PERFORMANCE STUDIES ON A LIQUID-LIQUID CONTACTOR
WITH OSCILLATING MULTIPLE BAFFLES

Thesis submitted for the degree of Doctor of
Philosophy in the Faculty of Engineering of the
University of Surrey

by

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1982
TO MY

MOTHER

A REMARKABLE WOMAN
Performance Studies on a liquid-liquid Contactor with oscillating multiple baffles are reported. Two three component liquid systems - CCL\textsubscript{4} - HAC - H\textsubscript{2}O and MIBK - HAC - H\textsubscript{2}O - were studied.

The mass transfer efficiency of the contactor have been evaluated with respect to the variation of operating conditions; flow ratio and solute concentration in the feed phase. Empirical correlations for the overall mass transfer coefficient have been proposed.

Drop size studies based on photographic techniques are reported. Sauter mean drop sizes were obtained from the photographs for a wide range of operating conditions. Using this data and the data for the dispersed phase hold-up, obtained by the manometric method, the interfacial area of the dispersion was calculated for the same operating conditions.

Axial mixing properties were investigated for single phase flow using a dye injection technique. Residence time and axial mixing parameters were obtained, and their significance discussed. A correlation for the axial mixing coefficient has been proposed.

The Miyauchi and Vermulen type one dimensional diffusion equation for predicting Number of Transfer Units was solved analytically and numerically. The limitation associated with the use of this equation especially with respect to (a) variation of NTU along the column length, and (b) boundary conditions has been discussed.

The maximum operating conditions have been investigated for a number of operating conditions, and the effect of solute concentration in the feed phase on maximum operating conditions have been demonstrated.
The power consumption characteristics have been investigated and correlation for total mixing power $P_M$ (i.e. excluding dry power), have been proposed. Due to a proportion of unswept volume, correlations for total mixing power $P_M$, total mixing power per unit active volume $P_{Va}$ and total mixing power per unit swept volume $P_{Vs}$, have been proposed.

Mass transfer and power studies are also reported for a smaller contactor with a single oscillating baffle. The experimental results of the two contactors have been compared for scale-up purposes.
Acknowledgments

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I wish to thank Mr John Darby and other members of the Audio-Visual Aids Unit for their help in photographing the drops and components in the extraction unit.

Special thanks to my wife Dolly who encouraged this work and assisted materially by typing the thesis.
CONTENTS

CHAPTER 1

INTRODUCTION

1.1 Basic Design Considerations For Liquid-Liquid Contactors. 1.2

1.2 Mass Transfer Rates. 1.3

1.2.1 Mass Transfer Theories 1.3

1.2.2 Mass Transfer Rate Equations 1.4

1.2.3 Mass Transfer Coefficients 1.5

1.2.4 Interfacial Area 1.6

1.3 Drop Dynamics 1.6

1.4 Axial Mixing 1.7

1.5 Dispersed Phase Hold-up 1.8

1.6 Maximum Operating Conditions - Flooding 1.9

1.7 Power Consumption in Liquid Mixing 1.9

1.8 Programme of the Research 1.10
# CHAPTER 2

## LITERATURE SURVEY

<table>
<thead>
<tr>
<th>Section</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1</td>
<td>Liquid-Liquid Contactors</td>
<td>2.2</td>
</tr>
<tr>
<td>2.1.1</td>
<td>Discrete Stage Contactors</td>
<td>2.2</td>
</tr>
<tr>
<td>2.1.2</td>
<td>Continuous Differential Contactors</td>
<td>2.3</td>
</tr>
<tr>
<td>2.2</td>
<td>Mass Transfer Studies in Extractors</td>
<td>2.4</td>
</tr>
<tr>
<td>2.2.1</td>
<td>Spray Columns</td>
<td>2.4</td>
</tr>
<tr>
<td>2.2.2</td>
<td>Rotating Disc Contactors</td>
<td>2.5</td>
</tr>
<tr>
<td>2.2.3</td>
<td>Pulsed Plate Extractors</td>
<td>2.5</td>
</tr>
<tr>
<td>2.3</td>
<td>Drop Studies</td>
<td>2.6</td>
</tr>
<tr>
<td>2.3.1</td>
<td>Drop Size and Size Distribution</td>
<td>2.6</td>
</tr>
<tr>
<td>2.3.2</td>
<td>Interfacial Area Measurements</td>
<td>2.7</td>
</tr>
<tr>
<td>2.3.3</td>
<td>Movement and Behaviour</td>
<td>2.9</td>
</tr>
<tr>
<td>2.3.3.1</td>
<td>Drop Velocities</td>
<td>2.9</td>
</tr>
<tr>
<td>2.3.3.2</td>
<td>Drop Interaction</td>
<td>2.10</td>
</tr>
<tr>
<td>2.3.4</td>
<td>Mass Transfer to and from Droplets</td>
<td>2.13</td>
</tr>
<tr>
<td>2.3.4.1</td>
<td>Mass Transfer in the Dispersed Phase</td>
<td>2.13</td>
</tr>
<tr>
<td>2.3.4.2</td>
<td>Mass Transfer in the Continuous Phase</td>
<td>2.15</td>
</tr>
<tr>
<td>2.3.4.3</td>
<td>Mass Transfer in Drop Swarms</td>
<td>2.16</td>
</tr>
<tr>
<td>2.3.5</td>
<td>Interfacial Turbulence</td>
<td>2.17</td>
</tr>
<tr>
<td>2.4</td>
<td>Axial Mixing</td>
<td>2.18</td>
</tr>
<tr>
<td>2.5</td>
<td>Hold-up Studies</td>
<td>2.20</td>
</tr>
<tr>
<td>2.6</td>
<td>Flooding Studies</td>
<td>2.21</td>
</tr>
<tr>
<td>2.7</td>
<td>Power Studies</td>
<td>2.23</td>
</tr>
<tr>
<td>Section</td>
<td>Title</td>
<td>Page</td>
</tr>
<tr>
<td>---------</td>
<td>-------</td>
<td>------</td>
</tr>
<tr>
<td>3.1.1</td>
<td>Equilibrium</td>
<td>3.2</td>
</tr>
<tr>
<td>3.1.2</td>
<td>Concentration Profile Based on Piston Flow Assumption</td>
<td>3.3</td>
</tr>
<tr>
<td>3.1.3</td>
<td>Number of Transfer Units - NTU</td>
<td>3.5</td>
</tr>
<tr>
<td>3.1.4</td>
<td>Height of Transfer Units - HTU</td>
<td>3.7</td>
</tr>
<tr>
<td>3.1.5</td>
<td>True Value of NTU</td>
<td>3.7</td>
</tr>
<tr>
<td>3.1.6</td>
<td>Measured and Apparent Values of NTU</td>
<td>3.7</td>
</tr>
<tr>
<td>3.1.7</td>
<td>Piston Flow Values of NTU</td>
<td>3.9</td>
</tr>
<tr>
<td>3.2.1</td>
<td>Types of Flow Patterns</td>
<td>3.9</td>
</tr>
<tr>
<td>3.2.2</td>
<td>Age Distribution Functions and F.Diagram</td>
<td>3.10</td>
</tr>
<tr>
<td>3.2.3</td>
<td>Analysis of Age-Distribution Functions</td>
<td>3.14</td>
</tr>
<tr>
<td>3.2.4</td>
<td>Model for Axial Mixing in Single Phase Flow</td>
<td>3.16</td>
</tr>
<tr>
<td>3.2.5</td>
<td>Peclet Number</td>
<td>3.20</td>
</tr>
<tr>
<td>3.2.6</td>
<td>Determination of Axial Mixing Parameters from R.T.D Studies</td>
<td>3.20</td>
</tr>
<tr>
<td>3.3.1</td>
<td>Concentration Profiles</td>
<td>3.23</td>
</tr>
<tr>
<td>3.3.2</td>
<td>Diffusion Model for Mass Transfer in Countercurrent Continuous Flow Operations</td>
<td>3.24</td>
</tr>
<tr>
<td>3.3.3</td>
<td>Interpretation of the Diffusion Model</td>
<td>3.28</td>
</tr>
<tr>
<td>3.3.4</td>
<td>Solutions to the Diffusion Model</td>
<td>3.30</td>
</tr>
<tr>
<td>3.4</td>
<td>Sauter Mean Drop Diameter, Interfacial Area and Hold-up</td>
<td>3.31</td>
</tr>
<tr>
<td>3.5.1</td>
<td>Speed Parameter for O.B.C</td>
<td>3.33</td>
</tr>
<tr>
<td>3.5.2</td>
<td>Power Correlations</td>
<td>3.34</td>
</tr>
</tbody>
</table>
CHAPTER 4

DESCRIPTION OF EXPERIMENTAL EQUIPMENT

4.1 Experimental Contactor 4.2
4.2 Pilot Plant 4.9
   4.2.1 General Description 4.9
   4.2.2 Baffle Drive 4.13
   4.2.3 Manometer 4.17
4.3 Equipment For Dye Study 4.17
   4.3.1 Photocell Unit 4.17
   4.3.2 Data Logger 4.19
   4.3.3 Tracers Used 4.19
4.4 Equipment For Power Measurement 4.19
   4.4.1 Torque Transducers 4.20
   4.4.2 Torque Indicator 4.20
   4.4.3 Torque Integrator Unit 4.20
   4.4.4 Shaft Speed Measurement 4.24
4.5 Refractometer 4.24
4.6 Particle Size Analyser 4.26
4.7 Tensiometer For Interfacial Tension Measurement 4.26
4.8 Stirrer For Mixing Solute And Solvent 4.26
4.9 Rotameters 4.27
4.10 Chemicals Used 4.27
CHAPTER 5

EXPERIMENTAL TECHNIQUE

5.1 General 5.1
5.2 Calibration of Instruments 5.2
  5.2.1 Kopp Speed Variator 5.2
  5.2.2 Refractometer 5.2
5.3 Droplet Studies 5.3
  5.3.1 Photography 5.3
  5.3.2 Counting and Sizing of Droplets 5.13
5.4 Mass Transfer Studies 5.14
  5.4.1 Preparation and Sampling 5.14
  5.4.2 Sample Analysis 5.17
    5.4.2.1 Refractometry 5.17
    5.4.2.2 Titration 5.17
5.5 Hold-up Measurement 5.18
  5.5.1 Manometric Method 5.18
  5.5.2 Setting Method 5.18
5.6 Power Measurement 5.19
5.7 Dye Injection Studies 5.20
5.8 Flooding Studies 5.20
CHAPTER 6

DIMENSIONAL ANALYSIS

6.1 Correlation for Sauter Mean Drop Diameter

6.2 Correlation for Overall Mass Transfer Coefficient

6.3 Correlation for Axial Dispersion Coefficient

6.4 Power Correlations for O.B.C
# CHAPTER 7

**RESULTS**

<table>
<thead>
<tr>
<th>Section</th>
<th>Page No</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.1 Experimental Results</td>
<td>7.2</td>
</tr>
<tr>
<td>7.1.1 Solute Concentration Profiles</td>
<td></td>
</tr>
<tr>
<td>7.1.2 Sauter Mean Drop Diameter and Interfacial Area</td>
<td></td>
</tr>
<tr>
<td>7.1.3 Contactor Overall Performance - Mass Transfer</td>
<td>7.3</td>
</tr>
<tr>
<td>7.1.4 Dispersed Phase Hold-up</td>
<td>7.3</td>
</tr>
<tr>
<td>7.1.5 Flooding</td>
<td>7.4</td>
</tr>
<tr>
<td>7.1.6 Residence Time and Axial Mixing</td>
<td>7.4</td>
</tr>
<tr>
<td>7.1.7 Power Study</td>
<td>7.5</td>
</tr>
<tr>
<td>Graphical Representation of Experimental Results</td>
<td>7.6-7.60</td>
</tr>
<tr>
<td>7.2 Theoretical Results</td>
<td>7.61</td>
</tr>
<tr>
<td>7.2.1 Concentration Profile from Diffusion Model</td>
<td>7.62</td>
</tr>
<tr>
<td>7.2.2 Calculation of Axial Mixing Coefficients from the Diffusion Model</td>
<td>7.63</td>
</tr>
<tr>
<td>7.3 Statistical Results</td>
<td>7.66</td>
</tr>
<tr>
<td>7.3.1 Correlation for Sauter Mean Drop Diameter</td>
<td>7.66</td>
</tr>
<tr>
<td>7.3.2 Correlation for Overall Mass Transfer Coefficients</td>
<td>7.67</td>
</tr>
<tr>
<td>7.3.3 Correlation for Axial Dispersion Coefficient</td>
<td>7.68</td>
</tr>
<tr>
<td>7.3.4 Power Correlations for O.B.C</td>
<td>7.71</td>
</tr>
</tbody>
</table>
CHAPTER 8

DISCUSSION

8.1 Hydrodynamic Results
   8.1.1 Drop Size Distribution
   8.1.2 Sauter Mean Drop Diameter
   8.1.3 Dispersed Phase Hold-up
   8.1.4 Interfacial Area

8.2 Mass Transfer Results
   8.2.1 Solute Concentration Profiles
   8.2.2 Effect of Operating Condition on Contactor Performance
   8.2.3 Empirical Correlation for $K_{Ca}$

8.3 Axial Mixing
   8.3.1 Residence Time and Axial Mixing Parameters
   8.3.2 Empirical Correlation for Axial Mixing Coefficient
   8.3.3 Effect of Axial Mixing on Mass Transfer - One Dimensional Diffusion Model
   8.3.4 Interpretation of the Diffusion Model

8.4 Maximum Operating Conditions

8.5 Power Studies
   8.5.1 Total Power
   8.5.2 Power Per Unit Volume
   8.5.3 Power Correlations
CHAPTER 9

CONCLUSIONS

REFERENCES

NOMENCLATURE

APPENDIX 1

Experimental Data

A.1.1 Physical Properties of the Chemicals Used
A.1.2 Equilibrium Data
A.1.3 Interfacial Tension Data
A.1.4 Refractive Index - Solute Concentration Calibration Charts
A.1.5 Determination of the Mean position along column length for carrying out drop size studies by photography

APPENDIX 2

Methods used for determining dispersed phase hold-up

A.2.1 Manometric Method
A.2.2 Displacement Method

APPENDIX 3

Theoretical Analysis

A.3.1 Solutions to the One-Dimensional Diffusion Equation
A.3.1.1 Analytical Solution
A.3.1.2 Numerical Solution

A.3.2 Calculation of Axial Mixing Coefficient from the One-Dimensional Diffusion Equation

A.3.3 List of Correlations for Mass Transfer Coefficients

APPENDIX 4

Computation of Results and Computer Programs

A.4.1 Mass Transfer Results
A.4.2 Residence Time and Axial Mixing
A.4.3 Runge-Kutta Numerical Method

APPENDIX 5

TABLES
# LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure No.</th>
<th>Description</th>
<th>Page No</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td><strong>CHAPTER 3</strong></td>
<td></td>
</tr>
<tr>
<td>3.1</td>
<td>Piston Flow Model for Evaluation of Extractor Efficiency</td>
<td>3.4</td>
</tr>
<tr>
<td>3.2</td>
<td>RTD Curve for Non-Ideal Flow</td>
<td>3.11</td>
</tr>
<tr>
<td>3.3</td>
<td>Typical C and F Diagrams for Ideal and Non-Ideal Flow</td>
<td></td>
</tr>
<tr>
<td>3.4</td>
<td>C-Curves for Large Values of Dispersion Numbers</td>
<td>3.19</td>
</tr>
<tr>
<td>3.5</td>
<td>C-Curves for Small Values of Dispersion Numbers</td>
<td>3.19</td>
</tr>
<tr>
<td>3.6</td>
<td>Effect of Axial Mixing on Concentration Profile</td>
<td>3.23</td>
</tr>
<tr>
<td>3.7</td>
<td>Schematic Diagram of Diffusion Model for OBC</td>
<td>3.25</td>
</tr>
<tr>
<td>3.8</td>
<td>Depletion and Accumulation for Solute Transferring in Feed Phase</td>
<td>3.29</td>
</tr>
<tr>
<td>3.9</td>
<td>Schematic Diagram for Determining Hold-up</td>
<td>3.32</td>
</tr>
<tr>
<td></td>
<td><strong>CHAPTER 4</strong></td>
<td></td>
</tr>
<tr>
<td>4.1</td>
<td>The Oscillating Baffle Contactors</td>
<td>4.4</td>
</tr>
<tr>
<td>4.1 (a)</td>
<td>Multiple Baffle Contactor (Large)</td>
<td></td>
</tr>
<tr>
<td>4.1 (b)</td>
<td>Complete Baffle</td>
<td></td>
</tr>
<tr>
<td>4.1 (c)</td>
<td>Single Baffle Contactor (Small)</td>
<td></td>
</tr>
<tr>
<td>4.2</td>
<td>Spacer Plates</td>
<td>4.6</td>
</tr>
<tr>
<td></td>
<td>(a) Top Plate</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(b) Bottom Plate</td>
<td></td>
</tr>
<tr>
<td>4.3a</td>
<td>Sketch of Contactor showing sampling points</td>
<td>4.7</td>
</tr>
<tr>
<td>4.3b</td>
<td>Section of Extraction Zone showing sampling unit</td>
<td>4.8</td>
</tr>
<tr>
<td>4.4</td>
<td>Pilot Plant</td>
<td>4.10</td>
</tr>
<tr>
<td>Figure No.</td>
<td>Description</td>
<td>Page No</td>
</tr>
<tr>
<td>-----------</td>
<td>-------------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>4.5</td>
<td>Flow Diagram</td>
<td>4.11</td>
</tr>
<tr>
<td>4.6</td>
<td>Bell Crank Assembly</td>
<td>4.15</td>
</tr>
<tr>
<td>4.7</td>
<td>Gear Box Unit</td>
<td>4.16</td>
</tr>
<tr>
<td></td>
<td>(a) Inside</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(b) Overall View</td>
<td></td>
</tr>
<tr>
<td>4.8</td>
<td>Dye Study Equipment</td>
<td>4.18</td>
</tr>
<tr>
<td></td>
<td>(a) Photocell Unit</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(b) Data Logger</td>
<td></td>
</tr>
<tr>
<td>4.9</td>
<td>Power Measurement Instruments</td>
<td>4.21</td>
</tr>
<tr>
<td></td>
<td>(a) Torque Meter</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(b) Torque Integrator</td>
<td></td>
</tr>
<tr>
<td>4.10</td>
<td>Circuit Diagram for Torque Integrator</td>
<td>4.22</td>
</tr>
<tr>
<td>4.11</td>
<td>Particle Size Analyser</td>
<td>4.25</td>
</tr>
</tbody>
</table>

**CHAPTER 5**

5.1 Layout of Photographic System 5.5

5.2 Photographs of Droplet Studies for 5.6
CCL₄-HAC-H₂O system, Contactor I \( R = 1 \)

5.3 Actual Size Photograph Used for Droplet 5.8
Analysis CCL₄-HAC-H₂O system. Contactor I \( N = 80 \text{ opm} \), \( Q_c = Q_d = 400 \text{ l/hr} \)

5.4 Photographs of Droplet Studies for 5.9
MIBK-HAC-H₂O system. Contactor I

5.5 Actual Size Photograph Used for Droplet 5.10
Size Analysis MIBK-HAC-H₂O system.
Contactor I \( N = 65 \text{ opm} \), \( Q_c = Q_d = 350 \text{ l/hr} \)

5.6 Photographs Showing the Effect of Solute Concentration on Droplet Density, 5.11
MIBK-HAC-H₂O, system, \( R = 1 \), Contactor I
<table>
<thead>
<tr>
<th>Figure No.</th>
<th>Description</th>
<th>Page No</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.7</td>
<td>Photographs of Droplet Studies for CCL\textsubscript{4}-HAC-H\textsubscript{2}O system, Contactor II</td>
<td>5.12</td>
</tr>
</tbody>
</table>

**CHAPTER 7**

<table>
<thead>
<tr>
<th>Section</th>
<th>Description</th>
<th>Page No</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.1.1</td>
<td>Concentration Profile of Continuous Phase for CCL\textsubscript{4}-HAC-H\textsubscript{2}O (System 2) at Constant Baffle Speed. N = 100 opm R = 1 Contactor I</td>
<td>7.6</td>
</tr>
<tr>
<td>7.1.2</td>
<td>Concentration Profile of Continuous Phase for CCL\textsubscript{4}-HAC-H\textsubscript{2}O system Q = 400 l/hr R = 1 Contactor I</td>
<td>7.7</td>
</tr>
<tr>
<td>7.1.3</td>
<td>Concentration Profile of Continuous Phase for CCL\textsubscript{4}-HAC-H\textsubscript{2}O system at varying Flow-ratio Q\textsubscript{d} = 400 l/hr, N = 100 opm Contactor I</td>
<td>7.8</td>
</tr>
<tr>
<td>7.1.4</td>
<td>Concentration Profile of Continuous Phase for CCL\textsubscript{4}-HAC-H\textsubscript{2}O system at varying Inlet Solute Concentration Q\textsubscript{c} = 400 l/hr R = 1, N = 100 opm Contactor I</td>
<td>7.9</td>
</tr>
<tr>
<td>7.1.5</td>
<td>Concentration Profiles of Continuous Phase for MIBK- Acetic Acid - Water System at Constant Baffle Speed Column I</td>
<td>7.10</td>
</tr>
<tr>
<td>7.1.6</td>
<td>Concentration Profiles of Continuous Phase for MIBK - Acetic Acid - Water System at Constant Flow Rate, Column I Q\textsubscript{c} = Q\textsubscript{d} = 350 l/hr</td>
<td>7.11</td>
</tr>
<tr>
<td>7.1.7</td>
<td>Concentration Profile of Continuous Phase at Constant Oscillation Rate R = 1, N = 80 opm, CCL\textsubscript{4}-HAC-H\textsubscript{2}O system Contactor II</td>
<td>7.12</td>
</tr>
<tr>
<td>Figure No.</td>
<td>Description</td>
<td>Page No</td>
</tr>
<tr>
<td>------------</td>
<td>-----------------------------------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>7.1.8</td>
<td>Concentration Profile of Continuous Phase at Constant Phase Flowrate R = 1</td>
<td>7.13</td>
</tr>
<tr>
<td></td>
<td>Q = 100 1/hr, CCL₄-HAC-H₂O system Contactor II</td>
<td></td>
</tr>
<tr>
<td>7.1.9</td>
<td>Comparison of Concentration Profiles for Contactors I and II, CCL₄-HAC-H₂O</td>
<td>7.14</td>
</tr>
<tr>
<td></td>
<td>R = 1, Q = 100 1/hr, N = 80 opm</td>
<td></td>
</tr>
<tr>
<td>7.2.1</td>
<td>Frequency Distribution Curves of Droplets at Different Oscillation Rates,</td>
<td>7.15</td>
</tr>
<tr>
<td></td>
<td>Contactor II R = 1, Q = 100 1/hr, CCL₄-HAC-H₂O system</td>
<td></td>
</tr>
<tr>
<td>7.2.2</td>
<td>Frequency Distribution of Droplets R = 1, Q = 350 1/hr MIBK-HAC-H₂O system</td>
<td>7.16</td>
</tr>
<tr>
<td></td>
<td>Contactor I</td>
<td></td>
</tr>
<tr>
<td>7.2.3</td>
<td>Variation of Interfacial Area with Oscillation Speed, R = 1</td>
<td>7.17</td>
</tr>
<tr>
<td></td>
<td>Contactor I, CCL₄-HAC-H₂O system</td>
<td></td>
</tr>
<tr>
<td>7.2.4</td>
<td>Variation of Interfacial Area with Dispersed Phase Hold-up for Contactor I</td>
<td>7.18</td>
</tr>
<tr>
<td></td>
<td>CCL₄-HAC-H₂O system</td>
<td></td>
</tr>
<tr>
<td>7.2.5</td>
<td>Variation of Measured Interfacial Area with Oscillation Rate R = 1, Q=100 1/hr</td>
<td>7.19</td>
</tr>
<tr>
<td></td>
<td>CCL₄-HAC-H₂O system Contactor II(Small)</td>
<td></td>
</tr>
<tr>
<td>7.2.6</td>
<td>Variation of Measured Interfacial Area with Oscillation Rate R = 1, Q=350 1/hr</td>
<td>7.20</td>
</tr>
<tr>
<td></td>
<td>MIBK-HAC-H₂O system Contactor I</td>
<td></td>
</tr>
<tr>
<td>7.2.7</td>
<td>Variation of Measured Interfacial Area with Phase Flowrate, R = 1, N = 70 opm</td>
<td>7.20</td>
</tr>
<tr>
<td></td>
<td>MIBK-HAC-H₂O system Contactor I</td>
<td></td>
</tr>
<tr>
<td>7.2.8</td>
<td>Comparison of the Variation of Measured Interfacial Area with Oscillation</td>
<td>7.21</td>
</tr>
<tr>
<td></td>
<td>Rate for Contactor I and II, Vₛ = 0.1572 cm/sec R = 1, CCL₄-HAC-H₂O system</td>
<td></td>
</tr>
<tr>
<td>Figure No.</td>
<td>Description</td>
<td>Page</td>
</tr>
<tr>
<td>------------</td>
<td>-------------</td>
<td>------</td>
</tr>
<tr>
<td>7.3.1</td>
<td>Variation of NTU&lt;sub&gt;cm&lt;/sub&gt; with Phase Flow Rate. R = 1, CCL&lt;sub&gt;4&lt;/sub&gt;-HAC-H&lt;sub&gt;2&lt;/sub&gt;O system Contactor I</td>
<td>7.22</td>
</tr>
<tr>
<td>7.3.2</td>
<td>Variation of Measured NTU&lt;sub&gt;dm&lt;/sub&gt; with Phase Flow Rate Contactor I R = 1 CCL&lt;sub&gt;4&lt;/sub&gt; - HAC - H&lt;sub&gt;2&lt;/sub&gt;O system</td>
<td>7.23</td>
</tr>
<tr>
<td>7.3.3</td>
<td>Variation of Measured Number of Transfer Units of Continuous Phase with Oscillation Rate R = 1 Q = 350 l/hr MIBK-HAC-H&lt;sub&gt;2&lt;/sub&gt;O system Contactor I</td>
<td>7.24</td>
</tr>
<tr>
<td>7.3.4</td>
<td>Variation of Measured Number of Transfer Units of Continuous Phase with Phase Flowrate R = 1 N = 70 opm MIBK - HAC - H&lt;sub&gt;2&lt;/sub&gt;O, Contactor I</td>
<td>7.24</td>
</tr>
<tr>
<td>7.3.5</td>
<td>Variation of Measured Number of Transfer Units for Continuous Phase with Phase Flowrate, R = 1 N = 80 opm CCL&lt;sub&gt;4&lt;/sub&gt; - HAC - H&lt;sub&gt;2&lt;/sub&gt;O system Contactor II</td>
<td>7.25</td>
</tr>
<tr>
<td>7.3.6</td>
<td>Variation of Measured Number of Transfer Units for Continuous Phsae with Oscillation Rate. R = 1 Q = 100 l/hr CCL&lt;sub&gt;4&lt;/sub&gt; - HAC - H&lt;sub&gt;2&lt;/sub&gt;O system Contactor II</td>
<td>7.25</td>
</tr>
<tr>
<td>7.3.7</td>
<td>Variation of Measured HTU&lt;sub&gt;cm&lt;/sub&gt; with Phase Flowrate R = 1, Contactor I CCL&lt;sub&gt;4&lt;/sub&gt; - HAC - H&lt;sub&gt;2&lt;/sub&gt;O system</td>
<td>7.26</td>
</tr>
<tr>
<td>7.3.8</td>
<td>Variation of Measured HTU&lt;sub&gt;dm&lt;/sub&gt; with Phase Flowrate R = 1 CCL&lt;sub&gt;4&lt;/sub&gt; - HAC - H&lt;sub&gt;2&lt;/sub&gt;O system Contactor I</td>
<td>7.27</td>
</tr>
<tr>
<td>7.3.9</td>
<td>Variation of HTU&lt;sub&gt;cm&lt;/sub&gt; with Flowrate 10 Q&lt;sub&gt;d&lt;/sub&gt; = 400 l/hr, N = 100 opm CCL&lt;sub&gt;4&lt;/sub&gt; - HAC - H&lt;sub&gt;2&lt;/sub&gt;O system Contactor I</td>
<td>7.28</td>
</tr>
<tr>
<td>7.3.10</td>
<td>Variation of HTU&lt;sub&gt;cm&lt;/sub&gt; with Dispersed Phase Inlet Solute Concentration. Q = 400 l/hr,</td>
<td>7.28</td>
</tr>
<tr>
<td>Figure No.</td>
<td>Description</td>
<td></td>
</tr>
<tr>
<td>------------</td>
<td>-------------</td>
<td></td>
</tr>
<tr>
<td>7.3.11</td>
<td>Variation of Measured Height of Transfer Units of Continuous Phase with Oscillation Rate, ( R = 1 ), ( Q = 350 \text{ l/hr} ) MIBK - HAC - ( H_2O ) Contactor I</td>
<td></td>
</tr>
<tr>
<td>7.3.12</td>
<td>Variation of Measured Height of Transfer Unit for Continuous Phase with Phase Flowrate ( R = 1 ), ( N = 70 \text{ opm} ) MIBK - HAC - ( H_2O ), Contactor I</td>
<td></td>
</tr>
<tr>
<td>7.3.13</td>
<td>Variation of Measured Heights of Transfer Units for Continuous Phase with Phase Flowrate ( R = 1 ), ( N = 80 \text{ opm} ) CCL(_4) - HAC - ( H_2O ) system Contactor II</td>
<td></td>
</tr>
<tr>
<td>7.3.14</td>
<td>Variation of Measured Height of Transfer Units for Continuous Phase with Phase Flowrate ( R = 1 ), ( Q = 100 \text{ l/hr} ) CCL(_4) - HAC - ( H_2O ) system Contactor II</td>
<td></td>
</tr>
<tr>
<td>7.3.15</td>
<td>Comparison of the Variation of HTU\text{cm} with Superficial Velocity for Contactors I and II, ( R = 1 ), ( N = 80 \text{ opm} ) CCL(_4) - HAC - ( H_2O )</td>
<td></td>
</tr>
<tr>
<td>7.3.16</td>
<td>Comparison of the Variation of HTU\text{cm} with Oscillation Rate for Contactors I and II, ( R = 1 ), ( V_S = 0.015716 \text{ cm/sec} ) CCL(_4) - HAC - ( H_2O )</td>
<td></td>
</tr>
<tr>
<td>7.3.17</td>
<td>Variation of ( K_a \text{cm} ) with Phase Flowrate ( R = 1 ), CCL(_4)-HAC-( H_2O ) system, Contactor I</td>
<td></td>
</tr>
<tr>
<td>7.3.18</td>
<td>Variation of Measured Mass Transfer Coefficient for Continuous Phase with Oscillation Rate. ( R = 1 ), ( Q = 350 \text{ l/hr} ) MIBK - HAC - ( H_2O ) Contactor I</td>
<td></td>
</tr>
<tr>
<td>Figure No.</td>
<td>Description</td>
<td>Page No</td>
</tr>
<tr>
<td>-----------</td>
<td>-------------</td>
<td>---------</td>
</tr>
<tr>
<td>7.3.19</td>
<td>Variation of Measured Mass Transfer Coefficient for Continuous Phase with Phase Flowrate $R = 1, N = 70$ opm MIBK - HAC - H$_2$O Contactor I</td>
<td>7.33</td>
</tr>
<tr>
<td>7.3.20</td>
<td>Variation of Measured Continuous Phase Mass Transfer Coefficient with Phase Flowrate $R = 1, N = 80$ opm CCl$_4$ - HAC - H$_2$O system, Contactor II</td>
<td>7.34</td>
</tr>
<tr>
<td>7.3.21</td>
<td>Variation of Measured Continuous Phase Mass Transfer Coefficient with Phase Flowrate $R = 1, N = 80$ opm CCl$_4$ - HAC - H$_2$O system Contactor II</td>
<td>7.34</td>
</tr>
<tr>
<td>(a)</td>
<td>Variation of $K_a$ with Superficial Phase Velocity, $R = 1, N = 80$ opm CCl$_4$ - HAC - H$_2$O system</td>
<td>7.35</td>
</tr>
<tr>
<td>(b)</td>
<td>Variation of $K_a$ with Oscillation Rate $R = 1, Q = 0.1572$ cm/sec CCl$_4$ - HAC - H$_2$O</td>
<td>7.35</td>
</tr>
<tr>
<td>7.3.22</td>
<td>Comparisons of Contactors I and II</td>
<td>7.35</td>
</tr>
<tr>
<td>7.3.23</td>
<td>Variation of Overall Mass Transfer Coefficient Along Contactor for CCl$_4$ - HAC - H$_2$O system $Q = 400$ l/hr, $R = 1$, Contactor I</td>
<td>7.36</td>
</tr>
<tr>
<td>7.4.1</td>
<td>Variation of Hold-up(percent of total volume) with Total Throughput, $R = 1$ CCl$_4$ - HAC - H$_2$O system, Contactor I</td>
<td>7.37</td>
</tr>
<tr>
<td>7.4.2</td>
<td>Variation of Dispersed Phase Hold-up with Flowratio. $Q_d = 400$ l/hr, $N = 100$ opm, CCl$_4$ - HAC - H$_2$O system Contactor I</td>
<td>7.38</td>
</tr>
<tr>
<td>7.4.3</td>
<td>Variation of Dispersed Phase Hold-up with Inlet Solute Concentration of Dispersed Phase. $Q = 400$ l/hr, $N = 100$ opm CCl$_4$ - HAC - H$_2$O system, Contactor I</td>
<td>7.38</td>
</tr>
<tr>
<td>Figure No.</td>
<td>Description</td>
<td>Page No</td>
</tr>
<tr>
<td>-----------</td>
<td>-----------------------------------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>7.4.4</td>
<td>Variation of Dispersed Phase Hold-up with Oscillation Rate. $R = 1$</td>
<td>7.39</td>
</tr>
<tr>
<td></td>
<td>$Q_c = 700 \text{ l/hr}$, MIBK - HAC - H$_2$O, Contactor I</td>
<td></td>
</tr>
<tr>
<td>7.4.5</td>
<td>Variation of Dispersed Phase Hold-up with Oscillation Rate. $R = 1$, $N = 70 \text{ opm}$</td>
<td>7.39</td>
</tr>
<tr>
<td></td>
<td>MIBK - HAC - H$_2$O, Contactor I</td>
<td></td>
</tr>
<tr>
<td>7.4.6</td>
<td>Variation of Dispersed Phase Hold-up with Phase Flowrate, $R = 1$, $N = 80 \text{ opm}$</td>
<td>7.40</td>
</tr>
<tr>
<td></td>
<td>$x_i = 30 \text{ g/l}$, CCL$_4$ - HAC - H$_2$O system, Contactor II</td>
<td></td>
</tr>
<tr>
<td>7.4.7</td>
<td>Variation of Dispersed Phase Hold-up with Oscillation Rate, $R = 1$, $Q = 100 \text{ l/hr}$</td>
<td>7.41</td>
</tr>
<tr>
<td></td>
<td>$x_i = 35 \text{ g/l}$, CCL$_4$ - HAC - H$_2$O system, Contactor II</td>
<td></td>
</tr>
<tr>
<td>7.4.8</td>
<td>Comparison of the Variation of Dispersed Phase Hold-up with Oscillation Rate for Contactor I and II. $R = 1$, $V_s = 0.1572 \text{ cm/sec}$, CCL$_4$ - HAC - H$_2$O</td>
<td>7.42</td>
</tr>
<tr>
<td>7.4.9</td>
<td>Comparison of Effect of Dispersed Phase Flowrate on Hold-up for Spray Towers and O.B.C</td>
<td>7.43</td>
</tr>
<tr>
<td>7.4.10</td>
<td>Comparison of the Effect of Continuous Phase Flowrate on Hold-up for Spray Towers and the O.B.C</td>
<td>7.44</td>
</tr>
<tr>
<td>7.5.1</td>
<td>Variation of Hold-up with Dispersed Phase Flowrate for High Solute Concentration ($x_i = 20 \text{ g/l}$) and Low Solute Concentration ($x_i = 0.5 \text{ g/l}$) CCL$_4$ - HAC - H$_2$O system, $N = 100 \text{ opm}$, $Q_c = 600 \text{ l/hr}$, Contactor I</td>
<td>7.45</td>
</tr>
<tr>
<td>7.5.2</td>
<td>Variation of Hold-up with Continuous Phase Flowrate Low Solute Concentration ($x_i = 0.5 \text{ g/l}$) CCL$_4$-HAC-H$_2$O system, $N = 100 \text{ opm}$, Contactor I</td>
<td>7.46</td>
</tr>
<tr>
<td>Figure No.</td>
<td>Description</td>
<td>Page No</td>
</tr>
<tr>
<td>-------------</td>
<td>-----------------------------------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>7.6.1</td>
<td>Variation of Means Residence with Continuous Phase Flowrate (single phase only) Contactor I</td>
<td>7.47</td>
</tr>
<tr>
<td>7.6.2</td>
<td>Variation of Dimensionless Variance with Peclet Number for Closed (Eqn.3.57) and Open (Eqn.3.58) Channels</td>
<td>7.48</td>
</tr>
<tr>
<td>7.6.3</td>
<td>Variation of Peclet Number with Continuous Phase Flowrate - Single Phase System, Contactor I</td>
<td>7.49</td>
</tr>
<tr>
<td>7.6.4</td>
<td>Variation of Axial Mixing Coefficient with Continuous Phase Velocity (single phase only) Contactor I</td>
<td>7.50</td>
</tr>
<tr>
<td>7.6.5</td>
<td>Profile of $E \frac{d^2 y}{dh^2}$ at Different Phase Flowrates. $R = 1, N = 100$ opm CCL$_4$ - HAC - H$_2$O system, Contactor I</td>
<td>7.51</td>
</tr>
<tr>
<td>7.6.6</td>
<td>Local Accumulation and Depletion Effects for Transferring Material for Continuous Phase, $N = 80, R = 1, Q = 400$ l/hr Contactor I, System I</td>
<td>7.52</td>
</tr>
<tr>
<td>7.7.1</td>
<td>Variation of Dry, Mixing, and Total Power Consumption with Oscillation Rate $R = 1, Q = 400$ l/hr, CCL$_4$-HAC-H$_2$O system Contactor I</td>
<td>7.53</td>
</tr>
<tr>
<td>7.7.2</td>
<td>Variation of Mixing Power per Unit Active Volume with Phase Flowrate. $R = 1$ CCL$_4$ - HAC - H$_2$O system Contactor I</td>
<td>7.54</td>
</tr>
<tr>
<td>7.7.3</td>
<td>Variation of Power per Unit Active Volume with Dispersed Phase Hold-up $R = 1$, CCL$_4$-HAC-H$_2$O system, Contactor I</td>
<td>7.55</td>
</tr>
<tr>
<td>7.7.4</td>
<td>Variation of Dry, Mixing and Total Power Consumption with Oscillation Rate $R = 1, Q = 350$ l/hr, MIBK - HAC - H$_2$O system, Contactor I</td>
<td>7.56</td>
</tr>
<tr>
<td>Figure No.</td>
<td>Description</td>
<td>Page No</td>
</tr>
<tr>
<td>-----------</td>
<td>-----------------------------------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>7.7.5</td>
<td>Variation of Mixing Power Consumption with Phase Flowrate. R = 1, N = 70 opm</td>
<td>7.56</td>
</tr>
<tr>
<td></td>
<td>MIBK - HAC - H$_2$O system, Contactor I</td>
<td></td>
</tr>
<tr>
<td>7.7.6</td>
<td>Variation of Dry, Mixing and Total Power Consumption with Oscillation Rate</td>
<td>7.57</td>
</tr>
<tr>
<td></td>
<td>CCL$_4$ - HAC - H$_2$O system, Contactor II</td>
<td></td>
</tr>
<tr>
<td>7.7.7</td>
<td>Variation of Power Per Unit Active Volume with Dispersed Phase Flowrate</td>
<td>7.58</td>
</tr>
<tr>
<td></td>
<td>R = 1, Q = 100 l/hr</td>
<td></td>
</tr>
<tr>
<td></td>
<td>CCL$_4$ - HAC - H$_2$O system, Contactor II</td>
<td></td>
</tr>
<tr>
<td>7.7.8</td>
<td>Comparisons of the Variation of Dry, and Total Power with Oscillation Rate</td>
<td>7.59</td>
</tr>
<tr>
<td></td>
<td>for Contactors I and II. R = 1, V$_S$ = 0.1572 cm/sec, CCL$_4$ - HAC - H$_2$O</td>
<td></td>
</tr>
<tr>
<td>7.7.9</td>
<td>Comparison of the Variation of Power per Unit Active and Swept Volume with</td>
<td>7.60</td>
</tr>
<tr>
<td></td>
<td>Oscillation Rate for Contactors I and II. R = 1, V$_S$ = 0.1572 cm/sec,</td>
<td></td>
</tr>
<tr>
<td></td>
<td>CCL$_4$ - HAC - H$_2$O system</td>
<td></td>
</tr>
<tr>
<td>7.8.1</td>
<td>Experimental and Theoretical Concentration Profiles for CCL$_4$ - HAC - H$_2$O</td>
<td>7.63</td>
</tr>
<tr>
<td></td>
<td>system, Contactor I, Q$_c$ = Q$_d$ = 400 l/hr</td>
<td></td>
</tr>
<tr>
<td></td>
<td>N = 80 opm</td>
<td></td>
</tr>
<tr>
<td>7.8.2</td>
<td>Effect of Boundary Condition on Concentration Profiles from the Solution</td>
<td>7.64</td>
</tr>
<tr>
<td></td>
<td>to the Diffusion Equation. CCL$_4$-HAC-H$_2$O system, Contactor I</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Q$_c$ = Q$_d$ = 400 l/hr, N = 80 opm</td>
<td></td>
</tr>
<tr>
<td>7.9</td>
<td>Comparison of Experimental and Calculated Sauter Mean Drop Diameter.</td>
<td>7.75</td>
</tr>
<tr>
<td></td>
<td>Contactor I, CCL$_4$ - HAC - H$_2$O</td>
<td></td>
</tr>
<tr>
<td>7.10</td>
<td>Comparison of Experimental and Calculated Mass Transfer Coefficients</td>
<td>7.76</td>
</tr>
<tr>
<td></td>
<td>for CCL$_4$-HAC-H$_2$O Contactor I</td>
<td></td>
</tr>
<tr>
<td>Figure No.</td>
<td>Description</td>
<td>Page No</td>
</tr>
<tr>
<td>-----------</td>
<td>-----------------------------------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>7.11.1</td>
<td>Variation of Axial Mixing Parameter with Reynolds Number for Oscillation</td>
<td>7.77</td>
</tr>
<tr>
<td>7.11.2</td>
<td>Variation of Axial Mixing Parameter with Reynolds Number for Flow</td>
<td>7.78</td>
</tr>
<tr>
<td>7.11.3</td>
<td>Determination of Constants for the Westerterp and Landsman - Type Correlation for Axial Mixing</td>
<td>7.79</td>
</tr>
<tr>
<td>7.11.4</td>
<td>Comparison of Experimental and Calculated Axial Mixing Coefficients for Contactor I</td>
<td>7.80</td>
</tr>
<tr>
<td>7.12</td>
<td>Comparison of Experimental and Calculated Power per Unit Volume for Contactor I</td>
<td>7.81</td>
</tr>
<tr>
<td>Figure No</td>
<td>Description</td>
<td>Page No</td>
</tr>
<tr>
<td>-----------</td>
<td>--------------------------------------------------------------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>A.1.1</td>
<td>Equilibrium Distribution of Acetic Acid Between Carbon Tetrachloride and water</td>
<td>A.1.6</td>
</tr>
<tr>
<td>A.1.2</td>
<td>Equilibrium Distribution of Acetic Acid Between Methyl Isobutyl Ketone and water</td>
<td>A.1.7</td>
</tr>
<tr>
<td>A.1.3</td>
<td>Refractive Index - Solute Concentration Charts</td>
<td>A.1.9</td>
</tr>
<tr>
<td>A.1.4</td>
<td>Variation of $d_{32}$ along Contactor Length CCL$_4$ - HAC - H$_2$O system. $R = 1$, $Q_C = 400$ 1/hr, Contactor I</td>
<td>A.1.13</td>
</tr>
<tr>
<td>A.2.1</td>
<td>Diagram showing Manometer Connections to Contactor</td>
<td>A.2.3</td>
</tr>
<tr>
<td>A.3.1</td>
<td>Boundary Condition Diagram for CCL$_4$ - HAC - H$_2$O system, Contactor I</td>
<td>A.3.6</td>
</tr>
<tr>
<td>A.4.1</td>
<td>Sectional Values of Transfer Units</td>
<td>A.4.2</td>
</tr>
</tbody>
</table>
Table No. | TABLES | Description | Page No
--- | --- | --- | ---
4.1 | Columns and Baffle Dimensions | | 4.3
4.2 | Details of Baffle Drive Systems | | 4.14

CHAPTER 7

7.1 | Values for Experimental and Theoretical Concentration Profiles at N = 80 opm Q_c = Q_d = 400 l/hr. Contactor I | | 7.62
7.2 | Axial Mixing Coefficient Values from Dye Studies, Empirical Correlation and Diffusion Equation | | 7.65
7.3 | Data for Westerterp and Landsman Type Correlation for Axial Mixing Coefficient | | 7.70

APPENDIX 1

A.1.1 | Physical Properties for the Chemicals used | A.1.2
A.1.2 | Equilibrium Distribution of Acetic Acid between water and Carbon Tetrachloride | A.1.3
A.1.3 | Equilibrium Distribution of Acetic Acid between water and Methyl Isobutyl Ketone | A.1.4
A.1.4 | Relationship between Solute Concentration and Interfacial Tension at 20°C (a) Carbon Tetrachloride - Water - Acetic Acid system | A.1.8
| (b) Methyl Isobutyl Ketone - Water - Acetic Acid system | A.1.8
A.1.5 | Variation of d_{32} along column length. Contactor I CCL_4-HAC-H_2O system, Q_c = Q_d=400 l/hr | A.1.11
<table>
<thead>
<tr>
<th>Table No.</th>
<th>Description</th>
<th>Page No</th>
</tr>
</thead>
<tbody>
<tr>
<td>Block A</td>
<td>CCL₄ - HAC - H₂O System</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Contactor I</td>
<td></td>
</tr>
<tr>
<td>1A</td>
<td>Concentration Profile for CCL₄ - HAC - H₂O System, ( R = \frac{Q_c}{Q_d} = 1 ), Constant ( x_i ), Contactor I (Large)</td>
<td>A5.2</td>
</tr>
<tr>
<td>2A</td>
<td>Concentration Profile for CCL₄ - HAC - H₂O System, Varying ( R ), Constant ( x_i ), Contactor I (Large)</td>
<td>A5.4</td>
</tr>
<tr>
<td>3A</td>
<td>Concentration Profile for CCL₄ - HAC - H₂O System, ( R = \frac{Q_c}{Q_d} = 1 ), Varying ( x_i ), Contactor I (Large)</td>
<td>A5.5</td>
</tr>
<tr>
<td>4A</td>
<td>Frequency of Droplet Sizes from Photographic Studies for CCL₄ - HAC - H₂O System, at Constant Flowrate, Varying Oscillation Speed, Contactor I ( Q_c = Q_d = 200 \text{ l/hr} )</td>
<td>A5.6</td>
</tr>
<tr>
<td>5A</td>
<td>Sauter Mean Drop Diameter, Interfacial Area and Mass Transfer Coefficient for Continuous Phase, Determined Experimentally from Photographic Studies for CCL₄ - HAC - H₂O System, Contactor I</td>
<td>A5.7</td>
</tr>
<tr>
<td>6A</td>
<td>Experimentally Determined Overall NTU, HTU, ( K_a ) Values for CCL₄ - HAC - H₂O System, Contactor I (Large) ( \text{Constant Flow Ratio, } R = \frac{Q_c}{Q_d} = 1 ) Constant ( x_i )</td>
<td>A5.8</td>
</tr>
</tbody>
</table>
Table No. | Description                                                                 | Page No
---|---|---
7A | Experimentally Determined Overall NTU, HTU, Ka Values for CCL₄ - HAC - H₂O system, Contactor I (Large) | A5.9
8A | Extraction Efficiency. Contactor I (Large) CCL₄ - HAC - H₂O system Constant xᵢ | A5.10
9A | Extraction Efficiency Contactor I (Large) CCL₄ - HAC - H₂O system | A5.11
10A | Sectional Values of Number and Heights of Transfer Units and Mass Transfer Coefficients R = Q_c/Q_d = 1 CCL₄ - HAC - H₂O system, Contactor I | A5.12
11A | Hold-up Studies for Low Solute Concentration (xᵢ 0.5 g/l), N = 100 opm, CCL₄ - HAC - H₂O system, Contactor I | A5.16
12A | Hold-up Studies for High Solute Concentration (xᵢ 20 g/l) and Low Solute Concentration ("Non M.T System") (xᵢ 0.5 g/l) CCL₄ - HAC - H₂O System, N = 100 opm, Contactor I | A5.17
13A | Residence Time Studies for Single (Continuous)Phase, Contactor I(Large) | A5.18
14A | Experimental and Calculated Axial Mixing Results for Single(Continuous) Phase. Contactor I (Large) | A5.19
15A | Variation of Dimensionless Variance with Peclet Number for Closed and Open Channels. | A5.20
16A | Axial Mixing Studies Based on the One-Dimensional Diffusion Equation for CCL₄ - HAC - H₂O system, Contactor I | A5.21
<table>
<thead>
<tr>
<th>Table No.</th>
<th>Description</th>
<th>Page No</th>
</tr>
</thead>
<tbody>
<tr>
<td>17A</td>
<td>Dry Power Study, Contactor I (Large)</td>
<td>A5.25</td>
</tr>
<tr>
<td>18A</td>
<td>Single Phase Mixing Power Study Contactor I (Large)</td>
<td>A5.25</td>
</tr>
<tr>
<td>19A</td>
<td>Two Phase Mixing Power Study Contactor I (Large) CCl₄-HAC-H₂O system</td>
<td>A5.26</td>
</tr>
<tr>
<td>20A</td>
<td>Two Phase Mixing Power per Unit Active and Swept Volume, CCl₄ - HAC - H₂O system, Contactor I</td>
<td>A5.27</td>
</tr>
</tbody>
</table>

**BLOCK B**

**MIBK - HAC - H₂O system**

**Contactor I**

<table>
<thead>
<tr>
<th>Table No.</th>
<th>Description</th>
<th>Page No</th>
</tr>
</thead>
<tbody>
<tr>
<td>1B</td>
<td>Concentration Profile for System MIBK - HAC - H₂O, R = Qc/Qd = 1 Contactor I</td>
<td>A5.29</td>
</tr>
<tr>
<td>2B</td>
<td>Frequency of Droplet Sizes from Photographic Studies for MIBK - HAC - H₂O system, at Constant Flowrate, Varying Oscillation Speed, Contactor I Qc = Qd = 350 1/hr</td>
<td>A5.30</td>
</tr>
<tr>
<td>3B</td>
<td>Sauter Mean Drop Diameter, Interfacial Area and Mass Transfer Coefficient for Continuous Phase, Determined Experimentally from Photographic Studies for MIBK - HAC - H₂O system, Contactor I</td>
<td>A5.31</td>
</tr>
<tr>
<td>4B</td>
<td>Extraction Efficiency, Contactor I MIBK - HAC - H₂O system</td>
<td>A5.32</td>
</tr>
<tr>
<td>5B</td>
<td>Experimentally Determined NTU, HTU, Ka Values for MIBK - HAC - H₂O system R = 1, Contactor I</td>
<td>A5.33</td>
</tr>
<tr>
<td>Table No.</td>
<td>Description</td>
<td>Page No</td>
</tr>
<tr>
<td>----------</td>
<td>-----------------------------------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>6B</td>
<td>Sectional Values of Number and Height of Transfer Units and Mass Transfer</td>
<td>A5.34</td>
</tr>
<tr>
<td></td>
<td>Coefficients $R = \frac{Q_c}{Q_d} = 1$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>MIBK - HAC - $H_2O$ system, Contactor I</td>
<td></td>
</tr>
<tr>
<td>7B</td>
<td>Power and Hold-up Study for MIBK-HAC-$H_2O$ system, Contactor I (Large)</td>
<td>A5.36</td>
</tr>
<tr>
<td>8B</td>
<td>Two Phase Mixing Power Per Unit Active and Swept Volume MIBK - HAC - $H_2O$</td>
<td>A5.37</td>
</tr>
<tr>
<td></td>
<td>system, Contactor I</td>
<td></td>
</tr>
</tbody>
</table>

**BLOCK C**

**CCL$_4$ - HAC - $H_2O$ System**

**Contactor II**

<p>| 1C       | Concentration Profile for System                                          | A5.39   |
|          | CCL$_4$ - HAC - H$_2O$, $R = \frac{Q_c}{Q_d} = 1$                         |         |
|          | Contactor II                                                               |         |
| 2C       | Frequency of Droplet Sizes from Photographic Studies for CCL$_4$ - HAC - $H_2O$ | A5.40   |
|          | system, at Constant Flowrate, Varying Oscillation Speed                    |         |
|          | Contactor II, $Q_c = Q_d = 100$ l/hr                                      |         |
| 3C       | Sauter Mean Drop Diameter, Interfacial Area and Mass Transfer Coefficient  | A5.41   |
|          | for Continuous Phase, Determined Experimentally and Photographic Studies   |         |
|          | for CCL$_4$ - HAC - H$_2O$ System, Contactor II                            |         |
| 4C       | Experimentally Determined Number and Heights of Transfer Units and Mass    | A5.42   |
|          | Transfer Coefficients $R = 1$                                              |         |
|          | CCL$_4$ - HAC - $H_2O$ system, Contactor II                                |         |</p>
<table>
<thead>
<tr>
<th>Table No.</th>
<th>Description</th>
<th>Page No</th>
</tr>
</thead>
<tbody>
<tr>
<td>5C</td>
<td>Extraction Efficiency, Contactor II CCL\textsubscript{4} - HAC - H\textsubscript{2}O system</td>
<td>A5.43</td>
</tr>
<tr>
<td>6C</td>
<td>Sectional Values of Number and Heights of Transfer Units and Mass Transfer Coefficients, ( R = Q_c/Q_d = 1 ) CCL\textsubscript{4} - HAC - H\textsubscript{2}O system, Contactor II</td>
<td>A5.44</td>
</tr>
<tr>
<td>7C</td>
<td>Dry Power Study Contactor II(Small)</td>
<td>A5.46</td>
</tr>
<tr>
<td>8C</td>
<td>Single Phase Mixing Power Study Contactor II (Small)</td>
<td>A5.46</td>
</tr>
<tr>
<td>9C</td>
<td>Two Phase Mixing Power Study Contactor II(Small) CCL\textsubscript{4} - HAC - H\textsubscript{2}O system</td>
<td>A5.47</td>
</tr>
<tr>
<td>10C</td>
<td>Two Phase Mixing Power Per Unit Active and Swept Volume CCL\textsubscript{4} - HAC - H\textsubscript{2}O system, Contactor II</td>
<td>A5.48</td>
</tr>
</tbody>
</table>
CHAPTER 1

INTRODUCTION
1.1 Basic Design Consideration For Liquid-Liquid Contactors.

The factors that govern the selection and design of liquid-liquid contactors for extraction purposes are numerous. Such factors include throughput, rate of mass transfer required, method for contacting phases, equilibrium conditions, floor space available, safety and environmental factors. A wide variety of contactors ranging from small sophisticated to large rugged types have been used for solvent extraction. Generally they can be classified under the following two categories (a) horizontal contactors and (b) vertical contactors.

For the majority of multistage liquid-liquid extraction processes, vertical columns are to be preferred as they occupy less floor space, no interstage pumping is required and intimate contacting of the two phases can be achieved mechanically by simple single drive mechanisms, thus making it more economical to operate. However, this type of equipment also has its drawbacks since it is not possible to control such important parameters as interfacial area which strongly influences the rate of mass transfer. They are characterized also by low throughput and extensive axial mixing which is dependent on the type of agitation and vessel internals.

For a countercurrent agitated contactor it is desirable that the interfacial area available for mass transfer be kept as high as possible, thus resulting in a high solute extraction rate. For processes where the solute has to be separated from the solvent after extraction it is necessary that the solvent flowrate be kept as low as possible. However, the interfacial area is fixed by the operating conditions such as throughput and agitator rate which is in turn fixed by the limiting flow conditions for the specific extraction system. Hence, for most contacting devices there are optimum operating conditions which are dependent on the nature of the extraction process.

An important parameter that must be considered when designing contactors, is the degree of axial mixing. Axial mixing lowers
the separation efficiency of contactors as compared to the values obtained from an assumption of piston flow. In counter-current contactors axial mixing is increased by increase in agitation rate and occurs at any flowrate.

Ideally a contacting device should be capable of handling high throughput and producing high separation efficiency. Also, it should be simple to construct and capable of being scaled up from small prototype to large industrial contactors with reliability.

The present work consists of a performance study on a oscillating Baffle Contactor O.B.C with multiple oscillating baffles. This contactor is the first of its type and the mixing system is unique to this particular device. It is a scaled up version - both in size and number of baffles - of the original O.B.C described by Thomas (T6 and British Patent 52556/66). The scale of the work can be regarded as close to semi-industrial scale.

1.2 Mass Transfer Rates.

1.2.1 Mass Transfer Theories.

Several theories for determining mass transfer rates in solvent extraction have been proposed. The most widely used of these theories for liquid-liquid extraction is the Lewis-Whitman two film theory(L7). This theory assumes that the solute concentration in each phase at the interface are in equilibrium. It also assumes mass transfer occurs by a turbulent mechanism in the main stream of each phase and by molecular diffusion through stagnant films on each side of the interface. Most of the resistance to mass transfer is assumed to be contained in these two films. The two films theory is by no means truly representative of the mass transfer mechanism across the interface and other theories for interphase mass transfer across an interface have been put forward. Two of these which are sometimes used as alternatives are the Higbie(H10) penetration theory and Danckwerts
(D2) surface renewal theory. Nevertheless, the two film theory has been used with remarkable success in interpreting the result of most two phase mass transfer operation on industrial importance. It provides the Chemical Engineer with a straightforward overall method for determining mass transfer rates in liquid-liquid contactors. This theory will be used to develop the mass transfer rate equation for the present work.

The Higbie (H10) penetration theory assumes that turbulent eddies travel from the bulk of the phase to the interface, where they remain for a short but constant time before moving back into the bulk of the phase. Solute is assumed to "penetrate" into a given eddy while it is at the interface by unsteady state molecular diffusion. The Surface Renewal Theory proposed by Danckwerts (D2) is a modification of Higbie's theory. This theory assumes an "infinite" range for the time spent by eddies at the interface and not constant times, as assumed for the penetration theory. An extensive treatment of the various theories are given in Coulson and Richardson (C19).

1.2.2 Mass Transfer Rate Equations.

These equations are developed from the two film theory. Consider solute being transferred from the dispersed to the continuous phase in the contactor. Then the rate of transfer of the solute can be represented by:

$$N = k_d a (x - x_i) \quad \ldots 1.1$$

or $$N = k_c a (y_i - y) \quad \ldots 1.2$$

where $N = \text{Mass transfer rate.}$

$k_c a, k_d a = \text{individual film mass transfer coefficient for continuous and dispersed phase respectively.}$

$y, x = \text{concentration of continuous and dispersed phase in the bulk of the phases respectively.}$

$y_i, x_i = \text{concentration of continuous and dispersed phase respectively at the interface.}$

$a = \text{interfacial area.}$
Due to the difficulty encountered in measuring the interfacial concentration of \( x_i \) and \( y_i \), overall mass transfer coefficients are used instead of individual coefficients. The overall mass transfer coefficient is defined as:

\[
N = K_{da} (x - x*) \quad \ldots 1.3
\]

\[
N = K_{ca} (y^* - y) \quad \ldots 1.4
\]

where \( x^* \) = concentration of solute in dispersed phase in equilibrium with concentration of solute in continuous phase \( y \).

\( y^* \) = concentration of solute in continuous phase in equilibrium with concentration of solute in dispersed phase \( x \).

\( K_{da}, K_{ca} \) = overall mass transfer coefficient.

1.2.3 Mass Transfer Coefficients.

As stated above it is normal to use overall mass transfer coefficients instead of individual coefficients when calculating mass transfer rates. The relationships between these two coefficients are dependent on whether the equilibrium relationship is linear or non-linear.

For a linear equilibrium relationship, the coefficients are related as follows:

\[
\frac{1}{k_c} = \frac{1}{k_{ca}} + \frac{m}{k_{da}} \quad \ldots 1.5
\]

\[
\text{and} \quad \frac{1}{k_d} = \frac{1}{k_{da}} + \frac{1}{mk_{ca}} \quad \ldots 1.6
\]

where \( m \) is the slope of the equilibrium curve and is referred to as the distribution coefficient. When the equilibrium line is non-linear, this coefficient varies with concentration and the values for \( m \) in equations 1.5 and 1.6 would be different.
1.2.4 Interfacial Area.

In liquid extraction processes where one phase is dispersed in another, the rate of mass transfer is directly related to the interfacial area, $a$ (equations 1.3 and 1.4). The magnitude of the interfacial area in an agitated contactor is highly dependent on the operating conditions, method of dispersion and physical properties of the liquid system.

The interfacial area is normally calculated from the Sauter mean drop diameter $d_{32}$, and the fractional hold-up of the dispersed phase $\phi$, both of which are measured experimentally. It is given by:

$$a = \frac{6\phi}{d_{32}} \quad \ldots 1.7$$

where $a$ is the specific interfacial area, $\text{cm}^2/\text{cm}^3$.

Equation 1.3 and 1.4 shows that a large interfacial area results in a high mass transfer rate and equation 1.7 show that this can be achieved at small drop diameters. However, too small a drop size can have an adverse effect on mass transfer rates. This is due to the effect of drop size on mass transfer coefficients. Associated with large drops are internal circulation and drop oscillation which enhance the mass transfer coefficients, while too small drops behave like rigid elastic spheres resulting in lower mass transfer coefficients. Therefore, there must be some minimum drop size beyond which further decrease would result in lower mass transfer coefficients. To put an actual value to this minimum size by experiment or other means is exceedingly difficult due to the complex behaviour of drop swarms.

1.3 Drop Dynamics.

The movement of drop swarms in a liquid media is an important aspect of liquid extraction. The drop size and velocity of the drop phase through the contactor determine the limiting flow
conditions and hence the contactor capacity. During the movement of the drop phase through the contactor drop interaction takes place.

Drop interaction is the combined process of collision, coalescence, break-up and redispersion of drops in a dynamic environment.

There are a number of factors which affect the movement of drop swarms in an agitated liquid media - liquid flowrates, agitation rate, drop size, drop interaction and the local eddy motions of the continuous phase. The combined effects of drop interaction is dependent on the nature of the system. For example, systems with low interfacial tension tend to coalesce and redispense more readily at lower agitation rates than systems with high interfacial tension. Another important factor affecting drop interaction is the hold-up of dispersed phase. For a system of low hold-up and high agitation rate, the break-up process will predominate with little or no interdrop coalescence. On the other hand, for contactors such as spray and packed towers, interdrop coalescence will predominate with little or no drop break-up, due to the lack of induced turbulence.

1.4 Axial Mixing.

The effect of axial mixing is to reduce the concentration driving force between the contacted phases and this lowers the separation efficiency of the contactor. Measurements(L5) show that up to 60 - 75% of the effective height of production size columns can be on account of axial mixing if they are scaled up from models where the effect of axial mixing was not considered. Thus an indepth study of the degree of axial mixing is crucial when designing countercurrent contactors.

Rod(R4) stated that by axial mixing, solute is transferred in the stripped phase in the direction of phase flow and in the enriched phase this proceeds against the direction of flow. It can be regarded as the combined effect of two phenomena, that
due to turbulent and molecular diffusion in the axial direction, and that resulting from a nonuniform velocity across the cross-section of the contactor (B2).

There appears to be some confusion in the literature with regards to the terms used to label axial mixing. Klinkenberg (K4) attempted to clarify this by defining two new terms, back mixing (1) - indicating a mechanism by which a phase can move against its main flow -, and back mixing (2) - indicating a mechanism by which a spread of residence time is brought about.

Backmixing in the continuous phase may result from turbulent eddies due to convective flow of the phase, or from entrainment of field liquid in the boundary layer surrounding the drops. Forward mixing in this phase may result from a velocity distribution in the continuous phase flow or from channeling effects due to the system geometry.

For the dispersed phase, axial mixing may be due to the spread of drop residence times within the contactor resulting in the distribution of drop sizes.

Several models have been proposed for studying the effect of axial mixing on extraction efficiency. One of the most widely used is that of Miyauchi and Vermulien (M8). The application of this model requires experimental values for axial mixing coefficients under the appropriate conditions, together with a knowledge of true values for mass transfer coefficients. Axial mixing coefficients can be determined from residence time distribution studies, and true values for mass transfer coefficients by Rod's (R4) method.

1.5 Dispersed Phase Hold-up.

Dispersed phase hold-up is the volume fraction of the total contactor volume occupied by the dispersed phase.

The hold-up is a very important parameter in liquid extraction
since it affects the mass transfer rates and the operating capacity of the system. The magnitude of the hold-up also affects the rate at which the various phases flow through the contactor. Too high a hold-up can result in hydrodynamic instability at which stage the contactor becomes inoperable.

1.6 Maximum Operating Conditions - Flooding.

In the operation of countercurrent liquid-liquid contactors it is generally recognized that for a given flowrate of one of the phases, continuous increase in the flowrate of the other may lead to a condition at which the lighter phase is swept out of the contactor by the heavy phase. At this stage the contactor is said to be "flooded" and the maximum operating conditions of the contactor is taken as these "limiting flow rates" or "flooding velocities".

At the flood point it can be observed that the droplet density within the contactor significantly increases. This "crowding" of the drops results in a decrease in interstitial area giving a high local continuous phase velocity which in turn increases the dispersed phase hold-up, and the density of dispersion still further. This ultimately lead to the contactor being filled up with dispersed phase and large amounts are carried over with the continuous phase.

There is no standard method for determining the flood point and it would appear from the definitions of "flooding" in the literature (B4, C12, H2, M2) the method used is dependent on the type of contactor and method of dispersion. Some of the methods widely used are observation of drop density build-up, carry over of dispersed phase and a marked change in the experimentally measured hold-up. Of these the last method is regarded as the most accurate, the other two being impractical.

1.7 Power Consumption in Liquid Mixing.

The power input into a mechanically agitated contactor is of
importance with regards to economical operation and successful scaling-up of the process.

The choice of a liquid contactor for solute extraction is dependent on the separation efficiency required and the cost in achieving this separation. For instance, it may be more economical to choose a contactor which consumes relatively little power and gives a 50 - 60% separation efficiency than a contactor which consumes a large amount of power and gives a 90% separation efficiency.

The power drawn by an agitator in a liquid mixing system is determined by its agitation rate and the environment in which it operates. As a result, the power consumed is correlated with these parameters as the independent variables. The correlation is in the form of an equation with dimensionless groups.

One of the important factors in the scaling-up of mixing equipment is the scaling-up of power data. This is normally carried out using the principle of similarity: geometric, kinematic and dynamic.

Miller and Mann(M1) reported that small and large mixers gave equal mass transfer coefficients and mixing rates at equal power input per unit volume. Thus, for reliable scaling-up of mixing equipment an in depth knowledge of the power consumption under different operating conditions is essential.

1.8 Programme of the Research.

The main variables affecting the performance of the oscillating baffle contactor with oscillating multiple baffles are (1) oscillation rate (II) amplitude of oscillation (III) liquid flowrates (IV) physical properties of the system (V) inlet solute concentration in the feed phase.

The effect of these variables has been investigated experi-
mentally, and computations based on derived theory were carried out. The sequence of the performance studies are as follows:

1. Solute concentrations at the inlet and outlet of both phases, and at equally spaced positions along the contactor length for the continuous phase were measured.

2. Computation of the apparent measured extraction efficiency based on the measured concentration profile for the continuous phase and including the effects of axial mixing.

3. Determination of axial mixing coefficients for continuous phase by residence time distribution studies.

4. Adaption of an existing mathematical model for predicting the solute concentration profile for the case in question, and computation of theoretical concentration profiles for some experiments using known parameters.

5. Determination of hold-up of dispersed phase using the manometric method.

6. Determination of Sauter mean drop diameter from photographic studies.

7. Determination of power characteristics of the O.B.C by electrical and electronic methods using a torque meter.

8. Flooding studies and the effect of solute concentration on flooding.

9. Generalised empirical correlations of measured extraction efficiency, Sauter mean drop diameter, power characteristics and axial mixing coefficient with their important variables using dimensional analysis technique.
Selected performance studies were also carried out on a small scale contactor with one oscillating baffle for comparison with the large scale multiple baffle system.
CHAPTER 2

LITERATURE SURVEY
2.1 Liquid-Liquid Contactors

The wide range of liquid-liquid contactors used both in industry and for research can be divided into two categories,

(I) Discrete Stage Contactors.

(II) Continuous Differential Contactors.

The two categories are further subdivided according to (a) the type of flow (i.e. countercurrent or co-current) and (b) method adopted for effecting the type of flow and interdispersing of the phases. An extensive classification of liquid-liquid contactors has been tabulated and discussed by Morello and Poffenberger(M10). A more recent classification has been presented by Logsdail and Lowes(L10).

2.1.1 Discrete Stage Contactors.

The discrete stage contactor consists of a fixed number of discrete stages wherein the immiscible (or partially miscible) phases are sufficiently well mixed to bring the liquids close to equilibrium. The liquids are then discharged into a settling tank for the phases to disengage. A well known extractor in which both of these steps are performed within a single shell is the Holley-Mott.

The discrete stage contactors are widely used in processes where a high purity of the product is essential such as in the pharmaceutical industry. They are also used in atomic energy processes where it is necessary to avoid the use of moving parts in the immediate vicinity of the unit.

Most of the discrete stage contactors employed in industry are arranged horizontally, however, there are cases, Hanson and Kaye(H4, H5) and Treybal(T13, T15) where vertical arrangement have been reported. In the present times where there is an urgent need for energy conservation and also the rapidly increasing cost of floor spaces, the vertical contactor would be much more economical to operate than the horizontal ones.
The advantages and disadvantages of the discrete stage contactors are given and discussed by Treybal (T14).

2.1.2 Continuous Differential Contactors (C.D.C's).

In continuous differential contactors extraction is effected by passing the phases countercurrently through the extractor which is vertically arranged. This type of flow is achieved by virtue of the density difference between the liquids being contacted.

The C.D.C's can be generally classified under two categories. (a) Unagitated Contactors and (b) Agitated Contactors. Voluminous research data have been reported on both categories.

The simplest form of the unagitated contactors is the spray column. Spray contactors are generally inexpensive and have high flow capacity but they have poor efficiencies. The efficiency of the spray could be increased by inserting packing, baffle-plates or sieve-plates in the empty shell of the contactor.

The agitated contactors such as the Rotary Disc Contactor, the York-Schiebal extractor, the Rotary Annular Contactor and the Oldshue-Rushton Contactor, which are rotary agitated, are widely used for liquid-liquid extraction in the petroleum industry. The performance of these column contactors indicates that they are more efficient and possess better operational flexibility than the unagitated contactors. In addition to rotary agitated contactors, there are the pulse agitated contactors. In these extractors, a rapid, reciprocating motion of relatively short amplitude is applied to the liquid contents.

A detailed description of both the discrete stage contactors and continuous differential contactor are given by Treybal (T14), Logsdail and Lowess (L10) and Laddha and Degaleesan (L2).
The present research was carried out on the oscillating baffle contactor which is classified as a continuous differential contactor. In order to make comparisons over the wide range of C.D.C's emphasis in this review will be placed on one of each type of extractor that falls in this category i.e un-agitated (spray contactor), rotary agitated (rotating disc contactor) and pulsed agitated (pulsed sieve plate) contactors.

2.2 Mass Transfer Studies in Extractors.

2.2.1 Spray Columns.

Elgin and Browning (El) were among the first of the many workers to report mass transfer data for Spray Columns. They studied the extraction of acetic acid from aqueous solution of isopropyl ether. Appel and Elgin (A1) reported on the extraction of acetic acid from water by benzene and methyl isobutyl ketone (M.I.B.K). Since the pioneering work of Elgin and co-workers, many workers have reported mass transfer studies for Spray Columns.

Laddha and Smith (LI) using the Colburn and Welsh (C8) technique studied the effect of flow rate on the individual mass transfer resistance for each phase, for counter diffusion of binary systems. They found that the HTU of the dispersed phase was independent of variations of continuous phase flow rate, while HTU for the continuous phase was dependent on both continuous and dispersed phase flow rates. Individual film resistances have also been reported by Smith and Beckam (S12) and Hong (H15) for transfer by the counter diffusion technique, with similar results. Ruby and Elgin (R7) studied unidirectional diffusion for several systems, and reported continuous phase mass transfer rate coefficients for a wide range of Reynolds number.

Hughmark (H17) reported mass transfer correlations in terms of the Sherwood, Reynolds and Schmidt dimensionless groups, he also compared correlations for mass transfer between single
drops and swarms of drops.

2.2.2 Rotating Disc Contactor – RDC.

Vermijs and Kramers (V3) reported performance studies on the RDC for the extraction of acetic acid from M.I.B.K. by water. They studied the effect of operating conditions – rotor speed, flowrates, and feed ratio – on contactor performance. For each of the rotor speeds studied the overall mass transfer coefficient of the dispersed phase increased to a maximum value (critical value) and then decreased with increase in hold-up. They ascribed this effect to back-mixing in the continuous phase due to entrainment of the dispersed phase.

Gel'Perin (G8) studied mass transfer rates in RDC for three systems with different physico-chemical properties. Local concentrations have been examined and smooth axial change in concentration was found. Optimum operating conditions were also reported. Reman and Olney (R1) correlated their mass transfer data with a power input factor.

Logsdail and Co-workers (L8) proposed a correlation for estimating the overall height of a transfer unit based on the continuous phase.

Strand, Olney and Ackerman (S17) analysed the data of several previous workers as well as their own and reported on the performance of R.D.C for several ternary systems. The behaviour of a swarm of drops in the rotating field was found to be adequately represented in most respects by single drop dynamics. They suggested a scale-up procedure based on the mass transfer data corrected for axial mixing for the design of industrial columns.

2.2.3 Pulsed Plate Extractors.

Sege and Woodfield (S5) reported on the extraction of uranyl nitrate with tributyl phosphate in a pulsed sieve plate column.
Effects of operating conditions and plate design on extraction efficiency were studied. It was found that extraction efficiency increased with increase in pulse frequency or amplitude.

Thornton (T12) and Logsdail and Thornton (L9) reported mass transfer data for solute transfer from dispersed to continuous phase and vice-versa for a number of aqueous-hydrocarbon solvent systems with water as the continuous phase. They proposed a correlation for \((HTU)_\text{oc}\) in terms of dispersed phase hold-up, characteristic velocity and physical properties.

Smoot and Babb (S13) studied mass transfer from organic to aqueous phases in a pulsed sieve plate column. The effects of column geometry and operating variables on the longitudinal solute concentration gradients for both phases of two ternary systems were reported.

2.3 Drop Studies.

2.3.1 Drop Size and Size Distribution.

The drop size distribution is an important aspect of liquid-liquid dispersions. It not only affects the physical behaviour of the dispersion, but also determines mass transfer rates in the system.

Research workers have used various relationships to express the size distribution. The relationships frequently used are the normal and log-normal distribution (B6, G9, K1, M11, N2, S15). It is quite common for the droplet size distribution to be represented by a median or by some mean diameter. For droplet analysis of liquid-liquid dispersions, the Sauter mean drop diameter \((d_{32})\) is widely used.

Thornton and Bouyatiotis (T13) developed a correlation for \(d_{32}\) in a fully baffled vessel, their correlation is as follows:
Keey and Glen (K1) studied drop size distribution in a continuously operated baffled vessels with six-blade paddle agitators. Diameter frequency plots showed drop size to have a log-normal distribution. Their correlation for diameter is:

\[ d_{32} = 3.165 \times 10^{-6} \frac{D_A^{0.4}}{(ND_A)^{1/2}} \left( \frac{D_V}{D_A} \right)^{1/2} \]  ...2.2

Skelland and Lee (S9) proposed a correlation for \( d_{32} \) when about 50% of the possible mass transfer had occurred during batch operation. Their correlation is:

\[ d_{32} = 6.713 \times 10^{-6} \phi^{0.188} \left( \frac{D_A}{D_V} \right)^{-1.034} \left( \frac{N_{Re}}{N_{oh}} \right)^{-0.558} \]  ...2.3

Tavlarides and Stamatoudis (T1) presented a tabulation of the most important reported correlation for Sauter mean drop diameter along with the ranges of operating variables and physical parameter over which they are valid. They reported that the most frequently reported correlation is of the form:

\[ \frac{d_{32}}{D_A} = k_1 (1 + k_2 \phi) (N_{we})^{-k_3} \]  ...2.4

where \( k_1, k_2 \) and \( k_3 \) are constants.

2.3.2 Interfacial Area Measurement.

Many methods have been reported in literature for the measurement of interfacial area. The various method can be generally classified into (a) the direct methods and (b) the indirect methods.

(a) Direct Methods

Direct methods for obtaining interfacial area are based on the measurement of the drop size distribution.
The simplest of these methods, used by a number of workers (C4, K1, R5, S4, S6), was to photograph directly through a window of the vessel. This technique can only be used for low hold-up fraction or for positions very close to the vessel walls due to the interference of the optical path by the presence of drops.

Some research workers (C10, G9, L3) have taken photomicrographs through a vessel window. Coulaloglou and Tavlarides used a photomicrographic probe assembly which enabled them to measure the drop size distributions at various depths in the vessel. Other workers (M9, S7) used the sample withdrawal method. By this method a sample is withdrawn, and a surfactant added to prevent coalescence. The sample is then analysed for drop size distribution by one of the methods above.

Park and Blair (P1), developed a new technique. They used a fibre optic probe in combination with cinephotography to measure drop size distributions.

(b) Indirect Methods.

Of the indirect methods used to measure interfacial area, the chemical method and the light transmittance method are considered to be the most important ones (T1).

The chemical method was first proposed, by Nanda and Sharma (N3). Liquid extraction accompanied by fast pseudo - first order reaction was used to evaluate the effective interfacial area. This method has since been used by a number of workers using esters of formic acid, chloroacetic acid and oxalic acid which are sparingly soluble in water.

Tavlarides and Stamatoudis (T1) suggested that the disadvantage of this method is the effect of the mass transfer on the physico-chemical properties of the dispersion since, mass transfer can affect the interfacial tension and thus the interfacial area.
The light transmittance method has been used by a number of research workers for estimating interfacial area. Tavlarides and Stamatoudis provided a list of most of these workers.

By this method a beam of light is passed through the dispersion, and due to the presence of the droplets only a small fraction of the light is transmitted. This transmitted light is a function of total interfacial area. Calderbank (C2) developed an expression for the transmitted light assuming uniform drop size.

2.3.3 Movement and Behaviour.

2.3.3.1 Drop Velocity.

The movement of drops through the liquid media is characterized by their terminal velocity. Drop velocities in liquid-liquid systems have been studied by many workers. A list of these workers are provided in the bibliographies of Treybal (T14), Heertjes and De Nie (H9) and Laddha and Degaleesan (L2).

The basic expression for the terminal velocity of drop was derived by Hadamard (H1) and Rybczynski (R9) working independently. They applied a viscosity correction factor to the well known Navier - Stokes equation. For the terminal velocity of a rigid sphere moving in a continuous liquid, the Hadamard and Rybczynski expression is:

\[ U_t = \frac{(\rho_d - \rho_c)gd^2}{18\mu_c} \quad \frac{3\mu_d + 3\mu_c}{3\mu_d + 2\mu_c} \quad \ldots 2.5 \]

Hu and Kintner (H16) studied the motion of single drops of a number of organic liquids falling through a stationary water phase. They presented a correlation in the form of a single curve which can be used directly to predict the terminal velocity for any given drop size.
Klee and Treybal (K2) measured terminal velocities of liquid drops rising and falling in liquid media. They studied eleven liquid systems covering a wide range of physical properties and, proposed correlations for each of the two velocity-drop diameter regions which can be used to compute terminal velocities of drops.

Their correlations are:

Region 1. where velocity increases with increasing drop diameter.

\[ U_{\text{tI}} = 38.3 \rho_C^{-0.45} \Delta \rho^{0.58} \mu_C^{-0.11} d^{0.70} \quad \ldots 2.6 \]

and Region II where velocity remains almost constant with increasing diameter.

\[ U_{\text{tII}} = 17.6 \rho_C^{-0.55} \Delta \rho^{0.28} \mu_C^{-0.10} \sigma^{-0.18} \quad \ldots 2.7 \]

Thornsen, Stordalen and Terjesen (T9) studied the terminal velocities for drops of organic liquids falling through water for seven high interfacial tension systems. They proposed the following empirical equation for terminal velocity.

\[ U_t = \left[ \frac{6.8}{1.65 - \Delta \rho / \rho_d} \sqrt{\frac{\sigma}{(3 \rho_d + 2 \rho_c)}} \right] / \sqrt{d} \quad \ldots 2.8 \]

They also showed that for very pure systems the value obtained for non-oscillating drops greatly exceeded those previously reported.

2.3.3.2 Drop Interaction.

Drop interaction in liquid-liquid dispersions is characterized by the breakage and coalescence of droplets. Drops break as a result of the turbulent eddies surrounding their surface which causes pressure fluctuations on their surfaces whereas coalescence occurs through the collision of two drops.
Drop Break-up.

The theory of drop break-up, based on the theory of homogenous, isotropic turbulence was developed by Kolmogoroff (K2), he derived the following relationship for maximum stable drop diameter.

\[ r_s = 2 \left( \frac{D_A}{U} \right)^{0.4} (\sigma / \kappa_f \rho_C)^{0.6} \] ...2.9

where \( U \) is the isotropic flow velocity, \( \kappa_f \) is a coefficient, \( r_s \) is the radius of a stable drop, the above equation was also developed independtly by Hinze (H12).

Sleicher (S11) tested equation (2.9) by studying the stable drop size of dilute dispersions flowing through pipes under turbulent conditions. He observed, using high speed photography, that there were at least two type of break-up mechanisms. He found that break-up of drops occurred by the drops stretching considerably and then fragmenting into two drops of approximately the same diameter. The second type of break-up mechanism observed was that of a small drop being stripped from a larger one (this type being less frequent than the first), and proposed the following correlation for maximum stable drop size:

\[ N_{we} (\mu_C U / \sigma)^{0.5} = 38 \left[ 1 + 0.7 (\mu_d U / \sigma)^{0.7} \right] \] ...2.10

It must be noted that throughout Sleicher's experimental work the dispersed phase hold-up never exceeded 1.7%, thus eliminating the possibility of coalescence.

Drop Coalescence.

The coalescence of two drops is accomplished by the draining and rupture of the film of the continuous phase. Most of the research work reported on coalescence was for coalescence between a drop and a flat interface. A list of this work is given by Tavlarides and Stamatoudis (Tl, pg.215), and a
summary of coalescence and the factors affecting coalescence is
given by Jeffreys and Davies (J5).

The basic expression describing drop coalescence rate is given
by Tavalides and Stamatoudis and is:

\[ F(a,a') \, d(a') = \lambda(a,a') \, z(a,a') \, d(a') \quad ...2.11 \]

where \( F(a,a') \, d(a') \) = No of coalescences per unit volume of
dispersion per unit time.

\( \lambda(a,a') \) = Collision efficiency of a collision
between droplets of sizes \( a \) and \( a' \).

\( z(a,a') \, d(a') \) = No of binary collision between drop of
size \( a \) and \( a' \) per unit volume of dis­
persion per unit time.

\( a,a' \) = drop diameter.

They also discussed the most important developments by various
workers for the collision efficiency and collision rate, point­
ing out that gravity can play an important role in collision
rates for systems with large density difference between con­
tinuous and dispersed phase.

Jeffrey and Hawksley (J4) studied the effect of physical pro­
erties on the rate of coalescence and proposed a correlation
of coalescence time and coalescence time distribution with
the physical properties of the system.

Delichatsios and Probstein (D6) derived an equation between
coalescence frequency resulting from binary collisions and an
effective break-up frequency which yielded a semiempirical re­
relationship for the increase in drop sizes with hold-up.

They reported that increased drop size with higher fractional
hold-up can only be accounted for by allowing for coalescence,
turbulence damping playing a secondary role. This is contrary
to the agruments put forward by Doulah (D6) who argued that
the increase in mean drop diameter can be adequately explained
by increased damping of the turbulence.
Johnson and Bliss (J8) reported that the rate of coalescence of drops increased when solute transfer occurred from the droplet phase into the continuous phase and decreased during the reverse transfer.

Groothius and Zuiderweg (G11, G12) demonstrated with the aid of photographs, the influence of direction of solute transfer. They showed that the addition of about 1.5 percent by weight of solute to the dispersed phase resulted in a twenty-fold increase in coalescence rates.

2.3.4 Mass Transfer To and From Drops.

The complex process by which solute is transferred from one liquid media to another liquid media has been investigated by numerous workers over the past two decades. Initially, workers studied mass transfer between a single solid particle and the surrounding fluid. The solid particle behaviour was then extended to liquid droplets, first to the single drop system and then to the multi drop system.

2.3.4.1 Mass Transfer in the Dispersed Phase.

The extent of mass transfer in the dispersed phase ranges from mass transfer from a small drop (rigid sphere) with no circulation, to transfer from a relatively large drop (non-rigid) with fully developed internal circulation.

(a) Rigid Drop.

Treybal (T14 pg.186) proposed the following correlation for the unsteady state variation of average solute concentration $x$ with $t$:

$$
\frac{x_0 - x}{x_i - x} = \left[1 - \exp\left(-4D \frac{2}{\rho d^2} t\right)\right]^{0.5} \quad \ldots 2.12
$$
where \( x_0 \) and \( x_i \) are the initial uniform solute concentration and the constant interfacial value respectively, \( D_d \) the diffusivity of solute in the dispersed phase, and \( d_p \) is the particle diameter. This relationship assumes that there is no continuous phase mass transfer resistance. For a linear concentration difference driving force they proposed the following relationship for the dispersed phase mass transfer coefficient \( k_d \):

\[
k_d = \frac{2\pi D_d}{3d_p}
\]

...2.13

(b) Non-Rigid Drop.

The transfer to and from non-rigid drops is more complex due to internal circulation and drop oscillation. As a result of this the mixing in the drop is by turbulent and laminar circulation.

Kronig and Brink (K8) proposed a relationship for the regime of laminar circulation using Hadamard stream lines for drop circulation, which when simplified (T14, T8) resulted in the following relationship for the dispersed phase mass transfer coefficient:

\[
k_d = 17.95 \zeta_d / d_p
\]

...2.14

Handlos and Baron (H3) considered the case of fully developed internal circulation (turbulent regime) and proposed the following relationship:

\[
k_d = \frac{0.00375 U_t}{1 + \left( \mu_d / \mu_c \right)}
\]

...2.15

This relationship was verified by Skelland and Welleck (S8) and by Johnson and Hamilec (J6). Orlander (O3) reported deviations on the Handlos and Baron's model for short contact times, and suggested a correction to their equation resulting in the following relationship:
\[ k_d = 0.972 \, k_{HB} + 0.075 \, \frac{d}{t} \quad ...2.16 \]

where \( k_{HB} \) is dispersed phase coefficient as calculated by the Handlos and Baron model.

Most of the workers combined the effect of oscillation with internal circulation. However, Heertjes and De Nie (H9) reported that there are cases in which the effect of oscillation is known to be much larger than the effect of circulation. Angelo et al (A3) using a model based on surface stretch and internal mixing of the drop, proposed the following empirical relationship for \( k_d \):

\[ k'_d = (4\Phi \omega (1+\varepsilon_0)/\pi)^\frac{1}{2} \quad ...2.16a \]

where \( \omega \) is the frequency of oscillation and \( \varepsilon_0 \) a dimensionless factor for the amplitude.

2.3.4.2 Mass Transfer in the Continuous Phase.

(a) Rigid Drops.

Griffiths (G10) studied mass transfer from a single stationary drop suspended in a flowing continuous phase media. He proposed the following correlation for the continuous phase mass transfer coefficient:

\[ (N_{Sh,c}) = 2 + \beta (N_{Re,c})^{\frac{1}{2}} (N_{Sc,c}) \quad ...2.17 \]

where the value of \( \beta \) varied from 0.63 to 0.70 and is dependent on the droplet-phase liquid. Similar correlations were also proposed by Rowe, Claxton and Lavis (R10) and Keey and Glen (K1) with a value of 0.76 for \( \beta \).

(b) Non-Rigid Drop.

Numerous workers (E4, G4, G5, H17, R7, W1) have reported continuous phase mass transfer coefficient correlations for circulating and oscillating drops.
Griffith (G10) used boundary theory and hypothetical streamline pattern inside the drop and proposed the following correlation for continuous phase film coefficient:

\[(N_{Sh})_c = 2 + 1.13 (N_{Re})^{1/2} (N_{Sc})^{1/2} (k_r)^{0.33} \]  \(2.18\)

or \[(N_{Sh})_c = 2 + 1.13 (N_{Pe})^{1/2} k_r^{0.33} \]  \(2.19\)

where \(k_r\) is a function of \((U_d/U_c)\) and \(N_{Re}\).

This equation reduces to the Boussinesq (B5) type equation for circulating drops at high \(N_{Pe}\) values as given below

\[(N_{Sh})_c = 1.13 (N_{Pe})^{1/2} \]  \(2.20\)

Hughmark (H17) reported correlations for mass transfer in a spray column. He suggested that continuous phase mass transfer coefficient can be predicted from correlation for single drops providing the viscosity ratio \(\mu_c/\mu_d\) is less than one. The following correlation was proposed for the oscillating liquid drop:

\[N_{Sh} = \left[ 2 + 0.084 (N_{Re})^{0.484} (N_{Sc})^{0.339} \left( \frac{d_p g}{\mathcal{D}} \right)^{0.072} \right]^{3/2} \]  \(2.21\)

### 2.3.4.3 Mass Transfer in Drop Swarms.

Ruby and Elgin (R7) were among to the first research workers to report data for mass transfer in drop swarms. They proposed a graphical correlation for continuous phase mass transfer coefficient which was modified by Treybal (T14) resulting in the following correlation:

\[(N_{Sh})_c = 0.725 (N_{Pe})^{0.57} (N_{Sc})^{-0.15} (1 - \phi_d) \]  \(2.22\)

Hughmark (H17) compared mass transfer data for single drop and swarms systems. By using the single drop correlation and the Ruby and Elgin correlation for a drop swarm, he found for viscosity ratios \((\mu_c/\mu_d)\) less than one, the mass transfer coefficients for single drop, and drop swarm, were approxi-
mately the same, while for viscosity ratios greater than one, interaction effect of the drop swarms reduced the coefficient from that of a single drop. Other workers who reported data on drop swarms include Gal-or and Hoelscher (G1), Calderbank and Moo-Young (C2), and Olney (O2).

More recently Korchinsky and Cruz-Pinto (K6) used local rather than average mass transfer coefficients to calculate mass transfer rates. They predicted extraction efficiencies for rigid and non-rigid isolated drops and compared the predictions and experimental data by applying the isolated drop model predictions to swarms of drops in a R.D.C. They reported that model predictions of extraction efficiencies for swarms of drops can only be obtained with sufficient accuracy (±20%) if the effects of varying continuous phase concentration and dispersed phase mass transfer coefficients, and of drop size distribution, are included in the theoretical model.

2.3.5 Interfacial Turbulence.

The effect of interfacial turbulence on mass transfer in liquid-liquid extraction is of prime importance, due to the substantial increase it induces on the rates of mass transfer between the two phases. This phenomena has been studied by numerous research workers over the past four decades. Wei (W4) reported a variety of interfacial disturbances, ranging from rippling and twitching of the interface to spontaneous emulsification in the vicinity of the boundary between phases.

Garner et al (G3), reported interfacial behaviour of pendant drops of various organic liquids containing 2 to 30% solute with distilled water as the surrounding liquid. They found that twitching and rippling of the interface occurred from time to time with periodic pulsations of the entire drop. This phenomena was also observed by Garner et al (G2) for drop moving slowly in a liquid media.

An excellent review of interfacial phenomena is provided by
Sternling and Scriven (S16). They suggested that interfacial phenomena can essentially be explained by the Marangoni effect, wherein movement in an interface is caused by longitudinal variations of interfacial tension. Using a simplified mathematical model they suggested a number of factors by which interfacial turbulence is promoted.

An extensive review of interfacial phenomena and the research work reported in this field is given by Sawistowski (S1), a more theoretical treatment of this phenomena is given by Davies and Rideal (D4).

2.4 Axial Mixing.

In liquid-liquid extraction a knowledge of the extent of axial mixing is important, since this is one of the important factors that affects the extraction efficiency of the contactor.

The literature on axial mixing is extensive, and only the work that is pertinent to the present study will be referred to. A good understanding of the subject can be obtained from Misek and Rod (M5) and Ingham (I1), who has written an excellent summary of the literature on axial mixing.

Levenspiel and Smith (L5) suggested that longitudinal mixing of fluid in flow can be characterized by a single parameter $E$, the longitudinal or axial dispersion coefficient. Methods for determining this parameter from residence time distribution studies are reported by Levenspiel and Smith (L5), Van de Laan (V1), Aris (A2) and Bischoff (B3).

Strand, Olney and Ackerman (S17) studied axial mixing in a R. D.C. They defined the axial mixing effect as a combination of the eddy diffusion effect, and a spreading effect that is specific in the forward direction, the latter effect being due to either channelling or to the Taylor type of diffusion (T2). They criticized the validity of applying a simplified dispersion coefficient to drop population and proposed the
following expression for single phase flow:

\[ \frac{E_c}{UH} = 0.5 + 0.09 \left( \frac{D_A N}{U} \right) \left( \frac{D_A}{D} \right)^2 \left[ \left( \frac{S}{D} \right)^2 - \left( \frac{D_A}{D} \right)^2 \right] \] ...

for \( D_A N / U < 30 \)

where \( H \) is the compartment height and \( S \) is the stator ring opening other variables are described in the nomenclature of the present work.

About the same time as Strand et al (S17), an independent paper by Westerterp and Landsman (W6) on axial mixing appeared. They showed that the apparent axial diffusivity may be considered as the sum of a flow contribution and a rotational contribution which can be represented by the following expression:

\[ E_c = C_1 N + C_2 U \] ...

Klinkenberg (K3) presented a detailed discussion of axial mixing studies reported by a number of workers. In a separate short note (K4) a few years later he discussed the concept of backmixing, which he divided into "backmixing(1) and "backmixing(2). He defined the former as a mechanism by which a phase moves against its flow and the latter as any mechanism by which a spread of residence time is brought about.

The effect of axial mixing on extraction efficiencies was studied by Sleicher (S10), Miyauchi and Vermulen (M7) and Bibaud and Treybal (B2). They all used the diffusion model as developed over a number of years by various research workers. Rod (R4) claimed that the methods used by the above workers are based on a simplified model and their application is limited, especially by the condition of linear equilibrium relationship. He proposed a finite difference method for solving the diffusion model. This solution can be used for non-linear equilibrium relationship and is straightforward in case of backmixing in single phase.
Hold-up of dispersed phase in liquid-liquid contactors has been studied by many workers, and the data reported is highly dependent on the method used to determine it. The methods widely used are (a) the drainage method (b) displacement and (c) the manometric method, which are adequately described in the text (L2,T14).

Minard and Johnson (M2) studied hold-up in a spray tower of the Elgin design (E3). They used a tetrahedron model with droplet fixed at each of the corners to express the hold-up in terms of drop diameter and interstice area as follows:

$$\phi = \frac{\sqrt{2\pi}}{6} \frac{(D/x)^3}{...2.25}$$

They used the manometric method to measure pressure drop across the contactor and found the pressure drop for the continuous phase expressed as feet of continuous phase per foot height of column is proportional to hold-up for any specific liquid system. Hughmark (H17) also studied hold-up in a spray column. He reported that dispersed phase hold-up can be predicted as a function of the ratio of the dispersed phase superficial velocity to the terminal velocity of the drop.

Misek (M3) reported hold-up studies for different types of agitated liquid extractors. He investigated the effect of flowrates, drop velocity and settling and coalescence of drops on dispersed phase hold-up. The following equation was proposed:

$$\frac{U_d/\phi}{U_c} = U_o (1-\phi) \exp \left[ \phi \left( Z_c - 4.1 \right) \right] \quad ...2.26$$

where $Z_c$ the coalescence coefficient and $U_o$ the characteristic velocity are constants for a given system and given mixing rate. He claimed that this equation is more accurate than the ones proposed by Thornton and co-workers (T11) and Pratt (P2)
in describing the hydrodynamic behaviour of an agitated extractor.

Gayler and Pratt (G6) working with packed columns, proposed a simple expression for hold-up as follows:

\[
\phi = \frac{U_d}{\left(\varepsilon U_0 - \left(U_c / \gamma \right)\right)} \tag{2.27}
\]

where \( U_0 \) is the mean drop velocity at \( U_c = 0 \), \( \varepsilon \) is the fractional free space of packing and \( \gamma \) is the fraction of free cross-sectional area of the column used for countercurrent flow of continuous phase.

Johnson and Lavergne (J7) suggested that this equation for calculating hold-up was overly simplified and they proposed a more complex equation which can be used for any liquid extractor.

With regards to the methods used for measuring hold-up, Gayler and Pratt (G6) found that the manometric method gave inaccurate results. He explained this algebraically by assuming the solvent is present as a continuous stream instead of discrete droplets. Their measurements were done by the drainage method.

On the other hand, Vermijs and Kramers (V3) working with the R.D.C found that the manometric method measurements were far more accurate than the displacement method. The manometric method was also used successfully by Minard and Johnson (M2), and Chiu (C5), Weng (W5), and Clare (C7) working with single oscillating baffle column of small sizes.

2.6 Flooding Studies.

Flooding in the various types of extractors has been investigated by numerous workers. The pioneering work on flooding in liquid-liquid contactors was done by Blanding and Elgin (B4) working with spray towers. They proposed three equations which
can be used to calculate flow rates at flooding.

Minard and Johnson (M2) carried out flooding studies in a spray tower and proposed the following correlation for limiting flow:

\[ V_c^{0.5} = \left[ -1.80 d_p^{0.050} \mu_c (\rho_d^{1.0}/\rho_c^{1.0})^{0.5} \right] V_d^{0.5} + \left[ 47 \mu_c^{0.070} \Delta \rho^{0.14} \rho_c^{0.50} \right] \]

...2.28

Logsdail, Thornton and Pratt (L8) reported flooding studies for a R.D.C using a number of solvent-water systems. Data was interpreted in terms of the characteristic droplet velocities \( \bar{U}_N \) (i.e. mean droplet velocity at substantially zero flow rates) and these were correlated with the column geometry and physical properties as follows:

\[ \frac{\bar{U}_N^{1/2}}{\sigma} = 0.012 (\Delta \rho)^{0.90} \left( \frac{g}{D_A} \right)^{1.0} \left( \frac{d_i}{D_A} \right)^{2.3} \left( \frac{h}{D_A} \right)^{0.90} \left( \frac{D_A}{D} \right)^{2.7} \]

...2.29

where \( d_i \) is the internal diameter of stator and \( h \) is the stator spacing.

Smoot, Mar and Babb (S14) proposed a generalized correlation for calculating flooding conditions in pulsed sieve-plate column. They derived a dimensionless equation and used the flooding data reported by four workers (665 experiments) to obtain the following correlation:

\[ U_c + U_d = 3.20 \times 10^5 \Delta \rho \mu_c^{0.63} d^{0.45} \frac{1}{(\psi_f \mu_d^{0.20} U_d^{0.14} \sigma^{0.14})} \]

...2.30a

where \( \psi \) is a power function defined by

\[ \psi = \frac{2^2 (1-\varepsilon)^2 (fa)^3}{2 \varepsilon \sigma_c L} \]

...2.30b
and \( f = \) pulse frequency
\( a = \) pulse amplitude
\( \varepsilon = \) fractional free space per plate
\( C_0 = \) orifice coefficient = 0.60
\( L = \) plate spacing

It was found that the total flooding velocity was independent of the extractor diameter.

Flooding studies have also been reported by Johnson and Bliss (J8), Elgin and Foust (E2) Dunn, Lapidus and Elgin (D9) for spray contactors, by Dell and Pratt (D9), Venkataraman and Laddha (V2) for packed contactors, by Reman (R2), Thornton and Pratt (T10) for rotary agitated contactors and by Jackson et al (J1), Hafez et al (H2), and Bender et al (Bl) for pulsed agitated contactors.

It is important to point out that the majority of flooding, or limiting flow studies which are the basic criteria used for fixing the optimum operating conditions for contactors, has mainly been done with non-transfer systems. The presence of solute can have a significant effect on interfacial tension and hence on the hydrodynamic conditions in the contactor. Thus, the use of solute free correlations for establishing optimum operating conditions can give inaccurate results, since the primary function of the contactor is the extraction of solute.

2.7 Power Studies.

White and co-workers (W7) were the first to point out the possibility and advantages of correlating the performance of mixing impellers by the use of dimensional analysis and a study of models. Further theoretical analysis were reported by Hixon and co-workers (H13, H14).

The classic work on industrial mixing operations was that of Rushton, Costitch and Everett (R8). They carried out a com-
prehensive study on the effect of physical properties of liquid, impeller size and type, tank size, and baffles on power consumption. They correlated their data in the form of dimensionless parameters and reported power data for Reynolds number ranging from 1 to $10^7$. They proposed a general correlation for power in terms of physical variables most often encountered in mixing with a single impeller, centered in a cylindrical, vertical axis, flat-bottomed tank. For a system with fixed geometric boundary conditions, the equation reduces to:

$$N_p = K(N_{Re})^m (N_{Fr})^n \quad \ldots 2.32$$

and for a fully baffled vessel where $(N_{Fr})^n$ is equal to 1 equation (2.32) becomes:

$$N_p = K(N_{Re})^m \quad \ldots 2.33$$

For baffled mixers, $m$ varies from -1.0 in the streamline region to zero for fully developed turbulence. The three regions are as follows:

$$\begin{align*}
\text{Re} &< 10 \quad \text{Streamline Region} \\
10 < \text{Re} &< 10^4 \quad \text{Transition Region} \\
\text{Re} &> 10^4 \quad \text{Turbulent Region}
\end{align*}$$

Flynn and Treybal (F1) carried out power studies on continuous flow six bladed turbine agitated extractors. The systems studied were toluene - water - benzoic acid and kerosene - water - benzoic acid. It was found that the power required for a given impeller speed was independent of the rate of flow of liquid. Extraction stage efficiencies were found to be appreciable at zero agitator speed, and the additional stage efficiency produced by agitation was found to be a function of the power input per unit volume of liquid flowing.

Miller and Mann (M1) reported power data for various designs of agitators operating in single and two-phase liquid mixtures in
unbaffled tanks. They found that the power required to agitate a system of two immiscible liquids may be correlated by a power function - Reynolds number plot, if the average density of the mixture, and a weighted mean of the viscosities of the two phase are used in calculating these functions. They also reported that successful scaling up of small scale mixing equipment can be achieved by designing large equipment which is geometrically similar and by applying equal power per unit volume in both sizes.

Olney and Carlson (01) basing their methods of analysis on that of Miller and Mann (M1) reported power data for disperser and spiral turbine impellers operating in several single liquid and two phase immiscible liquid systems. In order to bring the power equation (Equ.2.33) into a form comparable with the standard heat and mass-transfer equations, they modified it by multiplying both sides by the Reynolds number resulting in the following equation:

\[ \frac{P_v g_c}{N^2 \mu} = C_1 \left( \frac{N^2 \rho}{\mu} \right)^{X_f} \]

where \( P_v \) the power per unit volume replaces the \( P/d^3 \) term and \( x_f \), is the Reynolds index for fluid friction or momentum transfer. The modified power number is termed by "power function", and it represents the ratio of applied torque to viscous forces. By using average densities and viscosities for the mixtures, fair agreement between single and two phase power data has been obtained.

Jealous and Johnson (J2) studied power requirement for generating pulses in a 50 ft high and 24 inches diameter sieve plate liquid-liquid extraction column. They proposed an equation in differential form for calculating the theoretical power applied by the pulser.

Misek (M4) studied power consumption for discs rotating in geometrical arrangement, usually found in R.D.C's. The results has been reported in a form similar to Rushton et al (R8, equ.
2.33). They compared their results with previous research workers and suggested their results can be applied with confidence also to large diameter discs.

Some of the other workers to report power studies in mixers were Reiger and Novak (R3) who studied mixing of non-newtonian fluid with various types of conventional agitators, and Ulbrecht (U1) who studied agitation with flat-bladed turbines.
CHAPTER 3

THEORY
3.1 Analysis Of Extractor Efficiency

3.1.1 Equilibrium

In liquid-liquid extraction processes, the extent of transfer of solute from one liquid (feed) phase to another liquid (solvent) phase is dependent on how far removed from equilibrium the system is. Hence, accurate knowledge of phase equilibrium of the component involved is of prime importance in the determination of extraction efficiency.

For two phase ternary liquid systems, distribution curves are used to establish relationships between solute content in the phase at equilibrium. The simplest and most widely used distribution curve consists of a plot of the concentration of solute in the solvent phase against equilibrium concentrations of solute in the feed phase on rectangular co-ordinates.

Thus if \( y^* \) represents the solute concentration in the solvent phase on \( x^* \) the solute concentration in the feed phase at equilibrium, - for the present studies \( y \) represents solute concentration in the aqueous (continuous) phase and \( x \) the concentration in the organic (dispersed) phase - then from Nernst's distribution Law (N1). The two concentrations are related by the following equation;

\[
y^* = m \times^* \tag{3.1}
\]

i.e. \( \frac{y^*}{x^*} = m = \text{constant.} \)

Where \( m \) is the distribution coefficient.

However, this law in its simplest form has theoretical limitations in that it applies only to (a) dilute, almost ideal solutions, and (b) cases where the solute has the same molecular weight in both solvents. When the solute does not have the same molecular weight in the two liquid phases the simple form of the distribution law breaks down and allowance must be made for this in the equilibrium relationship for the law to be applicable.
One of the systems used in the present studies is \( \text{CCL}_4\text{-HAC-H}_2\text{O} \). Chaikhorskii et al. (C13) studied the behaviour of acetic and \( \text{HAC} \) in carbon tetrachloride \( \text{CCL}_4 \) under conditions of extraction and found that acetic and molecules are present in \( \text{CCL}_4 \) predominantly in a dimerized state. This presented a problem in the present studies, since the molecular weight of the solute in the two phases is not the same, thus making the simple form of the distribution law inapplicable (i.e. \( 'm ' \) is not constant, but varies with concentration).

In order to overcome this difficulty, equilibrium data was determined experimentally as described in Appendix A.1.2. This data was fitted to an equation of the form;

\[
y^* = ax^b
\]

which was used as the equilibrium relationship for the \( \text{CCL}_4\text{-HAC-H}_2\text{O} \) system where necessary.

It is important to point that this type of relationship is an empirical correlation of the equilibrium data and must not be confused with the simple relationship associated with the distribution law as described above.

3.1.2 Concentration Profile Based On Piston Flow Assumptions

Consider solute being transferred from the organic phase to the aqueous phase which are flowing countercurrently through the extractor as shown in Fig. 3.1. Let the solute concentration - based on solute free solvent - in the aqueous continuous and organic dispersed phases be \( y \) and \( x \) respectively and \( Q_c \) and \( Q_d \) the flowrates of the dispersed and continuous phases respectively.

Assuming piston flow, at steady state the material balance over the whole contactor (Fig. 3.1) is;

\[
\text{CCL}_4\text{-HAC-H}_2\text{O system} \quad \text{where} \quad y_B = 0
\]
Fig. 3.1 Piston Flow Model for Evaluation of Extractor Efficiency

- → CCL\textsubscript{4} - HAC - H\textsubscript{2}O system \( y_B = 0 \)
- → MIBK - HAC - H\textsubscript{2}O system \( y_T = 0 \)
\[ Q_c y_B + Q_d x_r = Q_c y_T + Q_d x_B \]  
\[ \text{putting } y_B = 0, \text{ phase ratio } \frac{Q_c}{Q_d} = R \text{ and rearranging} \]
\[ x_T = R y_T + x_B \]

Thus the general relationship between concentrations in the phase at any section in the contactor will be, for the nth section

\[ x_n = R y_n + x_B \]  
\[ \text{MIBK-HAC-H}_2\text{O system - where } y_T = 0 \]
\[ Q_c y_T + Q_d x_B = Q_c y_B + Q_d x_T \]
\[ \text{putting } y_T = 0, \text{ phase ratio } \frac{Q_c}{Q_d} = R \text{ and rearranging} \]
\[ x_B = R y_B + x_T \]

and the general relationship for any position n is;

\[ x_n = R y_n + x_T \]

### 3.1.3 Number of Transfer Units - NTU

From the two-film theory (Chapter 1.2.2) the rate of solute transfer in terms of the overall mass transfer coefficients are (Eqns. 1.3-1.4).

\[ N = K_c a \ (y^* - y) \]  
\[ N = K_d a \ (x - x^*) \]

where \( N \) = Rate of mass transfer per unit volume

\( K_c a, K_d a \) = Overall mass transfer coefficient base on continuous and dispersed phase respectively.

If A is the cross-sectional area of the contactor, then the
mass transfer rate in a differential section \( dh \) (Fig. 3.1) is given by

\[
dN = K_c a (y^* - y).A.dh \quad \ldots \ldots \ (3.11)
\]

and

\[
dN = K_d a (x - x^*).A.dh \quad \ldots \ldots \ (3.12)
\]

A material balance over the differential section \( dh \) gives;

\[
dN = Q_c (y + dy) - Q_c y = Q_d (x + dx) - Q_d x \quad \ldots \ldots \ (3.13)
\]
i.e. \( dN = Q_c dy = Q_d dx \quad \ldots \ldots \ (3.13a) \)

Substituting for \( dN \) in equations (3.11) and (3.12)

\[
Q_c dy = K_c a (y^* - y).A.dh \quad \ldots \ldots \ (3.14)
\]

\[
Q_d dx = K_d a (x - x^*).A.dh \quad \ldots \ldots \ (3.15)
\]

From (3.16) \[
\frac{dy}{(y^* - y)} = \frac{K_c a . A.dh}{\theta_c} \quad \ldots \ldots \ (3.16)
\]

From (3.15) \[
\frac{dx}{(x - x^*)} = \frac{K_d a . A.dh}{\theta_d} \quad \ldots \ldots \ (3.17)
\]

The left hand side of equations (3.16) and (3.17) may be recognized as the number of times the driving force \( (y^* - y) \) and \( (x - x^*) \) can divide the change in extract and raffinate compositions \( dy \) and \( dx \) respectively. The number of transfer units which is a measure of the difficulty of separation are obtained by integrating equations (3.16) and (3.17) giving;

\[
NTU_c = \frac{K_c a . A.H}{Q_c} = \int_{y_1}^{Y_5} \frac{dy}{y^* - y} \quad \ldots \ldots \ (3.18)
\]

and \( NTU_d = \frac{K_d a . A.H}{Q_d} = \int_{x_1}^{X_5} \frac{dx}{(x-x^*)} \quad \ldots \ldots \ (3.19) \)
3.1.4 Height Of Transfer Unit - HTU

For piston flow the length of transfer unit is defined as follows.

\[ \text{HTU}_C = \frac{H}{\text{NTU}_C} \]  \hspace{1cm} (3.21)
and \[ \text{HTU}_d = \frac{H}{\text{NTU}_d} \]  \hspace{1cm} (3.22)

From equations (3.18) and (3.19)

\[ \text{HTU}_i = \frac{Q_{i/A}}{K_i a} = \frac{U_i}{K_i a} \]  \hspace{1cm} (3.23)

where \( i \) refers to the particular phase.

Equation (3.23) can be recognized as the ratio of volumetric flow rate per unit cross section to the overall mass transfer coefficient.

3.1.5 True Value of NTU

The definition of the true value of NTU based on Colburn (144) is as follows;

\[ \text{NTU}_{ct} = K_{ca} \cdot \frac{H}{V_c} \]  \hspace{1cm} (3.24)
and \[ \text{NTU}_{dt} = K_{ca} \cdot \frac{H}{V_d} \]  \hspace{1cm} (3.25)

where \( K_{ca} \) is the true mass transfer coefficient for the system. True values of \( K_{ca} \) can be obtained by solving the one-dimensional diffusion model (equation 3.65).

The true height of transfer unit is defined as;

\[ \text{HTU}_t = \frac{H}{\text{NTU}} \]  \hspace{1cm} (3.26)

3.1.6 Measured and Apparent Measured Values of NTU

The concept of measured values of NTU has been introduced by Miyauchi and Vermulen (M8). Their purpose was to include the deviations from piston flow due to axial mixing. By
sampling both phases, they used real concentration profiles to calculate the number of transfer units by graphical integration of the following equation (See Fig. 3.1).

\[ NTU_{cm} = \int_{y_1}^{y_5} \frac{dy}{y^* - y} \quad \ldots \ldots (3.27) \]

and

\[ NTU_{dm} = \int_{y_1}^{y_5} \frac{dx}{x - x^*} \quad \ldots \ldots (3.28) \]

and the measured values of HTU are given by

\[ HTU_{cm} = \frac{z}{NTU_{im}} = \frac{K_{a_{im}}}{U_i} \quad \ldots \ldots (3.29) \]

i refers to the particular phase under consideration.

A more detailed treatment of true and measured number of transfer units is presented in Section 3.3.

Thomas (T6) and Thomas and Chiu (T5) introduced the concept of "Apparent Measured NTU" which can be regarded as an extension of Miyauchi and Vermulien (M8) work. The difference between the "Measured" and "Apparent Measured" NTU is mainly experimental. For the latter actual values of y were obtained from experiment and x was calculated from a material balance assuming piston flow, while for the former both were obtained from experiment. This was due to the practical difficulties encountered in trying to sample the dispersed or "x" phase directly.

The apparent NTU is evaluated by applying the piston flow model (Fig. 3.1) with graphical integration of the following equations;

\[ NTU_{c\, \text{App.m}} = \int_{y_1}^{y_5} \frac{dy}{(y^* - y)} = \frac{H}{HTU_{c\, \text{App.m}}} \quad \ldots \ldots (3.30) \]

and

\[ NTU_{d\, \text{App.m}} = \int_{y_1}^{x_5} \frac{dx}{(x - x^*)} = \frac{H}{HTU_{d\, \text{App.m}}} \quad \ldots \ldots (3.31) \]
### 3.1.7 Piston Flow Value of NTU

In the absence of axial mixing and if the operating and equilibrium lines are linear, equations (3.18) and (3.19) can be integrated analytically to yield a log mean NTU based on end conditions;

\[
NTU_{cp} = \int_{y_1}^{y_5} \frac{dy}{y - y^*} = \frac{y_6 - y_1}{(y^* - y)_{LM}} \quad \ldots \quad (3.32)
\]

and

\[
NTU_{dp} = \int_{y_1}^{y_5} \frac{dx}{x - x^*} = \frac{x_6 - x_1}{(x - x^*)_{LM}} \quad \ldots \quad (3.33)
\]

where

\[
(y^* - y)_{LM} = \frac{(y_5^* - y_5) - (y_1^* - y_1)}{\ln \left[ \frac{(y_5^* - y_5)}{(y_1^* - y_1)} \right]} \quad \ldots \quad (3.34)
\]

and

\[
(x - x^*)_{LM} = \frac{(x_5 - x_5^*) - (x_1 - x_1^*)}{\ln \left[ \frac{(x_5 - x_5^*)}{(x_1 - x_1^*)} \right]} \quad \ldots \quad (3.35)
\]

Hence piston flow HTU is defined by

\[
HTU_{cp} = H/NTU_{cp} \quad \ldots \quad (3.36)
\]

### 3.2 Residence Time Studies

#### 3.2.1 Types of Flow Patterns

When fluid flow through a vessel, the type of flow pattern can be either ideal or non-ideal. There are two types of ideal flow which are regarded as the limiting cases; these are "plug" or "piston flow" and "perfectly mixed" or "backmix" flow. Plug flow occurs when the fluid velocity gradient is uniform over the entire cross section of the vessel. Each element of fluid that enters the vessel moves through in "single-file" without intermingling with other fluid elements that entered
earlier or later. Perfectly mixed flow is the other extreme to plug flow and assumes that the vessel contents are completely homogenous down to a molecular scale - no differences at all exist between the various portions of the vessel, and the outlet stream properties are identical to the vessel-fluid properties.

For actual processes, such as the continuous differential contactor in which there is axial mixing, the flow patterns lie between these two extreme limits of ideal flow and is termed "non ideal flow". When studying the performance of these types of contactors it is important to study the effects of axial mixing on mass transfer since increasing axial mixing results in reduced mass transfer efficiency. The extent of axial mixing is usually evaluated from residence time distribution (R.T.D.) studies in the different phases. For the present studies R.T.D. studies are carried out in the continuous phase only. A detailed description of the method is given in Section 4.7.

3.2.2 Age Distribution Functions and F-Diagram

When fluid flows through a vessel elements of a stream of fluid entering the vessel at the same instant will take different routes through the vessel and hence different lengths of time to pass through the vessel. The distribution of these times when the stream of fluid leaves the vessel is called the residence time distribution - R.T.D. - or exit age distribution E, of the fluid. The "age" of an element of fluid is defined as the time elapsed since it entered the vessel, in the case of RTD the age is the time taken for the fluid element to pass through the vessel.

A typical RTD curve is shown in Fig. 3.2, and is described by the relationship;

\[ \int_{0}^{\infty} E(t) \, dt = 1 \quad \ldots \ldots (3.37) \]

The mean residence time is given by the relationship;

\[ \mu = \int_{0}^{\infty} t \, E(t) \, dt \quad \ldots \ldots (3.38) \]
The $E$-curve can also be defined in terms of dimensionless time, i.e.

$$\int_{0}^{\infty} E(\theta) \, d\theta = 1 \quad \text{......... (3.39)}$$

and

$$\mu = \int_{0}^{\infty} \theta \, E(\theta) \, d\theta \quad \text{......... (3.40)}$$

where $\theta$ is the dimensionless time defined as;

$$\theta = \frac{t}{\bar{t}} = \frac{vt}{V} \quad \text{......... (3.41)}$$

and $\bar{t}$ is the mean holding time or space time based on ideal flow.

The different methods for carrying out RTD studies are adequately described in the texts (F2, H11, L4). By these methods, which are classified as stimulus response techniques, the flow system is disturbed and the systems response to the disturbance is monitored. Usually the stimulus is a tracer input into the fluid entering the vessel (upstream) and the response is a record of the tracer leaving the vessel (downstream). Two types of input
signal widely used are (a) the step input signal and (b) the pulse input signal. Dankwerts (D3) introduced the notation that the dimensionless response to an up-step injection of tracer be called the F-curve and the dimensionless response for an impulse injection be called the C-curve. For the present studies the RTD is determined by the pulse input signal.

3.2.2.1 C-Curve

When an instantaneous pulse or shot of tracer is injected into the fluid entering the vessel, the response-time curve C is described by the following relationship:

\[ \int_{0}^{\infty} C \, dt = 1 \]  \hspace{1cm} \ldots \ldots (3.42)

using dimensionless time \( \theta \)

\[ \int_{0}^{\infty} C(\theta) \, d\theta = 1. \]  \hspace{1cm} \ldots \ldots (3.43)

where \( C(\theta) \) = fraction of tracer in exit stream at any instant \( \theta \) after tracer injection.

3.2.2.2 F-Curve

When a step function (in time) of tracer is introduced into the fluid entering the vessel, then the concentration time curve for tracer in the fluid stream leaving the vessel measured as \( C/Co \) is called the F-curve.

The C and F curves are usually plotted on a dimensionless basis and can be used to measure the deviation from ideal flow. Typical C and F curves for ideal and non-ideal flow are shown in Fig. 3.3.

Other distribution functions of interest in some applications are the "internal age distribution", I, and the intensity
Fig. 3.3a F - Diagrams

(a) Plug flow
(b) Plug flow with axial mixing
(c) Perfect mixing
(d) Dead water
function, \( A \). Both of these are adequately dealt with in the texts (F2, H11, L4).

### 3.2.2.3 Relations between the Age-Distribution Functions

It is obvious from the definitions above that the age distribution functions are related. The derivations of the relationships between the functions are given in the texts (F2, H11, L4) and can be summarized as follows:

For closed vessels

\[
E(\theta) = C(\theta) = \frac{dF(\theta)}{d\theta} \quad \cdots (3.44)
\]

or

\[
F(\theta) = \int_0^\theta E(\theta) \, d\theta = \int_0^\infty C(\theta) \, d\theta \quad \cdots (3.45)
\]

It is important to emphasize that these relationships were derived for closed vessels, and when the boundary conditions are different from that of a closed system, the C-curve may differ appreciably from the E-curve.

### 3.2.3 Analysis of Age Distribution Functions and F-Diagrams

#### 3.2.3.1 Mean Residence Time

For the present work two different notations are used for mean residence time and are defined as below;

**Ideal flow**

For ideal flow patterns, the mean residence, \( \bar{E} \), is defined as;

\[
\bar{E} = \frac{V}{v} \quad \cdots (3.46)
\]

where \( v = \) volumetric flow rate of fluid

\( V = \) volume of vessel
Non-Ideal Flow

For non-ideal flow where the age distribution function is a spread of elemental residence time, then using the C-curve, the mean residence time, \( \mu \), is defined as:

\[
\mu = \frac{\int_0^\infty t \ C \ dt}{\int_0^\infty C \ dt}
\]

\[
\ldots \ldots (3.47a)
\]

It is clear from the two equations that the values for residence for a given system would be different, and the difference between the two-values (\( t - \mu \)) can be used as a measure of the deviation from ideal flow and not the deviation from plug flow or mixed flow, unless it can be specified with accuracy whether the system is expected to behave as a piston flow system or mixed flow system, e.g. for liquid flowing through a pipe at high Reynolds No. the flow pattern would be expected to be close to "piston flow" while for liquid flowing through a stirred tank where the vessel contents are homogenous, the flow pattern would be close to "mixed flow".

If the distribution curve is only known at a number of discrete time values, \( t_i \), which is the case for the present work, then:

\[
\mu = \frac{\sum t_i \ C_i \ \Delta t_i}{\sum C_i \ \Delta t_i}
\]

\[
\ldots \ldots (3.47b)
\]

where \( \Delta t_i \) is the time interval between concentration readings.

3.2.3.2 Variance

The variance represents the square of the spread of the distribution, and is a measure of the degree of the spread of residence time about the mean and is defined as:

\[
\sigma^2 = \frac{\int_0^\infty (t - \mu)^2 \ C \ dt}{\int_0^\infty C \ dt}
\]

\[
\ldots \ldots (3.48a)
\]
In discrete form;
\[ \sigma^2 = \frac{\sum (t_i - \mu)^2 C_i \Delta t_i}{\sum C_i \Delta t_i} \] ........ (3.48b)

3.2.3.3 Holdback and Segregation

The concept of holdback and segregation was first introduced by Danckwerts (D3) and illustrated by the F-curve.

In a system which displays holdback, some elements of fluid spend more, others less, than the mean residence time \( \mu \) (equation 3.46) in the vessel. The magnitude of the holdback denoted by \( H \), can be defined as the area under the F-diagram between \( Q\theta/V = 0 \) and \( Q\theta/V = 1 \) and is given by;

\[ H = \frac{Q}{V} \int_{\theta=0}^{V/Q} F(\theta) \, d\theta \] ........ (3.49)

\( H \) varies from 0 for piston-flow to values approaching 1 when most of the space in the vessel is dead water. The magnitude of \( H \) is a measure of the deviation from piston-flow.

For the case of mixed flow a single quantity called the "segregation" is used to give an indication of the efficiency of mixing in a system. Again the F-curve is used to determine quantitatively the segregation. This is well described by Danckwerts (D3).

3.2.4 Model for Axial Mixing in Single Phase Flow

There are two approaches to the interpretation of the hydraulic behaviour of the continuous phase in the contactor.

The first method is one described by Levenspiel and Smith (L5) and originally by Geoffrey Taylor (T2, T3). They considered the superimposition of eddy diffusion on plug flow, known as the diffusion model. The method has been well described and
widely used. It has certain disadvantages, mainly concerned with the definition of the limits of use. There must come a point when mixing is so intense that it exceeds the limits of applicability of this model. This can be seen from quotations in the literature, although they are very few, for the values of the Dispersion and Peclet Numbers. It is stated that:

\[ \frac{E}{UL} \rightarrow 0 \text{ or } Pe \rightarrow \infty \text{ negligible dispersion - plug flow} \]

and \( \frac{E}{UL} \rightarrow \infty \text{ or } Pe \rightarrow 0 \) large amount of dispersion - mixed flow

and the intermediate values of \( \frac{E}{UL} \) or Pe are only qualitatively significant in helping to interpret the degree of intermediate mixing. However, this is a useful device when comparing the same apparatus under different conditions of operation.

The second method sometimes used uses a model of a series of perfectly mixed tanks. This is well described by Levenspiel (L4, p. 290) and Misek and Rod (M5). On balance, in the present case of a countercurrent extractor where the backmixing is small a reasonable conclusion is that this particular model is not really as applicable as the diffusion model. It is decided therefore that despite its limitations the dispersion model is to be preferred.

3.2.4.1 Diffusion Model for Axial Mixing

The theory behind the derivation of the diffusion model is well described in the literature and texts (L4, L5, C1l, S3, T2, T3).

When a tracer is injected into the main flow stream of a fluid flowing through a vessel and its behaviour as it moves through the vessel analysed, the differential equation describing axial diffusion superimposed on plug flow is given by:

\[ \frac{\partial c}{\partial t} = E \frac{\partial^2 c}{\partial x^2} + U \frac{\partial c}{\partial x} \quad \ldots \ldots (3.50) \]
In dimensionless form where \( z = x/L \) and \( \theta = t/E = tu/L \) the model becomes:

\[
\frac{E}{UL} \frac{\partial^2 c}{\partial \theta^2} - \frac{\partial c}{\partial z} - \frac{\partial c}{\partial \theta} = 0
\]

\[ (3.51) \]

The parameter \( E \) is called the axial dispersion coefficient and accounts for all the factors causing mixing in the axial direction such as transfer of material in and out of stagnant pockets and local channelling in addition to the effects of turbulent eddy diffusion. It characterizes the degree of axial mixing during flow and is analogous to the coefficient of molecular diffusion as defined by Fick's Law. The dimensionless group \( E/UL \) in equation (3.51) is called the vessel dispersion number which is the parameter that measures the extent of axial dispersion or mixing.

The solution to equation (3.51) based on a modification of Carslaw (C3) solution for a fluid at rest is given by Levenspiel and Smith (L5) as:

\[
\frac{CV}{Q} = \frac{1}{2\sqrt{\pi} \theta (E/UL)} e^{-\frac{(1-\theta)^2}{4 \theta (E/UL)}}
\]

\[ (3.52) \]

where \( CV/Q \) is a dimensionless concentration term. They (L5) went on to state that this solution is for systems where there is a large degree of axial mixing—when \( E/UL \) is of the order 0.01 and greater—presented as a family of C-curves for such a system as shown in Fig. 3.4.

For systems where there is a small degree of axial mixing the values of \( \theta \) must be taken close to 1 (otherwise the values of \( CV/Q \) are essentially zero). For \( \theta \) close to 1 equation (3.51) reduces to

\[
\frac{CV}{Q} = \frac{1}{2\sqrt{\pi} (E/UL)} e^{-\frac{(1-\theta)^2}{4 E/UL}}
\]

\[ (3.53) \]

He also presented a set of curves for this solution as shown in Fig. 3.5.
Levenspiel (L4) used equation (3.52) for the closed vessel system and equation (3.53) for the open vessel system.

![Diagram](image)

**Fig. 3.4 C** - Curves for Large Values of Dispersion Numbers. Eqn. 3.54 (Reproduced from Levenspiel and Smith (L5)).

![Diagram](image)

**Fig. 3.5 C** - Curves for small values of Dispersion Number Eqn. 3.55 (Reproduced from Levenspiel and Smith (L5)).
3.2.5 Peclet Number

The Peclet Number is a dimensionless group which is also used to measure the extent of axial mixing; it is the reciprocal of the dispersion number and is defined as:

\[
P_{e} = \frac{UL}{E} = \frac{1}{\text{Dispersion Number}} \quad \cdots \cdots (3.54)
\]

For a closed vessel Van der Laan's (VI) relationship between variance and Peclet Number is

\[
\sigma^2 = \frac{2}{P_{e}} \left( P_{e} - 1 + e^{-P_{e}} \right) \quad \cdots \cdots (3.55)
\]

and for the open vessel system Levenspiel and Smith's (L5) relationship is:

\[
\sigma^2 = \frac{2}{P_{e}} + \frac{8}{P_{e}^2} \quad \cdots \cdots (3.56)
\]

3.2.6 Determination of Axial Mixing Parameters from R.T.D. Studies

3.2.6.1 Mean Residence Time and Variance

The mean residence times are determined from impulse dye injection studies as follows;

\[
\mu = \frac{\sum t_i C_i \Delta t}{\sum C_i \Delta t} \quad \cdots \cdots (3.47b)
\]

where \( C_i \) is the measured response at time \( t_i \) after injection and \( \Delta t \) is the time interval between readings and is constant.

The variance is also calculated from the general equation (3.48b) in discrete form as follows:

\[
\sigma^2 = \frac{\sum (t_i - \mu)^2 C_i \Delta t}{\sum C_i \Delta t} \quad \cdots \cdots (3.48b)
\]

It can be seen from Fig. 3.4 that the experimental C-curve is
skewed with a "long tail" (approaches zero very slowly) and at this low concentration level the data are not accurate. This type of behaviour has been found experimentally by several workers (L5, D3, S3, T2, T3) and various explanations have been proposed to account for it.

Levenspiel and Smith (L5) have shown that the tail of the response to a true impulse would decay exponentially. Sater (S2) assumed that the response to an arbitrary pulse would also decay exponentially and can be represented by the relationship;

\[ C = Ke^{-qt} \]  

(3.57)

This was confirmed by plotting the data to the right of the maximum of the response curve to a "cut off point" on semi-log arithmetic coordinate paper and obtaining a straight line. This line is then extrapolated beyond the cut off point and used to calculate the contributions of that portion of the curve to the total moments. They proposed the following revised equation for the determination of mean and variance where the tail of the response curves are "long" - an extensive treatment on the derivation of these equations are given by Sater (S2);

\[ \mu = \frac{\Delta t \sum t_i C_i}{\Delta \sum C_i} + \frac{Ke^{-qt_{co}}(qt_{co}) + 1}{(Ke^{-qt_{co}})/q^2} \]  

(3.58)

and

\[ \sigma^2 = \frac{\Delta t \sum (t_i - \mu)^2 C_i}{\Delta \sum C_i} + \frac{Ke^{-qt_{co}}(q^2t_{co}^2 + 2qt_{co} + 1)}{(Ke^{-qt_{co}})/q^2} \]  

(3.59)

where \( t_{co} \) is the cut off point on the total end of the curve. Thus, the first term of each of the equations (3.58) and (3.59) represents the contribution of the first and second moments for \( t < t_{co} \) and the second term the contribution for \( t > t_{co} \).
The Peclet Number is a function of dimensionless variance and is dependent on the boundary conditions imposed on the system.

It is necessary at this point to define the dimensionless variance \( \sigma_\theta^2 \),

\[
\sigma_\theta^2 = \frac{\sigma^2}{\mu^2}
\]  .......... (3.60)

The two widely relationships for determining the Peclet Number from RTD studies are:

For a closed system - defined as one in which fluid enters and leaves solely by plug flow - Van der Laan (VI) proposed the following relationship:

\[
\sigma_\theta^2 = \frac{2}{\text{Pe}^2} \left( \text{Pe} - 1 + e^{\text{Pe}} \right)
\]  .......... (3.55)

For an open system Levenspiel and Smith (LS) proposed the following relationship:

\[
\sigma_\theta^2 = \frac{2}{\text{Pe}} + \frac{8}{\text{Pe}^2}
\]  .......... (3.56)

Solving for \( \text{Pe} \)

\[
\frac{1}{\text{Pe}} = \frac{1}{8} \left( \sqrt{8\sigma_\theta^2 + 1} - 1 \right)
\]  .......... (3.61)

The axial dispersion coefficient, \( E \), is calculated from the Peclet Number as follows;

\[
\text{Pe} = \frac{UL}{E}
\]  .......... (3.62)

i.e. \( E = \frac{UL}{\text{Pe}} \)  .......... (3.63)
3.3 Effect of Axial Mixing on Contactor Performance

3.3.1 Concentration Profiles

The effect of axial mixing is to lower the separation efficiency of the contactor as compared with values obtained from an assumption of piston flow (Section 3.1.2). Fig. 3.6 is a typical representation of the effect of axial mixing on the concentration profile along the contactor and hence on mass transfer efficiency.

**Fig. 3.6 Effect of Axial Mixing on concentration Profile**

- **ABCDE**: Actual concentration Profile - Aqueous Phase
- **FGHKL**: Actual Concentration Profile - Organic Phase
- **ABDE**: Hypothetical concentration Profile assuming Piston flow - Aqueous Phase
- **FGKL**: Hypothetical concentration Profile assuming piston flow - organic phase
From the above diagram (Fig. 3.6) it can be seen that axial mixing lowers the mean driving force for mass transfer. This type of diagram has been used by many authors (L2, M8, R4) to demonstrate the discontinuity of the concentration profile at the ends of the extractor caused by axial mixing.

3.3.2 Diffusion Model for Mass Transfer in Countercurrent Continuous Flow Operations

Miyauchi (M6) and Miyauchi and Vermulen (M8) were among the first workers to use the diffusion model for describing mass transfer in countercurrent continuous contractors with axial mixing. They extended and modified the Damköhler (D1) continuity equation for continuous flow system and derived an equation for two phase countercurrent flow operations. The theory of this work is based on the work of these two authors. Other workers to use the diffusion model were Sliecher (S10), Rod (R4) and Bibaud and Treybal (B2).

Figure 3.7 shows continuous differential contactors wherein the concentration of dissolved solute changes continuously with axial distance. It is necessary to point out at this stage that the axial distance is always measured from the physical bottom of the contacts and not from the inlet feed position which is the case for most of the other workers. Assuming that the axial diffusion coefficient $E_i$ adequately describes the axial mixing within the contactor, and the interphasial mass transfer can be represented by equations (3.9 and 3.10) (two film theory). Then from Fig. 3.7b a material balance of solute in the continuous phase over a differential height $dh$ is;

\[-E_c \left( \frac{dy}{dh} + \frac{d^2 y}{dh^2} \right) dh + U_c y + K_c a (y^* - y) dh =
\]

\[-E_c \frac{dy}{dh} + U_c (y + dy) \]

which, when simplified gives;
Fig. 3.7. Schematic Diagram of Diffusion Model for O.B.C
(a) MIBK - HAC - H₂O system x = x_F at h = 0
(b) CCL₄ - HAC - H₂O system x = x_R at h = 0
Similarly, for solute transfer the dispersed phase is:

\[-E_d \left( \frac{dx}{dh} + \frac{d^2x}{dh^2} + \frac{dh}{dh} \right) + U_d (x + dx) = U_d x - E_d \frac{dx}{dh} + K_c a (y^* - y)\]

giving

\[E_d \frac{d^2x}{dh^2} - U_d \frac{dx}{dh} - K_c a (y^* - y) = 0 \quad \cdots \cdots \quad (3.66)\]

Similar material balances for Fig. 3.7a where \(x = x_F\) at \(z = 0\) gives the following equations:

\[E_c \frac{d^2y}{dh^2} + U_c \frac{dy}{dh} + K_c a (y^* - y) = 0 \quad \cdots \cdots \quad (3.67)\]

and

\[E_d \frac{d^2x}{dh^2} - U_d \frac{dx}{dh} - K_c a (y^* - y) = 0 \quad \cdots \cdots \quad (3.68)\]

Since axial mixing studies were done on the continuous phase only the theory that follows is for this phase. Defining the following dimensionless parameters

\[Y = \frac{y}{x_F}\]

\[X_R = \frac{x_R}{x_F}\]

\[Z = \frac{h}{H}\]

\[N_{oc} = \frac{K_{oc} a H}{U_c}\]

\[Pe_c = \frac{U_c H}{E_c}\]

Then equations (3.65) and (3.67) can be written in dimensionless form as follows;

\[\frac{d^2Y}{dz^2} + Pe_c \frac{dY}{dz} - N_{oc} Pe_c (Y^* - Y) = 0 \quad \cdots \cdots \quad (3.69)\]

\[\frac{d^2Y}{dz^2} + Pe \frac{dY}{dz} + N_{oc} Pe (Y^* - Y) = 0 \quad \cdots \cdots \quad (3.70)\]
Boundary Conditions

The boundary conditions applicable to these cases were first proposed by Danckwerts (D3). Wehner and Wilhem (W3) later presented theoretical justification for the use of Danckwerts' boundary conditions. These boundary conditions can be obtained by doing material balances at the inlet and outlet places of the contactor. Assuming that the axial mixing outside the contactor (i.e. $Z < 0$ and $Z > 1$) is negligible compared to the axial mixing within the contactor (i.e. $0 < Z < 1$), then for Fig. 3.7b.

At $h = 0$

$$U_c y_s = U_c y_{h+0} - E_c \frac{dy}{dh} \quad \ldots \ldots \ (3.71a)$$

where $y_s$ is the solute concentration in the solvent stream as it approaches the plane $h = 0$ and $y_{h+0}$ is the solute concentration just within the plane $h = 0$. The difference between these two is due to presence of axial mixing within the contactor. The negative sign associated with the axial mixing term is due to diffusion taking place from a point of high concentration to a point of low concentration.

Writing equation (3.7a) in dimensionless form and rearranging;

$$\frac{dy}{dz} = Pe [Y_s - Y_{z+0}] \quad \ldots \ldots \ (3.71b)$$

At $h = H (Z = 1)$

Danckwerts (D3) established this boundary condition on intuitive grounds, this type of analysis was also used by Miyauchi and Vermulen (M8). The boundary condition is;

$$\frac{dy}{dh} = 0 \quad \ldots \ldots \ (3.72a)$$

In dimensionless form;

$$\frac{dy}{dz} = 0 \quad \ldots \ldots \ (3.72b)$$
Similarly, for Fig. 3.7a \((x = x_F\) at \(h = H\)) the boundary conditions are

At \(h = 0 \ (z = 0)\)

\[
\frac{dY}{dh} = 0 \quad \ldots \ldots \ (3.73a)
\]

or

\[
\frac{dY}{dz} = 0 \quad \ldots \ldots \ (3.73b)
\]

and at \(h = H \ (Z = 1)\)

\[
U_c \frac{Y_s}{y} = U_c \frac{Y_h}{y} - E_c \frac{dY}{dh} \quad \ldots \ldots \ (3.74a)
\]

or

\[
\frac{dY}{dz} = Pe \left[ Y_s - Y_{z-0} \right] \quad \ldots \ldots \ (3.74b)
\]

### 3.3.3 Interpretation of the Diffusion Model

The diffusion model as given by equation 3.65 in dimensional form is;

\[
E_c \frac{d^2Y}{dh^2} + U_c \frac{dY}{dh} - K_c a \ (y^* - y) = 0 \quad \ldots \ldots \ (3.65)
\]

Rearranging and integrating this equation between the limits \(h = 0\) and \(h = H\) and \(y = y_0\) and \(y = y_1\) (Fig. 3.6);

\[
\frac{K_c a}{U_c} \int_0^H dh = \int_0^{y_1} \frac{dY}{y_0} \left( y^* - y \right) + \frac{E_c}{U_c} \int_0^1 \frac{d^2Y}{dh^2} dh \ldots \ldots (3.75)
\]

This method of interpretation was first done by Miyauchi and Vermulun (M8) and later by Smoot and Babb (S13) who rewrote the equation in general form as follows;

\[
NTU_T = NTU_M + \text{Correction Factor} \quad \ldots \ldots \ (3.76)
\]

The correction factor term in equation (3.76) can be determined by graphical integration; however, this would be somewhat
difficult to achieve since the determination of the second derivative would require extremely accurate concentration profiles. It is important to point that equation (3.76) is applicable only when concentration profiles of both phases can be measured since \( y^* \) is determined from the measured \( x \) by the equilibrium relationship.

Thomas (T6) modified equation (3.76) for analysis in which \( y \) is obtained by experiment and \( x \) calculated from a material balance and proposed the following equation:

\[
NTU_T = NTU_{\text{App.M}} + (\text{Correction factor})_1 + (\text{Correction Factor})_2 \\
= NTU_M + (\text{Correction factor})_2 \\
\text{ (3.77)}
\]

\( NTU_{\text{App.M}} \) is defined in Section 3.2.6.

Miyauchi and Vermulen plotted (Fig. 3.8) each term of the diffusion model in dimensionless form for the feed phase \( X \) from which shows how the different terms seem to zero at each phase. They stated that the positive terms represent the rate of supply of solute at each phase and the negative term the rate of its removal by mass transfer.

![Fig.3.8 Depletion and Accumulation for Solute Transferring in Feed Phase (Reproduced from Miyauchi and Vermulen (M8) )](image)
It is not possible to carry out a similar analysis for the present work since this would require Peclet Number for the dispersed phase and direct measurement of the concentration profile for this phase. However, a similar analysis can be done by plotting the terms of the diffusion model for the continuous phase (Eqn.3.65) on a similar diagram. This will be dealt with further in Chapter 8.3.4.

3.3.4 Solutions to the Diffusion Model.

The diffusion model has been solved by Miyauchi and Vermulun (M8), Sleicher (S10), Smoot and Babb (S13) for systems with linear equilibrium relationship and by Rod (R4) for systems with curvilinear equilibrium relationship.

For the present studies two types of solution (Eqn.3.71, Fig. 3.7b) are carried out, an analytical solution for linear equilibrium relationship and a numerical solution for a curvilinear equilibrium relationship. The analytical solution is given in Appendix A.3.1 and the final exact solution is as follows:

\[ Y = \frac{pe^m e^{q_1 Z}}{q_1} + \frac{e^{q_2 Z}}{q_2} + \frac{q e^{q_1 Z}}{q_1} \frac{q_2 Y - pe^m}{q_1 q_2} + \frac{q_1 (mX_R + C)}{(q_1 - q_2)(m-1)} \]

\[ \frac{-mX_R + C}{m-1} \]

where

\[ q_1 = \frac{(-pe + r)}{2} \]
\[ q_2 = \frac{(-pe - r)}{2} \]
\[ r = pe^2 + 4Nocpe (m - 1) \]
\[ Y_M = Y_0 - Y^0 \] (as shown in Fig. 3.6)

This solution assumes a linear equilibrium relationship. For curvilinear equilibrium relationships such as the CCL4-HAC-H2O system a more accurate solution was obtained by dividing the equilibrium relationship into a number of sections and deriving linear relationships for each section. If the con-
centration profile is known from experiment, then the values of \( m \) and \( C \) can be varied with \( Z \) accordingly when solving for \( Y \).

The numerical solution is done by the Runge-Kutta method with the aid of a computer. The theory of this method is well described in Jenson and Jeffreys (J3) and the development and computer program for the solution is given in Appendix A.3.1.

It must be stated that solutions of the one-dimensional diffusion model by previous workers were oversimplified since constant Peclet Number and \( N_{oc} \) values were assumed for a given set of operating conditions. For the present studies, the variation of \( N_{oc} \) or \( NTU_c \) with \( Z \) (which was determined experimentally) was included in the solution of the model. However, a constant value of Peclet Number was used. The limitations associated with this solution will be dealt with in Chapter 8.3.3.

3.4 Sauter Mean Drop Diameter, Interfacial Area and Hold-up

In all types of contacting devices wherein one phase is dispersed in another, there exists a distribution of particle size. When there is mass transfer taking place, in order to establish the mass transfer efficiency of the contactor it is necessary to ascertain the magnitude of the interfacial area available for solute transfer between the two phases under given operating conditions.

Various methods have been used to determine interfacial area in contacting devices where there is a distribution of drop sizes. One of these methods that is widely used is the determination of a mean volume-surface diameter known as the Sauter mean drop diameter, \( d_{32} \) - the theory of which is well described in Orr (04). The Sauter mean drop diameter relates the surface area of the drop to the total volume as follows;

\[
d_{32} = \frac{\Sigma N_i d_i^3}{\Sigma N_i d_i^2} \quad \text{........ (3.79)}
\]
where $N_i$ is the number of droplets of diameter $d_i$. The interfacial area, $a$, is related to the Sauter mean drop diameter and the volume fraction of dispersed phase, $\phi_F$, in the contactor at dynamic equilibrium as follows:

$$a = \frac{6 \phi_F}{d_{32}} \quad \ldots \ldots \ (3.80)$$

The interfacial area as calculated from equation (3.80) is the specific interfacial area and has the units $m^2/m^3$ or $1/m$.

Hold-up

![Fig. 3.9 Schematic Diagram for Determining Hold-up](image)
Three of the most widely used methods for determining the volume fraction of dispersed phase hold-up in the contactor are:

(a) The manometric method
(b) The displacement method
(c) The drainage method.

For the present studies the manometric method was used to determine hold-up $\phi_F$, the theory of which is given in Appendix 2. The relationship for calculating hold-up is as follows (Fig. 3.9).

$$\phi_F = \frac{h_m (\rho_m - \rho_c)}{\Delta h (\rho_d - \rho_c)} \quad \ldots \ldots \ (3.81)$$

where
- $h_m$ = manometer reading, cm.
- $\Delta h$ = difference between the points on the contactor to which the arms of the manometer are connected, cm.
- $\rho_m$ = density of heavy liquid in manometer, g/cc.
- $\rho_c$ = density of the continuous phase which is also used as the light liquid in the manometer, g/cc.
- $\rho_d$ = density of the dispersed phase, g/cc.

If $\rho_m = \rho_d$, then

$$\phi_F = \frac{h_m}{\Delta h} \quad \ldots \ldots \ (3.82)$$

### 3.5 Power Requirements in Liquid Mixing

#### 3.5.1 Speed Parameter for O.B.C.

The correlation used by most workers to study power requirements in an agitated contactor with two phase systems is given by:

$$N_p = K N_{Re}^a N_{Fr}^b N_{We}^c \quad \ldots \ldots \ (3.83)$$

The dimensionless groups, $N_{Re}$, $N_{Fr}$ and $N_{We}$ are for rotating movement and the speed parameter is measured in revolutions.
The mode of agitation in the Oscillating Baffle Contactor is completely different from the rotational mode since the agitators are oscillating at fixed amplitudes and the speed parameter is measured in oscillation per unit time (osc/min.). The use of this type of speed parameter in the power correlation would make comparisons of the two modes of operation (i.e. rotating and oscillating) somewhat difficult. In order to facilitate this comparison, a new speed parameter as derived below will be used for the O.B.C. for the oscillating system:

Total angle swept in one complete oscillation = $2\theta$ radians
Fraction of circle swept in one complete oscillation = $\frac{2\theta}{2\pi}$
Distance travelled by tip in one oscillation = $\frac{2\theta}{2\pi} \times \pi D$ cm
For $N$ oscillations/sec, mean tip speed = $\frac{2\theta}{2\pi} \times D \times N$ cm/sec
Hence, mean tip speed = $\theta DN$ cm/sec ............. (3.84)

where $\theta$ = amplitude of oscillation, radians
$D$ = diameter of agitator, cm.
$N$ = oscillating rate, osc/sec.

For the rotating system, where $\theta = 1$.
Mean tip speed = $DN$ cm/sec ............. (3.85)

3.5.2 Power Correlations

3.5.2.1 Correlations for Total Power Consumption

The quantity of mechanical energy required to generate turbulence throughout a liquid mass is dependent on the vessel geometry, agitator geometry and the physical properties of the liquids.

This energy has been correlated in terms of the independent variables by dimensional analysis. From the extensively published literature, it has been stated that for two phase
systems the power consumed is dependent on the agitator speed, \( N \), agitator diameter, \( D_A \), liquid density, \( \rho_1 \), liquid viscosity, \( \mu \), interfacial tension, \( \sigma \), and gravitational acceleration, \( g \).

The generalised equation for power consumption in a mixer wherein there are two immissible phases is;

\[
\frac{P}{\rho N^3 d^5} = K \left( \frac{N d^2 \rho}{\mu} \right)^a \left( \frac{N^2 d}{g} \right)^b \left( \frac{N^2 d^3 \rho}{\sigma} \right)^c \quad \ldots \quad (3.86a)
\]

This equation can be written in the form

\[
N_p = K (N_{Re})^a (N_{Fr})^b (N_{We})^c \quad \ldots \quad (3.86b)
\]

The development of this equation by dimensional analysis is given in Chapter 6.4.

For a fully baffled vessel—this is achieved by the O.B.C.—vortexing due to gravitational forces is eliminated and the exponent of the Froude Number is zero. The flow dynamics is then controlled by the viscous and interfacial tension forces as follows;

\[
N_p = K (N_{Re})^a (N_{We})^b \quad \ldots \quad (3.87a)
\]

or

\[
\frac{P}{\rho N^3 d^5} = K_1 \left( \frac{N d^2 \rho}{\mu} \right)^a \left( \frac{N^2 d^3 \rho}{\sigma} \right)^b \quad \ldots \quad (3.87b)
\]

Miller and Mann (M1) recommended the use of a weighted mean viscosity and an arithmetic mean density for use in the power correlation, where;

\[
\mu_m = \mu_\phi \cdot \mu_c^{1-\phi} \quad \ldots \quad (3.88a)
\]

and

\[
\rho_m = \phi \rho_d + (1 - \phi) \rho_c \quad \ldots \quad (3.88b)
\]

where \( \mu_m \) and \( \rho_m \) are the mean viscosities and density respectively, \( \phi \) is the volume fraction of dispersed phase, \( \mu_d, \mu_c \) and \( \rho_d, \rho_c \) are viscosities and densities of the dispersed and continuous phase respectively.
It must be pointed out that the power used in the correlation is the total mixing power and excludes the power consumed in overcoming fractional and other forces. Thus for the O.B.C.

\[ P_T = P_{Dry} + P_{MT} \]  \quad \text{...... (3.89)}

and \[ P_M = P_{M1} + P_{M2} \]

where \( P_{Dry} \) is the power used in overcoming frictional forces in the bearing and drive unit. This quantity is determined by taking power measurements with the contactor empty.

\( P_{M1} \) is the power required to agitate the single continuous phase. This is found by subtracting \( P_{Dry} \) from the power consumed when the vessel is filled with continuous phase only.

\( P_{M2} \) is the extra power consumed when the dispersed phase is present in the vessel. It is found by subtracting \( P_{Dry} \) and \( P_{M1} \) from the power consumed when both phases are present in the vessel. The significance of this quantity will be discussed in Chapter 8.

3.5.2.2 Correlations for Power Consumption per Unit Volume

The power consumption per unit volume is particularly useful when doing scale-up studies on mixing equipment. In the case of the multiple mixer system of the O.B.C. which can be regarded as the first of its type to be studied, it is necessary to establish some basis for comparison with the single mixer system. Previous work on the O.B.C. has been done with single oscillating baffles, and in order to make comparisons between the single baffle and multiple baffles system, the following power terms are used;

(a) The power per unit swept volume, \( P_{VS} \). This is the total mixing power \( P_M \) divided by the total volume swept by the baffles, thus for the multiple baffle system,
where $P_{vs} =$ the power per unit swept volume

$P_M =$ total mixing power

$V_B =$ the volume of a single agitator $= \pi R_A^2 L_B$

$R_A =$ the radius of the agitator

$L_B =$ the length of the agitator

$N_B =$ number of agitators.

(b) The power per unit active volume, $P_{va}$. This is the total mixing power divided by the total active volume $V_A$ of the contactor and is given by

$$P_{va} = \frac{P_M}{V_A} \quad \text{.......... (3.91)}$$

Obviously a better value for the power term would lie between these two values since some fraction of the unswept volume would be mixed by the agitators, and it would be erroneous to put an actual value to this fraction.

Olney and Carlson (01) multiplied both sides of equation (3.8 b) by the Reynolds Number giving;

$$\frac{P}{N^2 d^3 \mu} = K_1 \left( \frac{Nd^3 \rho}{\mu} \right)^{x_f} \left( \frac{N^2 d^3 \rho}{\sigma} \right)^b \quad \text{.......... (3.92)}$$

where $x_f = a + 1 =$ Reynold index for fluid friction or momentum transfer

By replacing $\frac{P}{d^3}$ in (3.92) by the power per unit volume $P_v$, then

$$\frac{P_v}{N^2 \mu} = K_1 \left( \frac{Nd^3 \rho}{\mu} \right)^{x_f} \left( \frac{N^2 d^3 \rho}{\sigma} \right)^b \quad \text{.......... (3.93a)}$$

or

$$N_{pv} = K_1 (N_{Re})^{x_f} (N_{We})^b \quad \text{.......... (3.93b)}$$
Writing similar equations for the two power terms $P_{vs}$ and $P_{va}$, then

\[
\frac{P_{vs}}{N^2 \mu} = K_2 \left( N_{Re} \right)^x f \left( N_{We} \right)^b \\
\text{......... (3.94)}
\]

and

\[
\frac{P_{va}}{N^2 \mu} = K_3 \left( N_{Re} \right)^x f \left( N_{We} \right)^b \\
\text{......... (3.95)}
\]
CHAPTER 4

DESCRIPTION OF EXPERIMENTAL RESULTS
4.1 Experimental Contactor.

Thomas and Chiu (1969), Thomas and Weng (1970) and Thomas and Clare (1978), carried out research studies on an oscillating baffle contactor. Their studies were concentrated on a single oscillating baffle system, with 7.5 and 15 cm I.D contactors. Studies on the 7.5 cm I.D contactor were done with baffles made from perforated plate (¼ inch holes) and studies on the 15 cm I.D contactor were done with baffles made from 3/64" wire mesh.

The present studies were concentrated on a 30 cm I.D contactor with three single four-bladed baffles positioned on a triangular pitch inside the contactor. The baffles were oscillating in phase. A single baffle was made up from 5 x 20 cm long four-bladed sections, the blades were made from 3/64" stainless wire mesh. A single baffle is shown in Fig.4.1(b). The wire mesh provided a much greater percentage of free area per baffle blade than the perforated plate blades thus reducing the stirring effect caused by the solid part of the blade. In addition to this, the power consumed in oscillating the mesh baffles is less than that for the perforated plate baffles.

Studies were also carried out on the 7.5 cm I.D contactor with a single oscillating baffle.

A detailed description of the small contactor (7.5 cm I.D) was given by Thomas (T4). Details and dimensions of the present contactors are given in Table 4.1.

Since the basic construction of both contactors used for this study is the same, the only difference being the number of baffles and dimensions of the contactors, the description given below will be that of the large contactor (Contactor I).

The effective extraction zone of the contactor was a vertical borosilicate glass section, 120 cm long consisting of seven 2.5 cm bore side arms for sampling purposes. The side arms
Table 4.1 Column and Baffle Dimensions.

<table>
<thead>
<tr>
<th></th>
<th>Contactor I</th>
<th>Contactor II</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Column Body</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Outside diameter</td>
<td>31.5 cm</td>
<td>16 cm</td>
</tr>
<tr>
<td>Inside diameter</td>
<td>30 cm</td>
<td>15 cm</td>
</tr>
<tr>
<td>Effective Extraction Length</td>
<td>120 cm</td>
<td>120 cm</td>
</tr>
<tr>
<td>Length of Top Header</td>
<td>45 cm</td>
<td>15 cm</td>
</tr>
<tr>
<td>Length of Bottom Header</td>
<td>30 cm</td>
<td>20 cm</td>
</tr>
<tr>
<td>Thickness of Spacer Plates</td>
<td>1.27 cm</td>
<td>1.27 cm</td>
</tr>
<tr>
<td>Diameter of Spacer Plates</td>
<td>45 cm</td>
<td>30 cm</td>
</tr>
<tr>
<td><strong>Baffle Section</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Material of Construction</td>
<td>S.S*</td>
<td>S.S*</td>
</tr>
<tr>
<td>Number of Blades</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Width of Blades</td>
<td>5.56 cm</td>
<td>5.56 cm</td>
</tr>
<tr>
<td>Length of Blades</td>
<td>20 cm</td>
<td>20 cm</td>
</tr>
<tr>
<td>No of holes per Blades</td>
<td>2560</td>
<td>2560</td>
</tr>
<tr>
<td>Percentage free area per Blade</td>
<td>58</td>
<td>58</td>
</tr>
<tr>
<td>Mesh Aperture</td>
<td>1.211 mm</td>
<td>1.211 mm</td>
</tr>
<tr>
<td><strong>Complete Baffle</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>No of sections per complete baffle</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>No of complete baffles</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>Clearance between baffle and column wall</td>
<td>0.8 cm</td>
<td>1.25 cm</td>
</tr>
<tr>
<td>Percent of column X' section swept by baffles</td>
<td>56.76</td>
<td>82.6</td>
</tr>
</tbody>
</table>

S.S* = Stainless Steel
(a) Multi Baffle Contactor (Contactor I, large)
(b) Complete Baffle
(c) Single Baffle Contactor (Contactor II, small)

Fig. 4.1 The Oscillating Baffle Contactors
were equally spaced along the column length and positioned alternately on opposite sides along the column length. Each sampling unit consisted of a short length of \( \frac{1}{4} \) inch bore stainless steel tube fitted through a stainless steel disc which was made leakproof by welding the tube onto the disc. One end of the tube was positioned inside the contactor and a \( \frac{1}{4} \) inch bore glass sampling tap with P.T.F.E stopcork was connected to the other end by a short length of silicone tubing. Each sampling unit was bolted onto the side arms by flanges with a P.T.F.E o'ring between the glass and metal making the connection leakproof. An overall view of the contactors are given in Fig. 4.1(a) & (c).

In order to ensure that only the continuous phase was sampled, very fine stainless steel wire gauzes were placed on the ends of the sampling tube positioned inside the contactors. This prevented the small disperse phase droplets from entering the sampling unit. A sketch of the sampling unit is given in Fig. 4.3.b

At each end of the extractive section there was a stainless steel spacer plate (shown in Fig.4.2) \( \frac{1}{4} \) inch thick and 18 inches diameter separating the extractor from the header sections. Seventy two holes (9/16 inch) were drilled on each spacer plate over the diameter which was covered by the cross section of the contactor. The bottom plate served as a support for the baffle shafts, and both plates effectively distributed the phases evenly over the contactor cross-section. These plates were also used as sampling positions at the ends of the contactor, and manometer take off points for measuring the hold-up of the dispersed phase. For these points 5/16 inch holes were drilled horizontally through the plate (i.e from the outer edge of the plate through to one of the drilled holes just inside the column). Each point was fitted with \( \frac{1}{4} \) inch stainless steel tubes and wire mesh gauze to prevent the small droplets entering the take off points. Glass sampling taps were connected to the two sampling points as described above. The manometer was connected to the other two points by
Fig. 4.2 Spacer Plates (Contactor I)
Fig. 4.3a Sketch of Contactor Showing Sampling Points

Upper Header
Extraction Zone
Lower Header

30 cm
120 cm
Fig. 4.3b Section of Extraction Zone showing Sampling Unit
silicone tubing.

For the small contactor (Contactor II) one baffle (i.e. 5 x 20 cm long sections) was mounted on a shaft which was positioned centrally inside the contactor. Dimensions of the baffle are given in Table 4.1.

4.2 Pilot Plant.

4.2.1 General Description.

The main body of the pilot plant consisted of the two contactors, three feed supply vessels, five feed storage vessels, the baffle drive systems and two centrifugal pumps. The plant was situated in a flame proof laboratory and all electrical devices and connections were installed to comply with the flameproof standards of the Health and Safety at work acts.

Due to the corrosive nature of the chemical used, the materials used to construct the plant were restricted to glass and stainless steel. The organic solvents used also attacked most plastic and rubbers. Borosilicate glassware (Q.V.F) supplied by Corning Ltd., and 316 stainless steel were used. Seals and gaskets were made from P.T.F.E. Glass was used for the feed storage and supply vessels (6 x 100 litres and 2 x 200 litres) and for the body of the contactors. Stainless steel was used for the contactors internals (i.e. sampling tubes, baffles shafts and spacer plates), the stirrer for mixing the solute, pipelines between rotameters and phase inlet to contactor, and phase outlet from contactors to storage vessels. (½ inch and 1 inch piping in conjunction with unions, bends, tees etc.). P.T.F.E was used as gaskets for the glass pipelines, the rotor of the two Q.V.F centrifugal pumps and bushes for stirrer drive shaft and baffle drive shafts.

The general layout of the pilot plant is shown in Fig.4.4 and the plant flowsheet is shown in Fig.4.5.
Fig. 4.4 Photograph of Pilot Plant and Auxiliary Equipment
Key to Fig. 4.5

C I - Contactor I; Large contactor with oscillating multiple baffles

C II - Contactor II; small contactor with single oscillating baffle

P1 - Aqueous Phase centrifugal pump
P2 - Organic Phase centrifugal pump

V1, V2 - Organic Phase storage vessels (200 litres each)
V3 - Organic Phase storage vessel (100 litre)
V4 - Organic Phase feed supply vessel (100 litre)
V5, V6 - Aqueous Phase storage vessels (100 litres each)
V7, V8 - Aqueous Phase feed supply vessels

- Valves

- Reducers
4.2.2 Baffle Drive Systems.

The baffle drive system consisted of a motor, rotating through a Kopp Variator which rotated through a reduction gear box unit. All three devices were mounted horizontally in series. Table 4.2 shows the technical details of the three devices. The reduction gear box also changed the direction of rotation from horizontal to vertical.

The constant speed drive shaft from the motor rotated through the Kopp Variator which was used to vary the speed of rotation. The variator was followed by a 10:1 reduction gear box unit. From this unit the now vertical drive shaft was attached via a strain gauge transducer to a bell crank assembly which changed the rotational motion of the shaft to an oscillation motion of the baffle shaft.

The drive systems for both contactors were the same up to the bell crank assembly (shown in Fig. 4.6). For the small contactor (Contactor II) the collar of the bell crank assembly was connected directly into the single baffle shaft, while for the large contactor the bell crank assembly was connected to a straight through gear box assembly which was used to oscillate the three baffles.

The assembly consisted of two plates 30 x 30 cm held in parallel by four supporting pillars, one at each corner, between which were placed four spindles, the bearing end of which were let into brass bushes. The bell crank assembly collar was connected to the bearing end of one of the spindles, (the others in the same plane having free ends), which transmitted motion from the bell crank to the other three spindles. The four spindles were linked up by a chain, three of them (including the one connected to the bell crank) were used to oscillate the baffles, while the other was used as a chain tensioner, and is free moving. The other ends of three spindles were connected to the baffle shafts by couplings. The bell crank arrangement is shown in Fig. 4.6 and the gear box assembly in Fig. 4.7.
Table 4.2 Details of Baffle Drive System.

<table>
<thead>
<tr>
<th>Description</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reduction Gear Box</td>
<td>Reynolds Ltd., Croft Gear Works, Thornbury, Bradford, U.K.</td>
</tr>
<tr>
<td>Kopp Variator</td>
<td>Allspeeds Ltd., Accrington, Lancashire</td>
</tr>
<tr>
<td>Motor Contactor I</td>
<td>Brook-Crompton Parkinson Ltd.</td>
</tr>
<tr>
<td>Manufacturer</td>
<td>Electrical Power Engineering Co. Ltd.</td>
</tr>
<tr>
<td>Specifications</td>
<td>Contactor I: 0.5 h.p. 940 RPM Contactor II: 0.25 h.p. 950 RPM</td>
</tr>
<tr>
<td>Output Speed</td>
<td>[ \text{Output Speed} = \frac{\text{Input Speed}}{10} ]</td>
</tr>
<tr>
<td>Variation of Speed</td>
<td>Range from 1/3 to 3 times input speed</td>
</tr>
</tbody>
</table>

Changes direction of rotation from horizontal to vertical.
Fig. 4.6 Sketch Showing Bell Crank Arrangement
(a) Internal View

(b) Overall View

Fig. 4.7 Gear Box Assembly
4.2.3 Manometer.

The manometer used to measure the dispersed phase hold-up was a glass U-Tube. The limbs were approximately 50 cm long and 3 cm apart with a bore of 6 mm. The limbs of the manometer were half filled with dyed carbon tetrachloride. The dye used was Sudan IV supplied by B.D.H Chemicals Ltd., and the concentration of the dye solution was approximately 1g/litre of solvent. Due to the very low concentration of the dye solution, the change in density of the carbon tetrachloride was negligible. The limbs of the manometer was filled with the continuous phase.

The principle and theory of the manometric method is given in Appendix 4.2.1.

4.3 Equipment for Dye Study.

4.3.1 Photocell Unit.

The photocell unit used for the dye study was made by the University of Surrey, Dept of Chemical Engineering Technical Department.

The unit consisted of a concave mirror reflecting a parallel beam of light from a halogen lamp through perspex windows placed on either side of the flow path. As the dye solution flowed through the unit, the variation of the light intensity was detected by a selenium cell giving a proportional output current.

This output was monitored by passing the signal through an electronic amplified unit providing zero and full scale adjustments, to a data-logger taking readings at specified intervals and giving a permanent record on paper tape.

The unit was positioned so that it was close as possible to the outlet phase being studied.
Fig. 4.8 Dye Study Equipment
The photocell unit is shown in Fig.4.8(a).

4.3.2 Data Logger.

The data logger is a Solartron system based on a LM1420 Digital Voltmeter with a command unit, an Addo punch drive unit feeding into an Addo paper punch.

The data logger as standard is restricted by data collected to sample times of 2 per second to 1 sample every 3 seconds. A small unit was constructed to give a variable sampling period of 1 to 15 seconds interval and manual sampling via the data logger external triggering facility. The unit also had a manual overload input to mark the tape with an end of run marker.

4.3.3 Tracers Used.

It was necessary to use a tracer which had the following properties.

(a) it was soluble in the aqueous phase only.
(b) the colour intensity in the outlet stream was high enough to be detected by selenium cell.

For these reasons Nigrosine crystals supplied by B.D.H. Chemicals Ltd was used. A concentrated solution was made and used as the dye for the aqueous phase.

4.4 Equipment For Power Measurement.

For the power studies it was necessary to measure small changes in the power reading between single and two phase flow as accurately as possible. To achieve this, all the power measurement devices used (i.e. strain gauge transducers and torque meters) were recalibrated if necessary and tested for accuracy by the manufacturers.
4.4.1 Torque Transducers.

The power consumption was obtained by measuring the torque transmitted with the Saunders-Roe Type 2 MK4 supplied by British Hovercraft Corp. Ltd.

The very small angular deflection of the torque shaft resulting from the torsional stress, caused a change in the electrical resistance of the strain gauges, and produced an electrical output voltage from the bridge circuit which was directly proportional to the transmitted torque.

Separate transducers of the same type were used for the two contactors, the torque range of the transducers used for Contactor I (large) was 0-6 lb.ft. The total transducer error due to non-linearity, hysteresis and repeatability was not more than ± 0.2% full load output.

4.4.2 Torque Indicator.

The output voltage from the torque transducer was fed to a TM6/R indicator unit which was also supplied by British Hovercraft Corp. Ltd. The unit had facilities for connecting ancillary apparatus.

Since the system was operating in the oscillating mode the reading on the indicator was unstable, as a result of this the output from the indicator unit was fed to a Torque Integrator Unit (described in 4.4.3 below) which provided a stable average reading on a digital LED display.

4.4.3 Torque Integrator.

The torque integrator unit was built to operate with the TM6/R indicator (described in 4.4.2) and strain gauge transducers (4.4.1). The unit was built by University's Department of Chemical Engineering Electronics Workshop.
(a) Baffle Drive System

(b) Torque Meter and Integrator

Fig. 4.9 Baffle Drive and Power Measurement Instruments
Fig. 4.10 Circuit diagram for torque integrator
Several methods for measuring the torque by signal averaging and integrating were experimented on, but it was not possible to obtain readings for small changes in torque with any tolerable degree of accuracy (± 0.5%) that were reproducible, also constant calibration of the instrument was necessary. The problem was solved by using a dual slope integration technique as described below.

The circuit (Fig. 4.10) of the integrator device worked on the principle of dual slope integration whereby the input voltage from the indicator was applied to an integrator and simultaneously a counter counting clock pulse was started. After a predetermined number of counts (in this case over a 10 second period) a reference voltage of opposite polarity is applied to the integrator. At this stage the accumulated charge on the integrating capacitor was proportional to the average value of input over the 10 second period.

The integral of the reference was an opposite going ramp having a slope $V_{\text{REF}}/R_c$. At the same time the reference voltage was applied and counter again counted from zero. When the output from the integrator reached zero the counter is stopped and the analog circuit reseted. The relative count resulting in relation to the full count is proportional to $t/T$ or $V_{\text{in}}/V_{\text{REF}}$.

Since the $V_{\text{REF}}$ was set equal to the full scale reading (F.S.R) from the torque meter, the output was calibrated giving a direct representation of torque. The output was in the form of a digital LED display giving reading to four decimal places.

The dual slope integration has several attributes. Conversion accuracy was independent of both capacitor value and clock frequency since the effect on up slope and down ramp for both was of the same ratio. The integration provided rejection of high frequency noise and averaging of charges that occurred during the sampling period. The circuit diagram for the integrator unit is shown in Fig. 4.10 and the transducer and in-
4.4.4 Shaft Speed Measurement.

A digital tachometer was used to measure the shaft speed of the baffle. The tachometer was fitted on the same console as the integrator. The circuit was based on a 4046 CMOS phase locked loop integrator circuit.

A circular metal disc with a specific number of holes drilled around the circumference was mounted on the shaft and on an optical source and sensor used to detect the rotational frequency. The pulses from the opto detector was fed to the phase lock loop circuit which had a scaling factor for the number of holes in the disc and the resulting output counted over a known period giving an accurate indication of speed of rotation.

4.5 Refractometer.

A new Abbe Refractometer, supplied by Bellingham and Stanley Ltd. was used to determine the solute content of the continuous and dispersed phases samples. Its light source was a sodium lamp. The cell surrounding the two prisms, between which the samples were placed, was filled with water and maintained at a constant temperature of 26.5°C by circulating water from a thermostatically controlled water bath containing an immersion heater. The range of the refractometer scale was 1.30 to 1.70 which can be read accurately to three decimal places, and by estimation to four decimal places with an accuracy of ± 0.0001.
4.6 Particle Size Analyser.

The counting and sizing of droplets were done by a Particle Size Analyser No.2 supplied by Sondes Place Research Institute. The analyser consisted of a ruler blade connected to a counter unit. The analyser is shown in Fig. 4.11.

The ruler blade was placed on the photograph so that the edges of the tapered slot touched the edges of the droplet to be sized. The droplet was then marked and the nearest contact point touched with the pen. This registered a count on the appropriate size group counter and on the total counter.

The ruler blade used sized droplet from 1.5 mm to 16.5 mm diameter.

4.7 Tensiometer For Interfacial.

Tension Measurement.

A tensiometer was used to measure the interfacial tension of the systems containing different amounts of solute. Most of the interfacial tension data were already established by previous workers, however, some selected measurements were carried out and compared with the previous data. The two sets of data were in close agreement (+ 1%).

An 'O.S' - type torsion balance supplied by White Electrical Instruments Co.Ltd. was used in conjunction with a platinum ring of 4 cm circumference. It was calibrated from 0-120 mN/m (i.e 0-120 dyne/cm) with 240 equal divisions. The maximum overall error of the torsion balance was ± 0.2%. This method is known as the "ring" method.

4.8 Stirrer for Mixing Solute and Solvent.

It was important for the solute and solvent (i.e feed to the contactor) to be thoroughly mixed. This mixing was done by a three-bladed, stainless steel, marine type propeller attached
to the end of a vertical stainless steel shaft which was ro-
tated by a 0.25 h.p, 940 RPM motor supplied by Brook, Crompton
and Parkinson Motor Ltd. The stirrer shaft, 3/8 inch diameter,
was surrounded by a fixed ½ inch i.d stainless steel sleeve
with a brass bush at the top of the sleeve and a P.T.F.E. bush
at the bottom (i.e the side exposed to the vapour of the chemi-
cals).

The mixing assembly was positioned at the centre of the organic
phase storage tank.(VI Fig.4.5)

4.9 Rotameters.

The rotameters used were the Metric Series Type 907 supplied by
G.E.C Marconi Process Control Ltd. They consisted of flat
buttress ends borosilicate glass tubes with stainless steel
float stops, mounted in steel frames, with interchangable flow
scale plates.

Separate calibrated flow scales, supplied by the
manufacturers for the different types of liquids were used
when necessary.

The accuracy was ± 3% of the full scale reading and the pres-
sure loss due to the float weight was 15.5 cm w.g.

4.10 Chemicals Used.

All the organic chemicals used were supplied by B.D.H Ltd.
Due to the large quantities of chemicals used (i.e 350 litres
for organic phase) technical grade chemicals were used instead
of Analar grades. Glacial acetic acid was used as solute for
all mass transfer experiments.

The impurities in the chemicals were 0.05% and the effect of
the impurities on the physical properties of the chemical
were negligible.
For all the experimental studies carried out the organic-aqueous systems were used. The aqueous phase was de-ionized water. The water from the mains was de-ionized by a Permutit Mk 12 F-type portable de-ionizer. The conductivity of the water was maintained below 10 microhms/cm which was somewhat purer than distilled water.
CHAPTER 5

EXPERIMENTAL TECHNIQUE
5.1 General.

All the experimental work was carried out in a newly built flame proof laboratory, under atmospheric pressure. The surrounding air in the laboratory was constantly changed by extraction fans open to the outer atmosphere, thus, the room temperature was fixed by the prevailing outside temperature. Due to this it would have been practically difficult and quite costly to maintain a constant room temperature - for instance by lagging flow lines and storage vessels - so all experimental work was carried out only when the room temperature was between 13 - 18 °C. This temperature range was considered to be adequate enough so as not to have any significant effect on the accuracy of the results.

Experimental work were carried out on the two contactors details of which are given in Table 4.1.

In order to minimise experimental error the performance studies consisting of droplet studies, mass transfer, dye study, power and hold-up were carried out separately.

A large quantity of the organic liquids used were also used by a previous worker, therefore at the start of the experimental work the organics already had a small amount of solute present. As a result of this it was not possible to carry out hydrodynamic studies on the pure two component systems. However, it was possible to obtain a very low solute concentration - 0.5 to 1.0 g/l - in the organics by continuous extraction. This concentration was considered to be low enough to give an idea of the hydrodynamic properties of the solute free system.

All the storage vessels and flow lines were washed thoroughly with chromic acid, de-ionized water and acetone before experimental work was carried out. The contactor was drained and rinsed with de-ionized water at regular intervals every two days. The organic phase inlet and outlet lines were rinsed with organic phase of outlet composition after each experimental run.
5.2 Calibration of Instruments.

5.2.1 Kopp Speed Variator.

Separate variators of the same type were used to vary the shaft speed of the two contactors. Each variator was fitted with a handwheel incorporating a digital counter calibrated to one decimal place. The variator speed was changed by manually rotating the handwheel in either direction.

The variators were calibrated by measuring shaft speed equal to oscillation rate for a number of different counter settings.

Calibration graphs of variator setting against oscillation rate were plotted. It was found that for a given counter setting there was no measurable difference in oscillation rate when the column was full or empty.

5.2.2 Refractometer.

The water bath system and sodium lamp described in section 4.5 were switched on and allowed to warm up for at least half an hour before the instrument was used for Refractive Index measurements. This period was sufficient for the refractometer cell to reach a constant temperature which was monitored by a thermometer positioned in the circulating water. For the present work all R.I. measurements were done at 26.5°C.

Five standard solutions for each of the two component solvent-solute mixtures being studied were prepared with solute concentration ranging from 0 to 80 g/l. The Refractive Indecies of the standard solutions were measured on the refractometer and calibration curves of R.I is solute concentration for each of the two component mixtures. Fresh calibration curves for the system being studied over a period were prepared every two weeks.
A typical calibration procedure together with curves are given in Appendix A.1.4

5.3 Droplet Studies.

5.3.1 Photography.

The droplet size distribution and mean droplet sizes were obtained by photography. As the mean droplet sizes varied along the column length for a given flow condition, there were two methods by which the studies could have been carried out. The first was by photographing various zones along the contactor length which would have meant continuous refocusing and rearranging of camera and lighting system for each zone, the second was by photographing at one zone - which can be assumed to be representative of the whole contactor - along the contactor length. Since the first method was highly susceptible to experimental error, the latter was chosen for carrying out the studies. However, the zone to be photographed was determined by using the first method as described below.

Photographs were taken at four positions along the column length for three oscillation rates, at $Q_c = Q_d = 400$ l/hr, the sectional and hence, overall Sauter mean drop diameter ($d_{32}$) were calculated for each speed condition.

Curves of sectional $d_{32}$ against position along column length were plotted and the positions corresponding to the overall $d_{32}$ were determined.

The same procedure was used for droplet studies on the two systems and the two contactors. The mean positions used for the photographic studies are given in Appendix A.1.5.

Throughout the photographic studies a "Nikon F2" camera was used with a 55 mm "Micro Nikor" lens, 50 mm extension tube and a N.D 2.6 neutral density filter. The lens was stopped down
to F 5.6 giving a small depth of field - approximately 5 mm - at unit magnification. Lighting was by two "Metz Mecablitz" electronic flash heads. The camera, flashes and background layout is shown in Fig.5.1.

![Fig.5.1 Layout of Photographic System](Plan View)

The films used were 35 mm Ilford FP4, 125 ASA, and they were developed for 7 mins at 20°C in Kodak D76 developer, this was a 10% increase on the normal time and it enhanced the contrast slightly. A5 prints enlarged to between 6 and 7 times life size were used for droplet analysis.

In order to obtain photographs at a time when the droplet can be assumed to be stationary (i.e. negligible or no sideways
Fig. 5.2 Photographs of Droplet Studies for \( \text{CCl}_4 - \text{HAC} - \text{H}_2\text{O} \) system Contactor I \( R = 1 \)
Fig. 5.3 Actual size photograph used for droplet size analysis,
CCl₄ - HAC - H₂O system  Contactor I  N = 80 opm  Q_c = Q_d = 400 l/hr
Magnification = ? x Life Size
Fig. 5.4 Photographs of Droplet Studies for MIBK - HAC - H₂O system Contactor I
Fig. 5.5 Actual size photograph used for droplet size analysis,
MIBK - HAC - H₂O system Contactor I  N = 65 opm  Q_c = Q_d = 350 l/hr
Magnification = 6 x Life Size
Fig. 5.6 Photographs showing the effect of solute concentration on droplet density MIBK - HAC - H₂O system contactor I
Fig. 5.7 Photographs of Droplet Studies for CCL₄ - HAC - H₂O system Contactor II
movement) a microswitch system was used to trigger off the flash units. The arm of the bell crank closed the microswitch circuit which triggered off the flash when the baffle was at the end of an oscillation and was changing direction. At this point in time the droplets can be assumed to be "instantaneously stationary". A slow shutter speed of 1 second was used to encompass the microswitch relay and the effective exposure time was 1/500th sec.

Photographs were taken at a focal plane 28 mm x 18 mm just inside the wall of the column - approximately 2 mm - this prevented blurred or ill-defined photographs. For both of the contactors distortion of the photographs due to curvature was negligible, more so for the large contactor.

The magnification of the drops was obtained by marking out the focal plane on the body of the contactor at the start of the photographic sessions. This rectangular plane appeared around the edges of the A5 prints, the magnification was calculated from the enlarged rectangular plane and the life size plane on the contactor body, the value was adjusted to take into account the extra depth of field between outer column wall and focal plane inside column, it was found to be the very small - approximately 0.5% - compared with the total magnification.

Four photographs were taken for each experimental condition and the best one was chosen for carrying out the droplet analysis. Photographs of droplets are given in Fig.5.2 to 5.7.

5.3.2 Counting and Sizing of Droplets.

Counting and sizing of the droplets were done by the size analyser described in section 4.6.

A5 size photographs enlarged 6 to 7 times life size were used. The droplet sizes were measured by sliding the ruler blade over the photograph so that the edges of the tapered slot
touched the edge of the droplet to be sized. The droplet was marked and the nearest contact point was touched with the pen, registering a count on the appropriate size group counter and on the total counter. This process was repeated for all the droplets on the photograph that were in focus. The histogram displayed on the counter was recorded. Counting and sizing were done for three prints of each experimental condition and the mean value calculated.

5.4 Mass Transfer Studies.

5.4.1 Preparation and Sampling.

The systems studies were CCL₄ - HAC - H₂O and MIBK - HAC - H₂O. Throughout the experimental work the aqueous phase was de-ionized water and solute (glacial acetic acid) was transferred from the organic to the aqueous phase.

Acetic acid was added to the organic phase and the mixture thoroughly mixed for one hour. For most of the mass transfer runs mixing was carried out in the two, 200 litres storage vessels - due to the large quantities of organic feed required for high flowrate runs. As stirring was being done in only one of the vessels, a uniform solute concentration was achieved by continuously pumping the mixture through the two vessels. This uniformity was monitored by taking regular samples of the inlet feed to the contactor. A rough sketch of the mixing loop is given in Fig.5.8.

After mixing, the mixture was circulated through the organic phase lines by a centrifugal pump for a further 30 minutes. For all the mass transfer studies the solute concentration of the organic phase feed to the contactor was kept between 30 to 40 g/l.
The contactor was filled with aqueous phase after charging the feed supply vessels, and the baffles started at the desired speed. The continuous (aqueous) and dispersed (organic) phases were then introduced into the column via their respective rotameters at the desired flowrate. Constant flowrates were maintained by the control valves positioned just below the rotameters. For CCL₄ - HAC - H₂O system, the less dense continuous phase was introduced into the bottom header of the contacting unit and left from the top header, while the more dense dispersed phase was introduced into the top header. Being the denser of the two phases the drops fell from the top to the bottom where they coalesced in the bottom header.

The depth of the coalescence pool was maintained at 7.5 cm by the outlet control valve immediately below the bottom plate of the contacting unit. For the MIBK - HAC - H₂O system the less dense dispersed phase flowed from bottom to top and the denser continuous phase flowed from top to bottom, other principles
being the same as for the CCL$_4$ - HAC - H$_2$O system. At steady state the inlet and outlet flow rates for each phase were equal.

The continuous phase leaving the contactor flowed into a separation vessel and was eventually discarded (to drain), while the dispersed phase leaving was recycled for further use.

It was found that for the range of mass transfer runs studied, the system reached steady state - i.e when concentration profile along contactor was constant with respect to time - after 30 minutes.

After reaching steady state, eight samples of the two phases were taken at various positions along the column length. Two of these were samples of the dispersed phase at the inlet and outlet, while the remaining six were samples of the continuous phase, which was sampled at five equally spaced positions along the effective length of the contactor and at the outlet from the top header.

The procedure for sampling was by withdrawing approximately 10 cm$^3$/min (5 cm$^3$/min for the small contactor) of continuous phase for between 3 - 5 minutes and discarding them. This served to (a) remove the liquid present in the taps from previous runs and (b) prime the sampling taps with fresh liquid. After priming the taps with fresh liquid 50 cm$^3$ of sample was withdrawn from each position at the same rate as above. The slow withdrawal of liquid ensured that the hydrodynamic equilibrium in the contactor at steady state was maintained during the sampling process.

Two mass transfer runs were done for each of the experimental conditions studied.
5.4.2 Sample Analysis.

5.4.2.1 Refractometry.

The solute content in the samples of both phases were obtained by refractometry. This was carried out on an Abbe Refractometer - described in section 4.5 - in conjunction with the calibration curves for the relevant systems - described in section 5.2.2.

Three to four drops of the sample being analysed were placed between the refractometer prisms which were then clamped together. Viewing through the eye piece, the crosswires were brought into sharp focus by sliding the eye piece draw tube, and a contrasty, colourless borderline defining the light and dark yellow zones was obtained by rotating the dispersion drum. The borderline was then adjusted by the control knob until it was directly on the crosswire intersection. The refractive index of the sample was read directly in the scale telescope and recorded.

The samples were allowed to attain the cell temperature by leaving them for 60 - 90 secs before recording R.I values.

5.4.2.2 Titration.

Titration was used as a means of checking the accuracy of the refractometry method. The solute content of at least two samples for each mass transfer run - outlet continuous phase and inlet dispersed phase samples - already obtained by refractometry were also determined by titration. 10 mls. of each sample was titrated with 0.05 N sodium hydroxide solution with phenolphthalein as indicator.

On the whole solute content values obtained by the two methods were within ± 3%, when after allowing for experimental error confirmed the validity of the refractometry method for obtaining the solute content of the samples.
5.5 Hold-up Measurement.

The hold-up data used in the calculation and analysis of experimental results were those obtained by the manometric method. However, as a means of roughly checking the authenticity of these values, the displacement method was used.

5.5.1 Manometric Method.

The manometer used is described in section 4.2.3. The liquids in the manometer limbs were dyed carbon tetrachloride and water. Special care was taken to eliminate all air bubbles from the manometer lines. The two limbs were connected to the spacer plates at the ends of the contactor, one at each end. The contactor was then filled with continuous phase after which time the levels of organic phase in the two limbs were the same (i.e. zero difference).

The introduction of the dispersed phase into the contactor resulted in a difference in level. When the contactor had reached a state of dynamic equilibrium, the difference in levels was recorded and the fractional volume hold-up was calculated by the method described in Appendix A.2.1.

5.5.2 Displacement Method.

For this method the inlet and outlet flow lines close to the contactor were closed almost instantaneously (within 3 secs.) and the dispersed phase in the contactor was allowed to settle. At the time when the valves were being closed the height of the liquid pool (heavy phase) in the bottom header was noted. After all the dispersed phase had settled in the bottom header the new height of the liquid was recorded and the difference in height was calculated.

This difference in height $h_s$ was made up of (a) the equivalent height $h_1$ of the volume fraction of the heavy phase held up in the contactor (which should be equal to the difference in
height $h_m$ measured on the manometer under the same experimental conditions). (b) the equivalent height $h_2$ of the heavy phase that flowed in after the values were switched off and (c) the equivalent height $h_3$ of heavy phase present in the top and bottom headers of the contactor.

\[ i.e \quad h_s = h_1 + h_2 + h_3 \]  
\[ \text{where} \quad h_1 \quad h_m \]

By making justifiable assumptions, $h_2$ and $h_3$ were calculated, giving the value of $h_1$, details of these assumptions are given in Appendix A.2.2. In all the cases studied the values $h_1$ was within $\pm 10\%$ that of $h_m$ for the same experimental conditions. This can be considered good enough to justify the authenticity of the manometric method.

It must be emphasized that the displacement method is in no way an accurate method for measuring hold-up, in this case unless the dispersed phase present only in the contactor can be isolated instantaneously and settled. For the displacement method studies the experimenter was assisted by two members of the technical staff to obtain the almost instantaneous shut off of the flow lines.

5.6 Power Measurement.

Power measurements were carried out by using the instruments described in section 4.4. The power consumed in oscillating the baffles was measured for different baffle speeds and liquid flow rates.

Studies were carried out for the contactors empty, filled with the continuous phase only and for the two phase ternary systems by measuring the torque.

For each of the experimental conditions studied, the torque readings (to four decimal places) on the integrator unit was recorded every 15 secs over a fifteen minute period (i.e 60
readings for each operating conditions) from which the mean (arithmetic) value was calculated. The readings varied between \( \pm 0.5\% \) of the average value.

The power consumption was calculated from the torque measurements by the formula,

\[
P = 2\pi NT \tag{5.2}
\]

where \( N \) = shaft rotational speed (rev/sec)

\( T \) = Torque (Joules)

5.7 Dye Injection Study.

For mainly practical reasons dye studies were restricted to the continuous phase. If dye studies on the organic phase were to be carried out for all or even some of the operating conditions studies, two methods were available for doing this (1) the use of fresh liquid for each operating condition being studied or (2) the removal of the dye after each study. Due to the large quantities (up to 350 litres) of organic liquids used, it was obvious that the former would have been quite costly, while for the latter it was not possible to obtain a dye which could have been easily removed in a relatively short length of time, thus, the studies were restricted to the continuous phase.

20 cm\(^3\) of concentrated tracer solution (as in section 4.3.3) was rapidly injected (within 3 secs) into the inlet line of the continuous phase through a "subaseal". The injection point was positioned as close as possible to the bottom header of the contactor. At the same instant the dye was injected, the data logger and punch tape was started. These measured and recorded the dye concentration in the outlet stream.

5.8 Flooding Studies.

Flooding studies were carried out for two baffle speeds with the two phase ternary system \((x_i \approx 30 \text{ g/l})\). It was not
possible to carry out studies on the two phases binary system (the importance and usefulness of which is discussed in chapter 8.5), since the organic phase available at the time had traces of solute. However, it was possible to carry out studies with very small solute concentration (0.5 to 1 g/l). Only the CCl₄ -HAC - H₂O system was studied. As for the mass transfer runs, the contactor was filled with continuous phase and air bubbles eliminated from the manometer lines.

With the baffles oscillating at the desired rate, the continuous phase was introduced into the column at a constant flowrate, the dispersed phase flowrate was then increased from 200 l/hr in increments of 50 l/hr. After each increment the contactor was allowed to reach hydrodynamic stability (i.e. constant hold-up) which took between 10 - 15 minutes, after this time the hold-up was recorded, as the maximum operating condition was approached, the increments were reduced to 25 l/hr. This procedure was carried out for three different continuous phase flow rates.

The same procedure was repeated, but this time the dispersed phase flowrate was kept constant and the continuous phase rate was varied.

Maximum operating condition of flooding is said to be reached when the hold-up of dispersed phase increases quite rapidly with small changes in flowrate. At this point the dispersed phase gradually builds up in the contactor and it is not possible to maintain hydrodynamic equilibrium under constant operating conditions.

For the current work maximum operating conditions was taken to be the flowrates at which the difference in manometer levels continued to increase under constant operating conditions. These flowrates were recorded.
CHAPTER 6

DIMENSIONAL ANALYSIS
6.1 Correlation for Sauter Mean Drop Diameter

Several correlations for Sauter mean drop diameter have been proposed for various types of contacting equipment. These correlations together with the physical properties of the liquid systems and operating conditions are tabulated by Tavlarides and Stamatoudis (T1, Pg 230).

From the extensively published literature the factors affecting the Sauter mean drop diameter in the contactor are the physico-chemical properties of the system such as density, \( \rho \), and interfacial tension, \( \sigma \), the turbulence intensity caused by the rate of oscillation (speed parameter), \( \theta N \), diameter of agitator, \( D_A \), and fractional hold-up \( \phi_F \).

Thus

\[
d_{32} = f(D_A, \theta N, \rho, \sigma, \phi_F)
\] .......... (6.1)

Applying Buckingham's \( \Pi \) theorem and taking \( D_A, \theta N, \) and \( \rho \) (geometric mixing and fluid property characteristics) as primary quantities.

Then

\[
F(\pi_1, \pi_2, \pi_3) = 0
\]

Dimensionally

\[
D_A = L
\]
\[
\theta N = T^{-1}
\]
\[
\rho = ML^{-3}
\]

Expressing each of the dimensions \( M, L, T \) explicitly in terms of the primary quantities.

Then

\[
M = \rho D_A^3
\]
\[
L = D_A
\]
\[
T = (\theta N)^{-1}
\]

The three dimensionless groups are obtained by taking each of the remaining variables, \( d_{32}, \sigma, \phi_F \) in turn.
\(d_{32}\) has dimensions \(L\)
\[d_{32} \times L^{-1} \text{ is dimensionless}\]
\[\therefore \quad \pi_1 = d_{32} \times D_A^{-1} = \frac{d_{32}}{D_A}\]

Similar treatment of \(\sigma\) and \(\phi_F\) gives
\[\pi_2 = \frac{\sigma}{(\theta N)^2 D_A^3 \rho}\]
and \(\pi_3 = \phi\)

Thus \(F\left(\frac{d_{32}}{D_A}, \frac{\sigma}{(\theta N)^2 D_A^3 \rho}, \phi_F\right) = 0 \quad \ldots (6.2)\)

\[\therefore \quad \frac{d_{32}}{D_A} = F_1\left(\frac{(\theta N)^2 D_A^3 \rho}{\sigma}, \phi_F\right) \quad \ldots (6.3)\]

\[\frac{\sigma}{(\theta N)^2 D_A^3 \rho}\]
is arbitrarily inverted because Weber Number is usually expressed in the form \((\theta N) D_A^3 \rho / \sigma\).

Equation (6.3) can be written as

\[\frac{d_{32}}{D_A} = K\left(\frac{(\theta N)^2 D_A^3 \rho}{\sigma}\right)^m \phi^n \quad \ldots (6.4a)\]
or

\[\frac{d_{32}}{D_A} = K N_{We}^m \phi^n \quad \ldots (6.4b)\]

6.2 Correlation for Overall Mass Transfer Coefficient

Numerous dimensionless correlations for continuous and dispersed phase individual mass transfer coefficients \(k_c\) and \(k_d\) have been reported in the literature. The most important of these correlations are tabulated in Laddha and Degaleesan (L2 p. 199). For the present studies it was not possible to determine the individual or overall mass transfer coefficients directly. However, overall specific mass transfer coefficients \(K_c^a\) and \(K_d^a\) were determined indirectly from NTU values which were
calculated from the measured concentration profiles. The dimensionless correlation for \( K_{ca} \) as described below is found for the O.B.C.

The factor affecting overall specific mass transfer coefficient \( K_{ca} \) are the operating conditions - phase velocity \( U_c \) and oscillation rate \( \theta N \) -, physical properties - density \( \rho_c \), viscosity \( \mu_c \) and diffusivity \( \alpha_c \) -, and geometric factors such as vessel diameter, \( D \), and Sauter mean drop diameter \( d_{32} \). Thus

\[
K_{ca} = f (U_c, \theta N, \rho_c, \mu_c, \alpha_c, D, d_{32}) \quad \cdots \cdots \quad (6.5)
\]

Apply Buckingham's Pi theorem and taking, \( U_c \), \( D \) and \( \mu_c \) as primary quantities, then

\[
F(\pi_1, \pi_2, \pi_3, \pi_4, \pi_5) = 0
\]

Dimensionally

\[
U_c = LT^{-1}
\]

\[
D = L
\]

\[
\mu_c = ML^{-1}T^{-1}
\]

Expressing each of the dimensions \( M, L, T \) explicitly in terms of the primary quantities, then

\[
M = \mu_c U_c - D^2
\]

\[
L = D
\]

\[
T = U_c^{-1}D
\]

The dimensionless groups are obtained by taking each of the remaining variables in turn,

\[
K_{ca} \text{ has units } T^{-1}
\]

\[
\therefore \ K_{ca} \times T \text{ is dimensionless}
\]

\[
\pi_1 = \frac{K_{ca} D}{U_c}
\]

Similarly,

\[
\pi_2 = \frac{(\theta N) D}{U_c}
\]
\[ \pi_3 = \frac{U_c D \rho_c}{U_c} \]
\[ \pi_4 = \frac{\Omega_c}{U_c D} \]
\[ \pi_5 = \frac{d_{32}}{D} \]

\[ \therefore F \left( \frac{K_c a D}{U_c}, \frac{(\theta N)D}{U_c}, \frac{U_c D \rho_c}{\mu_c}, \frac{\xi_c}{U_c D}, \frac{d_{32}}{D} \right) = 0 \quad \ldots \ldots \ (6.6) \]

\[ \therefore \frac{K_c a D}{U_c} = F_l \left[ \left( \frac{(\theta N)D}{U_c}, \frac{U_c D \rho_c}{\mu_c}, \frac{U_c D}{\xi_c}, \frac{d_{32}}{D} \right) \right] \quad \ldots \ldots \ (6.7) \]

Equation 6.7 can be written in the form;

\[ \therefore \frac{K_c a D}{U_c} = K \left( \frac{(\theta N)D}{U_c} \right)^a \left( \frac{U_c D \rho_c}{\mu_c} \right)^b \left( \frac{U_c D}{\xi_c} \right)^c \left( \frac{d_{32}}{D} \right)^d \quad \ldots \ldots \ (6.8) \]

The first and third dimensionless groups \( \left( \frac{(\theta N)D}{U_c} \right) \) and \( \left( \frac{U_c D}{\xi_c} \right) \) on the form

\[ \left( \frac{(\theta N)D}{U} \right)^a = \left[ \left( \frac{U_c}{U} \right) \times \frac{(\theta N)D^2 \rho}{\mu_c} \right]^a \quad \ldots \ldots \ (6.9) \]

\[ \left( \frac{U_c D}{\xi_c} \right)^c = \left[ \left( \frac{U_c D \rho}{\mu_c} \right) \times \left( \frac{\mu_c}{\rho \xi_c} \right) \right]^c \quad \ldots \ldots \ (6.10) \]

Putting (6.9) and (6.10) into (6.8);

\[ \frac{K_c a D}{U_c} = K \left[ \left( \frac{(\theta N)D^2 \rho}{\mu_c} \right)^a \left( \frac{U_c D \rho}{\mu_c} \right)^e \left( \frac{\mu_c}{\rho \xi_c} \right)^c \left( \frac{d_{32}}{D} \right)^d \right] \quad (6.11) \]

where \( e = b - a + c \)

Equation (6.11) can be written in the form;

\[ \frac{K_c a D}{U_c} = K \left[ N_{Re} \right]_o^{a} \left[ N_{Re} \right]_f^{e} \left[ N_{Sc} \right]^{c} \left[ \frac{d_{32}}{D} \right]^{d} \quad \ldots \ldots \ (6.11a) \]
6.3.3 Correlation for Axial Dispersion Coefficient

The factors affecting the continuous phase axial dispersion coefficient in the O.B.C. are the operating parameter such as phase flow rate, $U_c$, oscillation rate, $\theta N$; the physical properties of the liquid such as density, $\rho_c$ and viscosity, $\mu_c$; and geometrical factors such as vessel diameter $D$, length of contactor $L$, and diameter of agitator $D_A$.

Thus

$$E_c = f(U_c, \theta N, \rho_c, \mu_c, D, D_A, L) \quad \ldots \ldots (6.12)$$

Applying Buckingham's $\Pi_i$ theorem and taking $U_c$, $D$ and $\rho_c$ as the primary quantities. Then,

$$F(\pi_1, \pi_2, \pi_3, \pi_4, \pi_5) = 0$$

Dimensionally,

$$U_c = LT^{-1}$$
$$D = L$$
$$\rho = ML^{-3}$$

Expressing each of the dimensions, $M$, $L$, $T$ explicitly in terms of the primary quantities, then

$$M = \rho_c D^{-3}$$
$$L = D$$
$$T = U_c^{-1} D$$

The dimensionless groups are obtained by a similar treatment as the above, i.e.

$E_c$ has units $L^2 T^{-1}$

$\therefore E_c \times L^{-2} T$ is dimensionless

$\therefore \pi_1 = E_c \times D^{-2} \times U_c^{-1} D$

i.e. $\pi_1 = \frac{E_c}{U_c D}$
Similarly,

\[ \pi_2 = \frac{(N)D}{U_c} \]

\[ \pi_3 = \frac{c}{U_c} \cdot \frac{D}{D} \]

\[ \pi_4 = \frac{DA}{D} \]

\[ \pi_5 = \frac{L}{D} \]

\[ F \left\{ \left( \frac{E_c}{U_c} \right), \left( \frac{(N)D}{U_c} \right), \left( \frac{\mu_c}{U_c\rho_c} \right), \left( \frac{DA}{D} \right), \left( \frac{L}{D} \right) \right\} = 0 \quad \ldots \quad (6.13) \]

Equation (6.13) can be written as:

\[ \frac{E_c}{U_c} = K \left( \frac{(N)D}{U_c} \right)^a \left( \frac{U_c\rho_c}{\mu_c} \right)^b \left( \frac{DA}{D} \right)^c \left( \frac{L}{D} \right)^d \quad \ldots \quad (6.14) \]

Expressing the groups \[ \left( \frac{E_c}{U_c} \right) \] and \[ \left( \frac{(N)D}{U_c} \right) \] in (6.14) as,

\[ \left( \frac{E_c}{U_c} \right) = \left( \frac{E_c}{U_c} \frac{\rho_c}{\mu_c} \right) \cdot \left( \frac{U_c\rho_c}{\mu_c} \right) \quad \ldots \quad (6.15) \]

\[ \left( \frac{(N)D}{U_c} \right)^b = \left[ \left( \frac{(N)D^2\rho_c}{\mu_c} \right) \times \left( \frac{\mu_c}{U_c\rho_c} \right) \right]^a \quad \ldots \quad (6.16) \]

and replacing these groups in (6.14) by (6.15) and (6.16)

\[ \frac{E_c}{v} = K_1 \left( \frac{(N)D^2}{v_c} \right)^a \left( \frac{U_cD}{v_c} \right)^e \left( \frac{DA}{D} \right)^c \left( \frac{L}{D} \right)^d \quad \ldots \quad (6.17a) \]

where kinematic viscosity \( v = \frac{\mu_c}{\rho_c} \)

and \( e = -a + b - 1 \)

Equation (6.17a) can be written in the form

\[ \frac{E_c}{v} = K_1 \left( N_{Re} \right)_{osc}^a \left( N_{Re} \right)_F^e \left( \frac{DA}{D} \right)^c \left( \frac{L}{D} \right)^d \quad \ldots \quad (6.17b) \]

Equation (6.17) was written with kinematic viscosity \( v \) as a variable to facilitate comparisons with published correlations on axial mixing coefficients (see Section 7.3.3)
6.4 Power Correlations for O.B.C.

From the experimental results (Table 19A and Fig. 7.54) it can be seen that the effect of continuous phase and dispersed phase (i.e. hold-up) flow through the contactor on power consumption is negligible. From the extensively published work on power consumption in mechanically agitated contactors, for the O.B.C. the factors affecting the power consumed, $P_M$, in mixing two immiscible phases are the oscillation rate, $\theta N$, agitator or baffle diameter, $D_A$, mean density, $\rho_m$, mean viscosity, $\mu_m$ and interfacial tension, $\sigma$.

Thus

$$P_M = f \left\{ (\theta N), D_A, \rho_m, \mu_m, \sigma \right\} \quad \ldots \ldots \ (6.18)$$

where $\rho_m$ and $\mu_m$ are mean density and viscosity as defined by equations (3.89a&b).

Applying Buckingham's Pi theorem and taking $(\theta N)$, $D_A$ and $\rho_m$ as primary quantities, then

$$F(\pi_1, \pi_2, \pi_3) = 0$$

Dimensionally

$$(\theta N) = T^{-1}$$

$$D_A = L$$

$$\rho_m = ML^{-3}$$

Similar treatment as above gives;

$$M = \rho_m D_A^3$$

$$L = D_A$$

$$T = (\theta N)^{-1}$$

The dimensionless groups are obtained as follows;

$P_M$ has units $ML^2T^{-3}$
\[ P_M \times M^{-1} L^{-2} T^3 \text{ is dimensionless} \]

\[ \pi_1 = P_M \times \rho_m^{-1} D_A^{-3} \times D_A^{-2} \times \sigma N^{-3} \]

i.e. \[ \pi_1 = \frac{\rho_m}{(\sigma N)^3 D_A^5 \rho_m} \]

Similarly
\[ \pi_2 = \frac{\mu_m}{(\sigma N) D_A^2 \rho_m} \]
\[ \pi_3 = \frac{\sigma}{(\sigma N)^2 D_A^3 \rho_m} \]

\[ F \left[ \left( \frac{P_M}{(\sigma N)^3 D_A^5 \rho_m} \right), \left( \frac{(\sigma N) D_A^2 \rho_m}{\mu_m} \right), \left( \frac{(\sigma N)^2 D_A^3 \rho_m}{\sigma} \right) \right] = 0 \]

Equation 6.19 can be written as:
\[ \frac{P_M}{(\sigma N)^3 D_A^5 \rho_m} = K_1 \left( \frac{(\sigma N) D_A^2 \rho_m}{\mu_m} \right)^a \left( \frac{(\sigma N)^2 D_A^3 \rho_m}{\sigma} \right)^b \]...

\[ (6.20a) \]

i.e. \[ N_p = K_1 \left( \frac{N_{Re}}{Re_{osc}} \right)^a \left( N_{We} \right)^b \]...

\[ (6.20b) \]

Applying Olney and Carlson method of analysis (Section 3.5.2.2) by which both sides of equation (6.20a) are multiplied by the Reynolds Number and the term \( \frac{P_M}{D^3} \) is replaced by the power per unit volume. Then

(a) For power per unit active volume, \( P_{VA} \) (equation 3.92)
\[ \frac{P_{VA}}{(\sigma N)^2 \mu_m} = K_2 \left( \frac{(\sigma N) D_A \rho_m}{\mu_m} \right)^x \left( \frac{(\sigma N) D_A^3 \rho_m}{\sigma} \right)^c \]...

\[ (6.21a) \]

or
\[ N_{p_{VA}} = K_2 \left( \frac{N_{Re}}{Re_{osc}} \right)^x \left( N_{We} \right)^c \]...

\[ (6.21b) \]

where \( x = a + 1 = \text{Reynolds Index} \).
(b) For power per unit swept volume, $P_{VS}$ (equation (3.91))

\[
\frac{P_{VS}}{(\theta N)^2 \mu_m} = K_3 \left( \frac{(\theta N) D A^2 \rho_m}{\mu_m} \right)^z \left( \frac{(\theta N) D A^3 \rho_m}{\sigma} \right)^f \quad \ldots \quad (6.22a)
\]

or

\[
N_{P_{VS}} = K_3 \left( N_{Re} \right)_{osc}^z \left( N_{We} \right)^f \quad \ldots \quad (6.22b)
\]

Summarising

\[
N_p = K_1 \left( N_{Re} \right)_{osc}^a \left( N_{We} \right)^b \quad \ldots \quad (6.20b)
\]

\[
N_{P_{va}} = K_2 \left( N_{Re} \right)_{osc}^x \left( N_{We} \right)^c \quad \ldots \quad (6.21b)
\]

and

\[
N_{P_{VS}} = K_3 \left( N_{Re} \right)_{osc}^z \left( N_{We} \right)^f \quad \ldots \quad (6.22b)
\]
CHAPTER 7

RESULTS
Layout of Experimental Results.

Two systems were studied and experiments were carried out on two sizes of contactors. From here on these will be referred to as follows:

- **CCL\_4 - HAC - H\_2O** - System 1
- **MIBK - HAC - H\_2O** - System 2
- Large O.B.C with Multiple Baffles - Contactor I
- Small O.B.C with Single Baffle - Contactor II

Figures - Chapter 7.

- Figs 7.1 to Figs 7.7: Experimental Results
- Figs 7.8: Theoretical Results
- Figs 7.9 to Figs 7.12: Statistical Results

Tables - Appendix 5.

- **Block A**: Tables 1A to 20A: Results for CCL\_4 - HAC - H\_2O systems, Contactor I
- **Block B**: Tables 1B to 8B: Results for MIBK - HAC - H\_2O system, Contactor I
- **Block C**: Tables 1C to 10C: Results for CCL\_4 - HAC - H\_2O system, Contactor II
7.1 Experimental Results.

7.1.1 Solute Concentration Profiles.

The solute concentration profiles along the contactor for Systems 1 and 2 and Contactors I and II are given in Tables 1A to 3A, 1B and 1C; graphical representation is given in Figures 7.1.1 to 7.1.9. The solute concentration in the continuous phase - y values - were obtained experimentally by direct measurements and the dispersed phase solute concentration - x values - were obtained by sectional mass balance. In this respect the profiles are termed "Apparent Measured" values (chapter 3.1.6). For Contactor I the effect of flowratio \( R = Q_c/Q_d \) - Fig.7.13 and inlet solute concentration in the feed - Fig. 7.1.4 on the concentration profile was studied. A comparison of profiles for Contactor I and II at the same phase flow velocity and oscillation rate is given in Fig.7.1.9.

7.1.2 Sauter Mean Drop Diameter and Interfacial Area.

The results for Sauter mean drop diameter \( d_{32} \) and interfacial area are given in Tables 4a - 5a, 2B - 3B, and 2C - 3C. Frequency distribution of droplets for one operating condition of each system are given in Table 4a and 2B and represented graphically in Fig.7.2.1 and 7.2.2. The \( d_{32} \) values were calculated from the frequency distribution of drop for each operating condition by Equation 3.80. Interfacial area were calculated from \( d_{32} \) values by Equation 3.81.

Values of Sauter mean drop diameter and interfacial area are given in Tables 5A, 3B and 3C and represented graphically in Fig.7.2.3 - 7.2.7. A comparison of interfacial area for Contactor I and II at the same operating condition is given in Fig.7.2.8.

Photographs of droplet size distribution for both contactors and systems are given in Fig.5.2 - 5.7 (Chapter 5)
7.1.3 Contactor Overall Performance - Mass Transfer.

The overall contactor mass transfer performance was obtained by calculation of the number of transfer unit NTU, overall mass transfer coefficients $K_a$ and height of transfer units $H_{TU}$ from the concentration profiles. Performance studies were done for both systems and both contactors. The results are given in Tables 6a - 10a, 4b - 6b and 4c - 6c and represented graphically in Fig. 7.3.1 - 7.3.24. Comparison of performance for Contactor I and II are given in Fig. 7.3.16 and 7.3.24.

The NTU values were calculated from equations 3.32 and 3.33. The method used together with the computer program for evaluating the integral are given in Appendix A.4.1. Heights of transfer units and overall mass transfer coefficient were calculated from the NTU values by the same computer program. The method used also provided sectional values of performance characteristics. All the values calculated are "Apparent Measured" values (Chapter 3.1.6).

7.1.4 Dispersed Phase Hold-up.

The dispersed phase hold-up was determined by the manometric method, these values were also checked at regular intervals by the displacement method. Both of these methods are detailed in Appendix A.2.

Hold-up measurements were carried out for both systems and contactors at various operating conditions. Results are given in Tables 6A, 7A, 5B and 4C and graphical representation are given in Fig. 7.4.1 - 7.4.8. The effect of flowrates and inlet solute concentration in the feed for system 1 and Contactor I are given in Fig. 7.4.2 and 7.4.3 respectively.

A comparison of hold-up for Contactors I and II at the same operating conditions are given in Fig. 7.4.8.
7.1.5 Flooding.

Flooding studies were limited to Contactor I and System I, the reason for which are purely practical and is discussed in Chapter 5.8. Studies were carried out with high inlet solute concentration (≈ 30 g/l) and very low inlet solute concentration (0.5 - 1.0 g/l). The latter approximated to "Non-Mass Transfer" system. The results of these studies are given in Tables 11a and 12a. The effect of dispersed phase flowrate on hold-up for mass transfer and "non transfer systems" at high constant continuous phase flowrate is given in Fig.7.5.1. The effect of continuous phase flowrate on hold-up - Continuous phase flow­ rate was taken to the maximum flowmeter range - at various dis­ persed flowrate for "non-mass transfer" system is given in Fig. 7.5.2.

7.1.6 Residence Time and Axial Mixing.

Mean residence and variance was calculated from impulse dye in­ jection studies by the method given in Chapter 3.2.6 - Equations 3.60 and 3.61 - with a computer. The computer program used is given in Appendix A.4.2. Due to practical limitations as discussed in Chapter 5.7, dye injection studies were only carried out on the continuous phase. Peclet numbers were calculated from dimensionless variance by Equation 3.57 and axial mixing coefficients from Peclet numbers by Equation3.65.

Results for mean residence time, variance and dimensionless variance are given in Table 13A and graphical representation of the variation of mean residence with phase flowrate and oscillation rare is given in Fig.7.6.1. Results for Peclet numbers and and axial mixing coefficients are given in Table 14A and plotted graphically in Fig.7.6.4 and 7.6.3 respectively.

The concentration profiles and mass transfer results (Tables 1A and 6A - 10A) enabled the axial mixing component of the diffusion model (Equation 3.67) \( \frac{\partial c}{\partial h^2} \) to be calculated.
These results are given in Table 16A and plotted graphically in Fig. 7.6.5.

7.1.7 Power Study.

Power consumption was calculated from torque measurement obtained from a strain gauge transducer by Equation 5.1. Torque measurement were carried out at various oscillation rate for the contactor empty $T_{Dry}$, filled with continuous phase $T_{Dry} + T_{M1}$ and for both phases in flow $T_T$. The values were also separated into power for mixing single continuous phase $P_{M1}$, and power mixing the two phases $P_M$—both of these power values excludes dry power.

The dry, single phase mixing and two phase mixing torque and power for Contactors I and II and System land 2 are given in Tables 17A - 20A, 7B, 8B, 9C and 10C. The variation of Dry mixing and total power with oscillation rate are given in Fig. 7.7.1 and 7.7.6 for Contactor I and II, system 1 respectively and by Fig. 7.7.4 for system 2. Variation of mixing power with phase flowrate for system 1 and 2 in Fig. 7.7.2 and 7.7.5 respectively. Comparison of power consumption for Contactors I and II at the same operating conditions are given in Fig. 7.7.8. Variation of power consumption with hold-up for Contactors I and II are given in Fig. 7.7.3 and 7.7.7.

To put the power consumed by single and multiple baffles mixing system in a form that is suitable for comparison and scale up the power per unit swept $P_{Vs}$ and per unit active $P_{Va}$ were also calculated. These values are given in Tables 20A, 8B and 10C and plotted graphically in Fig. 7.7.9 and 7.7.10.
FIG. 7.1.1 Concentration profile of continuous phase for CCL₄-HAC-H₂O at constant baffles speed. N = 100 OPM  R = 1  Contactor I
Sampling positions along column length
(scale 1 cm = 15 cm)

FIG. 7.1.2 Concentration profile of continuous phases for CCl₄-HAC-H₂O systems
Q = 400 L/hr  R = 1  Contactor I
FIG. 7.1.3 Concentration profile of continuous phase for CCL₄-HAC-H₂O system at varying flow ratio

Q_d = 400 l/hr  N = 100 OPM  Contactor I
FIG. 7.1.4 Concentration profile of continuous phase for CCL₄-HAC-H₂O system at varying inlet solute concentration
Qₐ = 400 1/hr   R = 1   N = 100 OPM
Contactor I
FIG. 7.1.5 Concentration profiles of continuous phase for MIBK-Acetic-Acid-Water system at constant baffle speed. Column I
Sampling positions along column length
(scale 1 cm = 15 cm)

FIG. 7.1.6 Concentration profiles of continuous phase for MIBK-Acetic Acid-Water System at constant flow rate. Contactor I

\[ Q_C = Q_D = 350 \text{ l/hr} \]
Concentration of continuous phase $y$ (g/L)

Sampling positions along contactor length
(scale 1 cm = 15 cm)

**FIG. 7.1.7** Concentration profile of continuous phase at constant oscillation rate
R = 1  N = 80 OPM  CCL$_4$-HAC-H$_2$O system  Contactor II
FIG. 7.1.8  Concentration profile of continuous phase at constant phase flowrate
R = 1  Q = 100 1/hr  CCL₄-HAC-H₂O
system  Contactor II
Concentration of continuous phase

Key

- Contactor I
- Contactor II

$X_i$ 35.0  g/L

$X$ 32.5  g/L

Sampling positions along contactor length
(scale 1 cm = 15 cm)

FIG. 7.1.9  Comparison of concentration profiles for contactors I and II, CCL$_4$-HAC-H$_2$O

$R = 1$  $U=0.1572$  cm/sec  $N = 80$  OPM
FIG. 7.2.1 Frequency distribution curves of droplets at different oscillation rates Contactor II.

R = 1  Q = 100 1/hr  CCL₄-HAC-H₂O system

Droplet size No. (see Table 2c)
FIG. 7.2.2 Frequency distribution of droplets
R = 1  Q = 350 1/hr
MIBK-HAC-H₂O system  Contactor I
FIG. 7.2.3 Variation of interfacial area with oscillation speed, R = 1
Contactor I CCL₄-HAC-H₂O system
FIG. 7.2.4 Variation of interfacial area with dispersed phase hold-up for contactor I CCl$_4$-HAC-H$_2$O system
FIG. 7.2.5 Variation of measured interfacial area with oscillation rate

$R = 1 \quad Q = 100 \text{ l/hr} \quad \text{CCL}_4$-$\text{HAC}$-$\text{H}_2\text{O}$ system

Contactor II (small).
FIG. 7.2.6 Variation of measured interfacial area with oscillation rate $R = 1$ $Q = 350 \text{ l/hr}$ MIBK-HAC-H$_2$O system Contactor I.

FIG. 7.2.7 Variation of measured interfacial area with phase flowrate $R = 1$ $N = 70$ OPM MIBK-HAC-H$_2$O system Contactor I.
FIG. 7.2.8 Comparison of the variation of measured interfacial area with oscillation rate for contactors I and II

U = 0.1572 cm/sec  R = 1  CCL$_4$-HAC-H$_2$O system
FIG. 7.3.1 Variation of $NTU_{cm}$ with phase flowrate

$R = 1$, CCL$_4$-HAC-H$_2$O system, Contactor I
FIG. 7.3.2 Variation of measured $\text{NTU}_{dm}$ with phase flowrate
Contactor I  $R = 1$  $\text{CCL}_4$-$\text{HAC}$-$\text{H}_2\text{O}$ system
FIG. 7.3.3 Variation of measured number of transfer units of continuous phase with oscillation rate $R = 1$ $Q = 350$ l/hr MIBK-HAC-H$_2$O system Contactor I

FIG. 7.3.4 Variation of measured number of transfer units of continuous phase with phase flow-rate $R = 1$ $N = 70$ OPM MIBK-HAC-H$_2$O Contactor I
FIG. 7.3.5 Variation of measured number of transfer units for continuous phase with phase flowrate $R = 1$, $N = 80$ OPM.
CCL$_4$-HAC-H$_2$O system Contactor II.

FIG. 7.3.6 Variation of measured number of transfer units for continuous phase with oscillation rate $R = 1$, $Q = 100$ l/hr.
CCL$_4$-HAC-H$_2$O system Contactor II.
FIG. 1.3.7 Variation of measured HTU with phase flow rate $R = 1$
Contactor I CCL$_4$-HAC-H$_2$O system
FIG. 7.3.8 Variation of measured HTU\textsubscript{dm} with phase flowrate
R = 1 CCL\textsubscript{4}-HAC-H\textsubscript{2}O system, Contactor I
Flow ratio $R = \frac{Q_c}{Q_d}$

FIG. 7.3.9 Variation of HTU with flow ratio
$Q_d = 400 \text{ l/hr}$ $N = 100 \text{ OPM}$ CCL$_4$-HAC-H$_2$O system Contactor I

FIG. 7.3.10 Variation of HTU with dispersed phase inlet solute concentration $Q = 400 \text{ l/hr}$ $N = 100 \text{ OPM}$ CCL$_4$-HAC-H$_2$O system Contactor I
FIG. 7.3.11 Variation of measured height of transfer units of continuous phase with oscillation rate $R = 1$, $Q = 350$ l/hr, MIBK-HAC-H$_2$O, Contactor I.

FIG. 7.3.12 Variation of measured height of transfer unit for continuous phase with phase flowrate $R = 1$, $N = 70$ OPM, MIBK-HAC-H$_2$O, Contactor I.
FIG. 7.3.13 Variation of measured heights of transfer units for continuous phase with phase flowrate
R = 1  N = 80 OPM
CCL₄-HAC-H₂O system  Contactor II

FIG. 7.3.14 Variation of measured height of transfer units for continuous phase with phase flowrate  R = 1  Q = 100 1/hr
CCL₄-HAC-H₂O system  Contactor II
FIG. 7.3.15 Comparison of the variation of HTU\textsubscript{cm} with superficial velocity for contactors I and II
\[ \text{R} = 1 \quad \text{N} = 80 \text{ OPM} \quad \text{CCL}_4-\text{HAC-}H_2O \]

FIG. 7.3.16 Comparison of the variation of HTU\textsubscript{cm} with oscillation rate for contactors I and II
\[ \text{R} = 1 \quad \text{U} = 0.015716 \text{ cm/sec} \quad \text{CCL}_4-\text{HAC-}H_2O \]
FIG. 7.3.17 Variation of $K_a^c_{cm}$ with phase flowrate

$R = 1$  CCL$_4$-HAC-H$_2$O system  Contactor I
FIG. 7.3.18 Variation of measured mass transfer coefficient for continuous phase with oscillation rate
R = 1  Q = 350 l/hr  MIBK-HAC-H₂O Contactor I

FIG. 7.3.19 Variation of measured mass transfer coefficient for continuous phase with phase flowrate
R = 1  N = 70 OPM  MIBK-HAC-H₂O Contactor I
FIG. 7.3.20 Variation of measured continuous phase mass transfer coefficient with phase flowrate

$R = 1 \quad N = 80 \text{ OPM}$

$\text{CCL}_4$-$\text{HAC}$-$\text{H}_2\text{O}$ system Contactor II

FIG. 7.3.21 Variation of measured continuous phase mass transfer coefficient with phase flowrate

$R = 1 \quad N = 80 \text{ OPM}$

$\text{CCL}_4$-$\text{HAC}$-$\text{H}_2\text{O}$ system Contactor II
FIG. 7.3.22a Variation of $K_a$ with superficial phase velocity $U$ (cm/sec).

$$K_a \times 10^6 \text{ (sec}^{-1})$$

Superficial phase velocity $U$ (cm/sec)

FIG. 7.3.22b Variation of $K_a$ with oscillation rate $N$ (OPM).

$K_a \times 10^6 \text{ (sec}^{-1})$

Oscillation rate $N$ (OPM)
FIG. 7.3.23 Variation of overall mass transfer coefficient along contactor for CCl₄-HAC-H₂O system
Q = 400 1/hr   R = 1   Contactor I

Sampling positions along contactor length
(scale 1 cm = 15 cm)
FIG. 7.4.1 Variation of Hold-up (percent of total volume) with total throughput

\[ Q_c = Q_c + Q_D \text{ - total throughput (1/hr)} \]

\[
\begin{array}{c}
\text{KEY} \\
N \quad 60 \quad 80 \quad 100 \quad 120 \quad \text{OPM}
\end{array}
\]

R = 1 CCL\(_4\)-HAC-H\(_2\)O system Contactor I
FIG. 7.4.2 Variation of dispersed phase hold-up with flow ratio $R = Q_c/Q_d$

$Q = 400$ l/hr $N = 100$ OPM CCl$_4$-HAC-H$_2$O system Contactor I

FIG. 7.4.3 Variation of dispersed phase hold-up with inlet solute concentration of dispersed phase

$Q = 400$ l/hr $N = 100$ OPM CCl$_4$-HAC-H$_2$O system Contactor I
FIG. 7.4.4 Variation of dispersed phase hold-up with oscillation rate
\[ R = 1 \quad Q_t = 700 \text{ l/h} \quad \text{MIBK-HAC-H}_2\text{O Contactor I} \]

FIG. 7.4.5 Variation of dispersed phase hold-up with oscillation rate
\[ R = 1 \quad N = 70 \text{ OPM} \quad \text{MIBK-HAC-H}_2\text{O Contactor I} \]
Dispersed phase hold-up (% of total volume)

Phase flowrate $Q_t = Q_c + Q_d$ (1/hr)

FIG. 7.4.6 Variation of dispersed phase hold-up with phase flowrate $R = 1$ $N = 80$ OPM $x_i = 30$ g/l
CCL$_4$-HAC-H$_2$O system Contactor II
FIG. 7.4.7 Variation of dispersed phase hold-up with oscillation rate

$R = 1$  $Q = 100$ $1/hr$  $x_i = 35$ $g/l$

$CCL_4$-$HAC$-$H_2O$ system  Contactor II
Comparison of the variation of dispersed phase hold-up with oscillation rate for Contactors I and II

$$R = 1 \quad U = 0.1572 \text{ cm/sec} \quad \text{CCl}_4 - \text{HAC} - \text{H}_2\text{O}$$
FIG. 7.4.9 Comparison of effect of dispersed phase flowrate on hold-up for spray towers and O.B.C.

<table>
<thead>
<tr>
<th>KEY</th>
<th>System</th>
<th>( U_c ) cm/sec</th>
<th>( d_{32} ) mm</th>
<th>Column</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Toluene-benzoic acid-water</td>
<td>0.79</td>
<td>1.52-3.04</td>
<td>spray</td>
<td>Appel Elgin (A1)</td>
</tr>
<tr>
<td></td>
<td>MIBK-HAC-H(_2)O</td>
<td>0.671</td>
<td>1.0</td>
<td>spray</td>
<td>Johnson Bliss (J8)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>CCl(_4) - HAC - H(_2)O</td>
<td>0.229</td>
<td>1.0</td>
<td>O.B.C.</td>
<td>Low solute concentration present</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>High solute concentration present</td>
</tr>
</tbody>
</table>
FIG. 4.4.10 Comparison of the effect of continuous phase flowrate on hold-up for spray towers and the O.B.C.

<table>
<thead>
<tr>
<th>KEY</th>
<th>System</th>
<th>$U_D$ (cm/sec)</th>
<th>$d_{32}$ (mm)</th>
<th>Column</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Toluene-benzoic acid-water</td>
<td>0.018</td>
<td>1.5-3.0</td>
<td>Spray tower</td>
<td>Appel &amp; Elgin (A1)</td>
</tr>
<tr>
<td></td>
<td>MIBK-HAC-H$_2$O</td>
<td>0.375</td>
<td>5</td>
<td>Spray tower</td>
<td>Johnson &amp; Bliss (J8)</td>
</tr>
<tr>
<td></td>
<td>CCl$_4$</td>
<td>0.76</td>
<td>1.0</td>
<td></td>
<td>O.B.C.</td>
</tr>
<tr>
<td></td>
<td>HAC</td>
<td>0.157</td>
<td>1.0 to 1.2</td>
<td>O.B.C.</td>
<td>Present</td>
</tr>
<tr>
<td></td>
<td>H$_2$O</td>
<td>0.196</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
FIG. 7.5.1 Variation of hold-up with dispersed phase flowrate for high solute concentration \( x_i = 20 \text{ g/l} \) and low solute concentration \( x_i = 0.5 \text{ g/l} \) CCL\(_4\)-HAC-H\(_2\)O system \( N = 100 \) OPM \( Q_C = 600 \) 1/hr Contactor I
FIG. 7.5.2 Variation of hold-up with continuous phase flowrate low solute concentration ($x_i = 0.5$ g/l) 
CCL$_4$-HAC-H$_2$O system $N = 100$ OPM Contactor I
FIG. 7.6.1 Variation of mean residence with continuous phase flowrate (single phase only) Contactor I
FIG. 7.6.2  Variation of dimensionless variance with Peclet Number for closed (Eqn. 3.57) and open (Eq. 3.58) channels.
FIG. 7.6.3 Variation of Peclet Number with continuous phase flowrate - single phase system
Contactor I
FIG. 7.6.4 Variation of axial mixing coefficient with continuous phase velocity (single phase only) Contactor I
Sampling positions along contactor length
(scale 1 cm = 15 cm)

FIG. 7.6.5 Profile of $E_c \frac{d^2 Y}{dh^2}$ at different phase flowrates
$R = 1$, $N = 100$ OPM
CCL$_4$-HAG-H$_2$O system Contactor I
FIG. 7.6.6 Local accumulation and depletion effects for transferring material for continuous phase

\[
N = 80 \quad R = 1 \quad Q = 400 \text{ l/hr}
\]

Contactor I System 1
FIG. 7.7.1 Variation of dry, mixing and total power consumption with oscillation rate

R = 1  Q = 400 l/hr  CCL₄-HAC-H₂O system
Contactor I
FIG. 7.7.2 Variation of mixing power per unit active volume with phase flowrate

R = 1  CCL₄-HAC-H₂O system  Contactor I
FIG. 7.7.3 Variation of power per unit active volume with dispersed phase hold-up
R = 1 CCL₄-HAC-H₂O system Contactor I
FIG. 7.7.4 Variation of dry, mixing and total power consumption with oscillation rate
R = 1  Q = 350 l/hr  MIBK-HAC-H₂O system
Contactor I

FIG. 7.7.5 Variation of mixing power consumption with phase flowrate  R = 1  N = 70 OPM
MIBK-HAC-H₂O system  Contactor I
FIG. 7.7.6 Variation of dry, mixing and total power consumption with oscillation rate

CCL₄-HAC-H₂O system  Contactor II
FIG. 7.7.7. Variation of power per unit active volume with dispersed phase flowrate

\[ R = 1 \quad Q = 100 \text{ l/hr} \quad \text{CCL}_4-\text{HAC-H}_2\text{O system} \]

Contactor II
FIG. 7.7.8 Comparisons of the variation of dry and total power with oscillation rate for Contactors I & II
$R = 1 \quad U = 0.1572 \text{ cm/sec} \quad \text{CCL}_4\text{-HAC-H}_2\text{O}$
FIG. 7.7.9 Comparison of the variation of power per unit active and swept volume with oscillation rate for Contactors I and II

$R = 1 \quad U = 0.1572 \text{ cm/sec} \quad \text{CCL}_4\text{-HAC-H}_2\text{O system}$
7.2 Theoretical Results.

7.2.1 Concentration Profile from Diffusion Model.

Theoretical concentration profiles for the continuous phase were computed for a number of experiments by solving the one-dimensional diffusion model for the O.B.C developed in Chapter 3.3.2. (Equation 3.71). Concentration profiles were calculated from both numerical and analytical solutions which are given in Appendix A.3.1.1 and A.3.1.2.

The effect of changing number of transfer units along the column length were taken into account in calculating profiles by fitting the sectional values of $N_{oc}$ (from Table 10A) to an exponential curve which was written into the computer program for the numerical solution (A.4.3). For the analytical solutions values were taken directly from Table 10A.

Both types of solution to Eqn.3.71 required initial boundary conditions. Since it was not possible to evaluate accurately, the theoretical boundary condition given by Eqn.A3.15, it was decided to solve the equation for the two boundary conditions as shown below.

Boundary Conditions 1 (B.C.1)

From Eqn. A3.15, at $Z = 0$

$$\frac{dY}{dZ} = P_e [Y_o - Y^O]$$

where $Y_o$, $Y^O$ is defined in Fig.A.3.1

The value of $Y_o$ was measured experimentally while the value of $Y^O$ was unknown. Most workers took the latter to be zero, and B.C.1 was evaluated by taking $Y^O$ as zero, giving

$$\frac{dY}{dZ} = P_e Y_o$$

also at $Z = 0$

$$Y = Y_o$$
Boundary Condition 2 (B.C.2)

For B.C.2 the value of \( \frac{dY}{dZ} \) at \( Z=0 \) was obtained by drawing a tangent at \( Z=0 \) to the experimental profile to get \( \frac{dY}{dZ} \) at this point. Also as for B.C.1, at \( Z=0 \)

\[ Y = Y_0 \]

Experimental and theoretical profiles - for both Analytical and Numerical solutions - at a typical operating condition are given in Table 7.1 and represented graphically in Fig.7.8.1.

The effect of boundary conditions on concentration profile - for the numerical solution - is given in Table 7.1 and represented graphically in Fig.7.8.2.

Table 7.1 Values for Experimental and Theoretical concentration profiles at \( N=80 \) opm \( Q_c=Q_d=400 \) l/hr Contactor 1.

<table>
<thead>
<tr>
<th>( Z=h/H )</th>
<th>Expt1</th>
<th>Dimensionless Concentration, ( Y )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Analytical</td>
</tr>
<tr>
<td></td>
<td></td>
<td>B.C.2</td>
</tr>
<tr>
<td>0</td>
<td>0.308</td>
<td>0.308</td>
</tr>
<tr>
<td>0.25</td>
<td>0.65</td>
<td>0.527</td>
</tr>
<tr>
<td>0.50</td>
<td>0.769</td>
<td>0.602</td>
</tr>
<tr>
<td>0.75</td>
<td>0.850</td>
<td>0.648</td>
</tr>
<tr>
<td>1.00</td>
<td>0.920</td>
<td>0.696</td>
</tr>
</tbody>
</table>

B.C.1 Boundary Condition from Eqn.A3.15 with \( Y_0=0 \)
B.C.2 Boundary Condition from slope of \( Y-Z \) curve at \( Z=0 \)
FIG. 7.8.1 Experimental and theoretical concentration profiles for CCL₄-HAC-H₂O system Contactor I

Qᵥ = Qᵥ = 400 l/hr  N = 80 OPM
FIG. 7.8.2 Effect of boundary condition on concentration profiles from the solution to the diffusion equation. CCL$_4$-HAC-H$_2$O system Contactor I
$Q_c = Q_c = 400$ l/hr $N = 80$ OPM
7.2.2 Calculation of Axial Mixing Coefficient from the Diffusion Equation.

Axial mixing coefficient for the continuous phase at various operating conditions were calculated from one dimensional diffusion equation (Eqn.3.67). The method used is given in Appendix A.3.2. These results are compared with the values obtained from dye studies and from the values calculated from the Westerterp and Landsman type correlation (Eqn.7.9a) in Table 7.2

Table 7.2 Axial Mixing Coefficients values from Dye Studies, Empirical Correlation and Diffusion Equation.

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>Qc (L/hr)</th>
<th>(E^*_c) (cm²/sec)</th>
<th>(E^*_c) (cm²/sec)</th>
<th>(E^*_c) (cm²/sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>200</td>
<td>1.002</td>
<td>1.218</td>
<td>1.13</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>1.67</td>
<td>1.437</td>
<td>1.09</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>2.002</td>
<td>1.655</td>
<td>1.89</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>2.149</td>
<td>1.874</td>
<td>1.74</td>
</tr>
<tr>
<td>80</td>
<td>200</td>
<td>0.985</td>
<td>1.476</td>
<td>1.31</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>2.112</td>
<td>1.699</td>
<td>1.69</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>2.319</td>
<td>1.914</td>
<td>1.74</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>2.463</td>
<td>2.132</td>
<td>4.84</td>
</tr>
</tbody>
</table>

where \(E^*_c\) = \(E^*_c\) obtained from Dye studies

\(E^*_c\) = \(E^*_c\) calculated from Westerterp and Landsman type correlation (Eqn.7.9a)

\(E^*_c\) = \(E^*_c\) obtained from Diffusion Equation (Eqn.3.65 and Appendix A.3.2)
7.3 Statistical Results.

7.3.1 Correlation for Sauter Mean Drop Diameter.

A dimensionless correlation for Sauter mean drop diameter was derived in Section 6.1.

\[ \frac{d_{32}}{D_A} = K N_{We}^m \phi^n \] ...6.4b

Using multiple regression analysis and the experimental data determined values of \( d_{32} \) and \( \phi \), the following correlations for the systems and Contactors were obtained:

**Contactor I (Large) CCL\textsuperscript{4} - HAC - H\textsubscript{2}O system:**

\[ \frac{d_{32}}{D_A} = 3.0 \times 10^{-3} N_{We}^{0.0492} \phi^{0.0017} \] ...7.1

**Contactor I (Large) CCL\textsuperscript{4}-HAC-H\textsubscript{2}O and MIBK-HAC-H\textsubscript{2}O systems**

\[ \frac{d_{32}}{D_A} = 2.24 \times 10^{-2} N_{We}^{-0.235} \phi^{0.0492} \] ... 7.2

**Contactor II (Small) CCL\textsuperscript{4} - HAC - H\textsubscript{2}O system**

\[ \frac{d_{32}}{D_A} = 2.23 \times 10^{-3} N_{We}^{0.102} \phi^{-0.588} \] ...7.3

For the above correlations the standard deviation of \( d_{32}/D_A \) about the regression line and the proportion of total variation about the mean explained by the regression equation, \( R^2 \) are given as follows:

<table>
<thead>
<tr>
<th>Equation No.</th>
<th>% Deviation</th>
<th>( R^2 ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.1</td>
<td>10.01</td>
<td>7.6</td>
</tr>
<tr>
<td>7.2</td>
<td>20.28</td>
<td>28.0</td>
</tr>
<tr>
<td>7.3</td>
<td>18.80</td>
<td>84.3</td>
</tr>
</tbody>
</table>

The values of \( d_{32}/D_A \) calculated from equation 7.1 are compared with the experimental values in Fig.7.9
7.3.2 Correlation for Overall Mass Transfer Coefficient.

A dimensionless correlation for overall mass transfer coefficient $K_a$, for the continuous phase was derived in Section 6.2:

$$\frac{K_a D}{U_c} = K \left[ \frac{(\text{Re}) \rho}{\mu_c} \right]^a \left[ \frac{U_c D \rho_c}{\mu_c} \right]^b \left[ \frac{\mu_c}{\rho_c} \right]^c \left[ \frac{d_{32}}{D} \right]^d \quad \ldots 6.11$$

or

$$\frac{K_a D}{U_c} = K \left( \frac{N_{Re}}{\text{osc}} \right)^a \left( \frac{N_{Re}}{\text{osc}} \right)^b \left( \frac{N_{Sc}}{\rho} \right)^c \left( \frac{d_{32}}{D} \right)^d \quad \ldots 6.11a$$

For the present studies the dimensionless group $\mu_c/D_c \rho$ (i.e., the Schmidt No. $N_{Sc}$) was taken to be same for both systems and contactors, as a result, this group was highly correlated with the other groups in Equation 6.11.

Using multiple regression analysis and the experimental values $K_a$ and $d_{32}$, the following correlation for the systems and contactors were obtained:

1. Contactor I (Large) CCL₄ - HAC - H₂O system

$$\frac{K_a D}{U_c} = 8.33 \times 10^{-8} (\text{Re})_f^{0.509} (\text{Re})_{osc}^{0.668} (d_{32})^{-0.237} \quad \ldots 7.4$$

2. Contactor I (Large) CCL₄-HAC-H₂O and MIBK-HAC-H₂O systems.

$$\frac{K_a D}{U_c} = 0.0237 (\text{Re})_f^{0.274} (\text{Re})_{osc}^{0.953} (d_{32})^{-2.40} \quad \ldots 7.5$$

3. Contactor II (Small) CCL₄ - HAC - H₂O system

$$\frac{K_a D}{U_c} = 3.0 (\text{Re})_f^{0.283} (\text{Re})_{osc}^{0.530} (d_{32})^{-0.272} \quad \ldots 7.6$$

For these correlation (Equations 7.4 - 7.6) the standard deviation of $K_a D/U_c$ about the regression line and the $R^2$ terms are as follows:
<table>
<thead>
<tr>
<th>Equation No</th>
<th>% Deviation</th>
<th>$R^2(%)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.4</td>
<td>12.41</td>
<td>83.3</td>
</tr>
<tr>
<td>7.5</td>
<td>28.79</td>
<td>95.1</td>
</tr>
<tr>
<td>7.6</td>
<td>6.76</td>
<td>77.5</td>
</tr>
</tbody>
</table>

The values of $K_c aD/u_c$ calculated from Equation 7.4 are compared with the experimental values in Fig. 7.10.

### 7.3.3 Correlation for Axial Dispersion Coefficient.

A dimensionless correlation for the axial dispersion coefficient $E_c$, for the continuous phase, was derived in Section 6.3:

$$\frac{E_c}{\nu} = K_1 \left[ \frac{\theta (N D^2)}{\nu_c} \right]^a \left[ \frac{D_A}{D} \right]^c \left[ \frac{L}{D} \right]^d \ldots 6.17a$$

or

$$\frac{E_c}{\nu} = K_1 \left( \frac{N_{Rec}}{osc} \right)^a \left( \frac{N_{Rec}}{f} \right)^e \left[ \frac{D_A}{D} \right]^c \left[ \frac{L}{D} \right]^d \ldots 6.17b$$

Westerterp and Landsman (W6) used a similar type correlation to study axial mixing in a R.D.C and abandoned this approach since they found that the exponents of $(N_{Re})_{osc}$ and $(N_{Re})_f$ were interdependent and it was difficult to attach a physical model to such a correlation. Instead Westerterp and Landsman (W6) state that from the previous work of Stemerding et al (S18) and direct observation of their own results, a plot of $E'$ against '$u$' at constant stirrer speed gave a straight line. This can be interpreted as the sum of two contributions:

(i) a contribution independent of '$u$' and approximately proportional to $N$.

(ii) a contribution independent of $N$ and proportional to $V$.

For a given contactor the relationship is given by (Chapter 2.4):

$$\frac{E_c}{\nu} = \frac{E_{cN}}{\nu} + \frac{E_{cu}}{\nu} \ldots 7.7$$

It is not known whether this method of analysis is directly applicable to the present studies. However, recognizing the
that the exponents of the dimensionless Reynolds Number are interdependent, and in the absence of an alternative method, it has been decided to adapt the same method of analysis (Westerterp and Landsman, W6) for the present studies.

Since the dimensionless terms $D_A/D$ and $L/D$ are constant for a given contactor, then putting equation 6.17b in the same form as equation 7.7:

$$\frac{E_{CN}}{\nu_c} = C_1 \frac{(\theta N)D^2}{\nu_c}^a \quad \ldots 7.8a$$

and

$$\frac{E_{cu}}{\nu_c} = C_2 \frac{u_cD}{\nu_c}^e \quad \ldots 7.8b$$

The values of $E_{CN}/\nu_c$ were obtained from the intercepts of the plot, $E_c/\nu_c$ vs $u_cD/\nu_c$ and $E_{cu}/\nu_c$ from the intercepts of the plot $E_c/\nu_c$ vs $(\theta N)D^2/\nu_c$ (Eqn.7.7) as shown in Figs.7.11.1 and 7.11.2 respectively. The constants $C_1$ and $C_2$ and the exponents 'a' and 'e' were found from plots of $E_{CN}/\nu_c$ vs $(\theta N)D^2/\nu_c$ and $E_{cu}/\nu_c$ vs $u_cD/\nu_c$ as shown in tables 7.2a & b below and Fig.7.11.3.

Westerterp and Landsman - Type Correlation for Axial Mixing Coefficient.

$$\frac{E_c}{\nu_c} = \frac{E_{CN}}{\nu_c} + \frac{E_{cu}}{\nu_c} \quad \ldots 7.7$$

$$\frac{E_{CN}}{\nu_c} = C_1 \left[ \frac{(\theta N)D^2}{\nu_c} \right]^a \quad \ldots 7.8a$$

$$\frac{E_{cu}}{\nu_c} = C_2 \left[ \frac{u_cD}{\nu_c} \right]^e \quad \ldots 7.8b$$
### Table 7.3a (From Fig. 7.11.1)

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>((E_{CN}/\nu) \times 10^{-2}) From Intercepts Fig. 7.11.1</th>
<th>((\theta N) D^2/\nu) \times 10^{-4})</th>
<th>(C_1) From slope Fig. 7.11.3a</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>0.50</td>
<td>4.87</td>
<td>1.325</td>
</tr>
<tr>
<td>60</td>
<td>0.84</td>
<td>5.85</td>
<td></td>
</tr>
<tr>
<td>80</td>
<td>1.16</td>
<td>8.78</td>
<td></td>
</tr>
</tbody>
</table>

### Table 7.3b (From Fig. 7.11.2)

<table>
<thead>
<tr>
<th>(u_c \times 10^{-2}) cm/sec</th>
<th>((E_{cu}/\nu) \times 10^{-2}) From Intercepts Fig. 7.11.2</th>
<th>((u_c D/\nu)</th>
<th>(C_2) From slope Fig. 7.11.3b</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.076</td>
<td>0.45</td>
<td>239.5</td>
<td>0.185</td>
</tr>
<tr>
<td>0.117</td>
<td>0.70</td>
<td>357.66</td>
<td></td>
</tr>
<tr>
<td>0.157</td>
<td>0.83</td>
<td>475.87</td>
<td></td>
</tr>
</tbody>
</table>

Table 7.3 Data for Westerterp and Landsman type Correlation for Axial Mixing Coefficient
From Fig. 7.11.3, a & b the slope of the plots in both cases are straight lines, thus

\[ a = e = 1 \]

Therefore, the dimensionless equation for continuous phase axial mixing coefficient (Equ. 7.7) becomes:

\[
\frac{E_c}{\nu} = 1.325 \times 10^{-3} \left( \frac{(\theta N)D^2}{\nu_c} \right) + 0.185 \left( \frac{u_c D}{\nu_c} \right) \quad \ldots 7.9a
\]

or

\[
\frac{E_c}{\nu} = 1.325 \times 10^{-3} \left( N_{Re} \right)_{osc} + 0.185 \left( N_{Re} \right) \quad \ldots 7.9b
\]

Putting equation 7.9a in the form of the Westerterp and Landsman equation:

\[
P_e = \frac{L/D}{(1.325 \times 10^{-3}) (\theta N)D^2/u_c} + 0.185 \quad \ldots 7.10
\]

The values of \( E_c \) calculated from equation 7.9a are compared with the experimental values in Fig. 7.11.4

7.3.4 Power Correlations for the O.B.C.

7.3.4.1 Correlations for Total Mixing Power.

A dimensionless correlation for the total mixing power \( P_M \) was derived in Section 6.4:

\[
P_M = K_1 \left[ \frac{(\theta N)D_A^2 \rho_m}{u_m} \right]^a \left[ \frac{(\theta N)D_A^3 \rho_m}{\sigma} \right]^b \quad \ldots 6.20
\]

or

\[
N_p = K_1 \left( N_{Re} \right)_{osc}^a \left( N_{We} \right)^b \quad \ldots 6.21
\]

For the present studies the interfacial tension was taken to be constant since over the concentration range studied, concentration had very little effect on interfacial tension.
As a result of this the Weber No. was constant for all the operating conditions studied, making it highly correlated with the Reynolds No., thus, reducing the correlation to:

\[ N_p = K_1 (N_{Re})^{a} \quad \ldots 7.11 \]

Using multiple regression analysis and the experimental values of \( P_M \), the following correlations were obtained:

**Contactor I (Large), CCL\(_4\) - HAC - H\(_2\)O system**

\[ N_p = 1.34 N_{Re}^{0.453} \quad \ldots 7.12 \]

**Contactor I (Large), CCL\(_4\) - HAC - H\(_2\)O and MIBK - HAC - H\(_2\)O systems**

\[ N_p = 4.904 N_{Re}^{0.320} \quad \ldots 7.13 \]

**Contactor II (Small), CCL\(_4\) - HAC - H\(_2\)O system**

\[ N_p = 1.47 \times 10^6 N_{Re}^{-0.895} \quad \ldots 7.14 \]

Analysis of the regression equation is given as follows:

<table>
<thead>
<tr>
<th>Equation No.</th>
<th>% Deviation</th>
<th>( R^2(%) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.12</td>
<td>15.93</td>
<td>52.0</td>
</tr>
<tr>
<td>7.13</td>
<td>10.09</td>
<td>49.8</td>
</tr>
<tr>
<td>7.14</td>
<td>5.64</td>
<td>97.1</td>
</tr>
</tbody>
</table>

**7.3.4.2 Correlations for Power Unit Active Volume.**

The dimensionless correlation developed in Section 6.4 for power per unit Active Volume \( P_{va} \) is:

\[ \frac{P_{va}}{\theta N } = K_2 \left[ \frac{(\theta N)^2 D_a^2 \rho_m}{\mu_m} \right]^{x} \left[ \frac{(\theta N)^3 D_a^3 \rho_m}{\sigma} \right]^{c} \quad \ldots 6.22a \]
or \[ N_{P, Va} = K_2 (N_{Re})^{x}_{osc} (N_{We})^c \quad \ldots 6.22b \]

For constant \( \sigma \), this correlation reduces to:

\[ N_{P, Va} = K_2 \ 1 (N_{Re})^{x}_{osc} \quad \ldots 7.15 \]

Using multiple regression analysis and the experimental values of \( P_{va} \), the following correlations were obtained:

Contactor I (Large), CCL\(_4\) - HAC - H\(_2\)O system,

\[ N_{P, Va} = 0.244 N_{Re}^{1.36} \quad \ldots 7.16 \]

Contactor I (Large), CCL\(_4\) - HAC - H\(_2\)O and MIBK - HAC - H\(_2\)O systems.

\[ N_{P, Va} = 0.232 N_{Re}^{1.36} \quad \ldots 7.17 \]

Contactor II (Small), CCL\(_4\) - HAC - H\(_2\)O system.

\[ N_{P, Va} = 5.96 \times 10^6 N_{Re}^{0.566} \quad \ldots 7.18 \]

Analysis of the regression equations is as follows:

<table>
<thead>
<tr>
<th>Equation No.</th>
<th>% Deviation</th>
<th>( R^2(%) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.16</td>
<td>1.14</td>
<td>99.9</td>
</tr>
<tr>
<td>7.17</td>
<td>1.25</td>
<td>99.9</td>
</tr>
<tr>
<td>7.18</td>
<td>6.28</td>
<td>89.7</td>
</tr>
</tbody>
</table>

7.3.4.3 Correlations for Power Per Unit Swept Volume.

From Section 6.4 the power per unit swept volume \( P_{vs} \) is given by the following correlation:

\[ \frac{P_{vs}}{\mu_m} \ 2 \left( \frac{\theta N \ D_A^2 \ \rho_m}{\mu_m} \right)^2 \left[ \frac{\theta N \ D_A^3 \ \rho_m}{\sigma} \right]^f \quad \ldots 6.23a \]
or \[ N_{PvS} = K_3 (N_{Re})^Z \left( N_{We} \right)^f \] \( \ldots 6.23b \)

For constant \( \sigma \), this correlation reduces to:

\[ N_{PvS} = K_3 (N_{Re})^Z \left( N_{We} \right)^f \] \( \ldots 7.19 \)

Using multiple regression analysis and the experimental values of \( P_{VS} \) (Tables 20A, 8B, 10C), the following correlations were obtained:

Contactor I (Large), CCL\(_4\) - HAC - H\(_2\)O system

\[ N_{PvS} = 0.530 (N_{Re})^{1.36} \] \( \ldots 7.20 \)

Contactor I (Large), CCL\(_4\) - HAC - H\(_2\)O and MIBK - HAC - H\(_2\)O systems

\[ N_{PvS} = 0.232 (N_{Re})^{1.38} \] \( \ldots 7.21 \)

The correlation for Contactor II was very poor with a large standard deviation.

Analysis of the regression equations is as follows:

<table>
<thead>
<tr>
<th>Equation No</th>
<th>% Deviation</th>
<th>( R^2(%) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.20</td>
<td>1.58</td>
<td>99.9</td>
</tr>
<tr>
<td>7.21</td>
<td>2.79</td>
<td>99.6</td>
</tr>
</tbody>
</table>

The values of \( N_{Pva} \) and \( N_{PvS} \) calculated from equations 7.16 and 7.20 are compared with the experimental values in Fig.7.12.
FIG. 7.9 Comparison of experimental and calculated Sauter mean drop diameter
Contactor I  CCL₄-HAC-H₂O

\( \frac{d_{32}}{D_A} \) calculated Eqn. 7.1
FIG. 7.10 Comparison of experimental and calculated mass transfer coefficients for CCL\textsubscript{4}-HAC-H\textsubscript{2}O Contactor I

\[ \left( \frac{K_c a D}{U_c} \right) \times 10^3 \] - calculated from Eq. 7.4
FIG. 7.11.1 Variation of axial mixing parameter with Reynolds Number for oscillation
FIG. 7.11.2 Variation of axial mixing parameter with Reynolds Number for flow
FIG. 7.11.3b

Determination of constants for the Westerterp and Landsman-type correlation for axial mixing.
FIG. 7.11.4 Comparison of experimental and calculated axial mixing coefficients for Contactor I
FIG. 7.12 Comparison of experimental and calculated power per unit volume for Contactor I

\[
\left( \frac{P_v}{(\theta)^2 \mu} \right) \times 10^{-4} \text{ calculated} \quad \text{(see Key)}
\]

\[
\left( \frac{P_v}{(\theta)^2 \mu} \right) \times 10^{-4} \text{ - Experimental}
\]
CHAPTER 8

DISCUSSION
8.1 Hydrodynamic Results.

Droplet studies were carried out by direct photographic techniques. The purpose of the study was to determine the effect of operating conditions on interfacial area which is an important factor with regards to the rate of mass transfer and hence extraction efficiency. From the photographic studies it was possible to establish the drop size distribution at different operating condition from which the Sauter mean drop diameter and interfacial area could be calculated.

8.1.1 Drop Size Distribution.

As would be expected in a contactor where one phase is dispersed in another, there exists a distribution of drop sizes, this was the case for the 0.B.C as shown by Figs.7.2.1 and 7.2.2 for systems 1 and 2. In these Figures, frequency distribution at different oscillation rates were plotted for constant phase flowrates. It can be seen that when the peaks of the distribution curves are joined together as indicated, they appear to lie on an exponential curve. This type of behaviour is expected since increasing oscillation results in a decrease in drop sizes, and it is in some agreement with the results of Ward and Knudson (W2) and Ross-reproduced in (T1) Pg.243. It can also be seen that the spread of distribution about the peak diameter decreases with increase in oscillation rate, thus indicating that the drop size become more uniform at high oscillation rate. It would appear from the two graphs that for the CCL$_4$ - HAC - H$_2$O system the distributions are skewed-normal distributions while for the MIBK- HAC - H$_2$O system they are closer to normal distributions.

8.1.2 Sauter Mean Drop Diameter.

From Tables 5A and 3B there appears to be no definite trend with regards to the effect of operating conditions - varying oscillation rate and phase flowrate - on Sauter mean drop diameter for Contactor I (Large) and Systems 1 and 2.
For system 1 drop sizes ranged from 0.114 to 0.153 cm and for system 2 from 0.073 to 0.088 cm over the range of operating conditions studies. However, on the whole it would appear that drop sizes increase with increasing flowrate and decreases with increasing oscillation rate to a minimum, then increases with further increase in oscillation rate. This type of behaviour would be expected since an increase in flowrate at constant oscillation rate results in an increased droplet hold-up, the effect of which is to promote coalescence. With increasing oscillation rate a decrease in drop size under constant flow conditions would be expected as a result of drop break-up, however, there appears to be some optimum oscillation rate at which the coalescence process predominates the break-up process due to increasing droplet density, thus, resulting in an increase in drop size. Conversely for Contactor II (Small) increasing oscillation rate resulted only in decreasing drop size, the extent of which is more marked than for the large contactor, while the effect of flowrate at constant oscillation rate is similar to that of Contactor I (Large). It therefore appears that the effect of the oscillation rate for the two contactors are different.

One of the important results from the photographic studies is that there was no visual evidence of drop coalescence. This is also true for all the previous photographic work carried out on the O.B.C - both stills and cine film - when operating under mass transfer conditions. It is widely accepted and quoted that droplets do coalesce in a drop swarm. The possible explanations for the lack of this coalescence are (1) the distance between droplets is very large as compared to the diameter of the drop by using equation 2.25, drops were found to be between 4-8 diameters apart for Contactor I system 1 - thus, greatly reducing the probability of collision which is the first stage of coalescence. (b) the diameter of drops are so small (<1 mm) that they behave like rigid elastic spheres and thus rebound off each other when they collide.

Correlations for Sauter mean drop diameter - single systems,
two systems and both contactors - obtained from experimental results are given by equations 7.1 to 7.3.

The agreement between experimental and calculated values for Contactor I (Fig.7.9) was quite poor. This is not surprising since the experimental results did not show any definite trend between $d_{32}$ and operating conditions. In addition the variation in $d_{32}$ over the range of operating conditions studied was very small (1.1 to 1.4 mm).

Although the agreement was poor, the exercise was useful inasmuch it showed that unlike most of the other types of contactors, $d_{32}$ for the O.B.C cannot be predicted with reasonable accuracy from empirical correlations. A table of such correlations for a wide range of contactors is given by Tavlarides and Stamatondis (T1 Pg.230).

8.1.3 Dispersed Phase Hold-up.

The effect of operating conditions on hold-up is given in Fig. 7.4.1, 7.4.2 and 7.4.3 for Contactor I (Large) system 1. It can be seen that hold-up increases with both oscillation rate and phase flowrates.

If the lowest oscillation rate and throughput are taken as the datum, then over the ranges of operating conditions studied, the effect of oscillation rate on hold-up is more pronounced than that of phase flowrate. This can be explained from the drop size studies which show that the oscillation rate has a more marked effect on drop size than flowrate. At a constant total throughput increasing oscillation rate and hence increasing energy input results in smaller drops with larger residence time, giving an increased drop hold-up. Constant oscillation rate and increasing flowrate also results in large drop residence times. However, due to the presence of more liquid in the contactor with the same energy input, the change in hold-up is not as pronounced as for oscillation rate increase.
The effect of flowratio on hold-up at a constant oscillation rate is shown in Fig.7.4.2. It can be seen that the hold-up decreases with increasing flowratio, $Q_c/Q_d$ (or when $Q_c/Q_d < 1$, the hold-up increases). This type of behaviour was also obtained by Thomas (T6) for the 3 inch I.D O.B.C and may be explained as follows: at constant oscillation rate and low flowratio most of the energy supplied by the baffles goes into dispersing the heavy phase, as the flowratio is increased - i.e. increase in total throughput the energy available for dispersing the heavy phase is less due to increasing continuous phase flowrate. This reduced energy per unit throughput results in larger drop sizes, hence a decrease in hold-up.

Fig.7.4.3 shows the effect of inlet solute concentration in the feed on hold-up at constant operating conditions. It can be seen that at very low solute concentration the hold-up is very high and then falls sharply within a range of 2 g/l. This type of behaviour can be explained by the effect acetic acid concentration has on interfacial tension. As shown in Appendix 1 the interfacial tension falls rapidly when the solute concentration increases from 0 to 1 g/l and remains almost constant with further increase in concentration. At high interfacial tension i.e very low solute concentration, the break-up of droplet predominates any other factor that affect drop interaction resulting in very fine dispersions, while at low interfacial tension i.e relatively high solute concentration the tendency of drop interaction and other factors such as drop oscillation and internal circulation can result in larger drops with a small residence time and hence low droplet hold-up. Therefore, it is obvious that feed solute concentration is an important factor in establishing the contactors maximum operating conditions.

Figs.7.4.4 and 7.4.5 shows the effect of operating conditions on hold-up for system 2. It can be seen that similar hold-up values can be achieved at a much lower oscillation rates than those for System 1. This is due to the much lower interfacial tension of MIBK - HAC - $H_2O$ which requires less energy to effect
a similar degree of dispersion.

Figs 7.4.6 and 7.4.7 shows the effect of operating conditions on hold-up for Contactor II (Small) System 1. Again as for Contactor I (Large) it can be seen that if the lowest oscillation rate and throughput are taken as the datum point, then over the ranges of operating conditions, the oscillation rate has a more marked effect on hold-up than throughput. A comparison of the two contactors Fig 7.4.8 show that at a given flowrate Contactor II is likely to approach it maximum operating condition at lower oscillation rate than Contactor I. On the whole it would appear that Contactor I can operate effectively at higher operating conditions than Contactor II, which is desirable with respect to scaling up.

Fig 7.4.9 and 7.4.10 show comparison of the effect of continuous and dispersed phase velocity (based on x-section area of contactor) respectively, on dispersed phase hold-up for spray towers and the O.B.C.

The graphs of Appel and Elgin (A1) and Johnson and Bliss (J8) are interesting in that they show hold-up to be a function of the system (interfacial tension), greater the value of interfacial tension, the greater the hold-up. It (hold-up) may also be related to drop size as well as \( u_d \) and \( u_c \), but to a lesser extent. The O.B.C shows that hold-up is definitely a function of \( u_d \), and increases with \( u_c \) up to a certain point (Appel show that \( \phi \) decreases when \( u_c > 1.0 \) cm/sec). Generally, the hold-up values for the O.B.C are in the same range as for the spray column.

From the Figures, comparing the two contactors, it can be concluded that a number of factors affect droplet hold-up in a contactor, namely, physico-chemical properties, operating conditions, and the internal design of the contactor. All these factors which are interdependent make the analysis of hold-up behaviour in contacting devices rather difficult, and must be taken into account when comparing hold-up values for various
types of contactor.

8.1.4 Interfacial Area.

The variation of interfacial area with operating conditions for Contactor I (Large), System 1 is shown in Fig.7.2.3. It can be seen that increasing oscillation rate at constant flowrate results in a marked increase in interfacial area.

Fig.7.2.4 shows interfacial area to be directly proportional to dispersed phase hold-up. Since, they are related by:

\[ a = \frac{6\phi}{d_{32}} \ldots 3.81 \]

It would appear that the interfacial area is independent of drop diameter. Similar relationships has been reported for spray and packed towers by Ladda and Degaleesan (L2 Pg.452 and 454). It is clear that the magnitude of the interfacial area is dependent solely on the hold-up, thus the factors affecting hold-up also apply to interfacial area.

Comparing the spray contactor with the O.B.C, for the spray column once the drops are formed by the nozzle there is no agitation to change their size. Some disruption will occur for low interfacial tension. In this case 'a' is much more dependent on \( \phi \) than \( d_{32} \). Therefore, it follows that 'a' depends on \( \phi \), and all the parameters, \( u_c \), \( u_d \) and \( \sigma \) which affect \( \phi \) will also affect 'a' almost in direct proportion. For the O.B.C the oscillating baffles act fairly quickly in producing a dispersion with almost constant drop sizes - i.e a state where the drops are formed is reached soon after the discontinuous phase enters the contactor. The lower hold-up values especially ensure the drop do not coalesce, resulting in a fairly constant drop size which is low in value (1 to 2mm). Again the hold-up determines 'a', while \( u_c \), \( u_d \) and \( \sigma \) fix the values of hold-up. Fig.7.2.4 show the same 'a' dependency for the O.B.C.

Fig.7.2.8 compares the effect of oscillation rate on inter-
facial area for Contactor I (Large) and II (Small). It can be seen that under similar operating conditions, increasing oscillation rate has a much more marked effect on 'a' for Contactor II than for Contactor I. This is due to the difference in the degree of mixing within the two contactors. For Contactor I (Large) only 52% of the active volume is directly mixed by the baffles while for Contactor II (Small), 82% of the active volume is directly mixed by the baffles. However, the behaviour of the curves (Fig.7.2.8) suggest that Column II is quite close to its maximum operating condition at 120 opm at the specified throughput, while it would appear that Contactor I can go on operating efficiently at much higher oscillation rates.

<table>
<thead>
<tr>
<th>N opm</th>
<th>u cm/sec</th>
<th>a cm^2/cm^3</th>
<th>Kc x 10^6 cm/sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>Contactor I (Large)</td>
<td>120</td>
<td>0.157</td>
<td>1.067</td>
</tr>
<tr>
<td>Contactor II (Small)</td>
<td>120</td>
<td>0.157</td>
<td>8.280</td>
</tr>
</tbody>
</table>

Table 8.1 (Data from Tables 5A and 3C)

It is interesting to note from Table 8.1 that under the same conditions of operating, the mass transfer coefficient obtained in Contactor I (Large) is approximately 7 times that of Contactor II (Small), while the interfacial area in the latter is 8 times that of the former. It is not obvious why Kc is improved to the extent it is in the larger contactor. Inspection of theoretical equations containing k_c (within the Sherwood No.) - given in Laddha and Degaleesan (L2 Pg.199 & 200) and listed in Appendix A.3.3 for convenience - is not all that helpful inasmuch as the equations are very limited in application. In reality in the present contactors, there is a range of drop sizes so that despite the fact d_{32} is small in value
(1 mm) there will be a range of drop sizes. In some of the drops there may be internal circulation and even oscillation, while other smaller drop behave like rigid elastic spheres. Referring to the Tables in Appendix A.3.3, within the present range of drop sizes there would be little effect of $d_{32}$ on $k_c$. This is also true of $k_d$. In one case of Handlos and Baron (H3), drop diameter is not included in the equation for $k_d$. There is the added difficulty that for most of these equations they apply to a single file of drops and therefore become uncertain when applied to swarm of drops. Clearly, as has been stated before there are many such equation for $k_c$, $k_d$ and $N_{Shc}$ but in practice they have limited value. Referring to the present case of Table 8.1 it can be stated with confidence that the measurement of $d_{32}$ experimentally is most unlikely to contain an error in $d_{32}$ to allow for the difference of 8 times the interfacial area in one contactor compared with the other. Investigation shows that as $a'$ is obtained from Equ.3.8.1 ($a = 6\phi/d_{32}$) then there is a compensating effect in $\phi_f$ (the hold-up) which within the limitations of the present drop sizes distribution, the magnitude of 'a' is not seriously affected. This in fact minimizes error of measurement.

Therefore, the trend in the value of $K_c$ is quite acceptable, the only comment is that it highlights the changes that can occur in mass transfer coefficients under real conditions that cannot be predicted with confidence from existing theoretical equations. The same remarks can be made about the problems of scale-up.

Clearly, from Table 8.1, with virtually the same value for $K_c a$ the large contactor is just as effective in the mass transfer as the smaller contactor, but of course it has a much higher duty. In this sense the present arrangement of three baffles in the larger contactor is most acceptable, however, because of the mode of operating of the large contactor (there is a proportion of unswept volume) one has to say that the problem of scale-up is a very difficult one.
8.2 Mass Transfer Results.

8.2.1 Solute Concentration Profiles.

The effect of operating conditions on concentration profiles for Contactor I (Large), System 1, is shown in Figs. 7.1.1 and 7.1.2. The concentration profiles are for the continuous phase since these were obtained by direct experimental measurements. The dispersed phase profiles were obtained from mass balance using a measured y and the terminal x values. Although, they have not been represented graphically, they have the same shape as their corresponding continuous phase profiles.

Taking the lowest phase flowrate and oscillation rates as the datum for comparison, it can be seen that there is a gradual change in the shapes of the profiles as the phase flowrates are increased at a constant oscillation rate and constant inlet solute concentration in the feed phase ($x_1$) Fig.(7.1.1). At low flowrates a large percentage of the solute extracted takes place in the lower part of the contactor, as the flowrate is increased the whole contactor become more effective in solute extraction.

From 7.1.2 it can be seen that there is a marked in oscillation rate which are more pronounced than those of changes in flowrate (Fig.7.1.1). At the lowest oscillation rate it can be seen that more than 75% of the solute extracted takes place within 35% of the active volume of the column, indicating that the contactor is under operated at this oscillation rate. As the oscillation rate is increased more of the contactor's active volume becomes effective in the extraction process -at the highest oscillation rate 100% of active volume is effective. This poor extraction at low oscillation rates can be attributed to poor dispersions resulting in large drop sizes, thus low interfacial area, and low mass transfer rates.

Fig7.1.3 show the effect of flowratio at constant $x_1$, at a
high oscillation rate. It can be seen that all the curves are similar in shapes and indicate a large percentage of the active contactor volume is effective in solute extraction.

Fig 7.13 shows the effect of inlet solute concentration \( x_i \) in the feed on concentration profiles. The shapes of the profiles are approximately the same.

Figs. 7.1.5 and 7.1.6 show the effect of operating conditions on concentration profile for System 2. It can be seen that the effect of the individual operating conditions on the concentration profile are the same as for System 1.

Fig 7.1.7 and 7.1.8 show the effect of operating conditions on profiles for Contactor II, System 1. Although the operating conditions has the same effect as for Contactor I, the shape of the profile are different (as shown in Fig.7.1.9 at similar operating conditions), they have a steeper slope and take the form of an S-shape. It is evident that the two end sections of the active volume is not as effective in extracting solute as the mid-section. The shapes of the profile may be explained by the variation in drop diameter along the contactor length. At the top of the contactor where the dispersed phase enters the drops are relatively large giving a low interfacial area for mass transfer. As they move through the contactor they are broken up, and by the time they reach the lower end of the contactor a large proportion becomes so small in size that they behave like rigid elastic spheres, which can have the effect of reducing the mass transfer rate due to reduced internal circulation. The difference in slope can be attributed to difference of active volume that is being swept by baffles in the two contactors - for Contactor I 52% of the active volume is swept by the baffles while for Contactor II 82% is swept by baffles.
8.2.2 Effect of Operating on Contactor Performance.

The mass transfer performance of the contactors was established by calculating Number of Transfer Units NTU, Heights of Transfer Units HTU and Overall Mass Transfer Coefficients Ka. For the present studies the NTU values were calculated from the continuous phase concentration profiles and the equilibrium relationship (Eqn.A.1.1) by the method described in Chapter 3.1.6. The other two performance characteristics were calculated from the NTU values by equations 3.2.1 and 3.25. It was decided that as the NTU values were obtained from direct experimental measurements, these values would take precedence over the others in discussing the performance characteristics.

Although the discussion that follows is based on the apparent measured continuous phase values of $NTU_{cm}$, $HTU_{cm}$ and $Ka_{cm}$, values for the dispersed phase $NTU_{dm}$, $HTU_{dm}$ and $Ka_{dm}$ were also calculated and are shown in Tables 6A, 7A, 5B, and 4C for the different contactors and systems. The dispersed phase values, although different in magnitude from the continuous phase values, show the same trend with variation in operating conditions (Fig.7.3.3) as the continuous phase values.

Overall Number of Transfer Units.

The overall number of transfer units is important since it is a measure of the difficulty of separation.

Fig.7.3.2 shows the effect of operating condition on NTU for Contactor I, System 1. It can be seen that NTU increase with both increase in flowrate and oscillation rate. At low oscillation rates the increase with flowrate is more pronounced than at high oscillation rates. This type of behaviour can be explained by two factors which counteract each other. It is known that increase in oscillation rate results in (a) increase in turbulence within the contactor which has the effect of increasing the extent of axial mixing, hence reducing the driving force for mass transfer and (b) decrease in drop
size giving an increase in interfacial area which results in higher mass transfer rates. These factors can be related to the experimental results as follows: At low oscillation rates the degree of turbulence is relatively low thus the effect of axial mixing is small, and the increase in interfacial area due to increase in flowrate results mainly in increased mass transfer rates. As the oscillation rate is increased, the degree of axial mixing and the interfacial area increases with increase in flowrate. However, from the graphs (Figs.7.3.1 and 7.3.2) it would appear that the loss in mass transfer (as shown by decrease in NTU) due to axial mixing increases at a faster rate than the gain in mass transfer due to increased interfacial area. This results in a smaller net increase in mass transfer rate which is reflected in the curvature of the graphs.

Figs.7.3.4 and 7.3.5 show the effect of oscillation rate and phase flowrate respectively on NTU for System 2. The shapes of the curve are similar to those of system 1, however, comparison of the two systems show that much higher NTU values for System 2 at lower oscillation rates. This can be attributed to the lower interfacial tension and the lower density of MIBK which requires much less energy for dispersion. These properties also promote drop oscillation and internal circulation which increases the mass transfer rates.

Figs.7.3.6 and 7.37 show the effect of phase flowrate and oscillation rate respectively on NTU for Contactor II (Small) and System 1. The effect of phase flowrate is similar to those for Contactor I. However, for increasing oscillation rate at a constant flowrate, the NTU increases to a maximum at 100 opm and then decreases with further increase in oscillation rate. It would appear that at oscillation rates above 100 opm the degree of axial mixing is high and counteracts the increase in mass transfer resulting from higher interfacial area, giving an overall decrease in mass transfer rates.
Heights of Transfer Units - HTU.

Figs 7.3.8 to 7.3.16 show the effect of operating conditions on HTU for Contactors I and II and Systems 1 and 2. At constant contactor length, HTU is inversely proportional to NTU, thus the explanation given for NTU would also apply to HTU. However, two variations in operating parameter not discussed above are the effects of flowratio and inlet solute concentration in the feed \( x_i \), on contactor performance.

From 7.3.9 it can be seen that HTU increases with increasing flowratio. This type of behaviour would be expected since, as shown in Fig.7.4.2 and discussed in Chapter 8.4, the decrease in dispersed phase hold-up with flowratio results in lower interfacial area, hence lower mass transfer coefficients. As HTU is inversely proportional to \( K_c a \), an increase in HTU is the result.

Fig.7.3.10 shows that HTU decreases with increasing \( x_i \).

A comparison of the effects of operating conditions on HTU for Contactor I and II is shown in Figs.7.3.15 and 7.3.16. In both cases it can be seen that at the same throughput velocities and oscillation rates, the HTU values for Contactor I are greater than those for Contactor II. This can be attributed to the difference in active volume swept (or effectively mixed) by the baffles. For Contactor I this value is 52% and for Contactor II it is 82%.

Overall Mass Transfer Coefficients, \( K_a \).

Figs 7.3.18 to 7.3.24 show the effect of the various operating conditions on overall mass transfer coefficient for the continuous phase \( K_{acm} \). In all cases (except Fig.7.3.22) it can be seen the \( K_{acm} \) increases with both oscillation rate and phase flowrate.
Sectional Values of NTU, HTU and Ka.

The overall values of NTU as shown in Tables 6A, 7A, 5B and 4C were a summation of sectional values calculated for each 30 cm section of extractor length as shown in Appendix 4.1.

Fig.7.3.24 show the variation of overall mass transfer coefficients along the column length. All the graphs show a trend of decreasing $K_{\text{ac,m}}$ values from the bottom to the top of the contactor. This type of behaviour is expected since the incoming feed phase is either in the form of large drops or streams of liquids, thus having a low interfacial area. As the phase descends through the contactor it is broken up into small droplets by the energy input from the agitator. This creates a higher interfacial area for mass transfer, resulting in higher mass transfer coefficients.

8.2.3 Empirical Correlations for $K_{\text{ca}}$.

Dimensionless correlations for $K_{\text{ca}}$ were developed in Chapter 6.2. Correlations for single system, two systems and for both Contactors are shown in Chapter 7.3.2. Since the present studies were restricted to acetic acid as solute and water as the continuous phase the correlations are limited in use to the present studies and the following cases where:

(a) Water is the solvent in one phase
(b) Direction of solute transfer is from the dispersed phase
(c) The organic phase is the dispersed phase

Fig 7.10 show a plot of the experimental Ka values against the calculated values for System 1 and Contactor I. Good agreement between experimental and calculated values is obtained, and it can be seen from the table analysing the regression equation (section 7.3.2) that the standard deviation of the experimental values about the regression line is 12.4% which is well within the limits of experimental error.
8.3 Axial Mixing.

The purpose for carrying out axial mixing studies was to obtain via the axial mixing parameters - axial mixing coefficient $E_c$, and Peclet Number $P_e$ - the extent of backing mixing in the contactor. This can only be done meaningfully by reference to the continuous phase. Such a study on the dispersed phase would involve mixing the dye with this phase which may affect the physical properties of the system such as interfacial tension, giving inaccurate experimental results. In addition to this, there are practical difficulties and economic factors which would be encountered in such a study as described in Chapter 5.7.

8.3.1 Residence Time and Axial Mixing Parameters.

The experimental method has been described in Chapter 5.7 and the results are given in Tables 13A - 15A. Dye studies were only carried out on the large contactor (Contactor I) since similar studies were carried out by previous workers - Weng (W5) on the 7.5 cm I.D O.B.C and Clare (C7) on the 15 cm O.B.C.

Fig 7.6.1 shows the effect of the single phase flowrate on mean residence time (M.R.T) at different oscillation rates. It is immediately evident that the increasing oscillation rate has very little effect on the M.R.T, for instance at a flowrate of 400 1/hr the M.R.T, varies 1133 to 1039 secs over a baffle range of 60 to 90 opm. However, there is a marked decrease in M.R.T with increasing flowrate and it would appear that at flowrates in excess of 600 1/hr the M.R.T would approach some constant value of about 500 - 600 secs. It can also be seen that the measured values for M.R.T. $\mu$, are quite close to the values calculated from the relationship $\bar{\xi} = V/\nu_c$. However, there is no definite trend with regards to the variation of $(\mu - \bar{\xi})$ as obtained by Weng (W5). He found that this difference $(\mu - \bar{\xi})$ approaches zero as the flowrate is increased. Any minor differences between the results of Weng's study and the present studies are not surprising since the hydrodynamic...
conditions under operation are totally different.

The difference \((\mu - \bar{t})\) can be used as a measure of the departure from both plug flow and mixed flow depending on the nature of the process. In the case of countercurrent continuous flow operations where the flow can be regarded as far removed from mixed flow one would tend to use this difference as a measure of the departure from plug flow. However, this type of analysis for establishing the type of flow is oversimplified and possibly apply to only ideal cases.

The axial mixing parameters obtained from RTD studies are the Peclet Number and axial mixing coefficient \(E_c\). The Peclet Number is obtained directly from the experimental studies by Van de Laan equation for a closed channel - equation 3.57. \(E_c\) values were calculated from the Peclet No. by equation 3.56. These values are plotted in Fig.7.6.3 and 7.6.4.

Fig.7.6.3 shows that at constant oscillation rate and increasing flowrate, the Peclet Number decreases at first - from 200 to 300 l/hr - and then increases quite rapidly with further increase in flowrate. This type of behaviour is somewhat strange and no logical explanation can be given at this point. However, it may be suggested that this can be due to experimental error at the very low flowrates, and it has been decided to ignore these points for the time being until a more extensive study can be carried out. Neglecting these points, it can be seen that the Peclet Number increases with both flowrate and oscillation rate. Taking the lowest flowrate and oscillation rate as the datum, the graphs show that the increase in \(P_e\) with flowrate is more pronounced than the increase with oscillation rate. It is also interesting to note that at the low oscillation rate the increase in \(P_e\) with flowrate is more marked than at the high oscillation rates. The reason for this is at low oscillation rates the degree of turbulence is quite low, giving a small degree of axial mixing, hence small \(E_c\) values. As the oscillation rate is increased \(E_c\) also increases, and since \(E_c\) is inversely proportional to
P_e, the P_e values increase with decrease in oscillation rate, the rate of increase being dependent on the degree of axial mixing. This is discussed in more detail in Section 8.3.3.

8.3.2 Empirical Correlation for Axial Mixing Coefficient.

A correlation for the axial mixing coefficient E_c, was obtained by using the Westerterp and Landsman (W6) type equation as described in Chapter 7.3.3 (Eqn.7.7, 7.8 a&b).

Experimental results were used to find the coefficients of the equation, and the values for E_c calculated from the correlation:

\[
\frac{E_c}{\nu} = 0.185 \left( \frac{u_c D}{\nu} \right) + 1.325 \times 10^{-3} \left( \frac{\theta N}{\nu} \right)^2 D \quad \ldots \quad 7.9a
\]

are compared with experimental values in Fig.7.11.3. The agreement between the experimental and calculated values was quite good considering the limited experimental data used to obtain the correlation. Better agreement can be achieved by doing a more extensive study.

E_c values were also calculated from the Miyauchi and Vermulen type one dimensional diffusion equation (Eqn.3.67) by the method given in Appendix A.3.2. These values are tabulated in Table 7.2 together with the values obtained from the dye studies and the empirical correlation (Eqn.7.9a). The E_c values obtained by the three methods are of the same order of magnitude. The difference in values can possibly due to the end effects associated with the dye studies.

Overall, as the values obtained by the different methods are close, it is reasonable to assume that the values obtained by the dye studies can be used as a measure of the degree of axial mixing within the contactor with confidence.
8.3.3 Effect of Axial Mixing on Mass Transfer - One Dimensional Diffusion Model.

It was not quite clear from the axial mixing studies whether the type of flow through the O.B.C is far removed from plug flow or not. This is due to the poor interpretation of the significance of the values attributed to the dispersion number $E_c/uL$, or Peclet Number $P_e$, for different conditions of mixing as already explained. However, on intuitive grounds with regards to the nature of the extraction system (i.e O.B.C being a countercurrent continuous contactor) one could reasonably assume that the system is far removed from perfectly mixed flow, thus permitting the use of the one dimensional diffusion model (which assumes plug flow with superimposed axial mixing) to study the effect of axial mixing on mass transfer for the continuous phase.

Solution of the Model.

The diffusion model for the continuous phase as developed in Chapter 3.3.2 and repeated below for convenience.

\[
\frac{d^2Y}{dz^2} + P_e \frac{dY}{dz} - N\text{oc} P_e (Y^* - Y) = 0 \quad \ldots 3.71
\]

has been solved by two methods - analytically and numerically. Both of these solutions are given in Appendix A.3.1.

An accurate solution to this equation required (a) fairly accurate boundary conditions (b) the variation of Number of Transfer Units ($N\text{oc}$ or NTU) and Peclet Number ($P_e$) with contactor length to be included in the solution and (c) the use of True Number of Transfer Units. Practically, it is exceedingly difficult to satisfy these requirements exactly, and although a number of solutions to this equation have been proposed in the literature, only one or two workers have applied it to actual experimental data. In solving the equation for the present work exact agreement between experimental and theoretical profiles was not expected.
Theoretical boundary conditions have been developed (D3,M8,W3) from material balances around the end sections of the contactor. Of the boundary conditions the most important are those associated with the active section of the contactor. It is very important to define whether the concentration of the solute in the continuous phase \( Y \) should be related to position \( Z \) just before entry or just after entry in the active contactor section. Most of the previous workers considered the former but if the latter is considered then it has a marked effect on the subsequent concentration profile in the contactor. In particular the boundary conditions at entry being referred to are:

1. Just before entry (see Fig. 8.1), \( Y = Y^0 \)
2. Just after entry (see Fig. 8.1), \( Y = Y_o \)

![Fig. 8.1](image)

These differ inasmuch as for (1) \( dY/dZ = 0 \) and for (2)

\[
\left( \frac{dY}{dZ} \right)_{Z=0} = P_e (Y_o - Y^0)
\]

of these definitions, (1) was not used in the present work. Method (2) was used in two ways:

(a) assuming \( Y^0 = 0 \), then

\[
\left( \frac{dY}{dZ} \right)_{Z=0} = P_e (Y_o)
\]

where \( Y_o \) was measured in practice.

(b) \( (dY/dZ)_{Z=0} \) obtained from a graphical plot of \( Y \) vs \( Z \) by extrapolating to \( Z = 0 \) at \( Z_o \) then drawing a tangent at that point to the curve to get \( (dY/dZ)_{Z=0} \) at \( Z^+ \)

The variation of \( N_{oc} \) along the contactor length was included in the solution by fitting a curves to sectional values given
in Table 10A. However, the $P_e$ values used were overall values obtained from dye injection studies.

Finally, although the solution required true values of $N_{O_C}$, it has been shown by Thomas and Weng (T7) that for the 7.5 cm i.d. O.B.C, the difference between true and measured values are within 10%. It can be assumed with some justification that the behaviour of the multiple baffle O.B.C would not be too far removed from the small O.B.C. Hence, the use of measured $N_{O_C}$ instead of true $N_{O_C}$ for the present studies can be used within reason.

Concentration profiles obtained from the solutions of the one dimensional diffusion equation (Eqn.3.71) are given in Table 7.1 and plotted graphically in Fig.7.8.1 for both types of solutions. Fig.7.8.2 shows the effect of boundary conditions on the theoretical profile. Superimposed on both of these Figures are the experimental profiles.

From Fig.7.8.1 it can be seen that the theoretical concentration profiles resulting from both types of solutions are removed from the experimental profile. The reason for this difference is not immediately obvious and can be due to the contribution of a number of factors such as (a) the variation of $P_e$ (and therefore $E_c$) along the contactor length - this is discussed further in Section 8.3.4 - (b) the use of experimental number of transfer units instead of the true values (c) the use of linear equilibrium relationships for the analytical solution - as described in Appendix A.3.1.1 and (d) other complex hydrodynamic factors which are reflected in the experimental profiles but not in theoretical profiles.

However, it is important to note that the shape of both of the predicted profiles in Fig.7.8.1 are similar to that of the experimental profile. This is quite remarkable and proves that the Miyauchi and Vermulen go a far way in describing the extraction process in the O.B.C. Fig.7.8.2 shows that boundary conditions has a definite effect on the predicted profile
and this can be another factor that contributes to the discrepancy between experimental and theoretical profiles.

Clearly, there are significant limitations associated with the use of the one dimensional diffusion model since it requires accurate boundary conditions, relationships for the variation of true \( N_{qc} \) with \( Z \) for the particular extraction system at different operating conditions, and the use of true equilibrium relationships. Further limitations can arise from the complex hydrodynamic factor that are associated with different systems and types of extractors.

The applicability of the diffusion model - although limited - to describe the extraction process in the O.B.C justifies the assumption made above (on intuitive grounds) that the flow through the contactor can be taken as piston or plug flow with superimposed axial mixing. Hence, the flow pattern in the O.B.C can be regarded as not far removed from plug flow.

This now throws wide open, the whole concept of the use of Peclet Number both quantitatively and qualitatively to measure a system's departure from plug flow or mixed flow. As stated in Chapter 3.2.4, the conditions necessary for plug flow (i.e. as quoted in the literature) is that \( P_e \to \infty \), however, as shown in the present studies Peclet Number of between 7 and 19 were obtained, while the application of the diffusion model as shown in the Miyauchi and Vermulen equation (equation 3.71) suggests that the system is not far removed from plug flow. While the numerical values of 7-19 are far removed from infinity, there may be circumstances when the effects associated with these values are not far removed from those which would occur when infinity was reached. However, this leads to some confusion and uncertainty.

While there is no doubt on the usefulness of the concept of \( P_e \) as a measure of mixing in the intermediate ranges, and to the performance of apparatus in which mixing occurs, but differs from the simple case of flow in pipes, for more obvious cases
it has value, especially in defining plug flow and perfect mixing.

8.3.4 Interpretation of the Diffusion Model.

Interpretation of the diffusion model was attempted by calculating the terms that make up the one dimensional diffusion model in dimensional form - equation 3.67. These values are shown in Table 16A. It can be seen that the values of $E_d^2\frac{d^2y}{dh^2}$ are quite small as compared the values of the other two terms.

Miyauchi and Vermulen (M8) stated that in the absence of axial mixing the second derivative term would be negligible compared with the others thus, the concentration term and first derivative term would then have equal and opposite slopes. Fig.7.6.6 shows the numerical values of these terms for a given operating condition sum to zero at each plane h. The negative term represents the rate at which solute is supplied to the plane by (a) mass transfer from the dispersed phase and (b) axial mixing while the positive term represents the rate at which it is removed by convective flow. It can be seen that the axial mixing contribution is comparatively small and approximately constant along the contactor. Fig.7.6.5 shows the variation of the $E_d^2\frac{d^2y}{dh^2}$ term along the contactor length. It can be seen that the values increases with increase in height from the bottom to a maximum value after which they decrease. This height at this maximum is different for different operating conditions and as discussed earlier this is probably due to variation in the degree of axial mixing along the contactor.
8.4 Maximum Operating Conditions.

The maximum operating conditions for a countercurrent liquid-liquid contactor is established by determining the flooding point. The whole concept of flooding and the point in operation at which flooding is said to occur is somewhat unclear. Normally the optimum operating condition is taken at approximately 75% of the maximum operating conditions.

In the present work, flooding studies were carried out at two inlet solute concentration in the feed phase \( x_i \). The flooding point was taken as the operating conditions where there was a marked increase in hold-up with a small change in phase flow-rate at a given oscillation rate. Fig.7.5.1 shows the variation of hold-up with liquid throughput at \( x_i \approx 0.5 \text{ g/l} \) and \( x_i = 20 \text{ g/l} \). It can be seen that the presence of solute in the CCL\(_4\) has a marked effect on the hold-up in the contactor and on the flooding point. As shown, while the column "approaches" flooding at about 600 - 700 l/hr at low \( x_i \), there is no indication of flooding at 1000 l/hr at high \( x_i \). Both studies were carried out at continuous phase flowrate of 600 l/hr. It is exceeding difficult to give an accurate explanation as why this is so. However, Ismail (I2.) showed experimentally by interferometric studies on interfacial mass transfer of acetic acid between CCL\(_4\) and H\(_2\)O that the interfacial tension of pure CCL\(_4\) decreases quite rapidly with the addition of a very small amount of acetic acid (small meaning 1 g/l), after which there is very little change in the interfacial tension as more acetic acid is added. It would appear that this phenomena is one of the factors that contributes to the hold-up behaviour as mentioned above.

Furthermore, since the prime function of a contactor is to extract solute from one liquid phase by another, the use of non mass transfer systems by a number of workers in establishing maximum operating conditions in contactors is questionable.

Fig.7.5.2 shows the effect of continuous phase flowrate on
hold-up at various dispersed phase flowrate for $x_i \approx 0.5$ g/l. It can be seen that at low dispersed phase flowrate $Q_d$, there was no indication of flooding as the continuous phase flowrate $Q_c$ was taken to the flowmeter limit. As $Q_d$ is increased maximum operating condition is approached and from the Figure it can be seen that at maximum operating conditions $Q_d$ is inversely proportional to $Q_c$. 
8.5 Power Studies.

8.5.1 Total Power.

The total power $P_T$, consumed by the contactor under two phase mixing operation can be divided as follows:

1. The dry power $P_{Dry}$. This is power consumed to overcome friction in the bearings and drive unit, and is measured when the column is empty.

2. Single continuous phase mixing power, $P_{M1}$. This is power consumed in mixing only the single continuous phase, and is obtained by difference.

3. Single dispersed phase mixing power, $P_{M2}$. This is the additional power over $P_{M1}$ consumed under two phase operation, and is also obtained by difference.

These power quantities are related as follows:

$$P_T = P_{Dry} + P_{M1} + P_{M2}$$

Tables 17A - 19A show the effect of operating conditions on the different power quantities. It should be noted that for the single phase mixing power there was no measurable variation in torque (hence power consumed) with phase flowrate. It can also be seen that under constant operating conditions the values of $P_{M2}$ are very small as compared with $P_{M1}$ ($P_{M2} \approx 0.5-1\% P_{M1}$). This is very interesting as it would appear at first sight that the extra power required to create the interfacial area is very small. However, more careful analysis would suggest that most of the energy that is required to disperse the phase is already present in the form of eddies created by just mixing the single continuous phase and the energy needed to disperse the phase is the sum of this extra energy $P_{M2}$, plus part of the energy already consumed in mixing the single
phase. If the "useful power" $P_u$ is defined as power necessary for dispersing the phase or creating interfacial area then,

$$P_u = C \times P_{M1} + P_{M2}$$

To obtain a value for $C$ by experiment is virtually impossible, as a result of this, it has been decided that a discussion with these as separate quantities would be meaningless. They have been combined and referred to as the Total Mixing Power $P_M$.

$$P_M = P_{M1} + P_{M2}$$

Fig. 7.7 shows the effect of oscillation rate on Dry, Mixing and Total power. It can be seen that the Dry Power relationship is almost linear and does not show the same marked change in shape with oscillation rate as the mixing and total power curves which are non-linear. It is evident that the energy consumed in overcoming frictional effects increases in a more uniform manner than the energy consumed in mixing. This can be attributed to the changing hydrodynamic conditions within the contactor which have a pronounced effect on mixing power and very little on the dry power. This type of behaviour is also shown by system 2 (Contactor 1) and Contactor 1 (System 1) as shown in Figs. 7.7.4 and 7.7.6 respectively.

8.5.2 Power per Unit Volume.

The power quantities discussed above are with reference to the specific contactor. In order to put these values in a form that would facilitate comparisons and scale-up, the power per unit volume is used instead of total power. It is normal to use the power per unit active volume, however, for the O.B.C wherein only part of the active volume is swept directly by the baffle, it was decided to use the power per unit active volume $P_{va}$ and power per unit 'swept' volume $P_{vs}$ for comparisons. Obviously a more representative value for the actual system would lie somewhere between these two extremes.
Fig. 7.7.2 shows the variation $P_{va}$ with flowrate at various oscillation rates for Contactor I System 1. As in the case of total mixing power it can be seen that the variation in $P_{va}$ with flowrate is very small as compared to its variation in oscillation rate. This type of behaviour is also shown by System 2 (Contactor I) and Contactor II (System2).

Fig. 7.7.3 shows the effect of dispersed phase hold-up on $P_{va}$. It can be seen that at constant flowrate and varying oscillation rate the power increases with increasing hold-up, however, it would appear that at high oscillation rates very small increases in hold-up results in a marked increase in $P_{va}$, making the contactor uneconomical to operate since it was shown earlier that the dispersed phase has very little effect on mixing power, it would appear that a high oscillation rates (>120 opm) the hydrodynamic conditions within the contactor is very sensitive to small increases in hold-up.

Fig. 7.7.9 shows the comparison of the effects of oscillation rate on $P_{va}$ and $P_{vs}$ for Contactor I and II at constant velocity throughput. It can be seen that for both cases the power consumed by the large contactor is in the region of 40 - 50% of the smaller contactor at the same oscillation.

<table>
<thead>
<tr>
<th>Interfacial Area</th>
<th>$K_a$</th>
<th>$P_{vs}$</th>
<th>$P_{va}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>cm$^2$/cm$^3$</td>
<td>sec$^{-1}$</td>
<td>J/sec m$^3$</td>
<td>J/sec m$^3$</td>
</tr>
<tr>
<td>Contactor I(Large)</td>
<td>0.650</td>
<td>69.6</td>
<td>160.99</td>
</tr>
<tr>
<td>Contactor II(Small)</td>
<td>0.690</td>
<td>123.1</td>
<td>278.77</td>
</tr>
</tbody>
</table>

Table 8.2

Table 8.2 is a comparison of mass transfer rates and power consumed by the two contactors at 80 opm and $u = 0.1572$ cm/sec. It can be seen that although only half the power is consumed by Contactor I at the same operating condition, the mass transfer coefficient for Contactor I is half that for Contactor II. Most interesting is that the interfacial area values for the two contactors are within 10%, while the $K_c$ value for Contactor II is higher than that of Contactor I (Large). This difference
can be attributed to the difference in swept volume. While 82% of the active volume of Contactor II is swept by the baffle, only 52% of the active volume of Contactor I is swept. The higher degree of turbulence within the smaller contactor - due to a smaller proportion of unswept volume - results in a $K_c$ value that is higher than that of the large contactor. However, this is compensated for by the fact that Contactor I is capable of handling much higher throughputs than Contactor II.

8.5.3 Power Correlations.

Dimensionless correlations for the various types of Power Nos. i.e $N_p$, $N_{pva}$ and $N_{pvs}$ were developed in Chapter 6.4. Correlations for a single system (System 1), two systems (Systems 1 and 2) and for both contactors are shown in Chapter 7.3.4.

For Contactor I (Large) the agreement between experimental and calculated power number - can be ascertained from the analysis of the regression equation - for the correlations based on power per unit volume $P_{v1}$ (Eqns. 7.16 & 7.17, 7.20 & 7.21) was much better than the correlations based on total mixing power $P_m$, (Eqns. 7.12 & 7.13) while the reverse was true for Contactor II (Small). This is not surprising since the mixing system for the two contactors are different.

It is interesting to note that for Contactor I, the index of the Reynold number for the correlations based on power per unit active volume $P_{va}$ and power per unit swept volume are the same i.e

$$N_{pva} = 0.244 \left( \frac{1.36}{N_{Re}} \right)_{osc} \quad \ldots 7.16$$

$$N_{pvs} = 0.530 \left( \frac{1.36}{N_{Re}} \right)_{osc} \quad \ldots 7.17$$

the only difference being in the constant term, which makes $N_{pvs}$ a simple multiple of $N_{pva}$. From the present studies it would appear that $N_{pvs} \approx 2 N_{pva}$, however, a more extensive
study would be necessary to confirm this.

Fig. 7.12 shows a comparison of experimental and calculated values for the power correlations given by equations 7.17 and 7.21. The agreement between the experimental and calculated values for both cases was exceptionally good, with a mean deviation of approximately 1.5%.
CHAPTER 9

CONCLUSIONS
The experimental investigation of the performance of the multiple baffle and single baffle contactors can be summarized as follows:

1. Values of NTU, HTU, \( K_c a \), \( K_d a \), \( a \), \( \phi \), \( d_{32} \), the extraction efficiency have been established experimentally for two extraction columns. The values were obtained for a series of flow rates of both phases.

2. Two liquid systems were used namely, CCL\(_4\)/HAC/water and MIBK/HAC/water, transfer taking place in the direction CCL\(_4\) - H\(_2\)O and MIBK - water where CCL\(_4\) and MIBK were the dispersed phase.

3. The two columns consisted of Oscillating Baffle Contactors as described originally by Thomas, BP 52556/1966. The respective diameters of the contactors were 15 cm and 30 cm. The purpose being to study scale-up. In addition the 15 cm contactor had just one oscillating baffle and the 30 cm contactor composed of 3 oscillating baffles of the same size as in the 15 cm contactor with a view to commercial application to even larger sizes.

4. Apart from the application of the well established mass transfer equations, correlations were developed for (a) \( K_c a \) (b) \( d_{32} \) (c) \( E_c \) and (d) power numbers \( N_p \), \( N_{pva} \) and \( N_{pvs} \), relative to operating conditions and the physicochemical properties of the liquids.

5. The significance of interfacial tension relative to all the above studies was examined. With high inlet concentration of HAC in CCL\(_4\) the change in interfacial tension is small until a concentration of about 1 g/l is reached after which further extraction is accompanied by a very rapid increase in interfacial tension. This leads to rapid increased in hold-up and ultimately to flooding. This lead to the conclusion that if the extraction reduces concentration in the disperse phase too low, then the operability of the extractor is affected.
Most previous workers studied flooding where no mass transfer was taking place. The value of such work is questionable as hold-up and flooding characteristics are a function of the transfer of solute occurring across the drop/continuous phase interface.

(6) Extensive studies were made using a dye technique to establish experimental values for the variance and mean residence time under different conditions of operation. The theory was thoroughly explained and important questions were raised on the significance the Dispersion No \((= E_c/uL)\), and the inverse Peclet No \((= uL/E_c)\) relative to the degree of mixing occurring in the contactors for different flow-rates of phases and oscillating rates.

This brings into question the validity of application of the Diffusion Model as a means of defining the degree of mixing occurring as distinct from a single case of flow liquid in a pipe. It was concluded that with Peclet Numbers of 7-19, contrary to definition the apparatus was essentially in a condition of 'near to plug flow'. The difficulties associated with the extreme conditions namely \(Pe \to \infty\) Plug Flow, \(Pe \to 0\) Perfect Mixing are not readily applicable but, this does not necessarily invalidate the use of the diffusion model.

(7) The one-dimensional diffusion equation as developed by Miyauchi and Vermulen (M8), and used in a limited way by Smoot and Babb was investigated theoretically and practically. Some previous workers have attempted to apply this equation analytically, but with limited success only, inasmuch as in the case of Smoot and Babb a constant value for \(N_{oc}\) along the column was assumed.

The prediction of \(N_{oc}\) along column has still not been
solved as it is an exceedingly complex problem which involves not only hydrodynamics but also physico-chemical change for different systems. Different extractors present different hydrodynamic properties.

In the present studies the diffusion equation of Miyauchi and Vermulien (M8) has been solved using a Prime 750 Computer and the method of Runge-Kutta, but unlike other workers, for the first time $N_{oc}$ has been substituted in the main equation by a relationship of the form:

$$N_{oc} = 0.064 e^{-3.5Z}$$

which was established from experimental results in Contactor I (Large). After overcoming problems associated with the inlet continuous phase boundary conditions (for the system CCL$_4$ -HAC -H$_2$O) that had not been dealt with previously by other workers, a solution of the equation was found. A plot of the experimental continuous phase concentration profile against contactor length ($Z$) was made and on this graph was superimposed a theoretical curve ($Y$ vs $Z$) derived by solving the differential diffusion equation. Agreement was extremely good.

The interfacial area and hold-up of dispersed phase are related as has been shown for the Spray Column by many workers.

The method of measuring interfacial area by photography has been described and does not suffer the disadvantage of chemical methods which may affect the interfacial surface tension. Even so, it has limitations but in many respects is still preferable. The drop size distribution was established for different operating conditions to obtain $d_{52}$. The contactor is highly efficient inasmuch as it readily produces drops of a small size approximately 1 mm. Little
or no coalescence occurs. The values of \(d_{32}\) and the hold-up lead to the interfacial area \((a = 6\phi/d_{32})\). A knowledge of 'a' and \(K_c\) gives values of \(K_c\).

The hold-up measured manometrically and by displacement compares in magnitude with spray column as has been shown.

The analysis shows a \(\propto\phi\) and this follows as there is little variation in \(d_{32}\) for both systems used which can be attributed to the action of the oscillating baffles.

Flooding studies were also related to system properties and flooding values obtained by the techniques are necessarily imprecise due to the complex nature of the drop coalescences.

(9) Dimensional Analysis was used to correlate the mass transfer coefficient \(K_c\), Sauter mean drop diameter \(d_{32}\), the eddy diffusion coefficient \(E_c\) and the power.

(10) An important feature of the efficiency of contactors is the backmixing they produce as a consequence of their mode of operation. This is because backmixing acts against the rate of mass transfer by virtue of the reduction in driving force. This has been discussed in detail. The results of the present studies show that for the O.B.C backmixing is comparatively small. Justification for this statement occur in the residence time study where the measured mean residence time is close in value to that for plug flow of the continuous phase in the apparatus.

\[
\text{Mean residence time} \quad \begin{cases} 2200 - 700 & \text{measured} \\ 2200 - 700 & \text{calculated for plug flow} \end{cases}
\]

Further confirmation is in the value of \(E_c \frac{d^2\gamma}{dh^2}\) in the Miyauchi and Vermulen equation. Not only is
this function small in value compared with the Connective and Mass transfer functions but it is also relatively constant throughout the contactor.

These studies involve very complex interactions between two immiscible phases, the hydrodynamic properties of which are also affected by the actual transfer of a third component across the drop interfaces. Necessarily, despite a considerable literature on liquid-liquid extraction as a phenomena, the performance characteristics of contactors must inevitably remain partly empirical. Considerable advances have been made in theoretical analysis but as demonstrated here we have still not reached a confident enough state of expertise to predict how $N_{oc}(NTU)$ varies along a column even the simplest kind. The OBC is more complex but, nevertheless is quite different in operation from say the RDC.

Considerable furtherwork is necessary, but the main value of the present study is the promising demonstration of the use of multiple single oscillating baffles in large columns which overcome the mechanical problems of large torque in large vessels. The fact that the contactor is capable of high throughput, small drop sizes and a minimum of backmixing has been demonstrated.
### Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
<th>Unit(s)</th>
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</thead>
<tbody>
<tr>
<td>a</td>
<td>Specific Interfacial Area; pulse amplitude: $\frac{cm^2}{cm^3}$, $\frac{cm}{cm}$</td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>Cross section area of vessel</td>
<td>$cm^2$</td>
</tr>
<tr>
<td>c</td>
<td>Concentration</td>
<td>$g\cdot mol/l$</td>
</tr>
<tr>
<td>$C(\theta)$</td>
<td>Fraction of tracer in exit stream at any instant $\theta$ after injection</td>
<td></td>
</tr>
<tr>
<td>$C_o$</td>
<td>Orifice Coefficient</td>
<td></td>
</tr>
<tr>
<td>$d_p$</td>
<td>Particle or drop diameter</td>
<td>$cm$</td>
</tr>
<tr>
<td>$d_{52}$</td>
<td>Sauter Mean drop diameter</td>
<td>$cm$</td>
</tr>
<tr>
<td>$d_{52}$</td>
<td>Sauter Mean drop diameter at zero hold-up</td>
<td></td>
</tr>
<tr>
<td>$D_A$</td>
<td>Agitator Diameter</td>
<td>$cm$</td>
</tr>
<tr>
<td>$D, D_V$</td>
<td>Vessel Diameter</td>
<td>$cm$</td>
</tr>
<tr>
<td>$E$</td>
<td>Axial Mixing Coefficient</td>
<td>$cm^2/sec$</td>
</tr>
<tr>
<td>$E(\theta)$</td>
<td>Fraction of tracer in exit stream at any instant $\theta$ after injection</td>
<td></td>
</tr>
<tr>
<td>$f$</td>
<td>Pulse Frequency</td>
<td>$cm$</td>
</tr>
<tr>
<td>$F(\theta)$</td>
<td>Cumulative fraction of tracer in the exit stream at any instant $\theta$ after injection</td>
<td></td>
</tr>
<tr>
<td>$g$</td>
<td>Acceleration due to gravity</td>
<td>$cm/sec^2$</td>
</tr>
<tr>
<td>$h$</td>
<td>Distance between two locations along the column length</td>
<td>$cm$</td>
</tr>
<tr>
<td>$h_m$</td>
<td>Manometer reading</td>
<td>$cm$</td>
</tr>
<tr>
<td>$\Delta h$</td>
<td>Difference between levels connecting manometer levels</td>
<td>$cm$</td>
</tr>
<tr>
<td>$H$</td>
<td>Total Active height or length of column; hold back</td>
<td>$cm$</td>
</tr>
<tr>
<td>HTU</td>
<td>Height of transfer unit</td>
<td>$M, cm$</td>
</tr>
<tr>
<td>$I(\theta)$</td>
<td>Fraction of tracer in the vessel at any instant $\theta$ after injection</td>
<td></td>
</tr>
</tbody>
</table>
k  Film mass transfer coefficient  cm/sec
K₁HB  Film mass transfer coefficient from Handlos and Baron Eqn.2.15 cm/sec
K  Overall mass transfer coefficient  cm/sec
L  Compartment height  cm
m  Equilibrium distribution coefficient
n  Mass flux across interface  gmol/cm² sec
Nₐ  Mass transfer rate dn/dh  gmol/cm³ sec
N  Oscillation rate, rotor speed  ose/sec, rev/sec
Nₐ  Number of agitators or Baffles
NTU, N₀  Number of transfer units
P₁Dry  Dry Power  Joules/sec
P₂T  Total Power = P₁Dry + P₂M  Joules/sec
P₂M  Total Mixing Power = P₂M₁ + P₂M₂  Joules/sec
P₂M₁  Single continuous phase mixing power  Joules/sec
P₂M₂  Additional Power of dispersing second phase
P₂va  Power per unit active volume  Joules/sec cm³
P₂vs  Power per unit swept volume  Joules/sec cm³
Q  Phase volumetric flowrate  l/hr
r  Radius of Particle  cm
R  Flowratio Qc/Qd, uc/ud
s  Stator ring opening  cm
\bar{t}  Mean residence time V/v  sec
t  Time  sec
u, U  Phase velocity (Superficial)  cm/sec
\bar{U}ₙ  Mean velocity at substantially zero hold-up, Eqn.2.29  cm/sec
U₀  Characteristic velocity of droplets in column  cm/sec
**Us** Relative slip velocity cm/sec

**Ut** Drop terminal velocity cm/sec

**v** Volumetric flowrate cm³/sec

**V** Active volume of vessel or column cm³

**VA** Active volume of O.B.C cm³

**VS** Swept volume of O.B.C cm³

**x** Solute concentration in dispersed phase g/mol/cm³, g/l

**X** Dimensionless concentration for dispersed phase

**y** Solute concentration in continuous phase g/mol/cm³, g/l

**Y** Dimensionless concentration for continuous phase

**z** Dimensionless height w.r.t to two positions along column length h/H

**Z** Dimensionless height H/H = 1

**Zc** Coalescence coefficient Eqn.2.26

**Greek letters**

**ε** Fractional free space of packing or plate

**ηE** Extraction efficiency

**θ** Dimensionless time, amplitude of oscillation rad

**μ** Dynamic viscosity, Mean Residence Time g/cm sec, sec

**μm** Mean viscosity Eqn.3.88a g/cm sec

**ν** Kinematic viscosity μ/ρ cm²/sec

**ρ** Density g/cm³

**ρm** Mean density Eqn.3.88b g/cm³
\( \sigma \) \hspace{1cm} \text{Interfacial tension, variance} \hspace{1cm} g/sec^2

\( \phi \) \hspace{1cm} \text{Dispersed phase hold-up} \hspace{1cm} cm^3/cm^3

\( \psi \) \hspace{1cm} \text{Power function Eqn.2.30b}

**Dimensionless Groups**

\[ N_{Fr} = \frac{u^2}{g} \text{Froude Number for flow} \]

\[ N_{Fr} = (\Theta N)^2 D/g \text{Froude Number for Oscillation} \]

\[ N_{Fr} = N^2 D/g \text{Froude Number for Rotation} \]

\[ N_p = \frac{P}{\rho N^3 D^5} \text{Power Number for Rotation} \]

\[ N_p = \frac{P}{\rho (\Theta N)^3 D^5} \text{Power Number for Oscillation} \]

\[ N_{PVA} = \frac{P_{VA}}{\rho (\Theta N)^3 D^5} \text{Power Number for Oscillation based on Active volume of O.B.C} \]

\[ N_{PVS} = \frac{P_{VS}}{\rho (\Theta N)^3 D^5} \text{Power Number for Oscillation based on swept volume of O.B.C} \]

\[ N_{Pe} = \frac{uL}{E} \text{Peclet Number} \]

\[ N_{oh} = \frac{\mu_c}{\rho_c D_A} \sigma \text{Ohesorge Number} \]

\[ N_{Re} = \frac{uD\rho}{\mu} \text{Reynolds Number for flow} \]

\[ N_{Re} = (\Theta N)D^2 \rho/\mu \text{Reynolds Number for Oscillation} \]

\[ N_{Re} = ND^2 \rho/\mu \text{Reynolds Number for Rotation} \]

\[ N_{Sc} = \frac{\mu \theta}{\sigma} \text{Schmidt Number} \]

\[ N_{Sh} = \frac{kd}{\sigma} \text{Sherwood Number} \]

\[ N_{We} = \frac{u^2 L\rho}{\sigma} \text{Weber Number for flow} \]

\[ N_{We} = (\Theta N)^2 D^3 \rho/\sigma \text{Weber Number for Oscillation} \]

\[ N_{We} = N^2 D^3 \rho/\sigma \text{Weber Number for Rotation} \]

**Subscripts**

\( \text{App.m} \) \hspace{1cm} \text{Apparent measured} \\

\( B \) \hspace{1cm} \text{Bottom}
c  Continuous phase

D  Dispersed phase

i  Interface

m  Measured

p  Piston

k  Raffinate

S  Solvent

t  True

T  Top

**Superscripts.**

*  Equilibrium Conditions
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<tr>
<td>Treybal, R.E.</td>
<td>U.S. Patent</td>
<td>3,325,255</td>
</tr>
<tr>
<td>Venkataramau, G. and Laddha, G.S.</td>
<td>A.I.Ch.E.Jl.</td>
<td>6, 335 (1960)</td>
</tr>
</tbody>
</table>
W5  Weng, P.J.K., Ph.D. Thesis, Univ. of Surrey (1968)
APPENDIX 1

EXPERIMENTAL DATA
### A.1.1 Physical Properties or the Chemicals Used.

#### Table A.1.1.

<table>
<thead>
<tr>
<th>Properties at 20°C</th>
<th>Carbon Tetra-Chloride</th>
<th>Methyl Isobutyl Ketone</th>
<th>Water</th>
<th>Acetic Acid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (Kg/m$^3$)</td>
<td>1594</td>
<td>801</td>
<td>997.0</td>
<td>1052</td>
</tr>
<tr>
<td>Viscosity (N.s/m$^2$) x10$^3$</td>
<td>1.030</td>
<td>0.582</td>
<td>1.000</td>
<td>1.229</td>
</tr>
<tr>
<td>Surface Tension (mN/m)</td>
<td>30.0</td>
<td>23.9</td>
<td>72.58</td>
<td>27.5</td>
</tr>
<tr>
<td>Interfacial Tension* (mN/m)</td>
<td>40.4</td>
<td>10.9</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Diffusivity** (m$^2$/s) x 10$^9$</td>
<td>1.460</td>
<td>1.440</td>
<td>1.040</td>
<td>-</td>
</tr>
<tr>
<td>R.I</td>
<td>1.460</td>
<td>1.396</td>
<td>1.333</td>
<td>1.371</td>
</tr>
<tr>
<td>% Purity</td>
<td>99.0</td>
<td>99.0</td>
<td>-</td>
<td>99.5</td>
</tr>
<tr>
<td>Mol.wt.(Kg)</td>
<td>0.154</td>
<td>0.100</td>
<td>0.018</td>
<td>0.060</td>
</tr>
</tbody>
</table>

* Denotes Interfacial Tension with water

** Denotes Diffusivity of Acetic Acid in Solvent

1 Kg/m$^3$ = 1000 g/cm$^3$
1 N.s/m$^2$ = 1 x 10$^{-3}$ cP
1 mN/m = 1 dyne/cm
A.1.2 Equilibrium Data.

Equilibrium data for the two three component systems used (i.e. CCl₄ - HAC - H₂O and MIBK - HAC - H₂O) was obtained experimentally. Varying amounts of acetic acid were added to mixtures consisting of equal known volumes of pure organic and water for the system under investigation. The three component mixtures were then brought to a state of equilibrium by agitating the mixtures in a constant temperature bath over a period of time. The two phase were then separated and samples were analysed for solute content by the methods described in Chapter 5.4.2.

The results for both systems are given and compared with equilibrium data published by previous workers in Tables A.1.2 and A.1.3.

Table A.1.2 Equilibrium Distribution of Acetic Acit between Water and Carbon Tetrachloride.

<table>
<thead>
<tr>
<th>Present Experimental Results</th>
<th>Data from J.B.Lewis*</th>
</tr>
</thead>
<tbody>
<tr>
<td>x</td>
<td>y</td>
</tr>
<tr>
<td>0.18</td>
<td>21.4</td>
</tr>
<tr>
<td>0.44</td>
<td>40.7</td>
</tr>
<tr>
<td>0.93</td>
<td>58.0</td>
</tr>
<tr>
<td>1.45</td>
<td>77.5</td>
</tr>
<tr>
<td>2.23</td>
<td>98.0</td>
</tr>
<tr>
<td>2.89</td>
<td>111.0</td>
</tr>
<tr>
<td>3.55</td>
<td>127.4</td>
</tr>
<tr>
<td>4.54</td>
<td>144.2</td>
</tr>
<tr>
<td>5.43</td>
<td>158.8</td>
</tr>
<tr>
<td>6.65</td>
<td>179.2</td>
</tr>
<tr>
<td>11.20</td>
<td>237.5</td>
</tr>
<tr>
<td>15.91</td>
<td>309.0</td>
</tr>
<tr>
<td>20.71</td>
<td>359.0</td>
</tr>
</tbody>
</table>

Table A.1.3 Equilibrium Distribution of Acetic Acid between Water and Methyl Isobutyl Ketone.

<table>
<thead>
<tr>
<th>Present Experimental Results</th>
<th>Data from Efce. Publn.**</th>
</tr>
</thead>
<tbody>
<tr>
<td>x</td>
<td>y</td>
</tr>
<tr>
<td>1.50</td>
<td>2.75</td>
</tr>
<tr>
<td>3.72</td>
<td>7.50</td>
</tr>
<tr>
<td>6.70</td>
<td>12.50</td>
</tr>
<tr>
<td>10.08</td>
<td>20.82</td>
</tr>
<tr>
<td>12.50</td>
<td>23.50</td>
</tr>
<tr>
<td>22.56</td>
<td>42.96</td>
</tr>
<tr>
<td>35.64</td>
<td>64.08</td>
</tr>
</tbody>
</table>

** 'Recommended Systems for Liquid Extraction Studies'
Ed. T. Misek.
Published on behalf of European Federation of Chemical Engineers by the Institution of Chemical Engineers, England.
Treatment of Experimental Data.

CCL$_4$ - HAC - H$_2$O System.

An empirical relationship between $x$ and $y$ was developed by regression analysis. (See Chapter 3.1.1). The relationship between $x$ and $y$ was given by:

$$y^* = 60.92 x^{0.579} \quad ... A.1.1$$

Results calculated by this equation are compared with the experimental results and the results of J.B. Lewis in Fig.A.1.1. The mean deviation of $y^*$ about the regression line was 3.33%.

MIBK - HAC - H$_2$O System.

As for the CCL$_4$ - HAC - H$_2$O system an empirical relationship was developed by regression analysis. In this case the relationship was linear and was given by:

$$y^* = 1.859 x \quad ... A.1.2$$

Results calculated by this equation are compared with the experimental result and the results of the EFCE Publication in Fig.A.1.2. The mean deviation of $y^*$ about the regression line was 4.3%.
Acetic acid in water phase, y (g/l)

Acetic acid in CCL$_4$ phase, x (g/l)

FIG. A.1.1 Equilibrium distribution of acetic acid between water and carbon tetrachloride
Acetic acid in MIBK Phase, x (g/l)

KEY

- Data calculated from Eq. A.1.2
- ▲ Present experimental data
- • Data from EFLE Publn.

FIG. A.1.2 Equilibrium distribution of acetic acid between water and methyl isobutyl ketone
Table A.1.4 Relationship between Solute Concentration and Interfacial Tension at 20°C.

(a) Carbon Tetrachloride - Water - Acetic Acid

<table>
<thead>
<tr>
<th>$y^*$ g/1</th>
<th>$x^*$ g/1</th>
<th>$\sigma$ mN/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>40.4</td>
</tr>
<tr>
<td>10</td>
<td>0.050</td>
<td>29.1</td>
</tr>
<tr>
<td>20</td>
<td>0.168</td>
<td>26.4</td>
</tr>
<tr>
<td>40</td>
<td>0.567</td>
<td>24.2</td>
</tr>
<tr>
<td>59</td>
<td>1.154</td>
<td>22.5</td>
</tr>
<tr>
<td>78</td>
<td>1.829</td>
<td>21.2</td>
</tr>
<tr>
<td>97</td>
<td>2.681</td>
<td>20.7</td>
</tr>
</tbody>
</table>

Data From: Ismail (12)

(b) Methyl Isobutyl Ketone - Water - Acetic Acid

<table>
<thead>
<tr>
<th>$y^*$ g/1</th>
<th>$x^*$ g/1</th>
<th>$\sigma$ mN/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>10.7</td>
</tr>
<tr>
<td>5.52</td>
<td>2.81</td>
<td>9.9</td>
</tr>
<tr>
<td>27.69</td>
<td>14.27</td>
<td>8.7</td>
</tr>
<tr>
<td>40.50</td>
<td>20.54</td>
<td>8.3</td>
</tr>
<tr>
<td>53.41</td>
<td>28.28</td>
<td>7.5</td>
</tr>
<tr>
<td>81.50</td>
<td>44.29</td>
<td>6.5</td>
</tr>
<tr>
<td>103.54</td>
<td>57.52</td>
<td>5.8</td>
</tr>
</tbody>
</table>

Data From: Recommended Systems for Liquid Extraction Studies
(a) Water-acetic acid calibration chart 
(26.5°C)

(b) CCL₄-Acetic acid calibration chart 
(26.5°C)

FIG. A1.3 Refractive index/solute concentration calibration charts
A.1.5 Determination of the Mean Position Along Column Length for Carrying out Drop Size Studies by Photography

The method used to determine the mean position along the column length for carrying out drop size studies by photography is discussed in Chapter 5.3.1.

To establish this mean position, photographs of drops were taken at four position along the column length for a selected number of operating conditions. The photographs for each position were analysed by counting and sizing the droplets by the method given in 5.3.2 and the $d_{32}^2$ (for each position) was calculated from the relationship:

$$d_{32}^{sect} = \frac{\sum N_i d_i^3}{\sum N_i d_i}$$  \hspace{1cm} A.3.3

where $d_{32}^{sect}$ is the Sauter mean drop diameter at each section.

The overall $d_{32}$ for a given set of operating conditions was then determined from the relationship:

$$d_{32}^{overall} = \frac{(\sum N) d_{32}^{section}}{\sum N}$$  \hspace{1cm} A.3.4

By plotting graphs of $d_{32}$ VS column length, the positions corresponding to the overall $d_{32}$ were obtained. A mean position for carrying out the photographic studies was then determined as shown below for Contactor I (Large). The same procedure was used for each system and contactor.
Table A.1.5 Variation of $d_{32}$ along Column Length
Contactor I, CCL$_4$ - HAC - H$_2$O
$Q_c = Q_d = 400$ l/hr

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>Position</th>
<th>Distance from Bot (cm)</th>
<th>$d_{32}$ (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>1(Bot)</td>
<td>30.0</td>
<td>0.110</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>50.4</td>
<td>0.123</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>76.0</td>
<td>0.148</td>
</tr>
<tr>
<td></td>
<td>4(Top)</td>
<td>101.4</td>
<td>0.159</td>
</tr>
<tr>
<td>80</td>
<td>1(Bot)</td>
<td>30.0</td>
<td>0.125</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>44.3</td>
<td>0.141</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>72.7</td>
<td>0.149</td>
</tr>
<tr>
<td></td>
<td>4(Top)</td>
<td>102.2</td>
<td>0.150</td>
</tr>
<tr>
<td>120</td>
<td>1(Bot)</td>
<td>30.0</td>
<td>0.117</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>44.3</td>
<td>0.134</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>72.7</td>
<td>0.144</td>
</tr>
<tr>
<td></td>
<td>4(Top)</td>
<td>102.2</td>
<td>0.143</td>
</tr>
</tbody>
</table>

Table A.1.5 show the experimental data for different oscillation rates which are plotted in Fig.A.1.4.

From the data it was found that for:

- 60 opm $d_{32}$ overall = 0.124 cm
- 80 opm $d_{32}$ overall = 0.137 cm
- 120 opm $d_{32}$ overall = 0.134 cm

and from Fig.A.1.4 the position along the column corresponding to these $d_{32}$ values for 60 and 80 and 120 opm are 43, 48 and 49 cm. The mean position used for taking the photographs was the arithmetic mean of these values which 47 cm from the
bottom of the contactor. Similarly the mean position for the MIBK - HAC - H$_2$O system (Contactor I) and Contactor II (CCL$_4$ - HAC - H$_2$O) was found to 72 and 45 cm respectively from the bottom of the contactor.
FIG. A.1.4 Variation of $d_{32}$ along column length
Contactor I  CCL$_4$-HAC-H$_2$O system
$Q_c = Q_d = 400$ l/hr
APPENDIX 2

METHODS USED FOR DETERMINING DISPERSED PHASE HOLD-UP
A.2.1 Manometric Method.

The volume fraction of dispersed phase held up in the contactor was determined from manometric measurements by equations 3.82 and 3.83. The theory behind the derivation of this equation is based on the Bernoulli equation as shown below.

From Fig.A.2.1, for the continuous phase, the total energy at $h_2$ is less than that at section $h_1$, by the friction loss $f_c$ i.e:

\[
\frac{u_1^2}{2g} + \frac{p_1}{\rho_c} + h_1 - f_c = \frac{u_2^2}{2g} + \frac{p_2}{\rho_c} + h_2 \quad ... A.2.1
\]

Assuming equal velocity heads in the same column, and negligible friction loss due to wall effects, equation A.2.1 reduces to:

\[
p_1 - p_2 = (h_2 - h_1)\rho_c g = \Delta h\rho_c g \quad ... A.2.2
\]

Similarly for the dispersed phase:

\[
p_1 - p_2 = (h_2 - h_1)\rho_d g = \Delta h\rho_d g \quad ... A.2.3
\]

Multiplying equations A2.2 and A2.3 by $(1-\phi)$ and $\phi$ respectively where $\phi$ is the fractional hold-up of the dispersed phase.

\[
p_1 - p_2 \phi - p_2 + p_2 \phi = \Delta h\rho_c g - \Delta h\rho_c \phi g \quad ... A.2.4
\]

and $p_1 \phi - p_2 \phi = \Delta h\rho_d \phi g \quad ... A.2.5$

Adding equations A2.4 and A2.5:

\[
p_1 - p_2 = \Delta h \rho_c (1-\phi)g + \rho_d \phi g \quad ... A.2.6
\]

Rearranging A2.6

\[
\phi = \frac{(p_1 - p_2) - \Delta h\rho_c g}{\Delta h(\rho_d - \rho_c) g} \quad ... A.2.7
\]
Fig.A.2.1 Diagram showing Manometer connections to Contactor
If the pressure difference between \( h_1 \) and \( h_2 \) is to be measured with a manometer, then

\[
p_1 - p_2 = \Delta h \rho_c g + h_m (\rho_m - \rho_c)g \quad \ldots \quad A.2.8
\]

where \( \rho_m \) is the density of the liquid used in the manometer and \( \rho_c \) is the density of the continuous phase. Substituting Equation A2.8 into A2.7 then

\[
\phi = \frac{\Delta h \rho_c + h_m (\rho_m - \rho_c)}{(\rho_d - \rho_c) \Delta h} - \Delta h \rho_c \quad \ldots \quad A.2.9
\]

which on simplifying gives

\[
\phi = \frac{h_m (\rho_m - \rho_c)}{\Delta h (\rho_d - \rho_c)} \quad \ldots \quad A.2.10
\]

If the dispersed phase is used as the manometer liquid, then \( \rho_m = \rho_d \) and equation A2.10 reduces to:

\[
\phi = \frac{h_m}{\Delta h} \quad \ldots \quad A.2.11
\]

where \( h_m \) is the manometer reading and \( \Delta h \) is the difference of levels connecting with both arms of the manometer.

Throughout the present studies carbon tetrachloride (CCL\textsubscript{4}) was used as the heavy manometer liquid and water as the light liquid. Thus, for the CCL\textsubscript{4} - HAC - H\textsubscript{2}O system equation A2.11 was applicable and for the MIBK - HAC - H\textsubscript{2}O system equation A2.10 was applicable.

A.2.2 Displacement Method.

This method was used purely as a means of checking the authenticity of the values obtained by the manometric method. Inlet and outlet flow lines close to the contactor were shut off almost instantaneously (within 3 secs). The dispersed phase in the contactor was allowed to settle and the height
of continuous phase displaced by the settled liquid $h_s$, was measured.

This height $h_s$ was made up of:

(a) the equivalent height $h_1$, of the volume fraction of the heavy phase hold-up in the active contactor.

(b) the equivalent height $h_2$, of the heavy phase that flowed in after the inlet valve was shut off.

(c) the equivalent height $h_3$, of heavy phase that were present in top and bottom headers of the contactor at the instant when the valves were closed.

therefore:

$$h_s = h_1 + h_2 + h_3 \quad \ldots \quad A.2.12$$

where

$$h_1 \approx h_m$$

By making the following assumptions, the value of $h_1$ was calculated

(1) The volume of liquid $V_2$ that entered the contactor after the valves were switched off is the volume of liquid present in the flow line between the shut off valve and the point of entry in the top header. This was calculated from the dimensions of this section of the flow lines and the valve, $h_1$ was determined by dividing this volume $V_2$ by the cross-sectional area of the bottom header (which is the same as for the active contactor).

(2) The volume of liquid in the bottom header $V_3$, is the volume fraction $\phi_F$ (i.e the manometric value) in the active contactor multiplied by the total free volume in the bottom header (i.e excluding the volume
occupied by the coalescing liquid pool). The volume of liquid in the top header was negligible. \( h_2 \) was obtained by dividing \( V_3 \) by the cross-sectional area of the bottom header.

thus,

\[
h_1 = h_s - (h_2 + h_3) \quad \cdots \quad A.2.13
\]

In all case studied the values of \( h_1 \) was \( \pm 10\% \) that of \( h_m \) for the same experimental conditions.
APPENDIX 3

THEORETICAL ANALYSIS
A.3.1 Solutions to the One Dimensional Diffusion Equation

A.3.1.1 Analytical Solution

The one-dimensional diffusion equation derived in Chapter 3.3.2 based on the work of Miayauchi and Vermulen (M8) is given in dimensional form for continuous phase mass transfer as follows:

\[ E_c \frac{d^2 y}{dh^2} + U_c \frac{dy}{dh} - K_c a (y^* - y) = 0 \]  \hspace{1cm} \ldots \ldots (3.67)

Defining the following dimensionless parameter (see Fig. 3.7b):

\[ Y = \frac{y}{x_F} \]
\[ X_R = \frac{x_R}{x_F} \]
\[ Z = \frac{h}{H} \]
\[ N_{oc} = \frac{K_{oc} a H}{U_c} \]
\[ Pec = \frac{U_c H}{E_c} \]

Putting equation (3.67) in dimensionless form:

\[ \frac{d^2 Y}{dZ^2} + Pec \frac{dY}{dZ} - N_{oc} Pec (Y^* - Y) = 0 \]  \hspace{1cm} \ldots \ldots (A.3.1)

For the CCL_4-HAC-H_2O system, the equilibrium relationship is not linear and takes the form \( y^* = ax^b \). It is exceedingly difficult to carry out an analytical solution with this type of relationship. To overcome this difficulty, the equilibrium curve was divided into sections over the range of concentration used. The sections were chosen so that they can be represented by an equation of the form \( y^* = mx + c \). Assuming that the concentration profile resulting from the solution of A.3.1 is not far removed from the experimental profile, then, equation A.3.1 can be solved by using a linear equilibrium relationship as above with the experimental profile serving as a guide for selecting the linear equilibrium relationship that was applicable over the section, when calculating the theoretical profiles. Although it is expected that this type of solution may result in profiles that are far removed from the experimental ones, it is useful in that it may give an idea of the limitations when solving the differential equation analytically for non
linear equilibrium relationships.

The equations of the form \( y^* = mx + c \) over the concentration range studied are:

\[
\begin{align*}
0 \leq x &< 5 \text{ g/l} \quad y^* = 27.67x \quad \ldots \quad (A.3.2a) \\
5 \leq x &< 10 \text{ g/l} \quad y^* = 13.41x + 71.38 \quad \ldots \quad (A.3.2b) \\
10 \leq x &< 15 \text{ g/l} \quad y^* = 10.68x + 98.6 \quad \ldots \quad (A.3.2c) \\
15 \leq x &< 20 \text{ g/l} \quad y^* = 9.22x + 120.45 \quad \ldots \quad (A.3.2d) \\
20 \leq x &< 25 \text{ g/l} \quad y^* = 8.27x + 137.57 \quad \ldots \quad (A.3.2e) \\
25 \leq x &< 30 \text{ g/l} \quad y^* = 7.58x + 156.72 \quad \ldots \quad (A.3.2f)
\end{align*}
\]

Values obtained from the equations above are compared with the values from the non-linear equilibrium relationship given in Appendix A.1.

<table>
<thead>
<tr>
<th>x</th>
<th>( y ) Eqn.A.3.2a</th>
<th>( y ) Eqn.A.3.2f</th>
<th>( y ) Eqn.A.1</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>138.43</td>
<td>138.43</td>
<td>138.37</td>
</tr>
<tr>
<td>10</td>
<td>205.40</td>
<td>205.40</td>
<td>205.42</td>
</tr>
<tr>
<td>15</td>
<td>258.75</td>
<td>258.75</td>
<td>258.83</td>
</tr>
<tr>
<td>20</td>
<td>302.97</td>
<td>302.97</td>
<td>304.95</td>
</tr>
<tr>
<td>25</td>
<td>346.22</td>
<td>346.22</td>
<td>346.31</td>
</tr>
<tr>
<td>30</td>
<td>384.12</td>
<td>384.12</td>
<td>384.24</td>
</tr>
</tbody>
</table>

Using \( y^* = mx + c \) :

From equation (3.10) (Chapter 10)

\[
x = y + x_R
\]

\[
:\quad y^* = m(y + x_R) + c
\]

\ldots \quad (A.3.2d)

In dimensionless form

\[
Y^* = m(Y + X_R) + C
\]

\ldots \quad (A.3.3e)

where

\[
Y^* = y^*/x_F \quad C = c/x_F \\
Y = y/x_F \quad X_R = x_R/x_F
\]
Substituting for \( Y^* \) in equation (A.3.1);

\[
\frac{dY}{dz} + Pe \frac{dY}{dz} - N_{oc} Pe \left[ \left( m (Y + X_R) + C \right) - Y \right] \quad \ldots \ldots (A.3.4)
\]

Simplifying and rearranging;

\[
\frac{d^2Y}{dz^2} + Pe \frac{dY}{dz} - N_{oc} Pe Y (m - 1) = N_{oc} Pe (m X_R + C) \quad (A.3.5)
\]

The complete solution of equation (A.3.5) is

\[ Y = Y_c + Y_p \]

where

\( Y_c \) = complimentary function, C.F.

\( Y_p \) = particular integral, P.I.

(a) Complimentary Function

\[
\frac{d^2Y}{dz^2} + Pe \frac{dY}{dz} - N_{oc} Pe Y(m - 1) = 0 \quad \ldots \ldots (A.3.6)
\]

The auxiliary quadratic equation is;

\[ q^2 + Pe q - N_{oc} Pe Y(m - 1) = 0 \quad \ldots \ldots (A.3.7) \]

and the roots are;

\[
q_1 = \frac{-Pe + r}{2} \quad \ldots \ldots (A.3.8)
\]

\[
q_2 = \frac{-Pe - r}{2} \quad \ldots \ldots (A.3.9)
\]

where \( r = \sqrt{Pe^2 + 4 N_{oc} Pe (m - 1)} \) \ldots \ldots (A.3.10)

Thus the complimentary function is

\[ Y_c = Ae^{q_1 Z} + Be^{q_2 Z} \]

where \( A \) and \( B \) are constants and \( m \) and \( m \) are given in equations (A.3.8, 3.9 and 3.10).
(b) Particular Integral

A the right hand side of equation (A.3.5) is a constant, then let the particular integral be

\[ Y_p = K \quad \ldots \quad (A.3.11) \]

\[ \frac{dY}{dZ} = 0 \]

and

\[ \frac{d^2Y}{dZ^2} = 0 \]

Substituting these into equation (A.3.5)

\[ 0 + 0 - KN_0 = N_0 Pe (m - 1) = N_0 Pe (m X_R + C) \]

giving;

\[ k = \frac{-(m X_R + C)}{(m - 1)} \quad \ldots \quad (A.3.12) \]

The complete solution to equation (5) is

\[ Y = Y_c + Y_p = Ae^{q_1Z} + Be^{q_2Z} - \left( \frac{(m X_R + C)}{m - 1} \right) \quad \ldots \quad (A.3.13) \]

where A and B are constant and q_1 and q_2 are given in equations (A.3.8, A.3.9 and A.3.10).

Boundary Conditions

Apply Miyauchi's (M8) boundary conditions for solute extraction (Fig. A.3.1).

(i) At \( Z = 0 \)

\[ Y^0 = Y_o - \frac{1}{Pe} \left( \frac{dY}{dZ} \right) \quad \ldots \quad (A.3.14) \]

\[ \frac{1}{Pe} \frac{dY}{dZ} = Y_o - Y^0 \]

\[ \Rightarrow \frac{dY}{dZ} = Pe \left[ Y_o - Y^0 \right] \quad \ldots \quad (A.3.15) \]
Fig.A.3.1 Boundary Condition Diagram
For CCL₄ - HAC - H₂O System
At \( Z = 0 \)

\[ Y = Y_0 \]  

\[ \ldots \ldots \text{(A.3.16)} \]

The general solution of the one dimensional diffusion equation is;

\[ Y = A e^{q_1 Z} + B e^{q_2 Z} - \frac{m X_R + C}{m - 1} \]  

\[ \ldots \ldots \text{(A.3.13)} \]

and

\[ \frac{dY}{dZ} = q_1 A e^{q_1 Z} + q_2 B e^{q_2 Z} \]  

\[ \ldots \ldots \text{(A.3.17)} \]

Applying the boundary conditions given by equations (A.3.15 and A.3.16) at \( Z = 0 \);

\[ A + B - \frac{m X_R + C}{m - 1} = Y_0 \]  

\[ \ldots \ldots \text{(A.3.18)} \]

and

\[ q_1 A + q_2 B = Pe (Y_0 - Y^0) \]

Putting \( Y_m = (Y_0 - Y^0) \)

Then

\[ q_1 A + q_2 B = Pe Y_m \]  

\[ \ldots \ldots \text{(A.3.19)} \]

Solving (A.3.18 and A.3.19) for \( A \) and \( B \) gives

\[ A = \frac{Pe Y_m}{q_1} - \frac{q_2}{q_1} \left[ \frac{q_1 Y_0 - Pe Y_m}{(q_1 - q_2)} + \frac{q (m X_R + C)}{(q_1 - q_2)(m - D)} \right] \]  

\[ \ldots \ldots \text{(A.3.20)} \]

and

\[ B = \frac{q_1 Y_0 - Pe Y_m}{q_1 - q_2} + \frac{q_1 (m X_R + C)}{(q_1 - q_2)(m - 1)} \]  

\[ \ldots \ldots \text{(A.3.21)} \]

Substituting for \( A \) and \( B \) in (A.3.13), and simplifying the general solution to one dimensional diffusion equation is;

\[ Y = \left[ \frac{Pe Y_m e^{q_1 Z}}{q_1} \right] + \left[ e^{q_2 Z} + \frac{q_2 e^{q_1 Z}}{q_1} \right] \left[ \frac{q_1 Y_0 - Pe Y_m}{q_1 - q_2} \right] + \left[ \frac{q_1 (m X_R + C)}{(q_1 - q_2)(m - 1)} \right] - \frac{m X_R + C}{m - 1} \]  

\[ \ldots \ldots \text{(A.3.22)} \]

and
\[
\frac{dY}{dZ} = \left[ Pe Y_m e^{q_1 Z} \right] + \left[ q_2 e^{q_2 Z} - q_2 e^{q_1 Z} \right] \left[ \left( \frac{q_1 Y_0 - Pe Y_m}{q_1 - q_2} \right) + \left( \frac{q_1 (m X_R + C)}{(q_1 - q_2)(m-1)} \right) \right] \]

where

\[ q_1 = -\frac{Pe + r}{2} \]
\[ q_2 = -\frac{Pe - r}{2} \]
\[ r = \sqrt{Pe^2 + 4 N_{oc} Pe (m - 1)} \]
\[ Y_m = (Y_0 - Y^0) \] as defined in Fig. A.3.1

A.3.1.2 Numerical Solution - The Runge-Kutta Methods

The one dimensional diffusion equation as given in dimensionless form by equation (A.3.1) is;

\[
\frac{dY}{dZ} + Pe \frac{dY}{dZ} - N_{oc} Pe_c (Y^* - Y) = 0 \quad \text{(A.3.1)}
\]

From equation (A.1.1);
\[ y^* = 60.92 x^{0.579} \quad \text{(A.1.1)} \]

and from equation (3.10) (Chapter 3) \[ x = y + x_R \quad \text{(A.3.23)} \]

where \[ x_R \] is the outlet dispersed phase concentration as shown in Fig. A.3.1 and is constant at steady state for a given operating condition.

Substituting for (A.3.23) in (A.1.1)
\[ y^* = 60.92 (y + x_R)^{0.579} \quad \text{(A.3.24)} \]

In dimensionless form
\[ Y^* = 60.92 (Y + X_R)^{0.579} \]
where
\[ Y^* = \frac{y^*/x_F}{X_R} \]
\[ Y = \frac{y/x_F}{X_R} \]
\[ X_R = \frac{x_R/x_F}{X_R} \]

Substituting for \( Y^* \) in equation (A.3.1)
\[
\frac{d^2Y}{dz^2} + \frac{Pe}{dZ} \frac{dY}{dz} - N_{oc} \frac{Pe}{C} \left[ 60.92 (Y + X_R)^{0.579} - Y \right] = 0 \quad (A.3.25)
\]

Several methods can be used to solve this equation numerically (J3). For the present studies the method of Runge-Kutta-Which is well described in Jenson and Jeffreys (J3) - for solving first order ordinary differential equation was used by reducing the above equation (A.3.25) to two simultaneous first order equations. The solution of these two first order equations with the original equation now becomes very similar to the solution of a single first order equation provided that the boundary conditions are of initial type value (J3, p. 261).

Thus,
Putting \( \frac{dY}{dz} = W \) \quad ...... (A.3.26a)

Thus \( \frac{d^2Y}{dz^2} = \frac{dW}{dz} \) \quad ...... (A.3.26b)

Substituting for \( \frac{dY}{dz} \) and \( \frac{d^2Y}{dz^2} \) and rearranging, equation (A.3.25) becomes
\[
\frac{dW}{dz} = \frac{Pe}{N_{oc}} (Y^* - Y) - W \quad \text{...... (A.3.27)}
\]

and
\[
\frac{dY}{dz} = W
\]

where
\[ Y^* = 60.92 (Y + X_R)^{0.579} \]

with initial boundary conditions
\[ W(0) = \frac{dY}{dz}_{z=0} \quad \text{...... (A.3.28a)} \]
\[ Y(0) = Y_{z=0} \]
The solution (J3, pp.378-386) was carried out by a Prime 750 computer using the Fortran programme given in Appendix A.4.3.
A.3.2 Calculation of Axial Mixing Coefficient from the One-Dimensional Diffusion Equation.

Axial mixing coefficients $E_c$, for the continuous phase (CCL$_4$ – HAC – H$_2$O system) was calculated from the one-dimensional diffusion equation by the method given below. The diffusion equation developed in Chapter 3.3.2 is:

$$E_c \frac{d^2y}{dh^2} + u_c \frac{dy}{dh} - K_c a(y^* - y) = 0 \quad ...3.67$$

From the concentration profile of $y$ against $h$, the operating equation 3.7 and the equilibrium equation A.1.1, values for $(y^* - y)$ were calculated.

Values of $\frac{dy}{dh}$ was established from $y$ against $h$ graphs using finite difference methods (J3 Pg.389) equation

$$\frac{dy}{dh} = \frac{1}{2s}(y_{n+1} - y_{n-1})$$

where $n$ refers to the level and $s$ an equal interval of $h$.

Using the same data, the second differential $\frac{d^2y}{dh^2}$ was computed from the equation:

$$\frac{d^2y}{dh^2} = \frac{1}{s^2}(y_{n+1} - 2y_n + y_{n+1})$$

By this method a high degree of accuracy could only be achieved if values of $s$ are very small (i.e a very large number of intervals, $n$). Such calculations would be rather tedious to carry out manually and therefore required the use of a computer. This would have meant the fitting of equations to the experimental data of $y$ against $h$ and $K_c a$ against $h$ which was attempted with very little success. In the end it was decided to divide the contactors length $H,$ into 5 cm intervals (i.e $s = 5$ cm) and calculations were done manually from
enlarged graphs.

The calculations were made for two oscillation rates and the results are given in Table 7.2

A.3.3 List of Correlations for Mass Transfer Coefficients.

The list given below is taken from Laddha and Degaleesan (L2 Pg.199-200)

Continuous Phase Coefficients.

\[ (N_{Sh})_c = 2 + 0.95(N_{Re})_{c}^{\frac{1}{4}} (N_{sc})_{c}^{\frac{1}{4}} \] Rigid Liquid Drops Garner and Suckling (G5)

\[ (N_{Sh})_c = 0.98(N_{Pe}) \] Non-circulating Ward et al Drops (W1)

\[ N_{Pe} > 1000 \]

\[ (N_{Sh})_c = 0.61 \left( \frac{\mu_d}{\mu_c + \mu_d} \right) (N_{Pe})^{\frac{1}{2}} \] Circulating Liquid Drops Ward et al (W1)

\[ N_{Pe} > 2.8 \left( \frac{\mu_d + \mu_c}{\mu_d} \right) \left( \frac{12\mu_c + 9\mu_d}{\mu_d} \right)^2 \]

\[ (N_{Sh})_c = 2 + 0.084 \left[ (N_{Re})_{c}^{0.484} (N_{sc})_{c}^{0.339} \right] \] Oscillating Drops Hughmark (H17)

\[ \left( \frac{d_p g}{D_c} \right)^{0.072} \] Penetration theory circulating and Oscillation drops

\[ k_c = 2 \left( \frac{\Delta U_t}{D_d p} \right)^{\frac{1}{4}} \]

\[ \frac{k_c}{U_s (1-\phi)} \left( \frac{\mu_c}{\rho_c \Omega_c} \right)^{0.58} = 0.725 \left( \frac{d_p U_s \rho_c}{\mu_c} \right)^{-0.43} \] Swarm of Drops Ruby and Elgin from Treybal (T14)
Dispersed Phase Coefficient.

\[ k_d = \frac{2\pi^2}{3} \frac{d_d}{d_p} \]  \hspace{1cm} \text{Rigid Spheres} \hspace{1cm} \text{Treybal (T14)}

\[ k_d = 2 \left( \frac{d_d U_t}{\Pi d_p} \right)^\frac{1}{2} \]  \hspace{1cm} \text{Penetration Theory}

\[ k_d = 17.9 \frac{d_d}{d_p} \]  \hspace{1cm} \text{Laminar Circulation} \hspace{1cm} \text{Kroing and Brink (K8)}

\[ k_d = 0.00375 U_t \left( \frac{\mu_c}{\mu_c + \mu_d} \right) \]  \hspace{1cm} \text{Fully Developed} \hspace{1cm} \text{Handlos and Baron (H3)}

\[ \text{Internal Circulation} \]
APPENDIX 4

COMPUTATION OF RESULTS - COMPUTER PROGRAMS

The overall values of NTU were calculated by adding NTU values calculated for each of the four 30 cm sections of extractor length (each section being bounded by sampling points as shown in Fig. 4.3a).

For the computation of the overall value for NTU, the equation

\[
NTU_{cm} = \int \frac{dy}{y^*-y} \quad 3.29
\]

became

\[
NTU_{cm(overall)} = NTU_{cm1} + NTU_{cm2} + NTU_{cm3} + NTU_{cm4}
\]

\[
= \frac{y_2}{y^*-y_1} + \frac{y_3}{y^*-y_2} + \frac{y_4}{y^*-y_3} + \frac{y_5}{y^*-y_4} \quad A4.1
\]

Each sectional NTU \(NTU_{cm}\) was calculated individually using a Simpson's rule integration. This integration was the calculation of the areas LMNP shown below in Fig. A4.1.

![Fig.A4.1 Sectional Values of Transfer Units](image-url)
For each calculation it was assumed that the concentration profile across each section was approximately linear (this assumption is justified from the profiles shown in Figs. 7.1.1 to 7.1.8).

The interval $y_n - y_{n+1}$ was divided into 201 equal strips and letting $1/(y^*-y) = Y$, the calculation was carried out as follows:

\[
\text{Area LMNP} = \frac{y_n - y_{n+1}}{3} \left[ Y_1 + \frac{Y_2}{1} + \frac{Y_3}{4} \right. \\
\left. \frac{Y_4}{+} \cdots + \frac{Y_{200}}{4} \right] \\
\left. + \frac{Y_{201}}{+} \frac{Y_3}{2} + \frac{Y_5}{+} \cdots + \frac{Y_{199}}{2} \right] 
\]

\[\approx \int_{y_n}^{y_{n+1}} \frac{dy}{y^*-y} \cdots A4.3 \]

Sectional values for $HTU_{cm}$ and $Ka_{cm}$ were calculated from $NTU_{cm}$ by equation 3.31.

A similar method was used to calculate $NTU_{dm}$, $HTU_{dm}$ and $Ka_{dm}$, and the computer program used to calculate these values is given below.
APPENDIX A.4.1

COMPUTER PROGRAM FOR CALCULATING MASS TRANSFER EFFICIENCY OF O.B.C
COMMENT START OF ICL I/O
    **********************;

PROCEDURE PICL (R, M, N);
    VALUE R, M, N;
    REAL R; INTEGER M, N;
BEGIN
    INTEGER I;
    IF M=0 THEN
        PRINT PREFIX(''), SCALED(N+1), R, ' ';
    ELSE IF N=0 THEN
        BEGIN
            I:=R;
            PRINT PREFIX(''), DIGITS(M), I, ' ';
        END;
    ELSE
        PRINT PREFIX(''), ALIGNED(M,N), R, ' ';
    END;
END ICLPRINT;

PROCEDURE SPACE (N);
    VALUE N; INTEGER N;
FOR N:=N STEP -1 UNTIL 1 DO
    PRINT PREFIX(''), ' ';

REAL PROCEDURE RICL;
BEGIN
    COMMENT SIMULATES ICL PROCEDURE READ;
    REAL R;
    READ R;
    RICL:=R
END;

BOOLEAN PROCEDURE READBOOLEAN (N);
    VALUE N; INTEGER N;
BEGIN
    COMMENT N IS INPUT CHANNEL NUMBER;
    L1:
    IF NOT (BUFFER(N,'T') OR BUFFER(N,'F')) THEN
        BEGIN
            ADVANCE (N); GOTO L1
        END;
    READBOOLEAN:=BUFFER(N,'T')
PROCEDURE WRITEBOOLEAN (B);
VALUE B; BOOLEAN B;
IF B THEN
   PRINT PREFIX(''), 'TRUE'
ELSE
   PRINT PREFIX(''), 'FALSE';

PROCEDURE OUTPUT (R);
VALUE R; REAL R;
PRINT PREFIX(''), SCALED(11), R, ''L'';

COMMENT END OF ICL I/O
**********;

REAL NTUCMS,HTUCMS,KACMS,NTUDMS,HTUDMS,KADMS,
QC,QD,UC,UD,SD,SUMEV,SUMOD,HOLDUP;
INTEGER N,P,I,K,M,RN;
READER(12);
PUNCH(13);
PRINT PREFIX(''),'MEASURED EFFICIENCIES OF O.B.C. EXTRACTOR';
NEWLINE(2);
PRINT PREFIX(''),' SYSTEM CCL4-HOAC-WATER';
NEWLINE(8);
RN:=1;
LO:N:=RCL;
IF N=0 THEN GOTO L5;
P:=RCL;
BEGIN
ARRAY YP,XP,Y,X[1:N];
ARRAY JC,VD,FM,QM,NTUCM,HTUCM,KACM,NTUDM,HTUDM,KADM[1:N-1];
ARRAY YS,XS,YM,XM,XE,YE,YEM,XEM,YX[1:F];
SD:=RCL;
QC:=QCL;QD:=RCL;
UC:=QC/(3.6*3.142*(15*15));
UD:=QD/(3.6*3.142*(15*15));
HOLDUP:=RCL;
PRINT PREFIX(''),''S22''RUN SPEED(OPM) QC(L/HR) '',
'QD(L/HR)' 'S7''UC(CM/S)' 'S6''UD(CM/S)' 'S7''HOLDUP'';
NEWLINE(2);SPACE(19);
PICL(RN+0.0,3.1);SPACE(7);
PICL(SD+0.0,3.1);SPACE(8);
PICL(QC+0.0,3.1);SPACE(6);
PICL(UC+0.0,1,5);SPACE(4);
PICL(UD+0.0,1,5);SPACE(5);
PICL(HOLDUP+0.0,1,6);
NEWLINE(6);
PRINT PREFIX(''), ' SECTION NTUCM HTUCM(CM) ',
'KACM(S) NTUDM HTUDM(CM) KADM(/S)';
NEWLINE(2);
NTUCMS:=HTUCMS:=KACMS:=NTUDMS:=HTUDMS:=KADM:=0;
Y[1]:=RILC;X[1]:=RILC;
L1:FOR I:=2 STEP 1 UNTIL N DO
  BEGIN
  Y[I]:=RILC;X[I]:=RILC;
  IF Y[I]=0 THEN GOTO L5;
  L2:FOR K:=1 STEP 1 UNTIL P DO
    BEGIN
    IF K=1 THEN YS[K]:=Y[I-1]ELSE GOTO L3;
    XS[K]:=X[I-1];
    GOTO L4;
    L3:YS[K]:=((K-1)*((Y[I]-Y[I-1])/(P-1)))+Y[I-1];
    XS[K]:=((K-1)*((X[I]-X[I-1])/(P-1)))+X[I-1];
    L4:YM[K]:=(YS[K]/60.05)/((YS[K]/60.05)+((1000-YS[K])/18.01534));
    XM[K]:=(XS[K]/60.05)/((XS[K]/60.05)+((1594-XS[K])/153.82));
    YE[K]:=(YS[K]*0.579)*60.92;
    XE[K]:=(YS[K]/60.92)+1.746;
    YEM[K]:=(YE[K]/60.05)/((YE[K]/60.05)+((1000-YE[K])/18.01534));
    XEM[K]:=(XE[K]/60.05)/((XE[K]/60.05)+((1594-XE[K])/153.82));
    YY[K]:=1/(YEM[K]-YM[K]);
    XX[K]:=1/(XEM[K]-XM[K]);
  END;
  JC[I-1]:=(YM[P]-YM[1])/(P-1);
  VD[I-1]:=(XM[P]-XM[1])/(P-1);
  SMEM:=SMOD:=0;
  FOR M:=2 STEP 2 UNTIL P-1 DO
    BEGIN
    SMEM:=SMEM+YY[M];
  END;
  FOR M:=3 STEP 2 UNTIL P-2 DO
    BEGIN
    SMOD:=SMOD+YY[M];
  END;
  PM[I-1]:=(JC[I-1]/3)*((SMEM*4)+(SMOD*2)+(YY[1]+YY[P]));
  NTUCM[I-1]:=PM[I-1];KACM[I-1]:=(UC*PM[I-1])/30;HTUCM[I-1]:=UC/(KACM[I-1]*30);
  SMEM:=SMOD:=0;
BEGIN
SUMEV:=SUMEV+XX[M];
END;
FOR M:=3 STEP 2 UNTIL P-2 DO
BEGIN
SUMOD:=SUMOD+XX[M];
END;
QM[I-1]:= (VD[I-1]/3)*((SUMEV*4)+(SUMOD*2)+(XX[I]+XX[P]));
NTUDM[I-1]:=QM[I-1];KADM[I-1]=(UD*QM[I-1])/30;HTUDM[I-1]:=UD/(KADM[I-1]*30);
SPACE(7);
PICL(I-1+0.0,1,1);SPACE(3);
PICL(PBCM[I-1]+0.0,1,6);SPACE(3);
PICL(HTUCM[I-1]+0.0,5,4);SPACE(4);
PICL(KACM[I-1]+0.0,1,7);SPACE(3);
PICL(NTUDM[I-1]+0.2,6);SPACE(3);
PICL(HTUDM[I-1]+0.0,5,4);SPACE(4);
PICL(KADM[I-1]+0.0,1,7);
NEWLINE(2);
NTUCMS:=NTUCMS+NTUCM[I-1];
NTUDMS:=NTUDMS+NTUDM[I-1];
END;
NEWLINE(5);
SPACE(15);
HTUCMS:=120/NTUCMS;
HTUDMS:=120/NTUDMS;
KACM:=MC/HTUCMS;KADM:=UD/HTUDMS;
PICL(NTUCMS+0.0,1,6);SPACE(3);
PICL(HTUCMS+0.0,5,4);SPACE(4);
PICL(KACM+0.0,1,7);SPACE(4);
PICL(NTUDMS+0.0,2,6);SPACE(3);
PICL(HTUDMS+0.0,5,4);SPACE(4);
PICL(KADM+0.0,1,7);
NEWLINE(6);
PRINT PREFIX(''),''S27''CONCENTRATION PROFILE'';
NEWLINE(2);
PRINT PREFIX(''),' REAL Y (G/L)';
FOR I:=1 STEP 1 UNTIL N DO
BEGIN
PICL(Y[I]+0.0,3,3);
SPACE(4);
END;
NEWLINE(2);
PRINT PREFIX(''),' REAL X (G/L)';
BEGIN
PICL(X[I]+0.0.3,3);
SPACE(4);
END;
PRINT "!B:214!";
END;
RN:=RN+1;
GOTO L0;
L5:END;
END;
APPENDIX A.4.2

COMPUTER PROGRAM FOR CALCULATING RESIDENCE TIME AND AXIAL MIXING PARAMETERS
**MEAN AND VARIANCE ANALYSIS USING THE SATER**
***********CORRECTION***********

WRITTEN BY C.A.MILLINGTON SEPT 72
MOD 11 NOV 80 (PRIME)
PLOTTING REMOVED
icl I/O REMOVED
SATER ABORTION INCLUDED;

PROCEDURE SPACE(N);
VALUE N; INTEGER N;
FOR N:=N STEP -1 UNTIL 1 DO
PRINT PREFIX(''),'';
REAL INT,A, SUM, SUMI, SUMXY, SUMXSQ, SUMC, SUMCT, SUMCTSQ, K,
CUTOFF, INTC, INTC, INTCT, INTCTSQ, P, Q, RATIO, SIGMU, PEC, UL, LL, FL, FU,
TBAR, B, CC, MAX;
INTEGER M, N, SYST, I, J, D, R;
REAL ARRAY C[0:300, 1:6], F, G, MEAN, VAR[1:6];
INTEGER ARRAY TITLE[1:60], LENGTH, TRACK, FAIL, DROP, TOP[1:6];
R:=0;
OPEN('TAPE', 11, 1);
OPEN('SYSTEM', 12, 1);
OPEN('TITLE', 13, 1);
OPEN('OUTPUT', 14, 2);
CLOSE(14);
OPEN('OUTPUT', 14, 5);
OPEN('OUTPUT', 14, 2);
OPEN('PLOT', 15, 2);
CLOSE(15);
OPEN('PLOT', 15, 5);
OPEN('PLOT', 15, 2);

START: FOR J:=1 STEP 1 UNTIL 6 DO
FOR I:=1 STEP 1 UNTIL 300 DO
BEGIN C[I, J]:=0;
END;
COMMENT DATA FROM FILE TITLE WILL BE READ;
READER(13);
M:=1;
INSTRING(TITLE, M);
COMMENT DATA FROM FILE SYSTEM WILL BE READ;
READER(12);
READ N;
IF N LE 0 THEN GOTO FINISH;
PUNCH(14);
PRINT ''B:214'';
PRINT PREFIX(''),'THE DATA BEING PROCESSED IS TITLED';
M:=1;
OUTSTRING(TITLE,M);
NEWLINE(1);
PUNCH(1);
PRINT PREFIX(''),'THE PROGRAM IS NOW PROCESSING DATA';
M:=1;
OUTSTRING(TITLE,M);
NEWLINE(2);
IF N>2 THEN GOTO FOOL;
READER(12);
READ INT;
READ SYST;
IF SYST>5 THEN GOTO FOOL;
J:=1;
COMMENT DATA IN FILE TAPE WILL BE READ AND TESTED FOR MAXIMUM LENGTH;
READER(11);
ALPHA: READ C[0,J];
MAX:=0;
FOR I:=1 STEP 1 UNTIL 301 DO
BEGIN READ A;
A:=ABS(A);
IF A>2200 THEN GOTO BETA;
IF I=300 THEN GOTO GAMMA;
IF I=1 THEN B:=A ELSE C[I,J]:=A-B;
IF C[I,J]<=0 THEN C[I,J]:=0;
IF C[I,J]>=MAX THEN BEGIN MAX:=C[I,J];
TOP[J]:=I;
END;
END;
BETA: LENGTH[J]:=I-1;
IF J=N THEN GOTO DELTA;
J:=J+1;
GOTO ALPHA;
GAMMA: PUNCH(14);
SPACE(14);
PRINT PREFIX(''),'DATA TRACK';
SPACE(5);
PRINT PREFIX(''),'OF THIS RUN HAS MORE THAN 299 VALUES';
NEWLINE(1);
SPACE(5);
NEWLINE(1);
GOTO QUIT;

DELTA: J:=1;
COMMENT THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO LEVEL
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

PHI: IF TOP[J]GE LENGTH[J] THEN GOTO NU;
    IF C[TOP[J],J]LE 300 THEN GOTO PEAK;
    D:=0;
    FOR I:=TOP[J] STEP 1 UNTIL LENGTH[J] DO
    BEGIN A:=C[I,J];
        D:=D+1;
        IF A<60 THEN
        BEGIN IF A=0 THEN C[I,J]:=C[I-1,J];
            TRAC[J]:=I;
            IF D LE 8 THEN GOTO NU;
            FAIL[J]:=1;
            GOTO TEST;
        END;
    END;
    IF I=LENGTH[J] THEN
    BEGIN PUNCH(14);
        SPACE(5);
        PRINT PREFIX(''),'DATA TRACK';
        SPACE(1);
        PRINT PREFIX(''),DIGITS(1),J;
        SPACE(1);
        PRINT PREFIX(''),'OF THIS RUN HAS NOT CONVERGED';
        NEWLINE(1);
        SPACE(5);
        PRINT PREFIX(''),'THE DATA TRACK HAS BEEN IGNORED';
        NEWLINE(1);
        FAIL[J]:=0;
        GOTO TEST;
    END;

END;

PEAK: PUNCH(14);
SPACE(5);
PRINT PREFIX(''),'DATA TRACK';
PRINT PREFIX(''),DIGITS(1),J;
SPACE(1);
PRINT PREFIX(''),'OF THIS RUN DID NOT HAVE ENOUGH DYE';
PRINT PREFIX(''),'INJECTED';
NEWLINE(1);
SPACE(5);
NEWLINE(1);
FAIL[J]:=0;
TEST: IF J=N THEN GOTO THETA;
J:=J+1;
GOTO PHI;
NU: PUNCH(14);
SPACE(5);
PRINT PREFIX(''), 'DATA TRACK';
SPACE(1);
PRINT PREFIX(''), DIGITS(1), J;
SPACE(1);
PRINT PREFIX(''), 'OF THIS RUN HAS TOO LITTLE DATA FOR ACCURATE';
PRINT PREFIX(''), 'ANALYSIS';
NEWLINE(1);
SPACE(5);
PRINT PREFIX(''), 'THEIR WAS AN EQUIPMENT FAULT OR THE PRF WAS';
PRINT PREFIX(''), 'TOO SMALL';
NEWLINE(1);
SPACE(5);
PRINT PREFIX(''), 'THE DATA TRACK HAS BEEN IGNORED';
NEWLINE(1);
FAIL[J]:=0;
GOTO TEST;
COMMENT A NUMBER OF VALID DATA TRACKS ARE NOW IN STORE IN MATRIX C[IJ]
A CHECK FOR A SUFFICIENT NUMBER OF VALID TRACKS WILL BE MADE;
THETA: A:=0;
FOR J:=1 STEP 1 UNTIL N DO
BEGIN A:=A+FAIL[J];
END;
A:=A/N;
IF A<0.5 THEN GOTO LAMBDA;
COMMENT THE TAIL OF VALID DATA TRACKS WILL BE FITTED TO AN EXPONENTIAL
DECAY AND THE POINTS SMOOTHED TO THE BEST LINE;
J:=1;
MU: SUMX:=SUMXY:=SUMXSQ:=SUMY:=0;
IF FAIL[J]=1 THEN GOTO RHO;
IF J=N THEN GOTO SIGMA;
J:=J+1;
GOTO MU;
RHO: D:=0;
PUNCH(14);
M:=TOP[J]+((TRACK[J]-TOP[J]) DIV 2);
IF (TRACK[J]-M) LE 6 THEN M:=TRACK[J]-6;
J - M) GE 40 THEN M := TRACK J - 0;
FOR I = M STEP 1 UNTIL TRACK[J] DO
BEGIN CC := LN(C[I, J]);
  SUMX := SUMX + ((I - 1) * INT);
  SUMY := SUMY + CC;
  SUMXY := SUMXY + (CC * (I - 1) * INT);
  SUMX2 := SUMX2 + ((I - 1) * (I - 1) * INT * INT);
D := D + 1;
END;
NEWLINE(1);
PRINT PREFIX(''), 'CURVE FIT DATA RUN', DIGITS(1), J;
NEWLINE(1);
PRINT PREFIX(''), 'SUM =', SCALED(8), SUMX;
SPACE(3);
PRINT PREFIX(''), 'SUM =', SCALED(8), SUMY;
SPACE(3);
PRINT PREFIX(''), 'SUM =', SCALED(8), SUMXY;
SPACE(3);
PRINT PREFIX(''), 'SUM =', SCALED(8), SUMX2;
SPACE(3);
PRINT PREFIX(''), 'D =', DIGITS(3), D;
NEWLINE(1);
F[J] := ((SUMY * SUMX2) - (SUMX * SUMXY)) / ((D * SUMX2) - (SUMX * SUMX));
PRINT PREFIX(''), '= ', SCALED(8), F[J];
SPACE(4);
G[J] := ((D * SUMXY) - (SUMX * SUMY)) / ((D * SUMX2) - (SUMX * SUMX));
PRINT PREFIX(''), G = ', SCALED(8), G[J];
NEWLINE(1);
IF G[J] GE 0.0 THEN BEGIN DROP[J] := 0;
GOTO REP;
END;
DROP[J] := 1;
FOR I = M STEP 1 UNTIL TRACK[J] DO
BEGIN C[I, J] := EXP(F[J] + (G[J] * (I - 1) * INT));
END;
REP: IF J = N THEN GOTO SIGMA;
J := J + 1;
GOTO MU;
LAMBDA: PUNCH(14);
SPACE(5);
PRINT PREFIX(''), 'MORE THAN HALF THE DATA TRACKS OF THIS RUN';
SPACE(1);
PRINT PREFIX(''), 'ARE INVALID';
SPACE(5);
PRINT PREFIX(''), 'THE RUN HAS BEEN ABANDONED';
NEWLINE(1);
SPACE(5);
PRINT PREFIX(''), 'CONSULT DR MILLINGTON';
NEWLINE(1);
GOTO START;
SIGMA: J:=1;
COMMENT THE MEAN AND VARIANCE OF THE VALID RESPONSE CURVES
WILL BE CALCULATED USING THE SATER CORRECTION;

CHI:
IF FAIL[J]=1 THEN GOTO TAU;
MEAN[J]:=0;
VAR[J]:=0;
J:=J+1;
IF J=N+1 THEN GOTO PI ELSE GOTO CHI;

TAU:
SUMC:=SUMCT:=SUMCTSQ:=0;
FOR I:=1 STEP 1 UNTIL (TRACK[J]-1) DO
BEGIN
    SUMC:=SUMC+(C[I,J]*INT);
    SUMCT:=SUMCT+(C[I,J]*INT*(I-1)*INT);
    SUMCTSQ:=SUMCTSQ+(C[I,J]*INT*(I-1)*(I-1)*INT*INT);
END;

K:=EXP(F[J]);
G[J]:=-G[J];
CUTOFF:=(HK/(2*INT))/(INT/2);
INTC:=(K/G[J]*EXP(-G[J]*CUTOFF));
INTCT:=(K/(G[J]*G[J])*EXP(-G[J]*CUTOFF))*((G[J]*CUTOFF)+1);
INTCTSQ:=(K/(G[J]*G[J]*G[J])*EXP(-G[J]*CUTOFF))*((G[J]*G[J]*
    CUTOFF*CUTOFF)+(2*CUTOFF*G[J])+2);
IF DROP[J]=0 THEN BEGIN INTC:=0.0;
    INTCT:=0.0;
    INTCTSQ:=0.0;
END;

PUNCH(14);
NEWLINE(1);
PRINT PREFIX(''), 'CALCULATION DATA FROM TRACK', Digits(1), J;
NEWLINE(1);
PRINT PREFIX(''), 'SUM='; Scaled(8), SUMC;
SPACE(4);
PRINT PREFIX(''), 'SUMCT='; Scaled(8), SUMCT;
SPACE(4);
PRINT PREFIX(''), 'SUMCTSQ='; Scaled(8), SUMCTSQ;
SPACE(4);
IF DROP[J]=0 THEN BEGIN PRINT PREFIX(''), '***SATER CORRECTION ABORTED***';
PRINT PREFIX(''), ALIGNED(7, 3), MEAN[J], ' '
PRINT PREFIX(''), 'SECS';
P:=P+MEAN[J];
SPACE(6);
PRINT PREFIX(''), 'VARIANCE OF TRACK';
SPACE(1);
PRINT PREFIX(''), DIGITS(1), J;
SPACE(1);
PRINT PREFIX(''), '=';
PRINT PREFIX(''), ALIGNED(7, 3), VAR[J], ' '
PRINT PREFIX(''), 'SECS2';
Q:=Q+VAR[J];
NEWLINE(1);
IF J=N THEN GOTO EPSILON;
J:=J+1;
GOTO KAPPA;

EPSILON: PUNCH(14);
NEWLINE(2);
SPACE(2);
PRINT PREFIX(''), 'AVERAGE MEAN ='
PRINT PREFIX(''), ALIGNED(7, 3), P/(A*N), ' '
PRINT PREFIX(''), 'SECS';
SPACE(12);
PRINT PREFIX(''), 'AVERAGE VARIANCE ='
PRINT PREFIX(''), ALIGNED(7, 3), Q/(A*N), ' '
PRINT PREFIX(''), 'SECS2';
NEWLINE(3);
T BAR:=P/(A*N);
IF SYST GE 4 THEN GOTO PLOT;

COMMENT IF THE SYSTEM CONTAINS A PLUG FLOW SECTION THEN THE PECLET
NUMBER FOR BOTH THE VAN DER LAAN OPEN AND CLOSED PIPE MODELS
WILL BE DETERMINED;
SIGMU:=(Q*N*A)/(P*P);
IF SIGMU<0.2500 THEN GOTO HELL;
M:=1;
PUNCH(14);
NEWLINE(2);
SPACE(20);
PRINT PREFIX(''), 'PECLET NUMBER FOR DOUBLY INFINITE SYSTEM ='
PEC:=(2-(4*SIGMU))-(((4*SIGMU)-2)^2)-
((16*SIGMU)*(SIGMU-2))^(0.5))/(2*SIGMU);
IF PEC>0 THEN GOTO PECPRINT;
PLUS: PEC:=((2-(4*SIGMU))+((((4*SIGMU)-2)^2)-...


```
      (16*SIGMU)*(SIGMU-2)) ^0.5)/(2*SIGMU);
M:=2;
IF PEC>0 THEN GOTO PECPRINT ELSE GOTO LOOK;
PECPRINT: PUNCH(14);
SPACE(1);
PRINT PREFIX(''), ALIGNED(4, 3), PEC;
SPACE(5);
IF M=1 THEN GOTO PLUS ELSE
IF M=2 THEN GOTO LOOK ELSE GOTO PLOT;
HELL: PUNCH(14);
NEWLINE(2);
SPACE(10);
PRINT PREFIX(''), 'SOLUTION OF EDDY DIFFUSION EQUATION IMPOSSIBLE';
NEWLINE(1);
SPACE(22);
PRINT PREFIX(''), 'CONSULT DR MILLINGTON';
NEWLINE(1);
GOTO PLOT;
LOOK: RATIO:=-0.5+((5^0.5)/2);
UL:=2/SIGMU;
LL:=0;
D:=0;
MAXSEEK: PEC:=UL-(RATIO*(UL-LL));
FL:=(2/PEC)-((2/(PEC*PEC))*(1-EXP(-PEC)));
PEC:=LL+(RATIO*(UL-LL));
FU:=(2/PEC)-((2/(PEC*PEC))*(1-EXP(-PEC)));
IF FL>FU THEN UL:=LL+(RATIO*(UL-LL)) ELSE
LL:=UL-(RATIO*(UL-LL));
IF UL-LL<0.01 THEN GOTO ZERSEEK ELSE
IF D>40 THEN GOTO LAM1;
D:=D+1;
GOTO MAXSEEK;
ZEROSEEK: LL:=UL;
UL:=2/SIGMU;
D:=0;
M:=3;
AGAIN: PEC:=UL-(RATIO*(UL-LL));
FL:=ABS((2/PEC)-((2/(PEC*PEC))*(1-EXP(-PEC))));
PEC:=LL+(RATIO*(UL-LL));
FU:=ABS((2/PEC)-((2/(PEC*PEC))*(1-EXP(-PEC))));
IF FL>FU THEN LL:=UL-(RATIO*(UL-LL)) ELSE
UL:=LL+(RATIO*(UL-LL));
IF UL-LL<0.01 THEN GOTO CALC ELSE
IF D>40 THEN GOTO LAM2;
```
: = +1;
GO TO AGAIN;
CALC: PEC:= (UL+LL)/2;
PUNCH(14);
NEWLINE(2);
SPACE(20);
PRINT PREFIX(''), 'PECLET NUMBER OF CLOSED PIPE SYSTEM =';
GO TO PEC;
PRINT;
LAM1: PUNCH(14);
NEWLINE(1);
SPACE(10);
PRINT PREFIX(''), 'MAXSEEK HAS NOT CONVERGED - CONSULT DR LAMB';
GO TO PLOT;
LAM2: PUNCH(14);
NEWLINE(1);
SPACE(10);
PRINT PREFIX(''), 'ZEROSEEK HAS NOT CONVERGED - CONSULT DR LAMB';
GO TO PLOT;
COMMENT THE FOLLOWING PACKAGE TAKES THE VALID DATA TRACKS AND
EVALUATES A MEAN DIMENSIONLESS DATA SET WHICH IS
USED TO EVALUATE THE F-CURVE;

PLOT: BEGIN REAL CZERO;
REAL ARRAY TOT, RATIO[1:6], CDIM, TDIM[1:300],
ACC[0:300];
PUNCH(14);
PRINT 'B: 214''
FOR I = 1 STEP 1 UNTIL 300 DO
BEGIN CDIM[I] := 0;
TDIM[I] := 0;
ACC[I] := 0;
END;
ACC[0] := 0;
M := 0;
J := 1;
TOTAL: IF FAIL[J] # 0 THEN GO TO CHECK;
M := M + 1;
FOR I = 1 STEP 1 UNTIL TRACK[J] DO
BEGIN TOT[M] := TOT[M] + C[I, J];
C[I, M] := 0[I, J];
END;
TRACK[M] := TRACK[J];
CHECK: J := J + 1;
IF J LE N THEN GO TO TOTAL;
FOR M = 1 STEP 1 UNTIL N DO
BEGIN RATIO[M] = TOT[I]/TOT[M];
FOR I = 1 STEP 1 UNTIL TRACK[M] DO
BEGIN C[I,M] = C[I,M]*RATIO[M];
   ACC[I] = ACC[I] + (C[I,M]/N);
END;
END;
CZERO = 0;
FOR I = 1 STEP 1 UNTIL 300 DO
BEGIN CZERO = CZERO + (ACC[I]*INT/TBAR);
END;
J = 0;
NEWLINE(1);
PRINT PREFIX(' '), 'READ ACROSS THE PAGE----------------->';
NEWLINE(1);
SPACE(4);
PRINT PREFIX(' '), 'F' 'S8' 'TDIM' 'S12' 'F' 'S8' 'TDIM';
SPACE(12);
PRINT PREFIX(' '), 'F' 'S8' 'TDIM' 'S12' 'F' 'S8' 'TDIM';
SPACE(7);
PRINT PREFIX(' '), 'F' 'S8' 'TDIM';
NEWLINE(1);
M = 0;
FOR I = 1 STEP 1 UNTIL 300 DO
BEGIN CDIM[I] = ACC[I]/CZERO;
   ACC[I] = ACC[I-1] + (CDIM[I]*INT/TBAR);
   TDIM[I] = (I-1)*INT/TBAR;
   IF ACC[I]GE 1 THEN ACC[I] = 1.0;
COMMENT F AND TDIM DATA OUTPUT TO THE PLOT FILE;
PUNCH(15);
NEWLINE(1);
PRINT PREFIX(' '), ALIGNED(2,3), TDIM[I];
SPACE(3);
PRINT PREFIX(' '), ALIGNED(1,3), ACC[I];
COMMENT F AND TDIM DATA OUTPUT TO THE OUTPUT FILE;
IF ACC[I] > 0.99999 THEN BEGIN M = M + 1;
   IF M > 3 THEN GOTO LOOP;
END;
PUNCH(14);
PRINT PREFIX(' '), ALIGNED(1,3), ACC[I];
SPACE(4);
PRINT PREFIX(' '), ALIGNED(2,3), TDIM[I];
SPACE(8);
J:=J+1;
IF J=5 THEN BEGIN NEWLINE(1);
J:=0;
END;

LOOP: END;
COMMENT THE F-CURVE WILL BE PLOTTED OUT ALONG WITH THE COMPLETE
MIXING AND PLUG FLOW CURVES USING THE SPLT PACKAGE;
GOTO START;
OUT:
PUNCH(14);
SPACE(5);
PRINT PREFIX(''),'MORE THAN 9 SETS OF DATA SUBMITTED';
NEWLINE(1);
SPACE(5);
PRINT PREFIX(''),'THIS HAS INFRINGED THE SPLT LIMIT';
NEWLINE(1);
SPACE(5);
PRINT PREFIX(''),'RESUBMIT REMAINING DATA INCLUDING THIS SET';
GOTO FINISH;
END;
FOOL: PUNCH(14);
SPACE(5);
PRINT PREFIX(''),'ERROR IN SYSTEM DATA - EITHER N>6 OR SYST>5';
NEWLINE(1);
SPACE(5);
PRINT PREFIX(''),'BE MORE CAREFUL IN FUTURE';
NEWLINE(1);
SPACE(5);
PRINT PREFIX(''),'THE PROGRAM HAS BEEN HALTED';
NEWLINE(1);
GOTO STOP;
QUIT: PUNCH(14);
SPACE(5);
PRINT PREFIX(''),'ERROR IN DATA ON TAPE';
SPACE(1);
PRINT PREFIX(''),'EITHER 2300 OMITTED OR TOO MANY READINGS';
NEWLINE(1);
SPACE(5);
PRINT PREFIX(''),'BE MORE CAREFUL IN FUTURE';
NEWLINE(1);
GOTO STOP;
FINISH: PUNCH(14);
NEWLINE(4);
PRINT PREFIX(''),'THE PROCESSING OF DATA IS NOW COMPLETE';
SPACE(1);
PRINT PREFIX(''), 'PLEASE STUDY THE PRINTOUT CAREFULLY';
NEWLINE(1);
SPACE(6);
PRINT PREFIX(''), 'DR MILLINGTON WILL ASSIST IN THE INTERPRETATION';
SPACE(1);
PRINT PREFIX(''), 'OF THE RESULTS';
NEWLINE(2);
STOP: SPACE(33);
PRINT PREFIX(''), '**********END OF PRINTOUT**********';
CLOSE(11);
CLOSE(12);
CLOSE(13);
CLOSE(14);
CLOSE(15);
PUNCH(1);
PRINT PREFIX(''), 'RUN COMPLETE''L'''
PRINT PREFIX(''), 'PLEASE EXAMINE FILE OUTPUT FOR YOUR RESULTS''L'''
END;
APPENDIX A.4.3

COMPUTER PROGRAM FOR SOLVING SECOND ORDER DIFFERENTIAL EQUATIONS BY THE RUNGE-KUTTA METHOD
DIMENSION PRMT(5), Y(2), DERY(2), AUX(8, 2)
EXTERNAL FCT, OUT
NDIM=2
PRMT(1)=0. DO
PRMT(2)=1. DO
PRMT(3)=0. 01D0
PRMT(4)=1. D-06
Y(1)=1. 75D0
Y(2)=0. 308D0
DERY(1)=0. 5D0
DERY(2)=0. 5D0
CALL DRKGS (PRMT, Y, DERY, NDIM, IHLF, FCT, OUT, AUX)
STOP
END
SUBROUTINE DRKGS(PRMT, Y, DERY, NDIM, IHLF, FCT, OUT, AUX)
IMPLICIT DOUBLE PRECISION (A-H, O-Z)
DIMENSION Y(NDIM), DERY(NDIM), AUX(8, NDIM), PRMT(5), A(4), B(4), C(4)
DO 1 I=1, NDIM
1 AUX(8, I)=DERY(I)/15. DO
X=PRMT(1)
XEND=PRMT(2)
H=PRMT(3)
PRMT(5)=0. DO
CALL FCT(PRMT, X, Y, DERY, NDIM)
IF(H*(XEND-X)) 38, 37, 2
2 A(1)=0. 5D0
A(2)=1. DO-DSQRT(2. DO)/2. DO
A(3)=1. DO*DSQRT(2. DO)/2. DO
A(4)=1. DO/6. DO
B(1)=2. DO
B(2)=1. DO
B(3)=1. DO
B(4)=2. DO
C(1)=0. 5D0
C(2)=A(2)
C(3)=A(3)
C(4)=0. 5D0
DO 3 I=1, NDIM
AUX(1, I)=Y(I)
AUX(2, I)=DERY(I)
AUX(3, I)=0. DO
3 AUX(6, I)=0. DO
IREC=0
IHLF=-1
ISTEP=0
IEND=0
4 IF(((X+H-XEND)*H) 7,6,5
5 H=XEND-X
6 IEND=1
7 CALL OUT(PRMT,X,Y,DERY,NDIM,IHLF,AUX)
   IF(PRMT(5)) 40,8,40
8 ITEST=0
9 ISTEP=ISTEP+1
   J=1
10 AJ=A(J)
   BJ=B(J)
   CJ=C(J)
   DO 11 I=1,NDIM
      R1=DERY(I)*H
      R2=AJ*(R1-BJ*AUX(6,I))
      Y(I)=Y(I)+R2
      R2=R2+R2+R2
   11 AUX(6,I)=AUX(6,I)+R2-CJ*R1
      IF(J-4) 12,15,15
   12 J=J+1
      IF(J-3) 13,14,13
   13 X=X+0.5D0*H
   14 CALL FCT(PRMT,X,Y,DERY,NDIM)
      GO TO 10
   15 IF(IWRITE) 16,16,20
   16 DO 17 I=1,NDIM
   17 AUX(4,I)=Y(I)
      ITEST=1
      ISTEP=ISTEP+ISTEP-2
   18 IHLF=IHLF+1
      X=X-H
      H=0.5D0*H
      DO 19 I=1,NDIM
         Y(I)=AUX(1,I)
         DERY(I)=AUX(2,I)
   19 AUX(6,I)=AUX(3,I)
      GO TO 9
   20 IMOD=ISTEP/2
      IF(ISTEP-IMOD-IMOD) 21,23,21
   21 CALL FCT(PRMT,X,Y,DERY,NDIM)
      DO 22 I=1,NDIM
22 AUX(7,I)=DERY(I)
   GO TO 9
23 DELT=0.D0
   DO 24 I=1,NDIM
24 DELT=DELT+AUX(8,I)*DABS(AUX(4,I)-Y(I))
   IF(DELT-PRMT(4))28,28,25
25 IF(IHLF=10) 26,36,36
26 DO 27 I=1,NDIM
27 AUX(4,I)=AUX(5,I)
   ISTEP=ISTEP+ISTEP-4
   X=X-H
   IEND=0
   GO TO 48
28 CALL FCT(PRMT,X,Y,DERY,NDIM)
   DO 29 I=1,NDIM
   AUX(1,I)=Y(I)
   AUX(2,I)=DERY(I)
   AUX(3,I)=AUX(6,I)
   Y(I)=AUX(5,I)
29 DERY(I)=AUX(7,I)
   CALL OUT(PRMT,X-H,Y,DERY,NDIM,IHLF,AUX)
   IF(PRMT(5)) 40,30,40
30 DO 31 I=1,NDIM
   Y(I)=AUX(1,I)
31 DERY(I)=AUX(2,I)
   IREC=IHLF
   IF(IEND) 32,32,39
32 IHLF=IHLF-1
   ISTEP=ISTEP/2
   H=H+H
   IF(IHLF) 4,33,33
33 IMOD=ISTEP/2
   IF(ISTEP-IMOD-IMOD) 4,34,4
34 IF(DELT-0.02D0*PRMT(4)) 35,35,4
35 IHLF=IHLF-1
   ISTEP=ISTEP/2
   H=H+H
   GO TO 4
36 IHLF=11
   CALL FCT(PRMT,X,Y,DERY,NDIM)
   GO TO 39
37 IHLF=12
   GO TO 39
CALL OUT(PRMT, X, Y, DRY, NDIM, IHLF, AUX)
RETURN
END

SUBROUTINE FCT(PRMT, Z, Y, DYDZ, NDIM)
IMPLICIT REAL*8 (A-H, O-Z)
DIMENSION PRMT(10), Y(NDIM), DYDZ(NDIM)
XM=60.92
C=0.579
XB=0.061
XNC=0.064*EXP(-3.5*Z)
XPE=8.13
YS=XM*(Y(2)+XB)**C
DYDZ(1)= XPE*XNC*(YS-Y(2))-Y(1))
DYDZ(2)=Y(1)
RETURN
END

SUBROUTINE OUT(PRMT, Z, Y, DYDZ, NDIM, IHLF, AUX)
IMPLICIT REAL*8 (A-H, O-Z)
DIMENSION PRMT(10), Y(NDIM), DYDZ(NDIM), AUX(8, NDIM)
WRITE(6, 1000) Z, Y(1), Y(2)
1000 FORMAT(1H, 'Z=', F6.3, 5X, 'DYDZ=', 1PD13.6, 5X, 'Y=', 1PD13.6)
RETURN
END
BLOCK A TABLES

CCL₄ - HAC - H₂O System

CONTACTOR I (LARGE)
### Table 1A  Concentration Profile For CCL$_4$ - HAC - H$_2$O System

$R = \frac{Q_c}{Q_d} = 1$, Constant $x_1$. Contactor I (Large).

<table>
<thead>
<tr>
<th>$N_{(opm)}$</th>
<th>$Q_c_{(l/hr)}$</th>
<th>Sampling Positions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Concentration (g/l)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Bot 1 2 3 4 5 Top</td>
</tr>
<tr>
<td>60</td>
<td>200</td>
<td>$y$  0.0 15.5 22.0 25.0 26.3 27.0 25.5</td>
</tr>
<tr>
<td></td>
<td>$x$ 4.5</td>
<td>20.0 24.5 29.5 30.3 31.5 30.5</td>
</tr>
<tr>
<td>300</td>
<td>$y$ 0.0</td>
<td>12.5 20.0 23.8 25.0 25.3 24.0</td>
</tr>
<tr>
<td></td>
<td>$x$ 3.5</td>
<td>16.0 24.0 27.3 28.5 28.8 28.0</td>
</tr>
<tr>
<td>400</td>
<td>$y$ 0.0</td>
<td>11.0 23.0 26.5 27.0 27.5 27.0</td>
</tr>
<tr>
<td></td>
<td>$x$ 2.0</td>
<td>13.0 25.5 28.5 29.0 30.5 29.0</td>
</tr>
<tr>
<td>500</td>
<td>$y$ 0.0</td>
<td>10.0 19.5 24.5 27.0 28.0 27.0</td>
</tr>
<tr>
<td></td>
<td>$x$ 2.0</td>
<td>12.0 21.5 26.5 29.0 30.0 30.5</td>
</tr>
<tr>
<td>80</td>
<td>200</td>
<td>$y$ 0.0 12.0 18.0 22.0 25.0 26.5 26.5</td>
</tr>
<tr>
<td></td>
<td>$x$ 3.5</td>
<td>15.5 21.5 25.5 28.5 29.5 30.5</td>
</tr>
<tr>
<td>300</td>
<td>$y$ 0.0</td>
<td>11.0 23.5 27.5 29.8 30.5 30.5</td>
</tr>
<tr>
<td></td>
<td>$x$ 2.0</td>
<td>13.0 25.5 29.8 31.8 32.5 32.5</td>
</tr>
<tr>
<td>400</td>
<td>$y$ 0.0</td>
<td>10.0 20.0 25.0 27.8 29.8 30.0</td>
</tr>
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Table 1A Continued.

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<th>Concentration (g/l)</th>
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Table 3A  Concentration Profile For CCl₄ - HAC - H₂O System  

\( R = \frac{Q_c}{Q_d} = 1 \), Varying \( x_i \), Contactor I (Large)

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<td>x</td>
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Table 4A Frequency of Droplet Sizes from Photographic Studies for CCL$_4$ - HAC - H$_2$O System, at Constant Flowrate, Varying Oscillation Speed. Contactor I $Q_c = Q_d = 200$ l/hr.

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<th>100</th>
<th>120</th>
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</thead>
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<td>Droplet Size (mm)</td>
<td>Frequency</td>
<td>Of Droplet</td>
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<td>18</td>
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<tr>
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<td>13</td>
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Table 5A Sauter Mean Drop Diameter, Interfacial Area and Mass Transfer Coefficient for Continuous Phase, Determined Experimentally from Photographic Studies for CCL₄ - HAC - H₂O System. Contactor I

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>Qc (l/hr)</th>
<th>d₃₂ (cm)</th>
<th>a (cm²/cm³)</th>
<th>Kₑ a x 10⁶ (sec⁻¹)</th>
<th>Kₑ x 10⁶ (cm/sec)</th>
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<tbody>
<tr>
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<td>0.581</td>
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<td>0.731</td>
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Table 6A  Experimentally Determined Overall NTU, HTU, Ka Values
for CCl₄ - HAC - H₂O System, Contactor I (Large)
Constant Flow Ratio, R = Q₀ / Q₀ = 1, Constant xᵢ

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>Q_c (l/hr)</th>
<th>NTU_cm</th>
<th>HTU_cm (M)</th>
<th>Ka_cm x10⁶ (sec⁻¹)</th>
<th>NTU_dm</th>
<th>HTU_dm (M)</th>
<th>Ka_dm x10⁶ (sec⁻¹)</th>
<th>φ_F</th>
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Table 7A Experimentally Determined Overall NTU, HTU, Ka Values
For CCl₄ - HAC - H₂O system, Contactor I (Large)

(a) Varying Flowratio R, Constant Inlet Solute Concentration

<table>
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<th>N (opm)</th>
<th>Qc (l/hr)</th>
<th>R=Qc/400</th>
<th>NTU cm</th>
<th>HTU cm</th>
<th>Ka_cm x 10^6 (sec^-1)</th>
<th>NTU dm</th>
<th>HTU dm</th>
<th>Ka_dm x 10^6 (sec^-1)</th>
<th>φ_F</th>
</tr>
</thead>
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(b) Constant Flowratio R = 1 Varying Inlet Solute Concentration

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<th>NTU cm</th>
<th>HTU cm</th>
<th>Ka_cm x 10^6 (sec^-1)</th>
<th>NTU dm</th>
<th>HTU dm</th>
<th>Ka_dm x 10^6 (sec^-1)</th>
<th>φ_F</th>
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Table 8A Extraction Efficiency. Contactor I (Large)
CCL₄ - HAC - H₂O System, Constant x₁.

<table>
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<th>N (opm)</th>
<th>Q (l/hr)</th>
<th>R</th>
<th>y₁ (g/l)</th>
<th>y₅ (g/l)</th>
<th>x₅ (g/l)</th>
<th>y₅ - y₁ (g/l)</th>
<th>nₑ (%)</th>
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Extraction Efficiency \( nₑ = R \left( \frac{y₅ - y₁}{x₅} \right) \times 100 \)

\( R = \frac{Q_c}{Q_d} \)
Table 9A  Extraction Efficiency Contactor I (large)  
CCl₄ - HAC - H₂O System.

(a) Varying Inlet Solute Concentration, x₁.

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<th>y₁ (g/l)</th>
<th>y₅ (g/l)</th>
<th>x₅ (g/l)</th>
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(b) Varying Flow Ratio, R.

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\[
\eta_E = \frac{R (y_5 - y_1)}{\chi_5} \times 100
\]

\[
R = \frac{Q_c}{Q_d}
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Table 11A  Hold-up Studies For Low Solute Concentration  
\( (x_i \approx 0.5 \text{ g/l}) \), \( N = 100 \text{ opm} \),  CCL\(_4\) - HAC - H\(_2\)O  
System, Contactor I.

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<th>500</th>
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Table 12A Hold-up Studies For High Solute Concentration
\(x_i = 20 \text{ g/l}\) and Low Solute Concentration
("Non M.T System") \(x_i \approx 0.5 \text{ g/l}\) \(CCl_4 - \text{HAC - H}_2\text{O}\)
System \(N = 100 \text{ opm, Contactor I.}\)

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<th>(Q_c) (l/hr)</th>
<th>(Q_d) (l/hr)</th>
<th>(U_d x 10^2) (cm(^3)/sec cm(^2))</th>
<th>Fractional Hold-up Non M.T Syst (\phi_F)</th>
<th>Fractional Hold-up M.T system (\phi_F)</th>
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*Limiting Flow Condition
Table 13A  Residence Time Studies for Single (Continuous) Phase. Contactor I (Large)

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<th>$\mu$ (secs)</th>
<th>$\bar{t}-\mu$ (secs)</th>
<th>$\sigma^2 \times 10^{-3}$ (sec$^2$)</th>
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Table 14A Experimental and Calculated Axial Mixing Results for Single (Continuous) Phase. Contactor I (Large).

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<th>Pe calc (Eqn 7.10)</th>
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Table 15A Variation of Dimensionless Variance with Peclet Number for Closed and Open Channels.

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For closed channels (Van der Laan (V 1 ) )

$$\sigma^2_{01} = \frac{2}{Pe} (Pe - 1 + e^{-Pe}) \quad \ldots 3.57$$

For open channels (Levenspiel and Smith (L 5 ) )

$$\sigma^2_{02} = \frac{2}{Pe} + \frac{8}{Pe^2} \quad \ldots 3.58$$
Table 16A Axial Mixing Studies Based on the One Dimensional Diffusion Equation for CCL\textsubscript{4} - HAC - H\textsubscript{2}O system, Contactor I

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### Table 17A  Dry Power Study Contactor I (Large)

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### Table 18A  Single Phase Mixing Power Study Contactor I (Large)

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<th>Mixing Power, $P_{M1}$ (Joules/sec)</th>
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Table 19A Two Phase Mixing Power Study Contactor I (Large) CCL\textsubscript{4} - HAC - H\textsubscript{2}O System.

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<th>(P_{T}) (J/sec)</th>
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\(P_{\text{Dry}}\) = Dry Power Consumption  
\(P_{M1}\) = Single Continuous Phase Mixing Power  
\(P_{M2}\) = Single Dispersed Phase Mixing Power (By difference)  
\(P_{M}\) = Two Phase Mixing Power = \(P_{M1} + P_{M2}\)  
\(P_{T}\) = Total Power Consumption = \(P_{d} + P_{M} = P_{d} + P_{M1} + P_{M2}\)
Table 20A  Two Phase Mixing Power Per Unit
Active and Swept Volume CCl₄-HAC-H₂O
System Contactor I.

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>Q_c = Q_d (1/hr)</th>
<th>P_{va} (J/sec·m³)</th>
<th>P_{vs} (J/sec·m³)</th>
</tr>
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<tbody>
<tr>
<td>60</td>
<td>200</td>
<td>26.10</td>
<td>59.12</td>
</tr>
<tr>
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<td>59.55</td>
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<td>26.33</td>
<td>59.70</td>
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<td>200</td>
<td>69.71</td>
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<td>160.99</td>
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<td>144.88</td>
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<td>146.37</td>
<td>331.95</td>
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<td>400</td>
<td>147.61</td>
<td>334.76</td>
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<td>500</td>
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<td>200</td>
<td>267.88</td>
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<td>269.48</td>
<td>611.15</td>
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<td>400</td>
<td>270.36</td>
<td>613.15</td>
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<tr>
<td></td>
<td>500</td>
<td>272.36</td>
<td>623.34</td>
</tr>
</tbody>
</table>

P_{va} = Power Per Unit Active Volume = P_m / V_a
P_{vs} = Power Per Unit Swept Volume = P_m / V_s
BLOCK B TABLES

MIBK - HAC - $H_2O$ System

CONTACTOR I (LARGE)
Table 1B Concentration Profile For System MIBK - HAC - H₂O
R = Q_c / Q_d = 1 Contactor I

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>Q_c (1/hr)</th>
<th>Sampling Points Concentration (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Bot 1 2 3 4 5 Top</td>
</tr>
<tr>
<td>50</td>
<td>350</td>
<td>y 27.6 27.5 25.4 21.3 14.2 5.5 0.0</td>
</tr>
<tr>
<td>60</td>
<td>350</td>
<td>x 33.7 35.0 33.0 28.8 21.7 13.0 7.5</td>
</tr>
<tr>
<td>65</td>
<td>350</td>
<td>y 26.1 26.0 23.0 18.2 11.2 5.1 0.0</td>
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<td>350</td>
<td>x 30.0 30.6 27.6 22.8 15.6 9.6 4.6</td>
</tr>
<tr>
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<td></td>
<td>y 27.2 27.1 24.4 17.0 9.1 3.8 0.0</td>
</tr>
<tr>
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<td></td>
<td>x 31.4 31.5 28.8 21.4 13.5 8.2 4.4</td>
</tr>
<tr>
<td></td>
<td></td>
<td>y 24.1 24.0 21.5 14.8 8.5 4.2 0.0</td>
</tr>
<tr>
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<td>350</td>
<td>x 27.1 27.2 24.7 18.0 11.2 7.2 3.2</td>
</tr>
<tr>
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<td></td>
<td>y 31.0 30.8 25.9 18.7 10.6 6.9 0.0</td>
</tr>
<tr>
<td>70</td>
<td>300</td>
<td>x 35.7 35.0 30.1 22.9 14.8 11.1 4.2</td>
</tr>
<tr>
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<td>y 24.1 24.0 21.5 14.9 8.1 4.0 0.0</td>
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<tr>
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<td>350</td>
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</tr>
<tr>
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<td></td>
<td>y 33.3 33.3 27.7 17.4 9.5 3.9 0.0</td>
</tr>
<tr>
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<td>400</td>
<td>x 38.5 38.2 32.6 22.3 14.4 8.9 4.9</td>
</tr>
<tr>
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<td></td>
<td>y 32.9 32.7 25.8 16.8 9.2 3.7 0.0</td>
</tr>
<tr>
<td>70</td>
<td>500</td>
<td>x 37.0 36.4 29.5 20.4 12.9 7.4 3.6</td>
</tr>
</tbody>
</table>
Table 2B Frequency of Droplet Sizes from Photographic Studies for MIBK - HAC - H₂O system, at Constant Flowrate, Varying Oscillation Speed Contactor I, \( Q_c = Q_d = 350 \text{ l/hr.} \)

<table>
<thead>
<tr>
<th>Drop Size No.</th>
<th>N (opm)</th>
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<th>60</th>
<th>65</th>
<th>70</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Droplet Size (mm)</td>
<td>Frequency of Droplets ( f_i )</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>1</td>
<td>0.250</td>
<td>15</td>
<td>10</td>
<td>6</td>
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</tr>
<tr>
<td>2</td>
<td>0.381</td>
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<td>46</td>
<td>27</td>
<td>68</td>
</tr>
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<td>0.512</td>
<td>34</td>
<td>81</td>
<td>106</td>
<td>124</td>
</tr>
<tr>
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<td>64</td>
<td>97</td>
<td>83</td>
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<td>32</td>
<td>53</td>
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<tr>
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<td>23</td>
<td>27</td>
<td>19</td>
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<td>3</td>
<td>10</td>
<td>5</td>
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<td>2</td>
</tr>
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<td>1</td>
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<td>-</td>
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<td>1</td>
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<td>-</td>
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<tr>
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<td>-</td>
<td>-</td>
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</table>
Table 3B  Sauter Mean Drop Diameter, Interfacial Area and Mass Transfer Coefficient for Continuous Phase, Determined Experimentally from Photographic Studies for MIBK - HAC - H₂O System, Contactor I.

<table>
<thead>
<tr>
<th>$Q_c = Q_d$ (l/hr)</th>
<th>N (opm)</th>
<th>$d_{32}$ (cm)</th>
<th>$a$ (cm²/cm³)</th>
<th>$K_a \times 10^6$ (sec⁻¹)</th>
<th>$K_c \times 10^6$ (cm/sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
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<td>0.0879</td>
<td>0.614</td>
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<tr>
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<td>11.472</td>
<td>8.574</td>
</tr>
<tr>
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<td>350</td>
<td>0.0848</td>
<td>1.592</td>
<td>13.193</td>
<td>8.287</td>
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Table 4B Extraction Efficiency, Contactor I
MIBK - HAC - H₂O System.

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>Q (l/hr)</th>
<th>R</th>
<th>( y_1 ) (g/l)</th>
<th>( y_5 ) (g/l)</th>
<th>( x_1 ) (g/l)</th>
<th>( y_1 - y_5 ) (g/l)</th>
<th>( \eta_E ) (%)</th>
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</thead>
<tbody>
<tr>
<td>70</td>
<td>300</td>
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<td>19.98</td>
<td>73.51</td>
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<td>21.95</td>
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<td>350</td>
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<td>4.02</td>
<td>27.18</td>
<td>19.98</td>
<td>73.51</td>
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</table>

Extraction Efficiency \( \eta_E = \frac{R(\frac{y_1 - y_5}{x_1})}{\times 100} \)

\( R = \frac{Q_c}{Q_d} \).
Table 58: Experimentally Determined NTU, HTU, Ka Values For MIBK - HAC - H₂O System R = 1, Contactor I

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>Qc (l/hr)</th>
<th>NTU&lt;sub&gt;cm&lt;/sub&gt;</th>
<th>HTU&lt;sub&gt;cm&lt;/sub&gt;</th>
<th>K&lt;sub&gt;acm&lt;/sub&gt; x10&lt;sup&gt;4&lt;/sup&gt; (sec&lt;sup&gt;-1&lt;/sup&gt;)</th>
<th>NTU&lt;sub&gt;dm&lt;/sub&gt;</th>
<th>HTU&lt;sub&gt;dm&lt;/sub&gt;</th>
<th>K&lt;sub&gt;adm&lt;/sub&gt; x10&lt;sup&gt;4&lt;/sup&gt; (sec&lt;sup&gt;-1&lt;/sup&gt;)</th>
<th>Φ&lt;sub&gt;F&lt;/sub&gt;</th>
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<tbody>
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<td>0.7398</td>
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<td>0.0220</td>
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<td>1.0279</td>
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<td>0.0225</td>
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</table>
Table 6B Sectional Values of Number and Height of Transfer Units and Mass Transfer Coefficients \( R = \frac{Q_c}{Q_d} = 1 \)

MIBK - HAC - H₂O system, Contactor I

<table>
<thead>
<tr>
<th>N</th>
<th>( Q_c = Q_d ) (opm)</th>
<th>Section</th>
<th>NTU cm</th>
<th>HTU cm (M)</th>
<th>( K_a_{cm} \times 10^6 ) (sec⁻¹)</th>
<th>NTU dm</th>
<th>HTU dm (M)</th>
<th>( K_a_{dm} \times 10^6 ) (sec⁻¹)</th>
</tr>
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<tbody>
<tr>
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<td>1 (Top)</td>
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<td>0.7829</td>
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Table 6B Continued
Table 7B  Power and Hold-up Study for MIBK - HAC - $H_2O$
System. Contactor I (large)

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<th>N (opm)</th>
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<th>$h_m$ cm of CCL$_4$</th>
<th>$T$ (Joules)</th>
<th>$\phi_F$</th>
<th>$P_T$ (J/sec)</th>
<th>$P_M$ (J/sec)</th>
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<td>0.0225</td>
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<td>3.740</td>
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$P_{Dry}$ = Dry Power Consumption  
$P_{M1}$ = Single Continuous Phase Mixing Power  
$P_{M2}$ = Single Dispersed Phase Mixing Power (By difference)  
$P_M$ = Two Phase Mixing Power = $P_{M1} + P_{M2}$  
$P_T$ = Total Power Consumption = $P_d + P_M = P_d + P_{M1} + P_{M2}$
Table 8B  Two Phase Mixing Power Per Unit Active and Swept Volume MIBK - HAC - H₂O System Contactor I

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<th>N (opm)</th>
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<th>P_{va} (J/sec m^3)</th>
<th>P_{vs} (J/sec m^3)</th>
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BLOCK C TABLES

CCL$_4$ - HAC - $H_2O$ System

CONTACTOR II (SMALL)
Table 1C Concentration Profile for System CCl₄ - HAC - H₂O
R = Qc / Qd = 1 Contactor II.

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Table 2C Frequency of Droplet Sizes from Photographic Studies for CCL₄ - HAC - H₂O System, at Constant Flowrate, Varying Oscillation Speed Contactor II. $Q_c = Q_d = 100$ l/hr.

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Table 3C Sauter Mean Drop Diameter, Interfacial Area and Mass Transfer Coefficient for Continuous Phase, Determined Experimentally and Photographic Studies for CCl₄ - HAC - H₂O System, Contactor II.

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<th>d₃₂ (cm)</th>
<th>a (cm²/cm³)</th>
<th>Kₐ x 10⁶ (sec⁻¹)</th>
<th>Kₐ x 10⁶ (cm/sec)</th>
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Table 4C  Experimentally Determined Number and Heights of Transfer Units and Mass Transfer Coefficients  \( R = 1 \), \( CCL_4 - HAC - H_2O \) System, Contactor II

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Table 5C Extraction Efficiency, Contactor II
CCL$_4$ - HAC - H$_2$O System.

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Extraction Efficiency $\eta_E = R \left( \frac{y_5 - y_1}{x_5} \right) \times 100$

$R = \frac{Q_c}{Q_d}$
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Table 7C Dry Power Study Contactor II (small)

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Table 8C Single Phase Mixing Power Study. Contactor II (small)

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<th>Mixing+Power $P_{M1}$ (Joules/sec)</th>
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Table 9C  Two Phase Mixing Power Study Contactor II (small) CCL₄ - HAC - H₂O System.

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<th>Q̇ P</th>
<th>P_T (J/sec)</th>
<th>P_M (J/sec)</th>
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P_Dry = Dry Power Consumption  
P_M1 = Single Continuous Phase Mixing Power  
P_M2 = Single Dispersed Phase Mixing Power (By difference)  
P_M = Two Phase Mixing Power = P_M1 + P_M2  
P_T = Total Power Consumption = P_d + P_M = P_d + P_M1 + P_M2
Table 10C  Two Phase Mixing Power Per Unit Active
and Swept Volume CCl₄ - HAC - H₂O
System Contactor II

<table>
<thead>
<tr>
<th>N (opm)</th>
<th>Q_c = Q_d (l/hr)</th>
<th>P_va (J/sec m³)</th>
<th>P_vs (J/sec m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>80</td>
<td>50</td>
<td>162.55</td>
<td>276.13</td>
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<td>75</td>
<td>162.93</td>
<td>276.77</td>
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<td>100</td>
<td>163.73</td>
<td>278.14</td>
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<td>125</td>
<td>164.11</td>
<td>278.77</td>
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<tr>
<td>60</td>
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<td>89.22</td>
<td>151.56</td>
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<tr>
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<td>100</td>
<td>307.13</td>
<td>521.75</td>
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<tr>
<td>120</td>
<td>100</td>
<td>500.33</td>
<td>849.95</td>
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