The reliability of experimental data on mass transfer obtained in packed absorption towers will depend on the satisfactory design of the absorption tower avoiding end-effects. The end-effects were either made negligible or altogether eliminated by proper design of the end selections, including the gas-distributor at the bottom end of the packed section and the liquid distributor at the top end of the packed section. If end-effects cannot be eliminated they have to be estimated and allowance made for correction of the same. The usual method to evaluate end-effects is to carry out experiments for different packed heights and plot $(HTU)_{OG}$ versus height for a given set of gas and liquid flow rates. Zero end-effect is indicated when the above plot shows no intercept.

In the present design, which was based on the design of King [1] and Shulman et al [2], the spray section at the bottom was eliminated by releasing the gas through a suitable distributor inside the packing and above the packing support. The spray effect or splashing at the top was also minimised by keeping the liquid distributor as close to the packings as possible, but without obstructing the flow of liquid.

The height of packing chosen should be such that there is enough solute in the effluent liquid to allow an accurate analytical determination of the driving force. The factors that may be considered are the size of packing,
the range of flow rates to be studied and the end effects. Generally, a short packed height may be sufficient for small packings and large height for larger packings. But when the height of packing is large inaccuracies in the driving force may occur at low liquid flow rates. On the other hand large heights are preferable so that the end effects taken as equivalent height of packing will be negligible compared to the total height. Taking all these factors into considerations a packed height of 70 cm was adopted in the present work. Heat effects due to humidification in the packed bed can be considerable and hence a separate provision was made for saturating the gas stream with water.

4.2 DESCRIPTION OF THE APPARATUS

The experimental set-up used is as shown in Fig. 4.1. Figure 4.2 shows a photograph of the experimental set-up. The absorption column is made of thick-walled Pyrex glass fitted with suitable packing support at the bottom. The packing support is positioned below the gas distributor. The liquid after flowing down the packed section was collected at the bottom end of the column in a specially devised liquid chamber with a 'U' bent outlet for constant withdrawal of the liquid. A separate stopcock was provided at the bottom of this chamber for withdrawal of liquid to facilitate hold-up measurement. HDPE tubes were used for gas and liquid lines. While handling chlorine gas stainless steel fittings for dry chlorine and glass fittings for wet chlorine were employed.

Thermometers (0-100 °C range) were used to measure the temperatures of the inlet and outlet streams. Air was supplied by a small air compressor fitted with an pressure reduction valve.
FIG. 4.1. SCHEMATIC DIAGRAM OF THE EXPERIMENTAL SET-UP.
Fig. 4.2  EXPERIMENTAL SETUP
Flow rates for gas and air were measured by calibrated capillary flow
meters. For the measurement of flow rate for liquid, rotameters and capillary
flow meters were used. Two specially devised glass chambers were attached
to the gas distributor for thorough mixing of the air-gas mixture.

The system chosen for the interfacial area measurement was
oxygen - sodium sulphite. The other systems chosen for mass transfer studies
were CO₂ - MEA and chlorine - water. In all the experimental runs the
effluent liquid from the tower was not recirculated but was collected
separately.

4.3 EXPERIMENTAL PROCEDURE

The following procedure was adopted for obtaining data on interfacial
area of contact using the system oxygen - sodium sulphite. Sodium sulphite
solution of the desired concentration was filled in the overhead tanks. The
oxygen cylinder was opened and the flow rate was adjusted to give the desired
value. It was saturated with water by passing it through a series of wash
bottles containing water. The liquid flow rate was then started and
distributed at the top of the column through the distributors. Initially a high
flow rate was used and after a few minutes it was brought down to the
desired value. The conditions were maintained for 20 minutes to establish
steady state conditions. Samples in duplicate were taken in 250 ml iodine
flasks. Temperatures of the inlet and outlet streams, flow rates were noted.
The samples were analysed by the procedure given in Appendix 9. The
interfacial areas were calculated by the method of Danckwerts et al[3] given
in Section 4.4.
The following procedure was adopted for obtaining data on mass transfer and holdup using the systems \( \text{CO}_2 \)-MEA and chlorine-water. The liquid was filled in the overhead tanks. The air compressor was started and the air rate was adjusted to the desired value by operating the pressure-reduction valve and the by-pass valve. The gas cylinder was opened and flow rate was adjusted to give the desired partial pressure by adjusting the needle valve. The liquid flow rate was then started to the experimental column. Initially the liquid flow rate was kept at a higher level and after about 5 minutes the liquid flow rate was brought back to the desired value. The level of the pool of liquid on the bottom section was maintained at a suitable level by adjusting the valve on the liquid outlet. The conditions were maintained for 20-30 minutes to establish steady-state conditions. Liquid samples in duplicate were taken in 250 ml iodine flasks. The outlet gas mixture was collected in a specially devised glass gas-holder. Temperatures of the inlet and outlet streams and flow rates were recorded. The data were computed according to the method given in Section 4.7.

The following procedure was adopted for measuring the operating holdup. After collecting the samples and noting the temperatures and the flow rates the steady level of the liquid pool in the bottom section of the column was marked. The liquid inlet and outlet valves were closed simultaneously. The liquid level was then allowed to rise in the bottom section due to draining of the liquid held-up in the interstices of the packing. After about 15 minutes of draining, the liquid from the bottom section was drained into a beaker until the liquid level again reached the steady state mark. The volume of the liquid gave the operating hold-up.
4.4 ANALYSIS OF SAMPLES

The samples obtained for the mass transfer runs were analysed for their composition as per the procedure given in Appendix 9.

4.5 PACKINGS AND SYSTEMS USED

4.5.1 Interfacial Area Measurements

In the present investigation runs were taken for the measurement of interfacial area in 58 mm i.d. Column made of Pyrex glass. The packings used were 6.35 mm Raschig rings. The packings were supplied by U.S. Stoneware Company, U.S.A. The system studied was oxygen - sodium sulphite. The physical properties of the liquid were measured for each experimental run.

4.5.2 Mass Transfer Studies

Mass transfer runs were obtained in 58 mm i.d. Column made of Pyrex glass using 6.35 mm ceramic Raschig rings. The systems studied were CO₂ - monoethanolamine and Cl₂ - water. The physical properties of the liquid such as density and viscosity were measured for each run. In the system where water was used as the absorbing liquid, double distilled chlorine free water was used.

4.5.3 Packing Characteristics

The packing characteristics such as voidage, number of pieces per cm³ and superficial area were experimentally determined.
The effective volume of the column was measured by filling the empty column with water up to the top end of the packed section and draining it to the level of the gas distributor. The effective volume of the column was then obtained from the volume of the water drained. The wet voidage was determined as follows. The column was filled with water and the packings were dropped a few at a time until it was filled to the desired level. The void volume was measured by slowly draining the column between two definite heights and finding the volume thus collected. The ratio of the volume of water collected and the empty volume of the column between the two fixed heights was reported as fractional voidage.

The number of pieces of packings per cm$^3$ was determined by counting the pieces as they were dropped into the column for a known volume. For small packings the total weight of packings filling the column for a known volume was noted and from the average weight of a definite number, the number of pieces per cm$^3$ was evaluated. The average surface area of a single unit of packing was determined by actually measuring the relevant dimensions of each of a representative number of the packings, by using a travelling microscope. From the knowledge of the number of pieces of packing per cm$^3$ the dry surface area $a_t$ expressed as cm$^2$/cm$^3$ was calculated. The values of the packing characteristics are given in Table 4.1.

4.6 EVALUATION OF PHYSICAL PROPERTIES

The average values of the physical properties such as density and viscosity of the gas mixture and the liquid at the measured temperatures and concentrations at the top and bottom ends of the column were calculated as follows.
<table>
<thead>
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<th>S.No.</th>
<th>Packing type</th>
<th>Packing used in the present work</th>
<th>Nominal size</th>
<th>Column diameter</th>
<th>dp, mm</th>
<th>Surface area</th>
<th>Packing used by Teller and Ford[10]</th>
<th>Packing used by Vivian and Whitney[13]</th>
<th>Packing used by Pearson et al[16]</th>
<th>Packing used by Haslam et al[17]</th>
<th>Packing used by Haslam et al[17]</th>
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<td>5.8</td>
<td>0.5764</td>
<td>6.6929</td>
<td>19.50</td>
</tr>
</tbody>
</table>

Surface area: $A_s = \pi dp^2$ cm$^2$
The values of density and viscosity of pure gases at different temperatures were taken from 'Fundamentals of Momentum, Heat and Mass Transfer' by Welty, J.R., Wilson, R.E. and Wicks, C.E. (p. 652). However, for the gas mixtures the properties of the gas mixtures at the respective compositions were calculated from the pure component properties of the two gases evaluated at the measured temperatures as a molar additive property knowing the mole fraction of each gas in the mixture. Thus, to obtain $\mu_G$ the viscosity of the gas mixture at the operating temperature, the pure component viscosities were used in the molar additive equation as given below,

$$\mu_G = \mu_A x + \mu_B (1-x) \quad (4.1)$$

where $\mu_A$ and $\mu_B$ are the viscosities of gases A and B in the gas mixture and $x$ is the mole fraction of A in the mixture. In a similar manner the average density of the gas mixtures were calculated from the pure component densities at the operating temperatures as follows,

$$\rho_G = \rho_A x + \rho_B (1-x) \quad (4.2)$$

The diffusivity of the solute gas in the gas phase at the operating temperature was estimated using the Wilke-Lee modification [100] of the Hirschfelder-Bird-Spotz method for mixtures of non-polar gas or of a polar with a non-polar gas as follows,

$$D = \frac{[0.00107 - 0.000246 \sqrt{\frac{1}{M_A} + \frac{1}{M_B}}] T^{3/2} \sqrt{\frac{1}{M_A} + \frac{1}{M_B}}}{P_t \left(r_{AB}^2 \frac{f(kT/e_{AB})}{T} \right)} \quad (4.3)$$

where $D$ - diffusivity, cm$^2$/sec

$T$ - absolute temperature, °K
The molecular diffusivity of the solute gas in the respective absorbing solvent at the operating conditions was estimated using the empirical correlation of Wilke and Chang [101] as follows,

\[
D_{AB} = \frac{7.4 \times 10^{-8} \phi M_B^{0.5} T}{V_A^{0.6} u_B^{1.0}} \tag{4.4}
\]

where
- \(D_{AB}\) - diffusivity of solute A in solvent B, cm\(^2\)/sec
- \(M_B\) - molecular weight of solvent
- \(\mu_B\) - viscosity of solvent, centipoises
- \(V_A\) - solute molal volume at normal boiling point, cm\(^3\)/gm mole
- \(T\) - absolute temperature, °K
- \(\phi\) - association factor for solvent
- 2.6, 1.9, 1.5, 1.0 for water, methanol, ethanol and unassociated solvents
4.7 METHOD OF CALCULATION

4.7.1 Calculation of Interfacial Area

The conditions of the experiment were chosen such that the following rate equation was valid,

\[ N_A^* a = a A^* \sqrt{\frac{2}{3}} k_2 D_{AB} A^* \]  \hspace{1cm} (4.5)

The values of solubility \( A^*/p \), the diffusivity and the rate constant \( k_2 \) at the operative conditions were taken from 'Gas-Liquid Reactions' by Danckwerts, P.V. (p. 255). Since the values of the reaction rate constant, the diffusivity and the solubility are available in C.G.S. units the interfacial area \( 'a' \) would have the dimensions \( \text{cm}^2/\text{cm}^3 \).

\( N_A^* a \) the rate of absorption of oxygen in sodium sulphite in gm moles per \( \text{cm}^3 \) per sec could be calculated as follows,

\[ N_A^* a = \frac{(C_1 - C_2)}{2} \times \frac{L}{V} \]  \hspace{1cm} (4.6)

where \( C_1 \) and \( C_2 \) - the concentrations of sodium sulphite at the bottom and top of the column, gm moles/\( \text{cm}^3 \)
\( L \) - volumetric flow rate of liquid, \( \text{cm}^3/\text{sec} \)
\( V \) - packed volume of the column, \( \text{cm}^3 \)

The interfacial area \( 'a' \) was estimated as,

\[ a = \frac{N_A^* a}{N_A^*} \]  \hspace{1cm} (4.7)
4.7.2 Calculation of Mass Transfer Rate Coefficients

Equations used in evaluating the overall mass transfer coefficients from the data of this investigation developed from the literature[81] are summarized here.

For the absorption of chlorine in water, the overall liquid phase mass transfer coefficient $K_{OL}^a$ is defined as follows,

$$K_{OL}^a = \frac{L}{\rho_L} \frac{c_1}{h} \frac{c_2}{C - C'} \frac{dC}{C'}$$

(4.8)

where $K_{OL}^a$ - overall mass transfer coefficient, 1/sec

$L$ - liquor rate, gm/cm$^2$ sec

$h$ - height of packing, cm

$C$ - concentration of chlorine in water, gm moles/cm$^3$

$\rho_L$ - density of liquor, gm/cm$^3$

subscripts 1 & 2 - conditions at the bottom and top of the tower

$*$ - concentration of chlorine in liquid in equilibrium with the partial pressure of chlorine in gas phase

A straight-line relationship for the equilibrium solubility of chlorine in water was assumed and the above equation was integrated and used in the following form,

$$K_{OL}^a = \frac{L}{\rho_L} \frac{C_1 - C_2}{(AC)_{Lm}}$$

(4.9)

where

$$(AC)_{Lm} = \frac{(C_1^* - C_1) - (C_2^* - C_2)}{\ln \left[ \frac{C_1^* - C_1}{C_2^* - C_2} \right]}$$

$C_1^*$ and $C_2^*$ - equilibrium concentrations of chlorine in water at bottom and top condition of the tower
The overall gas phase mass transfer coefficient for the absorption of CO$_2$ in monoethanolamine solution was estimated in a similar manner as follows,

$$K_{OGa} = \frac{G}{\rho_G h} \times \frac{C_{G1} - C_{G2}}{(\Delta C_{G})_{lm}}$$  \hspace{1cm} (4.10)

where

- $K_{OGa}$ - overall gas phase mass transfer coefficient, l/sec
- $G$ - gas rate gm/cm$^2$ sec
- $\rho_G$ - average gas density, gm/cm$^3$
- $C_{G1}, C_{G2}$ - concentration of CO$_2$ in the inlet and outlet gas mixtures gm moles/cm$^3$
- $(\Delta C_{G})_{lm}$ - log mean driving force, gm mole/cm$^3$