The University of British Columbia

FACULTY OF GRADUATE STUDIES

PROGRAMME OF THE

FINAL ORAL EXAMINATION

FOR THE DEGREE OF

DOCTOR OF PHILOSOPHY

of

JEFFREY ERNEST HENTON

B.Sc., University of Leeds, 1964

MONDAY, OCTOBER 2, 1967, AT 3:30 P.M.

IN ROOM 207 CHEMICAL ENGINEERING BUILDING

COMMITTEE IN CHARGE

Chairman: B. N. Moyls

Dr. K.L. Pinder Dr. N. Epstein
Dr. J.S. Forsyth Dr. E. Peters
Dr. R.M.R. Branion Dr. A. Mitchell

External Examiner: T. Vermeulen
Professor, Department of Chemical Engineering
University of California,
Berkeley, California; U.S.A.

Research Supervisor: S.D. Cavers
BACKMIXING IN LIQUID-LIQUID EXTRACTION SPRAY COLUMNS

ABSTRACT

Backmixing of the continuous phase was studied in liquid-liquid spray columns of various geometries, for various flowrates of the two phases, and for various drop size distributions.

The dispersion or eddy diffusion model was used to characterize the axial mixing of the continuous phase, from a distributor of sodium chloride tracer (soluble in the continuous phase only). The steady state form of the model was utilized to calculate axial eddy diffusivities from these results.

The tracer studies showed that the axial eddy diffusivity is independent of the continuous phase flowrate and the column height. Axial eddy diffusivities between 7-ft.²/hr. and 31-ft.²/hr. were obtained in a 1½-in. I. D. column. Low dispersed phase flowrates and large drop sizes resulted in high axial eddy diffusivities. Increasing the column diameter to 3-in. resulted in superficial axial eddy diffusivities between 6.3 and 17.3 times larger.

The hold-up of dispersed phase was measured by means of a piston sampler. The hold-up increases
approximately linearly with increasing dispersed phase superficial velocity and tends to be slightly higher for increased continuous phase superficial velocities. A smaller drop size resulted in an increased hold-up.

Drop size distributions were measured. They always show two peaks, one at 0.02-in. diameter, and the other at a much larger size, the actual value of which depends on the nozzle tip diameter used to disperse the drops.

The mixing cell-packed bed analogy was used to predict Peclet numbers in a spray column. The agreement between these and measured Peclet numbers is good for drops of about 0.15-in. equivalent diameter but becomes progressively worse as the drop size is reduced.

AWARDS

1964-1967 Commonwealth Scholarship
1966-1967 Finning Tractor Graduate Scholarship
GRADUATE STUDIES

Field of Study: Chemical Engineering

Related Studies

- Mass Transfer: S.D. Cavers
- Statistics: D.A. Ratkowsky
- Fluid and Particle Dynamics: R.M.R. Branion
- Linear Algebra: Wm.R. Simons

Other Studies

- Mass and Heat Transfer Analogies: N. Epstein
- Surface Effects: J. Leja
- Optimization: K.L. Pinder
- Computer Programming: K. Teng
BACK-MIXING IN LIQUID-LIQUID EXTRACTION SPRAY COLUMNS

by

JEFFREY ERNEST HENTON

B. Sc., University of Leeds, 1964

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF

THE REQUIREMENTS FOR THE DEGREE OF

DOCTOR OF PHILOSOPHY

in the Department

of

CHEMICAL ENGINEERING

We accept this thesis as conforming to the required standard

THE UNIVERSITY OF BRITISH COLUMBIA

September, 1967
In presenting this thesis in partial fulfilment of the requirements for an advanced degree at the University of British Columbia, I agree that the Library shall make it freely available for reference and study. I further agree that permission for extensive copying of this thesis for scholarly purposes may be granted by the Head of my Department or by his representatives. It is understood that copying or publication of this thesis for financial gain shall not be allowed without my written permission.

Department of **CHEMICAL ENGINEERING**

The University of British Columbia
Vancouver 8, Canada

Date 2 October 1967
ABSTRACT

Backmixing of the continuous phase was studied in liquid-liquid spray columns of various geometries, for various flowrates of the two phases, and for various drop size distributions.

The dispersion or eddy diffusion model was used to characterize the axial mixing of the continuous phase. Axial concentration profiles were measured upstream, with respect to the continuous phase, from a distributor of sodium chloride tracer (soluble in the continuous phase only). The steady state form of the model was utilized to calculate axial eddy diffusivities from these results.

The tracer studies showed that the axial eddy diffusivity is independent of the continuous phase flowrate and the column height. Axial eddy diffusivities between 7-ft²/hr. and 31-ft²/hr. were obtained in a 1½-in. I. D. column. Low dispersed phase flowrates and large drop sizes resulted in high axial eddy diffusivities. Increasing the column diameter to 3-in. resulted in superficial axial eddy diffusivities between 6.3 and 17.3 times larger.

The hold-up of dispersed phase was measured by means of a piston sampler. The hold-up increases approximately linearly with increasing dispersed phase superficial velocity and tends to be slightly higher for increased continuous phase superficial velocities. A smaller drop size resulted in an increased hold-up.
Drop size distributions were measured. They always show two peaks, one at 0.02-in. diameter, and the other at a much larger size, the actual value of which depends on the nozzle tip diameter used to disperse the drops.

The mixing cell-packed bed analogy was used to predict Peclet numbers in a spray column. The agreement between these and measured Peclet numbers is good for drops of about 0.15-in. equivalent diameter but becomes progressively worse as the drop size is reduced.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Pages</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>INTRODUCTION</strong></td>
<td></td>
</tr>
<tr>
<td>- Previous work</td>
<td>1</td>
</tr>
<tr>
<td>- Mathematical models</td>
<td>19</td>
</tr>
<tr>
<td>- Object of this work</td>
<td>27</td>
</tr>
<tr>
<td><strong>THEORY</strong></td>
<td></td>
</tr>
<tr>
<td>- Application of the dispersion model to runs</td>
<td></td>
</tr>
<tr>
<td>with no mass transfer</td>
<td>30</td>
</tr>
<tr>
<td>- Application of the dispersion model to</td>
<td></td>
</tr>
<tr>
<td>liquid-liquid extraction</td>
<td>32</td>
</tr>
<tr>
<td><strong>APPARATUS</strong></td>
<td></td>
</tr>
<tr>
<td></td>
<td>36</td>
</tr>
<tr>
<td><strong>EXPERIMENTAL PROCEDURE</strong></td>
<td></td>
</tr>
<tr>
<td>- Column operation</td>
<td>60</td>
</tr>
<tr>
<td>- Sampling technique studies with mass transfer</td>
<td>61</td>
</tr>
<tr>
<td>- Search for a suitable tracer</td>
<td>67</td>
</tr>
<tr>
<td>- Sampling technique studies with no mass</td>
<td>69</td>
</tr>
<tr>
<td>transfer</td>
<td></td>
</tr>
<tr>
<td>- Axial eddy diffusivity and dispersed phase</td>
<td></td>
</tr>
<tr>
<td>hold-up</td>
<td>70</td>
</tr>
<tr>
<td>- Concentration profiles with mass transfer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>84</td>
</tr>
<tr>
<td><strong>RESULTS AND DISCUSSION</strong></td>
<td></td>
</tr>
<tr>
<td>- Results of sampling technique studies</td>
<td>86</td>
</tr>
<tr>
<td>- Discussion of sampling technique studies</td>
<td>91</td>
</tr>
</tbody>
</table>
Axial eddy diffusivity, drop size distribution, and dispersed phase hold-up studies in the 1 \( \frac{1}{2} \)-in. I. D. column.......................... 104

- Axial eddy diffusivity and drop size distribution studies in the 3-in. I. D. column........................................... 143

- Visual observations of the motion of the drops 150

- Concentration profiles with mass transfer...... 151

CONCLUSIONS................................................................. 164

NOMENCLATURE............................................................... 168

LITERATURE CITED.......................................................... 173

APPENDICES

I DISPERSION MODEL THEORY............................................. 183

II MIXING CELL - PACKED BED ANALOGY and SPRAY COLUMN - PACKED BED ANALOGY...................................................... 189

III ANALYSIS OF PISTON SAMPLE RESULTS TO PRODUCE THE AVERAGE CONTINUOUS PHASE CONCENTRATION, EXCLUDING THE CONTRIBUTION FROM WAKES, IN THE PISTON SAMPLE AT THE TIME OF SAMPLING.................................................. 197

IV TABULATED RESULTS.................................................... 203

V DETAILS OF THE APPARATUS.......................................... 230

VI DIMENSIONS OF THE GLASS PORTIONS IN THE COLUMN TEST SECTIONS AND MEASUREMENT OF PURGE TIMES............. 256
LIST OF TABLES

1. Key to Figure 9................................. 38
2. Key to Figure 21............................... 56
3. Sampling Studies with Hook and Bell-Probes and Piston............................... 89
4. Sampling Studies with Hook and Bell-Probes, Hypodermic Needles, and Piston............. 92
5. Sampling Technique Studies with No Mass Transfer......................... 94
6. Time to Reach Steady State in the 1 1/2-in. I. D. Column under Conditions of no Mass Transfer................. 135
7. Effect of Tracer Feed Rate on Reduced Concentration Profiles and Axial Eddy Diffusivity in the 1 1/2-in. I. D. Column................................. 136
8. Reproducibility of Results for Axial Eddy Diffusivity in the 1 1/2-in. I. D. Column......................... 137
9. Cross-Sectional Homogeneity in the 1 1/2-in. I. D. Column................................. 139
10. Effect of Sampling Rate on the Reduced Concentration Profile in the 1 1/2-in. I. D. Column......................... 140
11. Effect of Order of Sampling on the Measured Concentration Profile in the 1 1/2-in. I. D. Column......................... 142
12. Effect of Column Height on the Measured Concentration Profile and Axial Eddy Diffusivity in the 1 1/2-in. I. D. Column................................. 142
13. Steady State Times for the 3-in. I. D. Column

14. Reproducibility of Results for the 3-in. I. D.
   Column

15. Cross-Sectional Homogeneity in the 3-in. I. D.
   Column

16. Comparison of Axial Eddy Diffusivity by Mass
   Transfer Studies and Tracer Studies

IV-1. Data Sheet

IV-2. Calculation of Quantities used for the Calculation
   of E

IV-3. Typical Computer Results

IV-4. Axial Eddy Diffusivity Results for the 1\(\frac{1}{2}\)-in.
   I. D. Column

IV-5. Superficial Axial Eddy Diffusivity Results for the
   3-in. I. D. Column

IV-6. Typical Drop Size Distribution Results

IV-7. Drop Size Distributions in the 1\(\frac{1}{2}\)-in. I. D. Column

IV-8. Drop Size Distributions in the 3-in. I. D. Column

IV-9. Concentration Studies with Mass Transfer in the
   1\(\frac{1}{2}\)-in. I. D. Column

IV-10. The Relation Between E and \(\Delta^2\)

IV-11. Calculated Values of E for Various Values of
       \(K_{p,a}\) and J
IV-12. Calculated Values of E for Various Values of m...... 228

IV-13. Equilibrium Data for Acetic Acid Distributed
Between MIBK-Saturated Water and Water-Saturated
MIBK at 70°F............................................. 229

VI-1. Dimensions of the Glass Portions in the Column
Test Sections.............................................. 256
## LIST OF FIGURES

1. Schematic Diagram of a Spray Column ....................... 2  
2. Solute Concentration Profile in a Spray Column........... 5  
3. Mass Balance Over a Section of a Spray Column .......... 6  
4. Continuous Phase Recirculation ............................ 7  
5. Hook and Bell-Probes ....................................... 12  
6. Concentration Profiles in a Spray Column ................. 14  
7. Concentration Profiles In a Spray Column ....... 15  
8. Piston Sampler ............................................... 17  
9. Schematic Flow Diagram for the 1\frac{1}{2}-in. I. D. Column.. 37  
10. 1\frac{1}{2}-in. I. D. Column .................................. 41  
11. Short 1\frac{1}{2}-in. I. D. Column ............................... 43  
12. Long 1\frac{1}{2}-in. I. D. Column ............................... 44  
13. 1\frac{1}{2}-in. I. D. Column for Comparing Hook and Bell-Probes and Piston Sampler Results ............................... 45  
14. 1\frac{1}{2}-in. I. D. Column for Comparing Hypodermic Needle, Hook and Bell-Probes; and Piston Sampler Results .... 46  
15. Nozzle Tip Patterns. I. D. = 0.126-in. (1\frac{1}{2}-in. I. D. Column) ......................................................... 48  
16. Nozzle Tip Patterns. I. D. = 0.103-in. (1\frac{1}{2}-in. I. D. Column) ......................................................... 49
17. Nozzle Tip Patterns. I. D. = 0.086-in. (1\(\frac{1}{2}\)-in. I. D. Column) ................................................................. 50
18. Nozzle Tip Patterns. I. D. = 0.053-in. (1\(\frac{1}{2}\)-in. I. D. Column) ................................................................. 51
19. Photographic Conditions ................................................................. 53
20. 3-in. I. D. Column ........................................................................ 54
22. Nozzle Tip Patterns. I. D. = 0.102-in. (3-in. I. D. Column) .......... 58
23. Optical Distortion Investigation, 1\(\frac{1}{2}\)-in. I. D. Column ............. 75
24. Optical Distortion Investigation, 3-in. I. D. Column .................. 81
25. Sampling Technique Studies with Hook and Bell-Probes and Piston ................................................................. 90
26. Sampling Technique Studies with Hook and Bell-Probes, Hypodermic Needles and Piston ................................................................. 93
27. Sampling Technique Studies with no Mass Transfer ............... 95
28. Reduced Concentration Profiles .................................................... 108
29. Axial Eddy Diffusivity in the 1\(\frac{1}{2}\)-in. I. D. Column ................. 110
30. Comparison of Axial Eddy Diffusivity as Determined in this Work with That of Other Workers ................................. 112
30a. Comparison of Dispersion Number in This Work with That for Packed Beds ................................................................. 113
31. Predicted and Calculated Peclet Numbers, 
\[ d_p = 0.155 - \text{in.} \]

32. Predicted and Calculated Peclet Numbers, 
\[ d_p = 0.135 - \text{in.} \]

33. Predicted and Calculated Peclet Numbers, 
\[ d_p = 0.125 - \text{in.} \]

34. Predicted and Calculated Peclet Numbers, 
\[ d_p = 0.095 - \text{in.} \]

35. Percentage Error in the Equivalent Diameter due to Optical Distortion in the 1\(\frac{3}{4}\)-in. I. D. Column

36. Drop Size Distribution for Run 50, Average Nozzle Tip Diameter = 0.103-in.

37. Photographs at Operating Conditions Corresponding to Runs Indicated. Magnification Factor = 3

38. Drop Size Distribution for Run 130, Average Nozzle Tip Diameter = 0.126-in.

39. Distribution of Total Percent of Volume of Drops for Run 50, Average Nozzle Tip Diameter = 0.103-in.

40. Dispersed Phase Hold-up, \[ d_p = 0.155 - \text{in.} \]

41. Dispersed Phase Hold-up, \[ d_p = 0.135 - \text{in.} \]

42. Dispersed Phase Hold-up, \[ d_p = 0.125 - \text{in.} \]

43. Dispersed Phase Hold-up, \[ d_p = 0.095 - \text{in.} \]

44. Superficial Axial Eddy Diffusivity in the 3-in. I. D. Column

45. Percentage Error in the Equivalent Diameter due to Optical Distortion in the 3-in. I. D. Column
46. Equilibrium Curve for Acetic Acid Distributed Between MIBK- Saturated Water and Water-Saturated MIBK at 70°F ........................................... 153
47. Minimizing $\Delta^2$ for Run Jl ......................... 158
48. The Effect on E of Varying the Method of Flux Calculation and of Varying $K_a$ for Run Jl ................. 159
49. The Effect on E of Variations in m for Run Jl ........ 160
50. Measured and Fitted Concentration Profiles for Run Jl .................................................. 161

I-1. Solute Mass Balance in the Continuous Phase .......... 184
III-1. Lower Portion of a Spray Column .................. 201
III-2. The Effect of Time on a Piston Sample .............. 202

V-1. Piston Sample Collection Flask for Large Hold-ups ... 233
V-2. Hypodermic Needle Installed for Sampling .............. 234
V-3. Sampling Valve for Hypodermic Needle ................ 235
V-4. Tracer Injection System .................................. 236
V-5. Tracer Distributor ............................................. 237
V-6. 0.126-in. I.D. Nozzle Tips, Nozzle Tip Support Plate, and Nozzle Tip Caps .......................... 238
V-7. 0.086-in. I.D. Nozzle Tips, Nozzle Tip Support Plate, and Nozzle Tip Plugs ..................... 239
V-8. 0.053-in. I.D. Nozzle Tips, Nozzle Tip Support Plate and Nozzle Tip Plugs ..................... 240
V-9. Perspex Box for the 1½-in. I.D. Column Photographs... 241
V-10. Light Shield for the 1\(\frac{3}{4}\)-in. I. D. Column Photographs. 242
V-11. Lower end of the 3-in. I. D. Column ................. 243
V-12. Special Pyrex Reducer for the 3-in. I. D. Column.... 244
V-13. Nozzle Shell for the 3-in. I. D. Column ............... 245
V-15. Flow Straightener for the 3-in. I. D. Column Nozzle.. 247
V-16. Nozzle Securing Plate and Retaining Nut for the 3-in. I. D. Column ........................................ 248
V-17. End Plate for the Bottom of the 3-in. I. D. Column... 249
V-18. Elgin Head for the 3-in. I. D. Column ................. 250
V-19. Upper End Plate for the Elgin Head of the 3-in. I. D. Column .................................................. 251
V-20. Lower End Plate for the Elgin Head of the 3-in. I. D. Column .................................................. 252
V-21. Lower End Plate Packing for the Elgin Head of the 3-in. I. D. Column ........................................ 253
V-22. Perspex Box for the 3-in. I. D. Column Photographs... 254
V-23. Flange for the Photographic Section of the 3-in. I. D. Column .................................................. 255
ACKNOWLEDGEMENTS

The author would like to express his sincere gratitude to Dr. S. D. Cavers for his assistance, encouragement, and helpful criticisms offered throughout the course of this project.

Thanks are extended to all members of the faculty and staff of the Department of Chemical Engineering, The University of British Columbia for their readiness and willingness in discussing both theoretical and practical problems associated with the work. Particular appreciation is offered to Dr. K. L. Pinder for the loan of a telephoto lens and to Mr. R. Brandt for making most of the machined parts essential for this study.

The author is indebted to Dr. Duncan of the British Columbia Research Council for his suggesting the use of sodium chloride as a tracer and for providing the use of the atomic absorption spectrophotometer.

Financial support was most gratefully received in the form of scholarships from the Commonwealth Scholarship and Fellowship Committee of the External Aid Office, Ottawa, and from the Finning Tractor Company, Ltd. Funds for the equipment were provided by the National Research Council.
INTRODUCTION

1. PREVIOUS WORK

Liquid-liquid extraction is used widely for separating components which are more difficult or expensive to separate by other methods such as distillation, evaporation, or precipitation. Extraction processes usually are preferred if the two components to be separated have similar boiling points, or if one of the components is heat sensitive or present in small amounts.

Countercurrent extraction in vertical towers can be carried out when the raffinate and extract phases differ appreciably in density. Although sieve-plate, bubble cap, and packed towers are more commonly found in industrial use "...the spray tower is the more attractive for experimentation because of its inherent simplicity, and also because of the greater possible range of flowrates of the two (phases)..." (1)

Figure 1 shows diagrammatically how each phase is introduced into and removed from a spray tower. In the system shown the less dense phase is dispersed through nozzle tips located at the lower end of the column. The dispersed phase drops rise through the descending continuous phase and coalesce at the interface at the upper end of the column. Many workers have studied spray column operation. The knowledge gained in the main represents an accumulation of small contributions.
Figure 1. Schematic Diagram of a Spray Column
The designs of the continuous phase inlet and dispersed phase outlet used in the present study were first proposed by Blanding and Elgin (2). Optimum nozzle tip diameters and dispersed phase flowrates in the nozzle tips for reproducible, more or less uniform drop size have been suggested by Johnson and Bliss (3) and later by others (4,5,6,7,8).

From about the early 1930's workers began to perform laboratory scale experiments in attempts to discover simple laws or to formulate correlations which applied to spray tower operation (2,3,9,10,11,12,13). Their results indicate that the extent of extraction is dependent upon the flowrates of the two phases, the direction of solute transfer (f.e. from dispersed phase to continuous phase or vice versa), which phase is dispersed, drop size, column dimensions, and sometimes upon inlet solute concentrations. All these workers analysed only the column inlet and outlet solute concentrations of each phase.

Licht and Conway (14) in 1950 pointed out that the mass transfer process should be considered as taking place in three separate stages - i) drop formation, ii) drop rise, and iii) drop coalescence. Geankoplis and Hixson (1), also in 1950, revolutionized techniques in spray column experimentation by taking samples of the continuous phase from within an operating column. This was accomplished by lowering a hook-shaped sampling probe, (as shown in the sketch below) into the column and then applying a slight vacuum to the upper end of the probe. Continuous phase was drawn into the probe without entraining any dispersed phase.
They observed that the basic flow pattern within the column did not appear to be affected by the probe. In 1951 Geankoplis, Wells and Hawk (15) extended Geankoplis and Hixson's work, again using an internal sampling probe, to measure the solute concentration profile in the continuous phase. A typical experimental solute concentration profile in the continuous phase is shown in Figure 2.

Geankoplis and co-workers noticed a sharp change, or end-effect, in solute concentration in the continuous phase at the interface. They thought "...that the location of the end effect at the continuous phase inlet may be caused by the inherent turbulence effect of coalescence of bubbles at the interface..." (15). They made an allowance for this end effect by calculating a fictitious height of column in which there would occur the same amount of mass transfer as appeared to occur at the interface. To calculate solute concentrations in the dispersed phase at various elevations in the column the use of mass balances was attempted. On the basis that the superficial flowrates of both phases
are not affected by solute concentration changes experienced in the column, a material balance on solute around the control zone shown in Figure 3 resulted in the following equation.

\[ L_D c_D + L_C c_C^o = L_C c_C + L_D c_D^i \]

Thus

\[ c_D = \frac{L_C}{L_D} (c_C - c_C^o) + c_D^i \]
FIGURE 3. MASS BALANCE OVER A SECTION OF A SPRAY COLUMN.

Although some turbulence was observed in the continuous phase (1), no allowance for its effect was included in the mass balance. Geankoplis continued his work involving continuous phase sampling with Kreager (16) and later with Vogt (17).

Morello and Poffenberger (18) were the first to suggest positively that the continuous phase did not move through the column in effective plug flow, but that there was recirculation within that phase. They said that the recirculation, which was later to be called backmixing, may be caused by thermal currents, density differences or by the friction of the drops carrying some of the continuous phase along with them.
They portrayed the idea diagrammatically as shown in Figure 4.

![Diagram of Continuous Phase Recirculation](image)

**FIGURE 4. CONTINUOUS PHASE RECIRCULATION.**

It can be seen that the simple mass balance given in Equation 1 based on plug flow of both phases is not valid.

Although Morello and Poffenberger (18) gave a physical picture of backmixing, Newman (19) pointed out that the end-effect which had been observed by earlier workers could be explained in terms of backmixing of the continuous phase. Also he presented the following argument to show that the solute concentration in the dispersed phase at some point in the column cannot be calculated by a mass balance over short sections of the column. Newman accounted for backmixing by considering a flow of continuous phase countercurrent to the main flow of continuous phase.
This backmixing flow was exactly compensated by an increased main flow of continuous phase. It was assumed that the resulting main flow of continuous phase was radially homogeneous at a given column elevation. The plug flow model and Newman's backmixing model are shown in the following sketch.

For plug flow of both phases, as shown in (a) of the above sketch, \( c_D \) is given by Equation 2.

\[
c_D = \frac{L_c}{L_D} (c_C - c_C^0) + c_D^i
\]

For backmixing of the continuous phase, as shown in (b) above, a solute mass balance over the lower section of column yields

\[
L_D c_D^i + (L_C + L_B) c_C = L_D c_C^D + L_C c_C^0 + L_C c_C
\]
Thus

\[ c_D = \frac{L_C}{L_D} (c_C^0 - c_C^0) + \frac{c_D^D}{L_D} + \frac{L_B}{L_D} (c_C - c_B) \]

As \( L_B \) and \( c_B \) cannot be determined it is not possible to use this equation to obtain \( c_D \). For the same reason capacity coefficients and H.T.U. values for short sections of column cannot be derived from the knowledge of terminal conditions and the solute concentration profile in the continuous phase.

Campos (20) calculated the height of tower in which there would occur the same amount of mass transfer as appeared to have produced the end-effect at the continuous phase inlet of the column. He noted also that backmixing of the continuous phase may cause end-effects in spray towers. The existence of a backmixing stream in the form of wakes travelling with the dispersed phase drops has been demonstrated photographically (21, 22, 23, 24), and Li and Ziegler (25) say "In general the process of backmixing in spray towers is believed to be initiated largely in the wakes of droplets". Letan and Kehat (26, 27) have worked with spray column heat exchangers. They explain continuous phase backmixing effects by means of a model in which continuous phase is supposed to be carried along in the form of wakes, with the dispersed phase drops.

Gier and Hougen (28) used hypodermic syringes to draw off continuous phase samples and bell-shaped probes to collect dispersed phase samples.
The accompanying sketch shows an example of each sort of probe used by them. Probes of each sort were distributed along the length of the column.

A bell-probe sample contained both dispersed and continuous phases. The sample was allowed to reach equilibrium* and then each phase was analysed for solute. A simple mass balance on solute in the bell-probe sample at the times of collection and analysis results in Equation 5.

\[ V_D c_D + V_C c_C = V_D c_D + V_C c_C \]

Therefore

\[ c_D = c_D^a + V_C \left( c_C^a - c_C \right) \]

In order to calculate \( c_D \) from Equation 6 an estimate of \( c_C \), at the elevation of the bell-probe, was made from a plot of the concentration of solute in the hypodermic syringe samples versus column height. 

* i.e. no change in concentration of either phase with time.
Gier and Hougen calculated values of H.T.U., based on concentration differences in the dispersed phase, by graphical integration using the measured concentration profiles. Their Figure 17 shows the solute concentration in the dispersed phase as determined by means of Equation 6 for one of their runs in which solute was transferred from the continuous phase to the dispersed phase. These concentrations are greater than those which could be obtained from calculation by means of Equation 2 which is based on plug flow of both phases. This result is in agreement with Newman's suggestions (Equation 4). As a result the H.T.U. values calculated by graphical integration were correspondingly lower than those calculated from the column terminal conditions assuming plug flow of both phases. Patton (29) took samples from an operating spray column using the hypodermic needle and inverted funnel technique of Gier and Hougen. He found a considerable drop in the continuous phase solute concentration at the interface which was not matched by a proportional drop in the solute concentration in the dispersed phase. He concluded that the discontinuity in the solute concentration profile in the continuous phase at the interface was due to bulk mixing of the continuous phase in the column.

A development of the sampling technique of Geankoplis and coworkers (1) and of Gier and Hougen (28) was used by Ewanchyna and Cavers (30, 31). They made use of a hook-shaped probe for continuous phase sampling and a bell-shaped probe for dispersed phase sampling. The probes were lowered into the operating column at the ends of stainless steel tubes as shown in Figure 5. Samples were drawn into the probes and
FIGURE 5. HOOK AND BELL-PROBES
and along the sample lines by the use of a water aspirator. Acetic acid was transferred between an aqueous continuous phase and a methyl isobutyl ketone (MIBK) dispersed phase. The aqueous phase was saturated with MIBK and the MIBK phase saturated with water. The solute concentration profile in each phase was measured at various combinations of phase flow rates. Plots were made showing the dependence of capacity coefficients on flow rates and also of H.T.U. values on flow rates. The causes of end effects in spray columns were explained clearly and verified experimentally.

The measured concentration profiles for a typical run with solute being transferred from the continuous aqueous phase to the dispersed MIBK phase are shown in Figure 6. The lines FB and GE are the measured concentration profiles for the dispersed phase and the continuous phase respectively. It was assumed that no backmixing took place in the dispersed phase. This assumption was based on the visual observation that the drops appeared to rise up the column without circulating back on their paths (28, 30, 31). If it is assumed in addition that no backmixing takes place in the continuous phase then Equation 2, which is based on plug flow of both phases, can be used to calculate the line GD. When the drops of dispersed phase arrive at the interface they do not coalesce immediately but remain as part of a drop layer there. Undoubtedly mass transfer takes place into these drops during their sojourn at the interface. Ewanchyna and Cavers attributed the concentration jumps BA (in the dispersed phase) and CD to this interface mass transfer.
They attributed the change DE to axial mixing of the continuous phase. The line GH is the concentration profile which would be expected for perfect mixing of that phase under conditions such that the solute concentration in the aqueous stream leaving the column was that given by point G. It can be seen that the measured aqueous phase solute concentration profile (GE) lies between that expected for perfect mixing (GH) and that for true countercurrent flow (GD). Evidently the continuous phase does undergo some axial mixing.
Ewanchyna and Cavers (30, 31) found that the results of runs with solute transferred from the dispersed phase to the continuous phase supported the above ideas. With solute transferred to the continuous phase drops coalesce immediately on reaching the interface (31, 32, 33, 34). As a result there was negligible mass transfer at the interface and hence no jump in the dispersed phase concentration profile there. The results of a typical run are shown in Figure 7.

**Figure 7.** Concentration profiles in a spray column.
The whole of the solute concentration change (DE) in the continuous phase at the interface was attributed to backmixing of the continuous phase in the column.

Choudhury (35) continued Ewanchyna's work. To allow for backmixing of the continuous phase in the derivation of an expression for (H.T.U.) overall values, based on a logarithmic mean dispersed phase concentration driving force, he introduced a correction factor, $F_m$, as Pratt (36) had done for packed towers.

The question was raised as to whether the solute concentration in the continuous phase entering the bell-probe with the drops was the same as that entering the hook-probe. It was thought that the bell-probe might sample preferentially continuous phase which was in the immediate vicinity of the drops. If this continuous phase were not of the same solute concentration as that in the main bulk of the continuous phase the use in Equation 6 of $c_c$ from a hook-probe sample in order to calculate $c_D$ in a bell-probe sample would be invalid.

In order to test the hook and bell sampling technique Hawrelak (37) designed and constructed a piston sampler. A sketch of this device is shown in Figure 8. By moving the piston from one side of the piston block to the other it was possible to remove a bulk sample of both phases from the column and allow the column to continue operating. With this device Hawrelak (37), and later Bergeron (38),
compared solute concentrations in the dispersed phase calculated using the bell and hook-probes with those calculated from results using the piston sampler and the hook-probe. It was necessary to assume that the hook-probe sample was representative of the continuous phase in the column at the sampling height. However, no definite conclusions were drawn, mainly because the column was run under such conditions that the two phases were near equilibrium at the sampling elevation.

Rocchini (39, 40) studied drop shape and measured drop size distributions in a spray column by examining close-up photographs taken of an operating column.
Dispersed phase hold-ups in spray towers have been calculated largely from Equation 7 (3).

\[ h = \frac{100 L_D}{u} \]

However, Weaver, Lapidus and Elgin (41) determined hold-ups by isolating a section of column between two quick-acting ball valves and measuring the volume of each phase collected. Hawrelak (37) and Bergeron (38) measured dispersed phase hold-ups, without disrupting the column operation, with the aid of a piston sampler described earlier. It has been found (3, 30, 35, 42) that the dispersed phase hold-up increases only slightly with increasing continuous phase flowrate and is nearly linearly dependent on the dispersed phase flowrate.

Hayworth and Treybal (4) and Johnson and Bliss (3) have observed that the length of a jet of dispersed phase leaving a nozzle tip increases to a maximum and then decreases as the flowrate of dispersed phase increases. They noticed that the size of the drops formed at the ends of the jets of dispersed phase also increases to a maximum with increasing dispersed phase flowrate. However, the dispersed phase flowrate at which the maximum drop size occurs is lower than that at which the maximum jet length occurs. The claim was made that uniformly sized drops are produced at dispersed phase flowrates lower than those which give the maximum drop size. The formation of many small drops along with larger drops has been
observed under some operating conditions. Weaver, Lapidus, and Elgin found no noticeable change in drop size distribution along the length of a spray column. Garwin and Smith report that the dispersed phase drop size is independent of the continuous phase flowrate.

2. MATHEMATICAL MODELS

In order to describe fully a turbulent field it is necessary to know the velocity vector at all points and at all times. However, such knowledge is unavailable. Therefore a measurement of some effect of the turbulence usually is made in order to characterize the extent of the turbulence. Even when the flow patterns in the turbulent field are of a complicated nature a simple mathematical model often adequately describes the mixing taking place. In the past the design of flow reactors has been based largely upon two idealized models: plug flow, and completely mixed flow. Plug flow assumes a flat velocity profile, whereas completely mixed flow assumes that the fluid in the vessel is perfectly mixed. The three most popular models which lie between the two extreme cases are the dispersion, mixing cell, and random walk models. For the case of fluid flow through a long column or bed the mathematical implications of all three models are essentially the same. However, there are fundamental differences between the premises upon which the models are based. Brief outlines of these models are presented below together with summaries of some of the more important researches.
which have utilized them for interpreting experimental data. Axial mixing of only one phase is considered in each case.

DISPERSION MODEL

It is assumed that axial mixing due to turbulence follows a law similar to Fick's Law for molecular diffusion. On this basis a mass balance on solute in the continuous phase over an incremental section of column yields Equation 8 for the case of no mass transfer to or from the continuous phase. (Also see Appendix 1.)

\[
\frac{\partial^2 c}{\partial z^2} - \frac{\partial c}{\partial z} \frac{\partial}{\partial t} = \epsilon \frac{\partial c}{\partial t}
\]

It is assumed that radial homogeneity prevails. Wilson (47) solved an equation similar to Equation 8 for molecular diffusion of heat from a point source into a flowing fluid. Wilson's solution has been used together with experimental data (48, 49, 50) to calculate eddy diffusivities in open ducts on the assumption of isotropic homogeneous turbulence. Bernard and Wilhelm (51) used a similar method to determine eddy diffusivities in packed columns.

In 1949 Gilliland and Mason (52) applied the model to the steady state operation of an air-fluidized bed. At steady state \(c_C\) is independent of \(t\) and Equation 8 can be integrated (Appendix I) to give

\[
\frac{E \epsilon c}{d} = \frac{L c}{C} - J
\]
where the constant of integration, \( J \), is the net flux of solute down the column. If the test section is, from the viewpoint of the continuous phase, upstream from the feed point of tracer, then

\[ J = 0 \]

and Equation 9 yields Equation 10 on integration.

\[
\ln \left( \frac{c_c}{c_{CO}} \right) = \frac{L_c z}{Ee}
\]

Gilliland and Mason (52, 53) injected a tracer gas at a constant rate into the bed and measured tracer concentrations at various points in the bed below the level of tracer injection. From the slope of a plot of \( \ln (c_c) \) versus \( z \) they were able to calculate the superficial axial eddy diffusivity, \( (eE) \). The above method for estimating axial eddy diffusivities cannot be utilized for the cases of single phase or co-current flows because the process of axial mixing would not, in this case, carry the tracer upstream in the continuous phase with respect to the tracer injection point.

The model has been applied subsequently to the study of pulsed sieve-plate extraction columns (54, 55), gas-sparged tubular vessels (56), rotating-disk contactors (57, 58), and commercial fluidized bed catalyst regenerators (59).

Continuous phase axial eddy diffusivities have been determined mostly by considering the effect downstream, with respect to the continuous phase, of an unsteady state injection of tracer. Equation 8 has been solved for injection of tracer according to a Dirac
Delta Function or single pulse, a step function, and a sinusoidal function. These three different methods of tracer injection are discussed briefly below.

Dirac Delta Function

With no axial mixing a transverse-homogeneous single pulse of tracer injected into a moving stream would appear downstream as a single pulse at all times. With some axial mixing, spreading with respect to distance within the column of the tracer pulse results. Danckwerts (60) and Levenspiel and Smith (61) showed how to interpret the plot of tracer concentration versus time for some point downstream from the place of tracer injection. Such a plot is called a C-curve (60) and the second moment or variance of this curve is related to the axial eddy diffusivity. Axial eddy diffusivities have been determined by the Dirac Delta Function method for packed columns (62, 63, 64), pulsed sieve-plate columns (54, 55, 100), coiled tube reactors (66), and liquid-fluidized beds (67).

Van der Laan (68) showed how to determine the axial eddy diffusivity from the variance of a C-curve for a finite length of vessel by using the appropriate boundary conditions (60, 69). Aris (70) pointed out that it is impossible to inject a perfect pulse of tracer into a column. Also he showed, however, that it is not necessary for the tracer injection to be in the form of a perfect pulse if one takes the difference of the second moments of the C-curves measured at two different points in the column. Aris'
theory was later corrected by Bischoff (71) who, together with Levenspiel (72, 73), presented the theory in detail. Axial eddy diffusivities in liquid-liquid spray columns at flooding conditions (74), in packed beds (75), and in orifice plate gas-liquid reactors (76) have been determined by taking the difference of the variances of C-curves.

Step Function

A step function of tracer can be introduced into a column by suddenly stopping or starting the flow of tracer. A plot of downstream tracer concentration versus time is called an F-curve (60) or breakthrough curve. The gradient of the F-curve at a particular value on the time axis is related to the axial eddy diffusivity. Gilliland and Mason (53) examined an F-curve for a fluidized bed in 1952, but at that time a theoretical analysis of such a curve had not been developed. In 1953 Danckwerts (60) showed how to calculate the axial eddy diffusivity from an experimental F-curve. Axial eddy diffusivities have been determined from breakthrough curves for packed beds (46, 60, 77, 78, 79, 80, 81, 82), spray columns (42), and rotating disk contactors (83). Brutvan (84) determined axial eddy diffusivities from breakthrough curves in a spray column where the dispersed phase was solid spheres. A statistical analysis of experimental breakthrough curves has been discussed by Klinkenberg (85). Levenspiel and Bischoff (73) indicated how to eliminate the effect on the F-curve of the particular method of tracer injection used. The procedure involved taking measurements at two elevations in a column. Miller and
King (86) examined the slopes of F-curves measured at two axial distances in a packed bed to calculate axial eddy diffusivities. It should be noted that the F-curve is the time integral of the C-curve (79). This fact has been demonstrated experimentally (87).

Sinusoidal Function

Under the influence of axial mixing in a column a sinusoidal function of tracer concentration suffers both attenuation and phase lag. Levenspiel and Bischoff (73) derived equations relating the Peclet number to the attenuation and phase lag of a sinusoidal input of tracer. The sinusoidal input or frequency response method has been used to determine Peclet numbers for gas flow and liquid flow through random and ordered packed beds (63, 88, 89, 90, 91, 92, 93, 94, 104). Ebach and White (63) showed how to evaluate results for periodic tracer input functions which are not sinusoidal.

In liquid-liquid extraction columns two phase countercurrent flow with mass transfer is encountered. The performance of such columns has been predicted on the basis of Equation 8 modified to include a mass transfer term (95, 96, 97, 98) as shown below:

$$\text{I-1}$$

$$\text{Ee} \left( \frac{\partial^2 c_C}{\partial z^2} \right) + L_C \left( \frac{\partial c_C}{\partial z} \right) - K_D a \left( \frac{c_C - c_D}{m} \right) = \delta \frac{\partial c_C}{\partial t}$$

Theoretical concentration profiles in extraction columns have been calculated for various operating conditions (99, 100, 101, 102, 103).
Theoretical concentration profiles have been compared with experimental profiles for various liquid-liquid extraction devices. Agreement was good for pulsed columns (58, 102) and moderately good for some conditions of spray column operation (103) and for gas absorption-tower operations (105).

Gottschlich (106) applied Equation 8 to a packed column. In doing so he modified the equation to take into account stagnant layers of fluid around the packing elements. He reports that the modification improves the agreement of experimental results and the theoretical model. Van Deemter, Zuiderweg, and Klinkenberg (107) used the dispersion model to correct for non-ideality in chromatography due to axial dispersion.

**MIXING CELL MODEL**

Kramers and Alberda (90) drew an analogy between single phase flow in a packed bed and a series of perfect mixing vessels. They assumed a long bed and restricted the magnitude of the particle Peclet number. On these bases they showed that the dispersion model and mixing cell model yield the same frequency response diagram for a sinusoidally varying tracer input. Other workers (44, 64, 92, 108, 109) have followed Kramers and Alberda's approach for single phase flow. It has been shown that for the analogy to be valid the Peclet number, $\text{Pe}'$, must be given by Equation 11,

$$\text{Pe}' = \frac{2}{\lambda}$$
where $\lambda$ is the ratio of the length of a mixing cell, $d_1$, to the characteristic packing dimension, $d_p$ (See Appendix II.) In a packed bed the axial distance between layers of packing is taken to be equal to the height of a mixing cell.

Aris and Amundson (108) and Jacques and Vermeulen (46) used the Poisson probability distribution function to describe the residence time distribution of a tracer molecule on the basis of the mixing cell model.

Epstein (110) has developed charts for a correction factor which modifies log mean driving forces for single phase flow in packed beds. The model is based on the analogy between a fixed bed of particles and a series of perfect mixers.

Theoretical arguments have been presented relating the dispersion model to a series of perfect mixing cells with backflow between mixers (45, 100, 111, 112, 113). Li and Ziegler (25) recently have published a review of the experimental and theoretical work done on the application of the dispersion and the mixing cell models to spray and packed towers.

RANDOM WALK MODEL

The theory for the random walk model is based on Einstein's statistical treatment (115). Small packets of fluid are assumed to move in an intermittent fashion, the motion being over a
distance, and lasting for a length of time, both of which are
taken to be purely random quantities. Jacques and Vermeulen (46)
show that the random walk model, the mixing cell model, and the
dispersion model result in the same mathematical equation for
residence time distributions. Axial eddy diffusivities for
packed beds have been determined using the random walk model and
input of tracer according to a step function (46, 116, 117).

OBJECT OF THIS RESEARCH

In 1963 Gerster (127) summarized the work which had been
done on the effect of axial mixing upon the performance of
extraction columns. He suggested that much further work in the
field of axial eddy diffusion was needed.

Previous work to determine axial eddy diffusivities in spray
columns has been carried out by Hazlebeck and Geankoplis (42)
and by Brutvan (84). The former investigation involved the use
of water as the aqueous phase and MIBK as the dispersed phase in
a 1½-in. I.D. column. Only one set of nozzle tips, which produced
drops of about 0.135-in. diameter, was used throughout the work.
The step function input application of the dispersion model was
used as described earlier. The axial eddy diffusivity was found
to increase linearly between 12-ft²/hr. and 22-ft²/hr. for
continuous phase superficial velocities between 10-ft³/hr. ft²
and 45-ft³/hr. ft² respectively. For a given continuous phase
superficial velocity the axial eddy diffusivity was constant for dispersed phase superficial velocities between $18.4 \text{-ft}^3/\text{hr. ft}^2$ and $50\text{-ft}^3/\text{hr. ft}^2$. Brutvan performed experiments in 1-in., $1\frac{1}{4}$-in., and 2-in. I.D. columns with water as the continuous phase and glass beads, of diameters 3, 4, 5, and 6-mm. respectively, as the dispersed phase. He, also, used the step function input application of the dispersion model. Various dispersed phase superficial velocities between $10\text{-ft}^3/\text{hr. ft}^2$ and $100\text{-ft}^3/\text{hr. ft}^2$ and continuous phase superficial velocities between $140\text{-ft}^3/\text{hr. ft}^2$ and $780\text{-ft}^3/\text{hr. ft}^2$ were studied. He found that the axial eddy diffusivity increased with increasing column diameter, increasing dispersed phase flowrate, decreasing continuous phase flowrate and decreasing dispersed phase particle size. Both the investigations of Hazlebeck and Geankoplis and of Brutvan did not take into account the problems associated with the production of a perfect step function as mentioned earlier. No work has been reported where axial eddy diffusivities in spray columns were determined by means of the steady state application of the dispersion model.

Accordingly, the object of the present work was to measure axial eddy diffusivities in an operating liquid-liquid spray column. The steady state form of the dispersion model was used in these investigations. Since little was known of the effect of various operating parameters on eddy diffusivity it was decided that the effects of flowrates of the two phases, column
length, and column diameter on the axial eddy diffusivity should be investigated. A minor objective in the form of a preliminary study was to find suitable methods for sampling the continuous and dispersed phases of an operating spray column.
THEORY

APPLICATION OF THE DISPERSION MODEL TO RUNS WITH NO MASS TRANSFER

In the present work the dispersion model described in the Introduction was used. The general assumptions upon which this model is based are listed below.

1. Backmixing of the continuous phase can be represented by Fick's Second Law of Diffusion with a constant axial eddy diffusivity throughout the column.
2. The molecular diffusivity of any solute considered is negligible compared to the axial eddy diffusivity.
3. Radial homogeneity prevails at constant axial position.
4. The velocity profile in the continuous phase is flat.
5. Volumetric flowrates are constant throughout the column.
6. A test section of column is considered in which no hydrodynamic property is affected by the column terminal conditions.
7. There is no backmixing of the dispersed phase.

Determination of Axial Eddy Diffusivities

In addition to the above assumptions the following assumptions were made for determining axial eddy diffusivities.

1. The axial eddy diffusivity in the continuous phase is not
affected by the chemical nature of the solute.

2. The solute dissolves only in the continuous phase.

The theory is developed for a test section of column which lies, from the viewpoint of the continuous phase, upstream from the point of injection of tracer. The downstream flow of tracer due to bulk flow of the continuous phase is equated to the backflow of tracer by axial eddy diffusion to give

\[ L_c c'_c = Ee \frac{dc_c}{dz} \]

Equation 12

\( E \) is the axial eddy diffusivity and \((Ee)\) is the superficial axial eddy diffusivity. Obviously Equation 12 results from Equation 8 at steady state. As mentioned earlier, also, the solution of Equation 12 is

\[ \frac{L_c}{Ee} \frac{z}{c_0} = \ln \left[ \frac{\left( \frac{c'}{c_0} \right)}{\left( \frac{c}{c_0} \right)} \right] \]

Equation 13

where \( c_{c0} \) is the value of \( c_c \) at \( z = 0 \). It can be seen from Equation 13 that a plot of \( \ln c_c \) versus \( z \) has a slope of \( \frac{L_c}{Ee} \) if the model holds good. Derivations of Equations 12 and 13 are given in Appendix I.

Prediction of Peclet Number by the Mixing Cell Analogy

As mentioned in the Introduction the analogy between single
phase flow through a packed bed and through a series of perfect mixers results in the following limitation on the particle Peclet Number, \( \text{Pe}' \). (See Appendix II.)

\[
\text{Pe}' = \frac{2}{\lambda}
\]

where

\[
\lambda = \frac{d_1}{d_p}
\]

where \( d_1 \) is the distance between layers of packing pieces.

It is suggested that the above analogy can be extended to spray column operation by allowing the co-ordinate axes of reference to move at the same velocity as the rising dispersed phase drops. As shown in Appendix II; on this basis a spray column tends, mathematically speaking, towards a packed bed. If the drops are assumed to be arranged in some simple lattice structure, \( \lambda \) can be expressed purely in terms of \( h \). Therefore the predicted drop Peclet number is a simple function of \( h \) only. Equations for predicting the drop Peclet number, \( \text{Pe} \), from the hold-up, \( h \), have been derived in Appendix II for six different lattice arrangements of drops.

APPLICATION OF THE DISPERSION MODEL TO LIQUID-LIQUID EXTRACTION

On the basis of the dispersion model the reduced concentration of solute in the continuous phase of a liquid-liquid extraction column is given by Equation 14.
\[ C_c = A \exp (\lambda_1 Z) + B \exp (\lambda_2 Z) - Q \]

where

\[ \lambda_1 = \alpha + \sqrt{\alpha^2 + \beta}, \]

\[ \lambda_2 = \alpha - \sqrt{\alpha^2 + \beta}, \]

\[ 2\alpha = \frac{L_c H + K_a H}{E_e} \frac{L_D}{L_D} \]

\[ \beta = \frac{(L_D - mL_c) K_a H^2}{mL_D E_e} \]

\[ Q = \frac{Jm}{c_{C0}} \frac{L_D - mL_c}{(L_D - mL_c)} \]

and A and B are constants of integration. Equations 14 to 19 inclusive correspond to Equations I-13, I-14, I-15, I-10, I-11, and I-16 in Appendix I.

If the solute concentration profiles are available for both phases of a liquid-liquid extraction spray column the capacity coefficient, \((K_D a)\), can be calculated by means of Equation 20.

\[ K_D a = \frac{L_D}{H} \int \frac{dc_D}{(c_C - c_D)} \]
The integral in Equation 20 is evaluated graphically over the length of test section, H. The volumetric fraction of continuous phase, \( e \), in the column can be measured by means of a piston sampler (37). For steady-state operation the net flux, \( J \), of solute down the column must be the same at any elevation in the column. \( J \) can be calculated from a knowledge of the superficial velocities of both phases and solute concentrations in the streams entering and leaving the column by means of Equation 21.

\[
J = \frac{1}{2} \left[ (L_1C^1 - L_2C^2) + (L_1C^1 - L_2C^2) \right]
\]

Boundary conditions for Equation 14 have been suggested by Danckwerts (60). These could be used to calculate the values of A and B in that equation. Then, if an estimate of the axial eddy diffusivity, \( E \), were available, the concentration profile of solute in the continuous phase could be predicted by means of Equation 14. However, the boundary conditions rely on the assumption that the dispersion model is applicable at the ends of the spray column. Some doubt exists as to whether this assumption is valid because the hydrodynamic flow patterns are much different in the Elgin head at the upper end of the column, and in the conical section at the lower end of the column, than in the column proper.

Another method for calculating values of A and B consists of fitting Equation 14 to a measured solute concentration profile in the continuous phase. In carrying out this curve fitting, an estimate of the axial eddy diffusivity, \( E \), is obtained. Since Equations
15 to 18 inclusive involve E, values of E can be chosen by trial
and error to produce the best fit of Equation 14 to the measured
ccentration profile.

One method of fitting Equation 14 to an experimental profile
is by means of the least squares technique. Estimates of the values
of A and B are obtained by minimizing the sum of the squares of the
differences between measured concentrations and those given by
Equation 14. That is, the value of Δ given by Equation 22, must
be a minimum.

\[ \Delta^2 = \sum \left[ C - A \exp(\lambda_1 Z) - B \exp(\lambda_2 Z) + Q \right]^2 \]  

If Equation 22 is differentiated partially with respect to A and
this result equated to zero, and then again with respect to B, and
that result equated to zero, two simultaneous equations in A and B,
Equations 23 and 24, result:

\[ A \sum (\exp(\lambda_1 Z))^2 + B \sum (\exp(\lambda_1 Z) \exp(\lambda_2 Z)) = \sum (C \exp(\lambda_1 Z)) + Q \sum \exp(\lambda_1 Z) \]  

\[ A \sum (\exp(\lambda_1 Z) \exp(\lambda_2 Z)) + B \sum (\exp(\lambda_2 Z))^2 = \sum (C \exp(\lambda_2 Z)) + Q \sum \exp(\lambda_2 Z) \]  

Equations 23 and 24 can be solved for A and B. This process can
be repeated for various assumed values of the axial eddy diffusivity,
E. The value of E which results in the lowest value of Δ² is one
estimate of the true value of E.
APPARATUS

The original apparatus was designed and built by Le Page (118). However, it has been modified by others (37, 38, 39) and by the author. A schematic flow diagram of one arrangement of the apparatus used in the present work is shown in Figure 9. This arrangement was used for tracer studies with no interphase mass transfer in a 1\(\frac{1}{2}\)-in. I.D. column. A key to Figure 9 is presented in Table 1.

All control valves were stainless steel needle valves with Teflon packing. The centrifugal pump, \(P_1\), supplies the constant head tank, \(E\), with de-ionised water from the storage tank, \(A\). Water flows from the constant head tank, \(E\), through the control valve, \(J\), and rotameter, \(G\), to the Elgin head, \(M\), of the column. Water flows down, as the continuous phase, through the column proper, \(R\), leaving at the lower end of the column and passing through the interface control valve, \(X\), and rotameter, \(Y\), to the receiving tank, \(B\). MIBK is supplied to the constant head tank, \(F\), by the centrifugal pump, \(P_2\), from the supply tank, \(D\). From the constant head tank, \(F\), MIBK flows through the control valve, \(K\), and rotameter, \(H\), to the dispersed phase nozzle, \(S\), at the lower end of the column. MIBK rises, in the form of drops, through the descending water in the column and coalesces at the interface, \(Q\). From the Elgin head MIBK flows directly to the receiving tank, \(C\). It is possible to
FIGURE 9. SCHEMATIC FLOW DIAGRAM FOR THE 1\textfrac{1}{2}-IN. I.D. COLUMN
TABLE 1

(Key to Figure 9)

<table>
<thead>
<tr>
<th>Letter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Continuous phase feed tank.</td>
</tr>
<tr>
<td>B</td>
<td>Continuous phase receiver and storage tank.</td>
</tr>
<tr>
<td>C</td>
<td>Dispersed phase receiver and storage tank.</td>
</tr>
<tr>
<td>D</td>
<td>Dispersed phase feed tank.</td>
</tr>
<tr>
<td>E</td>
<td>Continuous phase constant head tank.</td>
</tr>
<tr>
<td>F</td>
<td>Dispersed phase constant head tank.</td>
</tr>
<tr>
<td>G</td>
<td>Continuous phase feed rotameter.</td>
</tr>
<tr>
<td>H</td>
<td>Dispersed phase feed rotameter.</td>
</tr>
<tr>
<td>I</td>
<td>Continuous phase inlet sample valve.</td>
</tr>
<tr>
<td>J</td>
<td>Continuous phase flowrate control valve.</td>
</tr>
<tr>
<td>K</td>
<td>Dispersed phase flowrate control valve.</td>
</tr>
<tr>
<td>L</td>
<td>Dispersed phase inlet sample valve.</td>
</tr>
<tr>
<td>M</td>
<td>Elgin head.</td>
</tr>
<tr>
<td>N</td>
<td>Continuous phase inlet pipes.</td>
</tr>
<tr>
<td>O</td>
<td>Elgin head drain valve.</td>
</tr>
<tr>
<td>P₁</td>
<td>Centrifugal pump for continuous phase.</td>
</tr>
<tr>
<td>P₂</td>
<td>Centrifugal pump for dispersed phase.</td>
</tr>
<tr>
<td>PTS</td>
<td>Piston-type sampler.</td>
</tr>
<tr>
<td>Q</td>
<td>Interface.</td>
</tr>
<tr>
<td>R</td>
<td>Column proper.</td>
</tr>
<tr>
<td>S</td>
<td>Dispersed phase nozzle.</td>
</tr>
<tr>
<td>T₁, T₂</td>
<td>Thermometers</td>
</tr>
<tr>
<td>T₃, T₄</td>
<td>Thermometers</td>
</tr>
</tbody>
</table>
U  Bottom conical section.
V  Vent to atmosphere.
X  Interface level control valve.
Y  Interface level control rotameter.
Z  Tracer constant head tank.
a  Tracer flowrate control valve.
b  Tracer feed rotameter.
c  Tracer distributor.
d  22-gauge hypodermic needle samplers.
take a bulk sample from the column with the piston sampler, PTS, details of which are to be found elsewhere (37, 38). A drawing of the piston sample collection vessel for dispersed phase hold-ups greater than 12% is shown in Appendix V. The piston sample collection vessel for dispersed phase hold-ups less than 12% is similar and is described elsewhere (37). Both of the piston sample collection flasks were calibrated by weighing them when they contained various amounts of water.

Sodium chloride dissolved in MIBK-saturated water was used as tracer. A concentrated solution of this flows from the constant head tank, Z, through the control needle valve, a, and rotameter, b, to the tracer distributor, c, in the column. Time average point samples were taken from the column through 22-gauge, 3-in. long hypodermic needles, d. The flowrate of these samples was regulated by means of simple stainless steel-polyethylene stopcock valves. The hypodermic needles were inserted through polyethylene gaskets which were between 6-in. sections of the Pyrex glass column. (See Appendix V for detailed drawings of the tracer distributor and of the sample valves.) Samples containing tracer were analysed for sodium with a Perkin - Elmer 303 atomic absorption spectrophotometer.

The major part of the work was carried out with a Pyrex glass column of 1\(\frac{1}{2}\)-in. I.D. Details of the end sections of this column are to be found elsewhere (118, 35). Its vertical dimensions are shown in Figure 10. The arrangements shown in Figure 11 and 12
A 6" LONG, 1/2" I.D. PYREX "DOUBLE TOUGH" PIPE (SEE TABLE VI-1 FOR MEASURED DIMENSIONS)

B 1/8" THICK, 1/2" I.D. POLYETHYLENE GASKET THROUGH WHICH SAMPLING NEEDLES PASSED
(EACH SAMPLING POSITION IS INDICATED BY NUMBER)

D STYLE 1-2 TEFLOI GASKET
(CORNING)

FIGURE 10. 1 1/4-IN. I.D. COLUMN
were used together with that given in Figure 10 for investigating the effect of column length on the axial eddy diffusivity. Details of the column arrangements used for the part of the work concerning sampling techniques are shown in Figures 13 and 14. Two sampling techniques for measuring concentration profiles were investigated. The first method was the use of the hook and bell-probes mentioned earlier. Both the hook-shaped probe and the bell-shaped probe were made from stainless steel. Details of these probes are to be found elsewhere (30, 118), and a diagram is shown in Figure 5. Samples were syphoned out of the column through these probes and through small needle valves and rotameters connected in series with them. The needle valves were used to control the flows, and the rotameters to measure them. The syphon was started by means of a water aspirator. The second sampling method made use of hypodermic needles as mentioned earlier. The piston sampler, mentioned in the Introduction, was employed in both sampling investigations.

The drops were produced at a nozzle similar in design to that of Kreager and Geankoplis (16). With this sort of nozzle drops are formed at the ends of jets extending from short tubes press-fitted into a plate forming the end of the spray nozzle. These tubes were chamfered to sharp edges at their delivery ends. The dispersed phase nozzle used with the 1½-in. I.D. column was designed by Choudhury, and a detailed drawing is to be found in his thesis (35). The nozzle tips and nozzle tip support plate are described elsewhere (30, 35, 118). The average inside diameter of these nozzle tips was measured by the present author and was
INTERFACE

6" LONG, 6" I.D. PYREX "DOUBLE TOUGH" PIPE
10" LONG, 1½" I.D. PYREX "DOUBLE TOUGH" PIPE
WITH UNFLANGED UPPER END

A 6" LONG, 1½" I.D. PYREX "DOUBLE TOUGH" PIPE
(SEE TABLE VI-I FOR MEASURED DIMENSIONS)

B 1/16" THICK, 1½" I.D. POLYETHYLENE GASKET
THROUGH WHICH SAMPING NEEDLES
PASSED (EACH SAMPLING POSITION IS
INDICATED BY NUMBER)

1/8" THICK, 1½" I.D. TEFLO N GASKET THROUGH
WHICH THE TRACER INJECTION NEEDLE PASSED

CUSTOM MADE PYREX "DOUBLE TOUGH" 3" TO
1½" REDUCER (30)

STYLE 1-2 TEFLO N GASKET (CORNING)

**FIGURE 11. SHORT 1½-IN. I.D. COLUMN**
INTERFACE

6" LONG, 6" I.D. PYREX "DOUBLE TOUGH" PIPE

10" LONG, 1/2" I.D. PYREX "DOUBLE TOUGH" PIPE WITH UNFLANGED UPPER END

2' 6" LONG, 1/2" I.D. PYREX "DOUBLE TOUGH" PIPE

3' LONG, 1/2" I.D. PYREX "DOUBLE TOUGH" PIPE

A 6" LONG, 1/2" I.D. PYREX "DOUBLE TOUGH" PIPE (SEE TABLE VI-1 FOR MEASURED DIMENSIONS)

B 1/16" THICK, 1/2" I.D. POLYETHYLENE GASKET THROUGH WHICH SAMPLING NEEDLES PASSED (EACH SAMPLING POSITION IS INDICATED BY NUMBER)

D STYLE 1-2 TEFLOM GASKET (CORNING)

1/8" THICK, 1/2" I.D. TEFLOM GASKET THROUGH WHICH THE TRACER INJECTION NEEDLE PASSED

2' LONG, 1/2" I.D. PYREX "DOUBLE TOUGH" PIPE

1' 6" LONG, 1/2" I.D. PYREX "DOUBLE TOUGH" PIPE

CUSTOM MADE PYREX "DOUBLE TOUGH" 3" TO 1/2" REDUCER

FIGURE 12. LONG 1-3/4-IN. I.D. COLUMN
FIGURE 13. 1½-IN. I.D. COLUMN FOR COMPARING HOOK AND BELL-PROBES AND PISTON SAMPLER RESULTS
FIGURE 14. 1¼-IN. I.D. COLUMN FOR COMPARING HYPODERMIC NEEDLE, HOOK, AND BELL-PROBES, AND PISTON SAMPLER RESULTS
found to be 0.103-in. (See Appendix V.) This nozzle and these nozzle tips were used for much of the work described in the present thesis including all the work involving mass transfer between phases. However, the end plate and nozzle tip assembly was replaced for certain runs so that the effect of drop size on the axial eddy diffusivity could be studied. Thus sets of nozzle tips of average inside diameter
0.126-in., 0.086-in., and 0.053-in. respectively were used in addition to those of 0.103-in. already mentioned. A dispersed phase nozzle was designed for use in a 3-in. I.D. Pyrex glass column used for part of the work and described later in this section of the thesis. Detailed drawings of the 0.126-in., 0.086-in., and 0.053-in. I.D. nozzle tips appear in Appendix V. The average velocity of MIBK in the dispersed phase distributor nozzle tips was maintained constant for each set of nozzle tips by blocking off nozzle tips with Teflon caps or plugs as the MIBK flowrate was reduced. Patterns of open nozzle tips used in this work are shown in Figures 15, 16, 17, and 18, one for each set of nozzle tips and for the various dispersed phase flowrates used.

Drop size distributions in the 1½-in. I.D. column were determined by photographing a 4-in. length of column situated between 7-in. and 11-in. above the top of the piston sampler block. The camera used was an Exacta VX II a) with a 1.6-in. extension ring and a Telemegor 5.5/250 telephoto lens. The camera aperture was set at f22 to give a depth of focus greater than the inside diameter of the column. Adox KB-14 (20 ASA) film was used.
FIGURE 15. NOZZLE TIP PATTERNS. I.D. = 0.126-IN. (1\frac{1}{2}-IN. I.D. COLUMN)
FIGURE 16. NOZZLE TIP PATTERNS. I.D. = 0.103-IN. (1\(\frac{1}{2}\)-IN. I.D. COLUMN)
FIGURE 17. NOZZLE TIP PATTERNS. I.D. = 0.086-IN. (1/2-IN. I.D. COLUMN)
FIGURE 18. NOZZLE TIP PATTERNS. I.D. = 0.053-IN. (1\(\frac{1}{2}\)-IN. I.D. COLUMN)
The section of column photographed was surrounded by a parallel-sided Perspex box filled with distilled water to reduce the distorting effect of the round column. Back lighting was effected with a Braun Hobby F60 electronic flash unit with a one millisecond flash duration. The section of column photographed was shielded from extraneous light with a cardboard box which also supported the electronic flash head and a light-diffusing screen of eight sheets of tracing paper. Figure 19 shows the relative positions of flash, column, and camera. Detailed drawings of the Perspex box and the cardboard shield are given in Appendix V.

A few experimental runs were carried out with the 3-in. I.D. Pyrex glass column shown in Figure 20. In order to produce and maintain high continuous phase superficial velocities three continuous phase feed tanks, pressurized to about 15 p.s.i.g. with nitrogen, were used. A schematic flow diagram is presented in Figure 21. Detailed drawings of the end sections and dispersed phase distributor nozzle are given in Appendix V. Figure 22 shows the patterns of open and closed nozzle tips used for various dispersed phase flowrates. Sodium chloride tracer was injected into the column through the same tracer distributor as used in the 1 1/2-in. I.D. column. The tracer distributor was located on the centre line of the column and tracer flowed into it through a 3-in. long, 18-gauge hypodermic needle which passed through a 1/8-in. thick, 3-in. I.D., and 3 27/32-in. O.D. Teflon gasket. Samples were withdrawn as with the 1 1/2-in. I.D. column except that
FIGURE 19. PHOTOGRAPHIC CONDITIONS
5^ INTERFACE
9" LONG, 9" I.D. Q.V.F. PIPE

10 3/4" LONG, 3" I.D. PYREX "DOUBLE TOUGH" PIPE

A 6" LONG, 3" I.D. PYREX "DOUBLE TOUGH" PIPE (SEE TABLE VI-I FOR MEASURED DIMENSIONS)

B 1/16" THICK, 3" I.D. POLYETHYLENE GASKET THROUGH WHICH SAMPLING NEEDLES PASSED (EACH SAMPLING POSITION IS INDICATED BY NUMBER)

6" LONG, 3" I.D. PYREX "DOUBLE TOUGH" PIPE WITH BOTH ENDS UNFLANGED (SEE TABLE VI-I FOR MEASURED DIMENSIONS)

1/8" THICK, 3" I.D. POLYETHYLENE GASKET THROUGH WHICH THE TRACER NEEDLE PASSED

CUSTOM MADE PYREX "DOUBLE TOUGH" 6" TO 3" REDUCER

FIGURE 20. 3-IN. I.D. COLUMN
FIGURE 21. SCHEMATIC FLOW DIAGRAM FOR THE 3-IN. I.D. COLUMN
TABLE 2

(Key to Figure 21)

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A₁, A₂, A₃</td>
<td>Continuous phase feed tanks.</td>
</tr>
<tr>
<td>B</td>
<td>Dispersed phase feed and receiver tank.</td>
</tr>
<tr>
<td>C₁, C₂</td>
<td>Interface level control rotameters.</td>
</tr>
<tr>
<td>D₁, D₂</td>
<td>Interface level control valves.</td>
</tr>
<tr>
<td>G₁, G₂</td>
<td>Continuous phase feed rotameters.</td>
</tr>
<tr>
<td>H₁, H₂</td>
<td>Dispersed phase feed rotameters.</td>
</tr>
<tr>
<td>I</td>
<td>Continuous phase inlet sample valve.</td>
</tr>
<tr>
<td>J₁, J₂</td>
<td>Continuous phase flowrate control valves.</td>
</tr>
<tr>
<td>K₁, K₂</td>
<td>Dispersed phase flowrate control valves.</td>
</tr>
<tr>
<td>M</td>
<td>Elgin head.</td>
</tr>
<tr>
<td>N</td>
<td>Continuous phase inlet pipes.</td>
</tr>
<tr>
<td>NC</td>
<td>Nitrogen cylinder.</td>
</tr>
<tr>
<td>O</td>
<td>Elgin head drain valve.</td>
</tr>
<tr>
<td>P₁</td>
<td>Centrifugal pump for continuous phase feed.</td>
</tr>
<tr>
<td>P₂</td>
<td>Centrifugal pump for dispersed phase feed.</td>
</tr>
<tr>
<td>P₃</td>
<td>Centrifugal pump for continuous phase outlet.</td>
</tr>
<tr>
<td>PG</td>
<td>Pressure gauge.</td>
</tr>
<tr>
<td>PRV</td>
<td>Pressure relief valve.</td>
</tr>
<tr>
<td>Q</td>
<td>Interface.</td>
</tr>
<tr>
<td>R</td>
<td>Column proper.</td>
</tr>
<tr>
<td>S</td>
<td>Dispersed phase nozzle.</td>
</tr>
</tbody>
</table>
$T_1, T_2$
$T_3, T_4$  Thermometers

U  Bottom conical section.
V  Vent to atmosphere.
Z  Tracer constant head tank.
a  Tracer flowrate control valve.
b  Tracer feed rotameter.
c  Tracer distributor.
d  22-gauge hypodermic needle samplers.
FIGURE 22. NOZZLE TIP PATTERNS. I.D. = 0.102-IN. (3-IN. I.D. COLUMN)
the hypodermic needles passed through 1/16-in. thick, 3-in. I.D., and 3 27/32-in. O.D. polyethylene gaskets to the centre line of the column. The eighth 6-in. section of column above the tracer distributor was cut from a 1-ft. long piece of Pyrex glass pipe. The piece selected was remote from the ends so that its bore was reasonably uniform. This section of column was held in place by four aluminum tie rods between two large diameter aluminum flanges shown in Appendix V. A flat-sided Perspex box, also shown in Appendix V surrounded this 6-in. long piece of column. The space between the glass and the Perspex was filled with distilled water. Photographs were taken of the column through the Perspex box with the use of a cardboard light-shield, a tracing paper diffusing screen and an electronic flash as with the 1¼-in. column. Photographic conditions were the same as before except that lighting was by means of a Kakonet - II electronic flash unit with a flash duration of 0.5-millisecond.
EXPERIMENTAL PROCEDURE

1. COLUMN OPERATION

City water was passed through a Barnstead Bantam BD-1 mixed bed demineralizer to give better than 1 megohm-cm. resistivity water. In all experiments the continuous phase was demineralized water and the dispersed phase was technical grade methyl isobutyl ketone (MIBK) supplied by Chemcell Ltd. Each phase was kept saturated with the other by maintaining a layer of one phase in the feed tank of the other. The flowrates of both phases fed to the column were regulated with stainless steel needle valves and metered with rotameters. Four different drop size distributions were produced in the 1.2-in. I.D. column by means of four sets of nozzle tips of respective average inside diameters 0.126-in., 0.103-in., 0.086-in., and 0.053-in. Only one set of nozzle tips of average inside diameter 0.102-in. was used with the 3-in. I.D. column. The dispersed phase average velocity in the distributor nozzle tips was held at 0.36-ft./sec., 0.36-ft./sec., 0.38-ft./sec., 0.68-ft./sec., and 0.37-ft./sec. for the 0.126-in., 0.103-in., 0.086-in., 0.053-in., and 0.102-in. I.D. tips respectively by utilizing only a portion of the total nozzle tips available with the help of Teflon caps or plugs as described under the heading Apparatus. The higher nozzle tip
velocity of 0.68-ft./sec. in the 0.053-in. I. D. tips was found to be necessary in order to produce drops of a narrow range of sizes. The interface in the Elgin head was maintained steady to within $\pm$ 1/16-in. by controlling the flow of continuous phase leaving the column by means of a stainless steel needle valve. A rotameter, in series with this valve, indicated the flowrate. The dispersed phase flow from the column head to the receiving tank was unrestricted.

2. SAMPLING TECHNIQUE STUDIES WITH MASS TRANSFER

An attempt was made to relate the samples taken by the bell and hook-probes to samples taken by means of the piston. Early in the work each of the sampling lines leading from the bell and hook-probes, respectively, used by earlier workers (38) was replaced by 27-ft. of smaller bore (3/32-in. I.D.) Nylon tubing. It was found that a conservative estimate of the minimum purge time for each of these new sampling lines was given by the following equation. (See Appendix VI)

$$\text{Purge time} \ (\text{min.}) = \frac{120}{\text{sampling rate} \ (\text{ml./min.})}$$

In all of the work involving the hook or bell-probe described in this thesis these probes were positioned as closely as possible to the centre line of the column. The times for the column to reach steady state operating conditions at various flowrates of each phase was taken from the work of Bergeron (38).
In all experiments acetic acid was transferred from the continuous aqueous phase to the dispersed MIBK phase. At the end of an experiment the aqueous phase product was transferred to the aqueous phase feed tank and the aqueous phase product tank was filled with de-ionised water. This water was used to backwash MIBK from the previous run in the column in order to prepare a feedstock of MIBK low in acetic acid concentration for the next run. The aqueous phase backwashing product was fed to the aqueous phase feed tank until the liquid level in that tank was about 6-in. from the top and then the remainder was fed to the drain.

Reagent grade glacial acetic acid, manufactured by Nichols Chemical Corporation Ltd., was used for preparing the aqueous acetic acid solution used as continuous phase feed in the following manner. If necessary the aqueous phase feed tank was filled to within about six inches from the top by adding de-ionised water. The contents of this tank were pumped up to the aqueous phase constant head tank and allowed to run back into the feed tank until the solution was homogeneous. Homogeneity was checked by periodically titrating the flow from the constant head tank with carbonate-free standard sodium hydroxide solution. (The sodium hydroxide solution was prepared as recommended by Swift (119) and was standardized by titrating against standard potassium hydrogen phthalate solution.) From a knowledge of the volume of liquid in the aqueous phase feed tank and the concentration of acetic acid in solution in that tank the amount of glacial
acetic acid to be added in order to produce a suitable acetic acid concentration in the aqueous phase feed was calculated. This amount of glacial acetic acid was poured into the aqueous phase feed tank. The contents of this tank then were circulated to give a homogeneous solution as before.

In this study the set of nozzle tips of average inside diameter 0.103-in. was used. In most of these experiments all of the dispersed phase distributor nozzle tips were kept open. Therefore a high ketone flowrate was obtained with the use of an average nozzle tip velocity of 0.36-ft./sec. A high ketone flowrate was required in order to produce a high dispersed phase hold-up so that the application of Equation 6 in connection with the piston sampler would result in a small error in the calculated value of $c_D$. A high aqueous phase flowrate was used so that the column was operating under conditions far from equilibrium in the vicinity of the piston sampler. Under these conditions solute concentration gradients which are substantially different from zero in both phases over the piston height are to be expected. Without this condition a comparison between the various sampling techniques would be meaningless.

a) Sampling with hook and bell-probes and piston

The apparatus was set up as shown in Figure 13. Each of the bell and hook-probes were positioned so that their respective
entrances were 3-in. below the centre line of the piston. The desired MIBK flowrate into the empty column was established, and aqueous-feed phase was pumped into the column at the maximum possible rate. When the interface reached the desired level the flowrate of the aqueous continuous phase was reduced to the operating value and the valve controlling the aqueous phase flow rate from the foot of the column was opened and adjusted to maintain the interface level. After allowing the column to reach steady state conditions sampling through the probes was commenced. Samples were collected in 50-ml. graduated cylinders which were closed with ground-glass stoppers immediately after the samples were taken. Samples were collected with each probe at the location mentioned above. The probes then were moved 1-in. up the column, and a second set of samples was taken. Sets of samples were collected at an additional four 1-in. intervals up the column. When the probe samples had been taken the probes were raised above the interface, and a piston sample was collected after a half-hour interval in the calibrated custom-made vessel shown in Figure V - 1, Appendix V. The volumes of each phase in samples taken with the bell-probe and with the piston were recorded. Sometime during the course of steady state operation column inlet and outlet samples of both phases were taken.

All of the samples containing two phases were shaken vigorously many times in order to bring the two phases to equilibrium. The concentration of acetic acid in each phase of
each sample was determined by titrating with standard sodium hydroxide solution using phenolphthalein as indicator. When titrating the MIBK phase SDAG-1K* was added to render the MIBK and aqueous phases miscible.

In addition to the experiments described above an experiment was performed with a modification of the hook-probe as shown in the sketch below.

b) Sampling with hook and bell-probes, hypodermic needles, and piston

Short Pyrex spacers were included in the column above and below the piston block as shown in Figure 14. Hypodermic needles

* SDAG-1K is a mixture of 90% v/v ethanol and 10% v/v methanol.
were inserted through polyethylene gaskets between the spacers in a manner similar to that shown in Figure V - 2, Appendix V. The minimum purge time for these needles was found to be less than 45-sec. at a sampling rate of 3/4-ml./min. In all of the work involving hypodermic needles described in this thesis a needle was purged for at least 1½-min. before taking a sample. A hypodermic needle sample was taken always with the open end of the needle on the centre line of the column unless otherwise indicated.

The column was brought to steady state operating conditions and probe samples were taken as before. Then the probes were raised above the interface and the column was allowed to attain steady state operating conditions. Samples were collected by means of the hypodermic needles in the following manner. Two needles, separated by not less than 10 inches, were used concurrently, the other needles being withdrawn to the column wall so as not to protrude into the column. The valves at the end of each of the two needles were opened so as to give sampling rates of about 1-ml./min. The samples were collected in 10-ml. graduated cylinders. After the final hypodermic needle sample was collected the column was allowed to reach steady state operating conditions and then a piston sample was taken. The concentration of acetic acid in each phase of each sample was determined by titrating with sodium hydroxide solution as before.
3. SEARCH FOR SUITABLE TRACER

An investigation and preliminary tests were made to find a tracer for use in the determination of axial eddy diffusivities with no mass transfer between the dispersed and the continuous phases. The necessary tracer properties are listed below:

a) high solubility in MIBK-saturated water,

b) insolubility in water-saturated MIBK,

c) concentration must be measurable at low values,

d) must not disturb the fluid flow patterns when injected into an operating column,

e) must not adsorb at MIBK-water interface or at any solid-liquid interface in the column,

f) must not react chemically with water, MIBK, or any solid surface in the column, and

g) the molecular diffusivity in MIBK-saturated water must be negligible compared with the axial eddy diffusivity.

The tracers considered, the method of quantitative analysis for their concentration, and their shortcomings, if any, are listed below.

a) Ferric nitrate. Analysis for this compound would be by the thiocyanate method making use of colorimetry. Ferric nitrate hydrolyses in MIBK-saturated water, ferric hydroxide precipitating.

b) Hydrochloric acid. Analysis would be by titration. Very low concentrations are not easily measurable.

c) Potassium chloride. Analysis would be carried out by means
of conductimetry. Very low concentrations are not easily measurable.

d) Water soluble dyes. Solutions of these compounds would be analysed colorimetrically. Even completely ionised dyes, such as crystal violet, dissolve in water-saturated MIBK.

e) Cupric sulphate. Analysis would be by the dithizone method making use of colorimetry. Dithizone is quite unstable.

f) Sodium chloride. Analysis would be for sodium by means of atomic absorption spectrophotometry. The molecular diffusivity of sodium chloride in water is 0.00005-ft²/hr. at 65°F for concentrations between 0 and 1-molar (128). No data is available for this diffusivity in MIBK-saturated water, but it is expected that the value would be closely similar to that for pure water and this value has been used. Axial eddy diffusivities in this work were found to be always greater than 7-ft²/hr. Thus the condition that the molecular diffusivity is negligible compared with the axial eddy diffusivity is met.

A solution of sodium chloride in MIBK - saturated water was found to be a suitable tracer. The distribution coefficient* for sodium chloride between MIBK - saturated water and water - saturated MIBK was found to be 7060.

* The distribution coefficient is defined here as the concentration of sodium chloride in the aqueous layer divided by the concentration of that compound in the ketone layer where concentrations are expressed in the units of weight per unit volume of solution.
4. SAMPLING TECHNIQUE STUDIES WITH NO MASS TRANSFER

The apparatus was set up as shown in Figures 9 and 14 with the following modifications. The third polyethylene gasket below the piston was exchanged with the Teflon gasket further down the column. The hypodermic needle supporting the tracer distributor remained in this Teflon gasket so that there were only two hypodermic needle sampling positions between the tracer distributor and the piston. The MIBK leaving the Elgin head passed directly to the MIBK feed tank. The aqueous and MIBK feedstocks were free of acetic acid. The flows of MIBK and aqueous phases were started and the interface level was established as described in the present section of the thesis under the heading 1: Column Operation.

The experiments were performed under the following operating conditions:

<table>
<thead>
<tr>
<th>Continuous phase superficial velocity, ( \text{ft}^3/\text{hr. ft}^2 )</th>
<th>Dispersed phase superficial velocity, ( \text{ft}^3/\text{hr. ft}^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>36.5</td>
<td>128</td>
</tr>
<tr>
<td>36.5</td>
<td>30.4</td>
</tr>
<tr>
<td>27.7</td>
<td>128</td>
</tr>
</tbody>
</table>

A one molar sodium chloride tracer feed solution was prepared by dissolving a weighed amount of sodium chloride in MIBK-saturated water (119). This solution was started into the column as soon as the interface level had been established in the Elgin head. The column was run for one hour to allow steady state conditions to be attained. (Experiments for the study of steady state times are discussed later.) Five hook-
probe samples were taken from equally spaced positions throughout the height of the piston block. The hook-probe sampling rate was 5-ml./min. Four hypodermic needle samples were taken, one through each of the gaskets immediately above and below the piston block, and one through each of those approximately six inches above and below the piston block. Finally a piston sample was taken. Half hour time intervals were allowed between taking each probe sample, between taking the last probe sample and the first hypodermic needle sample, and between taking the last hypodermic needle sample and the piston sample. The aqueous phase of each sample was analysed for sodium by means of a Perkin-Elmer 303 atomic absorption spectrophotometer.

5. AXIAL EDDY DIFFUSIVITY AND DISPERSED PHASE HOLD-UP

a) Studies in the 1\(\frac{3}{4}\)-in. I.D. column.

Initially a few experiments were performed in order to see whether or not the dispersion model would describe the experimental data adequately and, therefore, whether or not axial eddy diffusivities could be determined from this model. These experiments were carried out in a manner identical to that for the main bulk of experiments which is described below. It was found that a plot of the logarithm of the concentration of tracer versus height up the column was linear; accordingly the dispersion model was adopted.

In addition, before the main bulk of experimental data was collected some experiments were carried out to justify some of the experimental techniques used. Since these experi-
ments usually were carried out in conjunction with axial eddy diffusivity determinations. Their description will be given following that of the main experiments. The apparatus used for the subsidiary experiments described under ii) to viii) inclusive below was identical to that used for the main experiments.

1) Axial eddy diffusivity determinations.

The column was set up as shown in Figures 9 and 10 except that the MIBK line from the Elgin head led directly to the MIBK feed tank so that MIBK recycled through the apparatus. As required, some nozzle tips of the dispersed phase distributor were blocked off with Teflon caps or plugs according to the patterns shown in Figures 15 to 18 to ensure the desired average velocity of dispersed phase in the nozzle tips. The flow of MIBK into the column was started, and thereafter maintained at the desired rate. Water was pumped into the column at the maximum possible rate until the interface in the Elgin head reached a predetermined level. The flowrate of water was then reduced to the experimental value and water was allowed to leave the column at such a rate so as to maintain the desired constant interface level. The tracer (1-molar sodium chloride solution in MIBK-saturated water) was fed to the column at approximately 1% of the volumetric flowrate of the aqueous phase. After allowing the column to reach steady state operating conditions ten aqueous phase samples were taken by means of the hypodermic needles, one at a time. The first
sample was taken with the lowest needle in the column, the second sample from the next needle up the column, and so on. The samples were withdrawn from the centre of the column and at a rate less than 1.5% of the volumetric flowrate of aqueous phase though the column. The description of experiments performed to investigate radial concentration gradients and also the effect of sampling rate upon the column operation are discussed later. When a hypodermic needle was not being used it was withdrawn to the column wall. The flowrate of each phase from the column was determined by weighing a sample collected over a suitable time interval. The average of the temperatures of the fluids in the upper and lower sections of the Elgin head, of the fluid at the lower end of the column, and of the ketone phase entering the column was recorded. Finally three piston samples were taken at intervals of ten minutes and the volumes of the two phases in each sample recorded. The tracer concentration in each of the ten hypodermic needle samples and in the aqueous phase leaving the column were measured by means of an atomic absorption spectrophotometer. (Analysis was for sodium.) The calibration line for the atomic absorption spectrophotometer always was found to be a straight line. The equation for this line was calculated by the method of least squares. Samples whose concentrations were less than 0.05-microgm. sodium/ml. were discarded on the grounds of unreliability. The probable error in the concentration of a sample determined by this method was less than 2%. 

Experiments were performed at all possible combinations of the following operating conditions.

<table>
<thead>
<tr>
<th>Continuous phase superficial velocity, ft³/hr. ft²</th>
<th>Dispersed phase superficial velocity, ft³/hr. ft²</th>
<th>Nozzle tip average inside diameter, in.</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.0</td>
<td>36.5</td>
<td>0.053</td>
</tr>
<tr>
<td>18.2</td>
<td>54.7</td>
<td>0.086</td>
</tr>
<tr>
<td>27.7</td>
<td>73.0</td>
<td>0.103</td>
</tr>
<tr>
<td>36.5</td>
<td>91.2</td>
<td>0.126</td>
</tr>
<tr>
<td>48.4</td>
<td>110</td>
<td></td>
</tr>
<tr>
<td></td>
<td>128</td>
<td></td>
</tr>
</tbody>
</table>

For each set of nozzle tips about 10 photographs of the drops within the column were taken as described earlier under the heading Apparatus at the operating conditions shown in each line shown in the following table.

<table>
<thead>
<tr>
<th>Continuous phase superficial velocity, ft³/hr. ft²</th>
<th>Dispersed phase superficial velocity, ft³/hr. ft²</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.0</td>
<td>36.5</td>
</tr>
<tr>
<td>27.7</td>
<td>36.5</td>
</tr>
<tr>
<td>48.4</td>
<td>36.5</td>
</tr>
<tr>
<td>27.7</td>
<td>54.7</td>
</tr>
<tr>
<td>27.7</td>
<td>91.2</td>
</tr>
</tbody>
</table>

Some, but not all, of the photographs were taken during runs for determining axial eddy diffusivities. However, those not taken during runs were taken under column operating conditions identical to those which applied during runs. The photographic negatives were examined by means of a Recordak model M.P.E. microfilm reader. The drop size distributions were determined by measuring the vertical and horizontal dimensions of the projected images of the drops. With the photographic
conditions described earlier the depth of focus included the whole of the column cross-section. Only the drops lying within the central 46% of the column image, as projected on the screen, were examined, because as determined by experiments described below, the drops falling within these limits were not distorted optically. Five hundred drops were measured for each set of column operating conditions given in the last table.

ii) Optical distortion of drops.

As mentioned earlier optical distortion was reduced considerably by surrounding the round glass column with a flat-sided Perspex box. Details of the box are presented in Appendix V. The space between the box and the column was filled with distilled water. The same photographic conditions were used for the calibration photographs as for the photographs taken to determine drop size distributions as described under the heading Apparatus. The same 6-in. long section of column was used for the calibrations as was used for the actual drop size distribution measurements. The Perspex box was not shielded from extraneous light as in the drop size distribution studies since the photographs were taken in a darkroom. Photographs were taken of a 5/32-in. diameter ball bearing silver soldered to a stainless steel wire. The ball was photographed at positions shown in Figure 23 for each of the horizontal planes located 1/2-in. above and 1/2-in. below the centre of the Perspex box. Two sets
FIGURE 23. OPTICAL DISTORTION INVESTIGATION, 1\(\frac{1}{8}\)-IN. I.D. COLUMN
of photographs were taken, one with MIBK - saturated water, and one with a solution of sodium chloride in MIBK - saturated water in the column. For this second solution the concentration of sodium was 100-microgm./ml.

iii) Effect of tracer feed rate

The feed rate of tracer solution into the column should be low enough so as not to disturb the fluid flow patterns within the column. The effect of tracer feed rate was studied using the superficial velocities of the two phases, and of the tracer feed solution supplied to the column, given in each line of the following table. Only the nozzle tips of 0.103-in. I. D. were used for this study.

<table>
<thead>
<tr>
<th>Continuous phase, ft³/hr. ft²</th>
<th>Dispersed phase, ft³/hr. ft²</th>
<th>Tracer solution, ft³/hr. ft²</th>
</tr>
</thead>
<tbody>
<tr>
<td>27.7</td>
<td>30.4</td>
<td>0.16</td>
</tr>
<tr>
<td>27.7</td>
<td>30.4</td>
<td>0.31</td>
</tr>
<tr>
<td>27.7</td>
<td>30.4</td>
<td>0.62</td>
</tr>
<tr>
<td>18.2</td>
<td>73</td>
<td>0.10</td>
</tr>
<tr>
<td>18.2</td>
<td>73</td>
<td>0.20</td>
</tr>
<tr>
<td>18.2</td>
<td>73</td>
<td>0.40</td>
</tr>
</tbody>
</table>

iv) Steady-state time.

The experimental conditions for sections iv) to viii) inclusive are shown in each line of the following table
The time taken for the column to reach steady state operating conditions was estimated by taking samples by means of the first, fourth, seventh, and tenth hypodermic needles above the tracer distributor, and also by collecting aqueous phase leaving the column. One of each of these samples was taken between 3-min. and 21-min. after start-up and then at intervals varying between 5-min. and 30-min. until 2-hr. after start-up.

v) Reproducibility of results.

Several of the experiments for determining axial eddy diffusivity and hold-up were repeated under identical operating conditions in order to test the reproducibility of results.

vi) Cross-sectional homogeneity.

In addition to the sample taken from the centre of the column by means of the hypodermic needle immediately above the tracer distributor four samples were taken at the same elevation but at positions on a 1-in. circle concentric with the column. This experiment was carried out for each of the runs listed in section iv) above.
vii) Effect of sampling rate.

Samples of the continuous phase were taken at 0.25-ml./min., 0.75-ml./min., and 1.5-ml./min. for the continuous phase superficial velocity of 9-ft³/hr. ft² and at 0.5-ml./min., 1.0-ml./min., and 2.0-ml./min. for the continuous phase superficial velocity of 27.7-ft³/hr. ft². Only the first four sampling positions above the tracer distributor were studied.

viii) Effect of order of sampling.

Samples were taken consecutively from the lowest sampling position up to the highest one. A second set of samples was taken with this sampling order reversed. Both sampling orders were used in each of the experiments described in the table given in section iv).

ix) Effect of column length.

In addition to the standard experiments performed with the column of height 10-ft. 3 1/8-in., as shown in Figure 10, a single experiment was performed in each of two other columns, one of height 6-ft. 3 1/2-in., and the other of height 16-ft. 4 1/2-in. as shown in Figures 11 and 12 respectively. Each of these last two experiments was carried out with a continuous phase superficial velocity of 18.2-ft³/hr. ft², and a dispersed phase superficial velocity of 54.7-ft³/hr. ft². The average I.D. of the
nozzle tips in both experiments was 0.103-in.

b) Studies in the 3-in. I.D. column.

Although the great bulk of the work described in this thesis was carried out in a 1\(\frac{1}{2}\)-in. I.D. column, some tracer experiments were carried out in a 3-in. I.D. column. This column is shown in Figure 20 and a flowsheet is given in Figure 21. In order to produce a continuous phase superficial velocity in the column greater than 100-ft\(^3\)/hr. ft\(^2\) it was necessary to pressurize the aqueous phase feed tanks to 15 p.s.i.g. with nitrogen. Only one set of nozzle tips of 0.102-in. average I.D. was used. It was not possible to measure the dispersed phase hold-up because a piston sampler for this column was not available. As with the 1\(\frac{1}{2}\)-in. I.D. column, experiments were carried out in conjunction with the axial eddy diffusivity experiments to justify some of the experimental techniques used for the 3-in. I.D. column. The experiments with this column are described below.

i) Optical distortion of drops.

The photographic test section was a 6-in. long, 3-in. I.D. length of Pyrex Double Tough glass pipe which was cut from a 1-ft. long flanged section as described earlier. This pipe was surrounded by a flat-sided Perspex box and the space between the pipe and the box was filled with distilled water.
photographic conditions were similar to those used for the 1\(\frac{1}{2}\)-in. I.D. column. A Kakonet - II electronic flash unit was used for lighting purposes. Photographs were taken of a 5/32-in. diameter ball bearing. The ball was photographed at positions shown in Figure 24 for each of the horizontal planes located 1\(\frac{1}{4}\)-in. above and 1\(\frac{1}{4}\)-in. below the centre of the Perspex box. Two sets of photographs were taken, one with MIBK - saturated water, and one with a solution of sodium chloride in MIBK - saturated water in the column. For this second solution the concentration of sodium was 100-microgm./ml.

ii) Steady-state time.

The experimental conditions for sections ii) and iii) are given in each line of the following table.

<table>
<thead>
<tr>
<th>Continuous phase superficial velocity, (\text{ft}^3/\text{hr. ft}^2)</th>
<th>Dispersed phase superficial velocity, (\text{ft}^3/\text{hr. ft}^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18.2</td>
<td>36.5</td>
</tr>
<tr>
<td>100</td>
<td>36.5</td>
</tr>
<tr>
<td>18.2</td>
<td>109</td>
</tr>
</tbody>
</table>

Samples were taken by means of the first, fourth, seventh, and tenth hypodermic needles above the tracer distributor, and also by collecting aqueous phase leaving the column. One of each of
FIGURE 24. OPTICAL DISTORTION INVESTIGATION, 3-IN. I.D. COLUMN
these samples was taken between 2-min. and 10-min. after start-up and then at intervals varying between 5-min. and 30-min. until 1.5-hr. after start-up.

iii) Reproducibility of results.

Several of the experiments for determining axial eddy diffusivity were repeated under identical operating conditions in order to test the reproducibility of results.

iv) Cross-sectional homogeneity.

In addition to the samples taken from the centre of the column by means of the first and fifth hypodermic needles above the tracer distributor, samples were taken from positions shown in the following sketch at each of the two above-mentioned sampling elevations. These sets of samples were collected for each of the column operating conditions shown in the following table.

<table>
<thead>
<tr>
<th>Continuous phase superficial velocity, ft³/hr. ft²</th>
<th>Dispersed phase superficial velocity, ft³/hr. ft²</th>
</tr>
</thead>
<tbody>
<tr>
<td>18.2</td>
<td>36.5</td>
</tr>
<tr>
<td>100</td>
<td>36.5</td>
</tr>
<tr>
<td>18.2</td>
<td>73</td>
</tr>
<tr>
<td>100</td>
<td>73</td>
</tr>
<tr>
<td>18.2</td>
<td>109</td>
</tr>
<tr>
<td>100</td>
<td>109</td>
</tr>
</tbody>
</table>
v) Axial eddy diffusivity determinations.

The experiments performed to determine axial eddy diffusivities were carried out in a manner similar to that for the 1\(\frac{1}{2}\)-in. I.D. column. The experimental conditions are given in Table IV-5, Appendix IV. A sampling rate of 3-ml./min. was used in all experiments in the 3-in. I.D. column.

Photographs were taken of the fluids within the calibrated glass section described earlier which was installed as the eighth 6-in. section above the tracer distributor. This section of column was surrounded by a flat-sided Perspex box as shown in Appendix V. The photographic conditions were similar to those used for the 1\(\frac{1}{2}\)-in. I.D. column. A Kakonet - II electronic flash unit was used for lighting purposes. The column operating
conditions for which photographs were taken are shown in each line of the following table.

<table>
<thead>
<tr>
<th>Continuous phase superficial velocity, ft.³/hr. ft.²</th>
<th>Dispersed phase superficial velocity, ft.³/hr. ft.²</th>
</tr>
</thead>
<tbody>
<tr>
<td>18.2</td>
<td>36.5</td>
</tr>
<tr>
<td>36.5</td>
<td>36.5</td>
</tr>
<tr>
<td>75</td>
<td>36.5</td>
</tr>
<tr>
<td>220</td>
<td>36.5</td>
</tr>
</tbody>
</table>

6. CONCENTRATION PROFILES WITH MASS TRANSFER

The 1 1/2-in. I.D. column was set up as shown in Figures 9 and 10. The tracer distributor, and the Teflon gasket through which the tracer supply hypodermic needle passed, were replaced by a standard 1/16-in. thick Teflon gasket. Acetic acid was transferred from the continuous phase to the dispersed phase. The concentration profile in each phase was measured by means of the bell-probe and hypodermic needles as described below.

The column was brought to the operating condition as described under section 2 of Experimental Procedure. The times at which steady state conditions were attained were determined by taking samples by means of the lowest and the highest hypodermic needles and of the aqueous phase leaving the column at various times after start-up. All hypodermic needle samples in this part of the work were taken at 3/4-ml./min. During actual runs samples were taken with the bell-probe at the levels
of the first, third, fifth, seventh, ninth, and tenth hypodermic needles up the column. A bell-probe sampling rate of 5.2-ml./min. was used for each sample taken. Samples were not taken by means of the bell-probe at all levels of the needles because of the lengthy procedure of sampling with the bell-probe. The bell-probe was raised above the interface and after a half-hour interval aqueous phase samples were taken with the hypodermic needles consecutively starting with the lowest one. Samples of the inlet and outlet streams of the column were taken at the beginning of steady-state operation, after the bell-probe samples had been taken, and after the hypodermic needle samples had been taken. Finally a piston sample was taken in order to measure the dispersed phase hold-up. Each bell-probe sample was shaken vigorously many times to bring the two phases to equilibrium. Each phase of each sample was analysed for acetic acid by titrating with standard sodium hydroxide solution as described earlier. The column operating conditions studied are given in each line of the following table.

<table>
<thead>
<tr>
<th>Run</th>
<th>Continuous phase superficial velocity, ( \text{ft}^3/\text{hr. ft}^2 )</th>
<th>Dispersed phase superficial velocity, ( \text{ft}^3/\text{hr. ft}^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>J1</td>
<td>36.5</td>
<td>54.7</td>
</tr>
<tr>
<td>J2</td>
<td>18.2</td>
<td>54.7</td>
</tr>
<tr>
<td>J3</td>
<td>36.5</td>
<td>91.2</td>
</tr>
<tr>
<td>J4</td>
<td>48.4</td>
<td>91.2</td>
</tr>
<tr>
<td>J5</td>
<td>48.4</td>
<td>128</td>
</tr>
</tbody>
</table>
RESULTS AND DISCUSSION

An initial investigation was undertaken to assess various methods of removing samples of either phase from the operating spray column. Previous measurements of concentration profiles within a liquid-liquid extraction spray column at The University of British Columbia involved the taking of samples by means of the hook and the bell-probes mentioned earlier (35, 37, 38, 39). As discussed under the heading Introduction some doubt existed as to whether the solute concentration in the continuous phase entering the hook-probe was the same as that in the continuous phase entering the bell-probe along with the drops. Accordingly various methods of sampling of the phases were investigated in this work.

A discussion of the results of the sampling technique studies is presented as a whole after all of these results are given. This procedure has been adopted in order that comparisons and contrasts between the results of various investigations can be seen more easily.

1. RESULTS OF SAMPLING TECHNIQUE STUDIES

a) Sampling with hook and bell-probes and piston with mass transfer between the phases.

Samples were taken from the operating spray column with the hook and the bell-probes at various locations shown for a
typical run in the abscissa of Figure 25. In each such run a
piston sample also was taken. The concentration of acetic acid
in the ketone phase of a bell-probe sample was calculated by
means of equation 6 as described earlier.

\[ c_D = C_D^a + \frac{V_C}{V_D} (C_C^a - C_C) \]

The value of \( c_C \) was taken to be the concentration of acetic
acid in the hook-probe sample taken from the same sampling
elevation as the bell-probe sample. An average concentration in
each phase over the piston height was determined from the
results obtained with each probe by graphical integration of
the respective concentration profiles of the sort shown in
Figure 25. The average concentration of acetic acid in the
ketone phase of the piston sample was calculated by means of
Equation 6 where \( c_C^a \) was taken to be the average concentration
in the aqueous phase over the piston height calculated from
the hook-probe sample results.

It is assumed that backmixing of the continuous phase can
be represented by "packets" of continuous phase rising with the
dispersed phase drops. Presumably these "packets" are, in fact,
mainly the wakes of rising drops*. On this basis it is shown in
Appendix III that for a piston sample the average concentration
of acetic acid in the continuous phase, excluding that in

* Note that the derivation of Equation III-8 in Appendix III is
general in that wakes are not mentioned. In the model used
there \( c_C \) is the average concentration of solute in the descending
continuous phase at the elevation under consideration.
such wakes, is given by Equation III-8.

\[
C_C = \left(\frac{V_C}{V_D}\right) C_C^a + C_D^a - C_D^i + \left(\frac{L_C}{L_D}\right) C_C^0 \left[\frac{L_D}{L_D} + \frac{V_C}{V_D}\right]
\]

The average concentration of acetic acid in the dispersed phase of each piston sample was calculated a second time by means of Equation 6. However, instead of using \(c_C\) from the hook-probe sample results as mentioned above, \(c_C\) was determined from Equation III-8 applied to the piston sample.

The results of sampling the phases in the operating spray column with the hook and the bell-probes and the piston for each of several runs are given in Table 3. The results of a typical experimental run are shown graphically in Figure 25.

b) Sampling with hook and bell-probes, hypodermic needles, and piston with mass transfer between the phases.

Further runs were made in which samples were taken as just described under a) but in which continuous phase samples also were withdrawn by means of hypodermic needles in order to provide an independent check on the hook-probe sample results. In addition to the average concentrations over the piston height calculated as described under a) all calculations involving the hook-probe were repeated for the hypodermic needle results.
TABLE 3. SAMPLING STUDIES WITH HOOK AND BELL PROBES AND PISTON

<table>
<thead>
<tr>
<th>Run</th>
<th>Superficial flowrates, ft³/hr.ft²</th>
<th>Concentration of acetic acid at the time of sampling, lb.-moles/ft³ x 10⁴</th>
<th>Bell and hook probes at position</th>
<th>Average in piston by hook and bell</th>
<th>Average over piston height by hook and bell</th>
<th>Average in piston by Equations III-8 and 6</th>
<th>Hook-probe sampling rate, ml./min.</th>
<th>Bell-probe sampling rate, ml./min.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Bell and hook probes at position</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
</tr>
<tr>
<td>2a</td>
<td>92.5</td>
<td>46.8</td>
<td>48.0</td>
<td>48.8</td>
<td>49.4</td>
<td>50.0</td>
<td>50.7</td>
<td>50.7</td>
</tr>
<tr>
<td></td>
<td>90.4</td>
<td>18.6</td>
<td>19.1</td>
<td>20.4</td>
<td>21.3</td>
<td>22.3</td>
<td>21.9</td>
<td>22.4</td>
</tr>
<tr>
<td>4</td>
<td>93.7</td>
<td>55.1</td>
<td>54.6</td>
<td>57.3</td>
<td>58.0</td>
<td>58.6</td>
<td>59.6</td>
<td>60.1</td>
</tr>
<tr>
<td></td>
<td>123.</td>
<td>21.6</td>
<td>23.5</td>
<td>23.4</td>
<td>23.9</td>
<td>24.0</td>
<td>24.2</td>
<td>25.0</td>
</tr>
<tr>
<td>5b</td>
<td>69.0</td>
<td>-</td>
<td>60.7</td>
<td>62.3</td>
<td>63.7</td>
<td>64.5</td>
<td>65.1</td>
<td>65.6</td>
</tr>
<tr>
<td></td>
<td>74.4</td>
<td>-</td>
<td>25.3</td>
<td>25.5</td>
<td>26.6</td>
<td>27.1</td>
<td>28.5</td>
<td>27.5</td>
</tr>
<tr>
<td>6</td>
<td>87.5</td>
<td>43.9</td>
<td>45.2</td>
<td>45.9</td>
<td>46.3</td>
<td>46.5</td>
<td>47.2</td>
<td>46.8</td>
</tr>
<tr>
<td></td>
<td>113.</td>
<td>18.2</td>
<td>18.7</td>
<td>19.5</td>
<td>19.7</td>
<td>19.4</td>
<td>20.1</td>
<td>20.7</td>
</tr>
<tr>
<td>7</td>
<td>87.5</td>
<td>34.8</td>
<td>39.6</td>
<td>40.5</td>
<td>40.7</td>
<td>41.3</td>
<td>41.7</td>
<td>42.3</td>
</tr>
<tr>
<td></td>
<td>113.</td>
<td>14.6</td>
<td>14.6</td>
<td>15.4</td>
<td>15.6</td>
<td>16.1</td>
<td>16.6</td>
<td>15.9</td>
</tr>
<tr>
<td>8c</td>
<td>87.5</td>
<td>40.1</td>
<td>40.2</td>
<td>42.2</td>
<td>41.7</td>
<td>43.2</td>
<td>41.7</td>
<td>41.7</td>
</tr>
<tr>
<td></td>
<td>113.</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>9d</td>
<td>87.5</td>
<td>33.0</td>
<td>35.5</td>
<td>35.0</td>
<td>35.7</td>
<td>36.2</td>
<td>36.7</td>
<td>36.9</td>
</tr>
<tr>
<td></td>
<td>113.</td>
<td>13.2</td>
<td>13.2</td>
<td>14.1</td>
<td>13.8</td>
<td>14.4</td>
<td>14.6</td>
<td>14.6</td>
</tr>
</tbody>
</table>

a For each run the upper number in each column refers to the continuous phase, the lower number to the dispersed phase. Each run was carried out at room temperature.

b The hook-probe was 0.9 inches above the sampling position indicated. The values shown for the respective concentrations of acetic acid in the hook probe samples have been taken from a plot of the measured concentrations versus distance up the column.

c The bell-probe was above the interface throughout the run.

d The hook-probe was modified as shown in the sketch on page .

e The positions at which samples were taken are indicated by number in the abscissa of Figure 25.
Figure 25. Sampling technique studies with hook and bell-probes and piston.
Table 4 shows the results of sampling with the hypodermic needles, and further results obtained with the hook and the bell-probes and the piston. The results of a typical experimental run are shown graphically in Figure 26.

c) Sampling with hook-probes, hypodermic needles, and piston with no mass transfer between the phases.

For each run a straight line was fitted by the method of least squares to a plot of the natural logarithm of the solute concentration versus height up the column for the hook-probe results and the hypodermic needle results respectively. The equation for this straight line was transformed into the equation for the concentration versus height up the column. Integration of the transformed equation produced an average concentration over the piston height. The average concentration of solute in the continuous phase of a piston sample was determined directly by analysis for solute. This concentration included the contribution of the wakes.

The results of sampling technique studies under conditions of no mass transfer are presented in Table 5. The results of a typical run are shown graphically in Figure 27.

2. DISCUSSION OF SAMPLING TECHNIQUE STUDIES

In order to understand fully what material enters a sampling device it would be necessary to know exactly the concentration of solute at every point in the column and
**TABLE 4. SAMPLING STUDIES WITH HOOK AND BELL-PROBES, HYPODERMIC NEEDLES, AND PISTON**

<table>
<thead>
<tr>
<th>Run</th>
<th>Superficial flowrate, ft²/hr.²</th>
<th>Concentration of acetic acid at the time of sampling, lb.-moles/ft³ x 10³</th>
<th>Hook-probe sampling rate ml./min</th>
<th>Bell-probe sampling rate ml./min</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Bell and hook probes at position</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>D</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>13a</td>
<td>87.5</td>
<td>28.9</td>
<td>29.6</td>
<td>29.9</td>
</tr>
<tr>
<td></td>
<td>113</td>
<td>9.87</td>
<td>10.6</td>
<td>10.9</td>
</tr>
<tr>
<td>15</td>
<td>87.5</td>
<td>26.8</td>
<td>27.3</td>
<td>28.2</td>
</tr>
<tr>
<td></td>
<td>113</td>
<td>9.83</td>
<td>10.2</td>
<td>10.5</td>
</tr>
<tr>
<td>16</td>
<td>87.5</td>
<td>27.3</td>
<td>28.1</td>
<td>29.7</td>
</tr>
<tr>
<td></td>
<td>113</td>
<td>9.6</td>
<td>10.2</td>
<td>10.9</td>
</tr>
</tbody>
</table>

Concentration of acetic acid at the time of sampling, lb.-moles/ft³ x 10³

<table>
<thead>
<tr>
<th>Concentration of acetic acid at the time of sampling, lb.-moles/ft³ x 10³</th>
<th>average over piston height by means of</th>
<th>average in piston by means of</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>hook probe and bell-probes</td>
<td>hypodermic needle and piston</td>
</tr>
<tr>
<td></td>
<td>hypodermic needles and bell-probes</td>
<td>hypodermic needles and piston</td>
</tr>
<tr>
<td></td>
<td></td>
<td>equations</td>
</tr>
<tr>
<td></td>
<td></td>
<td>III-8 and 6</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Run</th>
<th>hypodermic needle at position</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
<th>I</th>
<th>J</th>
</tr>
</thead>
<tbody>
<tr>
<td>13a</td>
<td></td>
<td>26.5</td>
<td>27.6</td>
<td>27.3</td>
<td>28.4</td>
<td>28.8</td>
<td>30.9</td>
<td>31.8</td>
<td>31.8</td>
<td>32.3</td>
<td>33.3</td>
</tr>
<tr>
<td>15</td>
<td></td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>16</td>
<td></td>
<td>-</td>
<td>25.3</td>
<td>26.0</td>
<td>26.3</td>
<td>26.3</td>
<td>26.3</td>
<td>27.0</td>
<td>28.9</td>
<td>29.4</td>
<td>29.4</td>
</tr>
</tbody>
</table>

For each run the upper number in each column refers to the continuous phase, the lower number to the dispersed phase. Each run was carried out at room temperature.

*Samples were taken 5/8-in. above the position indicated.*

*The positions at which samples were taken are indicated by number or letter in the abscissa of Figure 26.*
CONTINUOUS PHASE

DISPERSED PHASE

RUN 13
○ BY HOOK-PROBE
△ BY HYPODERMIC NEEDLES
● BY BELL-PROBE, HOOK-PROBE, & EQN. 6
□ AQUEOUS PHASE OF PISTON SAMPLE BY EQN. III-8
◆ KETONE PHASE OF PISTON SAMPLE BY NEEDLES & PISTON
◇ KETONE PHASE OF PISTON SAMPLE BY EQNS. III-8 & 6
▼ KETONE PHASE OF PISTON SAMPLE BY HOOK & PISTON

FIGURE 26. SAMPLING TECHNIQUE STUDIES WITH HOOK AND BELL-PROBES, HYPODERMIC NEEDLES AND PISTON
TABLE 5. SAMPLING TECHNIQUE STUDIES WITH NO MASS TRANSFER

<table>
<thead>
<tr>
<th>Run</th>
<th>$L_C$, $\text{ft}^3/\text{hr.}$</th>
<th>$L_D$, $\text{ft}^3/\text{hr.}$</th>
<th>Sodium concentrations, microgm./ml.</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hypodermic needle samples</td>
<td>Hook-probe samples</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Height above piston centre, in.</td>
<td>Average over piston</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>-8.96</td>
<td>-2.90</td>
</tr>
<tr>
<td>43</td>
<td>36.5</td>
<td>128</td>
<td>12.6</td>
<td>2.0</td>
</tr>
<tr>
<td>44</td>
<td>36.5</td>
<td>73</td>
<td>35.2</td>
<td>8.0</td>
</tr>
<tr>
<td>44a</td>
<td>36.5</td>
<td>128</td>
<td>15.8</td>
<td>1.6</td>
</tr>
<tr>
<td>44b</td>
<td>27.7</td>
<td>128</td>
<td>47.0</td>
<td>6.6</td>
</tr>
<tr>
<td>69</td>
<td>36.5</td>
<td>30.4</td>
<td>110.</td>
<td>61.3</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th>Piston sample</th>
<th>Temp. $^\circ F$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>43</td>
<td></td>
<td></td>
<td>0.67</td>
<td>74</td>
</tr>
<tr>
<td>44</td>
<td></td>
<td></td>
<td>3.62</td>
<td>73</td>
</tr>
<tr>
<td>44a</td>
<td></td>
<td></td>
<td>0.68</td>
<td>74</td>
</tr>
<tr>
<td>44b</td>
<td></td>
<td></td>
<td>3.9</td>
<td>73</td>
</tr>
<tr>
<td>69</td>
<td></td>
<td></td>
<td>46.3</td>
<td>72</td>
</tr>
</tbody>
</table>
FIGURE 27. SAMPLING TECHNIQUE STUDIES WITH NO MASS TRANSFER
from which point every volume element of the sample originated. Although it turns out that some conclusions can be drawn for the case of no mass transfer between the phases, sufficient knowledge is not available to do so for the case of mass transfer. Fortunately the bulk of the work described in this thesis deals with the case where conclusions can be drawn: measurement of axial eddy diffusivities where no mass transfer between the phases occurred. The comparative simplicity of the sampling techniques in the absence of mass transfer suggests that the results of these studies be discussed first.

Under conditions of no mass transfer the measured concentration of solute in the continuous phase of a piston sample is the average concentration in that phase over the piston height at the time of sampling. This average takes into account radial and axial variations in concentration, and includes the appropriate contributions from the descending continuous phase and from the rising aqueous phase in the wakes. It can be seen from Table 5 that the average solute concentration in a piston sample is found to be, to all intents and purposes, the same as the average solute concentration over the piston height calculated from the hook-probe or the hypodermic needle results. Therefore either the hook-probe or the hypodermic needles can be used to give the average concentration of solute in the continuous phase of a spray column under conditions applicable when tracer studies are being carried out.
Although, as mentioned under the heading Theory, the dispersion model envisages no radial concentration differences in the continuous phase, presumably such do occur in operating columns, for example in the wakes of rising drops. However, if the dispersion model is to be applied some average concentration must be used, and that obtained from a piston sample (and also, as shown in Table 5, by means of the hook-probe or the hypodermic needles) would seem to be a reasonable choice. Due to the many practical problems which would be encountered in incorporating several piston samplers along the length of the column and of the lengthy purge time needed in taking hook-probe samples hypodermic needles have been used for taking samples in the work involving tracer studies of axial eddy diffusivity.

With this rationale for the use of the hypodermic needles established, it is interesting to speculate on the mechanism lying behind the agreement noted in Table 5 between hypodermic needles, and hook-probe results under conditions of no mass transfer. Among the tenable postulates are the two alternatives that either the hook-probe and the hypodermic needles both sample the descending continuous phase and the rising wakes representatively, or the contribution of the wakes to the average solute concentration in a piston sample is negligible. The former explanation perhaps is possible. However, it seems unlikely that it is true because the physical natures of the hook-probe and of the hypodermic needles are different enough that these sampling devices would be expected to withdraw
continuous phase in different proportions from the wakes and from the descending aqueous phase. The second alternative, then, seems more likely to be valid than does the first and if this is accepted two possibilities arise. Either the volume of the wakes is small relative to the total volume of the continuous phase, or the concentration of solute in the bulk of the continuous phase is almost the same as that in the wakes. Letan and Kehat (121) suggest that the wakes have almost the same volume as the drops for heat transfer studies in a spray column. The present studies of sampling techniques for conditions of no mass transfer include conditions of dispersed phase hold-up as high as 16%. Therefore, accepting the views of Letan and Kehat, in tracer studies it would appear likely that the solute concentration in the wakes is almost the same as in the bulk of the continuous phase.

For continuous phase superficial velocities greater than about 36-ft.³/hr. ft² the rate of decrease of tracer concentration with height up the column is so great that the tracer concentration is measurable only in the samples taken at the first two or three sampling points above the tracer distributor. Because of this limitation the work of sampling under conditions of no mass transfer was restricted to low continuous phase superficial velocities. (The work includes both high and low dispersed phase superficial velocities.)
When the studies of sampling techniques for runs in which mass transfer took place between the two phases are considered, it is found that definite conclusions can not be drawn as to the exact significance of the results obtained. However, there are indications that the hypodermic needles withdraw continuous phase which is representative of the descending phase at the sampling elevation. A comparison between the hook-probe and the hypodermic needle results can be made with the help of Figure 26. From this figure it can be seen that the solute concentration profile in the continuous phase obtained by means of the hypodermic needles lies below that obtained with the hook-probe. The average solute concentration in the continuous phase over the piston height calculated by graphical integration of the hypodermic needle results lies very close to the average solute concentration of the descending continuous phase (i.e. wakes excluded) in the piston sample calculated by means of Equation III-8. This agreement of the results of the hypodermic needles with those calculated by means of Equation III-8 shows that, if the model of Appendix III is correct, either the hypodermic needle samples contain no continuous phase originating in the wakes, or continuous phase from this source in hypodermic needle samples does not contribute very much to the measured hypodermic needle sample concentrations. Again this small contribution could be the result of the wakes being of similar solute concentration to that of the bulk of the continuous phase, or the result of the inclusion of only a small relative volume of wake fluid in the hypodermic needle
samples. The first of these last two alternatives seem much less plausible than it did for sampling in tracer studies. In the mass transfer case, for transfer out of the continuous phase, the wake concentration would be low for two reasons: backmixing of dilute material from the lower end of the column, and transfer out of the wake into the dispersed phase. Only the first of these two factors operates in the case of the tracer studies. Unfortunately further information is needed to draw more definite conclusions for the runs with mass transfer. As there was no evidence which indicated that the needles gave erroneous results their use as sampling devices was adopted in the five mass transfer runs performed in order to investigate axial eddy diffusivity. However, this procedure perhaps was no worse in its effect than was the assumption (required for the dispersion model) of constant radial concentration in the continuous phase at a particular axial position which would not, of course, have been completely valid.

The solute concentration profile in the dispersed phase is substantially the same whether it is calculated from hook-probe and bell-probe results by means of Equation 6 or from hypodermic needles and bell-probe results by means of the same equation. (See Table 5) The reasons for this fact are that the values of $c_C$ from hypodermic needle results and from hook-probe results differ by roughly two percent only, and $\frac{V_C}{V_D}$ for a bell-probe sample is of the order of $1/7$ only. With this ratio of $V_C$ to $V_D$
a two percent change in \( c \) has only a negligible effect on the value of \( c_D \) calculated by means of Equation 6. Therefore no conclusions regarding \( c \) can be drawn from the fact that \( c_D \) from the bell-probe and the hypodermic needle results agrees with \( c_D \) obtained from the bell-probe and the hook-probe results. However, due to the calculated value of \( c_D \) for a bell-probe sample being insensitive to small errors in \( c \) it would be expected that this value of \( c_D \) is the true solute concentration in the dispersed phase entering the bell-probe if it can be assumed that the \( c \) used does involve only a small error. Now, as Table 4 shows, the average solute concentration in the dispersed phase of a piston sample calculated from hypodermic needle and piston results agrees with the average solute concentration in the dispersed phase over the piston height calculated from hypodermic needle and bell-probe results. Also, the average value of \( \frac{V_C}{V_D} \) for a piston sample is about 5, and therefore the value of \( c_D \) obtained from Equation 6 applied to a piston sample is much more sensitive to the value of \( c \) used in Equation 6 than it is for a bell-probe sample. Hence the agreement of the values of \( c_D \) as noted above implies the correctness of the value of \( c \) used in the piston case: the concentration given by the hypodermic needles. All this relies, of course, on the assumption that only small errors apply to the concentrations given by the hypodermic needles. Thus one might complain that the argument given here involves an assumption of what is being proved. However, although this
complaint is justified to a degree, it should be realized that in presenting the argument the very different $\frac{V_C}{V_D}$ ratio in the piston sample from that in the bell-probe sample strengthens the case for the correctness of the hypodermic needle samples. If these are correct, or even reasonably so, then the value of the dispersed phase concentration from the bell-probe results also is correct.

One further comparison should be made. Table 4 shows that $c_D$ obtained from the piston results in conjunction with the hook-probe results is lower than that obtained from the piston and hypodermic needle results or the piston results and Equation III-8. Table 3 shows similar low values of $c_D$ when the hook-probe analyses are used instead of continuous phase concentrations from Equation III-8 in the calculation of $c_D$ for the piston. These last observations, of course, are consistent with the fact mentioned earlier, that the value of $c_C$ obtained from Equation III-8 agrees with the hypodermic needle results.

Now, a low value of $c_D$ is obtained from a piston sample if a high value of $c_C$ is used in Equation 6. Recalling that $c_C$ from Equation III-8 assumes the inclusion of no wakes, and that if wakes were included, the value of $c_C$ would be reduced, and, therefore the value of $c_D$ calculated from piston results raised, we can see that if it were postulated that the hook-probe sample included an appreciable and effective contribution from the wakes, the value of $c_D$ calculated from piston and hook-probe results would be increased in comparison to the value of $c_D$. 
calculated from needle and bell, hook and bell, and Equation III-3 and piston, all of which are in reasonable agreement. However, \( c_D \) from hook-probe and piston results are lower than the values of \( c_D \) obtained by the other methods. Hence it seems impossible that the low \( c_D \) results from hook and piston measurements are due to the hook-probe concentrations reflecting appreciable contributions from drop wakes. In other words \( c_C \) for the hook-probe is high, not low. Furthermore in Run 4 \( c_D \) from the piston sample based on hook-probe analyses is lower, indeed, than the dispersed phase concentration at the dispersed phase inlet nozzle. This result, of course, is impossible, and lends strong support to the hypothesis that \( c_C \) from the hook-probe results generally is too high.

It seems advantageous to compare hook-probe results with reference to the dispersed phase concentrations, as has been done above, instead of comparing directly the hook-probe and hypodermic needle results. There are two reasons for this approach. First, the difference obtained between the values of \( c_D \) is numerically much larger than is the difference between the values of \( c_C \) from the hook-probe and the hypodermic needles respectively. Second, the impossible result of \( c_D \) being calculated as even lower than that in the dispersed phase entering the column is missed when the direct comparison is made between \( c_C \) from the hook-probe and \( c_C \) from the hypodermic needles.

The postulate could be put forward that the hook-probe
tends to suck in material from some elevation higher up the column than its actual position. However, when an attempt was made to lessen such an effect by directing the end of the hook horizontally, as shown in the sketch on page 65, no change in the results were obtained. Perhaps the comparatively large size of the hook-probe disturbs the flow patterns in the column to produce high continuous phase concentrations at its inlet. For example, if the hook-probe deflects drops in such a way that extraction is less complete in the neighbourhood of the hook-probe inlet, then high continuous phase concentrations would be measured by this probe.

From plots such as that shown in Figure 26, and assuming that the hypodermic needle samples are representative of the continuous phase in the column at the sampling height, it can be seen that the continuous phase entering the hook-probe appears to be representative of that phase in the column approximately 1-in. above the probe entrance. As a result the continuous phase concentration profiles given by Ewanchyna (30, 31) appear to be somewhat in error. However, this error is only slight and his conclusions mentioned under the heading Introduction regarding the end effect at the continuous phase inlet of the column are still substantially valid.

3. AXIAL EDDY DIFFUSIVITY, DROP SIZE DISTRIBUTION, AND DISPERSED PHASE HOLD-UP STUDIES IN THE 1½-IN. I.D. COLUMN

The axial eddy diffusivity, $E$, characterizes the extent to which solute is backmixed up the column, presumably through the
agency of wakes rising behind dispersed phase drops. In this work values of $E$ have been determined by means of tracer studies with no mass transfer between the phases. If mass transfer were present some degree of turbulence at the surfaces of the drops would be expected due to interfacial phenomena such as the Marangoni effect. However, the effect of this interfacial turbulence on the size of the wakes and on the manner in which solute is transferred out of and into the wakes would be expected to be negligible compared with the effect of the oscillating motion of the drops. Consequently the values of $E$ determined by tracer studies would be expected (subject to the limitations discussed on page 154) to be applicable for the case of mass transfer.

A discussion of the results of the main bulk of experiments precedes that of the preliminary experiments performed in order to test the applicability of the dispersion model and the suitability of the experimental method.

a) Axial eddy diffusivity, drop size distribution, and hold-up studies.

i) Determination of axial eddy diffusivities and Peclet numbers.

The calculations to be described here were performed on an IBM 7040 electronic computer. A data sheet, a hand calculation, and a computer output for a typical run are given in Appendix IV.

These calculations produced the following quantities: superficial axial eddy diffusivities, axial eddy diffusivities,
Peclet numbers, reduced concentrations, and mass balances. The natural logarithms of the concentrations of the samples were plotted versus height down the column. The equation for the best straight line through these points was calculated by the method of least squares. The validity of this method for calculating the straight line involved the usual assumptions concerning the normality of the distribution of the natural logarithm of the concentration (125). The superficial velocity of the continuous phase was divided by the absolute magnitude of the slope of this line to give the superficial axial eddy diffusivity, \((E_e)\). (See Equation 13.) The axial eddy diffusivity, \(E\), was calculated by dividing the superficial axial eddy diffusivity by the volumetric fraction, \(e\), of continuous phase in the column. The Peclet number, \(Pe\), was calculated by means of the following equation.

\[
Pe = \left( \frac{L_c}{e} + \frac{L_b}{1-e} \right) \left( \frac{d}{E} \right)
\]

The interpretation to be placed on the drop diameter, \(d\), is discussed below. In order that concentration profiles could be compared for various column operating conditions the reduced concentration at each sampling point was calculated by means of the following expression.

\[
\text{reduced concentration} = \frac{\text{actual concentration} \times 1000}{\text{concentration in the aqueous phase leaving the column}}
\]
Mass balances were calculated for the aqueous phase, the ketone phase, and the tracer, respectively, over the column. A set of reduced concentration profiles for one superficial velocity of ketone phase and various superficial velocities of aqueous phase is shown in Figure 28. The linearity of each plot in Figure 28 indicates that Equation 13, and hence the dispersion model concept, describe the backmixing of the continuous phase. The reduced concentration profile, the dispersed phase hold-up, the axial eddy diffusivity, the Peclet number and the mass balance results for each run are given in Table IV-4, Appendix IV.

As this table shows at the level of accuracy possible in the present experiments there was no dependence of axial eddy diffusivity on the continuous phase superficial velocity, $L_C$. Continuous phase has been found (21, 22, 23) to move up the column in the wakes of rising dispersed phase drops. The velocity of the drops is negligibly affected by a small change in the low values of $L_C$ used in the tracer studies. Correspondingly the flowrate of continuous phase up the column in the wakes of drops is substantially unaffected by such a change in the superficial velocity of the continuous phase. If the axial mixing of the continuous phase is caused by the drops and the associated wakes it is not surprising that the axial eddy diffusivity is independent of $L_C$ for the narrow range of low values of $L_C$ used in this work.
FIGURE 28. REDUCED CONCENTRATION PROFILES
Figure 29 shows the dependence of axial eddy diffusivity on the dispersed phase flowrate for one continuous phase flowrate and various drop sizes. The axial eddy diffusivity remained approximately constant as the dispersed phase flowrate was decreased from high values but at low dispersed phase flowrates the axial eddy diffusivity increased rapidly. This effect is less pronounced at small drop sizes. The effect of increasing the drop size for a given dispersed phase superficial velocity was to increase the axial eddy diffusivity.

At first it was expected that an increase in the number of drops of a given size per unit volume of column would result in a larger volume of continuous phase being carried up the column in their wakes and hence an increase in the axial eddy diffusivity, $E$. However, as mentioned above, the opposite effect is observed at low superficial velocities, $L_D$, of dispersed phase. The reason for the decrease in $E$ with an increase in the number of drops could be due to the increase in interference of the wakes of drops by neighbouring drops. This interference would tend to detach the wakes from the drops, resulting in a lower value of $E$. Apparently at low values of $L_D$ the decrease in $E$ due to interference of wakes predominates over the increase in $E$ due to the larger number of drops. At high values of $L_D$ the two effects on $E$ counterbalance each other.

An increase in the drop size, $d_D$, for a given value of $L_D$ would be expected to result in an increase in $E$ due to an increase in the size of each wake and also due to less interference of wakes by neighbouring drops. However, the value of $E$ would be expected to decrease due to a smaller
FIGURE 29. AXIAL EDDY DIFFUSIVITY IN THE 1½-IN. I.D. COLUMN
number of drops and hence fewer wakes. Apparently the effects resulting in an increase in $E$ when $d_p$ is increased predominate over those which would tend to decrease $E$.

Figure 30 shows the axial eddy diffusivity, $E$, plotted against the superficial velocity of continuous phase, $L_C$, for four different values of the superficial velocity of dispersed phase, $L_D$. The drop size, $d_p$, was 0.135-in. As mentioned earlier $E$ is independent of $L_C$ and decreases with an increase in $L_D$. Also shown in Figure 30 are the results of Hazelbeck and Geankoplis (42). They used a 1.41-in. I.D. column, a drop size, $d_p$, of 0.134-in. and water and MIBK as the continuous and dispersed phases respectively. They used an aqueous solution of potassium chloride as tracer and the step function method. They found no dependence of $E$ on $L_D$ for values of $L_D$ between 18.4-$\text{ft.}^3/\text{hr.}\cdot\text{ft.}^2$ and 49.5-$\text{ft.}^3/\text{hr.}\cdot\text{ft.}^2$, but found that $E$ increased linearly with $L_C$. As discussed on page 107 of this thesis it would be expected that $E$ should be nearly independent of $L_C$ for the narrow range of low values of $L_C$ used for the experiments described in this thesis and also those described by Hazelbeck and Geankoplis. The fact that these workers (42) found a marked increase in $E$ with an increase in $L_C$ may have been due to problems associated with transient response techniques discussed under the heading Introduction.

Figure 30a is a plot of the dispersion number, $\frac{E}{U_d d_p}$, against the Reynolds number, $\frac{d_p U_p}{\mu}$. This figure shows a summary, prepared
FIGURE 30. COMPARISON OF AXIAL EDDY DIFFUSIVITY AS DETERMINED IN THIS WORK WITH THAT OF OTHER WORKERS
FIGURE 30a. COMPARISON OF DISPERSION NUMBER AS DETERMINED IN THIS WORK WITH THAT FOR PACKED BEDS
by Levenspiel and Bischoff (73), of the data in the literature for
gas and liquid flow through packed beds. Also plotted are the
results for liquid-liquid spray tower operation determined in
this work. It can be seen from Figure 30a that the results
for spray tower operation more or less coincide with those for
liquid flow through packed beds, for the limited range of
Reynolds number investigated. This agreement supports the
correctness of the results of axial eddy diffusivity in a spray
column presented in this thesis. In addition it lends support
to the application of the mixing cell - packed bed analogy to
spray column operation described below.

Plots of drop Peclet numbers versus dispersed phase hold-up
for the four different drop size distributions studied are
presented in Figures 31, 32, 33, and 34 respectively. Peclet
numbers, predicted on the basis of the mixing cell - packed
bed analogy (44, 64, 90, 92, 108, 109) were calculated for
six lattice arrangements of drops as shown in Appendix II.
All of the predicted Peclet numbers lie in the range between
those for the lattice arrangements of orthorhombic - 2 and
rhombohedral - 1 respectively. The values of the Peclet
numbers for these two cases are plotted in each of Figures
31, 32, 33, and 34 in order that a comparison between the
calculated and the predicted Peclet numbers can be made.
The agreement between the experimental Pe and the predicted Pe
is very good for large drops and becomes progressively worse
FIGURE 31. PREDICTED AND CALCULATED PECLET NUMBERS, $d_p = 0.155$-IN.
FIGURE 32. PREDICTED AND CALCULATED PECLET NUMBERS, $d_p = 0.135$-IN.
FIGURE 33. PREDICTED AND CALCULATED PECLET NUMBERS, $d_p = 0.125$-IN.
FIGURE 34. PREDICTED AND CALCULATED PECLET NUMBERS, $d_p = 0.095$-IN.
as the drop size is reduced. A summary of the comparison is given in the table below. However, only by looking at Figures 31, 32, 33, and 34 can the trend of the experimental points away from the predicted values be seen clearly.

<table>
<thead>
<tr>
<th>$d_p$, in.</th>
<th>0.155</th>
<th>0.135</th>
<th>0.125</th>
<th>0.095</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage number of experimental points in the predicted Pe range</td>
<td>47</td>
<td>46</td>
<td>53</td>
<td>17</td>
</tr>
<tr>
<td>Percentage number of experimental points within 10% of the predicted Pe range</td>
<td>87</td>
<td>63</td>
<td>70</td>
<td>43</td>
</tr>
<tr>
<td>Largest percentage difference between the experimental Pe and the nearest side of the predicted Pe range calculated along a line of constant $h$</td>
<td>20</td>
<td>28</td>
<td>30</td>
<td>45</td>
</tr>
</tbody>
</table>

The drops, of course, do not lie in an ordered lattice arrangement but are positioned in a random fashion relative to one another at any instant in time. However, the above results indicate that the assumption of a simple lattice arrangement of drops is of some limited use in applying the mixing cell - packed bed analogy in order to make a first estimate of the Peclet number.

The mixing cell - packed analogy predicts a decrease in E with a decrease in $d_p$ at constant $h$ and it predicts a decrease in E with an increase in $h$ at constant $d_p$. The experimental results shown in Figure 29 bear out these predictions qualitatively.
Calibration for optical distortion and drop size measurements.

Figure 23 presented earlier shows the positions of the 5/32-in. ball bearing placed in the column when the optical distortion was being measured. The camera, of course, viewed the column in elevation from the front. With the camera lens used the depth of focus was such that all the drops in the column were in focus. Distortions were small in almost all cases, and changed only slightly in most cases when the ball was moved from one position to another. Hence it seemed reasonable to divide the cross-section of the column into regions in each of which the optical distortion was taken to be constant. The boundaries between regions are shown in Figure 35. These boundaries were placed equidistant from adjacent positions occupied by the ball during the taking of the photographs for calibration for optical distortion. The optical distortion of the ball was found to be independent of the height of the ball in the photographic test section of the column and also independent of the concentration of tracer in the column. By considering only the drops which were located in the central portions of the photographs it was possible to avoid making distortion corrections, since the corrections applicable to such drops were only $\pm 1\%$. Accordingly only drops in the region shown as "drop size measurement field" in Figure 35 were measured for calculating drop size distributions.

The part of the column shown in Figure 35 which was used for drop size measurements contained the whole of the central
FIGURE 35. PERCENTAGE ERROR IN THE EQUIVALENT DIAMETER DUE TO OPTICAL DISTORTION IN THE 1\frac{1}{2}-IN. I.D. COLUMN
portion, but not all of the peripheral area of the column cross-section. As a result a disproportionate number of drops appearing in the central portion of the column cross-section, as opposed to those near the column wall, were considered for drop size distribution measurements. If there were any wall effect on drop size distributions it was not taken into account.

With no liquid in the photographic test section of the column the ball was positioned at the centre of this test section. The glass column and Perspex box were removed without disturbing the ball. A photograph of the ball in air then was taken in the usual way. The diameter of the projected image of the ball in this photograph was measured. The enlargement factor for calculating the apparent dimensions of the ball during calibration studies and of drops during drop size distribution measurements was determined by dividing the measured diameter of the projected image of the ball photographed in air by the actual diameter of the ball.

The vertical and horizontal dimensions of the images of 500 drops were measured for each run in which drop size distributions were determined. Each drop was assumed to be an oblate spheroid. This assumption has been found to be reasonably good for drops issuing from the 0.103-in. I.D. nozzle tips (39). The measured drop dimensions were corrected for optical magnification, but no correction for optical distortion was applied. The equivalent drop diameter, $d_5$, was calculated for each drop by
means of the following equation

\[
d_s^3 = h_d p_d^2
\]

where

\[h_d = \text{the vertical dimension of the drop image,}
\]

and

\[p_d = \text{the horizontal dimension of the drop image.}
\]

Out of 13,000 drops measured only one drop was found to have an equivalent drop diameter of greater than 0.25-in. This drop was not considered to be typical and was not considered in drop size distribution calculations.

The range of \(d_s\) from 0.00-in. to 0.25-in. was divided up into increments of 0.01-in. An IBM - 7040 electronic computer was used to calculate the percentage of the total number of drops, and the percentage of total drop volume found in each of these increments. These calculations were performed for the first 100 drops measured, the first 200 drops measured, the first 300, the first 400, and the first 500 drops measured in each set of data. It was found that the calculated drop size distributions for 400 and 500 drops respectively were almost the same for each of the sets of data. Evidently a sample size of 500 drops is sufficiently large to be representative of the whole population of drops in the column. Accordingly all subsequent discussion involving drop size is based on a total of 500 drops for each set of data. A typical set of results for the drop size distrib-
ution calculations is given in Table IV-6 and plotted in Figure 36. A summary of all of the results is presented in Table IV-7. In each case there was a peak between 0.01-in. and 0.03-in. in the equivalent diameter on the drop size distribution plots. In addition a second peak appeared at a higher drop diameter. The mean of the two limits of the equivalent drop diameter range, of width 0.01-in., in which the second peak occurred was taken to be the drop diameter, $d_p$, used in the Peclet number calculations. For example, from Figure 36 the drop diameter of 0.135-in. was used to calculate $Pe$ for Run 50. Rocchini (39) took close-up photographs of a 1\(\frac{1}{2}\)-in. I.D. spray column using the 0.103-in. I.D. nozzle tips and with the transfer of acetic acid from the continuous phase to the dispersed phase. His photographic conditions resulted in a very short depth of focus in which drops were examined. Rocchini did not use any means for reducing optical distortion but he corrected the drop size measurements for optical distortion by means of a calibration graph. The drop size distributions which he presents (39) have peaks at exactly the same equivalent drop diameter as shown in Figure 36.

In the present work the second peak was very high and narrow for drops produced from the 0.053-in. I.D. nozzle tips and very low and broad for the 0.126-in. I.D. tips. Obviously the production of irregularly sized drops at the nozzle tips would result in a broad peak. However, it is felt that the main reason for the change in the shape of the peaks is the invalidity of the assumption that the larger drops are of oblate spheroid shape.
FIGURE 36. DROP SIZE DISTRIBUTION FOR RUN 50, AVERAGE NOZZLE TIP DIAMETER = 0.103-IN.
Typical photographs of drops produced in the $1\frac{1}{2}$-in. I.D. column by the four different sizes of nozzle tips are shown in Figure 37. From photographs such as these it can be seen that larger drops are more irregular in shape than smaller drops. Typical drop size distribution histograms for the 0.103-in. I.D. and the 0.126-in. I.D. nozzle tips are presented in Figures 36 and 38 respectively.

For each of the three smallest nozzle tip diameters the second peak in the drop size distribution plot was not influenced by run conditions. In the case of the nozzle tips of average I.D. 0.126-in. the location of the peak was influenced slightly by run conditions. The drop diameter, $d_p$, for these nozzle tips was taken as 0.155-in. Garwin and Smith (43) report that the drop size is independent of the continuous phase flowrate. This conclusion is consistent with the above observations. Figure 39 is a typical example of plots made to show the percent of total drop volume versus equivalent drop diameter. There was no noticeable peak for the very small drops in this sort of plot since the contribution of these to the total volume was negligible.

iii) Hold-up studies.

Plots are given in Figures 40, 41, 42, and 43 for the dispersed phase hold-up (measured by means of the piston) versus the dispersed phase superficial velocity for each of the four nozzle tip sizes studied. Weaver, Lapidus, and Elgin (41)
AVERAGE NOZZLE TIP DIA.  
= 0.053-IN.  
(RUN 148)

AVERAGE NOZZLE TIP DIA.  
= 0.086-IN.  
(RUN 96)

AVERAGE NOZZLE TIP DIA.  
= 0.103-IN.  
(RUN 65)

AVERAGE NOZZLE TIP DIA.  
= 0.126-IN.  
(RUN 126)

FIGURE 37. PHOTOGRAPHS OF DROPS AT OPERATING CONDITIONS CORRESPONDING TO RUNS INDICATED. MAGNIFICATION FACTOR = 3.
FIGURE 38. DROP SIZE DISTRIBUTION FOR RUN 130, AVERAGE NOZZLE TIP DIAMETER = 0.126-IN.
FIGURE 39. DISTRIBUTION OF TOTAL PERCENT OF VOLUME OF DROPS FOR RUN 50, AVERAGE NOZZLE TIP DIAMETER = 0.103-IN.
FIGURE 40. DISPERSSED PHASE HOLD-UP, \( d_p = 0.155 \)-IN.
FIGURE 41. DISPERSED PHASE HOLD-UP, \( d_p = 0.135 \text{-IN.} \)
FIGURE 42. DISPERSED PHASE HOLD-UP, \( d_p = 0.125 \text{-IN.} \)
FIGURE 43. DISPERSED PHASE HOLD-UP, $d_p = 0.095$-IN.
extended the theory relating slip velocity and hold-up in fluidized beds to spray column operation. They show that the slip velocity is expected to decrease with increasing hold-up. As a result a plot of hold-up versus dispersed phase superficial velocity, \( L_D \), is expected to be curved, concave upwards. Figures 40, 41, 42, and 43 bear out this prediction for values of \( L_D \) less than 60-ft\(^3\)/hr. ft\(^2\). However, no explanation could be found for the linearity of the hold-up curves for values of \( L_D \) greater than 60-ft\(^3\)/hr. ft\(^2\). The velocity, \( u \), of rise of the dispersed phase drops was of the order of 1000-ft./hr. and experiments were carried out for continuous phase superficial velocities, \( L_C \), between 9-ft\(^3\)/hr. ft\(^2\) and 48.4-ft\(^3\)/hr. ft\(^2\). Increasing \( L_C \) within this narrow range would be expected to result in only a small decrease in \( u \) and therefore only a slight increase in the hold-up. Although Figures 40, 41, 42, and 43 show no regular dependence of the hold-up on \( L_C \) there is a slight tendency for the hold-up to be somewhat higher at higher continuous phase flowrates. For a given dispersed phase flowrate decreasing the drop size should result in an increased hold-up since usually smaller drops have a smaller terminal velocity. However, no effect of this sort was noticed in going from a drop diameter, \( d_p \), of 0.155-in. to one of 0.135-in. as can be seen by comparing Figures 40 and 41. The reason for this might be that drops of about 0.155-in. equivalent diameter are so large that they are much distorted from the oblate spheroid shape. The distortion usually results in
a large frontal area of each drop (114) which in turn increases
the drag and lowers the terminal velocity. Comparison of
Figures 41, 42, and 43, however, shows an increase in the hold-
up as the drop size is decreased.

b) Results of the preliminary experiments.

The sampling positions 1 to 10 inclusive mentioned below
are shown in the abscissa of Figure 28.

i) Time to reach steady-state.

The solute concentrations in samples taken by means of the
first, fourth, seventh, and tenth hypodermic needles, shown in
Figure 9, above the tracer distributor, and in the aqueous phase
leaving the column were plotted versus time after start-up.
Table 6 shows the time to reach steady-state at the last sampling
position to do so.

**TABLE 6. TIME TO REACH STEADY-STATE IN THE 13-IN. I.D. COLUMN
UNDER CONDITIONS OF NO MASS TRANSFER.**

<table>
<thead>
<tr>
<th>Run</th>
<th>32</th>
<th>33</th>
<th>34</th>
</tr>
</thead>
<tbody>
<tr>
<td>( L_C ), ft(^3)/hr. ft(^2)</td>
<td>9.0</td>
<td>9.0</td>
<td>27.7</td>
</tr>
<tr>
<td>( L_D ), ft(^3)/hr. ft(^2)</td>
<td>128</td>
<td>30.4</td>
<td>30.4</td>
</tr>
<tr>
<td>time to reach steady-state, min.</td>
<td>100</td>
<td>115</td>
<td>45</td>
</tr>
<tr>
<td>Temperature, °F</td>
<td>71</td>
<td>68</td>
<td>69</td>
</tr>
</tbody>
</table>

Table 6 shows that the time to reach steady state varies little
when \( L_D \) is varied over a wide range. Hence the effect of \( L_D \) on
the time to reach steady state was not taken into account. Table 6 also shows that the time to reach steady state decreases rapidly with an increase in $L_c$. Consideration of Table 6 suggested that 1-hr. would be a conservative estimate of the time required to reach steady state when $L_c$ was greater than 30-ft$^3$/hr. ft$^2$ and that 2-hr. would be a conservative estimate of this time for values of $L_c$ between 9-ft$^3$/hr. ft$^2$ and 30-ft$^3$/hr. ft$^2$. These steady state times for various values of $L_c$ were adopted for all the studies involving tracer in the 1$\frac{3}{4}$-in. column.

ii) Effect of tracer feed rate.

Table 7 shows the results of tests performed to investigate any effects of tracer feed rate on the reduced concentration profiles or on the axial eddy diffusivity values.

**TABLE 7. EFFECT OF TRACER FEED RATE ON REDUCED CONCENTRATION PROFILES AND AXIAL EDDY DIFFUSIVITY IN THE 1$\frac{3}{4}$-IN. I.D. COLUMN.**

<table>
<thead>
<tr>
<th>Run</th>
<th>$L_c$/hr.</th>
<th>$L_D$/hr.</th>
<th>$L_T$/hr.</th>
<th>Reduced concentration at sampling positions</th>
<th>E. ft$^2$/hr.</th>
<th>Temp. °F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ft$^3$/hr.</td>
<td>ft$^3$/hr.</td>
<td>ft$^3$/hr.</td>
<td>1</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>35</td>
<td>27.7</td>
<td>30.4</td>
<td>0.16</td>
<td>599</td>
<td>411</td>
<td>271</td>
</tr>
<tr>
<td>40</td>
<td>27.7</td>
<td>30.4</td>
<td>0.31</td>
<td>557</td>
<td>380</td>
<td>246</td>
</tr>
<tr>
<td>36</td>
<td>27.7</td>
<td>30.4</td>
<td>0.62</td>
<td>621</td>
<td>389</td>
<td>251</td>
</tr>
<tr>
<td>67</td>
<td>18.2</td>
<td>73</td>
<td>0.10</td>
<td>350</td>
<td>157</td>
<td>50</td>
</tr>
<tr>
<td>57</td>
<td>18.2</td>
<td>73</td>
<td>0.20</td>
<td>433</td>
<td>166</td>
<td>76</td>
</tr>
<tr>
<td>68</td>
<td>18.2</td>
<td>73</td>
<td>0.40</td>
<td>307</td>
<td>142</td>
<td>48</td>
</tr>
</tbody>
</table>

The reduced concentration at a given sampling point varies considerably for various tracer feed rates in many cases.
However, the resulting overall concentration profile and axial eddy diffusivity are not affected appreciably by tracer-feed rate. It is concluded that the effect of tracer feed rate on the value of the axial eddy diffusivity is negligible for tracer flowrates of less than 2% of the continuous phase superficial velocity.

iii) Reproducibility of results.

Three runs were duplicated to provide a check on reproducibility of results. The reduced concentration profiles and the calculated values of the axial eddy diffusivity for these experiments are given in Table 8.

TABLE 8. REPRODUCIBILITY OF RESULTS FOR AXIAL EDDY DIFFUSIVITY IN THE 1\frac{1}{2}-IN. I.D. COLUMN.

<table>
<thead>
<tr>
<th>Run</th>
<th>$L_c$ ft$^3$/hr.</th>
<th>$L_p$ ft$^3$/hr.</th>
<th>Reduced concentration at sampling position</th>
<th>$E_a$ ft$^2$/hr.</th>
<th>Temp. °F</th>
</tr>
</thead>
<tbody>
<tr>
<td>32</td>
<td>9.0</td>
<td>128</td>
<td>575 256 155 79 48 28 14 9 3.8 2.0</td>
<td>8.6 71</td>
<td></td>
</tr>
<tr>
<td>47</td>
<td>9.0</td>
<td>128</td>
<td>529 308 157 86 46 28 14 7.8 5.3 2.9</td>
<td>8.9 71</td>
<td></td>
</tr>
<tr>
<td>33</td>
<td>9.0</td>
<td>30.4</td>
<td>755 644 566 487 391 330 297 259 223 179</td>
<td>29.3 68</td>
<td></td>
</tr>
<tr>
<td>42</td>
<td>9.0</td>
<td>30.4</td>
<td>754 668 571 492 397 252 282 250 221 179</td>
<td>28.7 73</td>
<td></td>
</tr>
<tr>
<td>34</td>
<td>27.7</td>
<td>30.4</td>
<td>523 408 257 166 93 58 38 28 19 12 33.0</td>
<td>32.6 69</td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>27.7</td>
<td>30.4</td>
<td>557 380 246 152 94 60 40 27 18 11</td>
<td>32.6 70</td>
<td></td>
</tr>
</tbody>
</table>

The reproducibility of the calculated value of the axial eddy diffusivity is good although point reduced concentrations are not exactly the same in duplicate runs.

iv) Cross-sectional homogeneity.

The dispersion model requires that at any elevation in the column the concentrations of solute in the continuous phase be
uniform. Accordingly it was necessary to check on whether or not cross-sectional homogeneity existed. Samples were withdrawn at the level of the first hypodermic needle above the tracer distributor (Figure 9) for each of the three runs listed in Table 9. This location was chosen because of all the sampling locations it would be the one most likely to exhibit non-uniformity of solute concentration. Samples were taken in each run at the positions shown in the sketch below:

![Diagram showing sampling positions](image)

The corresponding reduced concentrations appear in Table 9. Also shown is the reduced concentration at position 1 for a sample taken in each run about 40 minutes before the five samples mentioned above. Although the concentration of solute is not exactly uniform over the cross-section of the column, the variation is not large compared with the variation of concentration with time at the centre of the cross-section. Position 1 was the first sampling position above the tracer distributor. The small variation in concentration over the cross-section at this
TABLE 9. CROSS-SECTIONAL HOMOGENEITY IN THE 1/2-IN. I.D. COLUMN

<table>
<thead>
<tr>
<th>Run</th>
<th>(L_C^2/\text{ft}^3/\text{hr} \cdot \text{ft}^2)</th>
<th>(L_D^2/\text{ft}^3/\text{hr} \cdot \text{ft}^2)</th>
<th>Reduced concentration at sampling position</th>
<th>(T_{OF})</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Sampling position</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>la</td>
<td>lb</td>
</tr>
<tr>
<td>32</td>
<td>9.0</td>
<td>127.7</td>
<td>578</td>
<td>575</td>
</tr>
<tr>
<td>33</td>
<td>9.0</td>
<td>30.4</td>
<td>754</td>
<td>723</td>
</tr>
<tr>
<td>34</td>
<td>27.7</td>
<td>30.4</td>
<td>532</td>
<td>525</td>
</tr>
</tbody>
</table>

Sampling elevation indicates that tracer leaving the distributor spreads over the cross-section in a small incremental column height. Hawrelak (37) studied a 1/2-in. I.D. spray column with the transfer of acetic acid from the continuous phase to the dispersed phase. He took samples from various positions at a given cross-section by means of hypodermic needles and found no radial concentration gradients.

v) Sampling rate.

Table 10 shows the reduced concentrations of solute in samples taken from the first four sampling positions above the tracer distributor at various sampling rates. Sampling rates of 1.0-ml./min. for superficial velocities of continuous phase greater than 27.7 ft.\(^3\)/hr. ft.\(^2\) and of 0.75 ml./min. for superficial velocities of continuous phase between 9.0-ft.\(^3\)/hr. ft.\(^2\) and 27.7-ft.\(^3\)/hr. ft.\(^2\) appear to be small enough so as not to disturb the operation of the column. Accordingly these sampling rates were used for all
TABLE 10. EFFECT OF SAMPLING RATE ON THE REDUCED CONCENTRATION PROFILE IN THE 1/2-IN. I.D. COLUMN

<table>
<thead>
<tr>
<th>Run</th>
<th>( \frac{L_c}{ft^3/hr.ft.} )</th>
<th>( \frac{L_D}{ft^3/hr.ft.} )</th>
<th>Reduced concentration at sampling position</th>
<th>Temp. °F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Sampling rate</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.5 ml./min.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.0 ml./min.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2.0 ml./min.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Position 1</td>
<td>Position 2</td>
</tr>
<tr>
<td>34</td>
<td>27.7</td>
<td>30.4</td>
<td>523</td>
<td>388</td>
</tr>
<tr>
<td>33</td>
<td>9.0</td>
<td>30.4</td>
<td>749</td>
<td>642</td>
</tr>
<tr>
<td>32</td>
<td>9.0</td>
<td>128</td>
<td>755</td>
<td>575</td>
</tr>
<tr>
<td>31</td>
<td>9.0</td>
<td>128</td>
<td>751</td>
<td>573</td>
</tr>
<tr>
<td>30</td>
<td>9.0</td>
<td>128</td>
<td>751</td>
<td>573</td>
</tr>
</tbody>
</table>
of the tracer studies in the 1\(\frac{1}{2}\)-in. I.D. column.

vi) Order of sampling.

The usual order of sampling was to take a sample by means of the first hypodermic needle above the tracer distributor, then by means of the next higher needle, and so on. (See Figures 9 and 10 for sampling positions.) For three runs samples were taken in the reverse order, that is starting with the highest hypodermic needle. As well in these same runs samples were taken in the usual order. The results are given in Table 11.

It is evident from Table 11 that the order in which samples are taken does not affect the measured concentration profile.

vii) Effect of column height.

For one set of column operating conditions three experiments were performed with three different lengths of column. (See Figures 10, 11, and 12.) Table 12 shows the reduced concentration profile of solute in the test section and the calculated value of the axial eddy diffusivity for each run.

The length of the column appears to have no significant effect upon the axial eddy diffusivity.
### TABLE 11. EFFECT OF THE ORDER OF SAMPLING ON THE MEASURED CONCENTRATION PROFILE IN THE 1 3/4-IN. I.D. COLUMN

<table>
<thead>
<tr>
<th>Run</th>
<th>(L_C) ft(^3)/hr. ft(^2)</th>
<th>(L_D) ft(^3)/hr. ft(^2)</th>
<th>Order of sampling</th>
<th>Reduced concentration at sampling position</th>
<th>Temp. °F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>33</td>
<td>9.0</td>
<td>30.4</td>
<td>usual (1 to 10)</td>
<td>755</td>
<td>644</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>reverse (10 to 1)</td>
<td>747</td>
<td>642</td>
</tr>
<tr>
<td>32</td>
<td>9.0</td>
<td>128</td>
<td>usual (1 to 10)</td>
<td>574</td>
<td>256</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>reverse (10 to 1)</td>
<td>572</td>
<td>253</td>
</tr>
<tr>
<td>34</td>
<td>27.7</td>
<td>30.4</td>
<td>usual (1 to 10)</td>
<td>523</td>
<td>408</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>reverse (10 to 1)</td>
<td>526</td>
<td>374</td>
</tr>
</tbody>
</table>

### TABLE 12. EFFECT OF COLUMN HEIGHT ON THE MEASURED CONCENTRATION PROFILE AND AXIAL EDDY DIFFUSIVITY IN THE 1 3/4-IN. I.D. COLUMN

<table>
<thead>
<tr>
<th>Run</th>
<th>Column length (nozzle tips to interface)</th>
<th>(L_C) ft(^3)/hr. ft(^2)</th>
<th>(L_D) ft(^3)/hr. ft(^2)</th>
<th>Reduced concentration at sampling positions</th>
<th>E ft(^2)/hr.</th>
<th>Temp. °F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>174</td>
<td>6-ft. 3 1/8-in.</td>
<td>18.2</td>
<td>54.7</td>
<td>426</td>
<td>286</td>
<td>136</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>14.9</td>
<td>68</td>
<td></td>
</tr>
<tr>
<td>51</td>
<td>10-ft. 3 3/8-in.</td>
<td>18.2</td>
<td>54.7</td>
<td>561</td>
<td>298</td>
<td>138</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>14.5</td>
<td>71</td>
<td></td>
</tr>
<tr>
<td>175</td>
<td>16-ft. 4 3/8-in.</td>
<td>18.2</td>
<td>54.7</td>
<td>562</td>
<td>299</td>
<td>146</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>14.5</td>
<td>68</td>
<td></td>
</tr>
</tbody>
</table>
4. AXIAL EDDY DIFFUSIVITY AND DROP SIZE DISTRIBUTION STUDIES IN THE 3-IN. I.D. COLUMN.

The results of the preliminary experiments concerning steady-state time, reproducibility of results, and cross-sectional homogeneity are discussed after those of the main experiments.

a) Main Experiments.

The value of the superficial axial eddy diffusivity, \( E_e \), for each run was calculated in a manner similar to that already described for the 1\( \frac{3}{4} \)-in. I.D. column. A summary of the results for the 3-in. I.D. column is given in Table IV-5. Figure 44 shows the superficial axial eddy diffusivity plotted against dispersed phase superficial velocity. The curve shown in this figure was drawn through the points by eye. As with the 1\( \frac{3}{4} \)-in. I.D. column, the superficial axial eddy diffusivity decreases with increasing dispersed phase flowrate. However, there appears to be some tendency for higher superficial axial eddy diffusivity values for the 3-in. I.D. column to be associated with higher superficial velocities of the continuous phase. Comparison of the superficial axial eddy diffusivity results for the 3-in. I.D. column with those for the 1\( \frac{3}{4} \)-in. I.D. column shows that at a drop size, \( d_p \), of 0.135-in. and for a given superficial velocity of each phase the superficial axial eddy diffusivity in the 3-in. I.D. column was between 6.3 and 17.3 times that in the 1\( \frac{3}{4} \)-in. I.D. column. It was evident that the continuous phase underwent channelling. (See later.) This fact, no doubt, resulted in an increase in axial mixing of the continuous phase in the 3-in. I.D.
FIGURE 44. SUPERFICIAL AXIAL EDDY DIFFUSIVITY IN THE 3-IN. I.D. COLUMN
column. Gier and Hougen (28) say that the "... bulk mixing effect would be expected to be most serious in spray columns of high diameter to height ratio." However, the results of the experiments described in this thesis show that the diameter, but not the height, of the column is important in assessing the extent of the axial mixing of the continuous phase. Actual eddy diffusivities were not calculated from the superficial axial eddy diffusivities because the dispersed phase hold-up, \( h \), was not measured in the 3-in. I.D. column. Previous workers (3, 6, 30, 31, 43) have estimated the hold-up, \( h \), by means of Equation 7.

\[
h = \frac{100 L_D}{u}
\]

7

However, in the present work it was not possible to measure the velocity of rise of the dispersed phase drops with any accuracy due to the swirling motion of the drops (described later). As a result Equation 7 could not be used to estimate the hold-up, \( h \).

The percentage error in the equivalent diameter of drops due to optical distortion was determined in a manner similar to that used for the \( \frac{1}{2} \)-in. I.D. column. The optical distortion was independent of the vertical position of the ball and of the concentration of tracer in the column. Figure 45 shows this percentage error at various positions in the cross-section of the column. Also shown in Figure 45 is the portion of the cross-section in which drop size measurements were made. Just as for the \( \frac{1}{2} \)-in. I.D. column restricting the drop size measurements to the field shown in this figure made it unnecessary to apply any correction for optical distortion. A summary of the drop
FIGURE 45. PERCENTAGE ERROR IN THE EQUIVALENT DIAMETER DUE TO OPTICAL DISTORTION IN THE 3-IN. I.D. COLUMN
size distributions measured appears in Table IV-8. The second peak in each drop size distribution plot appeared between 0.13-in. and 0.14-in. equivalent drop diameter.

b) Preliminary Experiments.

i) Steady-state time.

The concentration of tracer in samples taken by means of the first, third, seventh, and tenth hypodermic needles above the tracer distributor (Figures 20 and 21) and in the aqueous phase leaving the column were plotted versus time after start-up. Table 13 shows the time to reach steady-state at the last sampling position to do so.

<table>
<thead>
<tr>
<th>TABLE 13. STEADY-STATE TIMES FOR THE 3-IN. I.D. COLUMN.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Run</td>
</tr>
<tr>
<td>L_G, ft^3/hr. ft^2</td>
</tr>
<tr>
<td>L_D, ft^3/hr. ft^2</td>
</tr>
<tr>
<td>time to reach steady-state, min.</td>
</tr>
<tr>
<td>Temperature, °F</td>
</tr>
</tbody>
</table>

Although the time to reach steady-state varied little for a wide range of dispersed phase superficial velocities it decreased markedly for an increase in the superficial velocity of continuous phase. The time for the column to reach a steady-state condition was taken to be 1-hr. for continuous phase superficial velocities of less than 100-ft^3/hr. ft^2 and 30-min. for continuous phase superficial velocities of 100-ft^3/hr. ft^2.
ii) Reproducibility of results.

The reduced concentration profiles and the superficial axial eddy diffusivities for duplicate runs are presented in Table 14.

**TABLE 14. REPRODUCIBILITY OF RESULTS FOR THE 3-IN. I.D. COLUMN**

<table>
<thead>
<tr>
<th>Run</th>
<th>$L_c$ ft$^3$/hr</th>
<th>$L_D$ ft$^3$/hr</th>
<th>Reduced concentration at sampling points</th>
<th>$E_\text{c}$ ft$^2$/hr</th>
<th>Temp. $^\circ F$</th>
</tr>
</thead>
<tbody>
<tr>
<td>177</td>
<td>18.2</td>
<td>36.5</td>
<td>933 863 844 711 707 680 656 636 593 188</td>
<td>70.188</td>
<td></td>
</tr>
<tr>
<td>186</td>
<td>18.2</td>
<td>36.5</td>
<td>921 883 819 785 761 731 689 656 620 595 190</td>
<td>70.190</td>
<td></td>
</tr>
<tr>
<td>191</td>
<td>48.4</td>
<td>54.7</td>
<td>1080 933 780 701 546 457 395 317 273 257 145</td>
<td>70.145</td>
<td></td>
</tr>
<tr>
<td>207</td>
<td>48.4</td>
<td>54.7</td>
<td>1077 892 731 608 532 444 367 310 249 218 138</td>
<td>69.138</td>
<td></td>
</tr>
<tr>
<td>208</td>
<td>100</td>
<td>109</td>
<td>1118 613 167 101 40 18.8 6.03 1.09 - -</td>
<td>53.6 67</td>
<td></td>
</tr>
<tr>
<td>208</td>
<td>100</td>
<td>109</td>
<td>1131 565 193 109 37 13.7 5.17 2.13 1.14 - -</td>
<td>56.5 68</td>
<td></td>
</tr>
</tbody>
</table>

Table 14 shows that, although point concentrations are not exactly reproducible, the reduced concentration profiles and superficial axial eddy diffusivities for duplicate runs are quite similar.

iii) Cross-sectional homogeneity.

The reduced concentrations of solute at the positions shown in the following sketch are given in Table 15 for the levels of
the first and fifth hypodermic needles above the tracer distributor. (See Figures 20 and 21.)

![Diagram of hypodermic needles and tracer distributor]

### TABLE 15. CROSS-SECTIONAL HOMOGENEITY IN THE 3-IN. I.D. COLUMN

<table>
<thead>
<tr>
<th>Run</th>
<th>( L_c ) ft³/ hr.ft²</th>
<th>( L_d ) ft³/ hr.ft²</th>
<th>Reduced concentration at sampling points</th>
<th>Temp. °F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>la</td>
<td>lb</td>
</tr>
<tr>
<td>177</td>
<td>18.2</td>
<td>36.5</td>
<td>937</td>
<td>933</td>
</tr>
<tr>
<td>180</td>
<td>100</td>
<td>36.5</td>
<td>838</td>
<td>833</td>
</tr>
<tr>
<td>197</td>
<td>18.2</td>
<td>73</td>
<td>980</td>
<td>988</td>
</tr>
<tr>
<td>195</td>
<td>100</td>
<td>73</td>
<td>1290</td>
<td>1263</td>
</tr>
<tr>
<td>203</td>
<td>18.2</td>
<td>109</td>
<td>916</td>
<td>911</td>
</tr>
<tr>
<td>201</td>
<td>100</td>
<td>109</td>
<td>1277</td>
<td>1198</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Run</th>
<th>( L_c ) ft³/ hr.ft²</th>
<th>( L_d ) ft³/ hr.ft²</th>
<th>Reduced concentration at sampling points</th>
<th>Temp. °F</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>5a</td>
<td>5b</td>
</tr>
<tr>
<td>177</td>
<td>18.2</td>
<td>36.5</td>
<td>807</td>
<td>798</td>
</tr>
<tr>
<td>180</td>
<td>100</td>
<td>36.5</td>
<td>230</td>
<td>225</td>
</tr>
<tr>
<td>197</td>
<td>18.2</td>
<td>73</td>
<td>664</td>
<td>682</td>
</tr>
<tr>
<td>195</td>
<td>100</td>
<td>73</td>
<td>194</td>
<td>184</td>
</tr>
<tr>
<td>203</td>
<td>18.2</td>
<td>109</td>
<td>611</td>
<td>599</td>
</tr>
<tr>
<td>201</td>
<td>100</td>
<td>109</td>
<td>41</td>
<td>40</td>
</tr>
</tbody>
</table>
Table 15 shows that the assumption of radial homogeneity is less valid for the 3-in. I.D. column than for the 1\frac{1}{2}-in. I.D. column. Visual observations of the very small dispersed phase drops during experimental runs showed that large scale axial swirling motions occurred in the continuous phase of the 3-in. I.D. column as mentioned later. However, the plot of the natural logarithm of the tracer concentration versus column height is straight for the runs in the 3-in. I.D. column as required by the dispersion model equation (Equation 13). Therefore a lack of cross-sectional homogeneity in the 3-in. I.D. column seems not to be too serious as far as the determination of superficial axial eddy diffusivities is concerned.

5. VISUAL OBSERVATIONS OF THE MOTION OF THE DROPS

The motion of the drops in the 1\frac{1}{2}-in. I.D. column was very erratic but always in an upwardly direction. There was no evidence of drops ever taking a downward course. The very small drops of about 0.02-in. diameter moved up the column very slowly. Due to the small size of these drops their motion would have reflected any large scale turbulence or channelling in the main bulk of the continuous phase. However no such effects were observed. These general flow characteristics were the same for the various flow-rates of the two phases and for the four different drop size distributions studied in the 1\frac{1}{2}-in. I.D. column. Evidently the drops moved up the column in effective plug flow. Thus the
assumption of no axial eddy diffusion in the dispersed phase (28, 31, 35) is valid.

In contrast to the observations of the drop motion in the 1\(\frac{1}{2}\)-in. I.D. column the drop behaviour in the 3-in. I.D. column was quite different. Not only did the very small drops exhibit an axial swirling motion but larger drops often took a downward course for a short distance along the column. Obviously the continuous phase underwent channelling. As discussed earlier this phenomenon resulted in radial inhomogeneity of the continuous phase. Due to the swirling motion of the drops the assumption of no backmixing of the dispersed phase is invalid.

6. CONCENTRATION PROFILES WITH MASS TRANSFER

Five runs were performed with the transfer of acetic acid from the continuous aqueous phase to the dispersed ketone phase in the 1\(\frac{1}{2}\)-in. I.D. column as described under Experimental Procedure. Samples were taken from within the operating column by means of the hypodermic needle samplers and also by means of the bell-probe. The concentration, \(c_D\), of acetic acid in the ketone phase of each bell-probe sample at the time of sampling was calculated by means of Equation 6 and the concentration, \(c_C\), of acetic acid in the hypodermic needle sample at the sampling elevation.

\[
c_D = c_D^a + \frac{V_C}{V_D} (c_C^a - c_C)
\]
For each run the concentration profiles of solute in both phases over the test section were plotted. Smoothed values of the concentrations of solute in each phase at the sampling positions were read from these plots. The value of the distribution coefficient, $m$, at each of the ten hypodermic needle sampling positions was calculated from the equilibrium curve for acetic acid distributed between MIBK-saturated water and water-saturated MIBK at 70°F. The arithmetic average of these ten values of $m$ was calculated and used in subsequent calculations. The equilibrium curve is shown in Figure 46 and the equilibrium data used to prepare this plot appear in Table IV-13, Appendix IV. The capacity coefficient, $K_{Da}$, was calculated by means of the smoothed concentrations and Equation 20. Integration was carried out over the test section only.

$$K_{Da} = \frac{I_D}{H} \int \frac{dc_D}{\left(\frac{c_C}{m} - c_D\right)}$$

The experimental results are presented in Table IV-9, Appendix IV.

The dispersion model characterizes the extent of backmixing of the continuous phase. It is likely that the continuous phase which is backmixed enters the wakes of drops and is transported some distance up the column as wake material before passing back into the main bulk of the continuous phase. In the tracer experiments described earlier this backmixing was studied by measuring the extent to which a solute tracer, soluble only in the continuous phase, was carried up the column.
FIGURE 46. EQUILIBRIUM CURVE FOR ACETIC ACID DISTRIBUTED BETWEEN MIBK-SATURATED WATER AND WATER-SATURATED MIBK AT 70°F.
When mass transfer can take place the simple conditions of the tracer work no longer obtain and caution must be exercised in using the tracer results and the dispersion model. The differences which can arise are that the wakes are no longer typical of the location from which they were drawn, and mass transfer can still occur from a wake while it is travelling with the drop.

In the case of mass transfer from the continuous to the dispersed phase, solute, in this case soluble in both phases, is transported along with continuous phase up the column in the wakes of the rising drops as was the case in the tracer studies. However, it is probable that the continuous phase, immediately prior to entering a wake, had been in the near vicinity of the associated drop. Consequently this continuous phase would be lower in solute concentration than the bulk of that phase at the same elevation. Therefore the amount of solute carried up the column in the rising wakes would be less than that predicted from the tracer studies wherein no such depletion of tracer could have taken place.

In addition to the above effect there is the dissimilarity of the diffusion pattern of solute between the wake and the surrounding continuous phase. In the case of the non-partitioned tracer diffusion must inevitably be from the wake to the continuous phase in which the concentration of tracer is less. However, in the case of mass transfer, as described in the present work, the wakes are moving towards a region of higher concentration of solute and therefore molecular diffusion of solute is from the continuous phase towards the wake. As has been discussed elsewhere in this thesis
molecular diffusion can be neglected with respect to eddy diffusion, and hence it is felt that this particular distortion of the wake composition can be neglected.

There is yet, however, one more factor to be considered. Mass transfer inevitably will occur between the wake and the drop itself by the transfer of the partitionable solute from the wake to the drop. For the moment our concern is with the wake itself and such mass transfer would still further deplete the solute in the wake and so when parts of this wake are cast off at higher levels the amount of solute transferred by the backmixing method will be lower than that had mass transfer not taken place. In passing one might mention that because part of the drop is exposed to the wake rather than to the surrounding continuous phase mass transfer to the drop will be lower than would have been true if the wake had not existed.

Evidently the dispersion model should be used with caution for the case of mass transfer between the phases. In spite of the above mentioned deficiencies of the dispersion model it was used in this work to analyse the results of five runs involving mass transfer.

For each run Equation 14:

\[ C_c = A \exp (\lambda_1 Z) + B \exp (\lambda_2 Z) - Q \]

was fitted to the measured concentration profile of acetic acid in the continuous phase over the test section for a value of 60-ft\(^2\)/hr. for the axial eddy diffusivity, \(E\). Values of \(A\) and \(B\) were estimated by means of the least squares technique as
described under Theory. These calculations were performed on an IBM-7040 electronic computer. The flux of solute, $J$, down the column was calculated by means of Equation 21:

$$J = \frac{1}{2} \left[ (L_D c_C^i - L_C c_D^o) + (L_C c_C^o - L_D c_D^i) \right]$$

The curve fitting was repeated for values of $E$ decreased by one for each successive trial (i.e. 59, 58, 57, etc.) until either $\lambda_1$ or $\lambda_2$ reached a value of $10^{37}$. Numbers greater than $10^{38}$ could not be handled by the IBM-7040 electronic computer. The lower limit of $E$ was between 5 and 9. The value of $E$ which resulted in the smallest sum of squares, $\Delta^2$, of the deviations between the calculated and measured values of $c_C$, was taken to be the estimate of $E$. The values of $E$ calculated by this method and the values of $E$ calculated by means of tracer studies for similar column operating conditions are given in table 16.

**TABLE 16. COMPARISON OF AXIAL EDDY DIFFUSIVITY BY MASS TRANSFER AND TRACER STUDIES.**

<table>
<thead>
<tr>
<th>Run</th>
<th>$L_0$, ft$^3$/hr*ft$^2$</th>
<th>$L_D$, ft$^3$/hr*ft$^2$</th>
<th>$E$, ft$^2$/hr.</th>
<th>Run</th>
<th>$E$, ft$^2$/hr.</th>
</tr>
</thead>
<tbody>
<tr>
<td>J1</td>
<td>36.5</td>
<td>54.7</td>
<td>16</td>
<td>49</td>
<td>15</td>
</tr>
<tr>
<td>J2</td>
<td>18.2</td>
<td>54.7</td>
<td>42</td>
<td>51</td>
<td>15</td>
</tr>
<tr>
<td>J3</td>
<td>36.5</td>
<td>91.2</td>
<td>31</td>
<td>138</td>
<td>11</td>
</tr>
<tr>
<td>J4</td>
<td>48.4</td>
<td>91.2</td>
<td>39</td>
<td>139</td>
<td>11</td>
</tr>
<tr>
<td>J5</td>
<td>48.4</td>
<td>127</td>
<td>53</td>
<td>143</td>
<td>9</td>
</tr>
</tbody>
</table>

Table IV-10 shows the point values of $\Delta$ and $E$ for the estimation...
of $E$ in each of the five runs. In each run the value of the axial eddy diffusivity, $E$, determined from concentration profiles with mass transfer was less than $E$ calculated from tracer studies for the same column operating conditions.

The estimation of $E$ for each run was repeated several times. The first repetition involved the replacement in the calculations of $J$ as given by Equation 21 by $J$ as given by Equation 25:

$$J = (L_C C^i_C - L_D C^o_D)$$

The second repetition consisted of replacing the value of $J$ used by that given by Equation 26:

$$J = (L_C C^O_C - L_D C^i_C)$$

Further repetitions involved the use of various arbitrarily chosen values of $m$ and $K_D a$. Tables IV-11 and IV-12, Appendix IV show the values of $E$ calculated for each of the three values of $J$ used and also for the various values of $m$ and $K_D a$ used for each run. Typical sets of results are shown graphically in Figures 47, 48, 49, and 50.

Figure 47 shows how $A$ passes through a minimum value as $E$ is varied arbitrarily. However, the minimum value of $A$ is not pronounced enough to enable $E$ to be estimated with any great accuracy. In addition, the true value of $E$ in any case may not correspond exactly with the minimum value of $A$ because of
\[ \Delta^2 \cdot (10^6) \text{LB-MOLES/CU.FT.}^2 \]

\[ E, \text{SQ.FT./HR.} \]

FIGURE 47. MINIMIZING \( \Delta^2 \) FOR RUN J1
FIGURE 48. THE EFFECT ON E OF VARYING THE METHOD OF FLUX CALCULATION AND OF VARYING $K_D \alpha$ FOR RUN J1
FIGURE 49. THE EFFECT ON E OF VARIATIONS IN m FOR RUN J1
FIGURE 50. MEASURED AND FITTED CONCENTRATION PROFILES FOR RUN J1
the fact that the underlying assumptions on which the least
squares fitting technique is based have no sound basis as applied
in this case. These assumptions are that the concentration of
solute in the continuous phase at each sampling point comes from
a normal distribution and that each such distribution has the
same variance at all sampling points (125).

Figure 48 shows the effect on E of calculating J by the
various methods. This figure also shows the effect of arbitrary
changes in the value of K_D used in the calculation.

The differences in the values of J used arise because the
flux of solute down the column as calculated at the upper and
at the lower ends of the column are not equal. These differences
are due solely to experimental error. Large differences result
between the values of E calculated from the various values of
J. The error in the value of K_D calculated by means of Equation
20 may be quite large due to the difference (\( \frac{c_C - c_D}{m} \)) being
small compared with \( \frac{c_C}{m} \) or \( c_D \). Figure 48 shows that the calculated
value of E is highly dependent upon the value of K_D used.
Hence small errors in \( c_C \), m, or \( c_D \) result in a large error in E.

The effect on the calculated value of E of the value of m
used is shown in Figure 49. Evidently this effect is quite
substantial. As mentioned earlier the value of m used initially
(Table IV-9) was the arithmetic average of m at the ten hypodermic
needle sampling positions. The values of \( m \) at the highest and lowest hypodermic needles for Run J1 were 1.86 and 1.99 respectively. The assumption of constancy of \( m \) over the test section obviously is not valid. Furthermore there are no theoretical grounds for taking the arithmetic average of \( m \) as that value of \( m \) which should be used. In the light of Figure 49 and these facts the calculated value of \( E \) can be considerably in error.

Figure 50 shows the measured concentrations for Run J1. Superimposed is the curve calculated from Equation 14 using the value of \( K_D \) calculated by means of Equation 20, the value of \( J \) calculated from Equation 21, and the value of \( m \) calculated as the arithmetic mean of \( m \) at the ten hypodermic needle sampling positions. Although the value of \( E \) corresponding to this curve may be in error, the agreement between the curve and the experimental points is good.
CONCLUSIONS

A better understanding of sampling methods has resulted from the work described in this thesis. At low continuous phase flowrates a hook-probe sample is representative of the continuous phase in the column at the sampling elevation. However, at high flowrates of the phases such a sample appears to be representative of continuous phase in the column at some height above the sampling elevation. Hypodermic needles (22-gauge) do, however, withdraw continuous phase which is representative of that in the column at the sampling height. The dispersed phase solute concentration obtained from the bell-probe sample is representative of the dispersed phase in the column at the sampling elevation. Equation III-8 in conjunction with the results of a piston sample and the terminal conditions of the column gives the average solute concentration in the continuous phase, excluding the contribution from the wakes, of the piston sample at the time of sampling. This calculated value of the solute concentration in the continuous phase together with Equation 6 results in the average solute concentration in the dispersed phase of the piston sample at the time of sampling.

This work has tested the dispersion model as a means of describing axial mixing of the continuous phase of a spray column. The prediction by this model of an exponential decay of solute concentration upstream, with respect to continuous phase flow,
from the injection point of a tracer soluble only in the continuous phase, is in agreement with experimental results. The axial eddy diffusivity, which characterizes the axial mixing of the continuous phase, was calculated from such results. In addition the effects of column diameter, column height, drop size, and flow-rates of the two phases have been measured experimentally. The axial eddy diffusivity of the continuous phase is independent of the superficial velocity of the continuous phase and of the column height and remains approximately constant as the dispersed phase superficial velocity is decreased from high values. However, the axial eddy diffusivity increases rapidly as the dispersed phase superficial velocity is decreased to low values. This effect is less pronounced at small drop sizes. The effect of increasing the drop size for a given dispersed phase superficial velocity is to increase the axial eddy diffusivity. The effect of increasing the column diameter is to increase the axial eddy diffusivity. For the same flowrates of respective phases in each of the two columns the superficial axial eddy diffusivity for the continuous phase is between 6.3 and 17.3 times greater in the 3-in. I.D. column than in the 1.5-in. I.D. column for the single value of \(d_p\) (0.135-in.) investigated.

The mixing cell-packed bed analogy, when applied to a spray column, predicts the Peclet number adequately for dispersed phase drops of about 0.155-in. \(d_p\). For drops of smaller equivalent diameter the agreement between the predicted Peclet
number and the measured Peclet number becomes worse.

In the present work drop size distributions were measured in order to define the physical systems existing in the various backmixing studies. This was an auxiliary study and not a complete investigation of drop size distributions for various nozzle tip sizes, nozzle tip velocities, and the like. For the restricted conditions investigated drop size distribution plots show two peaks. For all the drop formation conditions investigated in this work the first peak occurs at an equivalent drop diameter of about 0.02-in. and indicates a large number of drops of this size. The second peak is high and narrow for drops of about 0.095-in. equivalent diameter (formed at 0.053-in. I.D. nozzle tips) and becomes progressively flatter and broader as the equivalent diameter is increased to about 0.155-in. (formed at 0.126-in. I.D. nozzle tips).

The piston sampler proved to be an excellent device for use in measuring the dispersed phase hold-up. This hold-up was found to be almost independent of the continuous phase superficial velocity as, indeed, would be expected. The hold-up increases approximately linearly with dispersed phase flowrates above 60-ft$^3$/hr. ft$^2$. For a given dispersed phase flowrate the hold-up increases with decreasing drop size except when $d_p$ is greater than about 0.14-in.
Axial eddy diffusivities can be calculated from runs involving mass transfer by fitting the dispersion model equation to experimental concentration profiles. However, the eddy diffusivity values obtained were very sensitive to small changes in the flux of solute down the column, the value of the mass transfer capacity coefficient, and the value of the distribution coefficient. The effect of small changes in other parameters such as superficial velocities and dispersed phase hold-up on the calculated values of the axial eddy diffusivity were not investigated. Investigations into the validity of the boundary conditions proposed by Danckwerts (60) as applied to an experimental column might lead to the accurate prediction of solute concentration profiles by means of the dispersion model equation and axial eddy diffusivity values measured in this work by tracer experiments with no mass transfer.
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Constant of integration.</td>
</tr>
<tr>
<td>a</td>
<td>Interfacial area per unit volume of column, ( \text{ft}^2/\text{ft}^3 )</td>
</tr>
<tr>
<td>B</td>
<td>Constant of integration.</td>
</tr>
<tr>
<td>( c_B )</td>
<td>Average solute concentration in the continuous phase backmixing stream, lb.-moles/ft(^3)</td>
</tr>
<tr>
<td>( c_C = c_c/c_{CO} )</td>
<td>Reduced concentration in the continuous phase.</td>
</tr>
<tr>
<td>( c_c )</td>
<td>Solute concentration in the continuous phase, lb.-moles/ft(^3) or microgm./ml.</td>
</tr>
<tr>
<td>( c_c^a )</td>
<td>Solute concentration in the continuous phase of a bell-probe sample or a piston sample at the time of analysis, lb.-moles/ft(^3)</td>
</tr>
<tr>
<td>( c_{CO} )</td>
<td>Value of ( c_c ) when ( z = 0 ), microgm./ml.</td>
</tr>
<tr>
<td>( c_{C,j} )</td>
<td>Solute concentration in the continuous phase in the ( j^{th} ) cell of a series of perfect mixers, lb.-moles/ft(^3) or microgm./ml.</td>
</tr>
<tr>
<td>( c_c^i )</td>
<td>Solute concentration in the continuous phase inlet, lb.-moles/ft(^3)</td>
</tr>
<tr>
<td>( c_c^o )</td>
<td>Solute concentration in the continuous phase outlet, lb.-moles/ft(^3)</td>
</tr>
<tr>
<td>( c_D )</td>
<td>Solute concentration in the dispersed phase, lb.-moles/ft(^3)</td>
</tr>
</tbody>
</table>
\[ c_{D,j} \] Solute concentration in the dispersed phase in the \( j \)th cell of a series of perfect mixers, lb.-moles/ft\(^3\) or microgm./ml.

\[ c_{D}^a \] Solute concentration in the dispersed phase of a bell-probe sample or a piston sample at the time of analysis, lb.-moles/ft\(^3\).

\[ c_{D}^i \] Solute concentration in the dispersed phase inlet, lb.-moles/ft\(^3\).

\[ c_{D}^o \] Solute concentration in the dispersed phase outlet, lb.-moles/ft\(^3\).

\[ D \] Constant of integration.

\[ d_i \] Height of a mixing cell, ft.

\[ d_p \] Value of \( d_s \) at the second peak of a drop size distribution plot, in. or ft.

\[ d_s \] Equivalent drop diameter = the diameter of a sphere whose volume is the same as that of the drop, ft.

\[ E \] Continuous phase axial eddy diffusivity for the case of the dispersed phase moving relative to the co-ordinate axes, ft\(^2\)/hr.

\[ E \] Continuous phase axial eddy diffusivity for the case of the dispersed phase stationary relative to the co-ordinate axes, ft\(^2\)/hr.

\[ e = \frac{1-h}{100} \] Volumetric fraction of continuous phase in the column.

\[ H \] Test section height, ft.

\[ h \] Volume percentage of dispersed phase in the column, %.
Vertical dimension of a drop image corrected for magnification, \( ft \).

Net flux of solute down the column, \( \text{lb.-moles/(hr.} \cdot \text{ft.}^2 \).\)

Mass transfer coefficient based on \( \left[ \frac{c_C}{m} - c_D \right] \) driving force, \( \frac{\text{lb.-moles}}{(\text{hr.}) (\text{ft.}^2) (\text{lb.-moles/ft.}^3)} \).

Superficial velocity of the continuous phase back-mixing stream relative to the laboratory, \( \text{ft.}^3/(\text{hr.} \cdot \text{ft.}^2) \).

Superficial velocity of the continuous phase for the case of the dispersed phase moving relative to co-ordinate axes fixed with respect to the laboratory, \( \text{ft.}^3/(\text{hr.} \cdot \text{ft.}^2) \).

Superficial velocity of the continuous phase for the case of the dispersed phase stationary relative to co-ordinate axes. These may be either fixed or moving relative to the laboratory, \( \text{ft.}^3/(\text{hr.} \cdot \text{ft.}^2) \).

Superficial velocity of the dispersed phase relative to the laboratory, \( \text{ft.}^3/(\text{hr.} \cdot \text{ft.}^2) \).

Superficial velocity of the tracer feed relative to the laboratory, \( \text{ft.}^3/(\text{hr.} \cdot \text{ft.}^2) \).

Distribution coefficient for the solute between the continuous phase and the dispersed phase, \( \frac{\text{lb.-moles/ft.}^3}{\text{lb.-moles/ft.}^3} \).
\[
\begin{align*}
P_{e} &= \left(\frac{U_{d}}{E}\right) \\
& \quad \text{Peclet number for two phase flow.} \\
P_{e} &= \left(\frac{L_{d}}{E}\right) \\
& \quad \text{Peclet number for single phase flow.} \\
p_{d} &= \text{Horizontal dimension of a drop image corrected for}
\quad \text{magnification, ft.} \\
Q &= \frac{V}{S} \\
S &= \text{Cross-sectional area of the column, ft}^2 \\
t &= \text{Time, hr.} \\
U &= \left(\frac{L_{C} + L_{D}}{e} \right) \\
& \quad \text{Continuous phase interstitial velocity relative to}
\quad \text{the rising drops, ft./hr.} \\
u &= \left(\frac{L_{d}}{l - e}\right) \\
& \quad \text{Average linear velocity of the dispersed phase}
\quad \text{drops relative to the laboratory, ft./hr.} \\
V_{B} &= \text{Volume of the continuous phase backmixing stream}
\quad \text{in a piston sample, ft}^3 \\
V_{C} &= \text{Volume of the continuous phase in a bell-probe}
\quad \text{sample or a piston sample, ft}^3 \\
V_{D} &= \text{Volume of the dispersed phase in a bell-probe sample}
\quad \text{or a piston sample, ft}^3 \\
v_{B} &= \text{Average volume of a continuous phase backmixing}
\quad \text{"packet" associated with each dispersed phase}
\quad \text{drop, ft}^3 \\
v_{D} &= \text{Average volume of a dispersed phase drop, ft}^3 
\end{align*}
\]
\( y = z + ut \) Distance along the column, in the direction of the continuous phase flow, relative to co-ordinate axes stationary with respect to the dispersed phase drops, ft.

\( Z = z/H \) Dimensionless distance along the column in the direction of the continuous phase flow.

\( z \) Distance along the column, in the direction of the continuous phase flow, relative to co-ordinate axes stationary with respect to the laboratory, ft.

\[ \alpha = \frac{H}{2} \left[ \frac{L_C}{Ee} + K_D a \right] \text{ ft.}^{-1} \]

\[ \beta = \frac{(L_D - mL_C) K_D a H^2}{mL_D Ee} \text{ ft.}^{-1} \]

\[ \gamma = \frac{JK_D a H^2}{L_D Ee C_0} \]

\[ \Delta^2 = \sum \left[ C_C - A \exp(\lambda_1 Z) - B \exp(\lambda_2 Z) + q \right] \text{ (lb.-moles/ft.}^3) \]

\[ \lambda = \frac{d_1}{d_p} \]

\[ \lambda_1 = \alpha + \sqrt{\alpha^2 + \beta} \text{ , ft.}^{-1} \]

\[ \lambda_2 = \alpha - \sqrt{\alpha^2 + \beta} \text{ , ft.}^{-1} \]

\( \rho \) Density of the continuous phase, lb./ft.\(^3\)

\( \mu \) Viscosity of the continuous phase, lb./hr*ft.
LITERATURE CITED

40. Cavers, S. D., Birmingham University Chemical Engineer 14, 8 (1963)
67. Shemilt, L. W. and Krishnaswamy, P. R., 16th Canadian Chemical Engineering Conference, Windsor, Ontario (Oct. 1966)
73. Levenspiel, O. and Bischoff, K. B., Advan. Chem. Eng. 4, 95 (1963)


DISPERSION MODEL THEORY

The assumptions upon which the mathematical model is based are given under the heading of Theory.

Consider the control zone in the C-phase, shown in Figure I-1, during the incremental time $dt$. The case considered is one in which mass transfer is taking place between the continuous phase and the dispersed phase. A solute mass balance over the control zone gives the following terms.

i) Solute out due to mass transfer.

$$K_D \left( \frac{c_C}{m} - c_D \right) S_a (dz)(dt)$$

ii) Solute out due to C-phase flow.

$$L_C S \left( c_C + \left( \frac{\partial c_C}{\partial z} \right) (dz) \right) (dt)$$

iii) Solute in due to C-phase flow.

$$L_C S c_C (dt)$$

iv) Solute in due to eddy diffusion.

$$E \left( \frac{\partial}{\partial z} \left( \frac{c_C}{c_C} \right) \right) S_e (dt) + E e S \left( \frac{\partial c_C}{\partial z} \right) (dz) (dt) + E e S \left( \frac{\partial^2 c_C}{\partial z^2} \right) (dz) (dt)$$
FIGURE I-1. SOLUTE MASS BALANCE IN THE CONTINUOUS PHASE
v) Solute out due to eddy diffusion.

\[ E\left(\frac{\partial c_C}{\partial z}\right) (dz)eS(dt) \]

Terms i) to v) inclusive have been shown on Figure I-1 as arrows with the corresponding numbers as given here indicated beside them.

vi) Accumulation of solute.

\[ \left(\frac{\partial c_C}{\partial t}\right) Se(dz)(dt) \]

These terms combine into the following mass balance.

\[ K_D\left(\frac{c_C-c_D}{m}\right) Sa(dz)(dt)+L_C Sc(dz)(dt)+L_C S\left(\frac{\partial c_C}{\partial z}\right)(dz)(dt)+E\left(\frac{\partial c_C}{\partial z}\right) Se(dz)(dt) \]

\[ +\left(\frac{\partial c_C}{\partial t}\right) Se(dz)(dt)=L_C Sc(dz)(dt)+E\left(\frac{\partial c_C}{\partial z}\right) Se(dz)(dt)+E\left(\frac{\partial^2 c_C}{\partial z^2}\right)(dz)Se(dt) \]

The above equation can be rearranged to give

\[ Ee\left(\frac{\partial^2 c_C}{\partial z^2}\right) - L_C \left(\frac{\partial c_C}{\partial z}\right) - K_D a \left(\frac{c_C}{m} - c_D\right) = e \left(\frac{\partial c_C}{\partial t}\right) \]

I-1

At steady state Equation I-1 reduces to

\[ Ee\left(\frac{\partial^2 c_C}{\partial z^2}\right) - L_C \left(\frac{\partial c_C}{\partial z}\right) - K_D a \left(\frac{c_C}{m} - c_D\right) = 0 \]

I-2

Equation I-2 can be rearranged to give
The net flux, $J$, of solute down the column at the elevation $z$ is given by Equation I-4.

$$J = L_C c_C - \frac{B}{E} \frac{dc_C}{dz} - L_D c_D$$

I-4

The substitution of $c_D$ from Equation I-3 into Equation I-4 yields

$$J = L_C c_C - \frac{B}{E} \left( \frac{dc_C}{dz} \right) + \frac{L_D B}{K_D a} \left( \frac{d^2 c_C}{dz^2} \right) - \frac{L_D L_C}{K_D a} \left( \frac{dc_C}{dz} \right) - \frac{L_D c_C}{m}$$

I-5

Equation I-5 can be rearranged to give

$$\left( \frac{d^2 c_C}{dz^2} \right) - \left( \frac{K_D a}{L_D} \right) \frac{dc_C}{dz} - \left( \frac{L_C}{B} \right) \frac{dc_C}{dz} + \left( \frac{L_C c_K D a}{L_D B E} \right) - \left( \frac{c_K D a}{m B E} \right) = \left( \frac{J K_D a}{L_D B E} \right)$$

I-6

Concentrations and distances can be put on a dimensionless basis by means of Equations I-7 and I-8.

$$c_C = c_{CO} C_C$$

I-7

$$z = Z H$$

I-8

These two equations now can be used to transform Equation I-6 into Equation I-9.

$$\left( \frac{d^2 c_C}{dz^2} \right) - 2 \alpha \left( \frac{dc_C}{dz} \right) - \beta c_C = \gamma$$

I-9
where $\alpha, \beta$, and $\gamma$ are given by Equation I-10, I-11, and I-12.

\[
2\alpha = \left(\frac{L_C H}{B_0}\right) + \left(\frac{K_D H}{L_D}\right) \\
\beta = \frac{(L_D - mL_C)K_D aH^2}{mL_D B_0} \\
\gamma = \frac{JK_D aH^2}{L_DB_0 c_{CO}}
\]

I-10
I-11
I-12

If $\beta \neq 0$  
and  
\[(\alpha^2 + \beta) > 0\]

I-12a)
I-12b)

the solution of Equation I-9 is given by Equation I-13.

\[
C_C = A\exp (\lambda_1 Z) + B\exp (\lambda_2 Z) - Q
\]

I-13

A and B are constants of integration and $\lambda_1, \lambda_2$, and $Q$ are given by Equations I-14, I-15, and I-16 respectively.

\[
\lambda_1 = \alpha + \sqrt{\alpha^2 + \beta} \\
\lambda_2 = \alpha - \sqrt{\alpha^2 + \beta}
\]

I-14
I-15

\[
Q = \frac{\gamma}{\beta} = \frac{Jm}{c_{CO}(L_D - mL_C)}
\]

I-16

Other solutions of Equation I-9 are appropriate when the conditions given by Equation I-12a) and I-12b) are not met. However, the applicability of these other solutions is more limited than that of Equation I-13 and no extra information can be obtained by their use. When the condition given by Equation I-12b) is not satisfied problems involving instability may arise.

For a solute which dissolves only in the continuous phase  
$c_D = 0$.  

and Equation I-4 reduces to

\[ J = L_c c_c - Ee \frac{dc_c}{dz} \]  

I-17

If, in addition, continuous phase samples are withdrawn from the portion of the column which, for the continuous phase, is upstream from the feed point of tracer then

\[ J = 0. \]

Then Equation I-17 becomes

\[ L_c c_c = Ee \left( \frac{dc_c}{dz} \right) \]  

I-18

The solution of Equation I-18 is

\[ c_c = D \exp \left( \frac{L_c z}{Ee} \right), \]  

I-19

where D is a constant of integration. But

\[ c_c = c_{CO} \]  

when \( z = 0. \)

Therefore,

\[ c_c = c_{CO} \exp \left( \frac{L_c z}{Ee} \right) \]  

I-20

Equation I-20 can be rearranged to give

\[ \frac{L_c z}{Ee} = \ln \left( \frac{c_c}{c_{CO}} \right) \]  

I-21
APPENDIX II

A. MIXING CELL - PACKED BED ANALOGY \((44, 64, 90, 92, 108, 109)\)

Consider a series of perfect mixers as shown in the sketch below. Suppose that a solute is extracted from a moving continuous phase by a stationary dispersed phase. Let the inter-
stage superficial velocity of the continuous phase be \( L_C' \), the solute concentration for the continuous phase in the \( j \)th mixer be \( c_{C,j} \), and the solute concentration for the dispersed phase in the \( j \)th mixer be \( c_{D,j} \). Let the mass transfer coefficient, \( K_D \); the interfacial area per unit volume of mixer, \( a \); and the height of a mixing cell, \( d_i \), be the same for each mixer. At steady state a solute mass balance for the continuous phase of the \( j \)th mixer results in the following equation.

\[
(L_C')(c_{C,j-1}) = (L_C')(c_{C,j}) + K_D a \left( \frac{c_{C,j}}{m} - c_{D,j} \right) d_i
\]

Equation II-1 can be rearranged to give

\[
(c_{C,j-1} - c_{C,j}) - \frac{d_i K_D a}{L_C} \left( \frac{c_{C,j}}{m} - c_{D,j} \right) = 0
\]

II-2

If \( E \) is replaced by \( E' \) and \( L_C \) is replaced by \( L_C' \) Equation I-2 becomes Equation II-3 which is applicable to the continuous phase of a packed bed with solute transferred from the fluid phase to the packing.

\[
E' \left( \frac{d^2 C}{dz^2} \right) - L_C' \left( \frac{dC}{dz} \right) - K_D a \left( \frac{c_C}{m} - c_D \right) = 0
\]

II-3

It is assumed that the solute concentration in each packing piece is uniform throughout the piece at the value \( c_D \). Substitution of the central difference equivalents of differentials, as given in Equations II-4 and II-5, into Equation II-3 leads to Equation II-6 (8).
\[
\begin{align*}
\frac{dc}{dz} &= \frac{(c_{C,j+1} - c_{C,j-1})}{2d_1} \\
\frac{d^2c}{dz^2} &= \frac{(c_{C,j+1} - 2c_{C,j} + c_{C,j-1})}{d_1^2} \\
(c_{C,j-1} - c_{C,j}) \left( \frac{E_e}{d_1 L_C} \right) + (c_{C,j+1} - c_{C,j}) \left( \frac{E_e}{d_1 L_C} \right) - \frac{K_d a d_1}{L_C} \left( \frac{(c_{D,j} - c_{D,j})}{\lambda} \right) &= 0
\end{align*}
\]

By considering the interstices between the packing pieces of a packed bed as perfect mixing cells, an analogy can be drawn between a series of perfect mixers and a packed bed. For Equations II-2 and II-6 to be consistent, the following equality must be satisfied.

\[
\frac{c}{\lambda} = \left( \frac{L_C}{e} \right) \left( \frac{d_p}{E} \right),
\]

where

\[
\lambda = \frac{d_1}{d_p}.
\]

The right-hand side of Equation II-7 defines the Peclet number, Pe:

\[
Pe = \left( \frac{L_C}{e} \right) \left( \frac{d_p}{E} \right).
\]
The mixing cell length, \( d_1 \), is supposed (112) to be the vertical centre-to-centre distance between the layers of packing pieces in an ordered packing system. Although exact values for \( d_1 \) can be calculated for ordered packings, it would be expected that only an average value for \( d_1 \) could be calculated for random packings. For a packed bed the value of \( \lambda \) is approximately one. Thus for this case Equations II-7 and II-9 yield the following well known relationship (44, 64, 92, 108, 109, 122, 123).

\[ \text{Pe'} = 2 \]

B. SPRAY COLUMN - PACKED BED ANALOGY.

For a spray column operating at steady state and with a solute which dissolves only in the continuous phase Equation I-2 reduces to:

\[ \text{Be} \left( \frac{d^2 c}{dz^2} \right) = \frac{L}{U} \left( \frac{dc}{dz} \right) \]  

\[ \text{II-10} \]

Consider a spray column operating at steady state with axes of reference moving at the same velocity, \( u \), as the rising dispersed phase drops. The drops appear stationary, on a time-average basis, relative to the co-ordinate system. That is, the system appears as a packed bed with the packing pieces not touching each other. Let \( y \) be the axial position relative to the moving co-ordinate axes. Thus,

\[ y = z + ut \]

\[ \text{II-11} \]
Let the axial mixing of the continuous phase be characterized by an axial eddy diffusivity, $E'$, relative to the moving co-ordinate axes. Consider the control zone in the continuous phase as shown in the following sketch. Let the control zone be fixed relative to the moving co-ordinate system and be of incremental height $(dy)$.

A solute mass balance over the control zone for unit area of column and for an incremental time interval $(dt)$ gives

$$
\left[ (I_c + u_e)c_c + E' e \frac{\partial}{\partial y} \left( c_c + \frac{\partial c_c}{\partial y} \right)(dy) \right] (dt) = \left[ (I_c + u_e)c_c + \frac{\partial c_c}{\partial y} \right] (dy) + E' e \frac{\partial c_c}{\partial y} (dt) + \left[ u e c - u e \left( \frac{\partial c_c}{\partial y} \right)(dy) \right] (dt)
$$

The left-hand side of Equation II-12 represents the amount of solute entering the control zone. The terms in the first set of square brackets on the right-hand side of Equation II-12 represent the amount of solute leaving the control zone. The terms in the second set of square brackets on the right-hand side of Equation II-12 represent the accumulation of solute in the control zone.
Equation II-12 simplifies to

\[ E' \left( \frac{\partial^2 C}{\partial Y^2} \right) = L_c \left( \frac{\partial C}{\partial Y} \right) \]  

II-13

But

\[ \left( \frac{\partial C}{\partial Y} \right)_c = \left( \frac{dc_c}{dz} \right) \tag{124} \]

and

\[ \left( \frac{\partial^2 C}{\partial Y^2} \right)_c = \left( \frac{d^2 c_c}{dz^2} \right) \tag{124} \]

Therefore Equation II-13 becomes

\[ E' \left( \frac{d^2 C}{dz^2} \right)_c = L_c \left( \frac{dc_c}{dz} \right) \]  

II-14

A comparison of Equations II-10 and II-14 shows that

\[ E' = E \]  

II-15

The identities given in Equations II-15 above, and in II-16, and II-17 below:

\[ u = \frac{L_D}{1 - e} \]  

II-16

\[ L_c' = L_c + ue \]  

II-17

can be used with the following definition of Pe:

\[ Pe = \left( \frac{L_c'}{e} \right) \left( \frac{d_P}{E} \right) = \left[ \frac{L_c}{e} + \frac{L_D}{1 - e} \right] \left( \frac{d_P}{E} \right) \]  

II-18
and with Equation II-7 to produce Equation II-19.

\[ Pe = \frac{2}{\lambda} \]  

II-19

For simple ordered lattice arrangements of uniform spherical drops the vertical distance between layers of drops, \( d_1 \), is a function of the hold-up, \( h \), and the drop diameter, \( d_p \). The Peclet number, \( Pe \), calculated from Equation II-19, is a function of \( h \) only. The function giving \( d_1 \) and \( Pe \) are presented in the following table for six simple lattice arrangements of drops.

Each drop is supposed to be concentric with an imaginary larger sphere whose diameter is equal to the centre-to-centre distance between neighbouring drops. In the six lattice arrangements considered each imaginary sphere touches each of its nearest neighbours. These lattice arrangements represent the stable systems for beds of packed spheres and it is suggested by the author of this thesis that they are the ones most likely to be applicable for use in applying the mixing cell - packed bed analogy to a spray column.

It can be seen from the following table that for a given value of \( h \) all of the predicted \( Pe \) values lie in the range between the \( Pe \) values for the lattice arrangements of orthorhombic - 2 and rhombohedral - 1.
<table>
<thead>
<tr>
<th>LATTICE</th>
<th>$d_i$</th>
<th>Pe</th>
<th>NUMBER OF NEAREST NEIGHBOURS</th>
<th>DIAGRAM</th>
</tr>
</thead>
<tbody>
<tr>
<td>CUBIC</td>
<td>$\frac{3}{\sqrt{6}} \pi d_p^3$</td>
<td>$\frac{3}{\pi} \sqrt{48} h$</td>
<td>6</td>
<td>PLAN VIEW</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\frac{3}{\pi} \sqrt{64} h$</td>
<td></td>
<td>FRONT VIEW</td>
</tr>
<tr>
<td>ORTHORHOMBIC-1</td>
<td>$\frac{3}{\sqrt{2}} \pi d_p^3$</td>
<td>$\frac{3}{\pi} \sqrt{96} h$</td>
<td>8</td>
<td>PLAN VIEW</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\frac{3}{\pi} \sqrt{96} h$</td>
<td></td>
<td>FRONT VIEW</td>
</tr>
<tr>
<td>RHOMBOHEDRAL-1</td>
<td>$\frac{3}{\sqrt{12}} \pi d_p^3$</td>
<td>$\frac{3}{\pi} \sqrt{24 \sqrt{3}} h$</td>
<td>12</td>
<td>PLAN VIEW</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\frac{3}{\pi} \sqrt{24 \sqrt{3}} h$</td>
<td></td>
<td>FRONT VIEW</td>
</tr>
<tr>
<td>ORTHORHOMBIC-2</td>
<td>$\frac{3}{\sqrt{3}} \pi d_p^3$</td>
<td>$\frac{3}{\pi} \sqrt{32 \sqrt{3}} h$</td>
<td>8</td>
<td>PLAN VIEW</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\frac{3}{\pi} \sqrt{32 \sqrt{3}} h$</td>
<td></td>
<td>FRONT VIEW</td>
</tr>
<tr>
<td>TETRAGONAL</td>
<td>$\frac{3}{\sqrt{12}} \pi \sqrt{3} d_p^3$</td>
<td>$\frac{3}{\pi} \sqrt{32 \sqrt{3}} h$</td>
<td>10</td>
<td>PLAN VIEW</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\frac{3}{\pi} \sqrt{32 \sqrt{3}} h$</td>
<td></td>
<td>FRONT VIEW</td>
</tr>
<tr>
<td>RHOMBOHEDRAL-2</td>
<td>$\frac{3}{\sqrt{9 \sqrt{3}}} \pi d_p^3$</td>
<td>$\frac{3}{\pi} \sqrt{36 \sqrt{3}} h$</td>
<td>12</td>
<td>PLAN VIEW</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$\frac{3}{\pi} \sqrt{36 \sqrt{3}} h$</td>
<td></td>
<td>FRONT VIEW</td>
</tr>
</tbody>
</table>
APPENDIX III

ANALYSIS OF PISTON SAMPLE RESULTS TO PRODUCE THE AVERAGE CONTINUOUS PHASE CONCENTRATION, EXCLUDING THE CONTRIBUTION FROM THE WAKES, IN THE PISTON SAMPLE AT THE TIME OF SAMPLING

The analysis of piston sample results to produce the average continuous phase concentration, excluding the contribution from the wakes, in the piston at the time of sampling is based on a model in which it is pictured that each drop carries some continuous phase with it, for example in its wake (21, 22, 23, 24, 26, 121). It is assumed that on the average a volume $v_B$ of continuous phase of average solute concentration $c_B$ is carried up the column past a given elevation by each rising dispersed phase drop. Thus the frequency, $\frac{L_D}{v_D}$, of drops passing through unit area at a given elevation is equal to the frequency, $\frac{L_B}{v_B}$, of passage of volumes, $v_B$, of continuous phase in the form of a backmixing stream.

\[
\frac{L_D}{v_D} = \frac{L_B}{v_B}
\]

i.e.

Thus

\[
\frac{v_D}{v_B} = \frac{L_D}{L_B}
\]
The number of dispersed phase drops in a piston sample is

\[ \frac{V_D}{v_D} \]

and the number of packets of backmixed continuous phase in a piston sample is

\[ \frac{V_B}{v_B} \]

Therefore,

\[ \frac{V_D}{v_D} = \frac{V_B}{v_B} \]

or

\[ \frac{v_D}{v_B} = \frac{V_D}{V_B} \]

\[ \text{Equation III-2} \]

From Equations III-1 and III-2,

\[ \frac{V_D}{V_B} = \frac{L_D}{L_B} \]

or

\[ V_B = \frac{L_B V_D}{L_D} \]

\[ \text{Equation III-3} \]

Consider the lower portion of a spray column as shown in Figure III-1. It is assumed that the descending continuous phase is fully mixed at any given elevation. A mass balance on solute over the section of column shown in Figure III-1 yields
\[ L_B C_B + L_D C_D + L_C C_C^o = L_C C_C + L_B C_C + L_D C_D \]

The above equation can be rearranged to give
\[ \frac{L_B V_D (c_B - c_C)}{L_D} = \frac{L_C V_D (c_C - c_C^o) + V_D (c_D^i - c_D)}{L_D} \] III-4

Now consider a piston sample. Figure III-2a represents this sample at the instant of sampling. Figure III-2b represents the same sample but later in time: when the sample is analysed. A mass balance on solute in the piston sample over the time between sampling and analysis yields
\[ V_D C_D + V_B C_B + (V_C - V_B) c_C = V_D C_D^a + V_C C_C^a \] III-5

Substituting \( V_B \) from Equation III-3 in Equation III-5 and then rearranging gives
\[ \frac{L_B V_D (c_B - c_C)}{L_D} = \frac{V_D (c_D^a - c_D) + V_C (c_C^a - c_C)}{L_D} \] III-6

Equating
\[ \frac{L_B V_D (c_B - c_C)}{L_D} \]
as given by each of Equations III-4 and III-6 results in:
\[ \frac{V_D L_C (c_C - c_C^o) + V_D (c_D^i - c_D)}{L_D} = \frac{V_D (c_D^a - c_D) + V_C (c_C^a - c_C)}{L_D} \] III-7
Equation III-7 can be rearranged to give

\[ c_C = \frac{\left( \frac{V_C}{V_D} \right)^a c_C + c_D^a - \frac{1}{L_D} \left( \frac{I_C}{I_D} \right) c_D^o \right)}{\left( \frac{L_C}{L_D} + \frac{V_C}{V_D} \right)} \]

The concentration, \( c_C \), given by Equation III-8 is the average solute concentration in a piston sample, excluding the contribution from the wakes, at the time of sampling.
FIGURE III-1. LOWER PORTION OF A SPRAY COLUMN
FIGURE III-2. THE EFFECT OF TIME ON A PISTON SAMPLE
APPENDIX IV

CALCULATIONS AND TABULATED RESULTS FOR AXIAL EDDY DIFFUSIVITY DETERMINATIONS AND DROP SIZE DISTRIBUTIONS

a) CALCULATIONS.

A specimen hand calculation is presented starting from a data sheet shown in Table IV-1. (Actual data sheets were more abbreviated than the one shown in Table IV-1.)

RUN 50

Water flowrate = 27.7 ft³/hr. ft²
Ketone flowrate = 54.7 ft³/hr. ft²
Tracer flowrate = 0.310 ft³/hr. ft²
Column I.D. = 1.5 in. Cross-sectional area of column = 0.01227 ft²
Sampling rate = 1 ml./min. = 0.002119 ft³/hr. = 0.1727 ft³/hr. ft²
Ketone hold-up = 4.7%
### TABLE IV-1. DATA SHEET.

RUN NO. 50  
DATE: 25 AUG. 1966

<table>
<thead>
<tr>
<th>Column I.D.</th>
<th>Tracer feed conc.</th>
<th>Nozzle tip average I.D.</th>
<th>9 open nozzle tips</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5-in.</td>
<td>23,000-microgm./ml.</td>
<td>0.103-in.</td>
<td></td>
</tr>
</tbody>
</table>

Water rotometer reading = 86-mm.  
Ketone rotometer reading = 108-mm.  
Tracer rotometer reading = 7.5-mm.  

Average temperature of the fluids in the column = 72°F  
Steady state time = 1-hr.

ANALYSIS OF SAMPLES (See figure 10 for sampling positions)

<table>
<thead>
<tr>
<th>Sampling position</th>
<th>Sample collection tube</th>
<th>Dilution factor</th>
<th>Absorption reading on spectrophotometer, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>G1</td>
<td>25</td>
<td>54.7</td>
</tr>
<tr>
<td>2</td>
<td>G2</td>
<td>10</td>
<td>58.0</td>
</tr>
<tr>
<td>3</td>
<td>G3</td>
<td>5</td>
<td>48.3</td>
</tr>
<tr>
<td>4</td>
<td>G4</td>
<td>5</td>
<td>31.8</td>
</tr>
<tr>
<td>5</td>
<td>G5</td>
<td>1</td>
<td>40.6</td>
</tr>
<tr>
<td>6</td>
<td>G6</td>
<td>1</td>
<td>20.6</td>
</tr>
<tr>
<td>7</td>
<td>G7</td>
<td>1</td>
<td>8.1</td>
</tr>
<tr>
<td>8</td>
<td>G8</td>
<td>1</td>
<td>3.4</td>
</tr>
<tr>
<td>9</td>
<td>G9</td>
<td>1</td>
<td>1.6</td>
</tr>
<tr>
<td>10</td>
<td>G10</td>
<td>100</td>
<td>0.9</td>
</tr>
<tr>
<td>aqueous phase</td>
<td>G11</td>
<td></td>
<td>41.7</td>
</tr>
<tr>
<td>leaving column</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

CALIBRATION OF ATOMIC ABSORPTION SPECTROPHOTOMETER

<table>
<thead>
<tr>
<th>Tri-</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.95</td>
<td>6.45</td>
<td>0.95</td>
<td>6.25</td>
<td>116.4</td>
<td>5.3</td>
<td>4.55</td>
</tr>
<tr>
<td>2</td>
<td>0.95</td>
<td>6.85</td>
<td>0.95</td>
<td>6.65</td>
<td>116.4</td>
<td>5.7</td>
<td>4.90</td>
</tr>
<tr>
<td>3</td>
<td>1.00</td>
<td>6.60</td>
<td>1.00</td>
<td>6.40</td>
<td>116.4</td>
<td>5.4</td>
<td>4.64</td>
</tr>
</tbody>
</table>

A = reading of ketone/air interface level, ml.  
B = reading of water/ketone interface level, ml.  
C = corrected A from calibration curve, ml.  
D = corrected B from calibration curve, ml.  
E = total volume in sample, ml.  
F = volume of ketone in sample, ml.  
G = hold-up of dispersed phase, %

AQUEOUS PHASE OUTLET SAMPLE  
Collection time = 40-min.  
Weight of collection flask = 1-lb. 15-oz.  
Weight of flask + sample = 16-lb. 2-oz.  
Weight of sample = 14-lb. 3-oz.

KETONE PHASE OUTLET SAMPLE  
Collection time = 10-min.  
Weight of coll. flask = 1-lb. 5-oz.  
Weight of flask + sample = 6-lb. 14-oz.  
Weight of sample = 5-lb. 9-oz.
Calibration of atomic absorption spectrophotometer

<table>
<thead>
<tr>
<th>Sodium conc.</th>
<th>Absorption %</th>
<th>Absorbance</th>
<th>((\text{Absorbance})^2)</th>
<th>(Conc.) (Absorbance)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.0</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
</tr>
<tr>
<td>1</td>
<td>18.7</td>
<td>0.0899</td>
<td>0.0081</td>
<td>0.0899</td>
</tr>
<tr>
<td>3</td>
<td>46.7</td>
<td>0.2733</td>
<td>0.0747</td>
<td>0.8199</td>
</tr>
<tr>
<td>5</td>
<td>64.3</td>
<td>0.4473</td>
<td>0.2001</td>
<td>2.2365</td>
</tr>
<tr>
<td>7</td>
<td>76.2</td>
<td>0.6234</td>
<td>0.3886</td>
<td>4.3638</td>
</tr>
<tr>
<td>9</td>
<td>84.3</td>
<td>0.8041</td>
<td>0.6466</td>
<td>7.2369</td>
</tr>
<tr>
<td><strong>Total 25</strong></td>
<td></td>
<td><strong>2.2380</strong></td>
<td><strong>1.3181</strong></td>
<td><strong>14.7470</strong></td>
</tr>
</tbody>
</table>

Let the calibration line be

\[ \text{conc.} = (m_1)(\text{absorbance}) + k_1 \]

By least squares fit

\[ m_1 = \frac{(2.2380)(25) - (6)(14.7470)}{(2.2380)^2 - (6)(1.3180)} = 11.22 \]

and \[ k_1 = \frac{(25)(1.3181) - (2.2380)(14.7470)}{(6)(1.3181) - (2.2380)^2} = -0.018 \]

Therefore,

\[ \text{conc.} = (11.22)(\text{absorbance}) - 0.018 \]

Table IV-2 shows the calculation of the solute concentration at the sampling positions in the column. Also shown in Table IV-2 are quantities required for further computations. The dimensions of each piece of Pyrex pipe in the test section of the column are given in Table VI-1, Appendix VI.
Water balance

Density of MIBK-saturated water = 0.996 gm./ml. (Measured by means of specific gravity bottle)
Aqueous phase leaving the lower end of the column =
14 lb. 3 oz. in 40 min. = 27.897 ft\(^3\)/hr*ft\(^2\)
Sampling rate = 1 ml./min. = 0.1727 ft\(^3\)/hr*ft\(^2\)
Tracer feed rate = 0.310 ft\(^3\)/hr*ft\(^2\)
Aqueous phase fed to the column = 27.7 ft\(^3\)/hr*ft\(^2\)
Apparent loss = \[\frac{27.7 + 0.310 - 0.1727 - 27.897}{27.7}\] (100)\% = -0.22%

Ketone balance

Density of water-saturated MIBK = 0.806 gm./ml. (Measured by means of specific gravity bottle)
Ketone phase leaving the Elgin head of the column =
5 lb. 9 oz. in 10 min. = 54.07 ft\(^3\)/hr*ft\(^2\)
Ketone phase fed to the column = 54.74 ft\(^3\)/hr*ft\(^2\)
Apparent loss = \[\frac{54.74 - 54.07}{54.74}\] (100)\% = 1.22%

Tracer balance

The amount of tracer leaving the column due to sampling is neglected.
TABLE IV-2. CALCULATION OF QUANTITIES USED FOR THE CALCULATION OF E.

<table>
<thead>
<tr>
<th>Sampling position (Figs. 9 and 10)</th>
<th>Height above tracer distributor w, ft.</th>
<th>Dilution factor</th>
<th>Absorption %</th>
<th>Absorbance</th>
<th>Measured conc., microgm. per ml.</th>
<th>Sample conc. = C₀, microgm. per ml.</th>
<th>Logₑ(C₀) = q</th>
<th>( \frac{q}{w} )</th>
<th>( wq )</th>
<th>( \frac{q}{w} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.5115</td>
<td>25</td>
<td>54.7</td>
<td>0.3439</td>
<td>3.8406</td>
<td>96.015</td>
<td>4.564</td>
<td>0.2616</td>
<td>2.3345</td>
<td>20.8301</td>
</tr>
<tr>
<td>2</td>
<td>1.014</td>
<td>10</td>
<td>58.0</td>
<td>0.3768</td>
<td>4.2097</td>
<td>42.097</td>
<td>3.740</td>
<td>1.0282</td>
<td>3.7924</td>
<td>13.9876</td>
</tr>
<tr>
<td>3</td>
<td>1.517</td>
<td>5</td>
<td>48.3</td>
<td>0.2865</td>
<td>3.1965</td>
<td>15.9825</td>
<td>2.771</td>
<td>2.3013</td>
<td>4.2036</td>
<td>7.6784</td>
</tr>
<tr>
<td>4</td>
<td>2.020</td>
<td>5</td>
<td>31.8</td>
<td>0.1662</td>
<td>1.8468</td>
<td>9.2340</td>
<td>2.223</td>
<td>4.0804</td>
<td>4.4905</td>
<td>4.9417</td>
</tr>
<tr>
<td>5</td>
<td>2.527</td>
<td>1</td>
<td>40.6</td>
<td>0.2262</td>
<td>2.5200</td>
<td>2.2500</td>
<td>0.9423</td>
<td>6.3857</td>
<td>2.3812</td>
<td>0.8543</td>
</tr>
<tr>
<td>6</td>
<td>3.015</td>
<td>1</td>
<td>20.6</td>
<td>0.1002</td>
<td>1.1062</td>
<td>1.1062</td>
<td>0.1008</td>
<td>9.0902</td>
<td>0.3039</td>
<td>0.0102</td>
</tr>
<tr>
<td>7</td>
<td>3.518</td>
<td>1</td>
<td>8.1</td>
<td>0.0367</td>
<td>0.3938</td>
<td>0.3938</td>
<td>-0.9319</td>
<td>12.3763</td>
<td>-3.2784</td>
<td>0.8684</td>
</tr>
<tr>
<td>8</td>
<td>4.028</td>
<td>1</td>
<td>3.4</td>
<td>0.0150</td>
<td>0.1503</td>
<td>0.1503</td>
<td>-1.895</td>
<td>16.2248</td>
<td>-7.6331</td>
<td>3.5910</td>
</tr>
<tr>
<td>9</td>
<td>4.537</td>
<td>1</td>
<td>1.6</td>
<td>0.0070</td>
<td>0.0605</td>
<td>0.0605</td>
<td>-2.805</td>
<td>20.5844</td>
<td>-12.7263</td>
<td>7.8680</td>
</tr>
<tr>
<td>10</td>
<td>5.045</td>
<td>1</td>
<td>0.9</td>
<td>0.0039</td>
<td>0.0258</td>
<td>0.0258</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>11**</td>
<td>100</td>
<td>100</td>
<td>41.7</td>
<td>0.2343</td>
<td>2.6108</td>
<td>261.08</td>
<td>167.559</td>
<td>8.709</td>
<td>72.3329</td>
<td>-6.1317</td>
</tr>
</tbody>
</table>

* For positions 1 to 9 (incl.) only. (Sample conc. at position 10 is less than 0.05 microgm./ml.)

**Position 11 refers to the aqueous phase leaving the column.
Tracer leaving the lower end of the column =

(27.897)(261.08) = 7,283 (microgm./ml.)/(ft.³)/(hr.)/(ft.² of column)

Tracer fed to the column = (0.310)(23,000) = 7,130 (microgm./ml.)/(ft.³)/(hr.)/(ft.² of column)

Apparent loss = \[ \frac{7,130 - 7,283}{7,130} \times 100 \% = -2.15\% \]

**Axial eddy diffusivity**

The following statistical computations are based on the methods described by Bennett and Franklin (125).

n = the number of sample concentrations less than 0.05-microgm./ml. = 9

\[ S(q,w) = \frac{\sum(q)(w) - \left( \sum(q) \sum(w) \right)}{n} = \frac{-6.1317 - (22.6875)(8.709)}{9} = -28.0856 \]

\[ S(q^2) = \frac{\sum(q^2) - \left( \sum(q) \right)^2}{n} = 60.6297 - \frac{8.709^2}{9} = 52.2023 \]

\[ S(w^2) = \frac{\sum(w^2) - \left( \sum(w) \right)^2}{n} = 72.3329 - \frac{22.6875^2}{9} = 15.1415 \]

\[ \sqrt{S(w^2)} = 3.8912 \]

\[ S_{q,w}^2 = \frac{(S(q^2))(S(w^2)) - (S(q,w))^2}{(n-2)(S(w^2))} = \frac{(52.2023)(15.1415) - (-28.0856)^2}{(7)(15.1415)} = 0.01529 \]

\[ S_{q,w} = 0.1236 \]

A least squares fit (125) of the data to the equation

\[ q = (M)(w) + K \]

yields

\[ K = \frac{\left( \sum(q) \right)(\sum w^2) - \sum((q)(w)) \sum w}{n \left( \sum(w^2) \right) - (\sum w)^2} = \frac{(8.709)(72.3329) - (-6.1317)(22.6875)}{(9)(72.3329) - (22.6875)^2} = 5.640 \]
and

\[ M = S(q,w) = -1.8549 \]

\[ S(w^2) = 15.1415 \]

95% confidence limits on M are (125)

\[ M \pm t_{n-2, \alpha} \left( \frac{S_{(q,w)}}{S(w^2)} \right) \]

where

\[ t_{n-2, \alpha} = \text{the value of the t-distribution for (n-2) degrees of freedom and (100)(1-\alpha)\% confidence limits.} \]

\[ t_{7, 0.05} = 2.3646 \quad (125) \]

95% confidence limits on M are

\[ -1.8549 \pm \frac{(2.3646)(0.1236)}{3.8912} = -1.8549 \pm 0.07511 \]

\[ = -1.9300 \text{ to } -1.7798 \]

(It should be noted that for runs where n = 2 no statistical test regarding the confidence of results can be performed. For runs where n = 3 the 95% confidence limits on M are likely to be wide since

\[ t_{1, 0.05} = 12.71. \]

In fact the 95% confidence limits calculated for runs where n = 3 are not likely to represent the true situation because it is not possible to include non-statistical reasoning in the statistical analysis. For example, there is no reason to doubt the linearity of the relationship between \( q \) and \( w \) for runs where n is greater than 3.)

The superficial axial eddy diffusivity, \((Ee)\), is given by the following equation.
\( (Ee) = \frac{-L_c}{M} \)

\( (Ee) = \frac{-27.7}{-1.8549} = 14.93 \ \text{ft}^2/\text{hr.} \)

The flowrate, \( L_c \), of aqueous phase fed to the column can be measured and kept constant to within approximately 1% of \( L_c \).

On the assumption that the recorded value of \( L_c \) comes from a normal distribution an estimate of the standard deviation, \( \sigma_{L_c} \), of \( L_c \) is given by (125)

\[
\sigma_{L_c} = \frac{(L_c/100)}{3}
\]

Therefore

\[
\sigma_{L_c} = \frac{27.7}{300} = 0.09233
\]

An estimate of the variance, \( \sigma^2_M \), of \( M \) is given by

\[
\sigma^2_M = \frac{S^2_w}{\frac{1}{n-1} \sum (w^2)} = 0.01529 = 0.001010
\]

The variance, \( \sigma^2_{Ee} \), of \( (Ee) \) is given by the following equation (125)

\[
\sigma^2_{Ee} = (Ee)^2 \left[ \left( \frac{\sigma_{L_c}}{L_c} \right)^2 + \left( \frac{\sigma_M}{M} \right)^2 \right]
\]

\[
= (14.93)^2 \left[ \left( \frac{0.09233}{27.7} \right)^2 + \left( \frac{0.00101}{3.4407} \right)^2 \right] = 0.06791
\]

Therefore

\[
\sigma_{Ee} = 0.2606
\]

In calculating 95% confidence limits for \( (Ee) \) it must be remembered that \( M \) was determined from \( n \) data points. A reasonable approximation is to assume that \( (Ee) \) comes from a t-distribution.
with \( n - 2 \) degrees of freedom. Therefore the 95\% confidence limits on \( \bar{E}_e \) are

\[
(\bar{E}_e) \pm (t_{n-2, 0.05}) (\sigma_{\bar{E}_e})
\]

\[
= 14.93 \pm (2.3646)(0.2606) = 14.93 \pm 0.62
\]

\[
= 14.31 \text{ to } 15.55
\]

Now

\[
e = \left( \frac{1 - \text{hold-up}}{100} \right)
\]

The three values of \( e \) calculated from hold-up measurements are

\[
e = 1 - \frac{4.55}{100} = 0.9545
\]

\[
e = 1 - \frac{4.90}{100} = 0.9510
\]

\[
e = 1 - \frac{4.64}{100} = 0.9536
\]

The arithmetic average of \( e = 0.9545 + 0.9510 + 0.9536 = 0.9530 \)

The best estimate, \( \sigma_{e^2} \), of the variance of \( e \) is given by

\[
\sigma_{e^2} = \frac{(n)(\bar{E}_e)^2 - (\bar{E}_e)^2}{(n)(n-1)} = \frac{(3)(2.72482421) - (2.8591)^2}{(3)(2)}
\]

\[
= 0.000003303
\]

The axial eddy diffusivity, \( E \), is given by

\[
E = \frac{\bar{E}_e}{e} = \frac{14.93}{0.9530} = 15.67
\]

The variance, \( \sigma_E^2 \), of \( E \) is given by

\[
\sigma_E^2 = \left( \frac{E^2}{(\bar{E}_e)} \right) \left( \frac{(\sigma_{\bar{E}_e})^2}{(\bar{E}_e)} + (\sigma_e)^2 \right)
\]

\[
= (15.67^2) \left( \frac{(0.2606)}{14.93} \right)^2 + \frac{0.000003303}{(0.9530)^2} = 0.0748
\]
Therefore

\[ \sigma_E = 0.2735 \]

On the assumption that \( E \) comes from a t-distribution with \( (n-2) \) degrees of freedom the 95\% confidence limits for \( E \) are taken to be

\[ E \pm (t_{n-2,0.05})(\sigma_E) = 15.67 \pm (2.3646)(0.2735) \]

\[ = 15.67 \pm 0.65 = 15.02 \text{ to } 16.32 \]

The Peclet number, \( Pe \), is calculated from the following equation.

\[
Pe = \frac{(d_p) \left( \frac{L_D}{1 - e} + \frac{L_C}{e} \right)}{E} \quad \text{II-18}
\]

where \( d_p \) is the dispersed phase drop diameter.

\[
Pe = \left( \frac{0.135}{12} \right) \left( \frac{54.74 + 27.7}{0.047 \cdot 0.953} \right) = 0.857
\]

Table IV-3 shows the results of the above calculations as carried out on an IBM-7040 electronic computer.

b) TABULATED RESULTS

Table IV-4 gives the reduced concentration profiles, the dispersed phase hold-up, the number of experimental points used for the estimation of the axial eddy diffusivity, the axial eddy diffusivity, and the Peclet number for each run carried out with the 1\( \frac{1}{2} \)-in. I.D. column. The reduced concentration profile, the number of experimental points used for the estimation of the
### TABLE IV-3. TYPICAL COMPUTER RESULTS

**RUN 50**

- **WATER FLOWRATE** = 27.70 CU.FT./HR. 95 PC CONFIDENCE LIMITS ON SLOPE ARE -1.9404 TO -1.7693
- **KETONE FLOWRATE** = 54.74 CU.FT./HR. 95 PC CONFIDENCE LIMITS ON SLOPE ARE -1.9404 TO -1.7693
- **TRACER FLOWRATE** = 0.310 CU.FT./HR. 95 PC CONFIDENCE LIMITS ON SLOPE ARE -1.9404 TO -1.7693
- **NOZZLE TIP AVERAGE DIA.** = 0.75 IN. EQUIVALENT DROP DIA. = 0.14 IN.
- **AVERAGE VELOCITY OF DISPERSED PHASE IN NOZZLE TIPS** = 0.36 FT./SEC.
- **COLUMN I.D.** = 1.5 IN.
- **COLUMN HEIGHT IN NOZZLE TIPS TO INTERFACE 1** = 10 FT. 3/10-IN.
- **TEMPERATURE** = 72 °F

**CALIBRATION OF ATOMIC ABSORPTION SPECTROPHOTOMETER**

<table>
<thead>
<tr>
<th>SOD. CONC.</th>
<th>PER CENT ABSORPTION</th>
<th>CONCENTRATIONS ARE IN MICROGN./ML.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.0</td>
<td>0.000</td>
</tr>
<tr>
<td>1</td>
<td>18.7</td>
<td>0.090</td>
</tr>
<tr>
<td>2</td>
<td>46.7</td>
<td>0.273</td>
</tr>
<tr>
<td>3</td>
<td>64.3</td>
<td>0.447</td>
</tr>
<tr>
<td>4</td>
<td>72.2</td>
<td>0.623</td>
</tr>
<tr>
<td>5</td>
<td>84.3</td>
<td>0.804</td>
</tr>
</tbody>
</table>

**CONCENTRATION = 11.219*ABSORBANCE**

<table>
<thead>
<tr>
<th>POSITION</th>
<th>DILUTION</th>
<th>PER CENT ABSORPTION</th>
<th>MEASURED</th>
<th>SAMPLE</th>
<th>LOG.E</th>
<th>REDUCED</th>
</tr>
</thead>
<tbody>
<tr>
<td>101</td>
<td>25</td>
<td>54.7</td>
<td>0.344</td>
<td>3.840</td>
<td>96.000</td>
<td>4.566</td>
</tr>
<tr>
<td>102</td>
<td>10</td>
<td>50.0</td>
<td>0.277</td>
<td>4.209</td>
<td>42.087</td>
<td>3.740</td>
</tr>
<tr>
<td>103</td>
<td>20</td>
<td>46.3</td>
<td>0.166</td>
<td>1.847</td>
<td>9.233</td>
<td>2.223</td>
</tr>
<tr>
<td>104</td>
<td>40</td>
<td>31.8</td>
<td>0.077</td>
<td>1.647</td>
<td>9.233</td>
<td>2.223</td>
</tr>
<tr>
<td>105</td>
<td>60</td>
<td>20.6</td>
<td>0.033</td>
<td>0.393</td>
<td>0.393</td>
<td>0.393</td>
</tr>
<tr>
<td>106</td>
<td>70</td>
<td>12.5</td>
<td>0.015</td>
<td>0.150</td>
<td>0.150</td>
<td>0.150</td>
</tr>
<tr>
<td>107</td>
<td>30</td>
<td>9.9</td>
<td>0.007</td>
<td>0.026</td>
<td>0.026</td>
<td>0.026</td>
</tr>
<tr>
<td>108</td>
<td>40</td>
<td>1.7</td>
<td>0.004</td>
<td>0.026</td>
<td>0.026</td>
<td>0.026</td>
</tr>
<tr>
<td>109</td>
<td>50</td>
<td>1.6</td>
<td>0.004</td>
<td>0.026</td>
<td>0.026</td>
<td>0.026</td>
</tr>
<tr>
<td>110</td>
<td>60</td>
<td>1.2</td>
<td>0.004</td>
<td>0.026</td>
<td>0.026</td>
<td>0.026</td>
</tr>
</tbody>
</table>

**WATER MASS BALANCE APPARENT LOSS** = -0.201 PER CENT

**KETONE MASS BALANCE APPARENT LOSS** = 1.257 PER CENT

**TRACER MASS BALANCE APPARENT LOSS** = -2.139 PER CENT

**KETONE HOLD-UP** = 4.70 PER CENT

**LOG.E(CONC.) = -1.9404*(HEIGHT ABOVE TRACER INJECTION)+5.6416** 95 PC CONFIDENCE LIMITS ON SLOPE ARE -1.9404 TO -1.7693

**LOG.E(TODAY) = -4.216*(HEIGHT ABOVE TRACER INJECTION)+LOG.E(TODAY)**

**NUMBER OF POINTS FOR ESTIMATION OF EDDY DIFFUSIVITY = 9**

**SUPERFICIAL AXIAL EDDY DIFFUSIVITY** = 14.93 SQ.FT./HR. 95 PC CONFIDENCE LIMITS ARE 14.23 SQ.FT./HR. TO 15.63 SQ.FT./HR.

**AXIAL EDDY DIFFUSIVITY** = 15.67 SQ.FT./HR. 95 PC CONFIDENCE LIMITS ARE 14.93 SQ.FT./HR. TO 16.41 SQ.FT./HR.

**PECLET NUMBER** = 0.658
**TABLE IV-4. AXIAL EDDY DIFFUSIVITY RESULTS FOR THE 1/2-IN. I.D. COLUMN**

**AVERAGE INSIDE DIAMETER UP NOZZLE TIPS = 0.126 IN.**

**DISPERSED PHASE EQUIVALENT DROP DIAMETER = 0.195 IN.**

**AVERAGE VELOCITY OF DISPERSED PHASE IN NOZZLE TIPS = 0.36 FT./SEC.**

**COLUMN INSIDE DIAMETER = 1.5 IN.**

**COLUMN HEIGHT (IN NOZZLE TIPS TO INTERFACE) = 10 FT. 13/62 IN. (SEE FIG. 10 FOR DIAG. OF APPARATUS)**

<table>
<thead>
<tr>
<th>LV</th>
<th>CONTINUOUS PHASE SUPERFICIAL VELOCITY. CU.FT./HR./IN.</th>
<th>TRACER FEED SUPERFICIAL VELOCITY. CU.FT./HR./IN.</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.05 *</td>
<td>0.07</td>
</tr>
<tr>
<td>85</td>
<td>0.09</td>
<td>0.10</td>
</tr>
<tr>
<td>86</td>
<td>0.13</td>
<td>0.14</td>
</tr>
<tr>
<td>87</td>
<td>0.17</td>
<td>0.18</td>
</tr>
<tr>
<td>88</td>
<td>0.21</td>
<td>0.22</td>
</tr>
</tbody>
</table>

**NUMBER OF SAMPLE CONCENTRATIONS USED FOR ESTIMATION OF AXIAL EDDY DIFFUSIVITY: 95**

**WEIGHT BALANCE APPARENT LOSS, PER CENT**

<table>
<thead>
<tr>
<th>LV</th>
<th>WATER BALANCE APPARENT LOSS, PER CENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>10.86</td>
</tr>
<tr>
<td>85</td>
<td>12.27</td>
</tr>
<tr>
<td>86</td>
<td>14.25</td>
</tr>
<tr>
<td>87</td>
<td>15.65</td>
</tr>
<tr>
<td>88</td>
<td>17.06</td>
</tr>
</tbody>
</table>

**EFFECTIVE LENGTH OF COLUMN**

<table>
<thead>
<tr>
<th>LV</th>
<th>HOLD-UP</th>
<th>VOLUME PERCENTAGE OF DISPERSED PHASE IN COLUMN</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.02</td>
<td>0.04</td>
</tr>
<tr>
<td>85</td>
<td>0.03</td>
<td>0.05</td>
</tr>
<tr>
<td>86</td>
<td>0.04</td>
<td>0.06</td>
</tr>
<tr>
<td>87</td>
<td>0.05</td>
<td>0.07</td>
</tr>
<tr>
<td>88</td>
<td>0.06</td>
<td>0.08</td>
</tr>
</tbody>
</table>

**COLUMN VELOCITY, CU.FT./HR. SU.FT./HR.**

<table>
<thead>
<tr>
<th>LV</th>
<th>VELOCITY, CU.FT./HR. SU.FT./HR.</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.03</td>
</tr>
<tr>
<td>85</td>
<td>0.04</td>
</tr>
<tr>
<td>86</td>
<td>0.05</td>
</tr>
<tr>
<td>87</td>
<td>0.06</td>
</tr>
<tr>
<td>88</td>
<td>0.07</td>
</tr>
</tbody>
</table>

**CORRELATION FACTORS FOR AxIAL EDDY DIFFUSIVITY WITH 95 PER CENT CONFIDENCE LIMITS. SU.FT./HR.**

<table>
<thead>
<tr>
<th>LV</th>
<th>PE</th>
<th>EBE</th>
<th>EBE</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.05</td>
<td>0.06</td>
<td>0.07</td>
</tr>
<tr>
<td>85</td>
<td>0.08</td>
<td>0.09</td>
<td>0.10</td>
</tr>
<tr>
<td>86</td>
<td>0.10</td>
<td>0.11</td>
<td>0.12</td>
</tr>
<tr>
<td>87</td>
<td>0.13</td>
<td>0.14</td>
<td>0.15</td>
</tr>
<tr>
<td>88</td>
<td>0.15</td>
<td>0.16</td>
<td>0.17</td>
</tr>
</tbody>
</table>

**REDUCED CONCENTRATION AT SAMPLING POINTS (SEE FIG. 10)**

<table>
<thead>
<tr>
<th>LV</th>
<th>MET</th>
<th>NAW</th>
<th>MET</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>85</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td>86</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
</tr>
<tr>
<td>87</td>
<td>0.05</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>88</td>
<td>0.06</td>
<td>0.06</td>
<td>0.06</td>
</tr>
</tbody>
</table>

**HOLD-UP**

<table>
<thead>
<tr>
<th>LV</th>
<th>HOLD-UP</th>
<th>EBE</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>85</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td>86</td>
<td>0.04</td>
<td>0.04</td>
</tr>
<tr>
<td>87</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>88</td>
<td>0.06</td>
<td>0.06</td>
</tr>
</tbody>
</table>

**EFFECTIVE LENGTH OF COLUMN**

<table>
<thead>
<tr>
<th>LV</th>
<th>HOLD-UP</th>
<th>VOLUME PERCENTAGE OF DISPERSED PHASE IN COLUMN</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.02</td>
<td>0.04</td>
</tr>
<tr>
<td>85</td>
<td>0.03</td>
<td>0.05</td>
</tr>
<tr>
<td>86</td>
<td>0.04</td>
<td>0.06</td>
</tr>
<tr>
<td>87</td>
<td>0.05</td>
<td>0.07</td>
</tr>
<tr>
<td>88</td>
<td>0.06</td>
<td>0.08</td>
</tr>
</tbody>
</table>

**COLUMN VELOCITY, CU.FT./HR. SU.FT./HR.**

<table>
<thead>
<tr>
<th>LV</th>
<th>VELOCITY, CU.FT./HR. SU.FT./HR.</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.03</td>
</tr>
<tr>
<td>85</td>
<td>0.04</td>
</tr>
<tr>
<td>86</td>
<td>0.05</td>
</tr>
<tr>
<td>87</td>
<td>0.06</td>
</tr>
<tr>
<td>88</td>
<td>0.07</td>
</tr>
</tbody>
</table>

**CORRELATION FACTORS FOR AXIAL EDDY DIFFUSIVITY WITH 95 PER CENT CONFIDENCE LIMITS. SU.FT./HR.**

<table>
<thead>
<tr>
<th>LV</th>
<th>PE</th>
<th>EBE</th>
<th>EBE</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.05</td>
<td>0.06</td>
<td>0.07</td>
</tr>
<tr>
<td>85</td>
<td>0.08</td>
<td>0.09</td>
<td>0.10</td>
</tr>
<tr>
<td>86</td>
<td>0.10</td>
<td>0.11</td>
<td>0.12</td>
</tr>
<tr>
<td>87</td>
<td>0.13</td>
<td>0.14</td>
<td>0.15</td>
</tr>
<tr>
<td>88</td>
<td>0.15</td>
<td>0.16</td>
<td>0.17</td>
</tr>
</tbody>
</table>

**REDUCED CONCENTRATION AT SAMPLING POINTS (SEE FIG. 10)**

<table>
<thead>
<tr>
<th>LV</th>
<th>MET</th>
<th>NAW</th>
<th>MET</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>85</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td>86</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
</tr>
<tr>
<td>87</td>
<td>0.05</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>88</td>
<td>0.06</td>
<td>0.06</td>
<td>0.06</td>
</tr>
</tbody>
</table>

**HOLD-UP**

<table>
<thead>
<tr>
<th>LV</th>
<th>HOLD-UP</th>
<th>EBE</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>85</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td>86</td>
<td>0.04</td>
<td>0.04</td>
</tr>
<tr>
<td>87</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>88</td>
<td>0.06</td>
<td>0.06</td>
</tr>
</tbody>
</table>

**EFFECTIVE LENGTH OF COLUMN**

<table>
<thead>
<tr>
<th>LV</th>
<th>HOLD-UP</th>
<th>VOLUME PERCENTAGE OF DISPERSED PHASE IN COLUMN</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.02</td>
<td>0.04</td>
</tr>
<tr>
<td>85</td>
<td>0.03</td>
<td>0.05</td>
</tr>
<tr>
<td>86</td>
<td>0.04</td>
<td>0.06</td>
</tr>
<tr>
<td>87</td>
<td>0.05</td>
<td>0.07</td>
</tr>
<tr>
<td>88</td>
<td>0.06</td>
<td>0.08</td>
</tr>
</tbody>
</table>

**COLUMN VELOCITY, CU.FT./HR. SU.FT./HR.**

<table>
<thead>
<tr>
<th>LV</th>
<th>VELOCITY, CU.FT./HR. SU.FT./HR.</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.03</td>
</tr>
<tr>
<td>85</td>
<td>0.04</td>
</tr>
<tr>
<td>86</td>
<td>0.05</td>
</tr>
<tr>
<td>87</td>
<td>0.06</td>
</tr>
<tr>
<td>88</td>
<td>0.07</td>
</tr>
</tbody>
</table>

**CORRELATION FACTORS FOR AXIAL EDDY DIFFUSIVITY WITH 95 PER CENT CONFIDENCE LIMITS. SU.FT./HR.**

<table>
<thead>
<tr>
<th>LV</th>
<th>PE</th>
<th>EBE</th>
<th>EBE</th>
</tr>
</thead>
<tbody>
<tr>
<td>84</td>
<td>0.05</td>
<td>0.06</td>
<td>0.07</td>
</tr>
<tr>
<td>85</td>
<td>0.08</td>
<td>0.09</td>
<td>0.10</td>
</tr>
<tr>
<td>86</td>
<td>0.10</td>
<td>0.11</td>
<td>0.12</td>
</tr>
<tr>
<td>87</td>
<td>0.13</td>
<td>0.14</td>
<td>0.15</td>
</tr>
<tr>
<td>88</td>
<td>0.15</td>
<td>0.16</td>
<td>0.17</td>
</tr>
</tbody>
</table>
### Table IV - 4. Continued (1)

<table>
<thead>
<tr>
<th>RUN</th>
<th>L1</th>
<th>L2</th>
<th>LT</th>
<th>TEMP</th>
<th>CONC</th>
<th>REDUCED CONCENTRATION AT SAMPLING POINTS (See Fig. 10)</th>
<th>HOLD</th>
<th>FF</th>
<th>T</th>
<th>ER1</th>
<th>ER2</th>
<th>ER3</th>
</tr>
</thead>
<tbody>
<tr>
<td>41</td>
<td>9.0</td>
<td>10.0</td>
<td>0.012</td>
<td>72</td>
<td>595.60</td>
<td>554.02</td>
<td>264.35</td>
<td>399.85</td>
<td>341.54</td>
<td>183.84</td>
<td>19.78</td>
<td>24.00</td>
</tr>
<tr>
<td>41</td>
<td>9.0</td>
<td>10.0</td>
<td>0.002</td>
<td>72</td>
<td>595.60</td>
<td>554.02</td>
<td>264.35</td>
<td>399.85</td>
<td>341.54</td>
<td>183.84</td>
<td>19.78</td>
<td>24.00</td>
</tr>
<tr>
<td>41</td>
<td>9.0</td>
<td>10.0</td>
<td>0.012</td>
<td>72</td>
<td>595.60</td>
<td>554.02</td>
<td>264.35</td>
<td>399.85</td>
<td>341.54</td>
<td>183.84</td>
<td>19.78</td>
<td>24.00</td>
</tr>
<tr>
<td>41</td>
<td>9.0</td>
<td>10.0</td>
<td>0.012</td>
<td>72</td>
<td>595.60</td>
<td>554.02</td>
<td>264.35</td>
<td>399.85</td>
<td>341.54</td>
<td>183.84</td>
<td>19.78</td>
<td>24.00</td>
</tr>
<tr>
<td>41</td>
<td>9.0</td>
<td>10.0</td>
<td>0.012</td>
<td>72</td>
<td>595.60</td>
<td>554.02</td>
<td>264.35</td>
<td>399.85</td>
<td>341.54</td>
<td>183.84</td>
<td>19.78</td>
<td>24.00</td>
</tr>
<tr>
<td>41</td>
<td>9.0</td>
<td>10.0</td>
<td>0.012</td>
<td>72</td>
<td>595.60</td>
<td>554.02</td>
<td>264.35</td>
<td>399.85</td>
<td>341.54</td>
<td>183.84</td>
<td>19.78</td>
<td>24.00</td>
</tr>
<tr>
<td>41</td>
<td>9.0</td>
<td>10.0</td>
<td>0.012</td>
<td>72</td>
<td>595.60</td>
<td>554.02</td>
<td>264.35</td>
<td>399.85</td>
<td>341.54</td>
<td>183.84</td>
<td>19.78</td>
<td>24.00</td>
</tr>
<tr>
<td>41</td>
<td>9.0</td>
<td>10.0</td>
<td>0.012</td>
<td>72</td>
<td>595.60</td>
<td>554.02</td>
<td>264.35</td>
<td>399.85</td>
<td>341.54</td>
<td>183.84</td>
<td>19.78</td>
<td>24.00</td>
</tr>
</tbody>
</table>

**Notes:**
- L1, L2, LT, TEMP: Likely parameters for experiments or conditions.
- CONC: Concentration values.
- REDUCED CONCENTRATION DEPENDS ON (See Fig. 10): Specific concentration dependence.
- HOLD, FF, T, ER1, ER2, ER3: Values indicating performance or characteristics.
### Table IV-4. Continued (2)

<table>
<thead>
<tr>
<th>RUN</th>
<th>LW</th>
<th>LE</th>
<th>LT</th>
<th>V</th>
<th>H</th>
<th>E</th>
<th>PE</th>
<th>E2</th>
<th>E3</th>
</tr>
</thead>
<tbody>
<tr>
<td>96</td>
<td>9.0</td>
<td>36.5</td>
<td>0.112</td>
<td>70</td>
<td>766.39</td>
<td>662.35</td>
<td>496.47</td>
<td>433.99</td>
<td>282.18</td>
</tr>
<tr>
<td>97</td>
<td>18.2</td>
<td>36.5</td>
<td>0.200</td>
<td>70</td>
<td>361.62</td>
<td>271.11</td>
<td>167.93</td>
<td>106.22</td>
<td>63.15</td>
</tr>
<tr>
<td>98</td>
<td>27.7</td>
<td>16.5</td>
<td>0.310</td>
<td>70</td>
<td>314.16</td>
<td>149.25</td>
<td>95.05</td>
<td>51.11</td>
<td>5.10</td>
</tr>
<tr>
<td>99</td>
<td>36.5</td>
<td>36.5</td>
<td>0.350</td>
<td>70</td>
<td>235.63</td>
<td>74.26</td>
<td>20.27</td>
<td>10.11</td>
<td>7.00</td>
</tr>
</tbody>
</table>

**Note:**
- **LW** = Continuous phase superficial velocity, cu. ft./hr. 50 ft. P.T.
- **LE** = Dispersed phase superficial velocity, cu. ft./hr. 50 ft. P.T.
- **LT** = Tracer feed superficial velocity, cu. ft./hr. 50 ft. P.T.
- **V** = Velocity, (2)
- **H** = Column height (nozzle tips to interface) 10-FT. 1 1/8-IN. ISCE FIG. ID FOR 01AG. OF APPARATUS
- **HOLD-UP** = Volumetric percentage of dispersed phase in column
- **PP** = Number of sample concentrations used for estimation of axial eddy diffusivity
- **PE** = Peclet number based on dispersed phase diameter
- **E1** = Water balance apparent loss, per cent
- **E2** = Ketone balance apparent loss, per cent
- **E3** = Tracer balance apparent loss, per cent

**Legend:**
- **LO** = Continuous phase equivalent drop diameter = 0.125 in.
- **COLUMN INSIDE DIAMETER** = 1.5 in.
- **COLUMN HEIGHT** = Dispersed phase tips to interface = 10-FT. 3 1/2-IN. (See Fig. 10 for dia. of apparatus)
- **COLUMN INSIDE DIAMETER** = 1.5 in.
### Table IV-4. Continued (3)

<table>
<thead>
<tr>
<th>NUM</th>
<th>L</th>
<th>L</th>
<th>E</th>
<th>TEMP</th>
<th>REDUCED CONCENTRATION AT SAMPLING POINTS (SEE FIG. 10)</th>
<th>TOL</th>
<th>PP</th>
<th>E</th>
<th>PE</th>
<th>PCR</th>
<th>ECR</th>
<th>ECR</th>
</tr>
</thead>
<tbody>
<tr>
<td>145</td>
<td>9.0</td>
<td>36.6</td>
<td>0.012</td>
<td>60</td>
<td>511,76</td>
<td>819,54</td>
<td>0.95</td>
<td>0.94</td>
<td>0.95</td>
<td>0.94</td>
<td>0.95</td>
<td>0.94</td>
</tr>
<tr>
<td>146</td>
<td>26.3</td>
<td>36.6</td>
<td>0.012</td>
<td>60</td>
<td>511,76</td>
<td>819,54</td>
<td>0.95</td>
<td>0.94</td>
<td>0.95</td>
<td>0.94</td>
<td>0.95</td>
<td>0.94</td>
</tr>
<tr>
<td>147</td>
<td>26.3</td>
<td>36.6</td>
<td>0.012</td>
<td>60</td>
<td>511,76</td>
<td>819,54</td>
<td>0.95</td>
<td>0.94</td>
<td>0.95</td>
<td>0.94</td>
<td>0.95</td>
<td>0.94</td>
</tr>
<tr>
<td>148</td>
<td>36.5</td>
<td>36.5</td>
<td>0.015</td>
<td>60</td>
<td>89,06</td>
<td>16,72</td>
<td>1.67</td>
<td>0.31</td>
<td>0.08</td>
<td>0.06</td>
<td>0.06</td>
<td>0.08</td>
</tr>
<tr>
<td>149</td>
<td>48.4</td>
<td>38,6</td>
<td>0.080</td>
<td>60</td>
<td>21,28</td>
<td>1.96</td>
<td>0.80</td>
<td>0.07</td>
<td>0.05</td>
<td>0.07</td>
<td>0.05</td>
<td>0.07</td>
</tr>
<tr>
<td>150</td>
<td>9.0</td>
<td>36.6</td>
<td>0.012</td>
<td>60</td>
<td>693.70</td>
<td>923.70</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
</tr>
<tr>
<td>151</td>
<td>18.2</td>
<td>36.6</td>
<td>0.012</td>
<td>60</td>
<td>693.70</td>
<td>923.70</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
</tr>
<tr>
<td>152</td>
<td>26.3</td>
<td>36.6</td>
<td>0.012</td>
<td>60</td>
<td>693.70</td>
<td>923.70</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
<td>0.95</td>
</tr>
<tr>
<td>153</td>
<td>36.5</td>
<td>36.5</td>
<td>0.015</td>
<td>60</td>
<td>89,06</td>
<td>16,72</td>
<td>1.67</td>
<td>0.31</td>
<td>0.08</td>
<td>0.06</td>
<td>0.06</td>
<td>0.08</td>
</tr>
<tr>
<td>154</td>
<td>48.4</td>
<td>38,6</td>
<td>0.080</td>
<td>60</td>
<td>21,28</td>
<td>1.96</td>
<td>0.80</td>
<td>0.07</td>
<td>0.05</td>
<td>0.07</td>
<td>0.05</td>
<td>0.07</td>
</tr>
</tbody>
</table>

#### Notes
- AVERAGE INSIDE DIAMETER OF NOZZLE TIPS = 0.053 IN.
- REDUCED CONCENTRATION AT SAMPLING POINTS (SEE FIG. 10)
- TOL = TOLERANCE (IN.
- PP = NUMBER OF SAMPLE CONCENTRATIONS USED FOR ESTIMATION OF AXIAL EDDY DIFFUSIVITY
- E = WATER EDDY DIFFUSIVITY WITH 95 PER CENT CONFIDENCE LIMITS, SC.FT./SEC.
- PE = PEELERT NUMBER BASED ON REDUCED PHASE DROplet DIAmeter
- PCR = KETONE BALANCE APPARENT LOSS PER CENT
- ECR = TRACER BALANCE APPARENT LOSS PER CENT

---

**Column Inside Diameter** • I.* IN.

**Dispersion Phase Equivalent Drop Diameter** • 0.04* IN.

**Lumen Inside Diameter** • 1.5 IN.

**Lumen Height Inside Diameter to Interface** • 10-Ft. 3/8-In. (SEE FIG. 10 FOR DIAG. OF APPARATUS)

**L = Continuous Phase Superficial Velocity, C.U.F.T./IN. (SC.F.T.)

**Lp = Dispersion Phase Superficial Velocity, C.U.F.T./IN. (SC.F.T.)

**L = TRACER FEED SUPERFICIAL VELOCITY, C.U.F.T./IN. (SC.F.T.)

**HOLD-UP = VOLUMETRIC PERCENTAGE OF DISPERSED PHASE IN COLUMN

**P = Number of Sample Concentrations Used for Estimation of Axial Eddy Diffusivity**

**E = Axial Eddy Diffusivity With 95 Per Cent Confidence Limits, SC.FT./SEC.**

**PE = Peclet Number Based on Dispersed Phase Drop Diameter**

**PCR = Ketone Balance Apparent Loss Per Cent**

**ECR = Tracer Balance Apparent Loss Per Cent**
superficial axial eddy diffusivity, and the superficial axial eddy diffusivity for each run with the 3-in. I.D. column are given in Table IV-5. Dispersed phase hold-ups in the 3-in. I.D. column were not measured. Consequently it was not possible to reduce superficial axial eddy diffusivities to true axial eddy diffusivities or to calculate Peclet numbers.

Measurements of the dispersed phase drop sizes were analysed on an IBM-7040 electronic computer. No hand calculations are presented due to their voluminous nature. However, the algebraic equation used to process the data is given below.

Let \( r \) = the vertical dimension of the drop image as measured by means of the microfilm reader, mm.,

\( x \) = the horizontal dimension of the drop image as measured by means of the microfilm reader, mm.

\( f_1 \) = the conversion factor for mm. to in. = 0.03937,

\( f_2 \) = the enlargement factor = a linear dimension of the observed image divided by the same linear dimension of the actual object = 4.309,

and \( d_s \) = the equivalent diameter of the drop = the diameter of a sphere having the same volume as the drop.

Then,

\[
d_s = \frac{(f_1)(\sqrt[3]{r}x^2)}{f_2}
\]

The range of \( d_s \) between 0.00 and 0.25-in. was divided up into
### TABLE IV

**SUPERFICIAL AXIAL EDDY DIFFUSIVITY RESULTS FOR THE 3-IN. I.D. COLUMN**

**AVERAGE INSIDE DIAMETER OF NOZLE TIPS = 0.102 IN.**
**DISPERSED PHASE EQUIVALENT DROP DIAMETER = 0.395 IN.**
**AVERAGE VELOCITY OF DISPERSED PHASE IN NOZLE TIPS = 0.37 FT./SEC.**
**COLUMN INSIDE DIAMETER = 3.0 IN.**
**COLUMN HEIGHT NOZLE TIPS TO INTERFACE = 10 FT. 9 7/8 IN. (SEE FIG. 20 FOR DIAG. OF APPARATUS)**
**Lk = CONTINUOUS PHASE SUPERFICIAL VELOCITY (CP.FT./HR.) 50 FT./HR.**
**Lk = DISPERSED PHASE SUPERFICIAL VELOCITY (CP.FT./HR.) 50 FT./HR.**
**Tr = TRACER FEED SUPERFICIAL VELOCITY (CP.FT./HR.) 50 FT./HR.**
**Pp = NUMBER OF SAMPLE CONCENTRATIONS USED FOR ESTIMATION OF SUPERFICIAL AXIAL EDDY DIFFUSIVITY**
**Se = SUPERFICIAL AXIAL EDDY DIFFUSIVITY WITH 95 PER CENT CONFIDENCE LIMITS, 50 FT./HR.**
**E2 = TRACER BALANCE APPARENT LOSS, PER CENT**

#### TABLE IV.1

<table>
<thead>
<tr>
<th>RUN</th>
<th>LK</th>
<th>LT</th>
<th>TEMP °F</th>
<th>REDUCED CONCENTRATION AT SAMPLING POINTS (SEE FIG. 20)</th>
<th>Pp</th>
<th>SE</th>
<th>E2</th>
<th>ER2</th>
</tr>
</thead>
<tbody>
<tr>
<td>177</td>
<td>18.2</td>
<td>36.5</td>
<td>0.050</td>
<td>930.27</td>
<td>663.08</td>
<td>643.09</td>
<td>810.94</td>
<td>738.76</td>
</tr>
<tr>
<td>181</td>
<td>27.7</td>
<td>36.5</td>
<td>0.100</td>
<td>930.48</td>
<td>877.36</td>
<td>642.38</td>
<td>759.66</td>
<td>693.78</td>
</tr>
<tr>
<td>182</td>
<td>36.5</td>
<td>36.5</td>
<td>0.150</td>
<td>972.18</td>
<td>995.34</td>
<td>561.65</td>
<td>741.07</td>
<td>625.11</td>
</tr>
<tr>
<td>183</td>
<td>48.4</td>
<td>36.5</td>
<td>0.150</td>
<td>923.97</td>
<td>799.06</td>
<td>718.67</td>
<td>602.38</td>
<td>516.67</td>
</tr>
<tr>
<td>184</td>
<td>75.0</td>
<td>36.5</td>
<td>0.200</td>
<td>930.87</td>
<td>717.45</td>
<td>600.97</td>
<td>451.64</td>
<td>851.16</td>
</tr>
<tr>
<td>185</td>
<td>100.0</td>
<td>36.5</td>
<td>0.250</td>
<td>910.55</td>
<td>644.65</td>
<td>424.35</td>
<td>337.24</td>
<td>210.36</td>
</tr>
<tr>
<td>179</td>
<td>220.0</td>
<td>36.5</td>
<td>0.410</td>
<td>978.56</td>
<td>928.69</td>
<td>492.50</td>
<td>396.00</td>
<td>212.65</td>
</tr>
<tr>
<td>186</td>
<td>295.0</td>
<td>36.5</td>
<td>0.410</td>
<td>947.76</td>
<td>764.18</td>
<td>507.39</td>
<td>286.72</td>
<td>16.72</td>
</tr>
<tr>
<td>194</td>
<td>18.2</td>
<td>54.7</td>
<td>0.050</td>
<td>1028.76</td>
<td>974.61</td>
<td>936.07</td>
<td>864.67</td>
<td>818.58</td>
</tr>
<tr>
<td>193</td>
<td>27.7</td>
<td>54.7</td>
<td>0.100</td>
<td>1022.09</td>
<td>973.11</td>
<td>909.10</td>
<td>853.08</td>
<td>784.30</td>
</tr>
<tr>
<td>195</td>
<td>48.4</td>
<td>54.7</td>
<td>0.150</td>
<td>1026.55</td>
<td>945.14</td>
<td>765.26</td>
<td>364.91</td>
<td>234.51</td>
</tr>
<tr>
<td>197</td>
<td>95.0</td>
<td>54.7</td>
<td>0.200</td>
<td>1010.08</td>
<td>963.67</td>
<td>708.59</td>
<td>556.51</td>
<td>346.42</td>
</tr>
<tr>
<td>198</td>
<td>150.0</td>
<td>54.7</td>
<td>0.410</td>
<td>1015.60</td>
<td>923.03</td>
<td>920.26</td>
<td>649.34</td>
<td>72.90</td>
</tr>
<tr>
<td>201</td>
<td>128.0</td>
<td>37.0</td>
<td>0.050</td>
<td>1099.17</td>
<td>876.39</td>
<td>709.69</td>
<td>516.75</td>
<td>400.79</td>
</tr>
<tr>
<td>205</td>
<td>66.6</td>
<td>73.0</td>
<td>0.150</td>
<td>994.89</td>
<td>700.15</td>
<td>97.77</td>
<td>382.27</td>
<td>157.78</td>
</tr>
<tr>
<td>207</td>
<td>18.2</td>
<td>128.0</td>
<td>0.040</td>
<td>996.85</td>
<td>875.33</td>
<td>744.95</td>
<td>592.89</td>
<td>471.32</td>
</tr>
<tr>
<td>208</td>
<td>66.6</td>
<td>73.0</td>
<td>0.150</td>
<td>994.89</td>
<td>700.15</td>
<td>97.77</td>
<td>382.27</td>
<td>157.78</td>
</tr>
<tr>
<td>209</td>
<td>128.0</td>
<td>37.0</td>
<td>0.250</td>
<td>790.25</td>
<td>729.33</td>
<td>83.51</td>
<td>51.57</td>
<td>17.05</td>
</tr>
</tbody>
</table>
0.01-in. increments. The number of drops, and the total drop volume associated with the drops, appearing in each of these increments were calculated. For each increment the number of drops was converted into the percentage of the total number of drops measured. Similarly the sum of the volumes of the drops in each increment was converted into a percentage of the total drop volume associated with all of the drops. These calculations were performed for the first 100 drops examined and were extended to the first 200, 300, 400, and 500 drops examined. A typical set of results appears in Table IV-6. The results for the 500 drops examined for each run in which drop sizes were measured appear in Table IV-7 for the 1½-in. I.D. column and in Table IV-8 for the 3-in. I.D. column. Due to the overlapping of the drops in the photographs it was not possible to measure the drop sizes for runs when \( L_D \) was large. Therefore it was assumed that the drop size distributions were the same at high and low values of \( L_D \).

Table IV-9 shows the superficial flowrates, the measured concentrations, the calculated capacity coefficient, the hold-up, and the error in the mass balance for each run involving mass transfer. Table IV-10 shows the relation between \( \Delta^2 \) and \( E \) for the estimation of \( E \). The values of \( K_D a \) and \( m \) were taken from Table IV-9 and the values of \( J \) were calculated by means of Equation 21. Tables IV-11 and IV-12 show the effect on \( E \) of varying \( K_D a \), \( m \), and the method of calculation of \( J \). Table IV-13 gives the equilibrium concentration of acetic acid in MIBK - saturated water and water - saturated MIBK at 70°F as determined in this work.
**TABLE IV-6. TYPICAL DROP SIZE DISTRIBUTION RESULTS**

WATER SUPERFICIAL VELOCITY = 27.70 CU.FT./HR. SQ.FT.
KETONE SUPERFICIAL VELOCITY = 54.74 CU.FT./HR. SQ.FT.
NOZZLE TIP AVERAGE DIAMETER = 0.103 IN.
COLUMN DIAMETER = 1.5 IN.
CONDITIONS CORRESPOND TO RUN 50

<table>
<thead>
<tr>
<th>RANGE OF EQUIVALENT DROP DIAMETER IN INCHES</th>
<th>PERCENTAGE OF DROPS IN GIVEN SIZE RANGE FOR TOTAL NUMBER OF DROPS SHOWN AT HEAD OF LIST</th>
<th>PERCENTAGE OF TOTAL DROP VOLUME CONTRIBUTED BY DROPS IN GIVEN SIZE RANGE FOR TOTAL NUMBER OF DROPS SHOWN AT HEAD OF LIST</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00-0.01</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.01-0.02</td>
<td>15.0 15.5 15.7 16.0 16.6</td>
<td>0.1 0.1 0.1 0.1 0.1</td>
</tr>
<tr>
<td>0.02-0.03</td>
<td>25.0 24.5 25.0 24.2 23.0</td>
<td>0.4 0.4 0.4 0.4 0.4</td>
</tr>
<tr>
<td>0.03-0.04</td>
<td>6.0 7.0 5.3 6.3 7.4</td>
<td>0.2 0.3 0.2 0.2 0.3</td>
</tr>
<tr>
<td>0.04-0.05</td>
<td>5.0 4.0 3.3 3.5 3.0</td>
<td>0.4 0.4 0.3 0.3 0.3</td>
</tr>
<tr>
<td>0.05-0.06</td>
<td>3.0 3.0 2.7 2.2 1.8</td>
<td>0.5 0.5 0.4 0.3 0.3</td>
</tr>
<tr>
<td>0.06-0.07</td>
<td>0.0 0.5 0.7 0.5 0.4</td>
<td>0.0 0.1 0.2 0.1 0.1</td>
</tr>
<tr>
<td>0.07-0.08</td>
<td>1.0 2.0 1.7 1.2 1.6</td>
<td>0.4 0.8 0.7 0.5 0.6</td>
</tr>
<tr>
<td>0.08-0.09</td>
<td>0.0 2.0 2.3 2.2 2.2</td>
<td>0.0 1.1 1.2 1.2 1.1</td>
</tr>
<tr>
<td>0.09-0.10</td>
<td>2.0 2.0 2.3 2.0 2.0</td>
<td>1.6 1.7 1.9 1.6 1.6</td>
</tr>
<tr>
<td>0.10-0.11</td>
<td>3.0 3.5 4.3 3.7 3.0</td>
<td>3.1 3.9 4.6 4.0 3.1</td>
</tr>
<tr>
<td>0.11-0.12</td>
<td>4.0 2.5 3.0 3.0 3.6</td>
<td>5.4 3.6 4.3 4.2 5.0</td>
</tr>
<tr>
<td>0.12-0.13</td>
<td>3.0 5.0 6.3 7.0 7.2</td>
<td>5.0 9.4 11.6 12.7 12.8</td>
</tr>
<tr>
<td>0.13-0.14</td>
<td>16.0 13.5 12.3 13.2 13.8</td>
<td>33.7 31.0 27.8 29.5 30.0</td>
</tr>
<tr>
<td>0.14-0.15</td>
<td>9.0 9.5 10.0 10.0 9.2</td>
<td>23.2 26.9 27.7 27.3 24.5</td>
</tr>
<tr>
<td>0.15-0.16</td>
<td>6.0 4.0 3.7 3.5 3.6</td>
<td>18.3 13.3 11.8 11.3 11.6</td>
</tr>
<tr>
<td>0.16-0.17</td>
<td>2.0 1.5 1.0 0.7 0.6</td>
<td>7.7 6.4 4.1 3.1 2.4</td>
</tr>
<tr>
<td>0.17-0.18</td>
<td>0.0 0.0 0.0 0.2 0.6</td>
<td>0.0 0.0 0.0 1.3 2.8</td>
</tr>
<tr>
<td>0.18-0.19</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.19-0.20</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.20-0.21</td>
<td>0.0 0.0 0.0 0.3 0.2 0.4</td>
<td>0.0 0.0 2.7 2.0 3.0</td>
</tr>
<tr>
<td>0.21-0.22</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.22-0.23</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.23-0.24</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.24-0.25</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>RANGE OF EQUIVALENT DROP SIZE</td>
<td>PERCENTAGE OF DROPS IN GIVEN SIZE RANGE FOR A TOTAL OF 500 DROPS</td>
<td>CONDITIONS CORRESPOND TO RUN AT HEAD OF LISTED TIP 1.D. COLUMN</td>
</tr>
<tr>
<td>--------------------------------</td>
<td>---------------------------------------------------------------</td>
<td>---------------------------------------------------------------</td>
</tr>
<tr>
<td>TIP 1.D. = 0.126-1.9</td>
<td>TIP 1.D. = 0.103-1.9</td>
<td>TIP 1.D. = 0.086-1.9</td>
</tr>
<tr>
<td>126 128 130 75 86 92</td>
<td>65 63 66 50 55 45</td>
<td>96 98 100 103 113</td>
</tr>
<tr>
<td>0.00-0.01</td>
<td>0.7 0.0 0.0 0.0 0.0 0.0</td>
<td>1.0 0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.01-0.02</td>
<td>22.4 22.1 21.6 21.6 19.0</td>
<td>9.6 18.6 22.0 16.6 9.0 6.4</td>
</tr>
<tr>
<td>0.02-0.03</td>
<td>16.6 17.4 16.2 16.0 17.8 19.4</td>
<td>16.0 20.0 23.4 23.0 16.8 12.2</td>
</tr>
<tr>
<td>0.03-0.04</td>
<td>6.0 5.4 4.8 8.2 5.6 8.2</td>
<td>10.0 6.6 6.2 7.4 11.4 7.8</td>
</tr>
<tr>
<td>0.04-0.05</td>
<td>1.6 2.0 3.0 5.0 3.0 2.0</td>
<td>4.6 4.0 3.4 3.0 4.0 4.4</td>
</tr>
<tr>
<td>0.05-0.06</td>
<td>0.6 3.2 2.4 1.8 3.2 2.2</td>
<td>2.2 2.2 0.6 1.8 1.8 3.0</td>
</tr>
<tr>
<td>0.06-0.07</td>
<td>2.2 2.4 1.2 1.6 2.8 2.2</td>
<td>1.0 1.6 0.8 0.4 1.6 0.8</td>
</tr>
<tr>
<td>0.07-0.08</td>
<td>1.6 2.2 3.0 2.4 1.8 1.0</td>
<td>1.4 2.2 1.0 1.6 1.4 1.4</td>
</tr>
<tr>
<td>0.08-0.09</td>
<td>2.8 2.2 3.0 2.4 2.0 1.6</td>
<td>3.2 1.8 1.2 2.2 1.0 1.4</td>
</tr>
<tr>
<td>0.09-0.10</td>
<td>3.2 2.2 2.0 2.2 2.4 2.0</td>
<td>3.2 1.8 2.4 2.0 2.2 2.4</td>
</tr>
<tr>
<td>0.10-0.11</td>
<td>3.6 3.0 2.8 3.0 2.4 3.0</td>
<td>4.0 2.4 2.4 3.0 2.4 3.8</td>
</tr>
<tr>
<td>0.11-0.12</td>
<td>3.4 2.6 3.0 2.0 1.4 3.4</td>
<td>5.4 5.2 3.6 3.6 4.6 5.6</td>
</tr>
<tr>
<td>0.12-0.13</td>
<td>6.0 4.4 2.4 2.6 3.2 4.0</td>
<td>8.7 10.0 6.0 7.2 6.4 11.2</td>
</tr>
<tr>
<td>0.13-0.14</td>
<td>4.2 3.4 4.6 4.2 3.4 4.0</td>
<td>15.0 11.8 14.0 13.8 16.4 15.2</td>
</tr>
<tr>
<td>0.14-0.15</td>
<td>6.6 6.2 5.8 6.0 7.0</td>
<td>10.8 7.8 8.6 9.2 10.3 13.8</td>
</tr>
<tr>
<td>0.15-0.16</td>
<td>8.2 5.2 6.6 8.6 5.6 5.6</td>
<td>2.2 3.6 4.2 3.6 5.8 6.0</td>
</tr>
<tr>
<td>0.16-0.17</td>
<td>5.6 2.6 5.4 5.8 6.4 7.6</td>
<td>1.6 0.2 0.2 0.6 1.8 3.0</td>
</tr>
<tr>
<td>0.17-0.18</td>
<td>2.4 4.2 2.8 2.8 4.6 2.8</td>
<td>0.2 0.2 0.2 0.6 0.2 0.6</td>
</tr>
<tr>
<td>0.18-0.19</td>
<td>0.6 0.8 1.2 1.2 1.8 3.2</td>
<td>0.2 0.0 0.0 0.0 0.6 0.8</td>
</tr>
<tr>
<td>0.19-0.20</td>
<td>1.2 1.0 0.8 0.6 1.8 0.4</td>
<td>0.2 0.0 0.0 0.0 0.4 0.2</td>
</tr>
<tr>
<td>0.20-0.21</td>
<td>0.4 0.8 0.2 1.2 0.4 0.4</td>
<td>0.2 0.0 0.0 0.4 0.0 0.0</td>
</tr>
<tr>
<td>0.21-0.22</td>
<td>0.0 0.2 0.2 0.2 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.22-0.23</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.23-0.24</td>
<td>0.0 0.2 0.0 0.2 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>0.24-0.25</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0</td>
</tr>
<tr>
<td>RANGE OF EQUIVALENT DROP DIAM. IN INCHES</td>
<td>PERCENTAGE OF TOTAL DROP VOLUME CONTRIBUTED BY DROPS IN GIVEN SIZE RANGE FOR A TOTAL OF 500 DROPS. CONDITIONS CORRESPOND TO RUN AT HEAD OF LIST</td>
<td></td>
</tr>
<tr>
<td>----------------------------------------</td>
<td>-------------------------------------------------------------------------------------------------</td>
<td></td>
</tr>
<tr>
<td></td>
<td>AVERAGE NOZZLE TIP I.D. = 0.126-IN.</td>
<td>AVERAGE NOZZLE TIP I.D. = 0.103-IN.</td>
</tr>
<tr>
<td></td>
<td>126 128 130 75 86 92 65 63 66 50 55 45 98 96 100 103 110 113 148 146 144 151 149</td>
<td></td>
</tr>
<tr>
<td>0.00-0.01</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.01-0.02</td>
<td>0.1 0.1 0.1 0.1 0.1 0.1 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.02-0.03</td>
<td>0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2</td>
<td></td>
</tr>
<tr>
<td>0.03-0.04</td>
<td>0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4 0.4</td>
<td></td>
</tr>
<tr>
<td>0.04-0.05</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.05-0.06</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.06-0.07</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.07-0.08</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.08-0.09</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.09-0.10</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.10-0.11</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.11-0.12</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.12-0.13</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.13-0.14</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.14-0.15</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.15-0.16</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.16-0.17</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.17-0.18</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.18-0.19</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.19-0.20</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.20-0.21</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.21-0.22</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.22-0.23</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.23-0.24</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
<tr>
<td>0.24-0.25</td>
<td>0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0</td>
<td></td>
</tr>
</tbody>
</table>

TABLE IV-7. CONTINUED
**TABLE IV-8. DROP SIZE DISTRIBUTIONS IN THE 3-IN. I.D. COLUMN**

<table>
<thead>
<tr>
<th>RANGE OF EQUIVALENT DROP DIAMETER IN INCHES</th>
<th>PERCENTAGE OF THE DROPS IN GIVEN SIZE RANGE</th>
<th>PERCENTAGE OF THE TOTAL DROP VOLUME CONTRIBUTED BY DROPS IN GIVEN SIZE RANGE</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>177</td>
<td>182</td>
</tr>
<tr>
<td>0.00-0.01</td>
<td>8.4</td>
<td>2.4</td>
</tr>
<tr>
<td>0.01-0.02</td>
<td>29.8</td>
<td>32.8</td>
</tr>
<tr>
<td>0.02-0.03</td>
<td>5.6</td>
<td>12.4</td>
</tr>
<tr>
<td>0.03-0.04</td>
<td>3.6</td>
<td>2.2</td>
</tr>
<tr>
<td>0.04-0.05</td>
<td>1.8</td>
<td>1.0</td>
</tr>
<tr>
<td>0.05-0.06</td>
<td>1.2</td>
<td>1.4</td>
</tr>
<tr>
<td>0.06-0.07</td>
<td>1.4</td>
<td>2.4</td>
</tr>
<tr>
<td>0.07-0.08</td>
<td>1.6</td>
<td>0.8</td>
</tr>
<tr>
<td>0.08-0.09</td>
<td>3.2</td>
<td>2.8</td>
</tr>
<tr>
<td>0.09-0.10</td>
<td>2.8</td>
<td>2.6</td>
</tr>
<tr>
<td>0.10-0.11</td>
<td>2.8</td>
<td>1.4</td>
</tr>
<tr>
<td>0.11-0.12</td>
<td>5.4</td>
<td>4.0</td>
</tr>
<tr>
<td>0.12-0.13</td>
<td>11.2</td>
<td>10.0</td>
</tr>
<tr>
<td>0.13-0.14</td>
<td>12.2</td>
<td>13.2</td>
</tr>
<tr>
<td>0.14-0.15</td>
<td>7.4</td>
<td>7.8</td>
</tr>
<tr>
<td>0.15-0.16</td>
<td>0.8</td>
<td>2.0</td>
</tr>
<tr>
<td>0.16-0.17</td>
<td>0.6</td>
<td>0.6</td>
</tr>
<tr>
<td>0.17-0.18</td>
<td>0.2</td>
<td>0.0</td>
</tr>
<tr>
<td>0.18-0.19</td>
<td>0.0</td>
<td>0.2</td>
</tr>
<tr>
<td>0.19-0.20</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>0.20-0.21</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>0.21-0.22</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>0.22-0.23</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>0.23-0.24</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>0.24-0.25</td>
<td>0.0</td>
<td>0.0</td>
</tr>
</tbody>
</table>
### TABLE IV-9. CONCENTRATION STUDIES WITH MASS TRANSFER IN THE 1\frac{1}{2}-IN. I.D. COLUMN

<table>
<thead>
<tr>
<th>Run</th>
<th>L_C</th>
<th>L_D</th>
<th>Concentration at sampling points: (See Figure 10), $10^3$ lb.-moles/ft.</th>
<th>$c_C^0$</th>
<th>$c_C^0$</th>
<th>$K_B$</th>
<th>Average</th>
<th>h</th>
<th>M.B.</th>
<th>Time to reach steady state</th>
<th>Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td>7</td>
<td>8</td>
<td>9</td>
</tr>
<tr>
<td>J1</td>
<td>36.5</td>
<td>54.7</td>
<td>30.66</td>
<td>34.30</td>
<td>40.37</td>
<td>43.70</td>
<td>47.71</td>
<td>49.47</td>
<td>53.72</td>
<td>55.24</td>
<td>57.21</td>
</tr>
<tr>
<td></td>
<td>9.11</td>
<td></td>
<td>15.2</td>
<td>19.08</td>
<td>23.3</td>
<td>25.95</td>
<td>27.2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>J2</td>
<td>18.2</td>
<td>54.7</td>
<td>9.04</td>
<td>10.05</td>
<td>11.84</td>
<td>13.96</td>
<td>15.33</td>
<td>17.00</td>
<td>19.43</td>
<td>22.31</td>
<td>24.25</td>
</tr>
<tr>
<td></td>
<td>2.91</td>
<td></td>
<td>4.21</td>
<td>5.72</td>
<td>7.50</td>
<td>9.62</td>
<td>11.1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>J3</td>
<td>36.5</td>
<td>91.2</td>
<td>15.48</td>
<td>16.69</td>
<td>18.27</td>
<td>19.73</td>
<td>21.43</td>
<td>23.07</td>
<td>25.50</td>
<td>27.59</td>
<td>30.41</td>
</tr>
<tr>
<td></td>
<td>6.1</td>
<td></td>
<td>7.4</td>
<td>8.76</td>
<td>10.81</td>
<td>12.40</td>
<td>13.55</td>
<td>3.64</td>
<td>31.23</td>
<td></td>
<td></td>
</tr>
<tr>
<td>J4</td>
<td>48.4</td>
<td>91.2</td>
<td>25.25</td>
<td>27.71</td>
<td>30.35</td>
<td>32.78</td>
<td>35.42</td>
<td>37.51</td>
<td>40.06</td>
<td>42.49</td>
<td>45.25</td>
</tr>
<tr>
<td></td>
<td>9.16</td>
<td></td>
<td>12.2</td>
<td>15.0</td>
<td>17.9</td>
<td>20.9</td>
<td>22.7</td>
<td>4.02</td>
<td>37.03</td>
<td></td>
<td></td>
</tr>
<tr>
<td>J5</td>
<td>68.4</td>
<td>127.7</td>
<td>14.65</td>
<td>15.48</td>
<td>16.39</td>
<td>17.36</td>
<td>18.85</td>
<td>20.21</td>
<td>21.73</td>
<td>23.43</td>
<td>25.40</td>
</tr>
<tr>
<td></td>
<td>6.1</td>
<td></td>
<td>7.04</td>
<td>8.1</td>
<td>9.70</td>
<td>11.1</td>
<td>12.2</td>
<td>4.74</td>
<td>31.67</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1. The upper number refers to the continuous phase, the lower number to the dispersed phase.

2. M.B. is the error in the mass balance. This mass balance was calculated according to the following equation.

\[
M.B. = \left[ \frac{L_D(c_C^1 - c_C^0) - L_C(c_D^0 - c_D^1)}{L_C(c_C^0 - c_D^0)} \right] \times 100\%.
\]
TABLE IV-10. THE RELATION BETWEEN E AND $\Delta^2$. (The values of $K_D a$, and $m$ were taken from Table IV-9.

The values of $J$ were calculated from Equation 21.)

The numbers listed under the various values of $E$ tested $= 10^8 \Delta^2$.

| Run | $J$  | $K_D a$ | $m$ | $E$  | 60  | 59  | 58  | 57  | 56  | 54  | 53  | 52  | 51  | 50  | 49  | 48  | 47  |
|-----|------|---------|-----|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| J1  | 0.743| 42.54   | 1.92|      | 2800| 2800| 2700| 2700| 2600| 2600| 2500| 2500| 2400| 2300| 2300| 2200| 2200|
| J2  | 0.119| 56.24   | 2.04|      | 820 | 760 | 690 | 630 | 570 | 510 | 460 | 400 | 350 | 300 | 250 | 210 | 170 |
| J3  | 0.0943| 79.03  | 2.02|      | 1300| 1200| 1200| 1100| 1000| 990 | 930 | 880 | 820 | 770 | 720 | 660 | 610 | 560 |
| J4  | 0.477 | 94.63   | 1.96|      | 920 | 860 | 800 | 740 | 680 | 620 | 560 | 500 | 450 | 400 | 350 | 300 | 260 | 210 |
| J5  | 0.0017| 14.1   | 2.04|      | 58  | 47  | 38  | 31  | 25  | 20  | 18  | 18  | 18  | 23  | 29  | 38  | 50  | 65  |

| Run | $J$  | $K_D a$ | $m$ | $E$  | 46  | 45  | 44  | 43  | 42  | 41  | 40  | 39  | 38  | 37  | 36  | 35  | 34  | 33  |
|-----|------|---------|-----|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| J1  | 0.743| 42.54   | 1.92|      | 2100| 2100| 2000| 2000| 1900| 1800| 1800| 1800| 1700| 1700| 1600| 1600| 1500| 1400|
| J2  | 0.119| 56.24   | 2.04|      | 110 | 89  | 73  | 64  | 63  | 71  | 89  | 120 | 160 | 220 | 290 | 380 | 490 | 620 |
| J3  | 0.0943| 79.03  | 2.02|      | 510 | 460 | 410 | 370 | 320 | 280 | 240 | 200 | 170 | 140 | 110 | 65  | 65  | 51  |
| J4  | 0.477 | 94.63   | 1.96|      | 180 | 140 | 110 | 88  | 68  | 53  | 44  | 41  | 45  | 57  | 78  | 110 | 150 | 200 |
| J5  | 0.0017| 14.1   | 2.04|      | 83  | 100 | 130 | 160 | 190 | 230 | 270 | 320 | 370 | 430 | 500 | 570 | 650 | 740 |

| Run | $J$  | $K_D a$ | $m$ | $E$  | 32  | 31  | 30  | 29  | 28  | 27  | 26  | 25  | 24  | 23  | 22  | 21  | 20  | 19  |
|-----|------|---------|-----|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| J1  | 0.743| 42.54   | 1.92|      | 1300| 1300| 1200| 1100| 1100| 1000| 970 | 920 | 870 | 840 | 790 | 750 | 720 | 690 |
| J2  | 0.119| 56.24   | 2.04|      | 770 | 960 | 1200| 1400| 1700| 2000| 2400| 2900| 3400| 4000| 4600| 5400| 6300| 7300|
| J3  | 0.0943| 79.03  | 2.02|      | 42  | 39  | 43  | 55  | 76  | 110 | 150 | 200 | 270 | 350 | 440 | 560 | 700 | 850 |
| J4  | 0.477 | 94.63   | 1.96|      | 260 | 330 | 420 | 530 | 650 | 790 | 950 | 1100| 1300| 1600| 1800| 1800| 2100| 2400|
| J5  | 0.0017| 14.1   | 2.04|      | 840 | 940 | 1100| 1200| 1300| 1500| 1600| 1800| 2000| 2200| 2500| 2700| 3000| 3300|

| Run | $J$  | $K_D a$ | $m$ | $E$  | 18  | 17  | 16  | 15  | 14  | 13  | 12  | 11  | 10  | 9   | 8   | 7   | 6   | 5   |
|-----|------|---------|-----|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| J1  | 0.743| 42.54   | 1.92|      | 670 | 660 | 660 | 660 | 680 | 720 | 770 | 840 | 920 | 1000| 1200| 1300| 1500| 1500|
| J2  | 0.119| 56.24   | 2.04|      | 850 | 980 | 1100| 1300| 1500| 1700| 2000| 2300| 2700| 3100| 3600| 4200| 4900| 5700|
| J3  | 0.0943| 79.03  | 2.02|      | 1000| 1300| 1500| 1800| 2100| 2500| 2900| 3300| 3800| 4400| 5100| 5800| 6600| 5700|
| J4  | 0.477 | 94.63   | 1.96|      | 3200| 3600| 4100| 4700| 5200| 5900| 6600| 7400| 8200| 9100| 10000| 12000| 12000| 15000|
| J5  | 0.0017| 14.1   | 2.04|      | 3600| 4000| 4300| 4800| 5200| 5700| 6200| 6800| 7400| 8000| 8000| 8000| 8000| 8000|
TABLE IV-11. CALCULATED VALUES OF E FOR VARIOUS VALUES OF K_d a AND J. (The values of m were taken from Table IV-9)

<table>
<thead>
<tr>
<th>Run</th>
<th>J</th>
<th>m</th>
<th>K_d</th>
<th>25</th>
<th>30</th>
<th>35</th>
<th>40</th>
<th>42.54</th>
<th>45</th>
<th>50</th>
<th>55</th>
<th>60</th>
<th>65</th>
</tr>
</thead>
<tbody>
<tr>
<td>J1</td>
<td>0.743 a</td>
<td>1.92</td>
<td>E</td>
<td>60</td>
<td>5</td>
<td>5</td>
<td>12</td>
<td>16</td>
<td>20</td>
<td>25</td>
<td>29</td>
<td>33</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>0.782 b</td>
<td>1.92</td>
<td>E</td>
<td>60</td>
<td>5</td>
<td>12</td>
<td>22</td>
<td>26</td>
<td>29</td>
<td>34</td>
<td>38</td>
<td>41</td>
<td>43</td>
</tr>
<tr>
<td></td>
<td>0.704 c</td>
<td>1.92</td>
<td>E</td>
<td>60</td>
<td>60</td>
<td>5</td>
<td>6</td>
<td>6</td>
<td>10</td>
<td>16</td>
<td>21</td>
<td>24</td>
<td>27</td>
</tr>
<tr>
<td>J2</td>
<td>0.119 a</td>
<td>2.04</td>
<td>E</td>
<td>30</td>
<td>34</td>
<td>37</td>
<td>40</td>
<td>42</td>
<td>42</td>
<td>44</td>
<td>45</td>
<td>47</td>
<td>48</td>
</tr>
<tr>
<td></td>
<td>0.138 b</td>
<td>2.04</td>
<td>E</td>
<td>34</td>
<td>38</td>
<td>41</td>
<td>44</td>
<td>46</td>
<td>47</td>
<td>48</td>
<td>50</td>
<td>51</td>
<td>53</td>
</tr>
<tr>
<td></td>
<td>0.099 c</td>
<td>2.04</td>
<td>E</td>
<td>26</td>
<td>30</td>
<td>33</td>
<td>35</td>
<td>38</td>
<td>38</td>
<td>39</td>
<td>41</td>
<td>42</td>
<td>44</td>
</tr>
<tr>
<td>J3</td>
<td>0.094 a</td>
<td>2.02</td>
<td>E</td>
<td>22</td>
<td>26</td>
<td>29</td>
<td>31</td>
<td>32</td>
<td>34</td>
<td>37</td>
<td>39</td>
<td>41</td>
<td>42</td>
</tr>
<tr>
<td></td>
<td>0.099 b</td>
<td>2.02</td>
<td>E</td>
<td>23</td>
<td>27</td>
<td>30</td>
<td>32</td>
<td>33</td>
<td>35</td>
<td>38</td>
<td>40</td>
<td>42</td>
<td>44</td>
</tr>
<tr>
<td></td>
<td>0.089 c</td>
<td>2.02</td>
<td>E</td>
<td>21</td>
<td>24</td>
<td>28</td>
<td>30</td>
<td>30</td>
<td>33</td>
<td>35</td>
<td>37</td>
<td>39</td>
<td>41</td>
</tr>
<tr>
<td>J4</td>
<td>0.477 a</td>
<td>1.96</td>
<td>E</td>
<td>21</td>
<td>26</td>
<td>30</td>
<td>33</td>
<td>36</td>
<td>39</td>
<td>39</td>
<td>42</td>
<td>44</td>
<td>46</td>
</tr>
<tr>
<td></td>
<td>0.527 b</td>
<td>1.96</td>
<td>E</td>
<td>32</td>
<td>37</td>
<td>41</td>
<td>44</td>
<td>48</td>
<td>50</td>
<td>50</td>
<td>53</td>
<td>55</td>
<td>59</td>
</tr>
<tr>
<td></td>
<td>0.427 c</td>
<td>1.96</td>
<td>E</td>
<td>10</td>
<td>14</td>
<td>19</td>
<td>22</td>
<td>25</td>
<td>28</td>
<td>28</td>
<td>31</td>
<td>33</td>
<td>35</td>
</tr>
<tr>
<td>J5</td>
<td>0.0017 a</td>
<td>2.04</td>
<td>E</td>
<td>44</td>
<td>47</td>
<td>49</td>
<td>51</td>
<td>53</td>
<td>53</td>
<td>53</td>
<td>55</td>
<td>56</td>
<td>58</td>
</tr>
<tr>
<td></td>
<td>-0.0560 b</td>
<td>2.04</td>
<td>E</td>
<td>26</td>
<td>28</td>
<td>30</td>
<td>32</td>
<td>34</td>
<td>34</td>
<td>35</td>
<td>37</td>
<td>38</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td>0.0594 c</td>
<td>2.04</td>
<td>E</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
</tr>
</tbody>
</table>

a  The flux of solute down the column, \( J = \frac{1}{2} \left( L_C^i - L_D^p \right) + \left( L_C^o - L_D^p \right) \)

b  The flux of solute down the column, \( J = \left( L_C^i - L_D^p \right) \)

c  The flux of solute down the column, \( J = \left( L_C^o - L_D^p \right) \)
TABLE IV-12. CALCULATED VALUES OF $E$ FOR VARIOUS VALUES OF $m$. (The values of $J$ and $K_{D,a}$ were taken from Table IV-9.)

<table>
<thead>
<tr>
<th>Run</th>
<th>$J$</th>
<th>$K_{D,a}$</th>
<th>$m$</th>
<th>1.82</th>
<th>1.84</th>
<th>1.86</th>
<th>1.88</th>
<th>1.90</th>
<th>1.92</th>
<th>1.94</th>
<th>1.96</th>
<th>1.98</th>
<th>2.00</th>
<th>2.02</th>
</tr>
</thead>
<tbody>
<tr>
<td>J1</td>
<td>0.743</td>
<td>42.54</td>
<td>E</td>
<td>36</td>
<td>32</td>
<td>28</td>
<td>24</td>
<td>20</td>
<td>16</td>
<td>12</td>
<td>9</td>
<td>6</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>J2</td>
<td>0.119</td>
<td>56.24</td>
<td>E</td>
<td>48</td>
<td>46</td>
<td>45</td>
<td>44</td>
<td>43</td>
<td>42</td>
<td>41</td>
<td>40</td>
<td>39</td>
<td></td>
<td></td>
</tr>
<tr>
<td>J3</td>
<td>0.0943</td>
<td>79.03</td>
<td>E</td>
<td>44</td>
<td>41</td>
<td>39</td>
<td>36</td>
<td>34</td>
<td>31</td>
<td>29</td>
<td>26</td>
<td>24</td>
<td>22</td>
<td>19</td>
</tr>
<tr>
<td>J4</td>
<td>0.477</td>
<td>94.63</td>
<td>E</td>
<td>59</td>
<td>55</td>
<td>51</td>
<td>47</td>
<td>43</td>
<td>39</td>
<td>35</td>
<td>32</td>
<td>28</td>
<td>24</td>
<td>21</td>
</tr>
<tr>
<td>J5</td>
<td>0.0017</td>
<td>141.1</td>
<td>E</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>58</td>
<td>53</td>
<td>49</td>
<td>45</td>
<td>41</td>
<td>37</td>
<td>34</td>
<td></td>
</tr>
</tbody>
</table>
### TABLE IV-13. EQUILIBRIUM DATA FOR ACETIC ACID DISTRIBUTED BETWEEN MIBK-SATURATED WATER AND WATER-SATURATED MIBK AT 70°F. (MEASURED IN THIS WORK.)

<table>
<thead>
<tr>
<th>Concentration of acetic acid, lb.-moles/ft(^3)</th>
<th>Distribution coefficient, m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water phase</td>
<td>MIBK phase</td>
</tr>
<tr>
<td>0.001010</td>
<td>0.0005545</td>
</tr>
<tr>
<td>0.002218</td>
<td>0.001099</td>
</tr>
<tr>
<td>0.004089</td>
<td>0.001990</td>
</tr>
<tr>
<td>0.006307</td>
<td>0.003079</td>
</tr>
<tr>
<td>0.006743</td>
<td>0.003287</td>
</tr>
<tr>
<td>0.01127</td>
<td>0.005485</td>
</tr>
<tr>
<td>0.01385</td>
<td>0.006772</td>
</tr>
<tr>
<td>0.01763</td>
<td>0.008619</td>
</tr>
<tr>
<td>0.02564</td>
<td>0.01262</td>
</tr>
<tr>
<td>0.02109</td>
<td>0.01040</td>
</tr>
<tr>
<td>0.02802</td>
<td>0.01412</td>
</tr>
<tr>
<td>0.03728</td>
<td>0.01901</td>
</tr>
<tr>
<td>0.04663</td>
<td>0.02446</td>
</tr>
<tr>
<td>0.05794</td>
<td>0.03111</td>
</tr>
<tr>
<td>0.06683</td>
<td>0.03614</td>
</tr>
<tr>
<td>0.06802</td>
<td>0.03738</td>
</tr>
<tr>
<td>0.07376</td>
<td>0.04059</td>
</tr>
<tr>
<td>0.07891</td>
<td>0.04368</td>
</tr>
<tr>
<td>0.08381</td>
<td>0.04733</td>
</tr>
<tr>
<td>0.08807</td>
<td>0.05050</td>
</tr>
<tr>
<td>0.09625</td>
<td>0.05564</td>
</tr>
<tr>
<td>0.1008</td>
<td>0.05881</td>
</tr>
<tr>
<td>0.1051</td>
<td>0.06188</td>
</tr>
<tr>
<td>0.1104</td>
<td>0.06569</td>
</tr>
<tr>
<td>0.1150</td>
<td>0.06901</td>
</tr>
</tbody>
</table>
APPENDIX V

DETAILS OF THE APPARATUS

Figures V-1 to V-23 inclusive show the details of various portions of the 1\(\frac{1}{2}\)-in. I.D. and 3-in. I.D. spray columns and their accessories. All stainless steel parts were made from Type 304 stainless steel.

The piston sample collection flask shown in Figure V-1 was used for dispersed phase hold-ups greater than 12%. The collection flask for hold-ups less than 12% is described elsewhere (37). Figure V-2 is a diagram of the hypodermic needle sampling system and Figure V-3 gives details of the sampling valve. The tracer injection arrangement is given in Figure V-4 and details of the tracer distributor are shown in Figure V-5.

Figures V-6, V-7, and V-8 show the 0.126-in. I.D., 0.066-in. I.D., and the 0.053-in. I.D. nozzle tips together with the respective nozzle tip support plates and nozzle tip caps or plugs for use in the 1\(\frac{1}{2}\)-in. I.D. spray column. The nozzle tip support plates were press fitted into the nozzle designed by Choudhury (35). The flow straightener in the nozzle designed by Choudhury was not used in the present work. The 0.103-in. I.D. nozzle tips, the corresponding nozzle tip support plate and nozzle tip caps are described elsewhere (30, 35). The inside diameter of
each nozzle tip in each set of nozzles was measured by means of a travelling microscope. Two such diameters at right angles to each other were measured for each nozzle tip. The arithmetic average of the nozzle tip inside diameter was calculated for each set of tips.

The Perspex box used to reduce optical distortion in the 1\(\frac{1}{2}\)-in. I.D. column is shown in Figure V-9. An O-ring was press fitted in each of the top and bottom of the box to prevent the loss of water from between the box and the column. The cardboard light shield, which fitted over the Perspex box, is shown in Figure V-10. The inside surfaces of the light shield were painted with black ink to reduce unwanted light reflections. The eight sheets of tracing paper in the light shield served as a diffusing screen for the light.

Figure V-11 shows the nozzle and nozzle tips assembled in the lower portion of the 3-in. I.D. column. Details of the various parts of the assembly are given in Figures V-12 to V-17 inclusive. The diametrically opposite holes labelled B in Figure V-17 were outlets for the aqueous phase. The third hole labelled B was for a thermometer well. The nozzle support fitted into E. C was for a drain valve.

Figure V-18 shows the Elgin head for the 3-in. I.D. column and Figures V-19, V-20, and V-21 give details of various component parts. The aqueous phase inlet pipes, shown as A in Figure V-18,
were screwed into the holes marked C in Figure V-19. The two holes labelled B in Figure V-19 were the dispersed phase exits and E in the same Figure accommodated a thermometer well. F in Figure V-20 accommodated another thermometer well. The six holes, C, in Figure V-20 were for bolts which held a standard flange. This flange compressed the polyethylene packing shown in Figure V-21. E in Figure V-20 was for a drain valve.

Figure V-22 shows the Perspex box used to reduce optical distortion in the 3-in. I.D. column. O-rings were press fitted into the top and the bottom of the box to prevent the escape of water from between the box and the column. Drop photographs were taken through a 6-in. long section of 3-in. I.D. glass pipe cut from a longer piece. This 6-in. piece used for photography therefore had unflanged ends. It was held in place, concentric with the rest of the column, by means of two flanges, one of which is shown in Figure V-23. Four 9-in. long aluminum tie-rods held the flanges in position. Each end of these was threaded and carried a nut for tightening purposes.
FIGURE V-1. PISTON SAMPLE COLLECTION FLASK FOR LARGE HOLD-UPS

214-35 FULL LENGTH PYREX GROUND GLASS JOINT

0.2 ml. DIVISIONS
(APPROX. 2 mls./cm.)

25 mm. DIA.

0.2 ml. DIVISIONS
(APPROX. 2 mls./cm.)

HOOKS FOR SPRINGS

40 mm. DIA.

TEFLON STOPCOCK
(WITH LARGE DIA. HOLE FOR FAST DELIVERY)
3" LONG  22 GAUGE HYPODERMIC NEEDLE

\[ \frac{1}{16} " \text{ THICK, } \frac{1}{2} " \text{ I.D.,} \]
\[ 2\frac{5}{32} " \text{ O.D. POLYETHYLENE} \]
GASKET WITH 0.025" DIA. HOLE FOR NEEDLE

FIGURE V-2. HYPODERMIC NEEDLE INSTALLED FOR SAMPLING
SECTION ON A-A

BODY MATERIAL: POLYETHYLENE

STEM MATERIAL: STAINLESS STEEL

FIGURE V-3. SAMPLING VALVE FOR HYPODERMIC NEEDLE
FIGURE V-4. TRACER INJECTION SYSTEM
SECTION ON A-A

1 HOLE 0.043" D.

4 HOLES 0.063" D.

0.157" D.

SECTION ON B-B

FIGURE V-5. TRACER DISTRIBUTOR
STAINLESS STEEL NOZZLE TIP SUPPORT

STAINLESS STEEL NOZZLE TIP

TEFLON NOZZLE CAP

FIGURE V-6. 0.126-IN. I.D. NOZZLE TIPS, NOZZLE TIP SUPPORT PLATE, AND NOZZLE TIP CAPS
FIGURE V-7. 0.086-IN. I.D. NOZZLE TIPS, NOZZLE TIP SUPPORT PLATE, AND NOZZLE TIP PLUGS
73 HOLES 3/32" D. ON 1/8"
TRIANGULAR PITCH
STAINLESS STEEL NOZZLE TIP SUPPORT

STAINLESS STEEL NOZZLE TIP

45° CHAMFER

SECTION ON A-A

0.052" D.

TEFLON NOZZLE TIP PLUG

FIGURE V-8. 0.053-IN. I.D. NOZZLE TIPS, NOZZLE TIP SUPPORT PLATE, AND NOZZLE TIP PLUGS
SECTION ON A-A

SECTION ON B-B

MATERIAL: TRANSPARENT 3/8" PERSPEX UNLESS OTHERWISE SPECIFIED

FIGURE V-9. PERSPEX BOX FOR THE 1 1/2-IN. I.D. COLUMN PHOTOGRAPHS
PHOTOGRAPHIC SECTION

MATERIAL: $\frac{1}{32}$ PULPBOARD

FIGURE V-10. LIGHT SHIELD FOR THE 1$\frac{3}{8}$-IN. I.D. COLUMN PHOTOGRAPHS
FIGURE V-11. LOWER END OF THE 3-IN. I.D. COLUMN
FIGURE V-12. SPECIAL PYREX REDUCER FOR THE 3-IN. I.D. COLUMN
FIGURE V-13. NOZZLE SHELL FOR THE 3-IN. I.D. COLUMN

MATERIAL: STAINLESS STEEL
FIGURE V-14. NOZZLE TIPS, NOZZLE TIP SUPPORT PLATE, AND NOZZLE TIP PLUGS FOR THE 3-IN. I.D. COLUMN
MATERIAL: STAINLESS STEEL

FIGURE V-15. FLOW STRAIGHTENER FOR THE 3-IN. I.D. COLUMN NOZZLE
STAINLESS STEEL NOZZLE SECURING PLATE

STAINLESS STEEL RETAINING NUT

FIGURE V-16. NOZZLE SECURING PLATE AND RETAINING NUT FOR THE 3-IN. I.D. COLUMN
8 HOLES WITH CLEARANCE FOR 3/8" BOLTS ON 9 1/2" P.C.D.

4 1/2" P.C.D.
B, B, B, 3 HOLES DRILL AND TAP 1/4" N.P.T.
C, 1 HOLE DRILL AND TAP 1/8" N.P.T.
E, 1 HOLE 3/4" D.

MACHINE FLAT
SECTION ON A-A
MATERIAL: STAINLESS STEEL

FIGURE V-17. END PLATE FOR THE BOTTOM OF THE 3-IN. I.D. COLUMN
A ¼" STAINLESS STEEL PIPES (AQUEOUS PHASE INLET).

B UPPER END PLATE OF ELGIN HEAD.

C TEFELON COVERED GASKETS.

D 9" LONG, 9" I.D. Q.V.F. GLASS SECTION.

E LOWER END PLATE OF ELGIN HEAD.

F LOWER END PLATE PACKING.

G 10¾" LONG, 3" I.D. PYREX "DOUBLE TOUGH" PIPE WITH FLAT UNFLARED UPPER END.

FIGURE V-18. ELGIN HEAD FOR THE 3-IN. I.D. COLUMN
B, B, 2 HOLES DRILL AND TAP 1/4" N.P.T.
C, C, 2 HOLES DRILL AND TAP 1/4" N.P.T.
FROM BOTH SIDES
E, 1 HOLE DRILL AND TAP 1/2" N.P.T.

MACHINE FLAT

SECTION ON A-A

MATERIAL: STAINLESS STEEL

FIGURE V-19. UPPER END PLATE FOR THE ELGIN HEAD OF THE 3-IN. I.D. COLUMN
B  8 HOLES $\frac{7}{16}$" D. ON $12\frac{3}{16}$" P.C.D.
C  6 HOLES DRILL AND TAP $\frac{1}{2}$" DEEP FOR $\frac{5}{16}$" BOLTS ON 5$\frac{1}{4}$" P.C.D.
E  1 HOLE DRILL AND TAP $\frac{1}{4}$" N.P.T. ON 7$\frac{1}{2}$" P.C.D.
F  1 HOLE DRILL AND TAP $\frac{1}{8}$" N.P.T. ON 7$\frac{1}{2}$" P.C.D.

MACHINE FLAT

TAP E AND F FROM THIS SIDE

SECTION ON A-A

MATERIAL: STAINLESS STEEL

FIGURE V-20. LOWER END PLATE FOR THE ELGIN HEAD OF THE 3-IN. I.D. COLUMN
FIGURE V-21. LOWER END PLATE PACKING FOR THE ELGIN HEAD OF THE 3-IN. I.D. COLUMN

SECTION ON A-A
MATERIAL: POLYETHYLENE
MATERIAL: TRANSPARENT 5/16" PERSPEX

FIGURE V-22. PERSPEX BOX FOR THE 3-IN. I.D. COLUMN PHOTOGRAPHS
SECTION ON A-A
MATERIAL: ALUMINUM

FIGURE V-23. FLANGE FOR THE PHOTOGRAPHIC SECTION OF THE 3-IN. I.D. COLUMN
APPENDIX VI

DIMENSIONS OF THE GLASS PORTIONS IN THE COLUMN TEST SECTIONS AND MEASUREMENT OF PURGE TIMES.

a) Dimensions of the glass portions in the column test sections.

The length and inside diameter of each piece of Pyrex "Double Tough" glass pipe in the test sections of the 1\(\frac{1}{2}\)-in. I.D. and 3-in. I.D. columns were measured by means of vernier calipers. The I.D. was taken to be the average of six readings equally spaced along the length of the glass piece. These measurements are given in Table VI-1.

<table>
<thead>
<tr>
<th>Section of column between positions (See diags. 10 and 20)</th>
<th>Length (1(\frac{1}{2})-in. I.D. column) in.</th>
<th>I.D. (1(\frac{1}{2})-in. I.D. column) in.</th>
<th>Length (3-in. I.D. column) in.</th>
<th>I.D. (3-in. I.D. column) in.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tracer distributor and 1</td>
<td>6.044</td>
<td>1.500</td>
<td>5.983</td>
<td>2.992</td>
</tr>
<tr>
<td>1 and 2</td>
<td>5.967</td>
<td>1.500</td>
<td>6.022</td>
<td>3.026</td>
</tr>
<tr>
<td>2 and 3</td>
<td>5.969</td>
<td>1.497</td>
<td>6.020</td>
<td>2.985</td>
</tr>
<tr>
<td>3 and 4</td>
<td>5.978</td>
<td>1.503</td>
<td>5.975</td>
<td>2.989</td>
</tr>
<tr>
<td>4 and 5</td>
<td>6.024</td>
<td>1.492</td>
<td>6.020</td>
<td>3.008</td>
</tr>
<tr>
<td>5 and 6</td>
<td>5.793</td>
<td>1.497</td>
<td>6.014</td>
<td>3.009</td>
</tr>
<tr>
<td>6 and 7</td>
<td>5.975</td>
<td>1.504</td>
<td>6.008</td>
<td>2.992</td>
</tr>
<tr>
<td>7 and 8</td>
<td>6.050</td>
<td>1.595</td>
<td>6.044</td>
<td>3.020</td>
</tr>
<tr>
<td>8 and 9</td>
<td>6.049</td>
<td>1.508</td>
<td>6.018</td>
<td>2.998</td>
</tr>
<tr>
<td>9 and 10</td>
<td>6.032</td>
<td>1.500</td>
<td>5.996</td>
<td>2.995</td>
</tr>
</tbody>
</table>
The piston sampler block replaced a glass section. This piece of glass was used as the photographic test section. It was cut from a longer piece of Pyrex "Double Tough" pipe. The thickness of each polyethylene gasket was measured and was found to be 1/16-in. This measurement did not change when the gasket was compressed between two pieces of glass.

b) Measurement of purge times.

i) Hook and bell-probes sampling lines.

The column was brought to steady state operating conditions with the transfer of acetic acid from the continuous phase to the dispersed phase as described under Experimental Procedure. With the probes a few inches below the interface the flow of samples into the probes and along the sampling lines was started and maintained for about 2-hr. Without altering the sampling rates the probes were lowered to about 1-ft. above the nozzle tips. Samples from each probe were collected at time intervals of about 1-min. until 30-min. after moving the probes. The bell-probe samples were shaken vigorously many times to bring the two phases to equilibrium. The hook-probe samples and the ketone phase of the bell-probe samples were analysed for acetic acid as described earlier. For each probe a plot was made of the concentration of acetic acid in each sample versus the time after moving the probes. The purge time was taken to be that time when there was no change in concentration with increasing time. Purge times were determined for various sampling rates
between 4-ml./min. and 20-ml./min. It was found that a conservative estimate of the minimum purge time for each of the sampling lines was given by the following equation.

\[
\text{Purge time (min.)} = \frac{120}{\text{sampling rate (ml./min.)}}
\]

ii) Hypodermic needles.

The inside diameter of a 22-gauge hypodermic needle is so small that the purge time for such a sampling device is very small. A conservative estimate of the minimum purge time was estimated in the following manner. A hypodermic needle sampler was inserted through one of the 1\(\frac{1}{2}\)-in. I.D. polyethylene gaskets described earlier. This gasket was sandwiched between one end of a 3-ft. length of 1\(\frac{1}{2}\)-in. I.D. Pyrex pipe and a 1\(\frac{1}{2}\)-in. dia. disk of 1/16-in. thick polyethylene. The gasket and disk were clamped to the end of the pipe by means of an aluminum end plate and flange. The pipe was filled with water and a sampling rate of 3/4-ml./min. through the needle was established. The water was poured out of the pipe and then the pipe was filled with an aqueous solution of potassium permanganate. The flow of liquid through the hypodermic needle recommenced at 3/4-ml./min. The colour of the potassium permanganate showed up in the sample issuing from the needle in less than 45-sec. The change in colour was so rapid that the time between that when the colour first appeared and that when it reached full strength was considered to be negligible. A purge time of 1\(\frac{1}{2}\)-min. was considered to be conservative for sampling rates of at least 3/4-ml./min.