]

\[ V_s = \left( \frac{9g c^2}{8 \mu L \rho L} \right)^{1/3} \]

\[ k_L = 2 \sqrt{\frac{D_L}{\pi t}} \]

\[ \frac{k_L}{D_L} = 0.73 \text{Sc}_L^{1/2} \text{Re}_L^{1/3} \left( \left[ \frac{2 g \rho c L^2}{\mu L^2} \right]^{1/6} \right) \]
EXPLANATION OF METHOD OF CALCULATION

1. Find pressure drop in column. Using equations 5.10 and 5.10a
   calculate 'f' for different flow rates of vapour.

2. Plot 'f' vs. Re\(_G\). Establish equation 5.10a. From equation 5.10a
   and 5.9 find H\(_G\)\(_a\).

3. Calculate 'M' from equation 5.13. This requires experimental values
   of L\(_M\)', G\(_M\)', x, y and equilibrium data.

4. Calculate H\(_{OL}\) from equation 5.1.
   H\(_{OG}\) from equation 5.1.

5. Calculate H\(_L\) from equation 5.12. L\(_M\)', G\(_M\)' are measured, H\(_OG\)
   from equation 5.1, H\(_G\) from equation 5.11, m from equation 5.13.

6. This H\(_L\) is H\(_L\) EXPTL in Table 22.

7. Calculate k\(_L\) and H\(_L\) from equations 5.16 and 5.17. This H\(_L\)
   is H\(_L\)Theor. in Table 22.
POLYTECHNIC OF WALES

DEPARTMENT OF CHEMICAL ENGINEERING

Mass Transfer Studies In A

'Finnntak' Distillation Column

by

W. Llewellyn

A thesis submitted in part
fulfilment of the requirements for the Degree of
Philosophiae Doctor in the Polytechnic of Wales.
This thesis is a presentation of material submitted in candidature for a degree in the Polytechnic of Wales. No part of this thesis is being submitted to any other University or Polytechnic.
DECLARATION

This dissertation is based on work executed in the Department of Chemical Engineering at the Polytechnic of Wales during the period October, 1970, to October, 1973, and is the original and independent work of the author except where specifically acknowledged in the text.

........................Candidate

........................Director of Studies.

Date: July 1976
This thesis presents the results of an investigation of the behaviour of a new form of rectification device under vacuum distillation conditions.

The operating conditions were so varied as to not only provide a measure of 'Finpak' column efficiency under a variety of such conditions, but also to illustrate the effect such variables have on the overall distillation efficiency.

The results of the research indicate that the 'Finpak' fractionating column is a suitable contacting device for the investigation of mass transfer processes where low liquid rates are employed, such as in vacuum distillation.

With regards to the vacuum distillation process it was shown that column efficiency improved on reducing stillhead pressure, and it is proposed that the increase in efficiency occurs as a result of a reduction in the resistance to mass transfer within the liquid film, which may be as high as 90% of the total resistance to mass transfer under the low liquid flow conditions employed throughout the study.
The author wishes to thank Dr. C. Pryce, Lecturer at the Polytechnic of Wales, who directed this study, for his encouraging guidance, criticism, advice and discussion in a proper balance which ensured completion of the study.

Thanks are due to Mr. G.T. Dolan for making the department facilities available for this work.

Financial support from the Scientific Research Council is gratefully acknowledged.

Numerous phases of the study required assistance from the workshops and technical staff, and their co-operation is much appreciated. In particular, grateful thanks are extended to Mr. J. Lloyd and Mr. B. Evans for the procurement and manufacture of items of equipment.

The author wishes to express his gratitude to his wife, Susan, for her sustained tolerance and understanding, to his parents for their encouragement, and to Susanne for her long suffering patience in typing the script.
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NOMENCLATURE

A - cross sectional area of column \( (L^2) \)

\( A_H \) - area above the curve \( \log \frac{\gamma_1}{\gamma_2} = 0 \)
Herington Consistency Test \( (L^2) \)

\( a \) - surface area of packing per unit column volume \( (L^{-1}) \)

\( B_H \) - area below the curve \( \log \frac{\gamma_1}{\gamma_2} = 0 \)
Herington Consistency Test \( (L^2) \)

\( B_{11} \) - second virial coefficient of component 1

\( B_{22} \) - second virial coefficient of component 2

\( B_{12} \) - mixed virial coefficient of components 1 & 2

\( c_p \) - specific heat, vapour phase at constant pressure

D - molecular diffusivity \( (L^2T^{-1}) \)

\( D_e \) - equivalent diameter of column \( (L) \)

d - diameter of column \( (L) \)

\( \Delta F^E \) - excess Gibbs free energy change on mixing

\( f \) - friction factor

\( f_i^{CV} \) - fugacity

\( G \) - mass vapour rate per unit cross section \( (MT^{-1}L^{-2}) \)

\( G_m \) - molar vapour rate per unit cross section \( (MT^{-1}L^{-2}) \)

\( g \) - acceleration due to gravity \( (LT^{-2}) \)

\( g_c \) - gravitation constant

H - height of column \( (L) \)

\( H_G \) - height of transfer unit, gas phase \( (L) \)

\( H_{OG} \) - overall height of transfer unit, gas phase \( (L) \)

\( H_L \) - height of transfer unit, liquid phase \( (L) \)
NOMENCLATURE (continued)

- $H_{OL}$ - overall height of transfer unit, liquid phase
- $H^E$ - molal heat of mixing
- H.T.U. - height of transfer unit
- H.E.T.P. - height equivalent to a theoretical plate
- $h_f$ - loss of head due to friction through the column
- $J_D$ - mass transfer number
- $K_{i_1} = \frac{y_i}{x_i}$, equilibrium value
- $k_G$ - mass transfer coefficient, gas phase
- $k_L$ - mass transfer coefficient, liquid phase
- $L$ - volumetric liquid rate per unit column cross section
- $L_m$ - molar liquid rate per unit column cross section
- $m$ - slope of the equilibrium line
- $M$ - number of transfer units
- $N$ - number of transfer units, gas phase
- $N_A$ - molar rate of transfer per unit column cross section
- $N_C$ - number of transfer units, gas phase
- $N_{UG}$ - overall number of transfer units, gas phase
- $N_L$ - number of transfer units, liquid phase
- $N_{OL}$ - overall number of transfer units, liquid phase
- $P$ - pressure, total stillhead
- $P_i$ - vapour pressure of component i
- $\Delta P$ - pressure drop through column
NOMENCLATURE (continued)

$S_r$ - surface area of liquid film, laminar  

$S_w$ - surface area of liquid film, waves  

$S_r/S_w$ - enlargement factor relating to increase in surface area of liquid on production of waves  

$T$ - temperature, $^\circ$K  

$T_{r1}, T_{r2}$ - reduced temperature condition relating to components 1 & 2  

$T_{c1}, T_{c2}$ - critical temperature relating to components 1 & 2  

$t$ - time of contact  

$U$ - vapour velocity through column  

$V_E$ - volume heat of mixing  

$V_L$ - molal volume, liquid phase  

$v_s$ - liquid surface velocity  

$x$ - mole fraction m.v.c. in the liquid phase, unless otherwise stated  

$y$ - mole fraction of m.v.c. in the vapour phase unless otherwise stated  

$Z$ - height of column  

$\tau$ - mass liquid rate per unit periphery  

$\rho$ - density  

$\mu$ - viscosity  

$\sigma$ - surface tension  

$\gamma$ - activity coefficient, vapour phase  

$\psi$ - interaction parameter  

$l$ - $V_E/RT.dP/dx_1$, isothennic case  

$l$ - $H^E/RT^2.dT/dx_1$, isobaric case  

$\tau$ - shear stress
NOMENCLATURE (continued)

Subscripts

- equilibrium value
- more volatile component
- less volatile component
- inert gas
- component i
- component 1
- component 2
- vapour phase
- liquid phase
- overall value

Abbreviations

Gr - Grashof Number
Sc - Schmidt Number
Re - Reynolds Number
Pr - Prandlt Number
m.v.c. - more volatile component
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CHAPTER 1
INTRODUCTION.

1. Introduction

The distillation process has always been of paramount interest within chemical engineering, moreover, in recent years there has been a steadily growing increase in the use of vacuum distillation as an effective means of separation not only within the petrochemical industry but within the pharmaceutical and food industries.

The ever growing demand for more sophisticated and inherently thermally unstable compounds has focussed more attention on the vacuum distillation process as a means of separation and preparation, and development in producing equipment suitable for use under vacua, and in order to gain a better insight into the mechanics of the process as a whole.

The investigation described here was undertaken to establish the efficiency of a laboratory scale distillation column under a variety of operating conditions, the principle variable of which was the stillhead pressure employed.

As a consequence of employing low stillhead pressures with their associated low liquid flows and high vapour velocities, it is also possible to examine the mechanics of mass transfer within the
individual phases, especially the contribution of individual film resistances to the overall resistance to mass transfer, with reference to varying operating conditions.

It was attempted to employ two distinct yet inter-related techniques to illustrate the behaviour of individual phase resistances under as wide a range of operating conditions as was experimentally practical.
CHAPTER 2
LITERATURE SURVEY.

2.1. Introduction

In the past twenty years a vast amount of data relating to vacuum distillation study and application has been published. For the purpose of this survey, reference material is categorised into the following sections :-

(a) A review of the types of apparatus developed for and applicable to low pressure rectification.

(b) A review of published material relating to the effect of operating variables on distillation efficiency.

(c) A review of published data on the contribution of individual phase resistances to the total resistance to mass transfer.

2.2. Vacuum Distillation Equipment.

The separation by distillation of many of the complex chemicals now being produced is often complicated by their thermal instability. Such thermal degradation may be reduced either by lowering the operating pressure of the distillation process, and hence the temperature, or by reducing the residence time of materials in the reboiler. Hickman and Embree have shown that reduction in operating temperature and reduction in residence time are of equal importance in
minimising thermal breakdown. There are, however, many practical difficulties to be overcome to reduce residence time and maintain basic simplicity in reboiler design. For this reason most development effort has been concentrated on achieving a low reboiler pressure, and this, coupled with the increased use of the vacuum distillation process, has led to a wide variety of equipment becoming available.

Contacting devices at present available for use as distillation equipment fall into three broad groups. These are basically:

(a) Plate columns.
(b) Packed columns.
(c) Specialised columns.

The low reboiler pressure, which is a feature of vacuum distillation, is the sum of the column head pressure and the pressure drop across the column. The former can be controlled but the latter is a function of the characteristics and loading of the column. Additional features of the process are the low liquid rates and high vapour velocities experienced. For efficient separation at low pressures it is necessary that the equipment employed has the following characteristics:

(a) Low pressure drop per theoretical plate.
(b) High throughput per unit column cross section.
(c) Large interfacial area per unit volume.
(d) High separation efficiency per unit length.
(e) Low operating costs.

A consideration of the first of these characteristics excludes many distillation devices from use under vacuum.

2.2.1. Plate Columns.

The bubble-cap plate has enjoyed wide application as a distillation device for many years, but is unsuitable for use under low pressure conditions because of its inherent high pressure drop characteristic. In recent years the bubble-cap plate has been largely superseded by the valve plate and sieve plate, where the overall plate pressure drop is lower \(^{(7)}\) \(^{(106)}\) and hence may not preclude their use as vacuum distillation equipment.

Billet et al \(^{(7)}\) present the results of experimental studies involving the use of plate and packed columns under identical low pressure conditions. Their results indicate that under moderately low pressures, in excess of 100 Torr, when comparing the performance data of thin walled metal Raschig Rings of various diameters with those of sieve plates and T.F.
Plates, insignificant differences in the specific pressure drop for the Raschig Rings and the T.F. Plates exist only in the range of maximum permissible vapour load factors. Further, thick walled ceramic Raschig Rings have higher pressure drops\(^{(105)}\) than modern plates at any useful throughput.

The type B Kittel plate is a development of the Kittel plate to cater for the conditions prevailing in a high vacuum distillation process\(^{(91)}\). In order to overcome the difficulties of high vapour velocity and low liquid loading, the plates between the lower and upper grid of a Kittel plate are filled with one inch Raschig Rings. This has the effect of impeding the downflow of liquid and introducing an artificial liquid hold up. In addition, an antispray grid is invariably employed for vacuum operations and this permits very high vapour velocities to be used. The immediate effect of this grid is to flatten the pressure drop curve (Fig. 1) thus increasing the range of the plate\(^{(106)}\). In comparison to the sieve plate the Kittel plate has a lower pressure drop for a given vapour velocity, but is more sensitive to liquid loading.

For the APV-West plate the pressure drop is almost directly proportional to the vapour velocity\(^{(106)}\). At a given rate the pressure drop across such a plate is higher than that of a Kittel plate,
but less than that across a sieve plate.

The Turbogrid is the simplest in construction of all plates. In common with the sieve plate and Kittel plate it has a minimum vapour velocity below which it is unstable. For a given duty the pressure drop across the Turbogrid is less than that across the other types of plates. There is, however, an inherent danger of channeling and the plate is reported difficult to use (18). However, Zuiderweg et al. (131) comment on its usefulness as a vacuum distillation device.

Descriptions of the various other plate types available are given in standard textbooks (18) (75). The types considered here are, perhaps, the most promising for use under vacuum distillation conditions. Of these the APV-West plate is the most flexible, maintaining a high efficiency throughout its operating range. The Kittel Plate, the Turbogrid, and Sieve Tray have greater capacities than the APV-West, but have more restricted operating ranges. The Kittel Plate operates efficiently at lower pressure drops than the other plates, making it more suitable for vacuum distillation operations, where flexibility is not of prime importance (91) (106). The low pressure drop modification of the APV-West has greater flexibility but still retains a higher pressure
drop characteristic than the Type D Kittel over most of its range (106).

The opinion still prevailing that packed columns have a lower pressure drop per theoretical plate than plate columns is not always correct, as has been proved by recent research (7), and certain types of plate columns may be used for pressures as low as 2 Torr (91).

2.2.2. Packed Columns

Historically speaking this type of column has almost always been the first choice for use under vacuum. This has resulted in the development of a wide range of packing materials (13) (31) (72) (93) (111) (113). The simplest form of such packings is the randomly dumped rings. Thick walled Raschig Rings, as stated earlier, have excessive pressure drop characteristics, Sillet et al, (8) (9) (10), however, report considerable success in using thin walled metal Pall rings and claim them superior to other packing types. Another type of packing with outstanding separation properties is the gauze packing (13) (72) (35) (111) (113). The development of this packing is based on investigations into the formation and stability of liquid films. It is mainly the
capillarity of the gauze that is responsible for causing the reflux to spread out as a liquid film. These types of packing are often characterised by a large value of "Height equivalent to a theoretical plate" (H.E.T.P.) or Height of Transfer Unit (H.T.U.), compared with values achieved at atmospheric pressure (31). A further development of this type of packing is the expanded metal packing developed by Ellis et al (31) which is an improvement on the Knitmesh packing investigated by Taylor (113) and compares favourably with the gauze packing developed by Silzer (111).

Zuiderweg et al (131) emphasised the excellence of grid tray design of high open area in vacuum distillation studies. This design has been developed (h5) (91) to yield grid packing elements with a comparatively large open volume. Consequently it allows high throughputs at moderate pressure drops. Course and fine versions of these grids are available (h5).

The pressure drop characteristics of these packing materials are presented in Fig. 2. However, the optimum choice of packing type should be based on both the H.E.T.P. data and the pressure drop per theoretical plate (31).

2.2.3. Specialised Columns.

These are columns which were
developed for specific duties or which cannot be directly
categorised as either plate or packed in nature.

Bancroft and Rae (3) report the
performance of a wetted wall type of packing consisting
of closely spaced vertical sheets of aluminium. The
reported pressure drop per transfer unit is lower than
many other columns at the same throughput, although many
other packings give much lower H.T.U. values.

Rectification using mechanically
scraped surface equipment has been carried out (73),
(105);(138). Zuiderweg (128) reports the use of a
spinning band column over a wide range of pressures, and
equates its performance to that of a wetted wall column
of similar dimensions. Another such type of column is
the rotary thermal rectifying column of Byron et al (16).
This column operates satisfactorily only at intermediate
and low pressure ranges, failing to function usefully
at extremely low pressures.

There exists a miscellany of other
specialised equipment; the Kuhn column (64) helical
coil columns (73), Eldershaw sieve plate columns (50)
the plate type film contacting equipment of Leva (65)
and Linder (67), Hyflex sieve plate (4) and many others.
The majority of these columns have been utilised only at
laboratory or pilot plant level, and many of the references
do not provide sufficient data to extract comparative performance figures.

There is, therefore, a wide and diverse range of contacting equipment which have been developed or modified for specific use as vacuum distillation equipment. Comparative performances on a pressure drop per theoretical plate basis are presented in Figs 1 and 2. However, the final comparison and selection should be made utilising H.E.T.P. or H.T.U. values where available, and eventually on a cost basis.

2.3. **Effect of Reduced Pressure on Distillation Efficiency**

The major operating variable in vacuum distillation studies is the operating pressure. This section will, therefore, review the effects of reduced pressure on the distillation process. These effects can be broadly categorised into two groups, namely the effect on vapour flow and the effect on distillation efficiency.

2.3.1. **Vapour Flow and Pressure Drop**

Beaton et al (16) defined three pressure regions; pressures of the order of atmospheric and greater, where vapour flow is turbulent; pressures of the order $10^{-2}$ and $10^{-4}$ Torr, where Knudsen flow prevails; and thirdly intermediate pressures where
laminar flow exists.

As the pressure on a distillation system is reduced, then for any given mass rate, the vapour velocity increases. The vapour density, however, is decreased by the same factor if the slight temperature effects are neglected, and hence the Reynolds number of the vapour phase is largely unaltered.

From a consideration of the friction factor relationships

For turbulent flow

\[
\frac{\tau}{\rho u^2} \propto (\frac{\rho_d u}{\mu})^{-1.0}
\]

For laminar flow

\[
\frac{\tau}{\rho u^2} \propto (\frac{\rho_d u}{\mu})^{-0.2}
\]

As \( \mu \) is substantially independent of pressure for the reasons outlined above, a variation in pressure above causes these relationships to reduce to

\[ \tau \propto u \mu \quad \text{and} \quad \tau \propto u \mu^{0.2} \]

For operation above that region where the mean free path of the molecules is of the same order as the duct dimensions, then the change in viscosity is small compared to the change in velocity. Hence for a given throughput, the pressure drop increases proportionally to the decrease in pressure. For the
columns earlier described that have high pressure drop characteristics, it is this feature that restricts their use to moderate vacua.

The flooding rate is considerably decreased by pressure reduction, and Nemec (78) has discussed in detail the effect of pressure reduction on the capacity of high vacua distillation columns.

**2.3.2. Effect of Reduced Pressure on Distillation Efficiency.**

The confusing and contradictory evidence provided by the literature as to the effect of pressure reduction on H.E.T.P. and H.T.U. is best presented by categorising the survey into separate sections according to the effect found. According to published data, the effect of decreasing the operating pressure can cause distillation efficiency to decrease, increase, pass through a maximum or a minimum, or remain constant.

**2.3.2a. Efficiency decreases with decreasing pressure.**

Feldman et al (39) employed the system n-dodecane/cyclo hexylcyclopentane to study the efficiency of a 25cm x 91.5 cm high Fodbielbank column. They found that as the pressure was reduced from 760 Torr to 50 Torr the number of theoretical plates was reduced from 70 to 30. Walsh, Sugimura and Reynolds (119), employing the same test mixture in 1 inch and 2 inches diameter Heligrid packed columns, showed
that for a pressure reduction from 760 to 30 Torr
the K.E.T.P. increased from 0.5 inches to 2.0 inches
at a fixed vapour velocity.

Kirschbaum and David (62) used the
systems ethanol/water and benzene/toluene, over a
pressure range 760 to 250 Torr and 760 to 50 Torr
respectively, in a 100 mm diameter column packed to
a height of 1 metre with 8 mm Raschig Rings. They
showed that for the former system K.E.T.P. values
increased by approximately 20%, and by approximately
30% for the latter system. Bliss, Eschayer and
French (11) used a 3 inch diameter column packed with
1/2 inch Raschig Rings with the system
o-dichlorobenzene/o-diethylbenzene over a pressure
range 100 to 16 Torr. They found that the H.T.U.
increased from 1.16 ft. at 49.1 Torr to 1.64 ft. at
16.5 Torr.

Zelvenskii, Titov and Shalygin (127)
used a 25 mm diameter column packed with helices to
separate the systems dichloroethane/benzene and
chlorobenzene/ethylbenzene at various pressures in
the range 760 to 100 Torr. They found a reduction
in efficiency corresponding to a reduction in pressure
which though insignificant in the range 760 to 400
Torr was substantial below 300 Torr.
Zuiderweg (128) found that efficiency fell by 25% in a 11 mm diameter Vigreux column over the pressure range 760 to 1 Torr. A 6 mm spinning band column showed a 70% efficiency loss as pressure was reduced from 760 to 10 Torr.

Ellis (27) studied the effect of pressure on distillation efficiency in a 1½ inches diameter column packed with Knitmesh Multifill elements. Using methylcyclohexane/toluene he obtained H.E.T.P. values of 1½ inches - 2 inches at 760 Torr and 1½ inches - 2½ inches at 200 Torr. Ellis, Porter and Jones (33) used the same packing elements in a 6 inch diameter column and found that, at a constant boil up rate of 400 lbs./ft.²/hr., the H.E.T.P. for the separation of methylcyclohexane/toluene increased from 3 inches at 760 Torr to 5½ inches at 200 Torr.

Ellis and Contractor (30) studied pressure effect on distillation efficiency in a 1½ inches O1ershaw Column and found that plate efficiency fell from 82% at 760 Torr to 71% at 200 Torr whilst separating methylcyclohexane/toluene at an F factor of 0.3.

2.3.2b Efficiency increases with decreasing pressure.

Peters and Cannon (39) used n decane/trans decalin and chlorobenzene/ethylbenzene to study...
the effect of pressure on distillation efficiency in columns packed with Cannon protruded and McHahon packings over the pressure range 735 to 10 Torr. Comparing the efficiencies at 85% of the flooding rate they showed that the H.E.T.P. fell by about 30% as pressure decreased.

McLaughlin(76) investigated the efficiency of a helical coil distillation column in the pressure range 760 to 1 Torr using ethanol/water and chlorobenzene/ethylbenzene systems. He found that in columns of greater than 10 mm bore the efficiency doubled as pressure was reduced from 760 to 15 Torr.

Gelperin and Zelenetskii(42), studied the systems chlorobenzene/ethylbenzene, n-octane/toluene, diphenyloxide/ethyl salicylate, and menthol/menthane at pressures of 760 to 5 Torr in a 30 mm column packed with 4.5 mm Raschig Rings. They found that column efficiency in all cases increased, measured at a fixed proportion of flooding, with a decrease in pressure. They believed this to apply generally in view of the fact that the systems they employed varied widely in their chemical and physical properties and in their relative volatilities.

2.3.2c. Pressure regions of maximum or minimum efficiency.
Struck and Kinney\(^{(110)}\) concluded that the H.E.T.F. passed through a flat minimum between 50 and 100 Torr, after distilling n-decane/trans-decalin in 3/4 inch diameter column with various packings. Mylis et al\(^{(74)}\) used a 25 cm diameter column packed in turn with saddles, helices, spheres and heligrid packing elements with the system n-dodecane/cyclohexylcyclopentane in the pressure range 760 to 20 Torr. They suggested that at a pressure of around 200 Torr there existed the optimum pressure for securing maximum separating efficiency for all the packings.

Peters and Cannon\(^{(89)}\), as reported earlier, found that by comparing efficiencies at constant boil up rate there existed a maximum efficiency in the range 100 to 50 Torr.

Belvenskii, Titov and Shalygin\(^{(127)}\) defined the term "stage of separation" as the change in composition between the top and bottom products achieved in a column. They found that this stage of separation was at a maximum at an optimum pressure of about 150 Torr.

### 2.5.3d Efficiency independent of pressure.

Berg and Popovac\(^{(5)}\) used a 1 inch diameter column packed with 1/8 inch stainless steel helices.
for the distillation of n octane/toluene at pressures in the range 760 to 20 Torr and found the H.E.T.P. to be relatively independent of pressure.

Walsh, Sugimura and Reynolds (119) using n-dodecane/cyclohexylcyclopentane in 1 inch and 2 inch diameter columns packed with 3/8 inch helices found that efficiency was affected less by pressure than the variation between runs at the same pressure. They concluded that the efficiency of random packings is largely independent of pressure within the range 760 to 200 Torr.

Nandi and Jalota (77) found no effect on efficiency as a result of a pressure reduction from 760 to 200 Torr in a 2½ inch diameter column packed with 10 mm copper Lessing rings whilst distilling the systems methanol/water, ethanol/water, and benzene/toluene.

Using the system n-hexanol/aniline in a 1½ inch diameter column packed with Knitmesh Multifill elements Ellis (27) showed that the H.E.T.P. was independent of pressure over the range 300 to 50 Torr.

Hawkins and Brent (46), after extensive tests with dichlorobenzene/diethylbenzene in columns packed with spiral screen and 4 mm Raschig
Rings, showed these packings to be as efficient at 20 Torr as at 760 Torr.

Norman et al (81) showed H.E.T.P. to be substantially pressure independent in the range 50 to 5 Torr when distilling phenol/m-cresol in an 8 inch diameter column packed with 1 inch stainless steel Lessing rings.

Ellis and Contractor (30) using a 6 inch diameter sieve plate column distilling methylcyclohexane/toluene at 760 and 200 Torr found that the plate efficiency was largely unaltered.

Van Weber (120) found that a reduction in pressure affects the number of theoretical plates in a column to a negligible extent. Krell (63) states that the separation of chlorobenzene/ethylbenzene mixtures was largely unaltered by a pressure reduction from 760 to 20 Torr.

2.4. Effect Of Pressure Reduction on Distillation Variables.

The data sources reviewed in the preceding section show no fixed pattern as to the effect of pressure alone on distillation efficiency. The effect of pressure on those other variables which may have some bearing on the separation efficiency will now be discussed.

2.4.1. Temperature.

The overall purpose of vacuumum
distillation is to reduce, for many reasons, the prevailing temperatures in the distillation column and reboiler. The most important effect of the reduced temperature is that it modifies a variety of system variables which may have some further bearing on the efficiency. These system properties will be discussed individually.

2.4.2. Vapour Velocity.

From a consideration of the increased vapour velocity, Peters and Cannon\(^{(89)}\) have indicated that the vapour molecules hit the liquid phase with far greater momentum than in normal distillation. This provides far better penetration and hence better contact between vapour and liquid molecules. The higher vapour velocity also causes thinning of the vapour film at the interface thus decreasing the effective vapour film resistance. Both of these effects tend to increase the transfer rate. However, as they further indicate, vapour residence time is reduced producing a deleterious effect on efficiency.

Zelvenskii, Titov and Shalygin\(^{(127)}\) showed that at constant pressure there appeared to be two distinct regions of mass transfer around a critical point of \(\text{Re}/\text{Ra}_{\text{f}}\) of 0.6. In the first region increasing vapour velocity causes a decrease in
efficiency, and in the second the efficiency is independent of vapour velocity.

Ellis and Contractor\(^{(30)}\) in their studies using Oldershaw and sieve plate columns found that increased vapour velocities, as a result of decreased pressures, altered the foam structure, (for a foaming positive system) on the plate causing a reduction in interfacial contact area and hence a reduction in the overall plate efficiency. For a non-foaming negative system the effect on efficiency of the increased vapour velocity was smaller.

Gelperin and Zelvenetskii\(^{(42)}\) have shown that at a constant pressure the H.E.T.P. values obtained are characterised by two minima, one at low velocities, and at velocities close to flooding the second minima.

2.4.3. Relative Volatility

Generally, the relative volatility of a system varies with pressure, either increasing or decreasing. There are only a limited number of systems for which the relative volatility is pressure independent.

Berg and Popovac\(^{(5)}\) attribute any change in separation accompanying a pressure change as being primarily due to the change, with pressure, of the relative volatility.
studied the rectification of the system ethylbenzene/styrene at various pressures. This system has a relative volatility independent of pressure and they found only slight variation in column efficiency in the pressure range 30 to 130 Torr.

2.4.4. Viscosity

The accompanying temperature fall with a decrease in pressure causes the vapour viscosity to decrease resulting in a thinning of the vapour film at the interface, thus promoting mass transfer.

However, this same temperature effect also causes an increase in the liquid viscosity. Drickamer and Bradford (24) showed that plate efficiency decreased with increasing liquid viscosity. Lloyd (68) calculated that, in a particular separation plate, efficiency decreased from 56% to 31% due to the increasing liquid viscosity as a result of a pressure change from 740 to 10 Torr.

2.4.5. Diffusivity

The liquid phase diffusivities are unaffected by pressure changes but decrease with decreasing temperature, thus having a deleterious effect on the mass transfer conditions.

Vapour phase diffusivity increases
decreasing pressure. Byron et al (16) argue that this increase gives rise to two opposing effects. Firstly, the mixing rate normal to the interface is increased resulting in a lower vapour film resistance and promoting mass transfer. Secondly, however, mixing in the direction of flow increases and this may reach such an extent that the vapour stream in the column is incapable of producing a concentration gradient. In the extreme case, this would mean a column containing no plates, condenser and reboiler composition being identical. However, this would only happen at a pressure of zero Torr. Zuiderweg (128) and McLaughlin (76) both invalidate this latter point by suggesting that Byron et al ignored the correction factor which is the ratio of the diffusivity to the linear velocity. Westhaver (121) demonstrated that the back diffusion effect is controlled by this ratio which, as both terms are equally affected by pressure, is pressure independent.

2.4.5. **Surface Tension**

Dankwerts et al (21) considered the overall effect of interfacial tension in the distillation process. In packed or wetted wall columns interfacial effects may influence the degree
wetting, and thus the magnitude of the interfacial area, as well as the value of the mass transfer co-efficient.

The effect of variation of surface tension along a column has been studied by Zuiderweg and Harnens (130) and has been shown to have a profound effect on distillation efficiency. The slope of the surface tension/temperature curves of most substances is similar, thus with decreasing pressure the change in surface tension is unlikely to alter, positive systems are likely to remain positive and negative systems remain negative.

With regards to the absolute value of surface tension, however, this increases with a reduction in pressure and this in turn may lead to a reduction in wetting power of the liquid film. This, combined with the low liquid rates experienced in vacuum distillation, could give poor wetting in a packed vacuum distillation column resulting in a lower liquid/vapour contact area and hence lower efficiency.

This effect may explain the anomalous results obtained by Peters and Cannon (89) who found that for the same system on Cannon protruded and McMahon packings as on Berl saddles
found an increase in efficiency for the former and no effect for the latter. These results may indicate that saddles are more prone to incomplete wetting than either the protruded or McMahon packings.

Zuiderweg and Harmon (130) also found that the effect of concentration on surface tension gradients is greater than that of temperature. Liquid arriving at an interface from the bulk can be expected to have a lower surface tension than that which it replaces. Such local concentration changes will, therefore, introduce unbalanced surface tension forces which will lead to convection currents reinforcing the normal surface renewal mechanism and thus increase the mass transfer co-efficient (21).

2.4.6. Composition

In a given system the composition of a mixture, either in the vapour or liquid phase, is dependent upon temperature, which in turn is influenced by pressure.

There is a wealth of evidence in the literature (60) (82) (85) (109) (118) (130) as to the form and extent of the variation of efficiency with composition. Such variation in efficiency is not restricted to column type or system and must,
therefore, be general to the rectification process rather than specific to one set of circumstances.

Sowistowski and Smith (102) found H.T.U. values for the gas phase to be a minimum at the middle of the distillation range and to increase at both low and high concentrations of the more volatile component. Liang and Smith (66) found that efficiency should depend upon composition and should have a maximum value. Further, their results indicated a low efficiency when the mixture is very dilute.

Everitt (37) in introducing his "Composition Function" theory, classifies his explanations of this variation in efficiency into three categories:

1) Variation due to errors in measurement and calculation.

2) Equations of gas phase controlled mass transfer, do not adequately describe the transfer process.

3) There may exist some unsuspected supplementary transfer mechanism.

and in addition to these acknowledged the contribution of the surface tension effects described by Dankverts (21) and Zuiderweg (130).

2.4.7. Summary

Decreasing the pressure of a vacuum
distillation system results in a number of opposing effects on the distillation process, either as a direct result of pressure reduction or through the influence pressure exerts on the other distillation variables. It must be concluded, therefore, that the overall effect of pressure reduction depends upon which of these effects, or combination of effects, is predominant, and this in turn will largely depend upon the chemical systems involved and, in certain respects, upon the equipment employed. This conclusion, although not entirely satisfactory, underlines the complex pattern of the vacuum distillation process and from this it is possible to adopt a more lenient understanding to the contradictory evidence of the literature cited.

2.5. **Individual Phase Resistances**

Information on the contribution of the individual liquid and vapour phase mass transfer co-efficients to the overall resistance to mass transfer in various low pressure fractionating devices is conflicting. Johnstone and Pigford (61), Norman (79) and Jackson and Caeglski (60) found that for distillation in a wetted wall column the resistance to mass transfer was entirely in the vapour phase. Sawistowski et al (101) employed two experimental
techniques in an attempt to isolate the vapour and liquid phase resistances. Their results also indicated that the main resistance to mass transfer lay in the vapour phase.

Conversely, Furnas and Taylor, Deed, Schutz and Drew, and Zuiderweg for distillation in packed and Vigreux columns concluded that the liquid film resistance accounted for a significant proportion of the overall resistance.

In general it may be assumed that due to the presence of ripples in the liquid film the liquid phase resistance in distillation is small, but its effect in the non-rippling region has not been sufficiently explored. Lynn, Straatenier and Kramer, and Eamert and Pigford used short wetted wall columns in an attempt to produce non-rippling films and showed that the experimental values of liquid phase resistance in the non-rippling region agreed fairly closely with the values predicted by the theoretical equations. However, the possibility of error arising in these results through the end effects necessarily experienced in these type of columns cannot be ignored. There must exist a suitable length of column at entrance and exit to enable sufficient time for the flows of the
individual phases to smooth and settle into an organised pattern.

Grimley\textsuperscript{(46)} has shown that for liquid layers falling down smooth vertical walls, at certain liquid rates within the viscous flow regime, ripples appear in the liquid films. He also demonstrated that rippling persists over a range of Reynold's Numbers for the liquid phase, the upper and lower limits of which are dependent upon the liquids physical properties. Grimley\textsuperscript{(46)} correlated his data for the lower end critical limit at which the ripples first appeared by means of a dimensionless equation.

\[
\frac{3}{\mu^{4/3} \sigma^{3/2}} = 0.3 \left( \frac{\text{Re}_L}{\text{Crit}} \right)^{3.0} \quad \text{for} \quad \text{Crit} = 2.1
\]

Portalski\textsuperscript{(95)} in his examination of flow mechanism in wetted wall columns proposes that the three, hitherto, well defined regimes of flow (laminar, turbulent and transitional) are no longer sufficient to fully describe the flow mechanism. An additional implication arises from the wave motion or presence of ripples on the liquid surface at quite low Reynolds Numbers. Portalski divides the mode of flow into
the following regions:–

Laminar - pseudolaminar - transitional - pseudoturbulent

\[ \text{Re}_L = \text{Re}_i \quad \text{Re}_2 = \text{Re}_L = \text{Re}_t \quad \text{Re}_3 \]

turbulent.

Each regime is characterised by upper and lower limits of the Reynolds Number. He presents an equation for the prediction of \( \text{Re}_i \), which is the value of liquid Reynolds Numbers at the inception of ripples, as

\[ \text{Re}_i = \frac{\dot{Q}_i}{\sqrt{2}} \cdot 2.2 \]

The effects of such rippling is to increase the bulk mixing in the liquid phase, and to increase the available interfacial area for mass transfer. Sherba and Hunt\(^{(104)}\) speculated that it was more the presence of turbulence than the increase in interfacial area which accounted for the larger mass transfer rates experienced in such systems\(^{(61)}\)\(^{(112)}\). Fortalski\(^{(92)}\) developed a practical method for calculating this increase in interfacial area, and, in some cases, the enlargement factor \( S_f / S_w \) was as much as 2.5, thereby providing evidence for his disagreement with the explanation of Sherba and Hunt for the increase in mass transfer rates.
As a further attempt to explain the additional mass transfer, supplementary mass transfer mechanisms have been proposed. One such mechanism is usually suggested as "thermal distillation", caused by an imbalance in the heat transfer rates giving rise to additional vaporisation or condensation.

Ruckenstein (100) poses the question: "Are there cases in which an imbalance in heat flux affects the rate of mass transfer between the two phases?"

Dankwerts et al. (21) also stated the possibility of mass transfer being accomplished by virtue of heat transfer in addition to diffusional mass transfer.

Everitt (37) examined the possible mechanisms of thermal distillation in depth and concluded that normal distillation conditions are rarely severe enough for thermal distillation to have a significant effect on distillation efficiency. Only for example, in non-adiabatic column operation, where a considerable amount of evaporation does occur, could an affected efficiency be expected and then mainly in terms of altered flow rates, contact times and possibly interfacial area. However, even under normal distillation conditions, the use of systems where there is a considerable difference in the molal
latent heats of the pure components could result in significant net evaporation or condensation and care should be taken in the selection of test systems in order to further minimise this effect. Everitt concludes his analysis by saying that any variation in efficiency may be satisfactorily explained, not by thermal distillation, but by acknowledging the existence of a significant liquid film resistance. Other workers (21) (83) also minimise the contribution of thermal distillation in the overall mass transfer.

From the preceding discussion it is evident that the existence of a liquid film resistance may be determined by flow conditions within the column itself. Both Pigford and Johnston (61) and Norman (79), who discounted the existence of a liquid film resistance, operated in the turbulent regime, the existence of a liquid film resistance specifically in non-rippling films has not been reported. Accepting the possibility of a liquid film resistance at low liquid flow rates it is possible that in vacuum distillation with its low liquid loads the liquid phase resistance may attain significant proportions. Further, the effect of pressure reduction on vapour velocity, vapour diffusivity, vapour viscosity, and liquid viscosity may further enhance the existence of the
liquid phase resistance as the dominating resistance to mass transfer. Utilising the description of flow regimes given by Fortalaki\textsuperscript{(93)} it should be possible, in the vacuum distillation process, to fully describe the behaviour of the liquid film resistance, especially with regard to column flow conditions.
3.1. **INTRODUCTION**

One of the major problems in the field of vapour-liquid equilibria has been the inadequate testing, for accuracy and consistency, of published isothermal and isobaric data. This situation has developed not through a lack of consistency tests but through a lack of appreciation of the limits of some consistency tests.

All equilibrium results are subject to error, the magnitude of which depends both on the apparatus and experimental techniques employed, and also on the accuracy of the analysis. These errors can be classified into two groups, namely, random errors and systematic errors. Large random errors are easily found as they usually show up as deviations from the smooth curve when the data are plotted as vapour phase composition\( (y) \) versus liquid phase composition\( (x) \). They are even more apparent when, as suggested by Hala et al\(^{(47)} \), the data is plotted as \( (y - x) \) versus \( (x) \).

In certain cases, however, the smoothness of these two curves is not a guarantee
of absolute reliability for the results, since experimental data can be subject to a systematic error which does not show up as a scattering of the measured points. Systematic errors are most usually caused by improper functioning of the apparatus.

The consistency of measured data in such a case can be checked only by comparison with some thermodynamically exact relationship. The basis of many such relationships is the Gibbs-Duhem equation and, for the purposes of this presentation, a consistency test is defined as one based on the Gibbs-Duhem equation designed to test the accuracy of experimental vapour-liquid equilibria. All the thermodynamic equations are simpler to apply to isothermal than to isobaric vapour-liquid equilibria.

3.2. General Methods - The Gibbs-Duhem Equation.

For any liquid phase of a multi-component mixture the Gibbs-Duhem equation, in terms of activity coefficients, applicable to isothermal conditions is

$$\sum x_i \frac{d \ln \gamma_i}{dx} = \frac{V^E}{RT} \frac{dP}{dT} ; \quad dT=0$$  \hspace{1cm} (3.1)

For a binary system this simplifies to

$$x_1 \left( \frac{d \ln \gamma_1}{dx} \right)_T + x_2 \left( \frac{d \ln \gamma_2}{dx} \right)_T = \frac{V^E}{RT} \left( \frac{dP}{dx} \right)_T$$  \hspace{1cm} (3.2).

where $V^E$ is the volume heat of mixing $\gamma_1$ and $\gamma_2$. 
are the liquid phase activity co-efficients which may be calculated using equation 3.3.

\[ \gamma_1 = \frac{\exp(D - V_{\text{HP}})}{p_1^* x_1} \exp(\frac{(D - V_{\text{HP}}) (p - p^*)}{RT}) \exp(\frac{S_{12} y_2^2}{RT}) \]

where \( p^* \) and \( V_L \) are the vapour pressure and molal volume of liquid component 1, \( B \) is the second virial co-efficient of component 1, and \( S_{12} = 2B_{12} - B_{11} - B_{22} \) is the interaction parameter where \( B_{12} \) is the mixed virial co-efficient of components 1 and 2, \( \gamma_2 \) is found by interchanging the suffices. This equation allows for the deviation of vapour phase behaviour from the ideal.

The thermodynamic differential equation relating activity co-efficients to composition under isobaric conditions contain the term \( H^E \frac{dT}{dP} \), where \( H^E \) is the molal integral heat of mixing. For isobaric conditions, the Gibbs-Duhem equation for a binary system becomes

\[ x_1 \left( \frac{d \ln \gamma_1}{dx_1} \right)_p + x_2 \left( \frac{d \ln \gamma_2}{dx_1} \right)_p = \frac{H^E}{RT} \left( \frac{dT}{dx_1} \right)_P \]

Ellis and Bourne (29) pointed out that if the molal volumes of organic compounds do not differ by a factor of 2 to 4, the volume change of mixing is usually less than 0.5 cm\(^3\)/g.mole at
temperatures below the normal boiling point. Thus the right hand side of equation 3.2 does not exceed 0.002% of either term on the left hand side. Ibl and Dodge (58), for example, calculated a value of 0.0007% for equimolal mixtures of acetone and carbon disulphide at 35°C. Since it appears that the volume of mixing term is negligible compared with the left hand side of equation 3.2 it is usually neglected (29) (32), and equation 3.2 reduces to the common isobaric - isothermal Gibbs-Duhem equation given by

\[ x_1 \left( \frac{d \ln \gamma_1}{dx_1} \right) \tau, P + x_2 \left( \frac{d \ln \gamma_2}{dx_1} \right) \tau, P = 0 \]

3.5

Simplification of equation 3.2 was possible because of the negligible value of the volume change of mixing, unfortunately, such a simplification is not possible for equation 3.4, where the heat of mixing term on the right hand side is of significant proportions. Ibl and Dodge (58) found that for the system acetone/carbon disulphide the right hand side of equation 3.4 was 13%, 0.7% and 7% of either term on the left hand side when \( x_1 \) was 0.1, 0.5 and 0.9. This indicates a significant difference between equation 3.4 and the isobaric-isothermal Gibbs-Duhem equation (equation 3.5.) for
which the right hand side is zero, and to the
isothermal form (equation 3.2) for which the right
hand side is extremely small.

The right hand side of equation 3.4 becomes significant in any isobaric system where
there is a wide difference in the boiling points of
the components(29). Ibl and Dodge(53), and
Ellis and Spurr(54) have shown that in isobaric
systems where the boiling range exceeds 15-20°C
the right hand side of equation 3.4. is significant.
In isobaric systems where the boiling range does not
exceed 10-15°C the right hand side of equation 3.4.
is negligibly small and may be neglected(19) (23)
(95) (114).

Usually heat of mixing data at the
required temperature and composition are not available.
Such data can be calculated approximately from two
sets of isobaric equilibria data, but the accuracy
of this method is not known(32). The application
of equation 3.4. for the testing of consistency in
wide boiling point range isobaric mixtures is
therefore limited.

3.3. Specific Methods - Isothermal Data

Herington(49), Coulson, Males
and Herington(19), and Redlich and Kister(96) (97)
derived an equation which depends upon, but is more convenient than, the Gibbs-Duhem equation. For a miscible binary system at constant temperature

\[ \int_{x_1=0}^{x_1=1} \log \frac{\gamma_1}{\gamma_2} \, dx = \frac{1}{2.3026} \]

\[ \int_{p=p_1^*}^{p=p_2^*} V^g \, dp \quad \text{equation 3.6.} \]

The right hand side of equation 3.6 is frequently less than 0.001 as explained in an earlier argument, and can be neglected except in very accurate work \(^{(29)}\), therefore, equation 3.6 can be reduced to:

\[ \int_{x_1=0}^{x_1=1} \log \frac{\gamma_1}{\gamma_2} \, dx = 0 \quad \text{equation 3.7.} \]

If, therefore, the areas above and below the line \(\log \frac{\gamma_1}{\gamma_2} = 0\) are equal the binary data are thermodynamically consistent. An accurate method of numerical or graphical intergration should be used in the application of equation 3.6 and equation 3.7.

The largest contributions to the areas above and below the line \(\log \frac{\gamma_1}{\gamma_2} = 0\) (Fig. 3) arise from the most dilute regions, where experimental accuracy is lowest, this is the major fault of this important method \(^{(29)}\). A reliable curve can, however, be drawn when a substantial number of data points are
available, preferably at approximately equal intervals of concentration. If $1.05 > \frac{A}{B} > 0.95$, the data can be considered consistent \cite{12}.

Idler et al.\cite{1} derived an isothermal consistency test based on equilibrium $K$ values and compressibility factors.

\[ \int_{x_1=0}^{x_1=1} x_1 \ln K + \int_{x_1=0}^{x_1=1} x_2 \ln K_2 = \]

\[ \int_0^1 \left[ \frac{Z^L + Z_1 V}{P x_1} + \frac{1}{K_1} \left( \frac{1}{x_1} + \frac{1}{K_2} \right) \frac{z_v}{K} \right] d \ln P = 3.8 \]

where $K_1 = \frac{y_1}{x_1}$, $Z^L$ and $Z^V$ are the compressibility factors of the liquid and vapour phases, and $Z_1^V$ is the partial compressibility factor of pure component 1 in the vapour phase.

Again, the isothermal equation 3.8 is graphically integrated to test the consistency of the experimental data.

The area consistency tests here described apply over the whole composition range and are not capable of determining whether any point or composition region is consistent. Because the log
of activity co-efficients ratios are used equations 3.6 and 3.7. are less sensitive to temperature and pressure measurement errors and to vapour phase deviations from the ideal.

Another class of consistency tests for isothermal data are the slope consistency tests. The experimental vapour/liquid equilibrium results are tested for consistency by taking tangents at specific points on a plot of property, e.g. log activity co-efficient, temperature, etc., versus composition and substituting the results obtained into the Gibbs-Duhem equation (equation 3.2).

Van Ness derived an isothermal consistency test from the definition of excess free energy, $\Delta_{p}^{E}$, and the Gibbs-Duhem equation.

$$Q = \frac{\Delta_{p}^{E}}{RT} = x_{1} \ln \gamma_{1} + x_{2} \ln \gamma_{2} \quad \text{3.9}$$

Differentiating equation 3.9 and substituting equation 3.2.

$$\frac{dQ}{dx_{1}} = \ln \gamma_{1} - \ln \gamma_{2} + \frac{\Delta_{p}^{E}}{RT} \frac{dF}{dx_{1}} \quad \text{3.10}$$

Tangents are drawn to the curve of $Q$ versus $x_{1}$, and are extrapolated to the ends of the composition range to give values $Q_{0}$ and $Q_{1}$ at $x_{1} = 0$. 


and \( x = 1 \) respectively.

\[
Q_0 = \ln y_2 - x_1 \lambda \quad \text{3.11}
\]

\[
Q_1 = \ln y_1 + x_2 \lambda \quad \text{3.12}
\]

where \( \lambda = \frac{V^E}{RT} \frac{dP}{dx_1} \)

Differentiating equations 3.11 and 3.12 gives

\[
x_1 \frac{dQ_1}{dx_1} = x_2 \frac{dQ_0}{dx_1} \quad \text{3.13}
\]

Values of \( Q_0 \) and \( Q_1 \) are plotted against liquid composition \( x_1 \), tangents are drawn at specific points and the values substituted into equation 3.13 to test the data for consistency. Van Ness, realising that tangents to the \( Q \) versus liquid composition curve were difficult to draw, has proposed an alternative method for evaluating \( Q_0 \) and \( Q_1 \) from a plot of \( Q_0 / x_1 x_2 \) versus \( x_1 \) (Fig. 4).

Van Ness agreed that his test was a point consistency test. This is not strictly true in that the tangents are measured from smoothed curves drawn through experimental data. In the use of the slope consistency tests errors thus arise due to smoothing of the experimental results and due to the accuracy with which tangents can be drawn at specific points.
Herington (52) integrated equation 3.2., assuming the \( V^F \) term to be negligible, between the limits \( x_1 = x_1^{11} \) and \( x_1 = x_1^{1} \), thus

\[
\frac{1}{x_1} \int_{x_1 = x_1^{11}}^{x_1^{1}} x_1 \, d \ln \gamma_1 + \frac{1}{x_1} \int_{x_1 = x_1^{11}}^{x_1^{1}} x_2 \, d \ln \gamma_2 = 0 \quad 3.14
\]

It is proposed that integrals should be plotted against composition. For example, the integrals in equation 4.14 may be plotted against \( x_1 \) with \( x_1^{11} = 0 \) (Fig. 5), giving curves \( I_1 \) and \( \int_{x_1 = 0}^{x_1^{1}} y_1 \int_{x_1 = 0}^{x_2} d \ln \gamma_2 \).

Clearly if data satisfies equation 4.14 the sum of the integrals is zero and the curves are therefore symmetrical about the line \( \int x \, d \ln = 0 \).

Such a treatment does not make full use of the symmetry proposed by equation 4.2 and Herington proposed the use of the integrals.

\[
I_1 = \int_{x_1 = 0}^{x_1^{1}} x_1 \, d \ln \gamma_1 = \frac{1}{x_1} \int_{x_1 = 0}^{x_1^{1}} x_1 \, d \ln \gamma_1 \quad 3.15
\]

and

\[
I_2 = \int_{x_1 = 1}^{x_1^{1}} x_2 \, d \ln \gamma_2 = \frac{1}{x_1} \int_{x_1 = 1}^{x_1^{1}} x_2 \, d \ln \gamma_2 \quad 3.16
\]
The curves of these integrals are shown in Fig. 5. The sum $I_1 + I_2$ is constant as shown by the dotted line. The slopes of the $I_1$ and $I_2$ curves are equal but of opposite sign at every value of $x_1$. The $I_1$ curve is the mirror image of the $I_2$ curve about a line parallel to the $x_1$ axis, through the intersection of the $I_1$ and $I_2$ curves. The slope of the curves tend to zero when $x_1$ tends to zero or unity.

To apply the symmetrical area test for consistency, the quantities $I_1$ and $I_2$ are calculated from curves drawn through the experimentally observed values of $\ln|\phi|$ and $x_1$, and $I_1$ and $I_2$ and their sum are plotted against $x_1$. If, and only if, the data are consistent over a range of compositions will $I_1 + I_2$ be constant over that range. If the recorded vapour compositions are in error for part of the composition range, horizontal portions of the $I_1 + I_2$ curve will be connected by a smooth step for the region where the error exists.

A symmetrical area test can be applied to a restricted composition range (51). Integrals are constructed for the range available, and the properties described for $I_1$ and $I_2$ above are applied.
curves obtained from experimental results. However, it is possible to pinpoint errors to within a concentration range and can be used to revise values of \( \ln \) versus \( x \).

### 3.4. Specific Methods - Isobaric Data

As was stated earlier consistency testing is simpler for isothermal data than for isobaric data. This is because whereas the \( V^2 dP \) term in isothermal relationships (equation 3.2.) is insignificant, the \( H^2 dT \) term in isobaric relationships (equation 3.4.) is not.

Isobaric consistency tests tend to be extensions of isothermal tests differing from the latter by the need to account for the \( H^2 dT \) term.

Herington has proposed an empirical solution to the isobaric equation

\[
dP = 0 \int_0^1 \frac{1}{P^2} \ln \frac{y_1}{y_2} dx_1 - \int_{T_2}^{T_1} \left(\frac{H^2}{RT^2}\right)_P dT = 0 \quad 3.17
\]

As can be appreciated from a comparison with equation 3.7., this is an extension into the isobaric case from the isothermal case previously described (9)(10)(11)(12).

Herington's relationship is useful when heat of mixing data is unavailable. It is based on the assumptions that \( \frac{H^2}{\Delta F^2} \) is small and that equation 3.9.
is applicable to isobaric systems. Isobaric equilibrium data are considered consistent if:

\[ \frac{100}{T_{\text{min}}} < \frac{150}{T_{\text{min}}} \quad \text{3.18} \]

Where \( I = \int_{0}^{1} \ln \frac{y_1}{y_2} \, dx_1 = A - B \) (Fig. 3.1.)

\[ = \int_{0}^{1} \ln \frac{y_1}{y_2} \, dx_1 = A + B \]

\[ \Phi = T_{\text{max}} - T_{\text{min}} \]

\( T = \) Boiling point of component described by subscript.

Equilibrium results are also considered to be accurate if

\[ \frac{100}{T_{\text{min}}} - \frac{150}{T_{\text{min}}} < 10 \quad \text{3.19} \]

Areas \( A \) and \( B \) (Fig. 3) are determined in the same way as described earlier for equation 3.6.

Edminster and Robinson \( (26) \) have commented that the accuracy of Herington’s equation \( (50) \) is unknown.

Alder et al. \( (1) \) extended their isothermal consistency test to the isobaric case by assuming that the vapour phase obeys the ideal solution law. Edminster and Robinson \( (26) \) extended equation 3.8.
to isobaric conditions making allowance for vapour
phase deviations.

\[ T_{x_1 = 0} = \int_{x_1 = 0}^{1} x_1 \ln k_{1x_1} + \int_{x_1 = 0}^{1} x_2 \ln \delta_{2x_2} \]  

\[ = - \int_{T_{x_1 = 0}}^{T_{x_1 = 1}} \frac{H}{RT^2} dT \]  

where \( \phi_1 = \frac{f_{ov_1}}{p} \), \( \phi_2 = \frac{f_{ov_2}}{p} \), \( \chi_{ov_1} \) and \( \chi_{ov_2} \) are the
fugacities of pure components 1 and 2 at the temperature
and pressure of the system and \( \chi_{ov_1} \) and \( \chi_{ov_2} \) are the
activity co-efficients of the vapour phase.

Both equations 3.17 and 3.20 require
graphical integrations in their solutions. These
two tests are, therefore, examples of area consistency
tests available for isobaric data.

Van Ness's slope consistency test
for isothermal data (117) is also extended to cover
isobaric data.

\[ \text{At } dP = 0 \quad \frac{d\phi}{dx_1} = \ln \chi_1 - \ln \chi_2 - \frac{\delta^2}{RT} \frac{dT}{dx_1} \]  

\[ \text{At } dP = 0 \quad \frac{\delta^2}{RT} \frac{dT}{dx_1} \]  

\[ = - \int_{T_{x_1 = 0}}^{T_{x_1 = 1}} \frac{H}{RT^2} dT \]  

\[ \text{where } \phi_1 = \frac{f_{ov_1}}{p} \), \( \phi_2 = \frac{f_{ov_2}}{p} \), \( \chi_{ov_1} \) and \( \chi_{ov_2} \) are the
fugacities of pure components 1 and 2 at the temperature
and pressure of the system and \( \chi_{ov_1} \) and \( \chi_{ov_2} \) are the
activity co-efficients of the vapour phase.

Both equations 3.17 and 3.20 require
graphical integrations in their solutions. These
two tests are, therefore, examples of area consistency
tests available for isobaric data.

Van Ness's slope consistency test
for isothermal data (117) is also extended to cover
isobaric data.
According to previous equations 3.11 and 3.12

\[ Q_0 = \ln \gamma_2 - x_1 \alpha \]

and

\[ Q_1 = \ln \gamma_1 + x_2 \alpha \]

However, in the isobaric case, \[ \frac{\Delta H}{RT^2} = \frac{H^E}{RT^2} \frac{dT}{dx} \]

This testing procedure requires knowledge of the heat of mixing as a function of temperature and composition. In practice, this means the determination of the heat of mixing as a function of composition at two temperatures. Since the heat of mixing at a given composition is almost always essentially linear with temperature, interpolation for the effect of temperature on heat of mixing is possible. A correlation for the effect of composition on heat of mixing utilising a two constant type of equation has been proposed by Schnaible, Van Ness and Smith (105).

In common with the previous tests, Nortington's symmetrical area test (51) for isothermal data has also been extended for use on isobaric data.

Integrating equation 3.4., given that the boiling temperatures of components 1 and 2 are \( T_1 \) and \( T_2 \), respectively, and that of a mixture of composition \( x_1 \) is \( T_1 \) then
\[ \ln \gamma_1 \int_{x_1}^{1} x_1^1 \frac{d \ln \gamma_1}{x_1} = I_{1p} \]

\[ \ln \gamma_2 \int_{x_2}^{1} x_2^1 \frac{d \ln \gamma_2}{x_2} = I_{2p} \]

\[ \int_{T_1}^{T} \left( \frac{H^E}{RT^2} \right) dT = I_H \]

and further \( I_{1p} + I_{2p} + I_H = c_p \)

The recommended procedure is, therefore, to plot and examine the curves of \( I_{1p} \), \( I_{2p} \) and \( I_{1p} + I_{2p} \) against \( x_1 \). If the data are consistent these functions will possess all the appropriate symmetry properties discussed for \( I_1 \) and \( I_2 \). If the value of \( I_{1p} \) and \( I_{2p} \) appears to vary with composition an approximate calculation of \( H^E \) should be made by use of the equation 3.26.

\[ H^E = -RT^2 \left[ \frac{d(I_{1p} = I_{2p})}{dx_1} \right] \left( \frac{dT}{dx_1} \right) \]

In practice, if the value of \( H^E \) calculated from the isobaric data is excessively large then the data are suspect.

3.5. Isobaric Consistency Tests Employed

There are a great many consistency tests
for isobaric data in existence, it has only been possible to quote a few. It has been possible, however, to emphasise the drawbacks of each class of consistency test and from these appreciate the limitations of each type.

For the purposes of examining the isobaric equilibrium data available for the binary systems utilised in this research it was decided to employ one test from each of the three broadly defined classes described. These tests are:

1. Area Consistency Test - Herington
2. Slope Consistency Test - Van Ness
3. Joint or Restricted Area Test - Herington

Each test was applied to each set of equilibrium data, unless there was sufficient evidence to suggest that one or more of the selected tests had been employed by the author of the data and the results found to be conclusive.
4.1. Introduction

Kala \(^{(47)}\) presents a list of the binary systems for which vapour/liquid equilibria data has been published up to May 1965. This list has been further supplemented by Leiderweg et al \(^{(129)}\) who drew up a list of recommended test mixtures and standardised the methods of inspection of the equilibrium data, physical constants, and other required information. Williams \(^{(124)}\) had much earlier summarized the requirements which should be met by such test mixtures as follows:–

a) The boiling points of the components should be close together and should be in the range 100-200°C at the operating pressure.

b) The mixtures should not deviate too much from ideality.

c) Any mixture of the components should be accurately analyzable preferably by a simple and rapid method which requires only a small sample.

d) The components should remain liquid at temperatures well below the operating temperature, preferably even at room temperature.
e) The chemicals should be thermally and chemically stable at the operating temperature.

f) The components should be readily available and easy to purify.

Apart from the above requirements relating to the nature of the components to be selected, Zuiderweg makes the further demand that the vapour/liquid equilibrium data should be accessible to control on thermodynamic consistency. In general, the technique of determination of vapour/liquid equilibria is rather difficult, and this may be said particularly of the measurements of low pressure equilibria. Thermodynamic control is here a powerful expedient in estimating possible errors made during the determination. It is, therefore, accepted that all equilibrium data must be thermodynamically consistent within the context of the tests selected in Chapter 3.

4.2. Selection of Test Mixtures.

Five binary organic systems were chosen. They were selected not only because they satisfied the requirements of Williams and of Zuiderweg, but also by their careful selections they have enhanced the scope and nature of the research. Further, three of the five systems are suggested.
standard mixtures for the testing of distillation columns.

The five binary systems are:

a) Benzene/Toluene *

b) n-Heptane/Toluene

c) Chlorobenzene/EthylBenzene *

d) n-Decane/trans Dekalin

e) trans Dekalin/cis Dekalin *

* Suggested standard mixtures for the testing of distillation columns (129).

4.2.1. Benzene/Toluene

This system is described by Zuidervog (129) as an "ideal system". Employing the nomenclature of Zuidervog and Harnens (130) it is described as a surface tension negative system.

Equilibrium data at 760 Torr is readily available, Hala (137) lists eighteen references for such data whilst Zuidervog (129) also presents equilibrium data at this pressure. The components themselves were readily available in the "Analyler" grade of purity and subsequently did not require any further purification prior to use.

The system was selected for use at 760 Torr as an example of the surface tension negative class of system. The equilibrium data of
Zuiderweg (129) was accepted as consistent after application of the thermodynamic consistency tests described earlier.

4.2.2. n-Heptane/Toluene

Employing the nomenclature of Zuiderweg and Harrns (130), this system is described as surface tension positive. Sala (147) lists sixteen sources of equilibria data at 760 Torr. The components are readily available as purified grades.

The system was selected for use at 760 Torr as an example of the surface tension positive class of systems. Several sources of equilibrium data were tested (41)(57)(99)(107)(125) and compared. The data of Yerasanis (125) and that of Rose and Williams (99) compared excellently over the thermodynamic consistency tests, the data of Yerasanis (125) was selected.

4.2.3. Chlorobenzene/Ethylbenzene

A surface tension negative system that has been widely used in vacuum distillation studies (48)(39)(127), chlorobenzene/ethylbenzene systems are described as ideal (129). Equilibrium data at several pressures are available, Higgins (155) presents data at 760 and 300 Torr, Hawkins and Brent (48)
at 760, 300 and 50 Torr, and Suiderweg (129) at
760, 300 and 50 Torr.

The components are available only
in the "commercial" grade of purity, the major
impurities being ortho -, meta -, and para xylenes
(55). Both components were purified by fractional
distillation in a 3 foot x 1\(\frac{1}{2}\) inch diameter column
packed with 1/8 inch Raschig Rings, at a low boil
up rate. Fractions of both components were
collected and only those fractions of refractive
index, as determined using a Zeiss Abbe refractometer
at 25°C, \(\pm 0.0001\) of a constant value of 1.4931 for
ethylbenzene (55) and 1.5212 for chlorobenzene (55)
were retained. As a further check on purity,
samples of both components were analysed in a
GLC chromatograph before and after distillation.
The results showed the presence of the xylene
isomers in the crude compounds, which, although
almost impossible to eliminate completely, were
absent in the traces obtained for the distilled
components. Details of the GLC columns used here
and in the analysis of all other systems are given
in Table 1.

The system was selected for use at
760, 300 and 50 Torr. In testing the data of both
Higgins (55) and Zuidervog et al. (126) considered considerable difficulty was experienced in reaching a satisfactory conclusion. It can be seen from Fig. 6 that the log \( \gamma_1/\gamma_2 \) function can be represented by a straight line running parallel to the abscissa at a distance of approximately log \( \gamma_1/\gamma_2 = 0.02 \). This indicates that systematic errors are present in the determinations, and suggests that the chlorobenzene/ethylbenzene system behaves ideally. Guided by the opinions of Ellis and Bourne (25), and Higgins (55) it was concluded that the apparent failure of the tests to prove consistency was due to the closeness of the system to ideality, which tended to magnify the small errors beyond reasonable proportions.

The data of Zuidervog (129) was eventually selected for application to this system.

4.2.4. n-Decane/trans-Decalin

This is a surface tension positive system which has also been previously employed to test the efficiency of fractionating columns at reduced pressures (110)(128). Equilibrium data at various pressures are available (47), Zuidervog (127) presents data at 760 Torr, Struck and Kinney (110) at 50, 20 and 10 Torr, and Finske et al. (58) at 760 Torr. Both compounds are available in the
purified form, however, the trans dicalen was analysed by GLC (See Section 4.2.5.) for traces of the cis isomer.

As the system was required for use in comparison with the surface tension negative system chlorobenzene/ethylbenzene it was unfortunate that no published equilibrium data was available at a pressure of 300 Torr. This data was, therefore, obtained by calculation and is presented in Table 2. The data of Struck and Kinney (110) at 50 Torr was tested for thermodynamic consistency with a similar result as for chlorobenzene/ethylbenzene. From Fig. 6, it can be seen that the $\log \gamma_1/\gamma_2$ function is represented by a straight line at $\log \gamma_1/\gamma_2 = 0.02$. By a similar argument to that applied earlier the data was accepted and used. The Van Ness slope test for consistency was not applied to data for this system because of an absence of reliable heat of mixing data.

4.2.5. trans Dekalin/cis Dekalin

An "ideal" surface tension neutral system (129) that is especially suited for studying the performance of vacuum distillation columns. The lowest pressure that is attainable with normal cooling water in the condenser is about 5 Torr.
Zuiderweg (129) presents equilibrium data at 100, 50, 10 and 5 Torr, which is acceptable for reasons of thermodynamic consistency. Again the Van Ness test was not applied because of a lack of heat of mixing data.

Both components are available in the purified form, however, both components were tested for impurities using gas chromatography methods, the details of which are in Table 1. The major impurity in either component is the co-isomer, although no trace was evident in either of the pure samples.

4.3. Treatment Of Physical Data

The various equilibrium data are the primary physical data required in calculation. Such data has been obtained from references quoted and tested for thermodynamic consistency by application, where possible, of the tests selected in Chapter 3. The sources of other pertinent data, and their treatment is as follows.

4.3.1. Liquid and Vapour Densities

Data on liquid densities, for all the binaries used, are available in the literature (59)(87)(129). However, as an aid in calculation, it was desirable that the pure component density be
expressed as a function of temperature. Where such expressions were not available, they were constructed from all the available density data for such a compound, tested and found reliable enough, over the temperature range required, to enable use in calculation. Details of the method by which such expressions were obtained are given in Appendix A.

The density of liquid mixtures were calculated on a mole fraction basis.

The densities of vapours were determined by assuming that the perfect gas laws applied.

4.3.2. Liquid and Vapour Viscosities.

Data on liquid viscosities, for all the binaries used, are available in the literature\(^{(2)}(36)(59)(115)(116)(129)\). In the absence of information on this subject\(^{(98)}\), the viscosity of a liquid mixture was assumed to be the molal average of the values of the pure components. From experimental determinations and by analysis of published data on the molal average viscosity for light hydrocarbons\(^{(24)}\), compared to actual viscosity measurements it was calculated that the average error introduced in using molal averages is between...
The viscosities of pure vapours at their dew points, where not directly available from the literature, were determined by the method described by Bromley and Wilke (11) and the viscosities of vapour mixtures were estimated by the equations derived by Wilke (122).

### 4.3.3 Liquid and Vapour Diffusivities

The liquid diffusivities were calculated in two stages. Firstly, the method of Wilke and Chang (123) was used to determine the tracer diffusion coefficients. Their correlation is suitable for use only at low concentrations and hence is ideally suited for use in calculating these values. The trace diffusion coefficients are then used in the theoretical equation recommended by Reid and Sherwood (98) to calculate the overall liquid diffusion coefficients.

Vapour diffusivities were calculated using the empirical relationship derived by Gilliland (43). This method yielded more consistent results when a comparison was made with published diffusivities (128) for some of the mixtures used than did other empirical relationships available.

### 4.3.4 Surface Tension

Values for the surface tension of
all the pure components employed is available in the literature\(^{(59)(87)(129)}\). The equation

\[
\frac{\sigma_1}{\sigma_2} = \left(\frac{1 - T_{r1}}{1 - T_{r2}}\right)^{1.22}
\]

where \(\sigma_1\) surface tension at reduced temperature \(T_{r1}\), \(\sigma_2\) surface tension at reduced temperature \(T_{r2}\) was used to calculate the variation of surface tension with temperature\(^{(98)}\).

The surface tension of liquid mixtures were calculated using the theory devised by Eckert and Prausnitz\(^{(25)}\). The relationship was used in its simplified form which gave an acceptable average error of 4 to 6\(^{(25)}\).

4.3.5. Vapour Pressure

For all the pure components except \(n\)-Decane vapour pressure/temperature relationships of the Antoine type are available in the literature\(^{(129)}\). A similar relationship for \(n\)-Decane was constructed using vapour pressure/temperature data quoted by Zuiderweg\(^{(128)}\).

Where practical all physical properties were correlated as functions of temperature or composition to ease subsequent calculations. As stated earlier the method by which such correlations were obtained is described in Appendix A.
CHAPTER 5

APPARATUS AND EXPERIMENTAL TECHNIQUES

5.1. Introduction

In this study the performance of an internally finned glass tube has been investigated under low pressure distillation conditions. The presence of fins inside the glass tube considerably increased the interfacial area per unit volume (by about 250 per cent) compared to its value for a conventional wetted wall column, without altering the hydrodynamics of liquid films produced. In order to analyse apparatus performance, the effect of variables on distillation efficiency, and the behaviour of vertical liquid films and its effect on the relative resistance to mass transfer of individual phases, five different binary test mixtures were investigated at operating pressures ranging from 760 to 50 Torr. To investigate interphase transfer two distinct, yet inter-related, techniques were employed.

5.2. Apparatus – 'Finpak' Column

Basically the 'Finpak' column is a wetted wall column wherein the available surface area for liquid flow has been supplemented by the inclusion of a longitudinally finned central core.
To fully describe the apparatus it is convenient to subdivide it into four sections:

a) Stillpot and column base

b) Column centre and fin assembly.

c) Column head and condenser assembly.

d) Control systems - vacuum and heating.

5.2.1. Stillpot and Column Base

The stillpot was simply a 2 litre round bottomed flask with a short neck terminating in a standard 555/44 socket. The stillpot was heated using an Electrothermal heating mantle of 2 litre capacity and 1 Kw rating. As suggested by Krall (63) the stillpot charge did not exceed two-thirds of the volume of the flask for distillation at atmospheric pressure and not more than half in vacuum distillation conditions.

At atmospheric pressure, and at 300 Torr, "bumping" was prevented using 5 mm diameter glass beads, however, at pressures below 300 Torr this proved insufficient. An immersion heater was constructed through the walls of the flask. The heater consisted of 3 metres of 24 s.w.g. Nichrome resistance wire, coiled with an overall diameter of 3/8 inch brazed at either end to 0.014 inches diameter platinum wire which, in turn, was sealed,
using graded glass seals, into the walls of the flask. A sufficient length of platinum wire was exposed for connection to a variable voltage source. This immersion heater was not capable of supplying all the heating requirements of the stillpot, it did, however, prevent "bumping" at lower pressures by providing a surface for nucleate boiling just below the liquid surface. The flask was also partially filled with 1\frac{1}{2} mm diameter glass balls, instead of the 5 mm diameter beads previously mentioned, to help prevent excessive liquid superheating.

The stillpot was connected to a glass extension tube, the flask end of which terminated in a standard 55/44 cone, the other end in a standard 355/44 socket. When employing the "pressure drop" technique, (see Section 5.3.1.), this extension piece was merely a well lagged interconnecting tube. However, when using the "inert gas injection" technique (Section 5.3.2.), this section provided the inlet tube for the introduction of nitrogen into the base of the stillpot. This section was then packed to a length of 20 cm with gauge packing to ensure complete saturation of the inert gas and to prevent
excessive liquid entrainment.

The column base connected to these interconnecting tubes via a 1.55/4\(\frac{1}{4}\) cone. Liquid flows received into the column base from the fin assembly and column walls were collected separately by means of the arrangement detailed in Fig. 8, the flow of each stream was measured independently, samples independently, mixed and returned directly to the bottom of the stillpot. Vapour was introduced, from the stillpot, into the column base via the annular space shown in Fig. 8.

A pressure tapping was provided at the column base to facilitate the measurement of column pressure drop. The temperature of vapour into the column was also measured at this point using a single junction copper/constantan thermocouple.

An exterior view of the stillpot and column base assembly is provided in Plate 2.

5.2.2 Central Column and Fin Assembly

The central column assembly is indicated in the overall apparatus views of Plate 1 and Fig. 7.

The wetted wall section of the column consisted of a 23 mm internal diameter precision bore glass tube 1.095 metres in length.
The bottom end was fused directly to the column base assembly, the top end passed through a vacuum seal assembly (Section 5.2.3.) and the end ground flat at right angles to the tube length.

The fins were manufactured from three sheets of stainless steel, 1 mm thick, 22 mm wide and 1.09 metres in length. Two of the sheets were carefully bent at an angle of 60° along their centre line over their entire length and were spot welded on either side, at the centre line, of the flat third piece in such a way that they were equally spaced around the periphery of the tube. At their base the fins were tapered into a conical brass end piece to direct liquid flow into the assembly for independent measurement and sampling described earlier. At the top they were sealed into a brass distributor assembly, which suspended the fins centrally within the column whilst resting on the ground top surface of the precision bore tube. This distributor was designed by optimization through trial and error techniques, so that the amount of liquid reflux returned to the column was subdivided into a wall stream and fin stream in quantities proportional to the interfacial areas of the two assemblies. The principle difficulty encountered
with the distribution device was to achieve a seal between the distributor and the top of the glass tube and hence prevent seepage of liquid directly onto the column wall. This was eventually overcome by accurate machining of the brass distributor insert into the column section and by the use of a Viton rubber 'O' ring seal, the weight of the fin assembly being sufficient to compress the 'O' ring between the distributor and the ground glass to provide an adequate seal.

The vapour ascending from the reboiler should in general reach the condenser without the introduction or removal of heat. When the usual insulating materials are used this requirement can be satisfied sufficiently closely up to a temperature of about 100°C only (63), above this temperature heat must be supplied to counter balance that lost by convection and radiation to the surroundings. The stillpot and all parts of the apparatus up to the condenser were therefore insulated.

Of the various forms of insulation available all but one were discounted as insufficient for the required duty. The use of low heat conductivity materials to provide a column coating
was dismissed as these are only really effective up to 80°C (68). Vacuum jacketing, as had been previously used on an earlier model of this column (15), was not employed primarily through a consideration of its constructional difficulties, but also because of the difficulty in attaining and maintaining a sufficiently low vacuum to provide adequate insulation. The use of heating jackets through which a heating medium is passed (56)(94) was not considered because of the inherent danger of isothermal conditions being prevailed upon the column (63)(90). A satisfactory method for insulating columns is to use electric heat compensating jackets (63). This method has been widely employed (61)(66) and is particularly necessary in vacuum distillation, where the heat capacity of the vapour is small. The fact that a temperature gradient exists in a column must be taken into account. For this reason the insulation of the Finpak column was divided into three separately controlled regions over the column length, each of which could be adjusted to the corresponding interior temperature of the column. The column insulation was made up as follows:

1) Single point copper/constantan thermocouples
attached to the column wall every 300 m.n.

2) One layer of tightly wound asbestos rope
    1/8 inch wound overall.

3) One layer of tightly wound overlapping
    asbestos cloth, wound overall.

4) One layer of tightly wound asbestos rope
    1/8 inch diameter, wound overall.

5) One layer of tightly wound asbestos woven
    tape, wound overall.

6) One layer of tightly wound asbestos cloth,
    wound overall.

7) Single point copper/constantan thermocouples
    positioned to correspond to those on the
    column.

8) One layer of asbestos cloth, wound overall.

9) Three heating tapes, each wound over a length
    of 350 to 370 mm and each independently controlled.

10) One layer of tightly wound asbestos woven tape,
    wound overall.

11) One layer of tightly wound asbestos rope,
    wound overall.

12) One layer of tightly wound asbestos cloth,
    wound overall.

As stated each heating tape was
    independently controlled and so adjusted as to give
a temperature difference of 1°C across the intervening lagging between corresponding thermocouples.

Prior to lagging it was essential to determine that the central column section was vertical, to ensure correct liquid distribution and flow. This was achieved firstly by the use of travelling microscopes, and secondly by visual observation of water flow down column walls and fins.

5.2.3. Column Head and Condenser Assembly.

An overall view of this assembly is given in Plate 3. Figure 9 is an exploded diagram of the column head assembly and vacuum seal system.

The vacuum seal assembly consisted of four parts: -

a) Precision bore glass tubing constituting the actual column
b) Mild steel flange assembly.
c) Mild steel collar.
d) Viton rubber 'O' ring.

The precision bore tubing was found to be precise only with regards to its internal diameter, the external diameter varied to an extent which would not have permitted the use of an 'O' ring
assembly. It was, therefore, necessary to "lap" the top 100 mm of glass tube to give an external diameter of 32.0 ± 0.2 mm.

The mild steel flange assembly was machined to provide not only part of the vacuum seal arrangement but also a base for the condenser assembly, and a support for the column as a whole. The central area of this flange was part of the vacuum seal. Essentially it was merely a hole through which the precision bore tubing passed, but the diameter of this hole was critical; too small and the differences in thermal expansion of glass and metal meant a possibility of the flange crushing the tube, too large and the vacuum seal would be rendered ineffective. It was found that a clearance of 0.15 m.m. was sufficient to overcome both these problems. The lower section of this central area of the flange was internally threaded to correspond to the threaded portion of the moveable collar. In the interests of achieving an effective seal these threads had to be as accurate as possible, parallel to the axis of the precision bore glass tube and normal to the top face of the flange assembly. Plate 4 presents a top view of this flange assembly.

The liquid distributor can be seen at the centre
and the two concentric grooves provided locations for, in the inner groove, a glass smoothing collar which directed vapour into the condenser and smoothed the liquid flow returning from the condenser to prevent surges over the liquid distributor. The outer wider groove sealed the viton rubber sealing gasket which sealed between the flange and the spacer assembly complete with reflux heater.

The moveable mild steel collar presented a similar problem as did the flange with regards to the size of hole sufficient to take the precision bore tube and was solved by again leaving a 0.15 mm gap between steel and glass. The top surface and edge of the hole was chamfered (Detail Fig. 9) to provide the necessary retention and compression of the 'O' ring seal. To achieve a vacuum tight seal the moveable collar was screwed into the flange assembly trapping and compressing the 'O' ring between the chamfered shoulders of the collar, the walls of the precision bore tubing and the lower face of the flange assembly. This seal was capable of maintaining a leak free joint between these three surfaces down to 0.5 Torr.

Between the flange assembly and the lower flange of the condenser assembly was
placed a spacer around which was wound the reflux heater. Initially this spacer was manufactured of glass to permit observation of liquid flow behaviour over the liquid distributor and to check whether or not liquid was being returned to the column at its boiling point in a gently boiling condition and not in a violently boiling state. The spacer was later made of mild steel in the interests of strength and durability.

The reflux heater was so designed as to provide sufficient heat to raise any of the liquid mixtures employed to its boiling point, assuming they were returned to the column head at cooling water temperatures and at flow rates far in excess of those actually achieved. It was constructed of 5 metres of 24 s.w.g. Nichrome resistance wire connected to a constant voltage source.

The top edge of the spacer is located in a groove in the lower face of the condenser flange. A seal is provided by a viton rubber gasket as on the lower spacer surface. The condenser consisted of a 6 inch diameter copper body containing three interconnected coils of 1/2" diameter copper tube. At either end of the copper body were mild steel flanges, the lower one connected
to the flange assembly described earlier by eight long 5/16 inch bolts which, when tightened, compressed the seals at either end of the spacer. Centrally positioned through this lower flange was a pressure tapping, the point of which, by careful sizing of the spacer length, was just above the liquid distributor. Also through this lower condenser flange were inserted two single junction copper/constantan thermocouples, one to read vapour temperatures, the other to read reflux liquid temperature. The reflux heater was adjusted to provide reflux liquid at the same temperature as that indicated by the vapour temperature thermocouple.

The top end of the condenser was attached via a viton rubber 'O' ring seal to a cold finger assembly. The use of solid carbon dioxide in this cold finger completely minimised the likelihood of vapour leaving the column assembly. A connection was provided on the cold finger for connecting to the vacuum control systems.

This entire assembly was supported by the lower flange assembly which, in turn, was connected to a rigid framework using three equally spaced screws. These screws were so designed to permit accurate levelling of the assembly and aided
in achieving a vertical column. Samples of reflux liquid were obtained using a 5 m.m. metal Springham valve welded to 2 m.m. diameter hole drilled in the flange assembly between the glass smoothing collar position and the spacer position.

5.2.4. Control Systems

The principle controlled variable was the stillhead pressure at which distillations were performed. There are many methods of achieving such control \( \frac{44}{126} \), the simplest is the air-leak method. It was recommended \( \frac{28}{28} \) that the air-leak method was more suitable for the required duty. A schematic diagram of the vacuum control assembly is given in Fig. 10. The stillhead pressure was measured using two diaphragm vacuum gauges, one of range 0-760 Torr, the other 0-100 Torr, and a mercury 'U' tube manometer with one end open to the atmosphere. The other leg of the manometer and the two diaphragm gauges were connected to the pressure tapping at the column head. Using this control assembly it was possible to read the pressure to within \( \pm 0.5 \) mm and control pressure to within \( \pm 0.5 \) Torr.

The pressure drop across the column
length was obtained by connecting the upper and lower pressure tappings across two inclined manometers (Fig 7) of ranges 0-25 mm H₂O and 0-12.5 mm H₂O.

To prevent condensation of vapour within the manometer two small condensers were attached one at either of the two points of pressure tappings. It was felt that the error introduced into the pressure drop readings by the possible condensation of vapours within these condensers was negligible.

With regard to electrical control, the stillpot heater was controlled by an 8 amp Variac constant voltage regulator, the reflux heater used a similar regulator. The stillpot immersion heater was controlled using a 3 amp Variac, and each of the heating tapes wound as insulation on the column walls was controlled by simple variable relays. Control was also provided over the heating tape used to preheat the nitrogen prior to injection into the apparatus. The nitrogen was heated to within 1.0°C of the boiling point of the stillpot mixture using Electrothermal "Thermostat" heating elements wound around 2 metres of 1/2 inch diameter copper tube. This heating element was controlled using an adjustable voltage source.
The degree of control provided by all these systems was extremely good and by noting the readings of ammeters, voltmeters and dial settings it was possible to reproduce almost exactly column operating conditions of any given type.

5.3. Experimental Techniques

As stated earlier two experimental techniques were employed. They are distinct techniques but are interrelated in order to achieve a measure of the contribution of the liquid phase resistance to the total resistance to mass transfer. Here each technique will be described individually.

5.3.1. Pressure Drop Technique

This involved running the distillation column under constant stillhead pressure conditions for sufficient time to ensure steady state equilibrium had been achieved. Measurements of the composition of liquid on fins and wall, and a mixed stream of the two, and of reflux liquid were made. Liquid flow rates on the fins and on the wall were also measured as was column pressure drop.

In a laboratory scale distillation column of a wetted wall type, the concentration of the vapour and liquid streams vary continuously throughout
the length of the apparatus. The performance of such apparatus is usually expressed in terms of either the individual gas and liquid height of transfer unit or the overall height of transfer unit or the corresponding transfer co-efficients. In the present work the same criteria was used to determine the performance of the "Pinpak" fractionating column.

The relationship between the gas, liquid and overall heights of a transfer unit, and the gas and liquid compositions was derived by Chilton and Colburn\(^{(17)}\) and is expressed as follows:

\[
\frac{H_{OG}}{N_{OG}} = \frac{Z}{y_2} \int_{y_1}^{y_e-y} \frac{dy}{y_e-y} = \frac{Z}{N_{OG}} \quad (5.1)
\]

\[
\frac{H_{OG}}{N_{OG}} = \frac{Z}{y_2} \int_{y_1}^{y_e-y} \frac{dy}{y_e-y} = \frac{Z}{N_{OG}} \quad (5.2)
\]

\[
\frac{H_{OG}}{N_{OG}} = \frac{Z}{x_2} \int_{x_1}^{x_e-x_1} \frac{dx}{x_e-x_1} = \frac{Z}{N_{OG}} \quad (5.3)
\]
The equation for calculating $H_G$ for material transfer in a wetted wall column was modified for the case of a Finpack column. Fins restricted the cross sectional area of the column by forming a number of channels for vapour flow and also changed the inside diameter of column with a smaller equivalent diameter. The formation of channels was also expected to change the pattern of vapour flow which might have a marked influence on the rate of mass transfer.

The relationship between the gas phase mass transfer co-efficient $k_G$ and transfer unit $H_G$ can be found from the material balance over a differential height $dz$ of "Finpak" column i.e.

$$N_a \cdot A \cdot dz \cdot \alpha = k_G \cdot A \cdot (y_i - y) \cdot \lambda \cdot dz$$  \hspace{1cm} \text{5.5}

Also

$$G_m \cdot A \cdot dy = N_a \cdot \alpha \cdot dV = k_G \cdot A \cdot (y_i - y) \cdot dz$$  \hspace{1cm} \text{5.6}

Integrating equation 5.6. and combining with equation 5.4. gives

$$\int_{y_1}^{y_2} \frac{dy}{y_{1,2}-y} = \frac{Z}{N_G} = H_G = \frac{G_m}{k_G \cdot \alpha}$$  \hspace{1cm} \text{5.7}
The eddy motion that causes mass or heat transfer in a moving fluid also causes fluid friction owing to a change in momentum. The close similarity between the transfer of mass, heat, and momentum is illustrated by the modified Reynolds analogy (36) which is mathematically expressed as

\[
\frac{k_o}{G_m} \frac{Sc}{a}^{2/3} = \frac{h}{\frac{\rho u^2}{\mu}} \frac{Re}{2} = \frac{f}{2} = C \log^n \quad 5.8.
\]

Experimental data for mass transfer into gas streams agree fairly well with equation 5.8, when the value of the Schmidt group is near unity and when the friction factor is calculated from skin friction above. For flow through a straight tube, or across a flat plate placed parallel to the direction of flow, the pressure drop is entirely due to skin friction against the surface. This condition is also obeyed for flow through the "Fin-Pak" tube, though the values of the constants C and n will vary in each case and must be determined experimentally. Combining equations 5.7 and 5.8 we have

\[
\frac{1}{\sqrt[3]{a}} \frac{Sc}{a}^{2/3} = \frac{f}{2} = C \log^n \quad 5.9
\]

For very small pressure losses,
the friction factor for isothermal flow of vapour through straight tubes is correlated by a simplified Fanning equation (58).

\[ \Delta p = p_1 - p_2 = \frac{h_f \rho L}{\frac{2}{g_d} \rho_d u^2} \]

or \( f = \frac{h_f \rho L \rho_d u^2}{\frac{2}{g_d} \rho_d u^2} \) = \( C^1 Re_G^n \) \hspace{1cm} 5.10(a)

For the Finpack column temperature drop over the column length was small, the maximum recorded being only 2°C, and it may be supposed that for the purposes of utilising equation 5.10a, isothermal conditions existed.

By obtaining values of the friction factor, \( f \), and plotting against corresponding values of \( Re_G \) on suitable co-ordinates it is possible to obtain the values of \( C, C^1 \) and \( n \) in equations 5.9 and 5.10(a). It is possible, therefore, to construct a relationship between \( H_G \) and \( Re_G \) of the form

\[ H_G = C^{11} Sc^{2/3} Re_G^m \] \hspace{1cm} 5.11

where \( C^{11} \) and \( m \) are constants.

In order to compare the experimental values of \( H_G \) with the calculated values of \( H_G \) obtained from equations 5.11 and hence arrive at a value of \( H_L \), it was necessary to evaluate the slope
of the equilibrium line 'm' in equation 5.6. Since, in general, the slope changed over the operating range in each experiment, it was desirable to calculate its average value. This was determined by combining equation 5.1 with the following equation, for the overall height of the liquid phase transfer unit $H_{OL}$ due to Chilton and Colburn (17).

$$H_{OL} = H_L + \frac{L}{mG_m} \quad \therefore \quad H = \frac{2}{x_2} \int_{x_1}^{x_2} \frac{dx}{x-x_e}$$

The resultant equation for the average slope is,

$$m = \frac{L}{G_m} \quad \therefore \quad \frac{H_{OL}}{H_{0G}} = \frac{L_m}{G_m} \cdot \left[ \int_{x_1}^{x_2} \frac{dx}{x-x_e} \quad \text{and} \quad \int_{y_1}^{y_2} \frac{dy}{y-y_e} \right]$$

The value of the integrals in equation 5.13 was determined by the method described in Appendix D, and the average slope calculated for all the test runs for all the binary systems studied.
A value for $H_L$ may also be obtained by the following considerations. Higbie\(^{(54)}\) has shown that when a liquid surface is exposed to a gas for a short period of time say $t$, the liquid phase mass transfer co-efficient is given by the equation.

$$ \theta_L = 2 \left( \frac{D_L}{\pi t} \right)^{1/2} \tag{5.14} $$

Equation 5.14 may be applied to a moving film of liquid by substituting for the time of exposure, the distance $Z$ divided by the surface velocity $U_s$. For streamline flow over a vertical surface, the surface velocity is given by Nusselt's\(^{(34)}\) equation.

$$ V_s = \left[ \frac{g \gamma^2}{6 \mu_L} \right]^{1/3} \tag{5.15} $$

Equations 5.14 and 5.15 may be combined and re-arranged to give an expression for $L$ as follows.

$$ \frac{K_L}{D_L} Z = 0.73 \text{ Sc}^{1/3} \frac{K_L}{D_L} L \left[ \frac{g \left( \frac{P_e}{\mu_L} \right)^2}{Z} \right]^{1/6} \tag{5.16} $$

It can also be shown\(^{(17)}\) that the liquid phase transfer co-efficient is related to $H_L$ by the expression

$$ H_L = \frac{\varphi L}{K_L a} \tag{5.17} $$
Hence equations 5.16 and 5.17 give a value for $H_L$ which may be compared with that obtained from equation 5.4 after substitution of the values for $H_{OG}$, $H_G$ and $m$ obtained as described earlier.

From the empirical measurements and from all the subsequent calculations it is possible to utilise the pressure drop technique results in three ways.

Firstly, using pressure drop data and $H_{OG}$ values it is possible to describe fully the physical behaviour and efficiency of the Finpak column as a rectifying device and compare its performance with other fractionating devices.

Secondly it is possible to describe the vacuum distillation process and to comment on the effect of a number of operating variables on the process as studied using a Finpak column.

Lastly by an analysis of $H_{OG}$, $H_G$ and $H_L$ values it is possible to obtain a measure of the contribution of the liquid phase resistance to the total resistance to mass transfer.

5.7.2. Inert Gas Injection Technique

This technique was developed by Sawistowski et al. (101). The conditions under which inert gas injected distillations were performed
were such that the sum of the partial pressures of
the diffusing components, \( P_A + P_B \), remained constant
and equal to the operating pressure \( P \) in a vacuum
distillation run completed under pressure drop
technique conditions. Denoting the inert gas
by the subscript \( I \) we obtain
\[
P_A + P_B + P_I = p^I + P_I = P
\]  \[5.18\]

Under the above operating conditions
the temperature in the column was the same in the
vacuum runs and the nitrogen injection runs and the
mass transfer coefficient in the liquid film remained
unaltered. The gas phase mass transfer coefficient
will change, however, and the extent of its change
can be predicted theoretically.

Using the theory of Curtiss and
Hirschfelder\(^{(20)}\) and applying it for the tertiary
system considered.
\[
\frac{P}{RT} \frac{dP^*}{dz} = \frac{1}{D_{AB}} (P_A^* P_B^* - P_{AB}^*) + \frac{1}{D_{AC}} (P_A^* P_C^* - P_{AC}^*) \]
\[5.19\]
Since \( N_C = 0 \) and \( N_A + N_B = 0 \), assuming that equimolar
counter diffusion is not affected by the addition of
the inert gas,
then
\[
N_b = -\frac{P}{RT} \left( \frac{P_A^*}{D_{AB}} + \frac{P_B^*}{D_{AC}} \right)^{-1} \frac{dP^*}{dz} \]
\[5.20\]
For vacuum distillation

\[ N_p' = -\frac{D_{pe}(dP_e)}{RT} \]  \hspace{1cm} 5.21

From equations 5.20 and 5.21, compared in their integrated forms

\[ \frac{N_p}{N_a} = \frac{\rho D_{pe} (1 + \frac{P - P_e}{P_e}) \rho_{ae} - P_o}{P_{ae} - P_o} \frac{Z_o}{Z_1} \]  \hspace{1cm} 5.22

At constant temperatures $P_{ae} = P_{ae}$ and since the gas Reynolds number is approximately constant, the film thickness $Z_o$ and $Z_1$ can be considered equal. Hence

\[ \frac{N_p}{N_a} = \frac{\rho_{ae} - P_o}{P_{ae} - P_o} \]  \hspace{1cm} 5.23

Where $F = \left(1 + \frac{P - P_e}{P_e} \frac{D_{pe}}{D_{ae}}\right)$  \hspace{1cm} 5.24

But by definition

\[ \frac{N_p}{N_a} = \frac{K_{o} (\rho_{ae} - P_o)}{K_{a} (\rho_{ae} - P_o)} \]  \hspace{1cm} 5.25

Hence $\frac{K_{o}}{K_{a}} = F$ and

\[ K_L = \frac{m(F-1)}{\frac{F - 1}{K_{o} K_{a}}} \]  \hspace{1cm} 5.26

From which values of $K_L$ can be calculated. These values can be treated to yield values of $H_{OL}$ by the relationship

\[ H_{OL} = \frac{L}{K_L} \]  \hspace{1cm} 5.27
and these values of $H_{OL}$ can be compared with those calculated by integration of equation 5.28

$$H_{OL} = \int_{x_1}^{x_2} \frac{dx}{x-x_0} \quad 5.28$$

using the results obtained from the pressure drop technique of greater interest, however, is a comparison of the values given to the contribution of the liquid phase resistance by each experimental technique.

The two techniques can, therefore, be used to provide evidence as to the behaviour of phase resistances. Whilst the supply of such evidence is only part of the treatment of the pressure drop technique data, it is intended that the inert gas injection technique be used solely to provide such evidence and to serve as a check on the theory developed for the pressure drop technique which, it is supposed, could be used to measure the contribution of the liquid phase resistance to the overall resistance to mass transfer.
6. Results of Studies Utilising "Pressure Drop" Technique

6.1. Introduction

Most of the results obtained during the course of the research were obtained using the "pressure drop" technique, which is fully described in Chapter 5.

The results themselves have wide application in that they were obtained under a variety of operating conditions, using contrasting binary systems yet remaining comparable with the results published by others.

The experimental data obtained is used to describe all three facets of the research i.e.

a) To provide data on the "Finpak" column as a distillation apparatus.

b) To illustrate the effect of operating variables on distillation efficiency.

c) To measure the liquid phase resistance to mass transfer in rectification.

6.2. Performance of the "Finpak" Distillation Column

The method of presenting the results of the study of column characteristics is an adaptation of that used by Billet\(^{(5)}\) and is as follows.
1. Determination of the efficiency, $E$, of the theoretical number of transfer units per unit column height $n_t/H$, ($m^{-1}$) as a function of the vapour capacity factor, $U_v \sqrt{A_0}$, where $U_v$ is the superficial velocity of vapour of density, $\rho_v$ in the cross section of empty column.

2. Determination of the pressure drop per unit column height, $\Delta P/H$, as a function of the vapour capacity factor.

3. Determination of the specific pressure drop per transfer unit, $\Delta P/n_t$, as a function of the vapour capacity factor. The parameter $\Delta P/n_t$, is the main factor in assessing the suitability of a separation unit for rectifying thermally unstable mixtures.

The above results are presented in Tables 3 to 11 and graphically in Figures 11 to 16.

6.3. **Effect of Operating Variables on Distillation Efficiency**

The results presented in the above form are also suitable for indicating the effect of a number of operating variables on column efficiency.

This research investigation was aimed at providing data for the case of a vacuum distillation column operating with liquid flows within what had previously been regarded as the laminar flow condition.
As stated earlier it was felt that the sub-division of liquid flow regimes into Laminar/Transitional/Turbulent regions was imprecise. It has been decided to describe the various regions of mode of flow by the method used by Portalski, namely

Laminar - Pseudolaminar - Transitional - Pseudoturbulent - Turbulent.

As a result, the majority of results obtained during the course of this research programme were in the pseudolaminar region. To define the above regions the value of critical Reynolds number as defined by Grimley was used as the value for liquid Reynolds numbers within the pseudolaminar region, and the value of Reynolds number at the point of ripple inception, as defined by Portalski, as the upper value for liquid Reynolds numbers within the laminar region.

It was found that during the course of this work that the upper limit could be defined by the value of vapour phase Reynolds number of approximately 1300. This is borne out by a consideration of the values of friction factor, $f$, and corresponding values of vapour phase Reynolds number given in Tables 12 to 21 and shown in Fig. 17.
6.4. Liquid Phase Resistance to Mass Transfer

The flow conditions maintained within the liquid film during the majority of experiments were beneficial towards establishing and maintaining significant liquid phase resistances if such resistances existed.

As it was felt that liquid phase resistance would be affected by a variety of conditions, mainly the conditions relating to the liquid phase, the diverse nature of the systems and conditions employed would tend to optimise at some point the presence of liquid phase resistance.

Tables 22 to 30 present the values of individual and overall phase resistances to mass transfer and provide for a direct comparison with liquid phase flow conditions and surface tension variation.

The above data is also presented in Figs. 18 to 21.
CHAPTER 7

7. Results of Studies Utilising "Gas Injection" Technique

7.1. Introduction

As described in Chapter 5 it was intended that results obtained utilising this technique be utilised in supporting the information gained with regard to the contribution of liquid phase resistance to the total resistance to mass transfer employing the "pressure drop" technique.

The technique was developed by Savistowski et al\((101)\) who reported that results obtained with an injection of 21\% air were unsatisfactory since the change in performance when compared with results at 600 Torr was too small to allow for accurate comparisons. They obtained results with an injection of 47.5\% air compared with runs at 400 Torr that indicated that the liquid phase resistance passed through a minimum, and was always significantly lower than the gas phase resistance.

7.2. Results of "Gas Injection" Technique

In view of Savistowski's\((101)\) comments regarding the need to employ significant injections of inert gas it was decided to utilise results from the "pressure drop" technique whereby the percentage of air injected would be a maximum. This would be
true of runs conducted at stillhead pressures of 100 Torr and 50 Torr where the corresponding percentages of injected gas would be 86.84% and 93.42% respectively.

Further it was decided to utilise data from experimentation at those pressures using the surface tension positive system \((\text{decane}/\text{trans decalin})\) and the surface tension neutral system \((\text{trans decalin/cis decalin})\).

A line diagram of the layout of the apparatus employed is given in Fig. 27. Initially argon was employed as the inert gas in order to minimise the risk of oxidation of system components and of fire. However, the usage rate was such that the argon was substituted with high purity nitrogen.

The systems employed and the apparatus were first operated exactly as for the conventional vacuum distillation runs. After a period of four hours, during which time it was believed the system would have approached equilibrium, the inert gas was gently introduced into the system, via the stillpot, so as to raise the stillhead pressure within the column to 760 Torr, after a period of one hour. During the course of raising the pressure the
vacuum pump was kept running to maintain conditions exactly as for the vacuum distillation runs. The system was then monitored for a period of not less than a further three hours to ensure that equilibrium conditions were attained.

In view of the time factor involved for each run, and the early problems with the layout of the equipment it was not possible to complete more than thirty runs. Twenty of these runs were with the system trans decalin/cis decalin, ten each at injection percentages of 36.34 and 93.42. Ten runs were completed with the system n decane/trans decalin at an injection percentage of 36.34.

The results obtained using this technique are not suitable for inclusion. From a consideration of the calculated data it is believed that a dilution of 93.42% with inert gas is such that the vapour phase properties are essentially those of the inert gas employed. An assumption made by Savisto ski (101) in developing the technique was that the interaction of the vapour and liquid phases is not changed significantly by the addition of the inert gas. It is felt that at a dilution of 93.42%, conditions are such that the above assumption does not hold, and that the addition of
inert gas in such quantities renders a comparison of results with vacuum distillation techniques invalid.

It may be strongly contested that the effects of such a dilution could well have been appreciated beforehand and a lower dilution of say 60 - 70% should have been employed. In answer to this it must be realised that if this practice had been adopted it would have meant that a stillhead operating pressure would have been fixed in order to achieve the required dilution. This in turn would have meant searching for a binary system which complied with the majority of requirements listed in Chapter 4 for such a test mixture, and for which there had been published accurate, internally consistent data, or by lengthy experimentation establishing such equilibrium data, for the required stillhead pressure.

Further the importance of the inert gas injection technique must be correctly appreciated within the context of the proposed research. It was at all times intended that this technique should provide support data only, and that sufficient experience be gained with this technique to fully define its importance as a method of measuring the liquid phase resistance in any future work.

It is felt that the results obtained
have been of partial usefulness. It is true that in no way can they be used to support the results obtained using the pressure drop technique, but they do serve to indicate the maximum dilution permitted, and taken in conjunction with the results of Savistowski et al\(^{(11)}\), it is now possible to state that the optimum dilution with inert gas when employing this technique is in the range 60-70%.

A second contributory factor to the considerable inaccuracies in the results obtained lies in the lack of sensitivity of certain of the methods employed (as discussed in Chapter 4) to determine vapour phase properties. An example of this is the determination of vapour mixture viscosities. The method employed was a modification of the method described in Wilke\(^{(122)}\). This method was designed for binary vapour mixtures but was employed such that a viscosity was obtained for a mixture of two of the three constituents of the vapour phase, and this average viscosity, together with other overall properties of this theoretical binary mixture, was again used in Wilke's equations with the properties of the third constituent to give an overall viscosity for the tertiary vapour mixture. It is felt that some accuracy is lost in employing equations in this
way, and that this loss of accuracy results in
difficulty in showing small changes in vapour phase
properties, especially when one constituent is
present in concentrations as high as 85-95%.

Throughout all runs considerable
difficulty was experienced in maintaining a stable
flow condition within the liquid films, especially
those on the fins. Under normal vacuum distillation
condition, little difficulty was experienced in
maintaining such films and it is felt that there were
two causes for such instability.

Firstly, although the inert gas was
pre-heated prior to introduction below the boiling
liquid surface within the stillpot, considerable
difficulty was experienced in testing the equipment
with condensation of system components within the
apparatus. Additional heaters were fitted to ensure
that the gas was sufficiently pre-heated, but it is
felt that the means of controlling the temperature
of the injected gas by manual observation and method
was too inaccurate, and possessed an inherent lag which
prevented complete and accurate control.

The mis-match in temperatures manifests
itself in two ways. Either condensation of the vapour
is adversely increased, or additional vapourisation
of the liquid film occurs, because of too high an injection temperature. As stated, the control of temperature was inefficient and although every endeavour was made to avoid the described effects, the results obtained are such to suggest that the measures were not effective.

Secondly, the column did operate effectively at low liquid flow rates during the course of the "pressure drop" technique experiments, but as stated earlier, difficulty was experienced in maintaining an effective film on the finned surfaces.Acknowledging the contribution made by the effect of mismatch of temperatures, it is also felt that the additional "vapour" load of the inert gas affected the behaviour of the liquid distributor. On occasions it was observed that hold up within the stillhead was higher than previously experienced. This hold up tended to result in surges of liquid flow down the column.

Both the above points are indicative of inadequacies within the equipment, a drawback experienced by Savistowski (101). Further, the practice of calculating vapour phase concentrations from an analysis of liquid phase compositions would appear inadequate. As stated earlier the presence of such
large quantities of inert gas imposes the properties of that gas on the vapour phase. A direct analysis of the vapour phase should be made in situ in order to provide comparative measurements of vapour phase composition.

To summarise, therefore, it is felt that inadequacies in equipment design and analysis coupled with the use of too high a dilution figure resulted in data from runs utilising this technique being too inaccurate and misleading for use as a comparative experimental method. However, provided such design errors are eliminated, and a dilution of 60-70% with inert gas be employed, it is felt that this technique does present an ideal basis for further research.
CHAPTER 8

DISCUSSION OF RESULT

8.1. Performance Of The 'Finnak' Distillation Column

The original concept\textsuperscript{(15)} of the 'Finnak' column was based on the requirement to supply a rectification device possessing low pressure drop characteristics which provided extended surface area for mass transfer whilst studying vertically flowing liquid films.

The apparatus described in Chapter 5 was a modification of the original column designed to the above requirement.

The equipment is ideally suited to the study of the vacuum distillation process. The column possesses low liquid hold up in operation combined with relatively low column pressure drops. Further, the presence of flat, straight vertical surfaces provide for the study of liquid film behaviour at low liquid rates whilst also giving additional surface area without introducing the possibility of turbulence.

Prior to discussing the actual performance of the column and comparing this with the effectiveness of other types it is necessary to comment on the nature of the results obtained.

As stated earlier liquid flow
down the column was divided into fin and wall flows in a ratio determined by the proportionality of surface areas. To give a true analysis of results this means that two distinct Reynolds numbers existed for the liquid phase depending upon which surface was considered, whilst only one common value existed for the value of Reynolds number in the vapour phase.

Thermal gradients established within the column would be affected by the fact that the stainless steel finned surface possessed a higher thermal conductivity value than the lagged glass surface.

It was felt that as data was to be presented as an overall value it would be acceptable to employ an average value for liquid phase Reynolds number based on individual fin and wall values. Secondly as much of the work was undertaken in vacua or at relatively low boil up rates it was found that the temperature drop over the column length was relatively low, the maximum recorded being only 2°C, and that correspondingly any errors influenced by dissimilar thermal conductivities could be neglected.
During the course of experimentation it was found impractical to operate the column at stillhead pressures below 10 Torr. At such low stillhead pressures the vapour velocity within the column was such that it caused excessive liquid hold up and the subsequent occurrence of column flooding even at relatively low boil up rates.

Dilet (6) emphasised the rule that the lower the system pressure, the higher the maximum vapour capacity factor characterising the flood point at the end of the efficiency curve. Whilst this rule has some direct bearing on the limitation of the apparatus described above, it also describes a further limitation in the equipment design, inasmuch as both the stillpot heater and reflux heater assemblies were not adequate enough to provide for the necessary flow rates required to provide a full measure of efficiency up to the flood point at both high and low pressures.

To summarise, therefore, the design of the apparatus eventually proved to be such that it limited the scope of the research to an area of measurement, which although practical, does not fully describe the extreme ranges of flow conditions.
In assessing the performance of the 'Finpak' column two standpoints may be adopted. Firstly the efficiency of the column, expressed as values of \( n_t/H \), may be examined, and secondly the low pressure drop characteristics must be compared as the equipment is primarily designed for low pressure rectification studies.

In all cases the efficiency of the column decreased with increasing boil up rate. Referring to Figures 11 to 16, and considering values of \( n_t/H \) within the range of high loads of practical interest, i.e. \( \frac{W}{V} \leq 0.7 \text{ m}^{-1} \text{s}^{-1} \), the column efficiency is higher than that of an equivalent wetted wall column, comparable with the efficiency obtained in conventional packed columns using various sized Pall rings (7), but is much poorer than columns packed with gauze packings (7)(33). The rate of decrease in efficiency with increased loading is not as marked in the Finpak column as for gauze packings, and again is comparable with the more conventional packing materials.

The similarity of efficiency, and the effect on efficiency of increased boil up rate when comparing the results of the Finpak column
with the results obtained using conventional Pall
ring packings should be expected. Both types
of elements can be regarded as being basically
curved or flat surfaces either parallel to, or
inclined to, the direction of liquid flow and both
packings possess relatively high voidage factors.
The far higher efficiency of gauze packings and
their more marked decline on increasing column
loading is a direct function of the capillary
action of the gauze itself, an action not possessed
by the Finpak column.

The pressure drop characteristics
of the 'Finpak' column are typically those generally
experienced in other columns of 'open' tube design.
Whilst poorer than those of the
traditional wetted wall column, they are comparable
with the Kuhn multiple tube column ($C_k$), and are an
improvement over the characteristics of conventional
packing materials as illustrated in Fig. 2.

The results, as plotted in Figs.
11 to 16, show an increase in pressure drop with
increasing vapour rate, as is to be expected. This
rate of increase of pressure drop is generally lower
than for other distillation devices because the area
available for flow is higher in this type of column,
the increasing pressure drop being directly related to increasing skin friction, an argument employed in Chapter 5 when developing the experimental technique. Such skin friction is increased at higher throughputs not only as a result of higher vapour velocities, but also by an apparent "roughening" of the flow surface by the introduction of "ripples" and ultimately "waves" within the counter current liquid film.

From a consideration of Fig. 17, it can be seen that for runs where the Reynolds number for vapour phase was less than 1300, the relationship between skin friction and flow is essentially linear. Few results were obtained at higher values principally because this entailed work outside the field involving laminar liquid films, and practically because this regime was at the maximum loading of the apparatus. It was established utilising the technique described in Appendix A that two linear relationships existed, both holding to a maximum value of $Re = 1300$, as are shown in Fig. 17.

The uppermost of the two curves applies to surface tension negative systems, whilst the lower refers to surface tension positive systems.
It is proposed that skin friction is accountably higher in surface tension negative systems because of increased film thickness, and this point is developed further in Section 3.5. Points representing data achieved in runs where $Re < 1300$ have been omitted from Fig. 17, but are presented in the tables. As mentioned earlier linearity up to $Re = 1300$ was established by computer program, the considerable scatter for points greater than $Re = 1300$ suggests that these values lay in a transitional area.

An overall assessment of the Finpak column is that it provides a form of distillation device ideally suited to the study of vacuum distillation by virtue of its acceptable efficiency and excellent pressure drop characteristics. Its acceptance as a device for studying vacuum distillation is further enhanced by its ability to handle low liquid rates whilst still maintaining a fixed surface area for mass transfer. Further it is possible to examine behaviour on packing surfaces independently of behaviour on column walls, without the additional complication of liquid streams intermixing.
3.2. **Effect Of Operating Variables On Distillation Efficiency**

It is believed that a wide number of operating variables will affect distillation efficiency, either adversely or to the benefit of efficiency. The sum total of such effects is then given as the overall effect on efficiency, although only in the case of Galperin and Zelvenetskii \(^{(92)}\) was there any qualification to the statement that their measured effect of increased efficiency with reduction in pressure could be generally accepted because of the widely differing properties of the systems employed.

Examination of Figures 14 to 16 shows that there was, for a given vapour capacity factor, an increase in efficiency on reducing pressure. These curves, as indeed all plots presented were obtained using the computer method described in Appendix A, and represent the curve of "best fit" for all experimental runs relevant to that curve.

For the system chlorobenzene/ethylbenzene, on reducing the stillhead pressure from 750 Torr to 300 Torr it was found that the efficiency increased by an average of 30\% in the region of practical interest, i.e. for values of \(\sqrt[\frac{1}{2}]{\frac{\overline{p}}{v}}\) 2.0 but less than 2.7. This percentage increase in
efficiency, and all following percentage increases given, was calculated by employing the equations derived by the polynomial regression method described in Appendix A and substituting values of $U_{v}P_{v}^{1/3}$ to give values of efficiency which were arithmetically averaged to give the percentage increases quoted.

On reducing stillhead pressure to 50 Torr, the efficiency for the system chlorobenzene/ethylbenzene was found to have increased by a factor of approximately 54% over the efficiency at 760 Torr.

Such results for this system are slightly higher than those reported by Peters and Cannon (32), who used this same system with conventional packings at greater variations in stillhead pressure, and are very much lower than the results of McLaughlin (75), who predicted doubling efficiency when reducing pressure from 760 to 15 Torr in columns greater than 10 mm bore.

For the system n decane/trans decalin, efficiency increased from that at 300 Torr by a factor of 36%, when reducing pressure to 50 Torr in the range $2.0 < U_{v}P_{v}^{1/3} < 2.7$. This figure is greater than the 16% increase given
when reducing the stillhead pressure by the same values using chlorobenzene/ethylbenzene. However, as flow increases, at a given stillhead pressure, the efficiency of the n-decane-trans decalin system declines far more rapidly than that for the chlorobenzene/ethylbenzene system so that at values of $U_{w}^{1/2} \gg 2.7$ the percentage increase in efficiency on reducing pressure from 300 to 50 Torr is the same for both systems. This may suggest a maximum efficiency of operation when using n-decane/trans decalin and being mindful of the comments of Gelgerin and Selvenetskii concerning the influence of system properties this would seem to illustrate their case and to be of use in understanding the apparent contradictions in the wealth of data available as to the effect of reduced pressure on column efficiency.

For the system cis-decalin/trans decalin, on reducing pressure from 100 Torr to 50 Torr there is a 40% increase in efficiency within the practical range of operation. Again this increase in efficiency declines rapidly as flow increases, until at values of $U_{w}^{1/2} \gg 4.0$ there is no apparent variation in efficiency. It would
therefore seem necessary to not only fully define actual system properties but distillation conditions relating to vapour flow and liquid flow, and to aid in this it is suggested that the flow regime schematic proposed by Portalski\((22)\) with its greater subdivision and hence greater sensitivity to definition be employed.

Considering Figures 11 to 13 it can be seen that in the greater majority of cases systems of a surface tension negative character\((130)\) possess greater efficiencies for a given flow rate and stillhead pressure, that do surface tension positive systems. This result is the reverse of that expected whereby the better wetting characteristics of surface tension positive films might have given this type of system the improved efficiency.

Surface tension is dependent upon two basic factors, temperature and composition. As the effect of composition on distillation efficiency has been well examined\((60)(102)\) it was felt that this did not merit extensive investigation and hence, throughout the research, stillpot compositions were maintained in the middle of the distillation range to avoid maximising efficiencies; as is the case when
utilising compositions at extremes of concentration of the more volatile component.

Temperature was by far the more variable of these factors, being more directly affected by stillhead pressure. This is reflected in the fact that whilst overall column efficiency rose on reducing pressure the difference in efficiency when comparing the results of surface tension negative and positive systems fell whilst concentration and more specifically the concentration gradients were maintained at approximately the same level.

It is disappointing that the indications referring to the effect of surface tension character on efficiency are inconclusive. However, as the experiments were conducted such that stable films were maintained, it is probable that the major effect of surface tension whereby films of surface tension negative character break down to rivulets has been circumvented. If this point is valid there must be a further reason for the apparent variation in efficiency difference on comparing differing surface tension systems with a variation in stillhead pressure, allowing for the
temperature effect. As evidence that stable films were produced in all system types, considering Fig. 11 there is an indication of increasing efficiency with increasing boil up rate to a maximum, followed by the normal gradual decrease in efficiency for the system benzene/toluene. This maximum in the efficiency curve is present, because the benzene/toluene system was run at relatively low boil up rates in comparison with other systems and it is probable that there were breaks in the liquid film giving rise to unwetted areas which were more fully covered on increasing the boil up rate. Butt (15) in earlier work with the "Finpak" column reported considerable difficulty in maintaining stable liquid films at low boil up rates, especially on the finned surfaces. The design of the liquid distributor has been much improved on this later version of the equipment and, with the exception of the above observation, it is believed, accepting that visual observation is not a reliable means of distinguishing between fin areas covered with a flowing liquid film and areas covered with a stagnant film, that stable, whole films were produced and maintained.

These differences when comparing efficiencies between different systems may be
attributed to changes in viscosity or diffusivity in either the liquid or vapour phases. However, it has been effectively demonstrated\(^{(2)}(6)(7)(12)\) that both these properties can be regarded as pressure independent when considering their effect on overall efficiency, insomuch as a decrease in pressure, or more importantly the associated fall in temperature, will affect a change in efficiency through its influence on a physical property of one phase whilst simultaneously affecting an equal and opposite change in efficiency through its influence on a physical property of the second phase.

It can be accepted, therefore, that the difference in efficiencies for a given flow rate between surface tension negative and surface tension positive systems cannot wholly be attributed to experimentation, nor to a specific physical property and a possible reason may lie in the mechanics of the actual mass transfer between phases.

Two possible courses are, therefore, open, either to accept that a contribution is made by the existence of thermal distillation processes\(^{(21)(100)}\), a contribution varying according to system type, or by acknowledging the presence of a substantial
liquid phase resistance.

8.5. Individual Phase Resistances.

The conditions maintained within the "Finpak" column throughout the course of the research were such as to maximise the contribution of the liquid film resistance.

The use of the column under low stillread pressures tended to increase vapour velocity, resulting in thinning of vapour film, and also causing greater penetration of molecules from vapour phase to liquid phase because of their higher momentum.

Further, the accompanying fall in temperature caused a fall in vapour viscosity, again causing thinning of the vapour film. Correspondingly in the liquid film there was a rise in viscosity coupled with a fall in liquid diffusivity.

All these factors would tend to reduce the vapour film resistance and increase liquid film resistance. However, the factor most greatly influencing liquid film resistance was the flow condition of the liquid phase.

The results for all systems relevant to the liquid phase resistance are given in Tables 21 to 29, and presented in Figs. 17 to 25.
Considering Figures 22 to 26 for all systems it can be seen that values of $\frac{H_{OG}}{T}$ measured experimentally increase linearly for all systems, where flow rate permitted, up to a value of $Re_L$ of about 30-35. For $Re_L$ greater than 35, despite somewhat increased scatter, the results indicate that the rate of increase of $\frac{H_{OG}}{T}$ is lowered. Further, the rate of increase of $\frac{H_{OG}}{T}$ is greater for surface tension positive systems than for surface tension negative systems.

In order to evaluate the height of liquid phase transfer unit $H_L$ from the experimental value of $H_{OG}$, use was made of equation 4.1\(\frac{1}{2}\), in which the height of vapour phase transfer unit $H_G$ was calculated with equations 3.1 and 3.2., which are the forms of equation 2.11 obtained from Fig. 17. relating to surface tension negative and positive systems respectively.

\[
H_G = 3.269 \times 10^{-5} \frac{Sc_G}{Re_G} \quad 8.1
\]

\[
H_G = 3.49 \times 10^{-4} \frac{Sc_G}{Re_G} \quad 8.2
\]

The value of 'm' in equation 4.4 was calculated as described in Appendix D, and it is felt that this method was the most accurate available.
The equations relating to \( \lambda \) are obtained from Fig. 17 which is a plot of the friction factor of \( \eta \) versus \( Re \). As stated earlier the data for the systems chlorobenzene/ethylbenzene and benzene/toluene lie above the curve for surface tension positive systems.

Pressure losses in a film type vapour liquid contacting device occur as a result of the impact and sheering forces exerted by the vapour on the liquid phase. The degree to which the liquid film is agitated depends upon the liquid flow rate and the liquid properties. It is probable that the magnitude of the pressure losses in the Finpack column was influenced by the thickness of the liquid film. Stable films were maintained by visual examination and the evidence discussed in Section 3.4 suggests that such films were achieved. Averaged values of calculated film thickness indicate that the surface tension negative films were slightly thicker than the surface tension positive films, as was the average liquid viscosities.

Comparing Fig. 17 with Figs. 22 to 26 it can be seen that in all cases a linear correlation ends with either a marked change in slope, or a scatter of experimental data. The change in slopes
in Fig. 17 corresponds to a value of $Re_G$ of 1100 - 1200, in Figs. 22 to 25 the slopes change at $Re_L$ values of 30 - 35, and the changes may be attributed to a change in the pattern of vapour flow (transition region) through the packing. The use of equations 8.1 and 8.2 for values of $Re_G$ greater than 1300 - 1500 is doubtful because of the increased turbulence which is believed exists within the vapour phase.

Average values of $E_L$ obtained in the foregoing manner are presented as percentages of the total resistance to mass transfer related to values of $Re_L$ in Figures 18 to 21.

Considering the results for runs conducted at atmospheric pressure as presented in Tables 21, 22 and 23 and graphically in Fig. 18. It is apparent that the surface tension negative systems appear to possess the greater liquid phase resistance. At a $Re_L$ of 30 the value of % total resistance in the liquid phase for the system benzene/toluene is 72%, for the system n-heptane/toluene the corresponding value is 35.5%.

Both chlorobenzene/ethylbenzene and o-heptane/toluene exhibit short declines in the value of the resistance within the liquid phase tending to even out at values of 17% and 6%. 
respectively for values of $Re_L$ greater than 50-70.

Table 3 lists the Reynolds numbers for all systems relating to the point of ripple inception as given by Portalski (92) and the value of critical $Re_L$ as given by Grimley (46).

Considering these values in relation to Fig. 18 the sharp decline in liquid phase resistance for the system n heptane-toluene can be explained by the fact that the majority of results lie in the pseudolaminar region (92), a region where such a decline is to be expected. The same comments are applicable to the results of chlorobenzene/ethylbenzene at 760 Torr but the more gradual decline in the liquid phase resistance exhibited by the benzene/toluene runs cannot be easily explained.

Considering Fig. 19 and Tables and 23 and Table 24, the pattern described above is more clearly emphasised as there is a dramatic decline in liquid phase resistance between values of $Re_L = 20$ and 40. There is a greater similarity in the behaviour of the two systems at this pressure than hitherto.

Considering Fig. 20 and Tables
27 and 29 all the runs were conducted below values of critical \( Re_L \) given in Table 30 however, the decline in liquid phase resistance is as great as the other runs at higher pressures. In all cases it can be seen that the value of the liquid phase resistance is appreciable, declining rapidly above values of the \( Re_L \) calculated by the method prescribed by Portalski\(^{(92)} \) as the point of ripple inception. Further the liquid phase resistance is found to be higher in the surface tension negative systems possibly because of their slightly higher viscosity and their generally higher surface tension characteristics.

In view of these comments regarding viscosity and surface tension, two properties directly influenced by temperature, it would be expected that under vacuum distillation conditions the liquid phase resistance would be correspondingly higher. However, this is not the case as illustrated in Fig. 21. It is believed that the increases in vapour velocity as stillhead pressure is reduced influences the liquid film which is also more prone to rippling as shown by the gradual decline of values of \( Re_L \) in Table 30 as operating pressure...
is reduced. This tendency to rippling would result in a relatively large increase in the interfacial area for mass transfer.

Further, the tendency to rippling coupled with large increases in vapour velocity as pressure is reduced may result in a lateral spread accompanied with considerable mixing and/or penetration of the film resulting in the complete rupture of the boundary layer, especially under vacuum distillation conditions where the film thickness is reduced.

The improved penetration of the film proposed above would also explain the higher values of liquid phase resistance for surface tension negative systems where the film thickness was greater.

However, it is believed that the results indicate a trend for the liquid phase resistance which explains its dismissal by other workers.

For values of $Re_L$ greater than the critical value given by Grimley, the liquid phase resistance rapidly declines to an overall value which may be neglected. The value given for $Re_L$ by Grimley is much lower than the flow regimes of practical interest.
In agreement with Everitt (37) it is proposed that, in preference to thermal distillation, the existence of a significant liquid film resistance would serve as a more practical explanation of the difference between experienced distillation efficiencies and theoretical distillation efficiencies, especially under low liquid flow conditions as are experienced in vacuum distillation.
9. **Conclusions**

It has been demonstrated that the Finpak column is an effective rectification device under vacuum distillation conditions. The equipment possesses pressure drop characteristic and efficiencies comparable with those of accepted packing media. However, whilst the apparatus employed incorporated many improvements over the prototype equipment, especially in the distribution of liquid reflux, and in the direction of liquid and vapour flow in general, there are design modifications which are required prior to any future work.

Firstly it is necessary to increase column diameter to enable research at much lower stillhead pressures to be undertaken. Runs were attempted at 5 and 10 Torr with the system cis decalin/trans decalin, but these failed through excessive liquid hold up and entrainment which led to unstable liquid flow and intermittent flooding.

Secondly the capacity of stillpot and electrical heaters must be increased to permit a wider range of measurements to be made, especially
within a range of vapour Reynolds Number of 1300-1700, to fully establish the levels of the proposed transition regions.

Finally improvements must be made in the methods of sampling, especially incorporating a means of sampling the vapour phase directly. To achieve this end is exceptionally difficult, because the introduction of sampling points along the column will disturb the thin liquid film. The use of thermocouples, as described by Storrow, seems a practical proposition but one can never be sure that the temperatures measured are in fact saturation temperatures. If any liquid condenses on the thermocouple head the measured temperature will be a wet bulb temperature.

A general difficulty of either method of measuring concentration gradient by direct measurement is that one wants a separation sufficient to give a reasonable concentration difference whilst maintaining practical column lengths. It is believed that the column length employed was suitable in this respect and from a consideration of the influence of column end effects. However, there is ample scope to investigate the effect of varying column length
in order to establish the relative magnitudes of end effects in a column of this type.

The difficulties experienced in using the inert gas injection technique have been fully discussed and obviously steps need to be taken to ensure correct inert gas heating prior to injection and also the quantities of gas injected. It is strongly felt that this technique offers the soundest path for future work, and that provided all necessary equipment modifications are made, it will provide reliable and consistent data regarding liquid phase resistances.

The liquid phase resistance has been shown to exist as a relatively large percentage of the total resistance to mass transfer. However, it must be most strongly stressed that its existence as such is very much dependent on liquid flow conditions and on the nature of the binary systems employed.

Throughout the research it has been evident that there is a need to qualify exactly all operating parameters, and to allow that whilst much of the work undertaken, and the methods employed, were general, and that a wide range of possibilities existed, any future work must specify more precisely the region and system of interest.
In conclusion therefore it is felt that the contradictory nature of much of the research work reported in vacuum distillation is to a certain degree acceptable, but that the failure by authors to appreciate fully the exact area of their work has led to many over generalisations. In the present research an apparatus has been introduced which is an efficient vacuum distillation device, and which has been employed in work which was general in character, but which has provided the basis and experimental technique to enable more specific work to be undertaken. The liquid phase resistance has been shown to be a substantial contribution to the total resistance to mass transfer, and that it is concluded that this supports those workers who have suggested that it is the contribution of a liquid phase resistance, which affects distillation efficiency and not the alternative theory of the contribution of thermal distillation mechanisms.
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Fig 1 - VARIATION OF PRESSURE DROP WITH VAPOR LOADING
- PLATE COLUMNS
Fig 2 - VARIATION OF PRESSURE DROP WITH VAPOUR LOADING
- PACKED COLUMNS.
Fig. 3. Herington-Redlich and Kister test for thermodynamic consistency.

Fig. 4. Van Ness test for thermodynamic consistency.
Fig. 5 Hamilton Symmetrical Area Test

Curves of $\int x \, dx$ vs. $x$. 
Log $y/y_0$ vs $x$.

X, mole fraction in decane liquid.
Fig 7 - Flow Diagram of Finpak Column

- Cold finger assembly
- Condenser: 3 x 1/2" copper coils
- Pressure tapping
- 2.8mm bore glass tubing
- Stainless steel fin assembly
- Thermocouples (spaced over column length)
- Pressure tapping
- Calculated limits for measuring liquid flow
- Liquid return to still pit
For clarity tip of fin assembly has been omitted.

Fig. 8 - Cross sectional view of column base.
Fig 9 - Exploded View of Column Head Assembly
All pipework ½” copper tube
All valves, spring from gauges (close to 2.4 or 3.0) using either brass or nickel

Fig 10 - Schematic Diagram of Vacuum Control Assembly
Fig 12 Influence of different systems on 'Finn-Pak' column performance at a distillation pressure of 300 Torr.

- X Chlorobenzene/Ethylbenzene 300 Torr (-system)
- O n- Decane/Trans Decalin 300 Torr (+system)
Fig 13 Influence of different systems on Finpak column

Performance at a distillation pressure of 50 torr
Fig 14: INFLUENCE OF OPERATING PRESSURE ON 'FINPAR' COLUMN

PERFORMANCE FOR THE SYSTEM CHLOROBENZENE/ETHYLBENZENE
Fig 15  INFLUENCE OF OPERATING PRESSURE ON 'FINPON' COLUMN

PERFORMANCE FOR THE SYSTEM n-DECANE / n-METHYDECALIN

X n-DECANES n-METHYDECALIN
O n-DECANES n-METHYDECALIN

300 TBP + SYSTEM
50 TBP
Fig 16  INFLUENCE OF OPERATING PRESSURE ON 'FINPAX' COLUMN PERFORMANCE FOR THE SYSTEM cis DECALIN/\text{\textit{n}}-\text{DECALIN}.
Fig 17

Plot of friction factor, $f$, vs vapor phase

Reynold's number $Re_g$ for results obtained using both negative and positive surface tension systems with $Re < 1300$

\[ f = 16523 \cdot Re_g^{-1.89} \] (negative systems)

\[ f = 1587 \cdot Re_g^{-1.6} \] (positive systems)
Plot of liquid phase resistance, represented as percentage of total resistance to mass transfer, versus Reynolds number for liquid phase at a stillhead pressure of 760 torr.

- X: Benzene/Toluene
- O: Heptane/Toluene
- △: Chlorobenzene/Ethyl Benzene
Fig 19  Plot of liquid phase resistance, represented as a percentage of total resistance to mass transfer, versus Reynolds number for liquid phase at a stillhand pressure of 300 torr.

X chlorobenzene/ethylbenzene
O a decane/λom decalin
Fig 20  Plot of liquid phase resistance, represented as a percentage of total resistance to mass transfer, versus Reynolds number for liquid phase at a stillhead pressure of 50 Torr.

% Total resistance in liquid phase

Reynold's number, $Re_l$ (liquid phase)

- X Chlorobenzene/ethylbenzene
- O A decane/liquid decalin
- △ Waxes/decalin/c5 deca decalin
Plot of liquid phase resistance, represented as % of total resistance to mass transfer, versus Reynolds number for liquid phase to illustrate effect of still head pressure and differing surface tension quality of reflux employing classification of Zuiderweg and Marnens.

X chlorobenzene/ethylbenzene
O decane/napth decalin
Fig 22

Plot of $H_{o2,5}$ (expt) versus $R_{o1}$ for
benzene toluene
at 760 torr

\[ R_{eL} = \frac{uCL}{\muL} \]
FIG 23  PLOT OF HOG (EXPT.) VERSUS Re
FOR n HEPTANE/TOLUENE AT
760 TORR

\[ \text{Re} = \frac{4 \tau}{\mu L} \]
Fig 24  Plots of log (exp) VERSUS Re, for CHLORO BENZENE / ETHYL BENZENE AT 760 TORR, 300 TORR and 50 TORR

\[ R_e = \frac{4L}{vL} \]
Fig 25 Plot of $\log (\expil)$ versus $R_{EL}$ for $\alpha$ decane/immiscible decalin at 300 Torr and 50 Torr

$R_{EL} = \frac{4\gamma}{\mu_L}$
Fig 26  Plot of $\text{Hog (EXPTL)}$ versus $R_{\text{el}}$ for
Wax Decalin $\rightarrow$ Dicalin at 100 Torr
and 50 Torr

\[ R_{\text{el}} = \frac{4 \pi}{\mu L} \]
APPENDICES
Throughout the research it has been necessary for a number of reasons to express the relationship between variables as a polynomial expression. The majority of such expressions were constructed from empirical data and it was further desirable that not only an expression be produced, but a measure of the statistical worth of the data, and a comparison between the experimental values and the predicted values given by any such expression.

For such a treatment of data on occasions when between forty and hundred data points were available it was necessary to employ computer programs. It was fortunate that I.B.M. supplied, in their scientific subroutine package, the program 'PCLRG'. This program, and its associated programs, was capable of carrying out a polynomial regression on experimental data. As given by I.B.M. it suffered the drawback of only being able to handle up to sixty data points. By modifications to the relevant input dimensions for data in 'PCLRG' and its associated subroutines, it was possible to increase the program's capacity.
so that it was eventually able to treat up to
150 data points. To avoid confusion this modified

group of programs was named 'WLLRG'.

'WLLRG' provided an accurate expression

for the relationship between two variables, an

"Analysis of Variance" table, a table of comparison

between actual and predicted values and their
difference, and a plot of actual values and of
predicted values. The program was especially useful

when linked with the main computer's graph plotter

in that an automatic print out of the relationship

between variables was provided simultaneously with

a plot of the two variables.

Because of its length it is impossible

to reproduce here the 'WLLRG' program. As stated

earlier the program is merely a modification of the

more readily available 'POLRG' program, the
differences between the two merely being the size
of the relevant input dimensions for the amount of
data to be treated. Such modifications were readily
made to 'POLRG' because that program contained
concise details as to the nature of each dimension
statement it contained and noted the function and
inter-relationship of each such statement.
The program 'VLLAS' was used to determine and check relationships derived during the course of the research.
APPENDIX 'D'

AN ACCURATE METHOD FOR NUMERICAL OR GRAPHICAL INTEGRATION

Ellis and Bourne (29) in their discussion on the methods available for testing equilibria data for thermodynamic consistency stressed the need for an accurate method of numerical or graphical integration to be employed in such a situation. This is true not only there but anywhere where the value of an integral quantity is required in calculation. An example of this is equation 5.13 (Chapter 5), which is used to determine the average slope of the equilibrium line.

When it is not convenient or it is impossible to integrate a differential equation, some less exact method may be used as a substitute, which, however, for many cases, will give a sufficiently exact answer. Ferry (67) describes in detail some such alternatives, but in view of the often repetitive nature of many of the integrations required in the research it was felt that a computer program would offer the best and most accurate method of calculation. I.B.M. supply the program 'LATR' in their scientific
subroutine package. Basically it is a program which conducts a graphical integration between upper and lower limits using a modified form of the trapezoidal rule, given an expression for the relationship between the two variables concerned. To provide this expression, where one did not exist, the program described in Appendix 'A' was used.

This method of integration had many features to commend it over other alternative methods, it did, however, require the supply of the function of two variables and it may be argued that this could lead to inaccuracies caused by rounding errors. However, alternative methods would require the construction of curves whose accuracy could be doubtful. The expressions supplied by "ILLOG" were accompanied by an "Analysis of Variance" and a comparison of actual and predicted values, from which it was possible to appreciate the extent of error introduced through "rounding off" or "smoothing". Secondly 'MAT' itself provided a trapezoidal method of integration which was capable of calculation using a far higher degree of subdivision into trapezoidal strips than would otherwise have been possible. This resulted in greater accuracy, and because the program itself required a knowledge of
the degree of accuracy of measurement required it was possible to specify the accuracy of the whole integration technique.

In practice the technique was used in the thermodynamic consistency testing of equilibria data, and in the calculation of the average slope of the equilibrium line. It was, therefore, concerned entirely with integration from curves obtained from equilibria data. Such data had been tested for accuracy and was the most consistent available at the time. Further the integration program was required to work to an accuracy of 0.1%. This accuracy, although ridiculous when one considers the accuracy of the data principally supplied, was selected to gauge the usefulness of the technique. At no time was the program unable to meet the accuracy demanded of it. The degree of error resulting from the use of this technique was believed to negligible, and the technique itself judged to be the most accurate and suitable available. The program itself was supplied by I.S.M.
TABLES
<table>
<thead>
<tr>
<th>System</th>
<th>Col length</th>
<th>Col Diam</th>
<th>Carrier</th>
<th>Details</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
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<td>2m</td>
<td>20$</td>
<td></td>
<td>Squalane / Chromosorb</td>
<td>(129)</td>
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<td>2m</td>
<td>20$</td>
<td></td>
<td>Squalane / Chromosorb</td>
<td>(129)</td>
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<tr>
<td>Chlorobenzene / IDthyTbenzene</td>
<td>2m</td>
<td>20$</td>
<td></td>
<td>Squalane / Chromosorb</td>
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<td>5$</td>
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<td>Apiezon L + Chromosorb P60/80</td>
<td>(10)</td>
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<tr>
<td>trans Decalin / n-Paraffin</td>
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<td>5$</td>
<td></td>
<td>Apiezon L + Chromosorb P60/80</td>
<td>(10)</td>
</tr>
</tbody>
</table>

Reference:
- Huiderweg (029)
- van der Aar (125)
- Zuiderweg (129)
- Sruck et al (110)
- Zuiderweg (129)
- Verzezans (129)

Chromatography Details:
- T,7 degrees C
- 1.8 litres / hr
- 180°C
- 130°C
- 140°C
- 150°C
- 160°C

Pressure, working:
- 760 Torr
- 300, 50, 10, 5
**TABLE 2**

Vapour - Liquid Equilibria for the System

*n* Decane/trans Decalin at 300 Torr.

<table>
<thead>
<tr>
<th>Temp</th>
<th>Mole Fraction</th>
<th>Mole Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Vapour</td>
<td>Liquid</td>
</tr>
<tr>
<td></td>
<td>m.v.c.</td>
<td>m.v.c.</td>
</tr>
<tr>
<td>141.29</td>
<td>0.9546</td>
<td>0.9399</td>
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<tr>
<td>141.80</td>
<td>0.9086</td>
<td>0.8807</td>
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<tr>
<td>142.32</td>
<td>0.8622</td>
<td>0.8228</td>
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<td>142.83</td>
<td>0.8153</td>
<td>0.7661</td>
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<td>0.7105</td>
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<td>0.7201</td>
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<td>0.6028</td>
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<td>144.87</td>
<td>0.6231</td>
<td>0.5505</td>
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<td>0.5738</td>
<td>0.4993</td>
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<td>145.89</td>
<td>0.5241</td>
<td>0.4491</td>
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<td>0.4739</td>
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<tr>
<td>146.91</td>
<td>0.4233</td>
<td>0.3519</td>
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<td>147.42</td>
<td>0.3721</td>
<td>0.3047</td>
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<tr>
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<tr>
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<td>149.46</td>
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<tr>
<td>150.49</td>
<td>0.0548</td>
<td>0.0410</td>
</tr>
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</table>
### Table 3

Column Efficiency Data For the "Pinpek" Column

Using the System Benzene/Toluene at 760 Torr.

<table>
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<tr>
<th>Run No.</th>
<th>$U_t/p_v^{1/2}$</th>
<th>$P$</th>
<th>$n_t$</th>
<th>$P/n_t$</th>
<th>$n_t/H$</th>
<th>$P/H$</th>
</tr>
</thead>
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<tr>
<td>A760/1/P</td>
<td>0.709</td>
<td>1.625</td>
<td>4.24</td>
<td>0.384</td>
<td>3.872</td>
<td>1.464</td>
</tr>
<tr>
<td>A760/2/P</td>
<td>1.015</td>
<td>1.769</td>
<td>2.77</td>
<td>0.623</td>
<td>2.550</td>
<td>1.615</td>
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<tr>
<td>A760/3/P</td>
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<td>1.877</td>
<td>2.93</td>
<td>0.642</td>
<td>2.667</td>
<td>1.714</td>
</tr>
<tr>
<td>A760/4/P</td>
<td>0.880</td>
<td>1.814</td>
<td>2.66</td>
<td>0.681</td>
<td>2.429</td>
<td>1.656</td>
</tr>
<tr>
<td>A760/5/P</td>
<td>0.842</td>
<td>1.775</td>
<td>3.00</td>
<td>0.591</td>
<td>2.740</td>
<td>1.925</td>
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<tr>
<td>A760/6/P</td>
<td>1.774</td>
<td>2.103</td>
<td>2.30</td>
<td>0.916</td>
<td>2.100</td>
<td>1.973</td>
</tr>
<tr>
<td>A760/7/P</td>
<td>1.501</td>
<td>1.953</td>
<td>2.89</td>
<td>0.675</td>
<td>2.639</td>
<td>1.783</td>
</tr>
<tr>
<td>A760/8/P</td>
<td>1.091</td>
<td>1.900</td>
<td>3.50</td>
<td>0.759</td>
<td>2.263</td>
<td>1.734</td>
</tr>
<tr>
<td>A760/9/P</td>
<td>1.939</td>
<td>2.234</td>
<td>2.41</td>
<td>0.926</td>
<td>2.201</td>
<td>2.040</td>
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<tr>
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<td>3.50</td>
<td>0.667</td>
<td>2.374</td>
<td>1.534</td>
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<tr>
<td>A760/11/P</td>
<td>1.377</td>
<td>2.824</td>
<td>2.08</td>
<td>1.357</td>
<td>1.900</td>
<td>2.578</td>
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<tr>
<td>A760/12/P</td>
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<td>1.686</td>
<td>2.04</td>
<td>0.958</td>
<td>1.363</td>
<td>1.76</td>
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<tr>
<td>A760/13/P</td>
<td>1.803</td>
<td>2.035</td>
<td>1.95</td>
<td>1.056</td>
<td>1.763</td>
<td>1.858</td>
</tr>
<tr>
<td>A760/14/P</td>
<td>2.550</td>
<td>2.069</td>
<td>1.96</td>
<td>1.055</td>
<td>1.790</td>
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<tr>
<td>A760/15/P</td>
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<td>0.948</td>
<td>1.821</td>
<td>1.783</td>
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<tr>
<td>A760/16/P</td>
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<tr>
<td>A760/17/P</td>
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<td>2.155</td>
<td>1.71</td>
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<td>1.512</td>
<td>1.968</td>
</tr>
<tr>
<td>A760/18/P</td>
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<td>2.513</td>
<td>2.20</td>
<td>1.222</td>
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<td>1.668</td>
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<tr>
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<td>1.935</td>
<td>2.17</td>
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<td>1.222</td>
<td>1.765</td>
</tr>
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</table>
| A760/20/P | 2.305 | 2.024 | 1.99 | 1.817 | }
### Table 4

Column Efficiency Data For the "Finpak" Column

Using the System n-Heptane/Toluene at 760 Torr

<table>
<thead>
<tr>
<th>Run No.</th>
<th>( \frac{U}{V} )</th>
<th>( t )</th>
<th>( n_t )</th>
<th>( \frac{1}{n_t} )</th>
<th>( n_t / )</th>
<th>( \frac{r}{H} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>J760/1/P</td>
<td>1.749</td>
<td>0.254</td>
<td>2.370</td>
<td>0.127</td>
<td>2.164</td>
<td>0.252</td>
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<tr>
<td>J760/2/P</td>
<td>1.679</td>
<td>0.190</td>
<td>2.911</td>
<td>0.049</td>
<td>3.572</td>
<td>0.174</td>
</tr>
<tr>
<td>J760/3/P</td>
<td>1.313</td>
<td>0.679</td>
<td>3.398</td>
<td>0.206</td>
<td>3.012</td>
<td>0.620</td>
</tr>
<tr>
<td>J760/4/P</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>J760/5/P</td>
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<td>0.061</td>
<td>2.681</td>
<td>0.330</td>
<td>2.394</td>
<td>0.786</td>
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<tr>
<td>J760/6/P</td>
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<td>0.718</td>
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<td>0.291</td>
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<tr>
<td>J760/7/P</td>
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<td>3.678</td>
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<td>0.726</td>
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<td>J760/8/P</td>
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<td></td>
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</tr>
<tr>
<td>J760/9/P</td>
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<td>0.336</td>
<td>2.647</td>
<td>0.626</td>
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<tr>
<td>J760/10/P</td>
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<tr>
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<tr>
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<tr>
<td>J760/21/P</td>
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<td>0.282</td>
<td>3.200</td>
<td>0.928</td>
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<tr>
<td>J760/22/P</td>
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<td>3.665</td>
<td>0.346</td>
<td>2.799</td>
<td>0.968</td>
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<tr>
<td>J760/23/P</td>
<td>2.197</td>
<td>1.113</td>
<td>3.506</td>
<td>0.357</td>
<td>3.017</td>
<td>1.016</td>
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<td>( P )</td>
<td>( n_t )</td>
<td>( F/n_t )</td>
<td>( n_t/H )</td>
<td>( P/H )</td>
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</tr>
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<td>1.530</td>
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<td>0.123</td>
<td>5.676</td>
<td>0.490</td>
</tr>
<tr>
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<td>0.698</td>
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<td>5.206</td>
<td>0.096</td>
<td>4.754</td>
<td>0.456</td>
</tr>
<tr>
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<td>2.323</td>
<td>0.222</td>
<td>2.577</td>
<td>0.572</td>
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<td>B760/32/P</td>
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<td>1.395</td>
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<td>4.127</td>
<td>0.197</td>
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<td>0.741</td>
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<td>2.437</td>
<td>0.417</td>
<td>3.139</td>
<td>1.309</td>
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<td>0.396</td>
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<td>4.214</td>
<td>0.123</td>
<td>4.326</td>
<td>0.542</td>
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<td>1.167</td>
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<td>4.271</td>
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<td>2.505</td>
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<td>2.883</td>
<td>1.629</td>
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</table>

* Runs B760/1/P & B760/3/P - Samples Contaminated
** Run B760/25/P - Inaccurate Analysis.
### Table 5

**Column Efficiency Data For The "Finpak" Column**

Using the system Chlorobenzene/ethylbenzene at 760 Torr.

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<th>Run</th>
<th>$U_{y/\nu}$</th>
<th>$P$</th>
<th>$n_t$</th>
<th>$P/n_t$</th>
<th>$n_t/H$</th>
<th>$F/H$</th>
</tr>
</thead>
<tbody>
<tr>
<td>C760/1/P</td>
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<td>2.223</td>
<td>2.085</td>
<td>1.066</td>
<td>1.904</td>
<td>2.030</td>
</tr>
<tr>
<td>C760/2/P</td>
<td>1.254</td>
<td>1.341</td>
<td>3.22</td>
<td>0.416</td>
<td>2.941</td>
<td>1.225</td>
</tr>
<tr>
<td>C760/3/P</td>
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<td>2.682</td>
<td>0.629</td>
<td>2.449</td>
<td>1.540</td>
</tr>
<tr>
<td>C760/4/P</td>
<td>2.095</td>
<td>1.145</td>
<td>3.01</td>
<td>0.380</td>
<td>2.749</td>
<td>1.046</td>
</tr>
<tr>
<td>C760/5/P</td>
<td>1.464</td>
<td>1.271</td>
<td>2.56</td>
<td>0.496</td>
<td>2.338</td>
<td>1.161</td>
</tr>
<tr>
<td>Run No.</td>
<td>( \frac{U_p}{v} )</td>
<td>P</td>
<td>( n_t )</td>
<td>( P/n_t )</td>
<td>( n_t/H )</td>
<td>P/H</td>
</tr>
<tr>
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<td>----------------------</td>
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<td>---------</td>
<td>---------</td>
<td>-----</td>
</tr>
<tr>
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<td>3.153</td>
<td>1.040</td>
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<tr>
<td>C300/2/P</td>
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<td>3.309</td>
<td>1.011</td>
<td>3.095</td>
<td>3.128</td>
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<tr>
<td>C300/3/P</td>
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<td><strong>3.472</strong></td>
<td>3.438</td>
<td>1.010</td>
<td>3.138</td>
<td>3.171</td>
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<tr>
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<tr>
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<td>1.132</td>
<td>2.560</td>
<td>2.697</td>
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<td>0.734</td>
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<td>2.603</td>
</tr>
<tr>
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<td>2.508</td>
<td>2.198</td>
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<tr>
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<td>1.096</td>
<td>2.947</td>
<td>2.331</td>
</tr>
<tr>
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<tr>
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<td>$U_{v/p}^{1/2}$</td>
<td>F</td>
<td>$n_t$</td>
<td>$P/n_t$</td>
<td>$n_t/H$</td>
<td>F/H</td>
</tr>
<tr>
<td>----------</td>
<td>----------------</td>
<td>---------</td>
<td>--------</td>
<td>---------</td>
<td>---------</td>
<td>--------</td>
</tr>
<tr>
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<td>3.704</td>
<td>4.238</td>
<td>3.383</td>
<td>14.524</td>
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<td>15.115</td>
<td>3.090</td>
<td>4.893</td>
<td>2.882</td>
<td>13.805</td>
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<tr>
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<td>15.566</td>
<td>3.344</td>
<td>4.655</td>
<td>3.004</td>
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## COLUMN EFFICIENCY DATA FOR THE "FINPAN" COLUMN

### Using System n Decane/Trans Decalin at 300 Torr

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<th>Run No.</th>
<th>$U_{v}^{1/3}$</th>
<th>P</th>
<th>n$_t$</th>
<th>I/n$_t$</th>
<th>n$_t$/H</th>
<th>P/H</th>
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</thead>
<tbody>
<tr>
<td>D300/1/P</td>
<td>1.363</td>
<td>1.467</td>
<td>2.456</td>
<td>0.573</td>
<td>2.242</td>
<td>1.204</td>
</tr>
<tr>
<td>D300/2/P</td>
<td>2.572</td>
<td>2.270</td>
<td>2.425</td>
<td>0.936</td>
<td>2.214</td>
<td>2.073</td>
</tr>
<tr>
<td>D300/3/P</td>
<td>1.765</td>
<td>1.516</td>
<td>2.201</td>
<td>0.689</td>
<td>2.010</td>
<td>1.304</td>
</tr>
<tr>
<td>D300/4/P</td>
<td>2.175</td>
<td>1.572</td>
<td>2.311</td>
<td>0.711</td>
<td>2.019</td>
<td>1.435</td>
</tr>
<tr>
<td>D300/5/P</td>
<td>1.645</td>
<td>1.409</td>
<td>2.264</td>
<td>0.648</td>
<td>2.012</td>
<td>1.365</td>
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<td>2.106</td>
<td>0.830</td>
<td>1.921</td>
<td>1.595</td>
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<td>2.065</td>
<td>0.823</td>
<td>1.921</td>
<td>1.595</td>
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<td>0.649</td>
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<td>3.474</td>
<td>0.337</td>
<td>3.172</td>
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<td>0.549</td>
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<td>0.661</td>
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<td>0.556</td>
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<td>2.325</td>
<td>1.700</td>
<td>2.629</td>
<td>0.700</td>
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<td>2.551</td>
<td>1.057</td>
<td>2.329</td>
<td>2.463</td>
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Table 9

Column Efficiency Data For The "Finpak" Column

Using The System n Decane/trans Decalin at 50 Torr.

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<th>( u_{y} / P )</th>
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<th>( P/n_t )</th>
<th>( n_t/H )</th>
<th>( F/H )</th>
</tr>
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<tbody>
<tr>
<td>D50/1/P</td>
<td>2.393</td>
<td>4.573</td>
<td>3.757</td>
<td>1.217</td>
<td>3.431</td>
<td>4.176</td>
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<td>D50/2/P</td>
<td>4.236</td>
<td>5.574</td>
<td>3.534</td>
<td>1.534</td>
<td>2.318</td>
<td>5.090</td>
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<td>D50/3/P</td>
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<td>5.724</td>
<td>2.709</td>
<td>2.113</td>
<td>2.473</td>
<td>5.327</td>
</tr>
<tr>
<td>D50/4/P</td>
<td>2.954</td>
<td>5.032</td>
<td>2.028</td>
<td>1.719</td>
<td>2.673</td>
<td>4.595</td>
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<td>1.506</td>
<td>2.834</td>
<td>4.266</td>
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<td>2.753</td>
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<td>1.792</td>
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<td>1.341</td>
<td>3.175</td>
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<td>3.280</td>
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### Table 10

Column Efficiency Data for the "Finpak" Column

Using the System trans-Decalin/cis-Decalin at 100 Torr

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<th>$U_{\text{PP}}$</th>
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<th>$n_t/^c$</th>
<th>$P/H$</th>
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<td>E10C/2/P</td>
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<td>3.811</td>
<td>1.974</td>
<td>1.031</td>
<td>3.430</td>
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<td>1.640</td>
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<td>2.232</td>
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<td>2.038</td>
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<tr>
<td>E10C/6/P</td>
<td>2.237</td>
<td>3.676</td>
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### Table 11

Colomn Efficiency Data for the "Flinsen" COLUMN

Using The System trans Decalin/cis Decalin at 50 Torr

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* Runs B760/4/P & B760/8/P - Samples Contaminated.

** Run B760/25/P - Inaccurate Analysis.
TABLE 14

Pressure Drop, Friction Factor & Vapour Phase Reynold's Numbers Data For Runs Using The System Chlorobenzene/Ethylbenzene at 760 Torr.

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### Table 15

Pressure Drop, Friction Factor & Vapour Phase Reynolds's

Numbers Data For Runs Using The System Chlorobenzene/

Ethylbenzene At 300 Torr.

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Table 16

Pressure Drop, Friction Factor & Vapour Phase Reynolds's Numbers Data for Runs: Using the System Chlorobenzene/Ethylbenzene at 50 Torr

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**Table 18**

**Pressure Drop, Friction Factor & Vapour Phase Reynold's Numbers Data For Runs Using The System n Decane/trans Decalin At 50 Torr**

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<td>Friction Factor</td>
<td>Reynold's No. Vapour Phase</td>
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### TABLE 21

Overall & Phase Resistances Expressed As H.T.U.

Flow Conditions, Slope of Equilibrium Line, Surface Tension & % Resistance Liquid Phase For Runs Using The System Benzene/Toluene At 760 Torr

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<th>Re_{L}</th>
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<th>HoL</th>
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<th>M_{L} Theor</th>
<th>m</th>
<th>Base</th>
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TABLE 22

Overall & Phase Resistances Expressed As H.T.U.

Flow Conditions, Slope of Equilibrium Line, Surface Tension & % Resistance Liquid Phase For Runs Using The System n-Heptane/Toluene At 760 Torr

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<th>σ</th>
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### TABLE 23

Overall & Phase Resistances Expressed As H.T.U.

**Flow Conditions, Slope of Equilibrium Line, Surface Tension & % Resistance Liquid Phase For Runs Using The System Chlorobenzene/Ethylbenzene At 760 Torr.**

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Overall Phase Resistances Expressed As H.T.U.


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## TABLE 25

**Overall Phase Resistances Expressed As H.T.U.**

**Flow Conditions, Slope of Equilibrium Line, Surface Tension & % Resistance Liquid Phase For Runs Using The System Chlorobenzene/Ethylbenzene At 50 Torr**

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Overall & Phase Resistances Expressed As H.T.U.


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Overall & Phase Resistances Expressed As H.T.U.

Flow Conditions, Slope Of Equilibrium Line, Surface Tension & % Resistance Liquid Phase For Runs Using The System trans Decalin/cis Decalin At 100 Torr

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Overall & Phase Resistances Expressed as H.T.U.

Levels Conditions, Slope of Equilibrium Line, Surface Tension & % Resistance Liquid Phase For Runs Using The System trans Decalin/cis Decalin at 50 Torr

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|----------|----|----|-----|-----|-----|------|-------|-----|------|-----
| E50/1/P  | 685| 6  | 42.6| 42.8| 34.5| 21.2 | 0.996 | 23.2| 80.6 |
| E50/2/P  | 876| 7.9| 39.9| 40.1| 27.7| 24.8 | 0.995 | 23.2| 69.2 |
| E50/3/P  | 1183| 9.9| 45.6| 44.7| 25.2| 30.8 | 1.021 | 23.4| 56.3 |
| E50/4/P  | 1141| 10.0| 45.8| 45.8| 27.6| 29.8 | 0.999 | 23.2| 58.9 |
| E50/5/P  | 750| 6.9| 33.0| 33.4| 23.8| 22.2 | 0.937 | 23.1| 71.0 |
| E50/6/P  | 972| 8.5| 46.5| 46.9| 32.4| 27.0 | 0.992 | 23.1| 60.7 |
| E50/7/P  | 1130| 10.2| 46.3| 46.5| 25.5| 30.8 | 0.996 | 23.2| 57.1 |
| E50/8/P  | 1320| 10.3| 48.1| 47.5| 24.0| 34.0 | 1.013 | 23.3| 50.6 |
| E50/9/P  | 937| 8.5| 43.5| 43.8| 29.9| 30.0 | 0.992 | 23.1| 63.5 |