Mass transfer in a fixed baffle liquid-liquid extractor

by

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Summary

The main object of the present work is to study the characteristics of a Fixed Baffle Contactor. The liquid systems used were carbon tetrachloride - acetic acid - water and MIBK - acetic acid - water. The equilibrium relationships for each of these systems were established.

The investigation of the mass transfer properties of the contactor was centred on the variation of the baffle sizes and arrangements within the column, the physical properties and flowrates of the continuous and dispersed phases, and the initial solute concentration in the phases. The performance of the contactor was given in terms of mass transfer rates, interfacial areas and end effects.

The longitudinal dispersion(axial mixing) properties, as summarised by the Peclet number and the Variance, were studied for the continuous phase; the relationship between the mass transfer operation and the axial mixing was also investigated.

The Surface mean diameter of the droplet swarms was determined photographically under various flow conditions.

This Surface mean diameter was used in conjunction with the hold-up of the system to evaluate the interfacial area 'a' of the dispersion and subsequently the value of $(K)_{c}$ was also found. This made possible a comparison between experimental and theoretical $(K)_{c}$ values.

The interfacial turbulence, such as eruption of the droplets, was studied, and the relationship between the final concentration, the interfacial tension and the time was presented graphically.
Acknowledgements

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Chapter 1
General Introduction
1.1 Factors which influence the design of extractors

The separation of the constituents of a homogeneous liquid mixture is a problem frequently encountered in the chemical processing industry, and liquid-liquid extraction—sometimes called solvent extraction—has emerged as a very important method for separating liquid mixtures. In such a process, intimate contact of feed mixture with the solvent and the separation of the resultant mixture into two layers are essential, and equipment used for extraction should be capable of meeting these requirements. The solvents used should be immiscible or partially miscible with one of the components of the mixture in order to facilitate the separation of the liquid phases.

The equipment used for such separation may be classified, according to the construction and the operational characteristics, into two major groups, stagewise and differential contactors. Stagewise contactors can be either batch or continuous and an example would be one or more mixer-settler systems. The flow of the phases could be either co-current or counter-current although the latter is more usual. The differential contactors are of the types of packed, pulsed, spray columns and others. (Table 1.1)

Alternatively, liquid-liquid extraction equipment can be classified into vertical column type and horizontal extractors. The former are further divided, according to the mode of agitation provided for the interdispersion of liquids, as unagitated, rotor-agitated and pulsed-agitated columns.

The selection of a particular extractor is generally governed by economics and is based largely on experience. The physical and chemical properties of the system have
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of course to be known or need to be established and any special features such as the presence of solids and emulsion formation must be taken into account. The location of the extractor in terms of floor area or headroom available is also an important factor. This is why vertical columns are preferred for multistage extraction purposes as they occupy a smaller floor space. On the other hand, the cost of installation, maintenance and operation of the extractor must also be evaluated.

Before a final decision can be made, the process parameters such as phase flow ratio, solvent residence time and axial mixing, dispersed phase hold up and certain physical properties must be considered. This is because some of the parameters such as hold up, drop size and mass transfer coefficient are interrelated in such a fashion that the optimum condition must be carefully investigated if an acceptable contactor is to be found.

An acceptable contactor is one which exhibits high efficiency and a high capacity. Further, the construction has to be simple with no scaling-up problems.

Considerable effort has been made over the years to improve the design of contactors to give high performance for a wide variety of systems. Some of these contactors are mentioned here such as Rotating Disc, Pulsed, Packed etc. One of the governing factors is power input and even the existence of moving parts of the equipment. It is rather paradoxical that the fixed baffle column is in constant use in industry and yet has received little attention significantly. A considerable amount can be learnt from its mechanism of operation and the work is not only needed
but may yield a better understanding of the operation of contactor. In particular it is intended to compare its performance with that of an Oscillating Baffle Column designed and built in the present Chemical Engineering Department.

1.2 Equilibrium relationship in equilibrium system

In a liquid-liquid extraction process the solute is transferred from one liquid phase to another (solvent) phase. Accurate knowledge of phase equilibrium relationship of the components involved is therefore of prime importance in handling any extraction problems. From the equilibrium relationship, the real driving forces can be evaluated, which in turn determine the overall mass transfer rate.

1.3 Mass transfer

The mass transfer rate between two fluid phases will depend on the physical properties of the two phases, the concentration difference, the interfacial area and the degree of turbulence. Clearly, the physical properties are related to the mass transfer coefficients, while the concentration difference would give the effective driving forces.

The main feature of an extraction process is the transference of the solute from one liquid solvent to another across a phase boundary, i.e. the interface. This transference of material can take place under various conditions: it may be effected by a pure molecular transport, by eddy transport, or a combination of both.

For a fixed baffle contactor, there are four possible regions for mass transfer, viz (i) the region where the drops are formed, (ii) the region where the drops are broken,
(iii), the region where they travel through and (iv), the pool on the plate where re-coalescence occurs.

1.3.1. Mass transfer coefficient

From Whitman's classical two-film theory (or two-resistance concept) the mass transfer coefficient for the interphase mass transport is defined by the following equations:

\[ N_A = k_x (x - x_i) \]

and

\[ N_A = k_y (y_i - y) \]

where \( N_A \) is the mass flux per unit area, \( k_x, k_y \) are film coefficients for mass transfer, \( (x - x_i), (y_i - y) \) are the driving forces, \( x_i, y_i \) are the solute concentrations (mole fraction) in equilibrium across the two phases 1 and 2 at the interface, and

\[ y_i = m(x_i^n) \] (for non-linear equilibrium relationship)

or \( y_i = m(x_i) \) (for linear equilibrium relationship)

where \( n \) is a constant and \( m \) is the equilibrium constant.

Because of the difficulty in the experimental determination of the values of \( x_i \) and \( y_i \), modified equations are generally used which avoid the use of the interfacial equilibrium conditions.
\[ N_A = K_{ox}(x - x^*) = K_{oy}(y^* - y) \]

where \( K_{ox} \) and \( K_{oy} \) are overall mass transfer coefficients,
\( x^* \) represents the composition of the solute in phase 1 in equilibrium with the solute concentration in the bulk of phase 2, and
\( y^* \) represents the composition of the solute in phase 2 in equilibrium with the solute concentration in the bulk of phase 1.

In addition, the coefficients \( K_{ox} \) and \( K_{oy} \) are related to individual coefficients \( k_x \) and \( k_y \) through the equations:

\[ \frac{1}{K_{ox}} = \frac{1}{k_x} + \frac{1}{mk_y} \]

\[ \frac{1}{K_{oy}} = \frac{1}{k_y} + \frac{m}{k_x} \]

The actual area of interface between the two phases is generally not known in practice, the term \( \alpha \) is introduced as the interfacial area per unit volume of the column. As the value of \( \alpha \) is difficult to measure in practice, it is normally combined with the overall coefficient, and the product is known as "volumetric overall coefficient". For a countercurrent differential contactor, the following equation holds (assuming that the solute is extracted from the dispersed phase):

\[ d(L_c y) = d(L_d x) = K_{ox} a(x - x^*) dZ = K_{oy} a(y^* - y) dZ \]

where \( Z \) is the height of the tower,
\( L \) is the flowrate in \( \text{lb mole/hr ft}^2 \),
\( K_{ox} \) and \( K_{oy} \) are the overall area-based mass coefficients,

\( 'K_{ox}a' \) is the overall mass transfer coefficient based on the dispersed phase, and

\( 'K_{oy}a' \) is the overall mass transfer coefficient based on the continuous phase.

1.3.2. Interfacial area

As pointed out by some workers, it is possible to get an insight into the transport phenomenon if the interfacial area of contact between the two phases can be estimated or predicted. This is shown to be related to the dispersed phase hold-up and mean drop size as follows:

\[
a = \frac{6\phi}{d_{32}}
\]

where \( \phi \) is the hold-up, and

\( d_{32} \) is the Sauter mean drop diameter.

Sauter mean drop diameter is also known as volume-surface diameter or simply surface mean diameter and its value is related to the mass transfer rates, but not directly. This is due to the fact that other factors are also at work, such as the size of the drops, effects of axial mixing, internal circulation, etc. Very small drops are seen to behave like rigid spheres, but with larger drops under turbulent flow conditions the liquid drops may tend to deviate from this behaviour, as mobility inside the liquid drop sets in and the liquid elements inside the drop begin to circulate. This phenomenon of internal circulation is due to the frictional drag in the drop equator region.

1.3.3. Driving forces

The amount of solute in a solvent is measured in terms of concentration or activity. Where the solution is very
dilute then any association or dissociation occurring in the solution is very small and it is approximately true that the concentration and activity are of the same order of magnitude. That is, the activity coefficient approaches 1. However, in strong solutions, the solute suffers association. In this case, the activity coefficient is greater than 1 and the concentration differs in magnitude from the activity. Conditions are then non-ideal and a complex relationship exists between the chemical potential and the concentration. This is of importance not only in defining the strength of the solution but also in establishing the driving force that exists in mass transfer of solute. Here, the rate of transfer depends on the concentration difference \( C^* - C \) where \( C^* \) occurs at the phase interface and \( C \) in the bulk. It could happen therefore that \( C^* \) has a large value (when non-ideality exists) and \( C \) has a very low value (when ideality exists). The situation is therefore very complicated.

In practice, this is overcome by developing from experiment the actual curves of solute in each phase under identical conditions. In this way, it is not required to 'predict' the distribution function under different concentrations and hence the physical-chemical aspects can be avoided.

As the situation in most contactors deviates from the plug flow, i.e. the ideal case, due to the effects known as 'axial mixing', it is reasonable to expect the driving forces, and hence the mass transfer rate, to be somewhat reduced.

1.4. Drop behaviour in a fluid-fluid system

1.4.1. Droplet hydrodynamics
The study of drop behaviour in a liquid-liquid extraction is of primary importance, as the contact between the two phases normally takes the form of droplets.

To understand the complex hydrodynamic aspects of this problem, a thorough knowledge of the characteristics of single drops is necessary. This involves the shape of forming drops, the shape and velocity of moving drops, internal circulation and drop oscillation, the boundary layer around mobile drops and the effect of surfactants. Indeed many workers have applied the analysis of single drops to the behaviour of swarms when information on the swarms as an entity does not exist.

The shapes of drops largely depend on physical properties such as the density, viscosity and surface tension of the two fluid media, and also on the size of the nozzle tips or orifices where appropriate.

For a mobile drop under a fixed flow regime (i.e. having a certain value of Reynold Number), internal circulation starts to occur when the radius reaches a certain critical value (This critical value is related to the density difference of the two phases and the interfacial tension of the system). This implies that the mobile drops behave differently depending on the particular fluid systems.

As the drop size increases, it ultimately starts to flatten and shows a tendency to oscillate. This oscillatory movement causes an alternate creation and destruction of interfacial area and the internal circulation becomes somewhat dampened by this wobbling action.

Another factor which may enhance the mass transfer rate is that of interfacial effects or disturbances at the
interface between the drop and the field liquid. These are generally known as "Marangoni effects" and are normally due to variations of local interfacial tension or extension or contraction of an interface.

The flow pattern around the fluid drop is important for mass transfer study, and it is generally recognised that a thin laminar sub-layer adjacent to the interface and a thicker turbulent boundary layer between this and the bulk of the continuous phase are formed, provided the drop velocities are above the creeping flow regime.

In an extraction contactor the movement of a swarm of drops is very complex and the mass transfer process depends largely upon numerous factors, such as distribution of drop sizes and the repeated coalescence and redispersion of droplets. It is the distribution of drop sizes which gives some kind of quantitative measure of the interfacial area, as both of them are related to the mean drop size.

1.4.2. Flooding

One of the most important factors to be considered for any fluid-fluid system is the flooding characteristic of a contactor. If the flow rate of either the dispersed phase or the continuous phase is held constant and that of the other phase gradually increased, a point is reached where the dispersed phase coalesces, the hold up of that phase increases, and finally both phases leave together through the continuous phase outlet. This effect is called "flooding". At flooding, the dynamic equilibrium that normally exists between coalescence and redispersion breaks down. Instead it shifts completely towards coalescence and this results in reduction of interfacial area and mass
The flooding characteristic is important in liquid-liquid extraction in the sense that it is the point of maximum throughput at which the contactor can operate.

1.5 Hold up

With respect to a packed column, "hold up" refers to the liquid retained in the column as films wetting the packing and as pools trapped in the crevices between the packing materials. It consists of two parts, known respectively as "static" and "operating or moving" holdups.

For the purposes of the present study, hold up is usually referred to as "droplet hold up" and is defined as the relation of the volume of the droplets of the dispersed phase to the total volume of liquid hold-up in the extractor.

As hold up is directly proportional to the interfacial area, it is obvious that it relates to the overall mass transfer coefficient too. Hence, to a certain extent, it is a measure of mass transfer efficiency. Therefore an accurate knowledge of droplet holdup is desirable if a better understanding of the mass transfer process is to be achieved.

1.6 Axial Mixing

Axial mixing, also known as longitudinal mixing, is a common phenomenon encountered in a liquid-liquid contactor. Axial mixing occurs because of two phenomena. The first is the fact that the velocity profile of the liquid across the cross-section of the tower is not in the form of a plug flow. Some liquid is thus caused to circulate in the wrong direction which gives rise to radial mixing. This phenomenon
is generally known as "Taylor diffusion". The other phenomenon, turbulent and molecular diffusion in the axial direction, gives a further classification based on forward and backward mixing.

Axial mixing occurs in and applies to both the continuous and dispersed phases. Its occurrence is undesirable as far as the mass transfer process is concerned, since the difference between the solute concentrations in the two contacted phases is thereby reduced, in turn resulting in lower rates of extraction than would prevail under plug flow condition.

Similar effects apply to the dispersed phase. But the effects are mainly due to the spread of the residence time distribution, which in turn is affected by the drop interaction, and the actual backflow of some drops in a random manner.

The magnitude of the axial dispersion can be evaluated by measurements of residence time distribution in the continuous and dispersed phase. The most common method is by the so called "stimulus response" technique. This involves the injection of some kind of tracer into the inlet of the column and observation of the concentration at certain locations, such as the exit of the column, as a function of time. It is essential that the tracer should be soluble only in the phase under consideration.

1.7 Significance of the fixed baffle contactor

While there has been a lot of interest concentrated on the external power of the extractor, relatively little work has been done on the "static" side. However, the significance of the fixed baffle tower should not be
Some of the earliest work on this field was done by Morello & Poffenberger (M1) and Vulcan Company (V1) and little or no work has been carried out since! The fixed baffle contactor presents a challenge in the study of the mechanism of mass transfer. The two previous studies above were concerned only with performance overall for different systems and gave no indication of the mechanisms which form the basis for future design. The absence of knowledge or any fundamental understanding of mechanisms, both physical and hydrodynamic, prompted the undertaking of the present research.

 underestimated.
Chapter 2
Literature Review
2.1 Liquid-Liquid Extractor

A critical review of the classification of extraction equipment has been presented by Morello & Poffenberger (M1). The classification itself was further separated, depending on the construction and the operational characteristics, into two categories, stagewise and differential contactors.

The operation of the process is normally carried out either by an external power source or is simply gravitational. The performance of such contactors has been investigated by numerous workers.

2.1.1. Spray Column

The spray column is one of the simplest liquid-liquid contactors available. The movement of the dispersed phase droplet swarm is governed by the physical factors such as the distribution of drop sizes, drop interaction, the local velocities of the fluids and the eddy motion in the continuous phase. Its capacity of operation depends on the throughput of both phases and also on the size of the droplets of the dispersed phase. The extraction rates are somewhat reduced due to the effects of axial mixing, hence the spray column is generally employed when the requirement of the number of overall transfer units is not high. A tremendous amount of work involving spray columns has been done. Christiansen & Hixson (C1) have investigated in detail the drop breakup mechanism at single nozzle distributors. The hold up of the dispersed phase was studied fully and its numerical value was evaluated by a relationship given by Appel & Elgin (A1) and Johnson & Bliss (J1). Lapidus & Elgin (L1) considered the working of the spray column as a special case of a continuous fluidized bed and they also
defined a quantity known as "slip velocity" in terms of the superfacial velocities of the phases and the hold up of the fluid system.

Minard & Johnson (M2) have carried out studies covering a variety of liquid-liquid systems under so-called "flooding velocities" conditions and they proposed a correlation in terms of the slip velocity and the physical properties of the fluids.

Among the pioneers of mass transfer study in spray columns were Elgin & Browning (E1) who studied the extraction of acetic acid from an aqueous solution by isopropyl ether and vice versa. One of their resulting analyses is the well known square root plot for flooding velocities,

\[ \frac{U}{c}^{1/2} + \frac{1}{d}^{1/2} = \frac{1}{o}^{1/2} \]

where \( U_o \) is a constant.

The work done by Heertjes, Holve & Talsma (H1) involved a partially miscible system of isobutanol and water. Because of differences in density and movement of the drops the continuous phase (isobutanol) appeared to be thoroughly mixed and this gave a nearly constant concentration which was independent of the height of the column.

Sampling techniques have been investigated by Geankoplis & Hixon (G1) and Gier & Hougen (G2). The dependence of the mass transfer coefficients on the direction of solute transfer was analyzed by Satish et al (S1) with the aid of the experimental data of Gier & Hougen (G2) and Johnson & Bliss (J1) etc.

End effects, which were one of the causes of the
discrepancy in results have been studied by some workers such as Geankoplis et al (G 3).

The significance of the individual phase resistances has been investigated by Laddha & Smith (L 2) and Hong (H 2).

Generally speaking, most of the work involving the efficiency of mass transfer in spray columns was to correlate the data in terms of the overall height of transfer units and the extraction factor as proposed by Colburn (C 2).

2.1.2. Packed Tower

The packed tower is virtually identical to the spray column except for the introduction of packing material such as Raschig rings into the column. Most of the factors governing the mass transfer and the movement of the droplet swarm are the same as in the spray column. The main function of the packing pieces is to promote more intimate mixing and increase the local velocity of the continuous phase. This gives rise to a higher performance as compared with the spray column.

Extensive studies of the mass transfer rate and hold up in packed towers have been carried out by Varteressian & Fenske (V 2) and Allerton et al (A 2). The effect of packing size and column diameter on mass transfer has been investigated carefully by Leibson & Beckman (L 3) who expressed their results in terms of the extraction factor and the overall height of transfer unit.

The mass transfer rates of different systems have been predicted in terms of the physical properties of the fluids by various workers; amongst them were Degaleesan & Laddha (D 3) and Krishnan (K 1). The direction of solute
transfer was also taken into account in these correlations.

2.1.3. Plate Towers

2.1.3.1. Perforated Plate Towers

A perforated plate tower can be considered as a series of short and wide spray column sections or stages arranged one over the other in which the contacting phases flow countercurrently between the stages. The hole size of the perforated plate should lie within certain limits as found by Vedaiyan (V3), and these limits are linked somehow to the interfacial tension and densities of the fluids. Treybal (T1) has given a correlation involving the diameter of the liquid jet issuing out of the perforation of the plate, and in his mass transfer studies he argued that the plot of overall height of transfer units against extraction factor should be avoided and instead the performance should be compared in terms of stage efficiency. For any specific hole size, higher stage efficiencies are obtained for a system with low interfacial tension. Effect of the flow rate ratio \( U_c/U_d \) is an important quantity to be decided upon for the purpose of design. Other factors worthy of consideration are the direction of solute transfer, and the wetting characteristics of the plate: these have both been reviewed by Garner et al. (G4).

2.1.3.2. Baffle Plate Column

Very little work has been done with respect to this column, but Morello & Roffenberger (M1) may be considered as pioneers. Instead, one of the earliest experimental works was carried out by the Vulcan Company (V1) and some of the features and advantages of such column were given by them.
2.1.4. Rotary Agitated Column

2.1.4.1. Rotary Disc Contactor

Reman(R 1) and Reman & Olney(R 2) were the first to develop the rotary disc contactor(RDC) and it consists of a cylindrical column with rotor disc and stator rings arranged alternately. The rotor disc is driven by a central shaft and the dispersion is created by the shear forces set up between the discs. The design of such column can best be based on the correlation given by Misek(M 3).

Detailed study involving the dispersed phase hold up and characteristic velocity has been carried out by Logsdail et al(L 4), King & Beckmann(K 2), Strand et al(S 2) and Misek(M 4) etc.

Logsdail et al(L 4) correlated the characteristic velocity with the hold up and flow rates, as in the case of the spray column. An alternative expression of the characteristic velocity was also given in terms of the various column parameters and physical properties of the liquids. If the characteristic velocity is plotted against the rotor speed, two regions will be obtained. The boundary between these two regions is known as the critical rotor speed.

Mass transfer rates in such contactors have been investigated by many workers, such as Logsdail et al(L 4) and Vermijs & Kramers(V 4). They have given a correlation for the estimation of the overall height of transfer unit, based on the continuous phase. Vermijs & Kramers(V 4) plotted the $(Ka)_d$ against dispersed phase hold up with rotor speed as parameter. A maximum value of $(Ka)_d$ was obtained, and this was an indication of the fact that the drop size
distribution was affected.

Again, the direction of the solute transfer is another factor which should not be neglected. Also to be considered is the axial mixing, and this has been dealt with comprehensively by Strand et al (S2), Stainthorp & Sudall (S3) and Stemerding et al (S4). Stemerding proposed a correlation for the axial dispersion coefficient in the continuous phase and Stainthorp & Sudall introduced a quantity known as backmixing factor. However, Strand et al. proposed correlations for estimation of both the dispersion coefficients for the continuous and dispersed phases.

2.1.4.2. Other types of Agitated Column

(a) Oldshue-Rushton Contactor (O1) —-------- This is essentially the same as the RDC but with the rotating disc being replaced by impellers. High efficiency has been claimed for such column. Axial mixing characteristics have been investigated by Bibaud & Treybal (B1) and both the dispersion coefficients were given in the form of a correlation. Subsequent modifications of this contactor were made by Garner et al. (G5) and Treybal (T2) for the sake of reducing axial mixing. The unit developed by Treybal is generally known as "Treybal liquid-liquid contactor" and it consists of two vertical cylinders with the smaller one mount asymmetrically within the larger one. The dispersion, which is created by the centrally located turbine rotors inside the inner compartments, leaves through a port into the annular region.

(b) Scheibel Column (S5)

This column consists alternate packed and unpacked sections serving as settling and mixing chambers. The
dispersion is generally created within the unpacked section by a centrally mounted rotor shaft carrying a number of equally spaced turbine blade impellers. There is a critical agitator speed above which the stage efficiency decreases because of the possible formation of emulsion which could not be broken by the packing material.

Karr & Scheibel (K3) investigated the mass transfer rate and indicated that there is a critical flowrate of dispersed phase below which the overall mass transfer coefficients would be independent of the flow rates of both phases. Above the critical flowrate, the overall mass transfer coefficient obeys certain correlation with some of constants are specific for the particular system. The effect of solute transfer direction on efficiency was further studied by Scheibel (S6).

(c) Rotary Annular Column

The dispersion in this column is created by the shearing forces produced in the annular space between the column shell and the coaxial rotating cylinder. Hold up and characteristic velocity of the droplets below flooding have been investigated by Thornton & Pratt (T3) and Davis & Weber (D2) and correlations on such quantities were given. Mass transfer rates were studied in detail by Ney & Lochte (N1), Short & Twigg (S7) and Thornton & Pratt (T3). Thornton & Pratt indicated that the presence of an organic solute has a tremendous effect on the limiting throughput and they analysed their results by the Colburn method, i.e. in terms of the overall height of transfer units.

2.1.5. Pulse-Agitated Columns

These types of column were developed in view of the inferior capacity and throughput rates of the ordinary
rotor-agitated columns. The pulsing, which is normally generated by some sort of mechanical device, would give the advantage of high throughput and high transfer efficiency. One common example of such columns is the one devised by Van Dick (V 5) and it consists of reciprocating perforated plates moving vertically up and down the column. The alternative version of this type of column is that which allows stationary packing or sieve-plates: the liquids are pulsed hydraulically by an outside pulsing device.

The mass transfer rates, operational characteristics and dispersed phase hold-up of different pulsed columns have been investigated by various workers. For pulsed sieve plate column, the operational characteristics and mass transfer rate studies were carried out by Li et al. (L 5) and Cohen & Beyer (C 3), while the axial mixing characteristics were investigated by Smoot & Babb (S 8) and Vermeulen et al. (V 6).

The performance of the pulsed packed column has also been analysed in many works. Among these were Potnis (P 1) on the flooding rates, Wiegandt and coworkers (W 1) on the hold up, and Feick & Anderson (F 1) and Chantry et al. (C 4) on the mass transfer. Axial mixing in the continuous and dispersed phases has been reported by Vermeulen & coworkers (V 6).

2.1.6. Mixer-Settler

Mixer-settler is one of the simplest contactors, which largely explains why it was the earliest contacting device for fluid-fluid systems. The normal arrangement of such contactors consists of two tanks in series acting as a single stage. One of the tanks is used for mixing, which is
induced by some kind of paddle agitator, while the other is used for settling the mixed phase. One of the advantages of such contactors is the potential high efficiency. A full discussion on these types of apparatus can be found in Treybal's work (T1) and, in addition, the mass transfer aspects of such contactors have been studied by Calderbank (C5).

2.1.7. Miscellaneous types

Apart from the extractors mentioned above, some other types such as Podbielniak, Luwesta, De Laval and Graesser Raining-bucket contactor, etc are also available.

2.1.8. Selection of extractors

The selection of a particular apparatus for a given extraction process is normally governed by economic factors and also by the physical and chemical properties of the system. The question of the solvent recovery also looms large. Comprehensive guidance on this point is given by Hanson (H3).

2.2 Drop behaviour

The behaviour of the droplets can be considered in relation to the following four areas:

2.2.1. Drop formation

If the volume of a drop released from a capillary tube is plotted graphically against the time of formation, a relation such as shown in Fig.2.1. can be obtained. This curve can be divided roughly into four parts.

Regions I and II have been studied relatively rarely. However, surveys on region I have been carried out by Levich (L6) and Christiansan & Hixson (C1).

For region III, some relations have been established by
Fig. 2.1 The relation between drop volume and time of formation
workers such as Hayworth & Treybal (H 4) and subsequently reviewed by Kintner (K 4) and Jackson (J 2). This was further analysed by Heertjes & De Nie (H 5) and correlation on the release time were given in terms of the time of formation and the physical properties of the system.

In region IV, the drop volume is independent of the time of formation and its value can be predicted by Harkins & Brown's method (H 6). A good survey on this topic is contributed by Kintner (K 4).

2.2.2. Drop coalescence

Coalescence of liquid droplets is a subject of such complexity that it can be studied in its own right. A detailed analysis of this field is given by Jeffreys & Davies (J 3). They stated that the coalescence process takes place through five consecutive stages:

(i) The approach of the drop to the interface, hence resulting in deformation of the two surfaces coming together.

(ii) Damped oscillation of the drops.

(iii) The formation of a film of the continuous phase between the drop and its bulk phase.

(iv) After the drainage, rupture and eventual removal of the film, the proper coalescence process starts.

(v) Partial or total transference of the contents of the drop into its bulk phase.

Stages (i) and (ii) are assumed to be extremely fast and hence the time taken by stages (iii), (iv) and (v) is considered to be the time of coalescence. However, it has been found by research workers that most of the time taken for the coalescence of a drop to occur is the drainage time and this is known as "rest time" generally. This coalescence
time is such an important quantity that a considerable amount of work has naturally been done on it. One such study by Elton & Picknett (E 2) produced the following relationship:

\[ \log(N/N_0) = -Ct^n \]

where \( N \) is the number of drops after time \( t \),
\( N_0 \) is the total number of drops,
\( C \) is known as the coalescence constant, and
\( n \) is a constant which varies according to the system used.

A similar log. relationship has been given by Gillespie & Rideal (G 6), and there has been a dispute about which is the better representation of the quantity.

No doubt, the dominant factors governing the coalescence process are drop size, distance of fall of the drop, shape of the interface, densities and viscosities of the phases, interfacial tension, temperature and vibrational effects of the environment, the existence of a third component and the possible electrical effects. All the above factors have been discussed fully by Jeffreys & Davies (J 3), who also produced a correlation relating the coalescence time and the physical properties:

\[ t_{1/2} = 4.53 \times 10^5 \left[ \left( \frac{\mu^{1/2} \rho^{1/2}}{\delta^2} \right) (T/25)^{-0.7} \right] \left( \frac{d^{1/2}}{D} \right)^{0.02} \left( \frac{T^{1/2}}{\mu^{1/2}} \right)^{0.55} \left( \frac{\delta}{\mu^{1/2}} \right)^{0.001} \]

where \( t_{1/2} \) is the "half-life" rest time, and
\( T \) is the temperature.

This was further simplified to give:
\[ \frac{d^2 \Delta \rho g}{\delta} = 1.32 \times 10^5 \left( \frac{L}{d} \right)^{0.18} \left( \frac{d^2 \Delta \rho g}{\delta} \right)^{0.32} \]

Mathematical models have been devised by various workers to describe different coalescence situations. The concept of equating the mechanical work to the energy dissipated through viscous shear within the draining fluid has been applied to the case of the coalescence of single drops at plane interfaces (limiting case) by Gillespie & Rideal (G 6). The time of drainage was given as follows:

\[ t = \frac{\mu \Delta \rho g a^5}{4 \gamma^2 \left( h_1^2 - h_2^2 \right)} \]

where \( a \) is the radius of the droplet,

- \( h_1 \) is the thickness of the film when the drop initially comes to rest (apparently) on the interface, and the time is taken zero at this moment, and
- \( h_2 \) is the thickness at time \( t \) when the film ruptures.

The above equation was further modified and expressed in terms of the probability of rupture.

An alternative approach based on the equilibrium between the shape of the drop and the deformed interface was given by Bashforth & Adams (B 2) and refined further by Princen (P 2) and Hartland (H 7).

Inter-droplet coalescence is different from the above mentioned case, as the quantity of continuous phase trapped between the coalescing interfaces is larger and hence the drainage time will also increase. This problem has been tackled by McAvoy & Kintner (M 5) and the result is expressed
in terms of a differential equation. For two rigid spheres of radius \( a_1 \) and \( a_2 \), the rate of approach is:

\[
\frac{dh}{dt} = \frac{-F_h}{6\pi \mu} \left[ \frac{1}{a_1} + \frac{1}{a_2} \right]^2
\]

where \( h \) is the distance between the two spheres.

For equal sized spheres of radius \( a \),

\[
\frac{dh}{dt} = \frac{-8\pi \gamma^2 h^3}{3 \mu a^2}
\]

Further studies on this field such as stepwise coalescence between drops, collision and coalescence frequency and coalescence probability can be found in the work by Jeffreys, Davies & Ali (J 3), Mackay & Mason (M 6), Howarth (H 8) and Scheele & Leng (S 9) respectively.

2.2.3. Droplet breakup

For a two-phase system under turbulent flow conditions, the condition of a drop at breakup has been derived by Hinze (H 9) as follows:

\[
\tau > \frac{\left( \gamma + \mu_d \frac{\tau}{\rho_d} \right)}{d_e}
\]

where \( d_e \) is the diameter of volume-equivalent sphere and is equal to \((6V/\pi)^{1/3}\), and the \( \tau \) is the total local shear stress.

This local shear stress is imposed by the continuous phase and acts to deform a drop or bubble and to break it if the counterbalancing surface tension forces and viscous
stresses inside the fluid particle are overcome.

The above equation leads to a prediction of a critical (or "maximum stable") size if $\gamma$ can be evaluated. Indeed this was achieved by Kolmogoroff (K 5). By considering the difference in dynamic pressure exerted on opposite sides of a drop and the eddy velocity over a region of length equivalent to its diameter, he obtained the following expression for the stable drop:

$$ r_{\text{max}} = \sqrt{2(\gamma/k_f \rho)^{3/5}(D/U^3)^{2/5}} $$

where $k_f$ is a coefficient,

$D$ is the diameter of the agitator, and

$U$ is the velocity of the homogeneous turbulent flow.

However, this was criticised by Kagan et al. (K 6) and he proposed a correlation in terms of modified Reynolds and Froude Numbers.

Correlation involved Sauter mean drop diameter $d_{32}$ and the $N_{\text{We}}$ was given by Vermeulen et al. (V 7) and subsequently confirmed by Calderbank (C 5):

$$ d_{32}/D\phi = 0.055N_{\text{We}}^{-0.6} $$

where $D$ is the impeller diameter,

$N_{\text{We}}$ is the rotational Weber Number, and

$\phi$ is the hold up, which shows the significance of the coalescence on the mean drop size in the system.

For the breakup mechanism in a fragmentation process,
Sleicher(S 10) correlated the maximum stable drop size to the other parameters to account for the shear stress by the following expression:

\[ N_{We}(\mu_c U/\gamma)^{1/2} = 38 \left[ 1 + 0.7(\mu_d U/\gamma)^{0.7} \right] \]

An alternative concept based on the energy associated with eddies was also given by Hinze(H 9) and the work on the application of this theory has been carried out by Middleman(M 7).

2.2.4. Movement of the drops

For fluid particles rising or falling freely in infinite media it is possible to prepare a generalized graphical correlation in terms of the Reynolds Number, Morton Number and Eötvös Number:(Fig.2.2)(C 6)

\[ N_{ReP} = \frac{\rho_d d_c u}{\mu_c} \]
\[ N_M = g \frac{\mu_c}{\rho_c} \frac{2}{\rho_c} \]
\[ N_{Eo} = g \frac{\rho d_c^2}{\gamma} \]

The graphical plot cannot be applied to the case with extreme values of density ratio, \( \rho_d/\rho_c \), or viscosity ratio, \( \kappa = \mu_d/\mu_c \), where such a situation exists for liquid drops falling through gases. However, it can be used to estimate terminal velocities under various shape regimes. It is interesting to note that the viscosity of the droplets is relatively unimportant(except possibly with very pure (surfactant-free) systems or for large fluid particles in
Fig. 2.2  Shape regimes for bubbles and drops in unhindered gravitational motion through liquids. (The values along the axes are expressed in log scales.)
high Morton Number liquids) in determining terminal velocities and shape regimes, as it does not appear in any of the three dimensionless groups.

A complete analysis of the motion of the particles in fluids was given by Hadamard(H 10) and Rybczynski(R 3) and the terminal velocity of a fluid particle under the condition of creeping flow is:

\[
U_T = \frac{2}{3} g r^2 \frac{\Delta \rho}{\mu} \left( \frac{1+K}{2+3K} \right) \]

where \( \left( \frac{1+K}{2+3K} \right) \) is the viscosity correction factor.

For the steady creeping flow past a rigid sphere, Stokes(S 11) derived his equation for the terminal velocity by putting \( K \to \infty \), hence:

\[
U_{TS} = 2gr^2\Delta \rho/9\mu
\]

According to the Hadamard-Rybczynski theory, the terminal velocity of a fluid sphere should be up to 50% higher than that of a rigid sphere of the same size and density. However, it has constantly been observed that the small bubbles and drops tend to obey Stokes's law and have no internal circulation. Various explanations have been advanced by different workers, but all seem to be flawed in some respect. The most acceptable version was the surface contamination theory given by Frumkin & Levich (F 2, L 6) which states that the concentration gradient which, due to the accumulation of the contaminants, gives rise to a tangential gradient of surface tension, in turn
causes a tangential stress tending to retard surface motion. This theory implies that all bubbles and drops, irrespective of their sizes, will show internal circulation if the system is sufficiently free of surface-active contaminants.

Further expansion of this theory was given by Hu & Kintner (H 11) with reference to other factors such as drop deformation and oscillation.

The correlation relating the various physical parameters including the drag coefficient have been investigated and subsequently presented by various workers such as Krishna et al. (K 7).

The question of incorporating the single drop relationship as given above to the droplet swarms has also been studied by various researchers and one of many results was presented by Vedaiyan (V 3) as follows:

\[ U_S = \frac{U_d}{\phi} + \frac{U_c}{1 - \phi} = \bar{u}_o (1 - \phi) \]

where \( U_S \) is the slip velocity, and
\( \bar{u}_o \) is the "Characteristic velocity" of the dispersed droplets which also agrees well with the average velocity of the droplet swarm at all flow rates.

2.3 Mass transfer theories and related topics

2.3.1. Available theories

Various mass transfer theories have been put forward by many workers. Amongst the earliest ones were the film theory and the two-resistance (also known as two-film) theory. The two-resistance theory given by Lewis & Whitman (L 7) assumes that the only diffusional resistances are
those residing in the fluids themselves, and the solute concentrations in the interface are in equilibrium with each other. The oversimplication of this theory was criticized by Higbie (H 12) and hence he presented his Penetration theory. The main theme of this theory was the constant time of exposure of the eddies of fluid during the contact. However, this was later modified by Danckwerts (D 3) and he stated that Higbie's concept was only a special case of a more realistic picture in which the eddies are exposed for varying lengths of time, i.e. the time exposure forms some kind of distribution.

Recently, various combinations of the above theories have appeared. A combination of film-surface renewal theory was given by Dobbins (D 4) in which he proposed that the transfer coefficient is proportional to certain power of the diffusivity, and the value of this power varies according to the circumstances. Toor & Marchello (T 4) have made similar suggestions and further combinations were also presented by Kishinevskij (K 8) and Ruckenstein (R 4). The confirmation of Danckwert's theory was recently again given by Lamont & Scott (L 8) in their Eddy Cell Model.

The variation of mass transfer with time was investigated by Angelo et al. (A 3, A 4), finally leading to the development of the Surface-Stretch theory, which pays particular attention to the wobbling of the droplets.

2.3.2. Estimation of the mass transfer coefficients

It has been shown that the mass transfer rate during drop formation (mainly by diffusion) accounts for a large proportion of the overall process. So far, there are four available models which can be used to explain the physical
phenomena involved, i.e. the behaviour of the growing boundary layer, occurring in the drop with respect to the layer near the interface. These four models are generally known as aging rigid boundary layer model, balloon model, etc. and they all been tested by various workers. All four models state that the efficiency is proportional to the square-root of the time of formation and the value of the proportionality constant varies according to the models used. A fuller account can be obtained from the work of Licht & Pansing (L 9), Baird (B 3), Groothuis & Kramers (G 7). One interesting point to note is that these models are valid only for stable interfaces.

The biggest drawback of the above models is that they pay insufficient attention to the different regions of drop formation as stated in section 2.2.1.

Once the drop is formed, different considerations should be given to the process, and hence the study of mass transfer between a single particle and the surrounding fluid can be classified into two categories, viz. mass transfer in the dispersed phase and in the continuous phase.

2.3.2.1 Mass transfer in the continuous phase

(i) From and to rigid drops

A correlation was proposed by Linton and Sutherland (L 10).

\[ (N_{Sh})_c = 0.582(N_{Re})^{1/2}(N_{Sc})^{1/3} \]

This correlation describes the overall process in the light of the difficulty of estimating the contribution to mass transfer of the wake of the drop.
A modified form of the above correlation was given by Rowe, Claxton & Lewis (R 5) to include the diffusion process:

\[(N_{Sh})_c = 2 + 0.76(N_{Re})^{1/2}(N_{Sc})^{1/3}\]

The wake effect was investigated by Kinard, Manning & Manning (K 9) and their result was:

\[(N_{Sh})_c = 2 + (N_{Sh})_n + 0.45(N_{Re})^{1/2}(N_{Sc})^{1/3} + 0.0484(N_{Re})(N_{Sc})^{1/3}\]

where \((N_{Sh})_n\) is the contribution for natural convection,

\[(N_{Sh})_n = f(Nr, N_{Sc}), \text{ and}\]

the last term for the wake effect at the rear.

Further work on the wake was carried out by Linton & Sutherland and the angle of separation \(\theta_s\) for the rigid sphere was given as follows:

\[\theta_s = 83 + \frac{191}{N_{Re}^{1/3}}\]

According to Ward et al. (W 2), the viscosity factor \(\kappa = \mu_d/\mu_c\) plays an important role and for \(N_{Pe} > 1000\)

\[(N_{Sh})_c = 0.98(N_{Pe})^{1/3}\]

(ii) Non-rigid drops

For non-rigid drops, the Garner & Tayeban correlation (G 8), which takes account of the wake effect, can be used.

\[(N_{Sh})_c = 0.6(N_{Re})^{1/2}(N_{Sc})^{1/2}\]
OR with Ward et al.'s (W 2) correlation

\[
(N_{Sh})_c = 0.61 (N_{Pe})^{1/2} \left( \frac{\mu_d}{\mu_c + \mu_d} \right)
\]

for \( N_{Pe} \gg 2.8 \left( \frac{\mu_d + \mu_c}{\mu_d} \right) \left( \frac{12 \mu_c + 9 \mu_d}{\mu_d} \right)^2 \)

(iii) Oscillations of drops

Garner & Tayeban (G 8) derived the correlation for the oscillating drops with the aid of the experimental results of Elzinga & Banchero (E 3) who reported the large percentage increase in mass transfer due to oscillation.

\[
(N_{Sh})_c = 50 + 0.0085 (N_{Re}) (N_{Sc})^{0.7}
\]

Again, the effects of the wake is very difficult to estimate.

2.3.2.2 Mass transfer in the dispersed phase

(i) The rigid drops

For rigid drops with no internal circulation and with transfer by pure molecular diffusion, the film coefficient can be estimated by Treybal's relation (T 1):

\[
k_d \cong 2 \pi^2 D_d / 3 d_p
\]

(ii) Non-rigid drops

Two cases of non-rigidity can be considered, viz. laminar and turbulent circulations. Using the concept of Hadamard-Rybczynski, Kronig & Brink (K 10) gave the following formula for drops with laminar circulation:

\[
k_d \cong 17.9 D_d / d_p \quad \text{for } (N_{Re}) < 1.
\]
On the other hand, Handlos and Baron's relation (H 13) can be used for the drops with turbulent internal circulation:

\[
  k_d = 0.00375 \frac{U_t}{(1 + \frac{\rho_d}{\rho_c})} \quad \text{for long durations of contact.}
\]

For short contact durations:

\[
  k_d = 0.972 k_{HB} + 0.075 \frac{d}{t}
\]

This modified version was due to Olander(0 2) and \( k_{HB} \) is the film coefficient from Handlos & Baron's equation.

(iii) Oscillating drops

Oscillation can be induced by non-rigidity and it normally begins to occur in regimes of flow for which \( N_{Re} = 200 \) for systems with a low viscosity in the continuous phase. Rose and Kintner(R 6) have investigated such phenomena and their result was expanded further by Angelo et al.(A 4) as follows:

\[
  k_d = \left( \frac{4D_d w (1 + \xi_0)}{\eta} \right)^{1/2}
\]

where \( w \) is the frequency, and \( \xi_0 \) is a dimensionless factor for the amplitude.

2.3.2.3 Mass transfer in the swarm of drops

The most common correlation to be used for the swarm of droplets is the one due to Ruby & Elgin(R 7). This equation was recommended by Treybal(T 1) with the terminal velocity of the droplet(\( U_t \)) replaced by the slip velocity(\( U_s \))
\[(N_{Sh})_c = 0.725(N_{Pe})^{0.57}(N_{Sc})^{-0.15}(1 - \phi)\]

The effects of the purity of the system and the swarm of gas bubbles were considered by Waslo & Gal-Or (W 3) and Calderbank & Moo-Young (C 7) and their correlations are given in section 3.5.2.

However, for a pure system at low hold up, Levich's equation can be used:

\[(N_{Sh})_c = 0.653\left(\frac{\mu_c}{\mu_d + \mu_c}\right)^{1/2} (N_{Re})^{1/2} (N_{Sc})^{1/2}\]

Again, the effects of the viscosity ratio were pointed out by Hughmark (H 14).

2.3.3. Interfacial instabilities

One of the equilibrium forces acting in the interface is the interfacial tension, which is the characteristic feature of the interface. The local variations in its value are normally caused by the mass transfer process and are generally known as Marangoni effects. The hydrodynamic aspects of interfacial turbulence were analysed by Sternling & Scriven (S 12) with their roll cell model, paying particular attention to the direction of transfer and the physical properties of the system.

A mathematical analysis of this topic was presented by Sawistowski (S 13) who, with Maroudas (M 8), carried out studies of the process with simultaneous transfer of two solutes across an interface, emphasising the interrelationship between the two solutes.

2.3.4. Sizing and counting of the droplets

The most common used technique for sizing the droplets
is the photographic method. Either the droplets are photographed directly through the contactor or a sample of the dispersion is taken out and photographed immediately. All these techniques have been carried out in the past by workers such as Gucker & Rose (G 9) and Thomas & Weng (T 5).

The other techniques are chemical methods, which have been investigated extensively by Nanda & Sharma (N 2) and light transmittance method by Langlois, Gullberg & Vermeulen (L 11). The light transmittance method does away with the tedium of counting a large number of droplets, and it is based on the theory that the amount of light transmitted through a dispersion sample is proportional to the droplet density and hence the actual number of droplets in the sample.

2.4 Equilibrium study

Equilibrium study is an important part of any mass transfer process as it provides information for the eventual estimation of the overall efficiency of the apparatus.

The equilibrium study of the present system has been investigated by Lewis (L 12) and Thomas & Weng (T 5) and the actual chemistry of the equilibrium system was explained by Chaikhorskii (C 8). He pointed out that the acetic acid behaves predominantly as a dimer in carbon tetrachloride and as a monomer in water. (Appendix 2)

2.5 Flooding

There have been many studies of flooding, but the results achieve no solid consensus. The studies were largely carried out without mass transfer, and because of the influence of mass transfer on drop sizes, it is reasonable to suggest that there is an influence on flooding as well. The result
of Crawford & Wilke (C 9) on packed tower was presented in graphical form with various combination of the physical properties group.

Similar work on the packed tower has been done by Dell & Pratt (D 5) and their result was:

\[ 1 + 0.835 \left( \frac{U_d}{U_c} \right)^{1/2} \left( \frac{\rho_d}{\rho_c} \right)^{1/4} = C \left( \frac{u_c^2 a_p \rho_c}{\epsilon \epsilon^3 \Delta \rho} \right)^{-1/4} \]

where \( a_p \) is the surface of packing in sq ft/cu ft or its equivalent,
\( \epsilon \) is the void fraction based on the total column volume, and
\( C \) is a constant which varies according to the nature of the packings.

The results of the above four workers were combined together by Venkataraman & Laddha (V 8) and the correlation stated previously was modified.

Flooding studies on spray tower have been carried out by Elgin & Browning (E 1) and Minard & Johnson (M 2).

For perforated plate tower, the flow capacity might be expected to be limited in the same way as in packed and spray towers. Of course, the pressure drop developed for dispersed phase flow through the holes should not be neglected.

The flooding of the other types of column such as RDC has been investigated by Reman (R 8) and Logsdail et al. (L 4), and pulsed sieve-plate column by Smoot et al. (S 14), and pulsed packed column by Potnis (P 1).

2.6 Axial dispersion

It is generally held that in any continuous
countercurrent extraction process the effective mass transfer rate is reduced by axial dispersion in either phase. Axial mixing normally results from a combination of longitudinal dispersion along the direction of flow, both ahead of and behind the average flow, and it was treated as a diffusion process by Taylor (T 6) and Danckwerts (D 6).

Among the pioneers in carrying out a general theoretical analysis of this topic were Levenspiel & Smith (L 13), Klinkenberg (K 11), Miyauchi & Vermeulen (M 9), Hartland & Mecklenburgh (H 15) and Stepanek & Shilimkan (S 15). Other workers such as Vermeulen et al. (V 6), Gier & Hougen (G 2), Hazlebeck & Geankoplis (H 16), Westerterp & Landsman (W 4), Strand et al. (S 2), Weng (W 5) and Smoot & Babb (S 8) have all applied the analysis to specific contactors.

Strand et al. (S 2) proposed that the overall axial dispersion process for the continuous phase consists of two main components, viz. backmixing and forward contributions. Hence the overall apparent axial dispersion coefficient is the sum of the two. Westerterp & Landsman (W 4) put forward the similar argument with respect to the performance of their RDC, but including the contribution made by the impeller. Their result was based on the correlation given earlier by Nagata et al. (N 3).

The most common method of determining the axial mixing coefficient is to utilize the residence time distribution data. Relationships between the variance of the distribution curve and the Peclet Number, which is a measure of the degree of mixing, under various conditions have been derived by different workers such as Aris (A 5), Van der Laan (V 9), Bischoff (B 4), Levenspiel & Smith (L 13) and Sleicher (S 16).
It was also proposed by Jordan (J 4) that the axial mixing coefficient for the dispersed phase in a packed tower can be calculated as:

$$\frac{d_p U_d}{D} = 0.35$$

A similar correlation for the dispersed phase was obtained by Vermeulen et al. (V 6). However, the continuous phase Peclet Number in their work has been found to change markedly with Reynolds Number and phase flow rates. These two coefficients can be used in conjunction with Sleicher's equation (S 16) to get the necessary correction for the experimental mass transfer data.

A comprehensive survey on the longitudinal mixing in liquid-liquid contactors has been written by Ingham (I 1).

Equations for the overall transfer coefficient and the number of transfer unit corrected for axial mixing effects have been derived by Miyauchi & Vermeulen (M 9).

2.7 Hold up

Extensive study on the hold up of the fluid system has been carried out by a number of workers, and most of the correlations derived were based on the original idea by Gayler & Pratt's work (G 10) on the packed column, i.e.

$$U_d + U_c (\phi \bar{u} - \phi) = e \bar{u}_o (1 - \phi) \phi$$

where $e$ is the void fraction based on total column volume, and $\bar{u}_o$ is the characteristic velocity and it is defined as the limiting mean droplet velocity.
at zero continuous phase flowrate, 
    i.e. \( U_c = 0, U_d \to 0 \).

More general expressions were given by Johnson & Laverge (J 5), Chandrasekaran et al. (C 10), and Degaleesan & Laddha (D 7).

The correlation given by Laddha et al. can be summarized as follows:

\[
\left( \frac{U_d}{U_c} + \frac{\varphi}{1 - \varphi} \right) \left( \frac{U_c^2 a \rho_c}{g e^3 \Delta \rho} \right)^{1/2} = C \varphi (1 - \varphi)
\]

where \( C \) is a constant and its value varies according to the following situations:

For no solute transfer:
    \( C = 0.683 \) for Raschig rings, Lessing rings and Berl saddles.
    \( C = 0.973 \) for spheres.

For solute transfer from continuous phase to dispersed phase:
    \( C = 0.637 \)

For solute transfer from dispersed phase to continuous phase:
    \( C = 0.82 \)

This difference in hold up for the same liquid systems and column variables under various conditions of solute transfer indicates the existence of other factors such as Maragoni effects.

The relationship between the characteristic velocity, the superficial velocities and the hold up of the dispersed
phase of the other types of contactors such as spray tower, perforated tower and RDC follows the same pattern as the one of the packed tower. Of course, the estimation of the characteristic velocity itself varies according to the type of the contactor as the column parameters of which it is related are different.

Experimental hold up measurements using drainage and displacement methods have both been carried out by Gayler & Pratt (G 10), but the most common and accurate one is the manometric method, which has been used by Minard & Johnson (M 2) and Weng (W 5).
Chapter 3

Theoretical Considerations of Various Parameters
3.1 Axial Dispersion

3.1.1 Flow Patterns

Plug flow and complete mixed flow are generally referred to as the two idealized flow patterns. Any deviation from these two is taken as a measure of non-ideality. This deviation can be caused by the creation of stagnant regions in the vessel, some mixing in the longitudinal direction and incomplete mixing in the radial direction, etc. The importance of the effects of axial dispersion (longitudinal dispersion) on mass transfer has been increasingly recognised, as can be seen from the large volume of information available on the subject. The value of the axial mixing coefficient can be determined by the residence time distribution study, and its magnitude reflects the degree of deviation from the plug flow behaviour.

Mixing perpendicular to the direction of flow is very small, hence the radial dispersion coefficient is generally neglected.

3.1.2 Residence Time Distribution Study

The time of residence of an element of fluid in a reactor as a statistical mean is called the mean residence time $\bar{t}$ and it has two important properties: time elapsed since the molecule entered the reactor (its age), and the remaining time it will spend in the reactor (its residual lifetime). In the case of a plug flow reactor, there is no distribution or range of residence times, as all the fluid elements spend the same time interval in the reactor. On the other hand, with a well mixed reactor, some fluid will leave almost immediately after entering the reactor and it therefore has a very small residence time, while some
further fluid will be retained for a much longer period. Also, there will be elements of fluid with residence times between these two extremes. A practical reactor with a certain degree of backmixing will not have either zero or infinite residence times. The form of residence time distribution curve may therefore be used to characterise the reactor. The performance of the reactor can also be predicted with the aid of first order kinetic data. The residence time distribution of material at the reactor exit can normally be expressed as: (D 6)

(i) a point distribution function\( E(t) \) or \( C(t) \) diagram), and

(ii) a cumulative distribution function\( F(t) \) diagram).

Hence a convenient definition of residence-time distribution function is the fraction \( F(t) \) of the effluent stream that has a residence time less than \( t \). There is another function called internal age distribution function and is given the symbol \( I(t) \)\( dt \). It is defined as the fraction of material which has been in the reactor for a time since entry between \( t \) and \( (t+dt) \). \( E(t) \) and \( C(t) \) diagrams (for closed vessel) are identical if the form of the stimulus to the system is an impulse.

3.1.3. Mean Residence time

Assuming that the variation of density of each element of fluid is negligible as it passes through the reactor, then the mean residence time is defined as:

\[
t = \frac{V}{Q} \quad (3.1)
\]
where V is the volume of the reactor, and 
Q is the volumetric flow rate.

For constant density, Q is the same for the feed as for the effluent stream.

\( \bar{t} \) is also known as "nominal holding time" and "space time".

3.1.4. Mean Value and Variance

From the definition of \( F(t) \), one can also say that 
\( dF(t) \) is the volume fraction of the effluent stream that has a residence time between \( t \) and \( t+dt \). Hence the mean residence time is also given by:

\[
\bar{t} = \frac{\int_0^t t dF(t)}{\int_0^t dF(t)} = \int_0^t tdF(t) = \frac{V}{Q}
\]  

(3.2)

as \( \int_0^t dF(t) = 1 \)

Since \( E(t) = C(t) = dF(t)/dt \) (See eq. 3.13), then

\[ F(t) = \int_0^t E(t) dt = \int_0^t C(t) dt \]

\[ \text{Hence } \bar{t} = \frac{\int_0^\infty tC(t) dt}{\int_0^\infty C(t) dt} \]  

(3.3)

The above \( \bar{t} \) is called the mean value or the centroid of the distribution. It is also known as the first moment and is given the symbol \( \mu \), as it can easily be obtained by moment analysis, i.e.

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

(3.4)

The values of \( \bar{t} \) (given by \( V/Q \)) and \( \mu \) are the same for closed
vessels but different for open vessels.

Another important parameter is concerned with the spread of the distribution and is generally known as the variance \( \sigma^2 \). It is defined as:

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

\[
= \frac{\int_0^\infty t^2 c(t) \, dt}{\int_0^\infty c(t) \, dt} - \mu^2 \tag{3.5}
\]

The variance represents the square of the spread of the distribution and has units of \((\text{time})^2\). They are normally expressed in terms of the finite calculus for the sake of computation, i.e.

\[
\mu = \frac{\sum t_i c_i \Delta t_i}{\sum c_i \Delta t_i} \tag{3.6}
\]

and \( \sigma^2 = \frac{\sum (t_i - \mu)^2 c_i \Delta t_i}{\sum c_i \Delta t_i} \)

\[
= \frac{\sum t_i^2 c_i \Delta t_i}{\sum c_i \Delta t_i} - \mu^2 \tag{3.7}
\]

For closed vessel with normalized distribution these equations can be simplified further, i.e.
\[ \mu = \int_0^\infty tE(t)\,dt = \sum \frac{t_i E_i}{E_i} = \sum t_i E_i \Delta t \]

and \[ \sigma^2 = \int_0^\infty (t - \mu)^2 E(t)\,dt = \int_0^\infty t^2 E(t)\,dt - \mu^2 \]

\[ = \sum \frac{t_i^2 E_i}{E_i} - \mu^2 = \sum t_i^2 E_i \Delta t - \mu^2 \]

3.1.5. Various Curves (E(t), C(t), F(t) and I(t) curves)

Definition of External Age Distribution Function -- It is defined as the fraction of material in the exit stream of the system which has spent a time between \( t \) and \( (t + dt) \) within the system and is denoted as \( E(t)\,dt \). Hence,

\[ \int_0^\infty E(t)\,dt = 1 \quad (3.8) \]

Similarly, the internal age distribution function is defined as the fraction of material which has been in the reactor for a time since entry between \( t \) and \( (t + dt) \) and is labelled as \( I(t)\,dt \). Also,

\[ \int_0^\infty I(t)\,dt = 1 \quad (3.9) \]

One factor to note is that \( I(t) \) and \( E(t) \) distribution functions are not necessarily identical.

The \( F(t) \) diagram is known as the cumulative distribution function and is defined as the total amount of material which has spent a time less than \( t \) in the reactor and is therefore obtained by integration of \( E(t)\,dt \) over the time interval \( 0 \rightarrow t \), i.e.
\[ F(t) = \int_0^t E(t) \, dt \quad (3.10) \]

As \[ \int_0^\infty E(t) \, dt = 1 \]

\[ \Rightarrow F(t) = 1 \text{ at } t = \infty \quad (3.11) \]

As stated earlier, for impulse testing, the response at the reactor exit is called a \( G \)-diagram and is identical with the \( E \) diagram, i.e.

\[ \int_0^t E(t) \, dt = \int_0^t C(t) \, dt = F(t) \quad (3.12) \]

or \( E(t) = C(t) = \frac{dF}{dt} \quad (3.13) \)

Of course, this is true for closed vessels only. Various curves are shown in Fig. 3.1. These curves can be expressed in terms of both ordinary and dimensionless time units.

In the case of the \( E(t) \) and \( C(t) \) diagrams of the plug flow, the discontinuity is dealt with by the Dirac delta function, \( \delta \). This special function is defined as:

\[ \delta(t - t_0) = \begin{cases} \infty & t = t_0 \\ 0 & \text{elsewhere} \end{cases} \]

such that \[ \int_{-\infty}^{\infty} \delta(t - t_0) \, dt = 1 \]

and \[ \int_{a}^{b} \delta(t - t_0) f(t) \, dt = f(t_0) \quad \text{if} \quad a < t_0 < b \]

\[ = 0 \quad \text{if the interval does not contain } t_0 \]
Fig. 3.1 $E(t)$, $G(t)$, $F(t)$ and $I(t)$ diagrams for various systems.
If the $E(t)$ and $C(t)$ curves of the regions other than the inlet and outlet are required, then the Convolution Integral has to be used.

### 3.1.6. Dispersion Coefficients and Peclet Number

Of the many models used to characterize non-ideal flow pattern in the reactor, the one which draws on the analogy between mixing in actual flow and a diffusional process is called a "dispersion model". The basic criterion for such a model is the assumption that a certain degree of backmixing is superimposed on the plug flow in such a way that its magnitude is independent of its position within the vessel. This condition then implies that there is no "dead spot" or short circuiting of fluid in the vessel. This model is generally known as dispersed plug flow model. Mathematically, the model is expressed as an unsteady state diffusion type equation:

$$\frac{\partial C}{\partial t} = E \frac{\partial^2 C}{\partial x^2} \quad (3.14)$$

where the parameter $E$ is called the longitudinal or axial dispersion coefficient and its value signifies the degree of backmixing during flow.

The above equation can be drawn in parallel with the Fick's law differential equation and the analogous manner is quite apparent:

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} \quad (3.15)$$

where $D$ is the coefficient of molecular diffusion.

As the superimposed backmixing could bring out both
longitudinal and radial dispersion, hence there are two coefficients, one for each direction. Fortunately, the radial dispersion coefficient is negligible, as the mixing perpendicular to the direction of flow is usually very small and also accomplished by molecular diffusion.

Equation (3.14) is more conveniently expressed in terms of the dimensionless parameters, i.e.

$$\frac{\partial C}{\partial \theta} = \left(\frac{E}{uL}\right) \frac{\partial^2 C}{\partial Z^2} - \frac{\partial C}{\partial Z} \quad (3.16)$$

where $Z = x/L$, and 
$$\theta = t/t = tu/L$$

For an impulse or step change in the fluid properties, the solution of the above equation will give a family of theoretical $E(t)$ or $C(t)$ and $F(t)$ curves having the dimensionless group $(E/uL)$ as the parameter which measures the extent the axial dispersion. It is called "Dispersion Number" and its features are given by Levenspiel (L14) as follows:

$E/uL \to 0$ negligible dispersion, hence plug flow.

$E/uL \to \infty$ large dispersion, hence mixed flow.

The reciprocal of the dispersion number is known as the "Peclet Number", i.e.

$$N_{Pe} = uL/E \quad (3.17)$$

For small extents of dispersion of a closed vessel, the solution of the equation (3.16) gives the symmetrical (normal)
C(t) curves, with $E/uL$ as the parameter. (Fig. 3.2)

$$C_\theta = \frac{1}{2\sqrt{\pi E/uL}} \exp \left[-\frac{(1 - \theta)^2}{4(E/uL)}\right]$$  \hspace{1cm} (3.18)

The dimensionless mean and variance of such curves are:

$$\bar{\theta}_c = \mu/\bar{t} = 1$$ \hspace{2cm} (3.19)

$$\sigma^2_\theta = \sigma^2/\bar{t}^2 = 2(E/uL)$$

or $\sigma^2 = 2(E/L/u^3)$  \hspace{1cm} (3.20)

and the parameter $E/uL$ can be evaluated from the experimental curve using the fact that the maximum value of $C(t)$, i.e.

$$C_{\theta\text{max}} = \frac{1}{2\sqrt{\pi E/uL}}$$

One point to note is that the analysis for small extents of dispersion as given above is insensitive to the boundary condition imposed on the vessel, whether closed or open. On the other hand, if the dispersion is large, then the analysis has to be dealt with differently. For a closed vessel, the $C(t)$ curves are shown in Fig 3.3. The important parameter $E/uL$ affects the skewness of the curves directly and the mean and variance are given as:

$$\bar{\theta}_c = \mu/\bar{t} = 1, \quad \mu = \bar{t}$$  \hspace{1cm} (3.21)
Fig. 3.2 Dimensionless C curve for small extent of dispersion.
Fig. 3.3 C curves in closed vessel for various extents of backmixing.
\[
\sigma_\theta^2 = \frac{\sigma^2}{\bar{t}^2} = 2(E/\mu L) - 2(E/\mu L)^2(1 - e^{-uL/E})
\]

\[
= \frac{2}{(N_{Pe})^2} \left( N_{Pe} - 1 + e^{-N_{Pe}} \right) \quad (3.22)
\]

The derivation of the C curves and their parameters for open vessels are given by Levenspiel & Smith (113) and the resulting equations are:

\[
C_\theta = \frac{1}{2\pi \theta (E/\mu L)} \exp \left[ - \frac{(1 - \theta)^2}{4\theta (E/\mu L)} \right] \quad (3.23)
\]

with mean and variance:

\[
\bar{\theta}_c = \mu/\bar{t} = 1 + 2(E/\mu L), \quad \mu \neq \bar{t} \quad (3.24)
\]

\[
\sigma_\theta^2 = \frac{\sigma^2}{\bar{t}^2} = 2(E/\mu L) + 3(E/\mu L)^2 \quad (3.25)
\]

For a one-shot traces input, the following equation deduced by Aris (A 5) holds:

\[
\Delta \sigma_\theta^2 = \frac{\Delta \sigma^2}{\bar{t}^2} = \frac{\sigma_{out}^2 - \sigma_{in}^2}{\bar{t}^2} = 2(E/\mu L) \quad (3.26)
\]

Another case of interest is the variance of the open-closed vessel which has been developed by Van der Laan (V 9), i.e.

\[
\sigma_\theta^2 = 2(E/\mu L) + 3(E/\mu L)^2 \quad (3.27)
\]

With small deviations from plug flow, the partial differential equation (3.16) can be transformed into an
ordinary differential equation as shown by Danckwerts (D 6).
The solution of such ordinary differential equations, with
the necessary substitution and appropriate boundary conditions,
will give a family of $F(t)$ curves as follows: (Fig. 3.4)

$$F(t) = (C/C_0)$$

$$= \frac{1}{2} \left[ 1 - \text{erf} \left( \frac{1}{\sqrt{\pi}} \left( \frac{1 - t/\bar{t}}{\bar{t}} \right) \right) \right] \quad (3.28)$$

where $\bar{t} = V/Q = L/u$, and

\text{erf} is the error function and is defined as:

$$\text{erf}(y) = \frac{2}{\sqrt{\pi}} \int_0^y e^{-x^2} \, dx$$

$$\text{erf}(\pm \infty) = \pm 1, \quad \text{erf}(0) = 0,$$
$$\text{erf}(-y) = -\text{erf}(y)$$

Again, the dimensionless group $E/uL$ is the curve-shape
determining factor.

3.1.7. Hold-back and Segregation

As stated earlier, both $C(t)$ and $F(t)$ curves can be used to measure the deviation from ideal flow.

In the $C(t)$ curve, the magnitude of deviation is expressed in terms of mean residence time and the axial dispersion coefficient. However, in the $F(t)$ curve, the magnitude of deviation from plug flow is called hold-back and the departure from perfect mixing is termed "segregation".

(i) Hold back
Plug flow, $E/uL = 0.0$

Stirred tank, $E/uL = \infty$

$F(t) = (c/c_0)_{\text{step}}$

Fig. 3.4 Response curves for plug flow with small deviations.
Consider again the $F(t)$ diagram plotted on a
dimensionless time scale. Fig. (3.5) depicts the step
disturbance imposed on the system, and it has been shown
by Cooper & Jeffreys (11) that:

$$\int_{0}^{\infty} \left[ 1 - F(t) \right] \frac{dt}{t} = 1 \quad (3.29)$$

Geometrically the above expression means that the area
between the $F(t)$ curve and the line $F(t) = 1$ is unity. From
the definition of the $F(t)$ curve, the area between the axes
and the lines $F(t) = 1$ and $\theta = 1$ is also unity. This
implies that the two areas $A_1$ and $A_2$ are equal. If the area
$A_1$ is known, then the $F(t)$ diagram may be completed
approximately by extending the monotonic function in such
a way that the area $A_2$ would be equal to area $A_1$. This
property serves as a geometrical check on the $F(t)$ diagram.

In fact, the area under the curve between $\theta = 0$ to
$\theta = 1$, i.e. area $A_1$ is known as hold back $H$:

$$A_1 = H = \int_{0}^{1} F(\theta) d\theta \quad (3.30)$$

also $A_1 = H = 0$ implies plug flow

Physically, the hold back can be interpreted as follows:
if the inflowing fluid changes suddenly from colourless to
blue, then the hold back is equal to the fraction of the
volume of the colourless fluid after a volume of the blue
fluid equal to the vessel volume has passed through.

(ii) Segregation

The equation of the perfect-mixing curve in terms of
Fig. 3.5 Hold back
the dimensionless time scale is given as follows:

\[ F(\theta) = 1 - e^{-\theta} \quad (3.31) \]

where \( \theta = \frac{t}{\bar{t}} \)

If this curve is drawn with another \( F \) curve with dispersion, then the resultant is shown as in Fig.3.6. As the area between each curve and the line \( F(\theta) = 1 \) is unity, the two shaded parts have equal area, i.e. \( A_1 = A_2 \). The segregation, which is the departure from perfect mixing is quantitatively equivalent to the area difference between the two curves up to the intersecting point.

3.1.8. Direct application

(i)

One of the many applications of the residence time distribution (RTD) function is to get an insight into the first order reaction taking place in a reactor. It has been shown by Cooper & Jeffreys (C 11) that the conversion expression for such reaction is the same for both complete segregation and maximum mixedness. After time \( t \) the overall fractional conversion, \( f \) from the system is:

\[ f = 1 - \int_0^t e^{-kt} dF(t) \quad (3.32) \]

where \( k \) is the first order specific reaction rate.

Hence the fractional conversion can be deduced from the area under a plot of \( e^{-kt} \) against \( F(t) \).

3.1.9. Sater correction

Another point worthy of note is the long tail of the skewed \( C(t) \) curve. This tail was represented by the equation.
Fig. 3.6 Segregation

\[ F(\theta) = 1 - e^{-\theta} \]
as given by Sater (3.17) and is known as Sater correction:

\[ C(t) = Ae^{-qt} \]  \hspace{1cm} (3.33)

where A and q are constants.

The above equation gives a straight line with \( \log C(t) \) vs. t. When the deviation from the linearity starts to occur at a time \( t_n \), then for \( t < t_n \),

\[
\int_0^{t_n} C(t) \, dt \simeq \Delta t \sum_{i=0}^{t_n} C_i
\]  \hspace{1cm} (3.34)

\[
\int_0^{t_n} C(t) \, dt \simeq \Delta t \sum_{i=0}^{t_n} C_i \cdot t_i
\]  \hspace{1cm} (3.35)

\[
\int_0^{t_n} C(t) \, t^2 \, dt \simeq \Delta t \sum_{i=0}^{t_n} C_i \cdot t_i^2
\]  \hspace{1cm} (3.36)

and for \( t > t_n \),

\[
\int_{t_n}^{\infty} C(t) \, dt = \left[ -Ae^{-qt} \right]_{t_n}^{\infty} = \frac{Ae^{-qt_n}}{q}
\]  \hspace{1cm} (3.37)

\[
\int_{t_n}^{\infty} C(t) \, t \, dt = \left[ -Ae^{-qt} (qt + 1) \right]_{t_n}^{\infty} = \frac{Ae^{-qt_n} (qt_n + 1)}{q^2}
\]  \hspace{1cm} (3.38)

\[
\int_{t_n}^{\infty} C(t) \, t^2 \, dt = \left[ -Ae^{-qt} \left( q^2 t^2 + 2qt + 2 \right) \right]_{t_n}^{\infty}
\]

\[
= \frac{Ae^{-qt_n}}{q^3} \left( q^2 t_n^2 + 2qt_n + 2 \right)
\]  \hspace{1cm} (3.39)

As stated in equations (3.4) and (3.5)

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

i.e. with Sater Correction:

\[ \Delta t \sum_{i=0}^{t_n} \frac{C_i t_i}{q} + \frac{Ae^{-q t_n}}{q} (q t_n + 1) \]

\[ \mu = \frac{\Delta t \sum_{i=0}^{t_n} \frac{C_i}{q}}{\Delta t \sum_{i=0}^{t_n} \frac{C_i}{q} + \frac{Ae^{-q t_n}}{q}} \]  (3.40)

\[ \sigma^2 = \frac{\Delta t \sum_{i=0}^{t_n} \frac{C_i t_i^2}{q^3} + \frac{Ae^{-q t_n}}{q} (q^2 t_n^2 + 2q t_n + 1)}{\Delta t \sum_{i=0}^{t_n} \frac{C_i}{q} + \frac{Ae^{-q t_n}}{q}} - \mu^2 \]  (3.41)

### 3.1.10. Two-phase flow

With two-phase flow, the Peclet Number is slightly modified, i.e. (S 18)

\[ N_{Pei} = \frac{u_i I_i}{\phi_i E_i} \]  (3.42)

where \( \phi_i \) is the volume fraction of the phase i under consideration.

### 3.2 Equilibrium Study

For any extraction process with no chemical reaction, the components involved may be considered to form a stable system and the distribution of the solute between the phases exists at equilibrium. In this case, the Gibbs rule may be
applied:
\[ \varnothing + F = N + 2 \]  \hspace{1cm} (3.43)

where \( \varnothing \) is the number of the phases,
\( F \) is the degree of the freedom, and
\( N \) is the number of the constituent components.

From the above reasoning, it is apparent that at equilibrium the concentrations in the two phases are not equal, but rather the chemical potential of the solute in both phases is the same. In fact, it is the equality of the chemical potentials, not concentrations, which causes the net flow of solute to stop. Hence, it is no surprise to see that the equilibrium is defined in terms of the thermodynamic property of the system. For a ternary liquid system having two phases at equilibrium at any constant temperature and pressure, the activity of the solute \( \overline{a}_1 \) (component 1) in the component 2 is given as follows:

\[ \overline{a}_1 = \varphi_{12} \overline{x}_1 \]

where \( \varphi \) is the activity coefficient, and
\( \overline{x}_1 \) is the mole fraction of the component 1 in the component 2.

Similarly, the activity of the component 1 in the component 3 is:

\[ a_1 = \varphi_{13} x_1 \]

At equilibrium:

\[ \overline{a}_1 = \varphi_{12} \overline{x}_1 = a_1 = \varphi_{13} x_1 \]
The distribution ratio $m$ of the solute in component 2 to that in component 3 is given as:

$$m = \frac{x_1}{x_1} = \frac{\gamma_{13}}{\gamma_{12}} = \frac{c_1}{c_1} = \frac{c_y}{c_x}$$

The above relation is always expressed in terms of the equilibrium value, i.e.

$$c^*_y = m c_x \quad (3.44)$$

To account for the non-linearity of the equilibrium curve, the equation takes the more general form:

$$c^*_y = m c^n_x \quad (3.45)$$

i.e. $\log(c^*_y) = \log(m) + n \log(c_x) \quad (3.46)$

Applying the technique of the least square fit, two normal equations involving $m$ and $n$ can be obtained, i.e. for $N$ sets of data:

$$N \log(m) + n \sum \log(c_x) = \sum \log(c_y) \quad (3.47)$$

$$\left[ \log(m) \sum \log(c_y) + n \sum \left( \log(c_x) \right)^2 \right] = \sum \left( \log(m) \log(c_x) \right) \quad (3.48)$$

By solving the above two equations, the values of $m$ and $n$ can be found with the aid of the experimental data.

As a point of interest, the values of $\gamma_{13}$ and $\gamma_{12}$ can be estimated by the Redlich-Kister equation or other suitable equation for excess Gibbs energy. The excess Gibbs energy is related to the minimum Gibbs energy which
is in turn related to the stability of the system.

3.3 Hold up measurement

The hold up measurement is important in the sense that it gives an insight of the amount of the dispersed phase inside the contactor. It also serves as an indirect indication of the flooding point of the equipment under a particular flow condition.

Consider the flow arrangement of Fig. 3.7. Utilizing the Bernoulli equation, the difference in energy between the point 2 and point 1 is given by the friction factor, $f_c$, i.e. for the continuous phase:

$$\frac{U_1^2}{2g} + \frac{P_1}{\rho_c} + h_1 = \frac{U_2^2}{2g} + \frac{P_2}{\rho_c} + h_2 + f_c \quad (3.49)$$

Assuming equal velocity heads and negligible wall friction in the column, then:

$$P_1 - P_2 = (h_2 - h_1) \rho_c = \Delta h \rho_c \quad (3.50)$$

Similar argument holds for the dispersed phase, i.e.

$$P_1 - P_2 = (h_2 - h_1) \rho_d = \Delta h \rho_d \quad (3.51)$$

Multiplying eq. (3.50) by $(1 - \phi)$ and eq. (3.51) by $\phi$, i.e.

$$P_1 - P_1 \phi - P_2 + P_2 \phi = \Delta h \rho_c \phi - \Delta h \rho_c (1 - \phi), \quad \text{and} \quad (3.52)$$

$$P_1 \phi - P_2 \phi = \Delta h \rho_d \phi \quad (3.53)$$
Fig. 3.7 Hold up measurement
Adding eq. (3.52) to eq. (3.53):

\[ P_1 - P_2 = \Delta h \left[ \rho_c (1 - \phi) + \rho_d \phi \right] \]

\[ = \Delta h \rho_c + \phi (\rho_d - \rho_c) \Delta h \]

i.e. \( \phi = \frac{(P_1 - P_2) - \Delta h \rho_c}{(\rho_d - \rho_c) \Delta h} \) \hspace{1cm} (3.54)

If the pressure difference between point 1 and point 2 is to be measured with a manometer, then:

\[ P_1 - P_2 = \left[ \Delta h \rho_c + (\rho_m - \rho_c) h_m \right] \frac{\varepsilon}{\varepsilon_c} \]

\[ = \left[ \Delta h \rho_c + (\rho_m - \rho_c) h_m \right] \] \hspace{1cm} (3.55)

as \( \varepsilon/\varepsilon_c = 1 \)

and \( \rho_m \) is the density of the liquid used in the manometer.

Substituting eq. (3.55) into eq. (3.54), i.e.

\[ \phi = \frac{h_m (\rho_m - \rho_c)}{(\rho_d - \rho_c) \Delta h} \] \hspace{1cm} (3.56)

To account for the nature of the fluids used in the system, eq. (3.56) should be written as:

\[ \phi = \frac{h_m (\rho_m - \rho_c)}{|\rho_d - \rho_c| \Delta h} \] \hspace{1cm} (3.57)
If the liquid of the dispersed phase is used in the manometer, then \( \rho_d = \rho_m \). Hence the above equation can be simplified even further:

\[
\mathcal{G} = \frac{h_m}{\Delta h}
\]  

(3.58)

3.4 Classical Mass Transfer Theories

3.4.1. Film theory

This theory is based on the concept used for convective heat transfer and it is mainly applied to the case of a fluid flowing turbulently past a solid surface with the mass transfer occurring from the surface to the fluid. As the fluid velocity is zero at the surface itself, there must be a viscous layer or film in the fluid adjacent to the surface. Hence this film theory imagines the concentration difference between the bulk of the liquid and the solid surface, i.e. the entire concentration difference is held to be due to molecular diffusion within an "effective" film. It was recognised that if this picture were to be valid, the effective film would have to be very thin so that the accumulation of the solute within the film would be small compared with the amount passing through it.

3.4.2. Two film theory (Two resistance theory)

This theory assumes that the turbulence dies out, i.e. that there is no resistance at the interface separating the phases. Instead, the diffusional resistance is assumed to lie within two hypothetical laminar layers, one on each side of the interface, which in turn resides in the bulk of fluids. As a result, equilibrium is assumed to exist at the interface and the interfacial concentration of the two
phases is given by the system's equilibrium-distribution curve.

The rate of mass transfer per unit area is based on the following equation:

\[ N_A = k_x(C_x - C_{xi}) = k_y(C_{yi} - C_y) \]  \hspace{1cm} (3.59)

where \( C_{xi} \) and \( C_{yi} \) are the solute concentrations in equilibrium across the two phases 1 and 2 at the interface, and

\[ C_{yi} = f(C_{xi}) \]

The interfacial concentrations \( C_{xi} \) and \( C_{yi} \) are normally very difficult to determine, hence the film coefficients \( k_x \) and \( k_y \) are replaced by the overall mass transfer coefficients \( K_{ox} \) and \( K_{oy} \), i.e.

\[ N_A = K_{ox}(C_x - C_x^*) = K_{oy}(C_y^* - C_y) \]  \hspace{1cm} (3.60)

where \( C_x^* \) represents the composition of the solute in phase 1 in equilibrium with the solute concentration in the bulk of phase 2, and

\( C_y^* \) represents the composition of the solute in phase 2 in equilibrium with the solute concentration in the bulk of phase 1.

The film coefficient \( k \) is given by:

\[ k = \frac{D}{s} \]

where \( s \) is the film thickness, and
D is the diffusion coefficient of component species.

The above theory is quite useful in explaining the majority of mass-transport processes. The main criticism bears on the concept of the equilibrium of the interfacial concentrations. Also, the time taken to establish the concentration gradient has to be small compared with the time of transfer.

3.4.3. Penetration theory

The question of the interfacial resistance having been investigated by Higbie(12), the penetration theory was put forward. This theory assumes that the eddies in the fluid bring an element of fluid to the interface where it is in contact with the second phase for a period of time and a state of equilibrium obtains, after which the surface element is mixed with the bulk again.

By solving a unsteady state partial differential equation, the average transfer rate is:

\[ N_A = (C^* - C_0) \frac{1}{N} 4D(\pi t_e) \]

(3.61)

where \( C_0 \) is the initial concentration, and \( t_e \) is the time of exposure, i.e.

\[ k = 2 \sqrt{\frac{D}{\pi t^*}} \]

(3.62)

where \( t^* \) is the whole life of an eddy element.

The main assumption of this theory is that each element resides for the same period \( t_e \) in the surface.
3.4.4. Random surface renewal theory

Danckwerts (D 3) proposed this theory, suggesting that the time of exposure of each surface element would not be identical, but rather that a random distribution of ages exists. Hence the following relationship can be obtained:

$$k = \sqrt{Dr}$$  \hspace{1cm} (3.63)

where $r$ is the rate of production of fresh surface per unit total area of surface, or simply rate of surface renewal, and $1/r$ is the average life of surface elements.

One of the disadvantages of this theory is that the numerical value of $r$ cannot easily be estimated.

3.4.5. The two-film - penetration theory

This theory was proposed by Toor & Marchello (T 4) and it is a combination of the Two-film theory and Penetration theory. The mass transfer is treated as an unsteady state process. For short times of exposure, the process is identical to that postulated in the penetration theory while for long periods of exposure, the condition is similar to those of the two-film theory.

The time of exposure of the surface elements can be assumed to follow the penetration theory or a random age distribution as proposed by Danckwerts. The drawback of this theory is that the mass transfer rate equation involves both the film thickness and the exposure time, neither of which can readily be known.

3.4.6. Film-surface renewal theory

Dobbins (D 4) proposed this theory with the replacement
of one of the boundary conditions of the penetration theory, and with Danckwerts' rate of renewal of the surface elements, he obtained:

\[ k = \frac{D r \coth \sqrt{r z_b^2 / D}}{n} \]  

(3.64)

where \( z_b \) is the depth of penetration and has a finite value.

### 3.4.7. Further theories

Some more theories were developed by Kishinevskij(K 8) and Bakowski(B 5). In Kishinevskij's model, the molecular diffusion is assumed to play no part, while Bakowski's model concerns the mass transfer from a turbulent gas stream to a liquid surface and he proposed that the transfer rate will be a function of the number of molecules of the diffusing component in the gas layer in contact with the liquid.

### 3.5 Mass transfer theories related to drops

#### 3.5.1. Mass transfer coefficients

Liquid drops under certain conditions tend to oscillate when they rise or fall in another liquid medium and various correlations for oscillating liquid drops have been obtained. For an oblate-prolate oscillated droplet, the continuous phase coefficient was obtained by Garner & Skelland(G 11), which was derived from the penetration theory of Higbie(H 12), i.e.

\[ k_c = 2 \frac{D c}{n t_e} \]  

(3.65)
where \( t_e \) is the time of contact for solute transfer, and 

\( D_e \) is the diffusivity of the solute in the continuous phase.

The dispersed phase mass transfer coefficient was formulated by Angelo (A4) & Lightfoot in their surface-stretch theory, with special reference to large drops in liquid-liquid extraction system.

\[
\frac{k_d}{\pi} = 4D_d w \left( 1 + \frac{8}{3} + \frac{36}{\delta^2} \right)
\]  

(3.66)

where \( w \) is the oscillation frequency, i.e.

\[
w = \frac{1}{2\pi} \sqrt{\frac{192 \sigma e c b}{d^3 (3\rho_d + 2\rho_c)}}
\]

\[
b = 1.052 \quad d_p^{0.225}
\]

where \( \delta \) is a dimensionless amplitude factor characteristic of the oscillating system, and may be taken as 0.2, and \( \sigma \) is the interfacial tension.

This surface-stretch theory is an extension of the penetration-surface renewal concepts and it is being applied to the situations where the interfacial surface through which mass transfer occurs changes periodically with time.

On the other hand, the dispersed phase mass transfer coefficient during drop formation and coalescence was
evaluated under various considerations by Licht & Pansing (1999):

\[ k_d = 2 \sqrt{\frac{D_d}{\pi} t_e} = 2 \sqrt{\frac{D_d U_t}{d_p}} \]  \hspace{1cm} (3.67)

for complete internal circulation and the contact time \( t_e \) as the time taken by the drop to travel a distance equal to its diameter under its terminal velocity, i.e. \( d_p/U_t \).

If the drop has a central stagnant region and partial circulation at the periphery, then:

\[ k_d = 2 \sqrt{\frac{D_d}{\pi} \left( \frac{F_c^2}{t_e} \right)} \]  \hspace{1cm} (3.68)

where \( F_c^2 \) is a correction factor which is the ratio of the vertical velocity of the interface, \( U_i \) to that of the bulk of the continuous phase, \( U_c \), both taken with respect to the drop centre.

Sivaraman et al. (1999) proposed a generalised equation to take into consideration the turbulence caused by circulation within the drop, i.e.

\[ k_d = f_d \sqrt{\frac{D_d U_t}{d_p}} \]  \hspace{1cm} (3.69)

where \( f_d \) is a correction factor for the effect of circulation within the drop and it varies with the droplet's Reynolds Number. For complete internal circulation, it is equal to \( 2/\sqrt{\pi} \).
A similar relationship holds for the continuous phase coefficient, i.e.

\[ k_c = f_c D_c U_t / d_p \]  \hspace{1cm} (3.70)

Apart from Licht & Pansing's formula (L9), Coulson & Skinner (C12) proposed two equations which expressed the overall coefficients \( K_{oc} \) and \( K_{od} \) in terms of the time of formation or collapse, i.e.

\[ K_{oc} = 0.89 / t_f^{0.7} \]  \hspace{1cm} (3.71)

\[ K_{od} = 0.96 / t_f^{0.7} \]  \hspace{1cm} (3.72)

On the other hand, Treybal (T1) produced the alternative equation for \( K_{od} \) during formation or coalescence, i.e.

\[ K_{od} = \frac{0.805 \left( \frac{D_c}{m} \right)^{1/2}}{t_f} \]  \hspace{1cm} (3.73)

where \( m \) is the distribution coefficient, and \( t_f \) is the time of drop formation.

Contradictory results were reported by various workers, and these were largely due to the "interfacial turbulence" which was induced by local differences in concentration of the solute in the interface.

### 3.5.2. Various correlations for mass transfer coefficients

Correlations for the estimation of the mass transfer coefficients under different conditions have been proposed by various workers. These correlations are expressed in
terms of the physical properties of the fluid system and serve as an alternative means of predicting the value of the coefficients.

Continuous Phase Coefficients

Solid spheres and rigid liquid drop

\[(N_{Sh})_c = 2 + 0.95(N_{Re})_c^{1/2}(N_{Sc})_c^{1/4} \quad \text{Garner & Suckling (G 12)}\]

\[(N_{Sh})_c = 0.98(N_{Pe})_c^{1/3} \quad \text{Ward et al. (W 2)}\]

for \(N_{Pe} > 1000\) and \(N_{Pe} = (N_{Re})_c(N_{Sc})_c\)

Circulating liquid drops

\[(N_{Sh})_c = 0.61 \left(\frac{\frac{\mu_d}{\mu_c + \mu_d}}{(N_{Pe})^{1/2}}\right) \quad \text{Ward et al. (W 2)}\]

\[\text{for } N_{Pe} > 2.8 \left(\frac{\mu_d + \mu_c}{\mu_d}\right)^{\frac{12\mu_c + 9\mu_d}{\mu_d}}\]

\[\frac{k_c}{U_t} \left(\frac{\rho_c}{\rho_c D_c}\right)^{0.58} = 1.3 \left(\frac{d_p U_t \rho_c}{\mu_c}\right)^{-1/2} \quad \text{Garner et al. (G 13)}\]

\[\frac{k_c d_p}{D_c} = 5.52 \left(\frac{\mu_c + \mu_d}{2\mu_c + 3\mu_d}\right)^{3.47} \left(\frac{d_p \rho_c}{\mu_c^2}\right)^{0.056} (N_{Pe})^{0.5} \quad \text{Elzinga & Banchero (E 5)}\]

\[(N_{Sh})_c = 50 + 0.0085(N_{Re})_c \left(\frac{\frac{\rho_c}{\rho_c D_c}}{(\frac{\rho_c}{\rho_c D_c})^{0.7}}\right) \quad \text{Garner & Tayeban (G 8)}\]
Oscillating drops

\[
(N_{Sh})_c = 2 + 0.084 \left[ (N_{Re})_c^{0.484} (N_{Sc})_c^{0.339} \left( \frac{d_p g^1/3}{D_c^{2/3}} \right)^{0.072} \right]^{1.5}
\]

Hughmark (H 14)

Circulating & oscillating drops (Penetration theory)

\[
k_c = 2 \sqrt{\frac{D_c U_t}{\pi d_p}}
\]

Swarm of drops

\[
(N_{Sh})_d = 1.26 \left[ \frac{1 - \phi^{5/3}}{Y - \phi^{1/3} W} \right]^{1/3} \left( \frac{N_{Pe}}{N_{Re}} \right)^{1/3} + \frac{2}{1 - \phi^{1/3}}
\]

for \( \beta = 0 \) ------- impure system.

\[
(N_{Sh})_d = 0.923 \left[ \frac{B(1 - \phi^{5/3})}{Y - \phi^{1/3} W} \right]^{1/2} \left( \frac{N_{Pe}}{N_{Re}} \right)^{1/2} + \frac{2}{1 - \phi^{1/3}}
\]

for large \( \beta \) ------- pure system.

and \( Y = 2 + 2\beta + \phi^{5/3}(3 - 2\beta) \)

\( W = 3 + 2\beta + 2\phi^{5/3}(1 - \beta) \)

\( \beta \) is a parameter which accounts for the effects of the impurities.

Waslo & Gal-Or (W 3)

\[
\frac{k_c}{U_s(1 - \phi)} \left( \frac{\mu_c}{\rho_c D_c} \right)^{0.58} = 0.725 \left( \frac{d_p U_s \rho_c}{\mu_c} \right)^{-0.43}
\]

Ruby-Elgin (R 7)
Swarm of gas bubbles

\[ k_c (N_{Sc})^{2/3} = 0.31 \left( \frac{\Delta \rho \mu_c \sigma}{\rho_c^2} \right)^{1/3} \]

Calderbank & Moo-Young (C 7)

\[ k_c (N_{Sc})^{1/2} = 0.42 \left( \frac{\Delta \rho \mu_c \sigma}{\rho_c^2} \right)^{1/3} \]

Calderbank & Moo-Young (C 7)

for large gas bubbles which do not behave like rigid spheres.

Dispersed Phase Coefficients

Rigid spheres with no circulation

\[ k_d = 2 \pi^2 \frac{D_d}{3 d_p} \quad \text{Treybal (T 1)} \]

Circulation

\[ k_d = 17.9 \frac{D_d}{d_p} \quad \text{Kronig-Brink (K 10)} \]

\[ k_d = 0.00375 \frac{U_t}{(\mu_c + \mu_d)} \quad \text{Handlos-Baron (H 13)} \]

Oscillating drops

\[ k_d = \frac{4D_d w(1 + \delta + 3\delta^2)}{\pi} \quad \text{Angelo et al. (A 4)} \]

Circulating & oscillating drops (Penetration theory)

\[ k_d = 2 \frac{D_d U_t}{(\pi d_p)} \]
A solute material balance over the whole column for the flow system (Fig. 3.8) with two immiscible phases under steady state will be:

\[ Q_d C_{xt} + Q_c C_{yb} = Q_d C_{xb} + Q_c C_{yt} \]  \hspace{1cm} (3.74)

\[ \Rightarrow (C_{xt} - C_{xb}) = \frac{Q_c (C_{yt} - C_{yb})}{Q_d} \]
Put \( \frac{Q_c}{Q_d} = R \), the flow ratio, then:

\[
(C_{xt} - C_{xb}) = R(C_{yt} - C_{yb})
\]  \hspace{1cm} (3.75)

i.e. \( C_{xt} = RC_{yt} + C_{xb} \)

with \( C_{yb} = 0 \), for solute free continuous phase.

Hence a general expression for the \( n^{th} \) section is

\[
C_{xn} = RC_{yn} + C_{xb}
\]  \hspace{1cm} (3.76)

3.6.2. Number of transfer units and height of transfer units.

The basic mass transfer equation can be derived from the two-film (two resistance) theory, i.e.

\[
N = K_d(x - x^*) = K_d(C_x - C_x^*)
\]  \hspace{1cm} (3.77)

\[
N = K_c(y^* - y) = K_c(C_y^* - C_y)
\]  \hspace{1cm} (3.78)

If this equation is applied over a differential section \( dH \) of the column, then:

\[
dN = (Ka)_d(C_x - C_x^*) \, dH, \text{ and}
\]

\[
dN = (Ka)_c(C_y^* - C_y) \, dH
\]

A material balance of solute in the elemental section gives:

\[
S \, dN = Q \, dC_x
\]  \hspace{1cm} (3.79)
\[ i.e. \quad \frac{Q \, dC_x}{S} = (K_a)_d(C_x^* - C_x^0) \, dH \quad (3.80) \]

and similarly:

\[ \frac{Q \, dC_y}{S} = (K_a)_c(C_y^* - C_y^0) \, dH \quad (3.81) \]

where \((K_a)_d\) and \((K_a)_c\) are overall mass transfer coefficients, based on the superficial area per unit volume of the column and \(S\) is the cross-sectional area of the column. If \(Q\) is expressed in \(\text{ft}^3/\text{hr}\), and the concentration \(C\) is expressed in \(\text{lb mole}/\text{ft}^3\), say, then \(N\) is expressed in \(\text{lb mole}/\text{hr ft}^2\). Hence \((K_a)\) is expressed in \(\text{lb mole}/\text{hr ft}^3(\text{lb mole}/\text{ft}^3)\) and \(a\) is the interfacial area in \(\text{ft}^2/\text{ft}^3\).

The mass transfer rate expression can be given by an alternative form:

\[ Q \, dx = (K_a)_d(x - x^*) \, dH \quad (3.82) \]

If \(Q\) is in \(\text{lb mole}/\text{hr ft}^2\), say, then \(x\) is in mole fraction and \(K\) is in \(\text{lb mole}/\text{hr ft}^2(\text{mole fraction})\).

The Number of Transfer Unit , \(NTU\) is then:

\[ NTU_d = \frac{(K_a)_dSH}{Q_d} \int_{C_x}^{C_{x^*}} \frac{dC_x}{C_x^* - C_x} \quad (3.83) \]
The Height of Transfer Unit, HTU is then:

\[
\text{HTU}_c = \frac{H}{\text{NTU}_c} = \frac{HQ_c}{(K_a)_c SH} = \frac{Q_c}{(K_a)_c S} = \frac{U_c}{(K_a)_c}
\]  

(3.85)

and similarly:

\[
\text{HTU}_d = \frac{U_d}{(K_a)_d}
\]  

(3.86)

### 3.6.3. Different concepts of NTU

Because of the different definitions of the effective driving forces, different concepts of NTU can therefore be derived.

#### 3.6.3.1. "Linear" plug flow NTU

This simple model assumes a linear concentration profile and hence no axial mixing. This gives rise to a log-mean driving force and thus:

\[
\text{NTU}_{cl} = \int \frac{C_y}{C^*_y - C_y} dy = \frac{C_{yt} - C_{yb}}{(C^*_y - C_y)_{LM}}
\]  

(3.87)

where \((C^*_y - C_y)_{LM} = (C^*_y - C_{yt}) - (C^*_y - C_{yb})\)

\[
\ln \left( \frac{C^*_y - C_{yt}}{C^*_y - C_{yb}} \right)
\]
and similarly:

$$\text{NTU}_{cl} = \int_{C_{xb}}^{C_{xt}} \frac{dC_x}{C_x - C_x^*} = \frac{C_{xt} - C_{xb}}{(C_x - C_x^*)_{LM}} \quad (3.88)$$

and $(C_x - C_x^*)_{LM} = \frac{(C_{xt} - C_{xt}^*) - (C_{xb} - C_{xb}^*)}{\ln \left( \frac{C_{xt} - C_{xt}^*}{C_{xb} - C_{xb}^*} \right)}$

The linear plug flow HTU is then given by:

$$\text{HTU}_{cl} = H/\text{NTU}_{cl} \quad (3.89)$$

### 3.6.3.2. "Non-linear" Plug Flow NTU (measured NTU and apparent measured NTU)

The actual concentrations of the phases at different sampling points were obtained by Miyauchi & Vermeulen (M 9) in their work and these real local solute concentrations were used to promote the concept of the measured value of NTU, i.e.

$$\text{NTU}_{cm} = \int_{C_{yb}}^{C_{yt}} \frac{dC_y}{C_y^* - C_y} \quad (3.90)$$

and $\text{NTU}_{dm} = \int_{C_{xb}}^{C_{xt}} \frac{dC_x}{C_x^* - C_x}$

This idea was further expanded by Thomas (T 7) and Thomas & Chiu (T 8). Instead of sampling both phases as done by Miyauchi, Thomas sampled the continuous phase only and the
dispersed phase concentration was then obtained by a sectional mass balance. The NTU values were then known as apparent measured NTU according to Thomas, i.e.

\[ \text{NTU}_{CA} = \int_{C_{y0}}^{C_{y*}} \frac{dC_y}{C_y - C_y} \]

and \( \text{NTU}_{DA} = \int_{C_{x0}}^{C_{x*}} \frac{dC_x}{C_x - C_x} \) \hspace{1cm} (3.93)

Miyauuchi's measured NTU was meant to be close to the actual situation with axial mixing and Thomas suggested that the apparent measured NTU was even closer. His argument was based on the fact that experimental error could occur in the sampling of the \( C_x \), as mass transfer would take place during drop sampling.

The above non-linear NTU values are all evaluated using the method of graphical integration. One thing to be borne in mind is that these values are still not the true NTU.

3.6.4. The One Dimensional Diffusion Model

3.6.4.1. True number of transfer units

The deviation from ideal flow due to axial mixing is represented by a flux term and the true performance, hence the true NTU is then evaluated by using Sleicher's diffusion model (S16) as shown in Fig. 3.9. This concept was further expanded by Rod (R9).

If a mass balance of solute in the continuous phase is taken across the element \( dh \), then:

\[ \text{U}_c \frac{\partial C_y}{\partial x} - \frac{dC_y}{dh} = - (\text{Ka})_c (C_{y*} - C_y) = \text{U}_c (C_y + \frac{dC_y}{dh} - E_c \frac{dC_y}{dh}) \]
Fig. 3.9 The One-Dimensional Diffusion Model
i.e. \[ E_c \frac{d^2 C_y}{dh^2} + U_c \frac{dC_y}{dh} + (Ka)_c (C^*_y - C_y) = 0 \] (3.94)

Similar argument holds for the solute transfer in the dispersed phase, viz.

\[ E_d \frac{d^2 C_x}{dh^2} - U_d \frac{dC_x}{dh} - (Ka)_d (C_x - C^*_x) = 0 \] (3.95)

Combining these two equations:

\[ E_c \frac{d^2 C_y}{dh^2} + E_d \frac{d^2 C_x}{dh^2} + U_c \frac{dC_y}{dh} - U_d \frac{dC_x}{dh} = 0 \] (3.96)

i.e. \[ E_c \left[ \frac{d^2 C_y}{dh^2} \right] + E_d \left[ \frac{d^2 C_x}{dh^2} \right] + U_c \frac{dC_y}{dh} - U_d \frac{dC_x}{dh} = 0 \] (3.97)

If a further mass balance is taken over the top section and also defining:

\[ X = C_x - \frac{E_d}{U_d} \frac{dC_x}{dh} \] (3.98)

\[ Y = C_y + \frac{E_c}{U_c} \frac{dC_y}{dh} \] (3.99)

\[ U_d (C_x + dC_x) + U_c (C_y + dC_y) = U_d (C_x + dC_x) + U_c (C_y + dC_y) \]

\[ -E_d \frac{d(C_x + dC_x)}{dh} - E_c \frac{d(C_y + dC_y)}{dh} \]

(3.100)
i.e. \[ U_d C_{xt} - U_c C_{yt} = U_d \left[ C_x - \frac{E_d}{U_d} \frac{dc_x}{dh} \right] - U_c \left[ C_y + \frac{E_d}{U_c} \frac{dc_y}{dh} \right] \]
\[ - \left[ \frac{E_d}{dh} \frac{d^2 c_y}{dh^2} dh + E_d \frac{d^2 c_x}{dh^2} dh + U_c \frac{dc_y}{dh} - U_d \frac{dc_x}{dh} \right] \]

(3.101)

Substituting \( X, Y \) and equation (3.97) into equation (3.101), then:

\[ U_d C_{xt} - U_c C_{yt} = U_d(X) - U_c(Y) \]  

(3.102)

The boundary conditions are:

\[ h = 0, \quad \frac{-dc_x}{dh} = 0, \quad X = C_{xb}, \quad Y = C_{yb}. \]

\[ h = H, \quad \frac{-dc_y}{dh} = 0, \quad Y = C_{xt}, \quad Y = C_{yt}. \]

If the dimensionless column height, \( Z \) is given by

\[ Z = \frac{h}{H}, \quad \text{then} \]

\[ dZ = \frac{1}{H} \quad dh \]

i.e. \( H \quad dZ = \quad dh \)

(3.103)

Hence:

\[ X = C_x - \frac{E_d}{U_d} \frac{dc_x}{H \quad dZ} \]

(3.104)

i.e. \[ \frac{E_d}{H \quad U_d} \int_{C_{xb}}^{C_{xt}} \quad dc_x = \int_{0}^{1} (C_x - X) \quad dZ \]

i.e. \[ \frac{E_d}{H \quad U_d} \int_{C_{xb}}^{C_{xt}} \quad dc_x = \int_{0}^{1} (C_x - X) \quad dZ \]
\[
\Rightarrow \frac{H U d}{E d} = \frac{(C_{xt} - C_{xb})}{\int_0^l (c_x - x) \, dz}
\]  
(3.105)

Similarly:

\[
Y = C_y + \frac{E_c}{U_c} \frac{dC_y}{H \, dZ}
\]
(3.106)

i.e. \( \frac{E_c}{H \, U_c} \frac{dC_y}{dZ} = (Y - C_y) \, dZ \)

\[
i.e. \frac{E_c}{H \, U_c} \int_{C_y}^{C_y_t} dC_y = (Y - C_y) \, dZ
\]
(3.107)

Equation (3.95) can be rewritten as follows:

\[
\frac{E_d}{U_d} \frac{d^2 c_x}{dh^2} - \frac{dC_x}{dh} + \frac{(Ka)}{U_d} \frac{d}{dh} (C_x - C_x^*) = 0
\]
(3.108)

Differentiating eq. (3.98) with respect to h:

\[
\frac{dX}{dh} = \frac{dC_x}{dh} - \frac{E_d}{U_d} \frac{d^2 C_x}{dh^2}
\]
(3.109)

\[
\Rightarrow \frac{dX}{dh} = \frac{(Ka)}{U_d} \frac{d}{dh} (C_x - C_x^*)
\]

i.e. \( \frac{dX}{dh} = \frac{(Ka)}{U_d} (C_x - C_x^*) \, H \, dZ \)
\[ \int_{x}^{c} \frac{dX}{x} = \frac{H(K_a)}{U_d} \int_{c}^{l} (c_x - c_x^*) \, dz \]

\[ \frac{H(K_a)}{U_d} = \frac{c_{xt} - c_{xb}}{\int_{c}^{l} (c_x - c_x^*) \, dz} \quad (3.110) \]

But the number of transfer units is defined by equation (3.83):

\[ NTU_d = \frac{(K_a)_{d}SH}{Q_d} = \frac{(K_a)_{d}H}{U_d} \]

Hence the true NTU is given by:

\[ NTU_{dT} = \frac{c_{xt} - c_{xb}}{\int_{c}^{l} (c_x - c_x^*) \, dz} \quad (3.111) \]

In a similar manner, the \( NTU_{cT} \) for the continuous phase is:

\[ NTU_{cT} = \frac{c_{yt} - c_{yb}}{\int_{0}^{l} (c_y - c_y^*) \, dz} \quad (3.112) \]

where \( NTU_c = \frac{(K_a)_{c}SH}{Q_c} = \frac{(K_a)_{c}H}{U_c} \)

3.6.4.2. Relationship between the true and measured and apparent measured NTU

If the equation (3.94) is rearranged by multiplying
through by \( dh/[U_c(C^*_y - C_y)] \), then:

\[
E_c \frac{d^2 C_y}{dh^2} \left( \frac{dh}{U_c(C^*_y - C_y)} \right) + U_c \frac{dC_y}{dh} \left( \frac{dh}{U_c(C^*_y - C_y)} \right) - \frac{(Ka)_c}{U_c} \frac{dh}{dh} = 0
\]

\[
E_c \frac{1}{U_c(C^*_y - C_y)} \frac{d^2 C_y}{dh^2} \int_{C_y}^{C_y} + \frac{dC_y}{C^*_y - C_y} \int_{C_y}^{H} = \frac{(Ka)_c}{U_c} \int_{C_y}^{H} dh
\]

i.e.

\[
\frac{(Ka)_c}{U_c} \int_{C_y}^{H} = \int_{C_y}^{H} \frac{dC_y}{C^*_y - C_y} + \frac{E_c}{U_c} \int_{C_y}^{H} \frac{1}{C^*_y - C_y} \frac{d^2 C_y}{dh^2} dh
\]

Since here \((Ka)_c\) is a "true" \((Ka)_c\)

\[
\left[ \frac{(Ka)_c}{U_c} \right] = NTU_{cT}, \text{ or }
\]

\[
\frac{1}{U_c} \int_{C_y}^{H} (Ka)_c \frac{dh}{dh} = NTU_{cT} \text{ for variable } (Ka)_c
\]

and

\[
\int_{C_y}^{C_y} \frac{dC_y}{C^*_y - C_y} = NTU_{cm}, \text{ according to Miyauchi(M 9)}
\]

\[
\int_{C_y}^{C_y} \frac{dC_y}{C^*_y - C_y} = NTU_{cA}, \text{ according to Thomas(T 7, T 8)}
\]
Hence \( NTU_{CT} = NTU_{cm} + \frac{E_c}{U_c} \int_{0}^{H} \frac{1}{C^*_{y} - C_y} \frac{d^2C_y}{dh^2} \) dh (3.115)

or \( NTU_{CT} = NTU_{cA} + \frac{E_c}{U_c} \int_{0}^{H} \frac{1}{C^*_{y} - C_y} \frac{d^2C_y}{dh^2} \) dh (3.116)

and similarly:

\( NTU_{dT} = NTU_{dm} + \frac{E_d}{U_d} \int_{0}^{H} \frac{1}{C^*_{x} - C_x} \frac{d^2C_x}{dh^2} \) dh (3.117)

or \( NTU_{dT} = NTU_{dA} + \frac{E_d}{U_d} \int_{0}^{H} \frac{1}{C^*_{x} - C_x} \frac{d^2C_x}{dh^2} \) dh (3.118)

According to Miyauchi, the integral term of the right hand side can be treated as a correction factor. This correction factor is a complex quantity and although it does not represent all the effects of the longitudinal mixing, it does give a further contribution to the true NTU due to convection and axial mixing. The other contribution is given by \( NTU_{cm} \) or \( NTU_{cA} \) as stated previously.

Various attempts have been made by workers such as Hartland & Mecklenburgh(H 15), Miyauchi & Vermeulen(M 9), and Smoot & Baab(S 8) to solve the one dimensional diffusion equation(eq. 3.94 and 3.95) under different boundary conditions. The latest attempt was made by Clare(C 13) with the assumption of the linear equilibrium relationship. This
assumption is questionable as the linearity can only be assumed in the very low concentration region.

3.7 Effects of axial mixing

Axial mixing in a contactor creates a false countercurrent flow pattern and hence lowers the mean driving potential for mass transfer. Subsequently, the actual NTU required will be larger as compared to the case of plug flow and any interpretation based on ideal countercurrent pattern will be in error. A typical concentration profile with the effect of axial mixing upon mass transfer is shown in Fig.3.10.

A full analysis has been carried out by Sleicher (S 16) and a graphical presentation of the NTU under axial mixing and plug flow in terms of various parameters was also given.

3.8 End effects

The real concentration profile is different somewhat from the apparent concentration profile because of the end or entrance effects (Fig.3.11). The end effects can be depicted as an abrupt jump in concentration of the phases and be considered as an increase in the column height, i.e. inefficiency. They are shown as fictitious heights $Z'_{c2}$ and $Z'_{d1}$ in Fig.3.11.

One of the earliest studies on this topic was by Geankoplis & Hixon (G 1) and the situations which induce such behaviour have been investigated by Letan & Kehat (L 15). According to Letan and Kehat, the dispersed drops on reaching the main interface may suddenly release their wakes; and these wakes mix with the rich incoming stream of the continuous phase and hence dilute it.
Fig. 3.10 Effect of axial mixing on conc. driving force.
Fig. 3.11 End effects.
3.9 Features of Vulcan's tower

The baffle column developed by Vulcan Co. (V 1) can employ side to side flow or centre to side flow, and it operates on the fact that a major portion of the extraction occurs by diffusion of the solute through the liquid interface occurring between the baffles. This implies that the number of baffles required is determined by the area of contact between each tray and the number of theoretical trays.

Some extra extraction also occurs at the opening of the baffles where the descending heavy liquid and the ascending light liquid cross each other's path. Such remixing process provides a fresh surface for the next extraction stage.

According to Vulcan, the major limiting factor in the design of a baffle type column is the total allowable velocity of the two liquids through the opening. The magnitude of this maximum permissible velocity can be obtained by a calibration chart.

The main theme of the paper published by Vulcan was a set of semi-empirical formulae for the calculation of the size of the tower. Unfortunately, methods for the calculation of the mass transfer rate and interfacial area were not given.

3.10 Droplet interfacial area

The interfacial area, $a$, of a mass transfer contactor is normally given by:

$$ a = \frac{6\theta}{\text{mean diameter}} \quad (3.119) $$

where $\theta$ is the dispersed phase hold up.
The dimension of 'a' in this equation is \( \frac{L^2}{L^3} \) and it is the total contact area per unit volume of the contactor. It is equivalent to the specific interfacial area.

The mean diameter of the above equation can be defined in various ways, depending on the type of the equipment employed in the operation.

One of the mean diameters used is called Sauter mean (or surface mean) diameter and is defined as follows:

\[
\text{Surface mean diameter} = d_{32} = \frac{\sum n d^3}{\sum n d^2} \quad (3.120)
\]

This \( d_{32} \) is proportional to the ratio of the total volume to total surface of the drops.

### 3.11 Droplet behaviour

As stated by Bond & Newton\((B 5)\), the critical radius of the spherical drops moving in viscous media at \( N_{Re} < 1 \) in the streamline region at which the internal circulation takes place is given by:

\[
r = \left( \frac{\sigma}{\Delta \rho g} \right)^{1/2} \quad (3.121)
\]

where \( \sigma \) is the interfacial tension of the system.

It is apparent from the above statement that the drop size and hence the behaviour is related to the interfacial tension in some way.

Further work on this field, 'interfacial phenomena' as it is generally known, has been carried out by Davies & Rideal\((D 8)\) and Sawistowski\((S 13)\). They have investigated
the conditions under which various phenomena occur. These were 'kicking', 'eruptions' and interfacial instabilities, etc.

Inevitably, all these were related to the concentration and interfacial tension of the system.
Chapter 4
Experimental Apparatus for Various Operations
4.1 General layout of the equipment

The arrangement of the equipment is shown in Fig. 4.1 and Fig. 4.2. It consisted of a glass column in which the two liquid phases were made to flow countercurrently. All the storage tanks and most of the pipe work (0.7 inch i.d) were made of glass in view of the corrosive nature of the chemicals. P.T.F.E. was chosen as the material for all the gaskets.

4.2 The Fixed Baffle Contactor

The body of the F.B.C. itself is made of glass (borosilicate Q.V.F.) supplied by Corning Ltd. It is a vertical cylinder with a diameter of 6 in. The main part of the body is the extraction section which is 47.244 in. (120 cm) long. Above the extraction zone is the upper header and below, the lower header. Along the length of the column are seven equally spaced side arms in which the \( \frac{1}{4} \) in. bore stainless steel sampling tubes are fixed. (Three side arms are on one side and the rest are on the other.) Connected to each of these sampling tubes is a \( \frac{1}{4} \) inch bore glass sampling tap through a piece of short rubber tubing. The keys used for the sampling taps are 'Interflon P.T.F.E. Stopcock keys' made by G. Springham and Co. Ltd. The ends of the side arms are blanked off with small brass discs allowing only the sampling tubes through. The entrances of the sampling tubes are covered with very fine stainless steel wire meshes to prevent the dispersed droplets from getting through. Three side arms on one side were used for the main part of the experiment while some on the other side were used for the minor part of the project.

Both the top and bottom headers are held in position
Fig. 4.1 Flow diagram of the pilot plant
Key to Fig. 4.1

1 Continuous phase feed tank
2 Continuous phase storage tank (treated mains water)
3 Continuous phase collection vessel
4 Continuous phase centrifugal pump
5 Continuous phase rotameter
6 Fixed Baffle Contactor
7 Dispersed phase rotameter
8 U-tube manometer
9 Dispersed phase centrifugal pump
10 Dispersed phase collection vessel
11 Stirrer
12 Dispersed phase feed tank
a–h Dispersed phase sampling points
Continuous phase feed tank

Continuous phase storage tank

Continuous phase collection vessel

Dispersed phase feed tank

Fixed Baffle Contactor

Dispersed phase collection vessel
Fig. 4.2 Arrangement of the pilot plant.
with the extraction section by a stainless steel plate. This metal plate has a thickness of 3/8 in. and diameter of 12 in. Again, P.T.F.E. rings are used as a seal. Each plate has a hole drilled horizontally (along the plane of the plate) and a stainless steel tube connected to serve as the sampling point for the respective ends of the extraction zone. The metal plates also serve as manometer take-off points (i.e. a manometer for measuring the hold up is connected to these two points). Diagrams of the column are shown in Fig. 4.3 and 4.4.

A structure made of stainless steel baffle plates is placed inside the column and is shown in Fig. 4.5. Different sets of baffle plate with various sizes are used for the formation of the structure.

**4.3 The Baffle Plate (Fig. 4.6)**

The segmental baffle plate is made of stainless steel and has a thickness of 1/8 in. and three holes of diameter 2/5 in. are drilled through the plate. The baffle plates are removable and are held in position by accurately-machined spacer posts through the holes, which ensure levelness and flatness. The clearance between the baffle edge and the column wall is 1/8 in. and no external power is used for the extraction process. The detailed dimensions of the column and the baffle are shown in Table 4.1.

**4.4 Chemicals used in the experimental work**

Water from the mains is purified as an aqueous phase by a Permutit De-ionizer (Mk 12 F-type). The purity of the aqueous phase is constantly checked by measuring its conductivity which should be below 10 micromhos/cm (Compared with distilled water of approx. 30 micromhos/cm).
Fig. 4.3(A) Continuous phase sampling points (a - e) of the column.

Fig. 4.3(B) Continuous phase sampling points (a - h) of the column.
Fig. 4.4 The Fixed Baffle Contactor.
Fig. 4.5 The arrangement of the baffles
Fig. 4.6 A typical baffle.
<table>
<thead>
<tr>
<th>Column</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Column external diameter</td>
<td>6.5 in. (16.51 cm)</td>
</tr>
<tr>
<td>Column internal diameter</td>
<td>6.0 in. (15.24 cm)</td>
</tr>
<tr>
<td>Extractive length</td>
<td>47.24 in. (120 cm)</td>
</tr>
<tr>
<td>Length of top header</td>
<td>8.27 in. (21 cm)</td>
</tr>
<tr>
<td>Length of bottom header</td>
<td>6.3 in. (16 cm)</td>
</tr>
<tr>
<td>Number of sampling points</td>
<td>3</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Baffle</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Length of the segments along the diameter</td>
<td>3.75 in. to 4.75 in.</td>
</tr>
<tr>
<td>Thickness of the plate</td>
<td>0.125 in.</td>
</tr>
<tr>
<td>Clearance between the baffle edge and the column wall</td>
<td>0.125 in.</td>
</tr>
<tr>
<td>Material of the plate</td>
<td>Stainless steel</td>
</tr>
<tr>
<td>Material of the spacer posts</td>
<td>Stainless steel</td>
</tr>
<tr>
<td>Plate spacing</td>
<td>2 in. to 5 in.</td>
</tr>
</tbody>
</table>
The organic phases, viz., carbon tetrachloride and MIBK (methyl isobutyl ketone) and the solute, glacial acetic acid are all supplied by B.D.H. Chemicals Ltd. The carbon tetrachloride and MIBK are commercial grade while the acetic acid is Analar grade. Their properties are shown in Table 4.2.

4.5 Refractometer

The analysis in terms of refractive index measurement of the continuous and dispersed phase is carried out by means of an Abbé refractometer, supplied by Bellingham and Stanley Ltd. The cell consists of a fixed and hinged prism and both of the prism boxes are fitted with branches for water circulation in order to maintain the prism and sample at known temperatures. The light source for this instrument is a sodium lamp.

4.6 Equipment for axial mixing study

4.6.1 Tracers

The tracers commonly used for residence time study are (i) soluble dyes, (ii) radioactive tracers, or (iii) electrolyte solutions. It is important that tracers should be soluble only in the phase under consideration. The tracer used for the present study is a concentrated blue solution of nigrosine and a small quantity of it is injected into the phase under consideration by a hypodermic syringe through a 'subaseal' rubber bung.

4.6.2 Absorptiometer and Data-logger

The optical system of the absorptiometer is shown in Fig.4.7. The instrument was supplied by Hilger and Watts Ltd. and apart from the optical system, it consists of two absorption cells and a stabilized power supply for the lamp. A further feature of this instrument is the
Table 4.2

Physical properties of the chemicals used in the experimental studies at 20°C

<table>
<thead>
<tr>
<th></th>
<th>Carbon tetrachloride</th>
<th>MIBK</th>
<th>Water</th>
<th>Acetic Acid</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Formula</strong></td>
<td>CCl₄</td>
<td>(CH₃)₂CH.CH₂COOCH₃</td>
<td>H₂O</td>
<td>CH₃COOH</td>
</tr>
<tr>
<td><strong>Mass</strong></td>
<td>153.84</td>
<td>100.16</td>
<td>18</td>
<td>60.05</td>
</tr>
<tr>
<td><strong>Form</strong></td>
<td>liquid</td>
<td>liquid</td>
<td>liquid</td>
<td>liquid</td>
</tr>
<tr>
<td><strong>Density (gm/cc)</strong></td>
<td>1.59</td>
<td>0.8</td>
<td>1.0</td>
<td>1.049</td>
</tr>
<tr>
<td><strong>Melting pt.(°C)</strong></td>
<td>-22.60</td>
<td>-80.3</td>
<td>0</td>
<td>16.70</td>
</tr>
<tr>
<td><strong>Boiling pt.(°C)</strong></td>
<td>76.8</td>
<td>118</td>
<td>100</td>
<td>118.1</td>
</tr>
<tr>
<td><strong>Viscosity (cp)</strong></td>
<td>1.03</td>
<td>0.64</td>
<td>1</td>
<td>1.28</td>
</tr>
<tr>
<td><strong>Interfacial tension</strong></td>
<td>47</td>
<td>10</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td><strong>Diffusivity</strong></td>
<td>1.58</td>
<td>1.528</td>
<td>1.044</td>
<td></td>
</tr>
</tbody>
</table>

* Interfacial tension with water in dyne/cm
** Diffusivity (Acetic acid in solvent)(ft²/s)x10⁸
Fig. 4.7 Optical system and photocell circuit of the absorptiometer.
Key to Fig. 4.7

A and B  45° prisms
D and E  Collimating lenses
F and G  Heat absorbing filters
H        Lamp
I, J, Q and R  Windows
K        Programming filter
L        Calibrating filter
M and N  Colour filter
O        Reference absorption cell
P        Sample cell
S and T  Barrier layer photocells
V        Selector switch
U        Data-logger
Y        Galvanometer
X and W  'Set Full Scale' control
Z        'Set Zero' control
incorporated filter mounts, by which the required colour and neutral density filters may be introduced into the light beams.

One of the absorption cells contains pure water and acts as a reference while the other has dyed liquid passing through it continuously. A potential difference, which is proportional to the concentration of the dye in the liquids, is set up between these two cells. This potential difference is then transmitted, rather indirectly, into the LY 1470 Data-Logger system, made by the Solartron Ltd.

This Data-Logger system consists of two main parts, the LU 1461 Scanner and LM 1420 Digital Voltmeter. The scanner can be set in such a way that it gives from one reading every two seconds to one every twenty seconds.

These readings are sent to the D.V.M. in pulses. The output of the absorptiometer is displayed in millivolts on the D.V.M. panel and simultaneously on a paper tape by an 'Addo' punch machine linked to the D.V.M. The arrangement is shown in Fig. 4.8.

4.7 Manometer

The manometer for measuring the holdup of the dispersed phase takes the form of a glass U-tube, whose limbs are partly filled with carbon tetrachloride which is coloured with crystalline waxoline red dye. The remainder of the limbs is topped up with water. One limb is connected to the bottom of the extraction section through a plastic tube while the other is connected to the top.

4.8 Equipment for the interfacial tension measurement

The interfacial tension of the system varies with the amount of the solute in the phases and hence it has to be
Fig. 4.8 The data logger system
determined experimentally. There are various methods of
determination.

4.8.1. Torsion Balance

The torsion balance is generally used for the 'ring'
method. This torsion balance is an 'OS' type and is supplied
by the White Electrical Instrument Company Ltd. It consists
of a platinum ring of 4 cm circumference and is calibrated
from 0-0.12 N/m (0-120 dyne/cm) with 240 equal divisions. It
is shown in Fig. 4.9.

The ring method is normally employed for the system
which the lower liquid preferentially wets the platinum
ring. Water, with overlying oil such as MIBK, satisfies
this condition, but if the ring is wetted by the upper
liquid (e.g. water overlying CCl₄), the method requires
modification. Some authors have suggested that possibly a
stainless-steel ring coated with silicone to make it
preferentially oil-wetted would be satisfactory here.

4.8.2. Drop volume apparatus

This is known as the 'AGLA' Micrometer Syringe Outfit
and was developed by the Wellcome Research Laboratories,
Beckenham, England. This apparatus is particularly suitable
for water-CCl₄ system and it consists of a special all-
glass syringe which is connected to a micrometer by a metal
syringe holder. The micrometer is fitted with a ratchet
head and it operates the piston of the syringe. A glass
capillary needle in which the drop is formed at the tip is
joined to the all-glass syringe. The drop is formed inside
a glass vessel containing another liquid and its volume is
directly read from the micrometer. The glass vessel has a
dimension of 35mm x 35mm x 203mm. The advantage of this
Fig. 4.9 The torsion balance.
instrument is that the volume of 0.01 ml may be measured with an accuracy of ± 0.00005 ml. The arrangement is shown in Fig. 4.10 and it is generally used in conjunction with a chart of \( \phi \) against \( a/V^{1/3} \) as shown in Fig. 4.11. The correction factor \( \phi \) was known from the work of Harkins and Brown. (H 6)

4.8.3. Optical Apparatus

The optical apparatus is shown in Fig. 4.12a and 4.12c. It is the modified version of that developed by Andreas (A 6), and hence it can be used as an alternative to the drop volume apparatus. The optical components of the system were mounted on a one metre long optical bench with triangular cross-section. The drop was formed inside a rectangular vessel at the tip of the capillary needle of the 'AGLA' micrometer apparatus. The light from a 100 W high pressure mercury vapour lamp was focused by a condensing lens (L) of a short focal length, then passed through a neutral density filter F(ND 20), producing a parallel monochromatic light beam. This beam was then passed through the rectangular vessel and the microscopic objective M(48mm focal length) which was fixed with a telecentric stop. The image of the drop was focused on to the vidicon tube of the T.V. camera and the signal from this camera was then fed to the television monitor. The magnified image of the drop was displayed on the screen of the monitor.

An alternative arrangement is shown in Fig. 4.12b and 4.12d. The microscopic objective and T.V. camera were replaced by a 'Pentax' 35mm camera with a 50mm f.4 'Takumar' macro focusing lens. The camera was also supplemented with a 50mm extension tube and the image of the drop was directly recorded on it.
Fig. 4.10 The micrometer syringe.
The correction factor $\varphi$ is related to the interfacial tension $\gamma$ by the following equation:

$$\gamma = \varphi \rho_1 g / 2 \pi r$$

where $g$ is the gravitational acceleration, $v$ is the volume of the drop, $\rho_1$ is the density of the liquid, and $r$ is the radius of the tip of the tube.
Fig. 4.12a Optical apparatus.

Fig. 4.12b Alternative arrangement of the optical apparatus.
Fig. 4.12c Arrangement of the optical apparatus
Fig. 4.12d Alternative arrangement of the optical apparatus.
Chapter 5
The Equilibrium Distribution of Acetic Acid

(a) between Water and Carbon Tetrachloride

(b) between Water and MIBK
5.1 Prerequisite

Before any attempt at the determination of the equilibrium curve of the system can be made, the refractometer must be calibrated.

5.2 Calibration of refractometer

The thermostat was switched on to supply water at a constant temperature to the cell of the refractometer. It normally takes half an hour for the instrument to warm up. Samples containing different amounts of solute were placed between the refractometer prisms, and the corresponding refractive indices were read from the scale. This instrument was illuminated by a sodium light source and the temperature of the cell (i.e., temperature of the samples) was registered on a thermometer whose bulb was in the circulating water. Calibration curves were obtained by plotting refractive index against concentration of the solution. The shapes of the curves vary according to the nature of the solution and the temperature under which the solution was measured. The results are shown from Fig. 5.1 to Fig. 5.6 and in Table 5.1.

5.3 Experimental Procedure

Different amounts of acetic acid from about 1 to a very few cc. were added to bottles containing equal volumes (50 cc. each) of carbon tetrachloride and water. The bottles were shaken for 10 minutes then placed in a water bath at the required temperature for, say, 3 hours to allow the contents to reach equilibrium.

After the phases were separated, samples were taken and the acetic acid concentration was determined. For the aqueous phase (water), the acetic acid concentration was
Fig. 5.1 Refractive Index (R.I.) v. Temperature for water.
Fig. 5.2 Refractive Index (R.I.) v. Temperature for MIBK
Fig 5.3 Refractive Index (R.I.) v. Temperature for carbon tetrachloride
Fig. 5.4 Calibration curve- Acetic acid in water at 22°C.
Fig. 5.5 Calibration curve - Acetic acid in carbon tetrachloride at various temperatures.
Fig. 5.6 Calibration curve – Acetic acid in MIBK at various temperatures.
Table 5.1

Calibration data for CCl₄/ Acetic acid system

<table>
<thead>
<tr>
<th>Sample (25 ml CCl₄)</th>
<th>Mass of acid (gm)</th>
<th>Conc. (gm/litre)</th>
<th>R.I. (23.0°C)</th>
<th>R.I. (22.0°C)</th>
<th>R.I. (21.5°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure</td>
<td>0.0</td>
<td>0.0</td>
<td>1.4583</td>
<td>1.4592</td>
<td>1.4596</td>
</tr>
<tr>
<td>1</td>
<td>0.5</td>
<td>20</td>
<td>1.4560</td>
<td>1.4570</td>
<td>1.4572</td>
</tr>
<tr>
<td>2</td>
<td>1.0</td>
<td>40</td>
<td>1.4540</td>
<td>1.4550</td>
<td>1.4552</td>
</tr>
<tr>
<td>3</td>
<td>1.25</td>
<td>50</td>
<td>1.4530</td>
<td>1.4540</td>
<td>1.4542</td>
</tr>
<tr>
<td>4</td>
<td>1.50</td>
<td>60</td>
<td>1.4520</td>
<td>1.4530</td>
<td>1.4532</td>
</tr>
<tr>
<td>5</td>
<td>2.0</td>
<td>80</td>
<td>1.4501</td>
<td>1.4510</td>
<td>1.4512</td>
</tr>
</tbody>
</table>

R.I. = Refractive Index
measured using an Abbé refractometer. However, the acid content in the organic phase ($CCl_4$) was so dilute it could not be determined using this equipment; instead it was titrated against 0.01 N potassium hydroxide with phenolphthalein as indicator.

The whole procedure was repeated for the water and MIBK system, but with all the concentration determination carried out by the Abbé refractometer, i.e. no titration was necessary.

The different techniques employed for the measurements illustrated the very nature of the distribution of the solutes in various substance.

5.4 Experimental Results

(a) $CCl_4$-Acetic acid-Water system (Table 5.2(a))

The experimental data were plotted on a Cartesian coordinate graph (Fig. 5.7) whose equation can be assumed to take the form:

$$C_y = a(C_x^m)$$

where $C_y$ and $C_x$ are the concentrations of the acid in the continuous phase (water) and dispersed phase (carbon tetrachloride) respectively.

By using the method of least squares, the values of 'a' and 'n' were found to be 64.87 and 0.5357 respectively:

$$C_y^* = 64.87(C_x^{0.5357})$$

(on the basis of gm/litre)(Appendix 2)
(b) MIBK-Acetic acid-Water system (Table 5.2(b))

The results of this system give a straight line (Fig. 5.8) and its equation is:

\[ C^*_y = 1.64(C_x) \]  \hspace{1cm} (5.2)

Similar equations were derived by other workers such as Eshalomi (E 5) and Vermij and Kramers (V 4)

i.e. Eshalomi's equation: \[ C^*_y = 1.858(C_x) \]

Vermij and Kramers: \[ C^*_y = 1.8315(C_x) \]
Table 5.2(a)
Equilibrium data for CCl$_4$-Acetic acid-Water system

<table>
<thead>
<tr>
<th>$C_x$ (gm/l)</th>
<th>$C_y$ expt. (gm/l)</th>
<th>$C_y$ cal. (gm/l)</th>
<th>$C_y$ cal. - $C_y$ expt. (gm/l)</th>
<th>Weng's results (W 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.18</td>
<td>21.0</td>
<td>25.887</td>
<td>+4.887</td>
<td>0.08</td>
</tr>
<tr>
<td>0.3</td>
<td>38.5</td>
<td>34.035</td>
<td>-4.465</td>
<td>0.18</td>
</tr>
<tr>
<td>0.4359</td>
<td>44.0</td>
<td>41.577</td>
<td>-2.423</td>
<td>0.44</td>
</tr>
<tr>
<td>0.6319</td>
<td>53.0</td>
<td>50.728</td>
<td>-2.272</td>
<td>0.93</td>
</tr>
<tr>
<td>0.9159</td>
<td>66.0</td>
<td>61.887</td>
<td>-4.113</td>
<td>1.45</td>
</tr>
<tr>
<td>1.332</td>
<td>80.5</td>
<td>75.638</td>
<td>-4.862</td>
<td>2.23</td>
</tr>
<tr>
<td>2.472</td>
<td>102.0</td>
<td>105.341</td>
<td>+3.341</td>
<td>2.89</td>
</tr>
<tr>
<td>3.276</td>
<td>110.0</td>
<td>122.493</td>
<td>+12.493</td>
<td>3.55</td>
</tr>
</tbody>
</table>

Table 5.2(b)
Equilibrium data for MIBK-Acetic acid-Water system

<table>
<thead>
<tr>
<th>$C_x$ (gm/l)</th>
<th>$C_y$ (gm/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>34.0</td>
<td>64.0</td>
</tr>
<tr>
<td>51.0</td>
<td>93.0</td>
</tr>
<tr>
<td>76.0</td>
<td>120.0</td>
</tr>
<tr>
<td>93.0</td>
<td>144.5</td>
</tr>
<tr>
<td>110.0</td>
<td>179.5</td>
</tr>
</tbody>
</table>
\[ c_y = 64.87 c_x^{0.5357} \]

○ $c_y$ expt.

△ $c_y$ cal. (from $c_y^* = 64.87 c_x^{0.5357}$)

Fig. 5.7a Equilibrium curve for the carbon tetrachloride – acetic acid – water system.
Fig. 5.7b Equilibrium curve for the carbon tetrachloride - acetic acid - water system. (enlarged section)

Gradient of the section, \( m = 6.24 \)
Fig. 5.8 Equilibrium curve for the MIBK - acetic acid - water system.
Chapter 6
Experimental Technique
6.1 Scope of experimental work

Two countercurrent systems, CCl₄-Acetic acid -water and MIBK-Acetic acid-water were studied and all the experimental work was carried out with the contactor as described in section 4.2.

Water was made to flow upwards in the carbon tetrachloride system as it is the lighter phase. For the MIBK system, water was allowed to flow downwards as MIBK is lighter than water. Both carbon tetrachloride and MIBK were used as the dispersed phase.

Experimental work was carried out with different plate gaps and plate spacings. Plate gap is the maximum cut-off segmented width between the plate and the column and plate spacing is the difference in height between two plates. Hold up measurements and photographic studies (interfacial area measurement) were made for each arrangement and a dye study was carried out on continuous phase only. As to cleanness, the apparatus was kept at maximum purity. Further experiments on droplets were performed with optical studies.

6.2 Flooding Studies

Flooding conditions were investigated under two different arrangements of the baffle plates.

For one particular setting of the baffle plate, the column was filled with continuous phase and the dispersed phase was then entered the column at a constant flowrate. The difference in the manometer reading, i.e. the hold up was noted when the system was stabilized. The flowrate of the continuous phase was varied incrementally and the procedure of taking readings was repeated.
The flooding point was achieved when there was a gradual accumulation of the dispersed phase in the column, as indicated by the continuous increase in the hold up in the manometer.

If the contactor contains droplets with a wide range of sizes, then the very small drops would be carried over by the continuous phase while the system is still far from flooding. This is because the terminal velocity of the droplet is being exceeded by the upward velocity of the continuous phase.

The above phenomenon explains why the flooding point is difficult to determine by visual observation.

6.3 Calibration of instruments

6.3.1. Calibration of Absorptiometer

In the interest of maximum accuracy, the absorptiometer was calibrated every day as follows:

The absorptiometer was switched to 'on' and left to stabilize for one hour. The reference cell was filled with water (or any appropriate reference liquid) and it was used in conjunction with a neutral density filter. The optical density of this filter was equivalent to the reference liquid. The 'SET ZERO' control was then adjusted to give zero reading on the LM 1420 Digital Voltmeter.

For 'FULL SCALE' adjustment, the reference cell was left in the condition used for setting zero. The sample cell was filled with a coloured water (or coloured reference liquid). The colour was due to Nigrosine and a concentration of 0.06 gm/litre was used. This concentration was maximum for the absorptiometer as any value above this would make the response non-linear. The 'SET FULL SCALE' control
was then adjusted to give a reading of 2000 on the Digital Voltmeter.

6.3.2. Rotameter

Both of the rotameters for measuring the flow rates of the dispersed and continuous phase were calibrated by direct collection of the liquids in a graduated cylinders over three minutes against meter readings. The rotameter was recalibrated each time a different liquid was used.

6.4 Axial Mixing study

The dye injection study was carried out on the continuous phase under single phase flow only. This is because the study of the continuous phase with the system in two phase flow is inaccurate. This inaccuracy is due to the surfactant effect of the dye which lowers the interfacial tension and subsequently lowers the flooding point of the system.

For the continuous phase dye study, the absorptiometer was switched on for a warm up period of about one hour and calibrated. The continuous phase (water) was passed into the column which was inserted with the baffle plate structure. Five cc. of Nigrosine solution (about 0.02gm/l) was quickly injected with a hypodermic syringe through a rubber seal in the inlet line of the continuous phase (water). The tracer concentration in the outlet stream passing through the absorptiometer was simultaneously recorded in the form of a punched tape by the data-logger system.

6.5 Hold up measurement with manometer

The hold up or volume fraction of the dispersed phase was measured by the manometric method and the coloured carbon tetrachloride (carbon tetrachloride with a very small
quantity of crystalline Waxoline Red dye) was used as the manometer liquid.

The limbs of a U-tube were partially filled with this coloured carbon tetrachloride and one side of it was connected to the bottom of the extraction zone of the column, while the other was connected to the top. Air bubbles were expelled from the system by topping up the arms completely with the continuous phase. The column was then filled with continuous phase so that the levels of carbon tetrachloride in the manometer limbs were then equal.

As soon as the dispersed phase (carbon tetrachloride or MIBK) was introduced into the column, the manometer liquid levels gradually drifted apart and its difference was recorded under the condition of hydrodynamic equilibrium.

6.6 Mass transfer processes

The solute was added to the dispersed phase (which was mutually saturated with the continuous phase) in the storage vessel and the content was thoroughly mixed for about half an hour. This mixing process was carried out by a 3 inch diameter marine-type propeller at a speed of 950 r.p.m. The solute concentration in the dispersed phase was maintained at about 50 gm/l and the continuous phase was solute free. Before the start of the experiment, the dispersed phase was circulated through the piping system by a centrifugal pump for a short while and finally the feed tank was fully charged.

The column, which contained the specified baffle structure, was filled with the continuous phase (water) from its feed tank, which had previously been purified by a Permutit De-Ioniser MK 12 water treatment system. Dispersed
phase was then introduced into the column and the flow rates of both phases were recorded by the rotameters. All the operations were carried out in a counter-current manner and the coalescence pool was maintained at a constant level by means of an outlet needle valve.

The fluctuations in the 'steady' state created by the decrease in head, which was provided by the feed tanks were eliminated by the use of the stainless steel 'Bar Stock Ball' valves supplied by Hoke International Ltd., which have fine adjustment.

Samples of the dispersed and continuous phase were taken at their respective inlets and outlets under steady conditions. Continuous phase was further sampled at the sampling points along the column's extractive zone. Liquids remaining in the sampling devices were discarded by draining away a small amount of continuous phase. The sampling rate was kept slow in order not to disturb the hydrodynamic equilibrium that had been obtained in the column.

6.7 Photographic and droplet counting studies

Photographic work was carried out for all the mass transfer processes and subsequently the total interfacial area was evaluated.

6.7.1. Photographic Technique

A 'Pentax' 35 mm camera with a 50 mm f.4 'Takumar' macro focusing lens was used for the present study. The film was the FP4 125 ASA (in daylight and with electronic flash) and the aperture was set at f22. The exposure was controlled by a Sunpack Autozoon 2000 flash gun which was operated with a speed of 1/1500 of a second and the camera was focused to give maximum depth of field in the region of the place.
The droplets of the dispersed phase were illuminated indirectly by reflecting the light on a white cardboard hung behind the column. The flash gun was positioned in such a way that the upper part of the white cardboard was brighter than the lower part (Fig. 6.1). This was necessary in order to obtain maximum definition (i.e., contrast) of the droplets.

Minimum distortion of the picture due to the curvature of the glass column was considered to be negligible and pictures were taken for each flow condition with each baffle spacing.

6.7.2. Droplet counting studies

A5 size pictures were printed and the droplets were sized and counted by a S.P.R.I. particle size analyser. This analyser is shown in Fig. 6.2. The sizes of the drops on the photograph were measured by a graduated template and their actual magnitudes were calculated using the magnification factors.

Also, the drops that were not in focus and drops that were badly distorted were neglected and examples of the droplet photographs are shown in Fig. 6.3.

6.7.3. 'Cine' photography

Further photographic work was carried out with a Bolex H 16 R Clockwork Cine Camera with a 10 mm f1.6 switar lens. The process was filmed at a speed of 64 frames per sec (at f8) with Ilford Mk V negative (200 ASA). The lighting was provided by two 1200W Q.I. (Quartz Iodine) lamps positioned to give similar illumination to the electronic flash. Approximately 5 feet of films was exposed for each run and the results were analysed by a John Hadland Motion -
Fig. 6.1 Optical apparatus for the photographic work.
Fig. 6.2 The particle analyser
Gap = 1.62 in.
Spacing = 5 in.

$Q_c = Q_d = 100$ litres/hr.
Fig. 6.3.(A) A typical photograph used for determining the droplet size. 

($\text{CCl}_4$/Acetic acid/Water)
Gap = 1 in.
Spacing = 5 in.

\( Q_c = Q_d = 100 \text{ litres/hr.} \)
Fig. 6.3.(B) A typical photograph used for determining the droplet size.
(MIBK/Acetic acid/Water)
analysing projector (Mk III).

6.8 Refractometric analysis of Mass Transfer results

As all the experiments were carried out with single solute (acetic acid) transfer, a simple refractometric method was used for the analysis. This Abbe refractometer was used in conjunction with a sodium light source and the temperature of its cell was kept constant by a stream of warm water which was supplied by a thermostatically controlled bath. The instrument employs the critical angle effect marked by a demarcation line (i.e. borderline) between bright and dark portions of the telescope field.

A small amount of sample (about 2 cc.) was placed on the cell, which consists of the upper and lower prisms, and the instrument was manipulated until the demarcation line coincided with the telescope cross-wire. The refractive index was then read out directly from the scale.

6.9 Computation of the mass transfer results

The performance of the contactor was measured in terms of the process characteristics, NTU, HTU, and (Ka), and residence time characteristics, μ & 6. Their actual magnitudes were evaluated by the 'Basic' & 'Algol' programs (Appendix 3 and 4) respectively with the aid of the I.C.L. 1905F and Prime 750 computers.

6.10 Interfacial Tension

Interfacial tension was measured by different methods, depending the nature of the liquids.

6.10.1. Using Torsion Balance

After setting up, the balance was checked for zero with the platinum ring completely immersed in the upper (MIBK) of the two liquids. The platinum ring was then completely
immersed in the lower liquid (water) before the platform (P) was gradually lowered by means of the adjusting screw (D) and the platinum ring drawn through the interface. At the same time, the index pointer (B) was moved in an anti-clockwise direction so as to maintain the beam pointer (C) at zero. After a degree of movement that was dependent upon the interfacial tension of the liquids, the ring suddenly parted from the interface of the liquid. The value indicated by the index pointer (B) at that particular moment was the interfacial tension of the liquid and it was read directly from the balance dial.

6.10.2. Using Drop Volume Apparatus

With this apparatus (Fig. 4.10) the drop was formed extremely slowly at the tip of the capillary tube inside a cuboidal vessel containing water. The limiting volume of the carbon tetrachloride drop in water was measured directly by the micrometer. With the aid of the correction factor chart, (Fig. 4.11) the interfacial tension was calculated.

One important point to note is that the nature of the water and carbon tetrachloride used in this experiment. Equal volumes of these were equilibrated with different amount of acetic acids. Then, they were separated and used for their respective purposes.

6.11 Optical studies of droplets

The phenomena associated with the droplets were investigated further with the optical apparatus. These studies were necessary as they provide an insight into the characteristics of the droplets.

6.11.1. Interfacial Tension by Andreas Method
This method served as an alternative to the drop volume apparatus and the equipment required is shown in Fig. 4.12 b and d. The procedure of forming a drop was the same as described in section 6.10.2. The photograph of the drop was taken with the aid of a 'Pentax' camera (section 4.8.3). The camera was placed in such a way that the distance between the glass vessel and the end of the extension tube was about 70 mm. The aperture and the shutter speed were set at f22 and 1/15 of a second respectively. The depth of field was very small (about 1/2 mm) and no flash gun was used. The extension tube was necessary as it would make the distance between the lens and the film plane longer. The size of the drop was measured in terms of the equatorial diameter and other parameters from the photograph and the interfacial tension was evaluated with the help of the S vs. l/H table developed by Andreas et al. (Fig. 6.4)

6.11.2. Eruption Studies

The eruption studies were carried out with the same apparatus as described in section 6.11.1. (Fig. 4.12 c and d) but the nature of the continuous and dispersed phases was different. The dispersed phases contained different amount of solute were used to form the drops in the continuous phase (which was pure distilled water). The eruption and 'kicking' phenomena were again recorded by the 'Pentax' camera.
Fig. 6.4 $1/H$ as a function of $S$. (Developed by Andreas et al.)

$H$ is a function of the surface tension.

$S$ is the ratio of the maximum diameter of a drop to a linear dimension of the same drop at a distance equal to the maximum diameter from the bottom of the drop.
Chapter 7

Record of Experimental Results
7.1 Flooding studies

The results of the flooding studies for the carbon tetrachloride–water system with two different arrangements of the baffle plate are shown in Table 7.1(1) and Fig.7.1(1).

7.2 Axial mixing study

The results obtained from the dye injection experiment were used to calculate the mean residence time and variance (Eq.3.40 and 3.41 respectively), with the aid of a computer program.(Appendix 3).

The continuous phase Peclet number was evaluated from the Van der Laan relationship which was defined in equation 3.27. It involves the dimensionless variance (Eq. 3.19). The continuous phase axial mixing coefficient,$E_c$, was obtained from equation 3.17.

The experimental values of various parameters such as mean residence times, Peclet number, Dispersion number, axial mixing coefficient and variance under different operating conditions are given in Table 7.2(1).

The variation of the continuous phase flowrate with various parameters and a sample of the response curves for the experimental runs is shown in Fig.7.2(1) and Fig.7.2(2) respectively.

A comparison of the theoretical and the practical relationship between the dimensionless variance and the Peclet number is given in Fig.7.2(3). The theoretical equation is the one developed by Van der Laan(V 9), i.e.

$$\delta^2 = \frac{2}{N_{Pe}} (N_{Pe} - 1 + e^{-N_{Pe}})$$
7.3 Mass transfer

The solute concentration profiles of the two phases for both MIBK and carbon tetrachloride systems, under different flow conditions and for different initial solute concentrations, are shown in Table 7.3(1), with some of the graphical presentation in Fig. 7.3(1). As described in the experimental procedure section, the data are the 'apparent measured' values as defined by Thomas (T7).

The performance of the column, as generally expressed in terms of the sectional and total NTU, HTU and (Ka) was evaluated by the computer 'BASIC' program (Appendix 4) based on the integration of equation 3.92 (Simpson's rule) and they are also tabulated in Table 7.3(1) and displayed in Fig. 7.3(2). (In the case of the sectional values, only a sample of the data is shown.)

Again, the NTU, HTU and (Ka) are all 'apparent measured' values.

7.4 Hold up

The hold up of the dispersed phase for the two systems under different flow conditions was calculated by the equation 3.57 with the aid of the manometer. The results are incorporated in Table 7.3(1) and its influence on various parameters such as flowrates and (Ka) is shown in Fig. 7.4(1).

7.5 Photography and counting

The photographs of the droplets (Fig. 7.5(1)) under various mass transfer operations were used to calculate the Surface Mean diameter, $d_{32}$ which was defined by equation 3.120, this in turn being used to calculate the interfacial area based on equation 3.119. The overall
results are shown in Table 7.3(1).

7.6 End effects

When the liquids enter or leave the extraction column, there is an abrupt change in their concentrations. The change is greater on entry than on leaving. As already explained earlier the change is due to the difference in the hydraulic and other properties of the system outside the active region of the extraction column. The end effects have been calculated by the method described in section 9.6 and the values are given in Table 7.6(1).

7.7 Interfacial tension

The interfacial tension of the MIBK and carbon tetrachloride systems under different equilibrium concentrations are shown in Table 7.7(1) and Fig 7.7(1).

The tensions of the MIBK system was determined by a torsion balance while that of the carbon tetrachloride system was found by an 'AGLA' micrometer syringe (drop volume apparatus), the difference being due to the nature of the fluids.

7.8 Optical studies

7.8.1. Andreas Method

Fig. 7.8.1(1) and Table 7.8.1(1) show the results of the interfacial tension of the carbon tetrachloride droplet in pure water and these results were used to substantiate the ones carried out previously (section 7.7) for accuracy.

7.8.2. Eruption studies

The results of the eruption studies are shown in Fig. 7.8.2(1). These pictures indicate various stages of the eruption process accompanying the transfer of acetic acid from the carbon tetrachloride droplets.
Chapter 8

Evaluation of Various Overall Coefficients $(K)_c$

of the Continuous Phase
8.1 Various overall coefficients for the continuous phase \((K_c)\)

The coefficients given as \((K_c)_1\), \((K_c)_2\), \((K_c)_3\) and \((K_c)_4\) all refer to the mass transfer coefficients in the continuous phase and they are derived from different definitions as follows:

(i) Experimental \((K_c)_1\)

The experimental \(K_c\), which is denoted by \((K_c)_1\), is obtained directly from the formula:

\[
(K_c)_1 = \frac{(K_a)_{\text{expt}}}{a_{\text{expt}}} = \frac{(K_a)_{\text{expt}}}{\left[\frac{6\theta_{\text{expt}}}{d_{32 \text{ expt}}}\right]}
\]

where \(a\) is the interfacial area.

It is of importance to realise that in the fixed baffle column there are at least two well defined sources of interfacial area. As can be seen in the diagram (Fig. 8.1) the moving liquid film on the plate although disturbed is not the same as the discrete drops falling between the plates. The former can be referred to as '\(a_p\)' (plate) and the latter as '\(a_d\)' (drop).

Of course as measured, the combined coefficient \(K_c\) and total interfacial area '\(a\)' that is \((K_a)_{\text{expt}}\) includes all types of interfacial area. The resultant '\(a\)' is not
necessarily a simple summation of \(a_p\) and \(a_d\) as it is in association with \(K_c\).

As shown by Laddha[2] for the case of the performance of perforated plates, mass transfer is virtually negligible for the liquid film hold up on the plates compared with the rest of the dispersed phase drop system. This being so it is reasonable to suggest that the same result will apply to the present case where the plates are fixed but not perforated.

\[
(ii) \quad (K_c)^2
\]

\((K_c)^2\) can be evaluated with the aid of the expression for the individual phase coefficients \((k_c\) and \(k_d\)), developed by Sivaramen[19] and they are based on the experimental data using single file drops. The two expressions are:

\[
k_c = 0.023 \ U_s \left( \frac{\mu_c}{\rho_c D_c} \right)^{-1/2}
\]

\[
k_d = 0.023 \ U_s \left( \frac{\mu_d}{\rho_d D_d} \right)^{-1/2}
\]

where \(U_s\) is the slip velocity defined in section 2.2.4.

The above two expressions are further modification of equations (3.69) and (3.70). As explained in the section (i), the coalesced layer plays only a minor part in the process of mass transfer, the \(k_d\) value hence refers to the droplets in motion.

The \((K_c)^2\) is then obtained by the following equation:

\[
\frac{1}{(K_c)^2} = \frac{1}{k_c} + \frac{m}{k_d}
\]
where $m$ is the gradient of the concentration distribution curve along the gradient and it is an experimental value.

Even though the concentration distribution is not a straight line, however, it is reasonable to consider $m$ as a constant over a limited range under which the experiments were carried out.

$$(iii) \ (K_c)^3$$

The calculation of the $(K_c)^3$ is based on the penetration theory (H 12) and the individual coefficients $(k_c$ and $k_d)$ can be expressed as follows:

$$k_c = 2 \frac{D_c}{\pi d_p} \frac{U_t}{U_s} \quad (3.65)$$

$$k_d = 2 \frac{D_d}{\pi d_p} \frac{U_t}{U_s} \quad (3.67)$$

where $d_p$ is the diameter of the droplet.

The terminal velocity $U_t$ in the above formulae is for a single drop while the slip velocity $U_s$ (section 2.2.4) is for the droplets in a swarm. Such substitution is reasonable in view of the size and oscillation of the drops in the present experimental work. Similar arguments were put forward by Laddha (L 2) and Treybal (T 1). $(K_c)^3$ is also obtained by a similar expression as $(K_c)^2$, i.e.

$$1/(K_c)^2 = 1/k_c + m/k_d$$

and again, $k_d$ refers to the droplets in suspension.
$k_c$ for the present case was obtained by Ruby-Elgin's expression (R 7) for a swarm of drops and $k_d$ of the droplets in motion was calculated using Handlos & Baron's equation (H 13), which is basically for fully developed internal circulation (section 3.5.2). Again, the $U_t$ in the original expression was modified into $U_s$ because of the dispersion droplets, and $(K_c)_4$ is given by:

$$1/(K_c)_4 = 1/k_c + m/k_d$$

The values of various $(K_c)_4$ are given in Table 8.1(1).
Chapter 9
Discussion
9.1 Flooding

The data of the flooding rate studies for the carbon-
tetrachloride system are shown in Table 7.1.(1) and
Fig. 7.1.(1). The family of curves was obtained by plotting
\( \phi \), the dispersed phase fractional hold up below flooding,
against \( Q_c \), with the dispersed phase flowrate, the gap
width and the plate spacing as the fixed experimental
parameters. The resulting shape of the curves was not
unexpected due to the increasing interactions with
increasing flow rates. For a fixed spacing, the hold up is
directly proportional to the gap width, as a smaller gap
means that the compartment retains more dispersed phase in
a given period. (Fig. 7.1.(1A,1B))

The effects of the gap width on the hold up can be
easily seen by referring to Fig. 7.1.(1C). The flow
conditions for the two sets of experiments were identical,
except the gap width. By reducing the gap width by 1/2 in.,
the hold up was immediately increased.

An interesting comparison on the flooding characteristic
between the present fixed baffle contactor and a packed
column can be made in terms of the superficial velocity
of the dispersed phase and the resulting graphs are shown
in Fig. 7.1.(1D)(fixed baffle column) and Fig. 7.1.(1E)
(packed column). Fig. 7.1.(1E) is for a toluene–water system
and it was the result of the investigation by
Chandrasekaran(C 10). The diameter of Chandrasekaran's
column was 4 inches and the packing material he used was
spheres each with a diameter of 17.5 mm.

The similar trend of the two graphs are not unexpected
and such similarity is due to the fact that the flooding
rates are mainly governed by the drop size distribution (L 4) and this in turn is a function of the column geometry and the droplet formation mechanism. According to Laddha (L 16), similar hold up curves were obtained for spray tower and absorption column.

Fig. 7.1. (ID) indicates that the fixed baffle contactor will be less prone to flooding because of its mode of construction and the absence of any mechanical device such as stirrer or rotatory disc. Also the drops will be on the whole layer and this affects the coalescence process up to certain extent. This point was illustrated clearly by Logsdail (L 4) in his RDC studies. His RDC consists of a rotor and stirrer rings and these devices produced a 'vortex' flow pattern of the continuous phase in which the droplets were followed and also the coalescence of the solvents was found to be negligible.

An important factor which has to be considered carefully is the interfacial tension; it decreases with the concentration of the solute in the systems. A high interfacial tension results in the drop resisting deformation, and hence the dispersed phase droplets in a binary system with no solute transfer are more spherical than the ones in a ternary system. This was confirmed by the present study.

The changes in concentration of solute and hence the interfacial tension of the system influence the rate of drop coalescence and therefore must be of considerable importance to the inducement of flooding. This point was further illustrated by Logsdail (L 4) and he stated that the presence of an organic solute in the dispersed phase
would enhance the 'characteristic velocity' of the droplets, and such increase would be inevitably linked to the rate of droplet coalescence.

9.2 Axial mixing

The purpose of carrying out mixing studies was to obtain, through the values of the Peclet number, the degree of backmixing in the apparatus. This can only be done meaningfully by reference to the continuous phase. Such a study on droplets would involve using a dye in the discontinuous phase which may in fact affect the surface tension of the drops and hence affect the measured results.

The experimental method has been described (section 6.4) and in Table 7.2.1 the values of the variance and Peclet numbers have been given for different conditions of operation of the column.

9.2.1 Relation between the flowrate and mean residence time

The relationship between the flowrate and the mean residence time, \( \mu \), is shown in Fig.7.2.1. Fig.7.2.1A refers to the situation of fixed baffle gap width and variable spacing, while Fig.7.2.1B is the other way round. The curve shows an approximate half-rectangular hyperbola shape as expected, i.e. \( \mu \) increases as the flowrate decreases. It should be noted that the effect of the gap width (with constant spacing) on the value of \( \mu \) appears to be minimal. On the other hand, the effect of the spacing (constant gap width) is more prominent. This is understandable as the flow path of the fluid particle in the case of spacing being varied is affected to a greater extent. Hence it can be said that, if the spacing is fixed,
then there is a range of gap width in which the contactor can be operated with a constant $\mu$. The difference between $\mu$ and the theoretical (plug flow) mean residence time $\bar{t}$ indicates the degree of dispersion, i.e., backmixing, and its magnitude (absolute value) approaches zero as the flow rate $Q$ increases. This is expected and it can be confirmed by the increase in the value of the Peclet number.

One of the problems associated with the residence time measurements was the fact that the residence time as measured includes end effects as well as the main extraction column. This is of particular importance to the $\text{CCl}_4$/Acetic Acid/Water studies at the bottom of the column where the water enters. The water first enters an end chamber (bottom header) before entering the main column. The nature of the water flow in the end chamber is not known exactly but it differs from the main column because of the constructional difference. However, it can be shown quite simply that the residence time in the end chamber is comparatively low compared with the main column, and so the overall residence time can be taken reasonably safely as the time in the column.

Take the case of the water flowrate of 150 litres/hr (Table 7.2.(1A)) and as the volume occupied by the water in the bottom end section is approximately 2.65 litres, then this gives the residence time of $2.65 \times 3600 / 150$ sec, i.e., 63 sec. for plug flow. The measured overall mean residence time for such case was 600–700 seconds.

Hence the end effects is of the order of 10%, which is within the limit of accuracy required to decide the nature of the mixing in the main column.
The above estimation was substantiated by calculating the end effects independently in terms of the percentage extraction under mass transfer operation and the details are given in section 9.6. They are also in the same sort of order of magnitude.

9.2.2. Influence of the operating conditions on the other mixing parameters

9.2.2.1. Peclet number \(v\) flowrate

Continuous phase liquid flow rates from 50 to 150 litres/hr giving \(N_{Pe}\) values of 3.9 to 19.37 were obtained experimentally. These are shown in Fig. 7.2.(1c) for different baffle spacings and gap widths.

By definition (for continuous phase)

\[
N_{Pe} \rightarrow 0 \quad \text{perfect mixing}
\]

\[
N_{Pe} \rightarrow \infty \quad \text{plug flow}
\]

Below is a table of \(N_{Pe}\) values for various conditions taken from Levenspiel (L 14). However, no clear statements on the significance of the \(N_{Pe}\) values for intermediate mixing conditions was available in the literature.

<table>
<thead>
<tr>
<th>Dispersion no.</th>
<th>Peclet no.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(E/uL)</td>
</tr>
<tr>
<td>Plug flow</td>
<td>0.0</td>
</tr>
<tr>
<td>Small amount of dispersion</td>
<td>0.002</td>
</tr>
<tr>
<td>Intermediate amount of disp.</td>
<td>0.025</td>
</tr>
<tr>
<td>Large amount of dispersion</td>
<td>0.2</td>
</tr>
<tr>
<td>Perfect mixing</td>
<td>(\infty)</td>
</tr>
</tbody>
</table>

Table 9.1
With \( \text{N}_{\text{Pe}} \) values 3.9 to 19.37 it would appear that there is mixing taking place in the continuous phase, probably of an intermediate nature.

As the continuous phase flow rate \( Q_c \) increases, \( u_c \) increases and hence \( \text{N}_{\text{Pe}} \) increases. The rate of increase of \( \text{N}_{\text{Pe}} \) with \( u_c \) depends on the effect of flowrate on the eddy diffusivity \( E_c \) \( (\text{N}_{\text{Pe}} = u_c L/E_c) \).

The \( \text{N}_{\text{Pe}} \) values shown in Fig.7.2.(1C) were obtained experimentally from dye studies in the present experiments through the variance. From the experimental values of \( \text{N}_{\text{Pe}} \) and the known length \( L \), \( E_c \) was calculated \( (\text{N}_{\text{Pe}}/L_{\text{expt}} = u_c/E_c) \). Its relationship with \( Q_c \) is shown in Fig.7.2.(1E) and Fig.7.2.(1F), \( Q_c \) being directly proportional to \( u_c \). These graphs reflect the changes in Figs.7.2.(1C & 1D).

Clearly the greater the spacing \( S \) and gap width \( G \) the higher the \( \text{N}_{\text{Pe}} \) values and the less the comparative degree of mixing in the continuous phase. This is logical but the difficulty that arises is attaching real significance to the rather low \( \text{N}_{\text{Pe}} \) values between 3.9 and 19.37. For both \( S \) and \( G \) there will be an upper and a lower limit which influence the loading of the column. Small values of \( S \) and \( G \) give higher pressure drops and decrease the time of contact of phases. Plate length clearly could be of importance. On a fixed column diameter increase of gap width \( G \), decreases the plate length but the change is not large enough to be significant with respect to the film flow across the plate. Further extensive experiments would be required which would be quite expensive to carry out as it requires new apparatus each time.
The corresponding curves of the axial dispersion coefficient $E_c$ v. flowrate are shown in Fig. 7.2.(1E) and Fig. 7.2.(1F). As expected, the axial dispersion coefficient $E_c$, which uniquely characterizes the degree of backmixing during flow, increases with the flow rate. In practical terms, increase in flow rate means increase in turbulence and hence more eddies are formed. This produces bigger distortion of the plug flow profile. Another point to be noted from the present experimental results is that the spacing has a greater effect than the gap width (Table 7.2.(1)).

While these two sets of curves (Fig. 7.2.(1E) and Fig. 7.2.(1F)) indicate the extent of mixing qualitatively, the actual degree of mixing can be visualized by observing the magnitude of the coefficient $E_c$. By judging the shapes of the curves of $E_c$ v. the flowrate (Figs. 7.2.(1E & 1F)), it is reasonable to suggest that the relationship between these two quantities can be given as

$$E_c = \alpha (Q_c)^\beta$$

where $\alpha$ and $\beta$ are constants.

Similar relationship has been obtained for the spray column by Hazlebeck & Geankoplis (H 16).

The actual values of $E_c$ obtained for the present study were from approximately 0.001 ft$^2$/sec to 0.003 ft$^2$/sec (Tables 7.2.(1A & 1B)) and these low values of $E_c$ indicated that the degree of mixing was low in the contactor, and the spacing and gap of the plates had a limited influence on
the mixing process. Such reasoning concerning the degree of mixing can be supported by reviewing the work carried out by Smoot & Baab (S.8) with a pulsed column of diameter of 2 inches and height of 4 feet. The magnitude of their $E_c$ were in the same order as the ones obtained in the present study ($0.002 \text{ ft}^2/\text{sec}$ as compared to $0.003 \text{ ft}^2/\text{sec}$ under the similar conditions) and they also pointed out the important influence of the frequency and amplitude of the pulse externally induced; which the present column did not have. The magnitude of $E_c$ suggests that the Taylor-type diffusion (lateral molecular diffusion) is negligible.

When the four graphs (Fig. 7.2.(1C) to Fig. 7.2.(1F)) are compared, and bearing in mind the definition of the Peclet number ($u_c L / E_c$), it can be seen that the high flow rates ($Q_c$ or $u_c$) produce a counteracting effects, that is, an increase in $E_c$. However, $E_c$ does not increase rapidly enough with increase in $u_c$ to maintain $u_c / E_c$ constant. Hence, with increase in $u_c$ there will be a qualified increase in $N_{pe}$. Even though $E_c$ is used to characterise the degree of backmixing, the extent of axial dispersion is better measured by the Peclet number.

Fig. 7.2.(3A) shows the relationship between the dimensionless variance and the Peclet number. Again, the deviation of the experimental results from the theoretical value becomes less as the Peclet number becomes larger, i.e. plug flow. Such a condition is approached asymptotically.

Fig. 7.2.(2A) is a typical $F(t)$ curve obtained from the system and it shows clearly the degree of mixing, for it was observed that it lies between the two ideal cases.

9.3 Mass transfer
A sample of the concentration profiles of the systems is shown in Figs. 7.3. (1A-1N). These profiles refer to the concentration pattern of the continuous phase which was measured experimentally along the column for a fixed arrangement of the internal structure, while the flow ratio of the two phases served as a parameter. Of course, if the concentration of the dispersed phase could have been obtained successfully experimentally, then a similar pattern would have emerged.

Within the range of the operative flowrate, the variation in the top and bottom end effects was relatively small. With possible exception, the average bottom and top effect were about 12 and 8% respectively. Hence, the extraction efficiency over the extractive length was nearly constant. Such constancy was for different loadings and this implies that the performance of the column is better when the efficiency is maintained at higher flowrates. There is a certain influence exerted by small spacings and gap widths as these produce high hold up, which in turn produces higher interfacial area.

It should be pointed out that the effects exerted by both phase flowrate and the plate spacing on the various parameters such as Ka etc. are similar, and hence similar graphical trends appear.

9.3.1. Overall HTU, NTU, and (Ka)_{CA}

The values of the HTU (which is a reciprocal of the NTU) and Ka, can be considered as a measure of the efficiency of the column and are largely governed by factors like axial mixing, droplet interaction and interfacial tension. While droplet interaction promotes higher efficiency, axial mixing
actually reduces it and interfacial tension does both.

From Figs. 7.3.(2A-2I), two patterns seem to emerge, i.e. the one with the biggest gap width (2 inches) shows the lowest HTU (higher NTU) at a flowrate of 200 litres/hr, while the other two gap widths have their lowest NTU at about 150 litres/hr. Hence it can be said that the column has its highest efficiency at a flowrate range of 150-200 litres/hr. This is understandable, as a low flowrate creates a smaller interfacial area and a too-high flowrate would reduce the residence time considerably. An increase in flowrate would increase the effect of axial mixing too. Overall speaking, the efficiency is determined by a combination of factors and the prominence of such factors depends on the flow rate and the physical structure of the column.

The overall mass transfer coefficient (Ka)_{cA} is an 'apparent measured' one. From Figs. 7.3.(2J-2M), it can be seen that, for the carbontetrachloride system, the (Ka)_{cA} increases with the flowrate. For a fixed throughput, the variation in (Ka)_{cA} due to various gap widths and plate spacings is relatively small and hence has approximately constant value. However, the (Ka)_{cA} value in the MIBK system varies much more considerably with the throughput and the spacing. This difference in behaviour is largely due to the diffusivity of the liquid phases.

9.3.2. Sectional values of HTU, NTU and (Ka)_{cA}

With the aid of a computer program, the sectional values of HTU, NTU and (Ka)_{cA} were calculated, and then their algebraic sum was established. The sectional values were so-called because they referred to the various sections
between the sampling points.

The overall NTU is given by the following equation:

\[
NTU_{CA} = \int_b^t \frac{dC_y}{C_y^* - C_y} = \sum_{r=0}^{r=n} \int_r^{r+1} \frac{dC_y}{C_y^* - C_y}
\]

The calculation of each individual integral, corresponding to the sectional value of NTU, was based on the method of the Simpson's rule and it is the area beneath the curve obtained by plotting \(1/(C_y^* - C_y)\) against \(C_y\). Each section was divided into 201 equal strips and in carrying out such computation, an assumption was made that the concentration profile across each section was approximately linear. This point was borne out by the experimental data and subsequent calculation also confirmed this. The sectional values indicated a high extraction efficiency at the entrance of the feed (in this case it is the top of the column). This is understandable as the incoming feed has a much higher concentration gradient at the initial stages of the extraction compared with the lower section. In addition, the breakup of the dispersed phase and the oscillation of the droplets also play a key role in this aspect. On moving down the column, the concentration gradient is somewhat reduced and apart from that, the overall effect is also influenced by factors such as increase in interfacial area and coalescence. However, the individual effects are difficult to assess.

9.3.3. Experimental \(K_c\) v. Semi-theoretical \(K_c\)

A comparison was made of experimental \(K_c\) with various \(K_c\) values obtained by different theories. Experimental \(K_c\)
was so-called because it was calculated by using the experimentally measured \((K_a)\), and 'a', which itself was evaluated by photographic study. The results are shown in Table 8.1.(1)

9.3.3.1. Rigid sphere model

In analysing the present results, this model was not used due to the anticipated size of the droplets. It was suspected that they would be above the critical size of rigid spheres and these suspicions were subsequently confirmed by the visual observation of actual drops and on calculations based on the experimental results. The large size of the droplets was principally due to the lack of the break-up process by any mechanical device.

The difference between rigid and liquid droplet has long been a very important research topic, as these two droplets give different residence times in the system. Previous investigations were conducted by Hadamard(H 10), Rybczinski(R 3) and Boussinesq(B 7). Hadamard & Rybczinski's results were known as a circulation model, which is basically a Stokes' law with a viscosity factor, while Boussinesq model was similar but with a further "surface viscosity" factor.

From these results, it can be seen that the question of whether the drops are in fact rigid or not, and hence the possibility of having internal circulation, is itself an interesting and important one. This problem was studied by Bond & Newton(B 6) in 1928 and they proposed the following correlation:

\[
r_{\text{crit}} = (\gamma / \Delta \rho g)^{1/2} \quad \text{for } N_{Re} < 1, \text{ and}
\]

\[
N_{Re} = \frac{d_e U_{\text{target}}}{\nu_c}
\]
where \( r_{\text{crit}} \) is the critical radius, below which the 'particle' behaves almost like a rigid sphere.

In other words, internal circulation starts to set in when the critical radius is exceeded. However, to apply the criterion proposed by Bond & Newton, two factors have to be considered very carefully. One is the interfacial tension and the other is the Reynolds number. The interfacial tension value quoted by Bond & Newton refers to a system without mass transfer and it should be noted that its value in a system involving mass transfer process is quite different altogether. However, it is known that the droplets circulate less readily the higher the interfacial tension of the system.

The significance of the Reynolds number lies in the fact that it can indicate the transition from stagnancy to internal circulation, and its relation with the physical properties of the system was investigated by Garner & Skelland (11). The Reynolds number at which circulation begins is generally known as Transition Reynolds number and is given by:

\[
N_{\text{ReT}} = 100 \nu^{-4/3}
\]

where \( \nu \) is the kinematic viscosity of the continuous phase in centistokes.

For the carbon tetrachloride-acetic acid-water system:

\[
r_{\text{crit}} = \left( \frac{26}{0.59 \times 981} \right)^{1/2} = 0.2119 \text{ cm}
\]

Typical \( r_{\text{expt}} \) for run 1 (Table 7.3(1A)) = 0.23881 cm

\( r_{\text{expt}} \) for run 4 = 0.15593 cm
N_{ReT} = 100(1)^{-4/3} = 100

Experimental N_{ReT} = U_{t}d_{p}/U_{c}, where U_{t} is the terminal velocity of the particle obtained by the correlation proposed by Klee & Treybal (K 12). That is:

\[ N_{ReT} = 17.299x(0.23881x2)x1/0.0103 \]
\[ = 802.169 \text{ for run 1, and} \]
\[ N_{ReT} = 523.773 \text{ for run 4.} \]

For the MIBK/Acetic Acid/Water system:

\[ r_{crit} = (6/0.1792x981)^{1/2} = 0.1847 \text{ cm} \]

Typical \( r_{expt} \) for run 1 = 0.17565 cm
\( r_{expt} \) for run 4 = 0.1555 cm

\[ N_{ReT} = 9.517x0.3513x1/0.0103 = 324.59 \text{ for run 1, and} \]
\[ N_{ReT} = 9.517x0.3111x1/0.0103 = 287.45 \text{ for run 4.} \]

The above calculation indicates that the critical radius cannot be used as an overriding factor for stagnancy. Instead, the transition Reynolds number should be used to supplement the argument and give an approximate result. It should be noted that all the above results have ignored the purity of the system, which has been proved by Edge & Grant (E 6) to be a very important factor and its influence on the terminal velocity and frequencies of oscillation was illustrated.

9.3.3.2. Penetration (H 12) and other theories
From Figs. 9.3.3.2.(1A-1D)(data taken from Table 8.1.(1A-1D)), it can be seen that the experimental overall $K$ (i.e. $K_c^*$) is considerably less than the other three semi-theoretical values ($K_{c2}^*$, $K_{c3}^*$, $K_{c4}^*$). However, they do show the same trend. This shows that the extent of internal circulation within the droplets is less than that predicted by these theories, and the turbulence contributed by the circulation was not as great as Sivaraman argued in his theory. Strictly speaking, his correlation was based on the single file drops, and in the present study this was not the case. Apart from that, the distribution coefficient between the two phases plays an important part too. For a linear equilibrium curve, this was a simple matter, but complication arises for the case of non-linearity. Undoubtedly, the degree of error involved depends on the choice of the gradient along the equilibrium curve, which represents the distribution coefficient. Ultimately, this affects the accuracy of the overall coefficient too as these two are linked by the following equation:

$$\frac{1}{K_c} = \frac{1}{K_c^*} + \frac{m}{k_d}$$

Other factors such as the temperature changes and condition at the interface (interfacial turbulence) can also affect the accuracy to a certain extent.

For the carbon tetrachloride-acetic acid-water system, the slope $m$ is small in the experimental range, since at equilibrium a low dispersed phase concentration corresponds to a large continuous phase concentration. Hence the dispersed phase resistance controls.

$K_{c4}^*$ in the table (Table 8.1.(1)) was obtained by using Ruby-Elgin (for swarms of drops) and Hanlos-Baron equations
(for fully developed internal circulation). Again, its value is substantially higher than the experimental value, but shows the same trend. One of the possible sources of error is the using of $U_s$, the slip velocity between the two phases. Under certain circumstances, it can be substituted with the terminal velocity of the droplets.

The previous section (Table 7.3.1A) shows that the values of $(Ka)_c$ increase with the flowrate. However, Fig.9.3.3.2.1A indicates that the value of $K_c$ decreases with the flowrate. This phenomenon illustrates how the interfacial area, $a$, plays such an important part in mass transfer. Its effect overshadows the other factors to a great extent.

The discrepancy in Figs.9.3.3.2.1 lies in the fact that all the theoretical curves were based on the studies of single drops and the present study is concerned with a swarm of droplets.

Fig.9.3.3.2.2A and Fig.9.3.3.2.2B show the effect of the initial concentrations (from 11 gm/litre to about 40 gm/litre) on the Ka values. This set of data was obtained with constant flowrates, gap width and spacing between the baffles. As the interfacial area 'a' is constant in this case, it can be said that $K$ varies linearly with the concentration under such situation. $Ka$ (Fig.9.3.3.2.2A) increases with increase in initial solute concentration.

This phenomenon can possibly be explained in terms of the high degree of convection, which was produced by the high driving force, i.e. the large difference in concentration-density.

9.3.4. Mass transfer with axial mixing
9.3.4.1. Consideration of the Peclet number

It is known that mass transfer coefficients determined in model equipment, without consideration of longitudinal mixing, do not match the coefficients determined in large equipment. Hence, the scale-up of the extractors becomes a difficult task.

It has been concluded that the industrial column has to be considerably taller to allow for the inefficiencies introduced by longitudinal mixing. Parameters such as the Peclet number, \( N_{pe} \), and the dispersion coefficient \( E_0 \) of the dispersion model, in no way describe fully the actual nature of the process, as they are used mainly to specify the physical and hydrodynamical condition of the system and do not refer directly to the mass transfer process. It is true that the creation of the concentration jump at the inlet of the phases and the longitudinal transport of the solute would decrease the mass transfer driving force and hence reduce the efficiency of the extraction process. Even though a qualitative representation of the degree of mixing is given by the Peclet number, its magnitude unfortunately does not give a direct indication of the level of reduction in extraction.

One interesting point deserving attention is the comparison of the Peclet number obtained in the present study with the ones quoted by Clare (C 13). Their magnitudes are of the same order, i.e. the degree of mixing in these two systems would appear to be similar. This is surprising, in view of the different internal construction of the two contactors, as Clare's contactor has a mechanical device and the contactor of the present study hasn't.
This is possibly due to the fact that, since the sizes of the two columns are virtually identical, the stagnant regions are equal in dimensions. That leaves only the comparison between the effects due to the oscillating baffles in Clare's study, and the static baffles plate in the present, and it is very likely that these two external media created the same degree of mixing. However, this degree of mixing should not be used to correlate the effect on mass transfer efficiency as the artificial turbulence created in Clare's study would contribute a large proportion of mass transfer, which is independent of the axial direction. Another comparison can be made by observing the results of the axial mixing study of the perforated plate tower.

Treybal (T 1) and Krishnan (K 13) reported that, for a small diameter laboratory-type column, axial mixing in the dispersed phase could be assumed to be absent and complete mixing exists in the continuous phase flow. The low value of the Peclet number obtained in the present study indicated that the hydrodynamic mixing pattern in the fixed baffle contactor is quite similar to the one of the perforated plate tower. On the other hand, if the fixed baffle contactor is considered to be a series of mixing stages, then it should be possibly compared with the RDC instead.

9.3.4.2. Analysis of the One-Dimensional Diffusion Equation (eq. 3.94)

Various mathematical models for describing mass transfer with longitudinal mixing in countercurrent extraction column have been developed by different workers. These models are the stage model, the Backflow model and the One-
Dimensional Diffusion model. However, all these models have their own advantages and disadvantages. Recent experimental work\((0.3)\) and \((10)\) has shown that neither the Backflow nor the Diffusion models could describe the longitudinal mixing in the continuous and dispersed phase adequately, especially in a mechanically agitated system which has no or little coalescence and redispersion. Another phenomenon affects the residence time distribution of the dispersed phase is the forward mixing, which is due to the drops of different sizes having different velocities.

Irrespective of its shortcomings, the diffusion model (eq. 3.94 or eq. 3.95) is still being widely used, albeit with its validity being constantly questioned. The solution of such diffusion equation (eq. 3.94) has been tackled by various workers. For example, Smoot & Babb (8) tried to solve the problem utilizing the differential equation for both phases and the equilibrium distribution curve where the gradient \(M\) was taken as constant, rearranging the resulting equation in dimensionless form and finally obtained the subsidiary equation:

\[
M \left[ M^3 + \left\{ (N_{Pe})c - (N_{Pe})d \right\} M^2 - \left\{ (N_{Pe})c (N_{Pe})d + NTU_{oc} (N_{Pe})c + NTU_{oc} (N_{Pe})c (N_{Pe})d (1 - R) \right\} \right] = 0
\]

If \(M_1, M_2, M_3, M_4\) are roots of the above equation, which can be evaluated by trial and error, then:

\[
C_y = A_1 e^{M_1 Z} + A_2 e^{M_2 Z} + A_3 e^{M_3 Z} + A_4
\]

where \(C_x = MC_y\),

\(R\) is the extraction factor, i.e. \(U_y M / U_x\),
CY is the dimensionless concentration of the aqueous phase, \( \frac{C_y}{C_y \text{ inlet}} \).

\( A_1, A_2, A_3, A_4 \) are constants which can be determined with the aid of the appropriate boundary conditions, and

Z is the dimensionless height from organic inlet nozzle.

The chief criticism of the Smoot & Babb's work is the lack of the derivation of the subsidiary equation, and again the main limitation of its applicability is the assumption of the constant value of the distribution coefficient. This can not be so for a great many systems.

Other workers such as Hartland & Mecklenburgh (H 15) and Miyauchi & Vermeulen (M 9) have all attempted to solve this equation (eq. 3.94) and analytical solutions (but not experimental data) were given for linear and special cases. Approximate calculation methods for the models were proposed in terms of the actual and apparent height of transfer units. Rod (R 9) also proposed a very good method of calculating the effects of dispersions for the diffusion model, provided the concentration of the dispersed phase can be measured experimentally.

Diffusion model can be used to describe the present system in view of the fact that, (i) it is not mechanically agitated, (ii) certain degree of coalescence and redispersion does take place.

The low value of the Peclet number indicated that there is a degree of mixing. This seems reasonable as similar phenomenon occurs in a perforated plate tower.

Another point deserves special attention is the
quantity $E_c d^2 C_y / dh^2$ itself in the one-dimensional diffusion equation (eq. 3.94). This quantity has a unit of mass per unit volume per unit time and theoretically, it is the mass flux due to the axial mixing in a mass transfer situation. However, judging by the results obtained (Table 9.3.4.2.1), it seems the whole situation is more complicated than this.

As suggested by Smoot & Babb (S8) who presented a similar argument in their research, the quantity $E_c d^2 C_y / dh^2$, they called a correction term, can be zero, positive or negative depending on the value of the Peclet number of both phases, the extraction factor and the magnitude of the NTUc.

9.3.5. Mass transfer under various regimes

9.3.5.1. Mass transfer during drop formation and coalescence

Mass transfer during drop formation is a very complex process and it is influenced by many phenomenon. The overall mass transfer coefficient during drop formation ($K_d$)$_f$ is related to the flux, and its average value is taken over the time of drop formation $t_f$. Hence the time of formation itself is a very important factor. However, because of the complexity of the process, the values of various parameters are only rough estimates.

Assuming the ideal case, the drop comes down to a plate, coalesces with the layer, then travels a distance equal to the length of the plate and then finally breaks off. The time it has taken to perform such activities could be considered as the time of formation $t_f$.

The individual coefficient during drop formation $k_{df}$ is given as:
The constant is normally in the range of 1.3 to 1.8 and 1.5 was taken as it is the arithematic mean of the two. However, its choice is not unquestionable as there is possible existence of interfacial turbulence in the system (T 9). The overall coefficient $K_{df}$ is then given by:

$$\frac{1}{K_{df}} = \frac{1}{k_{df}} \left[ 1 + \frac{1}{m} (D_d/D_c)^{0.5} \right]$$

where $m$ is the experimental equilibrium distribution coefficient.

For the present research work, the 'linearized' value of $m$ is taken as 6.24 over the experimental range. Another point should be noted is that in the above equation, it is assumed that the mechanism of mass transfer is the same for both sides of the interface.

Mass transfer during coalescence is very difficult to be measured experimentally and results are difficult to reproduce. One of the approximate relationships (T 9) is given as:

$$K_{dc} = 0.1 K_{df}$$

9.3.5.2. Mass transfer during the motion of the drops

Because of the droplets in the present experimental work were fairly large, and they tend to oscillate during the motion, the surface-stretch theory (A 4) was employed, i.e.

$$k_{dr} = \sqrt{\frac{4D_d}{\pi} \left( 1 + \frac{1}{3} \delta^2 \right)_w}$$
where \( \delta \) is the dimensionless amplitude factor and it can be taken as 0.2(T 9), and the oscillation frequency, \( w \) is:

\[
w = \frac{1}{2\pi} \sqrt{\frac{192 \delta \varepsilon \sigma d_p^{0.225}}{(3\rho_d + 2\rho_c)d_p^3}}
\]

The \( K_{Dr} \) is calculated by a similar relationship:

\[
\frac{1}{K_{Dr}} = \frac{1}{k_{dr}} \left[ 1 + \frac{1}{m} \left( \frac{D_d}{D_c} \right)^{0.5} \right]
\]

and \( m \), the 'linearized' experimental equilibrium distribution coefficient of the present work, is taken as 6.24 as before.

Again, it is assumed that the mechanism of mass transfer for the liquids on both sides of the interface is the same. The results are shown fully in Table 9.3.5.(l).

9.4 Hold up

The hold-up measured manometrically(experimental) was an operating dynamic or 'effective' hold up. In addition, there was a hold up of the liquid film(coalesced liquid drop) on each baffle surface. This is a static hold-up which makes no contribution to the manometric head. It appears in most cases to be less important than dynamic hold up, despite the fact that it does provide interfacial area for mass transfer. The mass transfer to this film appears to be much less than in other parts of the extraction column. This is supported by the observations of Laddha(L 16) on the performance of sieve plates—although this is not strictly analogous to the present case.
Fig. 7.4.(1A) shows an interesting difference in hold up between the two systems, one with mass transfer and one without. At low flowrates, the difference in hold up between these two systems is not very apparent, but the hold up increases more sharply for the system without mass transfer as the flowrate increases. This phenomenon is largely due to the decrease in interfacial tension due to the presence of the solute— a decrease from 47 to 26 dyne/cm. This low interfacial tension promotes deformation of the droplets and consequently its low hold up. The explanation of such phenomenon is as follows: The system without solute would produce spherical droplets and as soon as solute is added, the spherical tendency is partially destroyed. This gives more highly deformed, elongated and necking drops, and protuberances and cell-like surface structures resting on the plate. Subsequently, the operating hold up is drastically reduced. Such condition is enhanced further by high flowrates of the phases.

Fig. 7.4.(1B) shows the combined effect of the spacings between the baffles and gap width on the hold up for a fixed throughput. It also shows the considerable effects of these two constructional factors. In general, it can be said that the hold up increases with the gap width and decreases with spacings, while the other factors are kept constant. This is understandable as these two parameters have a direct influence on the drop sizes, and they can be considered as the equivalent droplet-maker such as the stirrer or rotatory baffle. For a constant throughput, a decrease in the magnitude of these two parameters (gap and spacing) decreases the mean drop sizes (as the frequency of the
break-up processes increases) which increases the mean residence time due to the lower of the terminal velocities. This affects the hold up directly and hence its increase.

Figs. 7.4. (1E, 1F, 1G) (CCl$_4$/acetic acid/water) show the relationship between $(Ka)_{cA}$ and $\phi$. Many more points would be of advantage but the additional experimental work would be very considerable with doubtful immediate benefit. The curves obtained are reasonable and they indicate that increase in hold up provides increase in interfacial area and hence larger values of $(Ka)$. However, there is a limit to which the value of 'a' can be increased and there is also a limit to any increase in $K_c$ in which case the curves approach the asymptotic values for $(Ka)$.

Fewer results were obtained for $\phi$ for the system MIBK/acetic acid/water (Fig. 7.4. (1D) and Fig. 7.4. (1H)) than for the CCl$_4$ system. The fractional hold up is much less for the MIBK than the CCl$_4$ system due to the considerable difference in the surface tension values and the $(Ka)_{cA}$ values are at least an order of magnitude less for CCl$_4$/acetic acid/water than MIBK/acetic acid/water at the same spacing and gap values.

9.5 Photography and counting

The size of the dispersed phase droplets, which is expressed in terms of the Sauter mean drop diameter, was determined by the photographic method. The Sauter mean drop diameter was used to find the interfacial area available for the mass transfer process.

Some typical results of the carbon tetrachloride–acetic acid–water system are shown in Fig. 9.5. (1A) and Fig. 9.5. (2A). All the values of $d_{32}$ were determined at a
distance of about three quarters way down the column. This position was chosen because of its convenience and it was found that the size of the droplets was relatively uniform throughout the main part of the column. Fig. 9.5.(1A) shows a family of curves of $d_{32}$ against flowrate under the condition of the fixed gap width and variable baffle spacing. The pattern obtained was expected as the impact exerted on the dispersed phase for a fixed flowrate would be greater in the case of a column with bigger baffle spacing and hence it produces smaller drops.

For a higher flow rate, smaller droplets would be produced and in this case the controlling factor is the higher shear between the two phases.

Fig. 9.5.(2A) is a plot of $a'$ against the flowrate, and $a'$ itself is obtained by using the formula $a = 60/d_{32}$. It is interesting to note that $a'$ increases with flowrates in an approximate linear fashion, under the condition of fixed gap width and variable baffle spacing. The family of curves shows that, for a constant flowrate, a smaller baffle spacing gives a larger value of $a'$ as the dispersed hold up would be higher in this case. The bigger value of $d_{32}$ obtained under such situation would be offset by the product of $60$.

The droplets were sized both horizontally and vertically, and the average was taken to reduce the errors. The droplets in the present study were relatively large and it is fairly safe to assume that the error involved was quite small. However, the chief source of error was due to the coalescence of the droplets. This effect of interaction is the main difference showing on the photograph between the
9.6 End effects

It is a well known fact that the formation of a concentration jump on the inlet of the phases, known as 'end effects', has a detrimental influence on the mass transfer efficiency as it decreases the mass transfer driving force. So these effects are one of the unwanted, limiting factors for the mass transfer.

One of the end effects such as 'Bottom effects' can be defined as the following: (Ref. Fig. 4.3)

\[
B^* = 100 \left( \frac{C_{x_a} - C_{x_B}}{C_{x_T} - C_{x_B}} \right)
\]

where \( C_{x_a} \), \( C_{x_B} \) and \( C_{x_T} \) are the concentrations of the dispersed phase at the position \( a \), bottom and top respectively.

Of course, other quantities such as HTU can be used to represent too.

The results (Table 7.6.1) of the end effects of the present study show a similar pattern as the spray column ((G 1), (G 3)), i.e. it has higher degree of end effects at the bottom (entrance of continuous phase) than the top (dispersed phase entrance). This high bottom effect indicates a high degree of mixing, which was proved independently by the low value of the Peclet number obtained during the residence time study. Again, this depicts a picture of deviation from plug flow. Another factor which makes certain contribution to the bottom effect is the
coalescence of the droplets at the accumulated bottom pool. However, the extent of the contribution is difficult to be quantified.

The smaller top effects (end effect at the dispersed phase inlet) were probably due to the actual increases in the rate of mass transfer caused by the process of droplet formation, instead of the mixing effect in the dispersed phase. Also, the effect of coalescence was absent.
Chapter 10
Conclusion
10.1

The present research can be considered as one of the series of projects carried out by Thomas & Co. (e.g. T 3), but with different apparatus. In other words, it is an extension of the previous studies. The experimental work was centred on a six-inch diameter column with fixed baffles and various parameters such as the size of the baffles and spacing between them were investigated.

10.2

Equilibrium distribution of acetic acid in the carbon tetrachloride-acetic acid-water and MIBK-acetic acid-water systems were again determined experimentally and this gives further substantiation to the results quoted by previous workers. Interfacial tension measurements were also repeated.

10.3

Flooding point of the Fixed Baffle Contactor (FBC) was evaluated under various operating conditions and the influence of the physical properties of the system such as with and without solute was made clear.

10.4

Axial mixing of the continuous phase in the column under various arrangements of the baffles was studied. The axial mixing coefficient and its related parameter such as Peclet number, mean residence time and variance were calculated. The effects of the flowrate on these parameters were established and a general discussion of the validity of the $N_{Pe}$ to describe mixing in the apparatus was also given.
The extraction performance, i.e. efficiency of the contactor was evaluated in terms of the overall NTU, HTU and \((\text{Ka})\) of both continuous and dispersed phases. Sectional values of these quantities were also calculated for the sake of analysis. End effects of the column were also estimated.

Simultaneously, the surface mean diameter of the drop swarms was determined photographically for various flow conditions. With the aid of the hold up of the dispersed phase, its value was used to calculate the interfacial area of the droplets and subsequently the \((\text{K})_c\). The experimental \((\text{K})_c\) was compared with various \((\text{K})_c\) obtained by different theoretical models. An attempt was made to explain the discrepancy and the difference between them. In calculating various \((\text{K})_c\), the gradient \(m\) of the equilibrium curve was taken as a constant over the experimental range.

The effect of the axial mixing on the mass transfer process was investigated, utilizing the One-Dimensional equation. The quantity \(E_c d^2 C_y / dh^2\) can be viewed as an indication of the degree of mixing, even though the One-Dimensional equation is inadequate for describing the process fully.

The results of the extraction studies in the fixed baffle contactor were comparable for the same systems (\(\text{CCl}_4\)-acetic acid-water and MIBK-acetic acid) with those obtained with some mechanically agitated extractor ((T 5) (T 8) (T 10)). Very probably this would not be true for
other chemical systems which are more viscous and with different surface tension properties. However, the fixed baffle contactor (FBC) is more widely used than is generally appreciated especially in cellulose acetate rayon plants, hardwood carbonization plants and food industries. (V 1).

10.8

The experimental work described is the first to be reported on a simple fixed baffle extractor. Very few performance figures are given anywhere and then they are restricted to extraction duty without any means of a detailed analysis to show how the mechanisms of transfer take place. In this sense the thesis is definitive. The complexities of the mechanisms become clearer and although it is still not possible to proceed to large scale design directly, it opens up the subject and forms the foundation for further work and a better understanding of the behaviour of the fixed baffle extractor.
NOMENCLATURE

A  a constant
a  interfacial area  ft²/ft³
B  bottom effect
C  concentration  g.mol/litre, gm/litre
C* equilibrium solute concentration  g.mol/litre, gm/litre
C₀ initial concentration  g.mol/litre, gm/litre
Cₑ dimensionless concentration
C(t) C diagram, fraction of material in exit stream at any time t after injection
D  molecular diffusion coefficient  ft²/sec
d  diameter  ft
\(d_{32}\)  Sauter mean drop diameter  ft
E  axial dispersion coefficient  ft²/sec
E(t) E diagram, fraction of material in exit stream at any time t after injection (external age distribution function)
e  void fraction based on total column volume
F  force  dyne, lb_f, N
F(t) F diagram, cumulative fraction of material which has spent a time less than t (cumulative distribution function)
f  fractional conversion
f  friction factor
f  function
G  gap width  inch
g  acceleration due to gravity  ft/sec², cm/sec²
\(g_c\) conversion factor  32.174 ft.lbm/lb_f.sec²
H  holdback
H  height of a column  ft, cm
HTU  height of a transfer unit            ft
h  height                ft, cm
hm  manometer reading            ft, cm
Δh  difference between levels connecting manometer arms            ft, cm
I(t)  I diagram, fraction of material in reactor at any time t after injection (internal age distribution function)
K  overall mass transfer coefficient            ft/hr, cm/sec
Ka  overall mass transfer coefficient with interfacial area            hr⁻¹, sec⁻¹
k  first order specific reaction rate            sec⁻¹
k  film mass transfer coefficient            lb mole/hr(ft²)(unit mole fraction),            ft/hr
L  characteristic length            ft, cm
L  distance of fall of drop            ft, cm
m  equilibrium distribution coefficient, dC_y/dC_x
N  number of constituent components
NTU  number of transfer units
n  number of particles
P  pressure            N/m², lb_f/ft²
Q  phase volumetric flowrate            litre/hr
r  radius of a particle            ft, cm
S  cross-sectional area            ft², in²
S  spacing            inch
s  film thickness            ft
T  temperature            deg.C
T*  top effect
T  time            sec
t  residence time  sec
U  superficial velocity  ft/sec
U_s  slip velocity or relative velocity of the drop phase  ft/sec
U_t  terminal velocity of a drop  ft/sec
u  velocity or relative velocity  ft/sec
V  volume of a reactor  ft^3
v  volume of a droplet  ft^3
w  oscillation frequency (equation 3.66)  sec^-1
X  quantity defined in equation 3.98
x  distance in x direction  ft, cm
x  mole fraction (solute concentration in dispersed phase)
Y  quantity defined in equation 3.99
y  mole fraction (solute concentration in continuous phase)
Z  dimensionless length
Z  dimensionless height
z  length or height  ft, cm

Greek symbols

\( \alpha \)  activity  g.mole/litre, g.ion/litre
\( \beta \)  a parameter accounts for the degree of purity of the system (Section 3.5.2. (W 3))
\( \gamma \)  activity coefficient
\( \sigma \)  interfacial tension  dyne/cm
\( \delta \)  amplitude factor (equation 3.66)
\( \theta \)  dimensionless time
\( \kappa \)  viscosity ratio (\( \mu_d/\mu_c \))
\( \mu \)  viscosity  lb_m ft^-1 sec^-1, poise, cp
\[ \mu \] mean or centroid of the \( G(t) \) or \( E(t) \) curve  
\[ \mu \] mean residence time  
\[ \phi \] kinematic viscosity (viscosity \( \mu \)/density \( \rho \))  
\[ \rho \] density  
\[ \sigma^2 \] variance  
\[ \sigma^2_{\text{out}} \] variance at the output position  
\[ \sigma^2_{\text{in}} \] variance at the input position  
\[ \sigma^2_{\theta} \] dimensionless variance  
\[ \phi \] number of phases  
\[ \phi \] correction factor (Fig. 4.11)  
\[ \phi \] hold-up

**Dimensionless groups**

\[ N_{Fr} \] Froude number \( u^2/gL \) (for flow in a circular tube, \( L \) is usually taken to be the tube diameter)

\[ N_{Re} \] Reynolds number \( uL\rho/\mu \)

\[ N_{Rep} \] particle Reynolds number \( u_d\rho_e/\mu_c \)

\[ N_M \] Morton number \( g\mu_\epsilon^4\Delta\rho/\rho_c^2 \sigma^3 \) (Section 2.2.4)

\[ N_{Eo} \] Botvos number \( g\rho \sigma d_e^2/\sigma \) (Section 2.2.4)

\[ N_{Pe} \] Peclet number \( uL/E \)

\[ N_{Sc} \] Schmidt number \( \mu/\rho D \)

\[ N_{Sh} \] Sherwood number \( kL/D, k_d/\rho D \)

\[ N_{We} \] Weber number \( u^2d_e\rho_e/\sigma \)

\[ N_{Wer} \] rotational Weber number \( N^2L^3\rho_c/\sigma \) where \( N \) is the rotor speed and \( D \) is the impeller diameter (Section 2.2.3)
<table>
<thead>
<tr>
<th>Subscripts</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>apparent measured value</td>
</tr>
<tr>
<td>c</td>
<td>continuous phase</td>
</tr>
<tr>
<td>c</td>
<td>coalescence</td>
</tr>
<tr>
<td>d</td>
<td>dispersed phase</td>
</tr>
<tr>
<td>d</td>
<td>droplet</td>
</tr>
<tr>
<td>e</td>
<td>droplet</td>
</tr>
<tr>
<td>e</td>
<td>volume-equivalent sphere</td>
</tr>
<tr>
<td>f</td>
<td>formation</td>
</tr>
<tr>
<td>l</td>
<td>linear plug flow</td>
</tr>
<tr>
<td>LM</td>
<td>log mean value</td>
</tr>
<tr>
<td>m</td>
<td>measured value</td>
</tr>
<tr>
<td>o</td>
<td>overall</td>
</tr>
<tr>
<td>p</td>
<td>particle</td>
</tr>
<tr>
<td>r</td>
<td>rise or fall(motion of drops)</td>
</tr>
<tr>
<td>T</td>
<td>true value</td>
</tr>
<tr>
<td>T</td>
<td>transitional value</td>
</tr>
<tr>
<td>x</td>
<td>dispersed phase (organic phase)</td>
</tr>
<tr>
<td>y</td>
<td>continuous phase (aqueous phase)</td>
</tr>
<tr>
<td>Θ</td>
<td>dimensionless</td>
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REFERENCES

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Appendix 1

A.1 Interfacial turbulence

A.1.1. Brief account of the mechanism of eruptions

Consider an eddy reaches the interface from the raffinate (dispersed) phase of a two phase system, which has a characteristic of the interfacial tension decreasing with solute concentration. The interfacial tension of the affected region will be lowered, if surface excess concentration is established there practically instantaneously. The next thing happens is the expansion of the affected area peripherally and liquid of lower subinterfacial concentration is brought to the spot. This results in higher interfacial tension at the region than at the periphery. With the compression and dilatation effect, a gradient of interfacial tension is produced and acting in the same direction. The induced gradient of the interfacial tension together with the viscous friction will soon overcome the momentum of the initial motion and the direction of motion will be reversed.

The flow towards the centre will produce a jet-like ejection of material rich in solute into the bulk of the extract phase. Similar ejection into the bulk of the raffinate phase will occur. This phenomenon, known as eruption, represents the final stage in the mechanism of a transient disturbance with no eruption of the interface.

A.1.2. Concentration dependency

Apart from localized forced convection, interfacial disturbance can also be produced by high concentration difference, and hence this gives rise to the concentration dependency of the phenomenon. The transfer of the solute
from the organic phase into the aqueous phase represents, in terms of the Sternling-Scriven criteria, a stable direction of transfer. Similarly, a system is unstable for the transfer of the solute from water into the organic phase. For a transfer in the stable direction, high concentration is required for the initiation of the disturbances. This point was made clear by Sawistowski (13) and further confirmation can be seen from the results of the present work (Figs. A.1.2.1A to 1D). These results give no visible disturbance and all their concentrations were less than 10 gm/litre. As soon as the concentrations were increased, visible disturbances and 'kicking' were detected. (Figs. A.1.2.1E and 1F)

Of course, the interfacial turbulence still existed in the case of the low concentration, even though it was invisible. Hydraulically speaking, the process of mass transfer then takes place under a transition regime at the interface. Transition regime, as its name suggests, is the region separating the diffusional and turbulent regimes.

The presence of the interfacial turbulence, as in the case of the high initial concentration, is the characteristic feature of the turbulent regime. Under such regime, the rate of mass transfer is governed mainly by the intensity of the spontaneous interfacial convection and less so by the hydrodynamic conditions of the bulk phases.

A.1.3. The relationship between the final concentration, the interfacial tension and the time

The values of the interfacial tension and final concentration under turbulent regimes were calculated by
means of the Andrea's formula and they are displayed in Figs. A.1.3.(1A & 2A). The work was carried out with the initial concentration of the acid ranging from 10 to 50 gm/litre, i.e. it was above the transition regime. These two curves can be considered simultaneously, as they are connected reciprocally.

Consider the curve of the interfacial tension against time. Theoretically, the interfacial tension should increase as the time proceeds, as this represents the decrease of the final concentration of the solute inside the droplet. However, the experimental curves indicate that there is a maximum on each curve and thereafter, the interfacial tension approaches a constant value.

A possible explanation for this phenomenon is the accumulation of foreign agents such as dust particles, dirt, etc., which serve as surface active materials. These films of surface-active materials have long been recognized as a barrier to mass transfer. The main effect of such surfactants on a system undergoing mass transfer is to reduce the interfacial tension and make it less sensitive to variations in solute concentration. The interfacial motion is also reduced due to the creation of resistance to motion at the interface.
Appendix 2

A.2 Distribution of a solute between two immiscible solvents. (P.3)

A.2.1. Dimerization of an organic acid in a nonpolar solvent.

Consider a solute \( s \) distributed between two liquid phases \( A \) and \( B \) and assuming that the mole fraction of the solute in either phase is very small, i.e. the two solutions are ideal. Then:

\[
\begin{align*}
f_s^A &= H_s^A x_s^A \\
f_s^B &= H_s^B x_s^B
\end{align*}
\]

(A.2.1.1)

(A.2.1.2)

where \( f_s \) is the fugacity of the component \( s \),
\( H_s \) is the Henry's constant for the solute \( s \),
\( x_s \) is the mole fraction of the solute \( s \) and
superscripts \( A \) and \( B \) represent phases \( A \) and \( B \) respectively.

The partition coefficient \( m \) is obtained by equating fugacities of the component \( s \) in the two phases and the resulting equation is known as Nernst distribution law,

\[
m = x_s^A / x_s^B = H_s^B / H_s^A
\]

(A.2.1.3)

where \( m \) is independent of composition under constant temperature and pressure.

Eq. (A.2.1.3) can be modified further as very small mole fraction \( x \) can be replaced by concentrations \( C \), i.e.
\[ m' = \frac{C_A^s}{C_B^s} = \frac{\rho_A x_A^s}{\rho_B x_B^s} \quad \text{(A.2.1.4)} \]

where \( \rho_A \) and \( \rho_B \) are the molar densities of phases A and B respectively, and for very small \( x_s \), \( \rho_A \) and \( \rho_B \) are the densities of the pure solvents.

However, it has been known in many cases that the experimental results did not obey Nernst's distribution law and such deviation may be attributed to chemical effects. One of such cases is the carbon tetrachloride - acetic acid - water system, in which the acid is distributed between the two immiscible solvents water and carbon tetrachloride under room temperature. The explanation for the failure of Nernst's law, in this case, can be found by taking into account the tendency of the acetic acid to dimerize in the nonpolar solvent carbon tetrachloride by forming hydrogen bonding:

\[
2\text{CH}_3\text{C}(-\text{OH}) \rightleftharpoons \text{CH}_3\text{C}\text{C}(-\text{OH})\text{CH}_3
\]

The situation can be visualized by the following diagram, Fig. A.2.1.1:

![Diagram showing the distribution of acetic acid between CCl₄ and water.](image)

Fig. A.2.1.1. Distribution of acetic acid between CCl₄ and water.
and the two equilibria are:

I. Phase-distribution equilibrium between the two phases:

\[
\text{Acid in water} \rightleftharpoons \text{Monomer acid in } \text{CCl}_4
\]

II. Chemical equilibrium in the \text{CCl}_4 phase:

\[
\text{Monomer acid} \rightleftharpoons \text{Dimer acid}
\]

For conditions I and II:

\[
\begin{align*}
\text{I: } & \quad m_I = \frac{C_{xM}}{C_y} \quad (A.2.1.5) \\
\text{II: } & \quad m_{II} = \frac{C_{xD}}{(C_{xM})^2} \quad (A.2.1.6)
\end{align*}
\]

where \(m_I\) and \(m_{II}\) are equilibrium constants, and subscripts \(xM\), \(xD\) and \(y\) stand for carbon tetrachloride monomer, carbon tetrachloride dimer and water respectively.

If the total concentration of the acetic acid in the carbon tetrachloride is \(C_x\), then:

\[
C_x = 2C_{xD} + C_{xM} \quad (A.2.1.7)
\]

Substituting equations A.2.1.5 and A.2.1.6 into A.2.1.7 gives:

\[
\frac{C_x}{C_y} = \frac{2m_{II}C_y + m_I}{m_I} = \frac{2m_{II}}{m_I} \frac{C_y}{C_y} + \frac{m_I}{m_I} \quad (A.2.1.8)
\]

The above equation indicates that the distribution coefficient \(m\) is not constant, contrary to Nernst's law, but varies in certain manner with the concentration of acetic acid in water.

For a system where there is a great tendency of the solute to dimerize, \(m_{II}\) is very large, i.e.
The numerical values of \( m_1 \) (dimensionless) and \( m_{II} \) (in units of litre/gm) for the present system (CCl\(_4\)-acetic acid-water) were obtained by plotting \( C_x/C_y \) against \( C_y \) (eq. A.2.1.8), using the experimental data taken from Chapter 5. They were found to be approximately 0.0014 and 51 respectively (Fig. A.2.1.(2A)).

Alternatively, the high degree of dimerization of the acetic acid in the carbon tetrachloride can be seen by observing the straight line in Fig. A.2.1.(3A), which was plotted according to equation A.2.1.9.

Substituting the values \( m_1 \) and \( m_{II} \) into eq. A.2.1.8 gives:

\[
\frac{C_x}{C_y} = 2(51)(0.0014)^2 C_y + m_{II}(0.0014)
\]

i.e. \( (C_y + 3.5)^2 = 5002(C_x + 0.0025) \) (A.2.1.10)

The parabolic shape of the eq. A.2.1.10 can be traced out as shown in Fig. A.2.1.(3B), which is virtually identical to Fig. 5.7a. In other words, the equilibrium curve can be obtained by considering the thermodynamics of the system. The calculated values of \( C_y \) (from eq. 5.1 of Chapter 5 and eq. A.2.1.10) are shown in Table A.2.1.

<table>
<thead>
<tr>
<th>( C_x ) (gm/l)</th>
<th>( C_y ) cal.1 (gm/l)</th>
<th>( C_y ) cal.2 (gm/l)</th>
<th>( C_x ) (gm/l)</th>
<th>( C_y ) cal.1 (gm/l)</th>
<th>( C_y ) cal.2 (gm/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.18</td>
<td>25.887</td>
<td>26.714</td>
<td>0.9159</td>
<td>61.887</td>
<td>64.277</td>
</tr>
<tr>
<td>0.3</td>
<td>34.035</td>
<td>35.398</td>
<td>1.332</td>
<td>75.638</td>
<td>78.202</td>
</tr>
<tr>
<td>0.4359</td>
<td>41.577</td>
<td>43.328</td>
<td>2.472</td>
<td>105.34</td>
<td>107.75</td>
</tr>
<tr>
<td>0.6319</td>
<td>50.728</td>
<td>52.832</td>
<td>3.276</td>
<td>122.49</td>
<td>124.55</td>
</tr>
</tbody>
</table>

\( C_y \) cal.1 from \( C_y = 64.87(C_x^{0.5357}) \)

\( C_y \) cal.2 from eq. A.2.1.10
Appendix 3  Program to calculate $\mu$ and $\sigma^2$

```
'REAL' INTO A, SUMX, SUMY, SUMXY, SUMSQ, SUMC, SUMCT, SUMCTS, K,
' =OFF, INTO, INTCT, INTCTS, P, Q, RATIO, SIGNI, PEC, UL, L
TRAP, B, TOP;
'INTEGER' M, N, SYST, I, J, D, R;
'REAL' 'ARRAY' C[0:300,1:6], F, G, 'REAL', SAP[1:6];
'INTEGER' 'ARRAY' 'INT' HEADFR, M;
'PROCEDURE' 'INT' 'IF' 'THEN' 'GOTO' 'PROCEDURE' 'EXTERN';
'PROCEDURE' 'OUTSTRING(A, M)' 'ARRAY' 'INTEGER' 'EXTERN';
'PROCEDURE' 'IF' 'THEN' 'GOTO' 'EXTERN';
'R: =0;
START: 'FOR' J :=1 'STEP' 1 'UNTIL' 6 'DO'
FOR 1:=1 'STEP' 1 'UNTIL' 300 'DO'
'REAL' C[1,J]: =0;
END;
'COMMENT' 'DATA FROM CARD WILL BE READ;
SFLFCTINPUT(3);
M:=1;
INSTRING(HEA, M);
N:=READ;
IF N LE 0 THEN 'GOTO' FINISH;
M:=1;
SFLFCTOUTPUT(2);
PAPERTHROW;
'WRITE' ('THE PROGRAM IS NOW STARTING TO PROCESS DATA', 1);
SPAC(1);
OUTSTRING(HEA, M);
'WRITE' ('DATA ON TAPE WILL BE READ IN AND TESTED FOR MAXIMUM', 1);
SFLFCTINPUT(1);
ALPHA: (I,J): READ;
TOP:=0;
FOO: 'FOR' J:=1 'STEP' 1 'UNTIL' 301 'DO'
'REAL' A:=READ;
IF A=ABS(A)
IF A>220 THEN 'GOTO' BETA;
IF I=300 THEN 'GOTO' GAMMA;
IF I=1 THEN R:=A "FALSE" C[I,J]: =A-B;
IF C[I,J] LE 0 THEN C[I,J]: =0;
IF C[I,J] GE TOP THEN 'BEGIN' TOP:=C[I,J];
TOP:=TOP+1;
'END';
BETA: 'LENGTH': M[I]=I-1;
'IF' J=1 THEN 'GOTO' DELTA;
J:=J+1;
'GOTO' ALPHA;
GAMMA: SFLFCTOUTPUT(2);
SPACE(5);
WRITE"('DATA ON TRACK')";
SPACE(1);
PRINT(J, 1, 0);
SPACE(0);
WRITE"('OF THE THIS RUN HAS MORE THAN 999 VALUES')";
NFILFCT(1);
```
DELTA: J:=1;

'COMMENT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

PHI: 'IF' LIMIT[J] < LIMIT(J) 'THEN' 'GOTO' PHIF;

••COMMT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

DELTA: J:=1;

'COMMENT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

PHI: 'IF' LIMIT[J] < LIMIT(J) 'THEN' 'GOTO' PHIF;

••COMMT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

DELTA: J:=1;

'COMMENT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

PHI: 'IF' LIMIT[J] < LIMIT(J) 'THEN' 'GOTO' PHIF;

••COMMT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

DELTA: J:=1;

'COMMENT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

PHI: 'IF' LIMIT[J] < LIMIT(J) 'THEN' 'GOTO' PHIF;

••COMMT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

DELTA: J:=1;

'COMMENT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

PHI: 'IF' LIMIT[J] < LIMIT(J) 'THEN' 'GOTO' PHIF;

••COMMT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

DELTA: J:=1;

'COMMENT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

PHI: 'IF' LIMIT[J] < LIMIT(J) 'THEN' 'GOTO' PHIF;

••COMMT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

DELTA: J:=1;

'COMMENT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;

PHI: 'IF' LIMIT[J] < LIMIT(J) 'THEN' 'GOTO' PHIF;

••COMMT' THIS LOOP TESTS DATA FOR CONVERGENCE TO INITIAL ZERO
MINIMUM PERMISSIBLE LENGTH AND MINIMUM DEFLECTION;
A CHECK FOR A SUFFICIENT NUMBER OF VALID TRACKS WILL BE

THETA: A:=0;
* FOR* J:=1 *STEP* 1 *UNTIL* N *DO*
* BEGIN* A:=A+FAIL[J];
* END*;

A:=A/N;
* IF* A<0.5 *THEN* *GOTO* LAMBDA;
*COMMENT* THE TAIL OF VALID DATA TRACKS WILL BE FITTED TO AN EX
DECAY AND THE POINTS SMOOTHED TO THE BEST INF;

J:=1;
MU:
SUMX:=SUMXY:=SUMSQ:=SUMY:=0;
* IF* FAIL[J]=1 *THEN* *GOTO* RHO;
* IF* J=N *THEN* *GOTO* SIGMA;
J:=J+1;
*GOTO* MU;

RHO: D:=0;
* FOR* I:=(TRACK[J]-8) *STEP* 1 *UNTIL* TRACK[J] *DO*
* BEGIN* cr[J]:=IF(TRAC[J]=1) THEN GOTO RHO;
J:=J+1;
END;

* BEGIN* cr[J]:=IF(TRAC[J]=1) THEN GOTO SIGMA;
J:=J+1;
*GOTO* MU;

LAMBDA: SELECT OUTPUT(2):
SPACE(5);
WRITETEXT("MORE THAN 50% OF THE DATA TRACKS ARE IN THIS RUN")
SPACE(1);
WRITETEXT("ARE IN VALID");
NEWLINE(1);
SPACE(5);
WRITETEXT("THE RUN HAS BEEN ABANDONED");
NEWLINE(1);
SPACE(5);
WRITETEXT("CONSULT");
NEWLINE(1);
*GOTO* start;

SIGMA: J:=1;
*COMMENT* THE MFAN AND VARIANCE OF THE VALID RESPONSE CURVES
WILL BE CALCULATED USING THE SATER CORRECTION;

CHI; * IF* FAIL[J]=1 *THEN* *GOTO* TAU;
MFAN[J]:=0;
VAR[J]:=0;
J:=J+1;
* IF* J=N *THEN* *GOTO* PT *ELSE* *GOTO* CHI;
TAU: SUMC:=SUMCT:=SUMCTS0:=0;
* FOR* I:=(TRACK[J]-10) *STEP* 1 *UNTIL* TRACK[J] *DO*
* BEGIN* SUMC:=SUMCT+INTCT;
INTC:=SUMC*(C[I,J]+INT);
SUMCT:=SUMCT+C[I,J]+INT*(I-1)*INT;
SUMCTS0:=SUMCTS0+C[I,J]+INT*(I-1)*INT*INT;
* END*;

* BEGIN* cr[J]:=EXP(-G[1]*CUTOFF);...
MFAN[J]:=(SUMC+INTC)/(SUMC+INT*INT);
VAR[J]:=(SUMCTS0+INTCTS0)/(SUMC+INT)-MFAN[J]*P2;
* IF* J=N *THEN* *GOTO* PI;
J:=J+1;
*GOTO* CHI;
PI: SELECTOUTPUT(2);
253 NEWLINE(2);
254 SPACE(53);
255 WRITEFXT("*************RESULTSOFTHISSRUN*************");
256 NEWLINE(2);
257 J: 1;
258 P: 0;
259 Q: 0;
260 KAPPA: 'IF' FAILED('J')='1' 'THEN' 'GOTO' OMEGA;
261 SELECTOUTPUT(2);
262 SPACE(10);
263 WRITEFXT("(\"TRACK\")");
264 SPACE(1);
265 PRINT('J/1.0');
266 SPACE(1);
267 WRITEFXT("(\"GIVES\%VALID\%RESULTS\")");
268 NEWLINE(1);
269 J: = J+1;
270 'IF' J<=1 'THEN' 'GOTO' EPSILON 'ELSIF' 'GOTO' KAPPA;
271 OMEGA: SELECTOUTPUT(2);
272 SPACE(10);
273 WRITEFXT("(\"MEAN\%OF\%TRACK\")");
274 SPACE(9);
275 PRINT('J/1.0');
276 SPACE(1);
277 WRITEFXT("(\"=\")");
278 PRINT('J/1.0');
279 WRITEFXT("(\"\"SECS\")");
280 P: P*MEAN(J);
281 SPACE(6);
282 WRITEFXT("(\"VARIANCE\%OF\%TRACK\")");
283 SPACE(1);
284 PRINT('J/1.0');
285 SPACE(1);
286 WRITEFXT("(\"=\")");
287 PRINT('J/1.0');
288 WRITEFXT("(\"\"SECS+2\")");
289 O: = Q*VAR(J);
290 NEWLINE(1);
291 'IF' J<=1 'THEN' 'GOTO' EPSILON;
292 J: = J+1;
293 'GOTO' KAPPA;
294 EPSILON: SELECTOUTPUT(2);
295 NEWLINE(2);
296 SPACE(10);
297 WRITEFXT("(\"MEAN\%\")");
298 PRINT('J/1.0');
299 WRITEFXT("(\"SECS\")");
300 SPACE(12);
301 WRITEFXT("(\"VARIANCE\%\")");
302 PRINT('J/1.0');
303 WRITEFXT("(\"\"SECS+2\")");
304 NEWLINE(3);
305 TRAKL: P/C(A*N);
306 'IF' SYSTEM='4' 'THEN' 'GOTO' PLOT;
307 'COMMENT': IF THE SYSTEM CONTAINS A PLUG FLOW SECTION THEN THE F
308 NUMBER FOR BOTH THE VAN DER LAAN OPEN AND CLOSED PIPE
309 WILL BE DETERMINED;
310 SIGMU: = (Q*N*A)/(P*P);
311 'IF' SIGMU<0.2500 'THEN' 'GOTO' WELL;
312 M: = 1;
313 SELECTOUTPUT(2);
314 NEWLINE(2);
315 SPACE(20);
316 WRTIFXT("(\"MEAN\%\")");
317 PRINT('J/1.0');
318 WRTIFXT("(\"\"SECS\")");
319 SPACE(12);
320 WRTIFXT("(\"VARIANCE\%\")");
321 PRINT('J/1.0');
322 WRTIFXT("(\"\"SECS+2\")");
323 NEWLINE(3);
324 TRAKL: P/C(A*N);
325 'IF' SYSTEM='4' 'THEN' 'GOTO' PFC;
326 PRINT;
327 NEWLINE(2);
328 SPACE(20);
329 WRTIFXT("\"MEAN\%\")");
330 PRINT('J/1.0');
331 WRTIFXT("(\"\"SECS\")");
332 SPACE(12);
333 WRTIFXT("(\"VARIANCE\%\")");
334 PRINT('J/1.0');
335 WRTIFXT("(\"\"SECS+2\")");
336 NEWLINE(3);
337 TRAKL: P/C(A*N);
338 'IF' SYSTEM='4' 'THEN' 'GOTO' PFC;
339 PRINT;
340 NEWLINE(2);
341 SPACE(20);
342 WRTIFXT("(\"MEAN\%\")");
343 PRINT('J/1.0');
344 WRTIFXT("(\"\"SECS\")");
345 SPACE(12);
346 WRTIFXT("(\"VARIANCE\%\")");
347 PRINT('J/1.0');
348 WRTIFXT("(\"\"SECS+2\")");
349 NEWLINE(3);
350 TRAKL: P/C(A*N);
M:=2;

318 IF PFC>0 THEN GOTO PFCPRINT ELSE GOTO 1O0K;

319 PFCPRINT: SELECTOUTPUT(3);

320 SPACE(1):

321 PRINT(PFC,3.3):

322 SPACE(5):

323 IF M=1 THEN GOTO PLUS ELSE

324 IF M=2 THEN GOTO LOOK ELSE GOTO 1O0K;

325 MXF: SELECTOUTPUT(2):

326 NFWLINE(1): SPACE(10):

327 WRITETEXT("(CONSULT)");

328 NFWLINE(1):

329 *GOTO* PICT.

330 LOOK: RATI:=0.5*(50.5/2):

331 UI:=P/SIGMU:

332 LI:=0:

333 D:=0:

334 MAXSFEK: PEC:=UI-(RATIO*(UL-LI));

335 FL:=(2/PEC)-(2/(PEC*PEC)+(1-EXP(-PEC))):

336 PFC:=(L*(RATIO*(UL-LU))):

337 IF FL>FL THEN UL:=UL+(RATIO*(UL-LU)) ELSE

338 LI:=UL-(RATIO*(UL-LI)):

339 IF UL-LU<0.01 THEN 'GOTO' ZEROSFEK ELSE

340 IF D>40 THEN 'GOTO' LAM3:

341 D:=D+1:

342 'GOTO' MAXSFEK:

343 ZEROSFEK: LL:=UL:

344 UL:=2/SIGMU:

345 D:=0:

346 M:=3:

347 AGAIN: PEC:=UL-(RATIO*(UL-LI));

348 FL:=(2/PEC)-(2/(PEC*PEC)+(1-EXP(-PEC))):

349 PFC:=(L*(RATIO*(UL-LU))):

350 IF FL>FL THEN UL:=UL+(RATIO*(UL-LU)) ELSE

351 LI:=UL-(RATIO*(UL-LI)):

352 IF UL-LU<0.01 THEN 'GOTO' CALC ELSE

353 IF D>40 THEN 'GOTO' LAM3:

354 D:=D+1:

355 'GOTO' AGAIN:

356 CALC: PFC:=(UL+LI)/2:

357 SELECTOUTPUT(1):

358 NFWLINE(2):

359 SPACE(20):

360 WRITETEXT("(CONSULT)");

361 'GOTO' PFCPRINT;

362 LAM1: SELECTOUTPUT(1):

363 NFWLINE(1):

364 SPACE(10):

365 WRITETEXT("(CONSULT)");

366 'GOTO' PICT.

367 LAM2: SELECTOUTPUT(2):

368 NFWLINE(1):

369 SPACE(10):

370 WRITETEXT("(CONSULT)");

371 'GOTO' PICT.

373 'COMMENT' THE FOLLOWING PACKAGE TAKES THE VALID DATA TRACKS AND

374 EVALUATES A MEAN DIMENSIONLESS DATA SET WHICH IS

375 USED TO EVALUATE THE F-CURVE:

377 PLOT: 'BEGIN' 'REAL' 4720:

378 'REAL' 4 'ARRAY' INT,PATI(1:6),COI,TDM(1:300),

379 ACC(0:300):

380 R:=0.1:

381 IF D>GEQ13 THEN 'GOTO' OUT ELSE

382 IF R>1 THEN 'GOTO' NFWPICTURE:
*END*;
CZERO := 0;
*FOR* I := 1 'STEP' 1 'UNTIL' 300 'DO*;
  *BEGIN* CZERO := CZERO + ACC[I] * INT / TBAR;*
*END*;

*FOR* I := 1 'STEP' 1 'UNTIL' 300 'DO*;
  *BEGIN* C0M[I] := ACC[I] / CZERO;
  ACC[I] := ACC[I] - 11 * (C0M[I] * INT / TBAR);
  TDIM[I] := TDIM[I] - INT / TBAR;
  IF ACC[I] * 1.0 < Then ACC[I] := 1.0;
*END*;

*COMMENT* THE E-CURVE WILL BE PLOTTED OUT ALONG WITH THE COMPL.
MIXING AND PLUG FLOW CURVES USING THE SPLIT PACKAGE;

PLOTSCALE(0,0,3.1,**(DIMENSIONLESS TIME)**,**(DIMEN
CONCENTRATION)**,**(**%**))**;

PENDUP;
SCALEPEN(0,0);
PENDOWN;
1:=0;
*FOR* I := 1 'WHILE' TDIM[I] * 1.0 < Then 1 < 0.0 'DO*;
  *BEGIN* SCALEPEN(TDIM[I],ACC[I]);
  *END*;

PENDUP;
SCALEPEN(1,0);
PENDOWN;
*SCALEPEN(1,1)*
SCALEPEN(2.9,1);
PENDUP;
pLUTFXT(2200,1400,2.0,**('PLUG')**);
PLOTFXT(2200,1450,2.0,**('FLOW')**);
PENDUP;
SCALEPEN(0,0);
PENDOWN;
1:=0;
*FOR* I := 1 'WHILE' TDIM[I] * 1.0 < Then 1 < 0.0 'DO*;
  *BEGIN* ACC[I] := 1 - EXP(-TDIM[I]);
  SCALEPEN(TDIM[I],ACC[I]);
  *END*;

PENDUP;
PLOTFXT(2200,1340,2.0,**('COMPLETE')**);
PLOTFXT(2214,1350,2.0,**('MIXING')**);
PLOTFXT(1070,1500,4.0,HEADER,6);
PENDUP;
*IE* SYS=1 'THEN' PLOTFXT(1200,1500,2.0,**('PLUG FLOW')**);
451 IF SYST=2 THEN PlotText(1200,1500,2,0,*(plUGCflow+cst
452 IF SYST=3 THEN PlotText(1200,1500,2,0,*(cst+plUGCflow
453 IF SYST=4 THEN PlotText(1200,1500,2,0,*(cSTF))
454 IF SYST=5 THEN PlotText(1200,1500,2,0,*(cSTF+cSTP))
455 PENUP;
456 'GOTO' START;
457 SELECTOUTPUT(2);
458 OUT: SELECTOUTPUT(2);
459 WRITEFTXT('MOdeXGaNSx125% Sets%0 of 02AxSurnitted')
460 NEWINF(1);
461 SPACE(5);
462 WRITEFTXT('This has infringed the plot limits')
463 NEWINF(1);
464 SPACE(5);
465 WRITEFTXT('Resuming remaining data including this')
466 'GOTO' FINISH;
467 'FND';
468 FOOL: SELECTOUTPUT(2);
469 SPACE(5);
470 WRITEFTXT('Error in data on cards - Either wrong syst
471 NEWINF(1);
472 SPACE(5);
473 WRITEFTXT('Be more careful in future')
474 NEWINF(1);
475 SPACE(5);
476 WRITEFTXT('This program has been halted')
477 NEWINF(1);
478 'GOTO' STOP;
479 QUIT: SELECTOUTPUT(2);
480 SPACE(5);
481 WRITEFTXT('Error in data on tape')
482 SPACE(1);
483 WRITEFTXT('Either too many or too many x repeatings')
484 NEWINF(1);
485 SPACE(5);
486 WRITEFTXT('Be more careful in future')
487 NEWINF(1);
488 'GOTO' STOP;
489 FINISH: SELECTOUTPUT(2);
490 NEWINF(4);
491 WRITEFTXT('This processing of data is now complete')
492 SPACE(1);
493 WRITEFTXT('')
494 NEWINF(1);
495 SPACE(4);
496 WRITEFTXT(''
497 SPACE(1);
498 WRITEFTXT(''
499 NEWINF(2);
500 STOP: SPACE(33);
501 WRITEFTXT('###########FND%OF%PRINTOUT###########')
502 NEWINF(2);
503 'END';
504 ;PLOTEROFF: WRITEFTXT('END OF PROGRAM')
505 PAUSE(77);
506 'FND';
Appendix 4  
Program to calculate NTU, HTU and (Ks) for the carbon tetrachloride-acetic acid-water and MIBK-acetic acid-water systems.

SLIST NTUMIBK
10 REM CY IS THE CONC. OF THE CONT. PHASE IN GM OF SOLUTE /LITRE
20 REM CX DISP
30 REM DIM A IS THE POINT VALUE OF CY ALONG THE COLUMN
40 REM DIM B IS THE VALUE OF CX WHICH IS IN EQUILIBRIUM WITH CX
50 REM DIM C IS THE VALUE OF CX
60 REM DIM D IS THE STRIP WIDTH FOR INTEGRATION FOR THE CONT. PHASE
100 REM DIM E IS THE SECTIONAL VALUE OF N3 (NTUCM)
110 REM DIM F IS THE EQUILIBRIUM CY EXPRESSED IN MOLE FRACTION
200 REM DIM G IS THE CYO EXPRESSED IN MOLE FRACTION
300 REM DIM H IS THE GAP BETWEEN THE PLATE AND THE COLUMN
310 REM DIM I IS THE SPACING BETWEEN THE PLATES
320 REM DIM J IS THE TOTAL NTUCM (NTUCM)
330 REM DIM K IS THE TOTAL NTUCM (NTUCM)
340 REM DIM L IS THE CY EXPRESSED IN MOLE FRACTION
350 REM DIM M IS THE CY EXPRESSED IN MOLE FRACTION
360 REM DIM N IS THE NTUCM OBTAINED BY SIMPSON'S RULE
370 REM DIM O IS THE NTUCM OBTAINED BY SIMPSON'S RULE
380 REM DIM P IS THE NTUCM OBTAINED BY SIMPSON'S RULE
390 REM DIM Q IS THE NTUCM OBTAINED BY SIMPSON'S RULE
400 REM DIM R IS THE NTUCM OBTAINED BY SIMPSON'S RULE
410 REM DIM S IS THE NTUCM OBTAINED BY SIMPSON'S RULE
420 REM DIM T IS THE NTUCM OBTAINED BY SIMPSON'S RULE
430 REM DIM U IS THE NTUCM OBTAINED BY SIMPSON'S RULE
440 REM DIM V IS THE NTUCM OBTAINED BY SIMPSON'S RULE
450 REM DIM W IS THE NTUCM OBTAINED BY SIMPSON'S RULE
460 REM DIM X IS THE NTUCM OBTAINED BY SIMPSON'S RULE
470 REM DIM Y IS THE NTUCM OBTAINED BY SIMPSON'S RULE
480 REM DIM Z IS THE NTUCM OBTAINED BY SIMPSON'S RULE
490 REM DIM A(201) IS THE NAME OF DATA FILE
500 REM DIM B(201) IS THE NAME OF DATA FILE
510 REM DIM C(201) IS THE NAME OF DATA FILE
520 REM DIM D(201) IS THE NAME OF DATA FILE
530 REM DIM E(201) IS THE NAME OF DATA FILE
540 REM DIM F(201) IS THE NAME OF DATA FILE
550 REM DIM G(201) IS THE NAME OF DATA FILE
560 REM DIM H(201) IS THE NAME OF DATA FILE
570 REM DIM I(201) IS THE NAME OF DATA FILE
580 REM DIM J(201) IS THE NAME OF DATA FILE
590 REM DIM K(201) IS THE NAME OF DATA FILE
600 REM DIM L(201) IS THE NAME OF DATA FILE
610 REM DIM M(201) IS THE NAME OF DATA FILE
620 REM DIM N(201) IS THE NAME OF DATA FILE
630 REM DIM O(201) IS THE NAME OF DATA FILE
640 REM DIM P(201) IS THE NAME OF DATA FILE
650 REM DIM Q(201) IS THE NAME OF DATA FILE
660 REM DIM R(201) IS THE NAME OF DATA FILE
670 REM DIM S(201) IS THE NAME OF DATA FILE
680 REM DIM T(201) IS THE NAME OF DATA FILE
690 REM DIM U(201) IS THE NAME OF DATA FILE
700 REM DIM V(201) IS THE NAME OF DATA FILE
710 REM DIM W(201) IS THE NAME OF DATA FILE
720 REM DIM X(201) IS THE NAME OF DATA FILE
730 REM DIM Y(201) IS THE NAME OF DATA FILE
740 REM DIM Z(201) IS THE NAME OF DATA FILE
750 REM DIM A(1) IS THE NAME OF DATA FILE
760 REM DIM B(1) IS THE NAME OF DATA FILE
770 REM DIM C(1) IS THE NAME OF DATA FILE
780 REM DIM D(1) IS THE NAME OF DATA FILE
790 REM DIM E(1) IS THE NAME OF DATA FILE
800 REM DIM F(1) IS THE NAME OF DATA FILE
810 REM DIM G(1) IS THE NAME OF DATA FILE
820 REM DIM H(1) IS THE NAME OF DATA FILE
830 REM DIM I(1) IS THE NAME OF DATA FILE
840 REM DIM J(1) IS THE NAME OF DATA FILE
850 REM DIM K(1) IS THE NAME OF DATA FILE
860 REM DIM L(1) IS THE NAME OF DATA FILE
870 REM DIM M(1) IS THE NAME OF DATA FILE
880 REM DIM N(1) IS THE NAME OF DATA FILE
890 REM DIM O(1) IS THE NAME OF DATA FILE
900 REM DIM P(1) IS THE NAME OF DATA FILE
910 REM DIM Q(1) IS THE NAME OF DATA FILE
920 REM DIM R(1) IS THE NAME OF DATA FILE
930 REM DIM S(1) IS THE NAME OF DATA FILE
940 REM DIM T(1) IS THE NAME OF DATA FILE
950 REM DIM U(1) IS THE NAME OF DATA FILE
960 REM DIM V(1) IS THE NAME OF DATA FILE
970 REM DIM W(1) IS THE NAME OF DATA FILE
980 REM DIM X(1) IS THE NAME OF DATA FILE
990 REM DIM Y(1) IS THE NAME OF DATA FILE
1000 REM DIM Z(1) IS THE NAME OF DATA FILE
1100 REM PRINT "INPUT THE NAME OF DATA FILE".
1200 REM PRINT "INPUT THE NUMBER OF STRIP".
1300 REM PRINT "INPUT THE NAME OF DATA FILE".
1400 REM PRINT "INPUT THE NUMBER OF STRIP".
1500 REM PRINT "INPUT THE NAME OF DATA FILE".
1600 REM PRINT "INPUT THE NUMBER OF STRIP".
1700 REM PRINT "INPUT THE NAME OF DATA FILE".
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1900 REM PRINT "INPUT THE NAME OF DATA FILE".
2000 REM PRINT "INPUT THE NUMBER OF STRIP".
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2500 REM PRINT "INPUT THE NUMBER OF STRIP".
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2700 REM PRINT "INPUT THE NUMBER OF STRIP".
2800 REM PRINT "INPUT THE NUMBER OF STRIP".
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3000 REM PRINT "INPUT THE NUMBER OF STRIP".
3100 REM PRINT "INPUT THE NUMBER OF STRIP".
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3300 REM PRINT "INPUT THE NUMBER OF STRIP".
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3500 REM PRINT "INPUT THE NUMBER OF STRIP".
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3700 REM PRINT "INPUT THE NUMBER OF STRIP".
3800 REM PRINT "INPUT THE NUMBER OF STRIP".
3900 REM PRINT "INPUT THE NUMBER OF STRIP".
4000 REM PRINT "INPUT THE NUMBER OF STRIP".
4100 REM PRINT "INPUT THE NUMBER OF STRIP".
4200 REM PRINT "INPUT THE NUMBER OF STRIP".
4300 REM PRINT "INPUT THE NUMBER OF STRIP".
4400 REM PRINT "INPUT THE NUMBER OF STRIP".
4500 REM PRINT "INPUT THE NUMBER OF STRIP".
4600 REM PRINT "INPUT THE NUMBER OF STRIP".
4700 REM PRINT "INPUT THE NUMBER OF STRIP".
For CCl₄-Acetic acid-Water system, following alterations are required:

840 \( A_5 = 0.5357 \log(B(K)) \)

850 \( A_5 = \exp(A_5) \)

860 \( C(K) = A_5 \times 64.87 \)

870 \( D(K) = 1.8667 \times \log((A(K)/64.87)) \)

880 \( D(K) = \exp(D(K)) \)
Appendix 5

Tables
<table>
<thead>
<tr>
<th>$Q_d$ (1/hr)</th>
<th>$Q_c$ (1/hr)</th>
<th>$\Delta h$ (cm)</th>
<th>$\phi_0$</th>
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<td>50</td>
<td>1.0</td>
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</tr>
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<td>$Q_e$ (l/hr)</td>
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Table 7.2 (1A)

Axial mixing study on the continuous phase (water).

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Table 7.2.(IB)

Axial mixing study on the continuous phase (water)

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### Table 7.3(1A)

**CCl₄/Acetic acid/Water (equal flowrates)**

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### CCl₄/Acetic acid/Water (unequal flowrates)

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* For more details of the sampling, see Table 7.3(1E)
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### Table 7.3(1E)
Full data of the sampling from run 57 to 72 (Ref. Fig. 4.3(B))

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Table 7.3

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Table 7.3(IF)

Sectional values of the NTU, HTU and Ka (Ref. Fig. 4.3(A)).

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### Table 7.3C-1F

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Table 7.6.1

End effects (Ref. Sec. 9.6 and Fig. 4.3)

Bottom effects, \( B^* = \frac{100(C_{x_a} - C_{x_B})}{(C_{x_T} - C_{x_B})} \)

Top effects, \( T^* = \frac{100(C_{x_T} - C_{x_e})}{(C_{x_T} - C_{x_B})} \)

(Equal flowrates) Table 7.6.1-A — CCl₄/Acetic acid/Water

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Table 7.6.1(1A)—CCL₄/ Acetic acid/ Water

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(Equal flowrates)
Table 7.6.(1B)—CCl₄/Acetic acid/water

(Unequal flowrates)

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<th>Qd (l/h)</th>
<th>B* %</th>
<th>T* %</th>
<th>B* + T* %</th>
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Table 7.6. (LC) -- CCl₄/Acetic acid/Water

(Low concentration)

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<th>Spac. (in)</th>
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<th>( B^* ) %</th>
<th>( T^* ) %</th>
<th>( B^* + T^* ) %</th>
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Table 7.6.(1D)—MIEK/Acetic acid/Water

(Equal and unequal flowrates)

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<th>( Q_d ) (l/h)</th>
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<th>( T^* )</th>
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### Table 7.7. (l)
Interfacial tension measurement

#### Table 7.7. (lA) -- MIBK/Acetic acid/Water (Using torsion balance

<table>
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<th>Equil. con. of the acid in the MIBK phase (gm/l)</th>
<th>Equil. con. of the acid in the water (gm/l)</th>
<th>Interfacial tension $\gamma$ (dyne/cm)</th>
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#### Table 7.7. (lB) -- CCl₄/Acetic acid/Water (Using an 'AGLA' micrometer syringe outfit and an 'AGLA' straight capillary glass needle)

<table>
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<tr>
<th>$C^*$ (gm/l)</th>
<th>$C^{**}$ (gm/l)</th>
<th>Volume V (cm³)</th>
<th>$r/V^{1/3}$</th>
<th>$\phi$</th>
<th>$\gamma$ (dyne/cm)</th>
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$C^*$ and $C^{**}$ are the equilibrium cons. of the acid in the CCl₄ and water respectively.

$2r$ is the contact-diameter of the capillary glass needle $r = 0.05$ cm and $\phi$ is obtained from Fig. 4.11.
Table 7.8.1.(l)(Ref. Fig.7.8.1.(l))

Interfacial tension measurement

Pure CCl₄ droplet in water—Optical(Andreas) method(A 6)

<table>
<thead>
<tr>
<th>Sample</th>
<th>dₑ (mm)</th>
<th>dₛ (mm)</th>
<th>S = dₛ/ᵈₑ</th>
<th>1/H</th>
<th>Interfacial tension $\phi$</th>
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H is obtained from Fig.6.4 and $d_e$ and $d_s$ are defined as shown in the following diagram of a hanging drop:
Table 8.1.(1A)

Various overall coefficients for the continuous phase \( K_c \) (defined in sections 8.1 and 9.3.3).

CCl\(_4\)/Acetic acid/Water (equal flowrates)

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<th>( (K_c)_1 \times 10^4 ) (cm/sec)</th>
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### Table S.1.1A

**CCl₄/Acetic acid/Water (equal flowrates)**

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<th>Spac. (in)</th>
<th>(Q_c = Q_d) (litre/h)</th>
<th>((K_c)_1 \times 10^4) (cm/sec)</th>
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<th>((K_c)_3 \times 10^4) (cm/sec)</th>
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Table 8.1.(IC)

CCl<sub>4</sub>/Acetic acid/Water (low concentration)

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Table 8.1.(ID)

MIBK/Acetic acid/Water (equal and unequal flowrates)

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Table 9.3.4.2.(1) (Ref. Section 9.3.4.2)

Numerical values of various terms in the One-Dimensional Diffusion equation (eq. 3.94).

Table 9.3.4.2.(1A) (Ref. Fig. 4.3(A))

CCl₄/Acetic acid/Water (equal flowrates)

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Table 9.3.4.2.(10)

**MIBK/Acetic acid/Water (equal and unequal flowrates)**

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Values of the overall mass transfer coefficient $K$ under various regimes. (The definition of $K$ in each case is given in Section 9.3.5)

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### Table 9.3.5.13

CCl₄/Acetic acid/Water (unequal flowrates)

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<th>Run</th>
<th>Gap (in)</th>
<th>Spac. (in)</th>
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<th>Qd (l/h)</th>
<th>tₚ (s)</th>
<th>K_{dc}x10^3 (cm/s)</th>
<th>K_{dr}x10^3 (cm/s)</th>
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**Table 9.3.5.**(1G)

\[\text{CCI}_4/\text{Acetic acid}/\text{Water (low concentration)}\]

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<tr>
<th>Run</th>
<th>Gap (in)</th>
<th>Spac. (in)</th>
<th>(Q_c = Q_d) (1/h)</th>
<th>(t_f) (s)</th>
<th>(K_{df} \times 10^3) (cm/s)</th>
<th>(K_{dc} \times 10^3) (cm/s)</th>
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Table 9.3.5.(1D)

MIBK/Acetic acid/Water (equal and unequal flowrates)

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<th>Qd (l/h)</th>
<th>t_f (s)</th>
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Appendix 6

Figures
Fig. 7.1.(l.A) Flooding curves for the carbon tetrachloride-water system. (Ref. Table 7.1.1.A)
Gap between the column and the baffle, G=1 in.
Spacing between the baffles, S=5 in.
$Q_c$ is the water flowrate
$Q_d$ is the carbon tetrachloride flowrate in l/hr.

- $Q_d = 50$
- $Q_d = 100$
- $Q_d = 150$
- $Q_d = 200$
- $Q_d = 250$
Fig. 7.1.(1.B) Flooding curves for the carbon tetrachloride - water system. (Ref. Table 7.1.1.B)

\( G = 2 \text{ in.} \)
\( S = 5 \text{ in.} \)

- \( Q_d = 50 \)
- \( Q_d = 100 \)
- \( Q_d = 150 \)
- \( Q_d = 200 \)
Fig. 7.1.1.(c) Effects of the gap width, G, on the holdup \( \phi_0 \) and the flowrate of the continuous phase.

(Ref. Table 7.1.1.A and Table 7.1.1.B)

- \( G = 1 \) in. \( Q_d = 50 \)
- \( G = 2 \) in. \( Q_d = 50 \)
- \( G = 1 \) in. \( Q_d = 100 \)
- \( G = 2 \) in. \( Q_d = 100 \)
Fig. 7.1. (ID) Holdup versus Dispersed Phase velocity below flooding. (Ref. Table 7.1. (1A))

- G = 1 in.
- S = 5 in.
- $U_c = 17.986$ ft/hr ($Q_c = 100$ l/hr)
- $U_c = 26.976$ ft/hr ($Q_c = 150$ l/hr)
- $U_c = 44.965$ ft/hr ($Q_c = 250$ l/hr)
Toluene/Water system

Diameter of the packed column = 4 in.

Diameter of the sphere (packing material) = 17.5 mm

Fig. 7.1 (1.E) Holdup versus Dispersed Phase velocity at and below flooding ($C_d$) for a packed column.

$U_c$ and $U_d$ are superficial velocity of the continuous and dispersed phases respectively, ft/hr.

$U_c$

- ○ 28.83
- × 39.58
- Δ 48.86
- ○ 56.68
- ☓ 64.50
Fig. 7.2 (1.A) The experimental mean residence time $\mu$ as a function of the flowrate of the continuous phase $Q_c$.

- $\times$ $G = 2$ in. $S = 5$ in.
- $\circ$ $G = 2$ in. $S = 4$ in.
- $\triangle$ $G = 2$ in. $S = 3$ in.
- $\square$ $G = 2$ in. $S = 2$ in.
Fig. 7.2(1.B) The experimental mean residence time $\mu$ as a function of the flowrate of the continuous phase $Q_c$.

- $\times$ G = 7/8 in. S = 3 in.
- $\bigcirc$ G = 11/8 in. S = 3 in.
- $\triangle$ G = 13/8 in. S = 3 in.
- $\square$ G = 2 in. S = 3 in.
Fig. 7.2(1C) The Peclet Number $N_{Pe}$ as a function of the flowrate of the continuous phase $Q_c$.

- $\times$ $G = 2$ in. $S = 5$ in.
- $\bigcirc$ $G = 2$ in. $S = 4$ in.
- $\triangle$ $G = 2$ in. $S = 3$ in.
- $\square$ $G = 2$ in. $S = 2$ in.
Fig. 7.2(1.D) The Peclet Number $N_{Pe}$ as a function of the flowrate of the continuous phase $Q_c$.

- $\times$ $G = 7/8$ in. $S = 3$ in.
- $\bigcirc$ $G = 11/8$ in. $S = 3$ in.
- $\triangle$ $G = 13/8$ in. $S = 3$ in.
- $\square$ $G = 2$ in. $S = 3$ in.
Fig. 7.2.(1.E) The Axial Dispersion Coefficient of the continuous phase $E_c$ as a function of the flowrate of the continuous phase $Q_c$.

$\times$ $G = 2$ in. $S = 5$ in.
$\bigcirc$ $G = 2$ in. $S = 4$ in.
$\triangle$ $G = 2$ in. $S = 3$ in.
$\square$ $G = 2$ in. $S = 2$ in.
The Axial Dispersion Coefficient of the continuous phase $E_c$ as function of the flowrate of the continuous phase $Q_c$.

- $\times$ $G = 7/8$ in. $S = 3$ in.
- $\bigcirc$ $G = 11/8$ in. $S = 3$ in.
- $\triangle$ $G = 13/8$ in. $S = 3$ in.
- $\blacksquare$ $G = 2$ in. $S = 3$ in.
Fig. 7.2(2A) A typical response curve from the experiment on axial mixing.
(Ref. Table 7.2.(1A))

G = 2 in. S = 2 in.

Q_c = 150.02 litres/hr
Fig. 7.2(2B) A typical response curve from the experiment on axial mixing.

(Ref. Table 7.2.(1A))

G = 2 in. S = 2 in.

Q_c = 90.92 litres/hr
Fig. 7.2(3A) Comparison of the theoretical and the practical relationships between the dimensionless variance $\sigma_\theta^2$ and the Peclet Number $N_{Pe}$.

- $\times$ G = 2 in. S = 5 in.
- $\bigcirc$ G = 2 in. S = 4 in.
- $\triangle$ G = 2 in. S = 3 in.
- $\blacksquare$ G = 2 in. S = 2 in.

- Theoretical equation due to Van der Laan: $\sigma_\theta^2 = \frac{2}{N_{Pe}} (N_{Pe} - 1 + e^{-N_{Pe}})$. 
Fig. 7.3(1.A) Concentration Profiles of the continuous phase along the column. (Ref. Fig. 4.3(A))

'Z' is the distance of the sampling points from the bottom of the column.

'B' is the bottom sampling point.

Region 'B-16' represents the bottom section.

'T' is the top sampling point.

Region '136-T' represents the top section.

'R' is the flow ratio and:

\[ R = \frac{\text{Continuous phase flow rate in litre/hr.}}{\text{Dispersed phase flow rate in litre/hr.}} \]

G = 1 in. S = 5 in.

\[ \times \quad R = 100/100 \]

\[ \circ \quad R = 150/150 \]

\[ \triangle \quad R = 200/200 \]

\[ \diamond \quad R = 250/250 \]

\( C_y \) is the concentration of the acetic acid in water and the dispersed phase is carbon tetrachloride.
Fig. 7.3(a,b) Concentration Profiles of the continuous phase along the column.

$G = 1.62$ in. $S = 5$ in.

- $\times$ $R = 100/100$
- $\circ$ $R = 150/150$
- $\triangle$ $R = 200/200$
- $\blacksquare$ $R = 250/250$
Fig. 7.3 Concentration Profiles of the continuous phase along the column.

G = 1.62 in. S = 2 in.

\( \times \) R = 100/100
\( \circ \) R = 150/150
\( \triangle \) R = 200/200
\( \square \) R = 250/250
\( \blacksquare \) R = 200/200
CCl₄/Acetic acid/Water

\[ C_y (\text{gm/l}) \]

0.0 10.0 20.0 30.0 40.0 50.0

\( Z(\text{cm}) \)

B 16 46 76 106 136 T

Fig. 7.3(D) Concentration Profiles of the continuous phase along the column.

G = 2 in. S = 4 in.

- \( \times \) R = 100/100
- \( \bigcirc \) R = 150/150
- \( \triangle \) R = 200/200
- \( \square \) R = 250/250
Fig. 7.3(1.E) Concentration Profiles of the continuous phase along the column.

$G = 2$ in. $S = 2$ in.

$\times$ $R = 100/100$

$\odot$ $R = 150/150$

$\triangle$ $R = 200/200$

$\blacksquare$ $R = 250/250$
Fig. 7.3(1.F) Concentration Profiles of the continuous phase along the column.

$G = 1 \text{ in. } S = 5 \text{ in.}$

$\times \quad R = 200/100$

$\bigcirc \quad R = 100/200$
Fig. 7.3(1.G) Concentration Profiles of the continuous phase along the column.

G = 1.62 in. S = 5 in.

X  R = 200/100

O  R = 100/200
Fig. 7.3(1.H) Concentration Profiles of the continuous phase along the column.

G = 2 in. S = 4 in.

- $R = 200/50$
- $R = 210/70$
- $R = 200/100$
- $R = 100/200$
- $R = 70/210$
Fig. 7.3(1.1) Concentration Profiles of the continuous phase along the column.

G = 2 in. S = 2 in.

- $R = 200/50$
- $R = 210/70$
- $R = 200/100$
- $R = 100/200$
- $R = 70/210$
- $R = 50/200$
Fig. 7.3(1.J) Concentration Profiles of the continuous phase along the column. (The initial concentration of the dispersed phase was fairly low.)

G = 2 in. S = 5 in. (Ref. Fig. 4.3(B))

$R = 250/250$
Fig. 7.3(1.K) Concentration Profiles of the continuous phase along the column. (The initial concentration of the dispersed phase was fairly low.)

G = 2 in. S = 5 in.

R = 100/100
MIBK/Acetic acid/Water

Fig. 7.3(1. L) Concentration Profiles of the continuous phase along the column. (Ref. Fig. 4.3(B))

$C_y$ is the concentration of the acetic acid in water and the dispersed phase is MIBK.

$G = 2$ in. $S = 5$ in.

- $\times$ $R = 50/50$
- $\bigcirc$ $R = 70/70$
- $\Delta$ $R = 100/100$
- $\square$ $R = 150/150$
Fig. 7.3(1.M) Concentration Profiles of the continuous phase along the column.

G = 2 in. S = 5 in.

- X R = 100/50
- R = 50/100
Fig. 7.3(1.N) Concentration Profiles of the continuous phase along the column.

- $G = 2$ in. $S = 3$ in.
- $\times$ $R = 50/50$
- $\bigcirc$ $R = 70/70$
- $\triangle$ $R = 100/100$
- $\square$ $R = 150/150$
Fig. 7.3(2.A) (HTU)$\_cA$ as a function of the flowrate of the continuous phase, $Q_c$.

'(HTU)$\_cA$' is the total apparent measured Height of Transfer Unit of the continuous phase.

$Q_c = Q_d$

$G = 1$ in. $S = 5$ in.
Fig. 7.3(2.B) $(HTU)_c A$ as a function of the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

$\times$ $G = 1.62$ in. $S = 5$ in.

$\bigcirc$ $G = 1.62$ in. $S = 4$ in.

$\bigtriangleup$ $G = 1.62$ in. $S = 3$ in.

$\Box$ $G = 1.62$ in. $S = 2$ in.

CCl₄/Acetic acid/Water
Fig. 7.3 (2.C) $(HTU)_{CA}$ as a function of the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

$\times$ G = 2 in. S = 5 in.

$\bigcirc$ G = 2 in. S = 4 in.

$\triangle$ G = 2 in. S = 3 in.
Fig. 7.3(2.D) \((\text{NTU})_{cA}\) as a function of the flowrate of the continuous phase, \(Q_c\).

\((\text{NTU})_{cA}\) is the total apparent measured Number of Transfer Unit of the continuous phase.

\[Q_c = Q_d\]

\(G = 1\) in, \(S = 5\) in.
Fig. 7.3 (2.E) \( (\text{NTU})_{cA} \) as a function of the flowrate of the continuous phase, \( Q_c \).

\[ Q_c = Q_d \]

- \( \times \) \( G = 1.62 \text{ in.} \) \( S = 5 \text{ in.} \)
- \( \bigcirc \) \( G = 1.62 \text{ in.} \) \( S = 4 \text{ in.} \)
- \( \triangle \) \( G = 1.62 \text{ in.} \) \( S = 3 \text{ in.} \)
- \( \square \) \( G = 1.62 \text{ in.} \) \( S = 2 \text{ in.} \)
Fig. 7.3(2.8) $\text{NTU}_{dA}$ as a function of the flowrate of the dispersed phase, $Q_d$.

'$(\text{NTU})_{dA}$' is the total apparent measured Number of Transfer Unit of the dispersed phase.

$Q_d = Q_c$

$\times$ $G = 1.62$ in. $S = 5$ in.
$\bigcirc$ $G = 1.62$ in. $S = 4$ in.
$\triangle$ $G = 1.62$ in. $S = 3$ in.
$\square$ $G = 1.62$ in. $S = 2$ in.
Fig. 7.3(2.G) 

(HTU)$_{dA}$ as a function of the flowrate of the dispersed phase, $Q_d$.

'(HTU)$_{dA}$' is the total apparent measured Height of Transfer Unit of the dispersed phase.

$Q_d = Q_c$

$\times \ G = 1.62 \text{ in.} \ S = 5 \text{ in.}$
$\bigcirc \ G = 1.62 \text{ in.} \ S = 4 \text{ in.}$
$\triangle \ G = 1.62 \text{ in.} \ S = 3 \text{ in.}$
$\square \ G = 1.62 \text{ in.} \ S = 2 \text{ in.}$
Fig. 7.3(2.H) A comparison of the $(HTU)_{CA}$ values of the two systems.

$Q_c = Q_d$

$G = 2$ in. $S = 5$ in.

○ Carbon Tetrachloride/Acetic acid/Water

× MIBK/Acetic acid/Water
Fig. 7.3(2.1) A comparison of the \((HTU)_{dA}\) values of the two systems.

\[ Q_d = Q_c \]

\( G = 2 \text{ in.} \quad S = 5 \text{ in.} \)

○ Carbon Tetrachloride/Acetic acid/Water

✗ MIBK/Acetic acid/Water
Fig. 7.3(2.J) Apparent measured (Ka) of the continuous phase v. the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

$G = 1 \text{ in. } S = 5 \text{ in.}$
Fig. 7.3(2.K) Apparent measured $(K_a)$ of the continuous phase v. the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

- $\times$ $G = 1.62$ in. $S = 5$ in.
- $\bigcirc$ $G = 1.62$ in. $S = 4$ in.
- $\triangle$ $G = 1.62$ in. $S = 3$ in.
- $\square$ $G = 1.62$ in. $S = 2$ in.
Fig. 7.3(2.L) Apparent measured (Ka) of the continuous phase v. the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

- $\times \ G = 2 \text{ in.} \ S = 5 \text{ in.}$
- $\bigcirc \ G = 2 \text{ in.} \ S = 4 \text{ in.}$
- $\bigtriangleup \ G = 2 \text{ in.} \ S = 3 \text{ in.}$
- $\square \ G = 2 \text{ in.} \ S = 2 \text{ in.}$
Fig. 7.3(2.M) Apparent measured (Ka) of the continuous phase v. the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

$\times$  $G = 2$ in. $S = 5$ in.

$\bigcirc$  $G = 2$ in. $S = 3$ in.
Fig. 7.4(1.A) The variation of the hold up $\phi$ with the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

$G = 1$ in. $S = 5$ in.

$\times$ with acetic acid

$\bigcirc$ with no acetic acid
Fig. 7.4(1.B) The variation of the hold up $\phi$ with the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

- $G = 1.62$ in. $S = 5$ in.
- $G = 1.62$ in. $S = 4$ in.
- $G = 1.62$ in. $S = 3$ in.
- $G = 1.62$ in. $S = 2$ in.
Fig. 7.4(1.0) The variation of the hold up $\phi$ with the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

- $\times \quad G = 2 \text{ in.} \quad S = 5 \text{ in.}$
- $\bigcirc \quad G = 2 \text{ in.} \quad S = 4 \text{ in.}$
- $\triangle \quad G = 2 \text{ in.} \quad S = 3 \text{ in.}$
- $\blacksquare \quad G = 2 \text{ in.} \quad S = 2 \text{ in.}$
Fig. 7.4 (1. D) The variation of the hold up $\phi$ with the flowrate of the continuous phase, $Q_c$. 

$Q_c = Q_d$

$\times$ $G = 2$ in. $S = 5$ in.

$\circ$ $G = 2$ in. $S = 3$ in.
Fig. 7.4.(l.E) The variation of the hold up $\varnothing$ with the apparent measured $(Ka)$ of the continuous phase.

$G = 1$ in. $S = 5$ in.
Fig. 7.4. (1. F) The variation of the hold up \( \phi \) with the apparent measured \((K_a)\) of the continuous phase.

- \( \times \) \( G = 1.62 \) in. \( S = 5 \) in.
- \( \bigcirc \) \( G = 1.62 \) in. \( S = 4 \) in.
- \( \triangle \) \( G = 1.62 \) in. \( S = 3 \) in.
- \( \square \) \( G = 1.62 \) in. \( S = 2 \) in.
Fig. 7.4. (1.G) The variation of the hold up $\phi$ with the apparent measured $(Ka)$ of the continuous phase.

- $\times$ G = 2 in. S = 5 in.
- $\ominus$ G = 2 in. S = 4 in.
- $\triangle$ G = 2 in. S = 3 in.
- $\square$ G = 2 in. S = 2 in.
Fig. 7.4.(1.H) The variation of the hold up \( \phi \) with the apparent measured \((K_a)\) of the continuous phase.

- \( \times \) G = 2 in. S = 5 in.
- \( \circ \) G = 2 in. S = 3 in.
Gap = 1 in.

Gap = 1.62 in.

Gap = 2 in.

Spacing = 5 in. 

\( Q_c = Q_d = 100 \text{ litres/hr.} \)

\( Q_c = Q_d = 150 \text{ litres/hr.} \)

Gap = 2 in.

Spacing = 5 in.
Fig. 7.5.(1A)  
Droplets under various mass transfer operations.

CCl₄/Acetic acid/Water system.

MIBK/Acetic acid/Water system.
Fig. 7.7(A) The variation of the interfacial tension $\gamma$ with the concentration of the acetic acid in the MIBK phase.
Fig. 7.7(1,B) The variation of the interfacial tension $\gamma$ with the concentration of the acetic acid in the Carbon Tetrachloride phase.
The determination of the interfacial tension of the carbon tetrachloride droplet in pure water by Andreas method.
Fig. 7.8.2.(1A) Initial conc. of the acetic acid in the carbon tetrachloride phase was 10 gm/litre.

Fig. 7.8.2.(1) Eruption accompanying the transfer of acetic acid from a stationary CCl$_4$ drop into pure water.
Fig. 7.8.2.(1B) Initial conc. of the acetic acid in the carbon tetrachloride phase was 30 gm/litre.

Fig. 7.8.2.(1) Eruption accompanying the transfer of acetic acid from a stationary CCl₄ drop into pure water.
Fig. 7.8.2.(1C) Initial conc. of the acetic acid in the carbon tetrachloride phase was 40 gm/litre.

Fig. 7.8.2.(1) Eruption accompanying the transfer of acetic acid from a stationary CCl₄ drop into pure water.
Initial conc. of the acetic acid in the carbon tetrachloride phase was 50 gm/litre.

Eruption accompanying the transfer of acetic acid from a stationary CC\textsubscript{4} drop into pure water.
Fig. 9.3.3.2.(1A) Various $(K)_c$ against the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

$G = 1$ in. $S = 5$ in.

- $\times$ (K)$_{c1}$ (Experimental)
- $\bigcirc$ (K)$_{c2}$ (Sivaraman equation)
- $\triangle$ (K)$_{c3}$ (Modified Penetration Theory)
- $\blacksquare$ (K)$_{c4}$ (A combination of Ruby-Elgin and Handlos-Baron).
Fig. 9.3.3.2.(IB) Various $(K)_c$ against the flowrate of the continuous phase, $Q_c$. 

$Q_c = Q_d$

$G = 1.62$ in. $S = 4$ in.

$X (K)_c1$

$O (K)_c2$

$\Delta (K)_c3$

$\Box (K)_c4$
Fig. 9.3.3.2 (10) Various $(K)_c$ against the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

$G = 1.62$ in. $S = 2$ in.

$\times$ $(K)_{c1}$

$\bigcirc$ $(K)_{c2}$

$\triangle$ $(K)_{c3}$

$\blacksquare$ $(K)_{c4}$
Fig. 9.3.3.2 (1D) Various $(K)_c$ against the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

$G = 2$ in. $S = 5$ in.

$\times (K)_{c1}$  $\triangle (K)_{c3}$

$\circ (K)_{c2}$  $\square (K)_{c4}$
The variation of the $(Ka)_c$ of the continuous phase with the initial concentration of the acetic acid in the dispersed phase.

$G = 2 \text{ in.} \quad S = 5 \text{ in.}$

- $Q_c = 100 \text{ litre/hr} = Q_d$
- $Q_c = 250 \text{ litre/hr} = Q_d$
Fig. 9.3.3.2.(2B) The variation of the \((Ka)_{d}\) of the dispersed phase with its initial concentration of the acetic acid. 

\(G = 2\) in. \(S = 5\) in. 

\(\times Q_c = 100\) litre/hr = \(Q_d\) 

\(\bigcirc Q_c = 250\) litre/hr = \(Q_d\)
Fig. 9.5.(1A) The Surface Mean Diameter of the droplet, \(d_{32}\) as a function of the flowrate of the continuous phase, \(Q_c\).

\[ Q_c = Q_d \]

- \(\times\) \(G = 1.62\) in. \(S = 5\) in.
- \(\bigcirc\) \(G = 1.62\) in. \(S = 4\) in.
- \(\triangle\) \(G = 1.62\) in. \(S = 3\) in.
- \(\square\) \(G = 1.62\) in. \(S = 2\) in.
Fig. 9.5.(2A) The interfacial area of the droplets, $a$, as a function of the flowrate of the continuous phase, $Q_c$.

$Q_c = Q_d$

- $\times$ G = 1.62 in. S = 5 in.
- $\circ$ G = 1.62 in. S = 4 in.
- $\triangle$ G = 1.62 in. S = 3 in.
- $\square$ G = 1.62 in. S = 2 in.
Fig. A.1.2.(1A) Initial conc. of the acetic acid in the carbon tetrachloride phase was 1.396 gm/litre.

Fig. A.1.2.(1) The concentration dependency of the interfacial disturbance.
Fig. A.1.2.(1B) Initial conc. of the acetic acid in the carbon tetrachloride phase was 4.488 gm/litre.

Fig. A.1.2.(1) The concentration dependency of the interfacial disturbance.
Initial conc. of the acetic acid in the carbon tetrachloride phase was 6.092 gm/litre.

The concentration dependency of the interfacial disturbance.
Fig. A.1.2.(1D) Initial conc. of the acetic acid in the carbon tetrachloride phase was 7.296 gm/litre.

Fig. A.1.2.(1) The concentration dependency of the interfacial disturbance.
Instant disturbance

Fig. A.1.2.(1E) Initial conc. of the acetic acid in the carbon tetrachloride phase was 210 gm/l.

Fig. A.1.2.(1F) Initial conc. of the acetic acid in the carbon tetrachloride phase was 420 gm/l.

Fig. A.1.2.(1) The concentration dependency of the interfacial disturbance.
Fig. A.1.3. (1A) The interfacial tension $\gamma$ as a function of the time.

Initial concentration of the acid: 10 gm/l to 50 gm/l.
Fig.A.1.3.(2A) The final concentration of the acid in the droplet as a function of the time. Initial concentration of the acid:
10 gm/l to 50 gm/l
Fig.A.2.1(2A) Linearization of distribution data with a 'chemical' theory. (eq. A.2.1.8)
Fig. A.2.1(3A) Relationship between the concentration of the acid in CCl₄ and water (the acid has a great tendency to dimerize in the CCl₄ phase).

(eq. A.2.1.9)
Fig.A.2.1(3B) Relationship between the concentration of the acetic acid in CCl₄ and water.

\[ C_y^* = 64.87 \cdot C_x^{0.5357} \]

or \( (C_y^* + 3.5)^2 = 5002(C_x + 0.0025) \)

- \( C_y \) expt.
- \( C_y \) cal. 1 (from \( C_y^* = 64.87 \cdot C_x^{0.5357} \))
- \( C_y \) cal. 2 (from \( (C_y^* + 3.5)^2 = 5002(C_x + 0.0025) \))