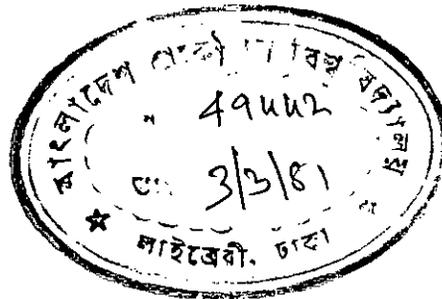


A STUDY OF MASS TRANSFER IN PACKED COLUMN

A thesis submitted in partial fulfilment of the requirements for the Degree of Master of Science in Engineering in the Department of Chemical Engineering.

T. 104



by

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#49442#

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CERTIFICATE OF THESIS WORK

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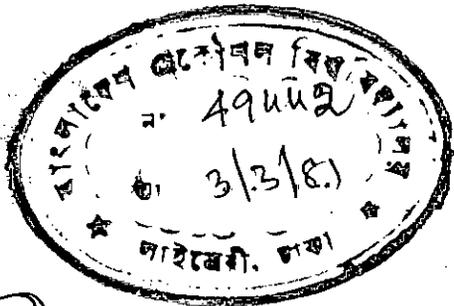
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I



ABSTRACT

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A mass transfer study in a packed column of diameter 5.287 cm was undertaken for $\text{CO}_2\text{-Na}_2\text{CO}_3$ system with 1N Na_2CO_3 solution. To test the usual hydrodynamic characteristics of packed column the pressure drop measurements with air-water system were undertaken and $\Delta P\text{-G}$ plots were drawn with L as the parameter. These plots showed usual trend as found in literature.

The mass transfer co-efficient was determined for $\text{CO}_2\text{-Na}_2\text{CO}_3$ system. The data show that the mass transfer rate increases with the liquid mass flux upto certain point, i.e., flooding point, and then decreases with the rise of liquid flow rate. As small amount of data were collected no general correlation for mass transfer was attempted.

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CHAPTER 1

1.1.0 INTRODUCTION

The separation of a selected component from a mixture of gases by a suitable solvent or liquid is an important chemical engineering unit operation/process encountered in process industries and chemical engineering design. The separation operation is usually accomplished by absorption of the desired component in the liquid solvent in a suitable contacting equipment. The absorption may be solely a physical phenomena or it may involve a chemical reaction. Equipments commonly used for various absorption operations are packed columns, plate columns or stirred tanks.

The rate of absorption for a given system is determined by the equilibrium relationship between the gas and the liquid concerned. It is also affected by pressure and temperature of the system. The separation of carbondioxide from a mixture is often encountered in many chemical processes. In some chemical processes, carbondioxide is itself the end product, e.g., dry ice or it is to be used as a raw material in other processes, e.g., use of carbon dioxide in urea synthesis. Whereas in other cases, carbondioxide removal is essentially a purification step and the product is discarded.

In Bangladesh, separation of CO_2 from gas mixtures is carried out in a number of process industries such as beverage, urea, dry ice using packed columns employing solutions of potassium carbonate, vetrocoke solution, and various organic amines.

This work presents an experimental study into the absorption of CO_2 by dilute Na_2CO_3 solution at room temperature and atmospheric pressure in a 5.287 cm diameter packed column.

CHAPTER - 2

2.1.0 LITERATURE REVIEW:

Packed column is frequently used in many chemical process industries to separate a component from a mixture of gases or liquid by another liquid. In general packed tower is preferred for corrosive service, liquid with tendency to foam, very high liquid-gas ratio; and applications where low pressure drops are required. A large volume of work has been carried out on mass transfer in packed tower for various systems(1,2).

The available literature on mass transfer related to absorption of carbondioxide by alkaline solution in packed column is reviewed in the following order.

- a. Equilibrium Relationships
- b. Mechanism of Mass Transfer.
- c. Absorption of Carbondioxide in Packed Column.
- d. Mass Transfer Equipments.

2.1 EQUILIBRIUM RELATIONSHIP.

2.1.1 Definition of Equilibrium

Equilibrium is a word denoting a static condition, the absence of change. In thermodynamics it is taken to mean not only the absence of change but absence of any tendency toward change on a macroscopic scale. Thus a

system at equilibrium is one which exists under such conditions that there is no tendency for a change of state to take place. Since any tendency toward change is caused by a driving force of one kind or another, the absence of such tendency indicates also the absence of any driving force. Hence a system at equilibrium may be described as one in which all the forces are in exact balance.

2.1.2 Underlying Principles

The equilibrium relationships are based on the laws of ideal solution modified according to certain generalizations of the thermodynamic properties of the system encountered. At low pressure, Raoult's law $p = P_v \cdot x$ and Dalton's law $P = \sum p$ may be combined with Avogadro's law to give the equilibrium relation;

$$P_v \cdot x = Py \quad \dots \quad (2.1a)$$

Under conditions where the equation (2.1a) applies, the compositions of vapors and liquids in equilibrium may be calculated. Even under atmospheric conditions, the deviations from Raoult's law may be appreciable. The equation may be used to give correct results, however, if fugacities are employed in place of p and P . However, at lower concentration, equilibrium relationship between a gas and a liquid may be obtained by Henry's law stated as

$$p = H \cdot x_e \quad \dots \quad (2.1b)$$

The term fugacity is defined by a thermodynamic equation for partial molal free energy or escaping tendency. Its use is explained by the fact that the partial molal free energies of a compound in the vapor and liquid phases must be equal at equilibrium. If it is assumed that the fugacities follow simple mixture rules in both phase, P_v in equation (2.1a) can be substituted by the fugacity f_P of the liquid, and for P , the fugacity f_π corresponding to the total pressure. The solution law then becomes,

$$f_P x = f_\pi \cdot y \quad \dots \quad (2.2)$$

The fugacities to be used in equation (2.2) can be determined in two ways, from p-v-T data of the substance and by direct determination of vapor-liquid equilibria. Various authors (3) have calculated the value of equilibrium constants for hydrocarbons and they are available in literature (2,3,4,32).

McCoy (5) presented a theoretical relationship for equilibrium between carbondioxide and sodium-carbonate-bicarbonate along with other variables such as temperature, concentration etc. This relationship was used and extended by Harte, Baker and Purcell (6) for the same system and by Siverts and Fritzsche (7) for carbondioxide and potassium carbonate-bicarbonate system.

Mathews and Williamson (8) describes a method for determining equilibrium relationship for potassium carbonate, potassium bicarbonate and carbon dioxide at different temperatures with a minimum amount of experimental data by modifying McCoy and Smith's (5) correlation.

2.1.3 Equilibrium Data

Equilibrium data are sometimes given in terms of solubility. The solubility factor of a gas in a liquid is defined in terms of temperature, the equilibrium partial pressure of the solute gas in the gas phase, and the concentration of the solute in the liquid phase. The total pressure of the system is also specified if the total pressure of the system exceeds 5 atm (2). In systems where the Henry's law does not hold, the solubility data is expressed as weight per cent of substance dissolved in liquid phase.

If Henry's law holds, solubility is defined by giving the Henry's law constant H and the temperature. The solubility data can be obtained for various systems from literature (2,3).

The systems for which the most reliable data have been obtained thus far are those which involves no chemical reaction with the solvent whatsoever, such as the absorption or desorption of oxygen or hydrogen in water, or those involving a small equilibrium degree of completion of the chemical reaction, such as the absorption of carbondioxide in water (1).

The solubility of carbon dioxide in sodium carbonate and potassium carbonate depends on the ratio of carbonate to bicarbonate, the total amount of salt in the solution, the temperature and partial pressure of the carbondioxide in the gas. The relationship encompassing the above variables at equilibrium between the phases was first worked out by McCoy (5) from theoretical consideration by the following relation,

$$\frac{2 X^2 C}{S P (1-X)} = M \quad \dots \quad (2.3)$$

Harte, Baker, Purcell (6) worked on $\text{CO}_2\text{-Na}_2\text{CO}_3$ systems and obtained more data and extended the relation (2.3) in the following form,

$$\text{CO}_2 = \frac{137f^2 N^{1.29}}{S(1-f)(365-t)} \quad \dots \quad (2.4)$$

The above relationship is valid only in the temperature range from 65° to 150°F and sodium normalities from 0.5 to 2.0.

Benson et al (9) studied extensively absorption of carbon dioxide by employing hot potassium carbonate solutions. They used the equilibrium data obtained by W.P. Haynes (10) which showed that equilibrium partial pressure at 248°F and 230°F of carbondioxide in the original carbonate solution as shown in Fig. (2.1)

Tosh et al (11) studied equilibrium for potassium carbonate, potassium bicarbonate, carbondioxide and water. The equilibrium data covered operating range of hot carbonate scrubbing system for removing carbondioxide from gas mixtures.

2.1.4 Absorption of Carbondioxide in Packed Column:

Williamson and Mathews (8) studied the effect on the rate of absorption of carbondioxide by the rate of flow of alkaline solution in three types of equipments, namely pebble packed tower, baffle tower, and special absorption box.

They concluded that for an increase of 100% in the rate of liquid flow, there is substantial increase in the rate of absorption in all three types of equipments. They also found that the rate of absorption of carbondioxide in potassium carbonate solution was practically independent of the concentration of potassium carbonate. The

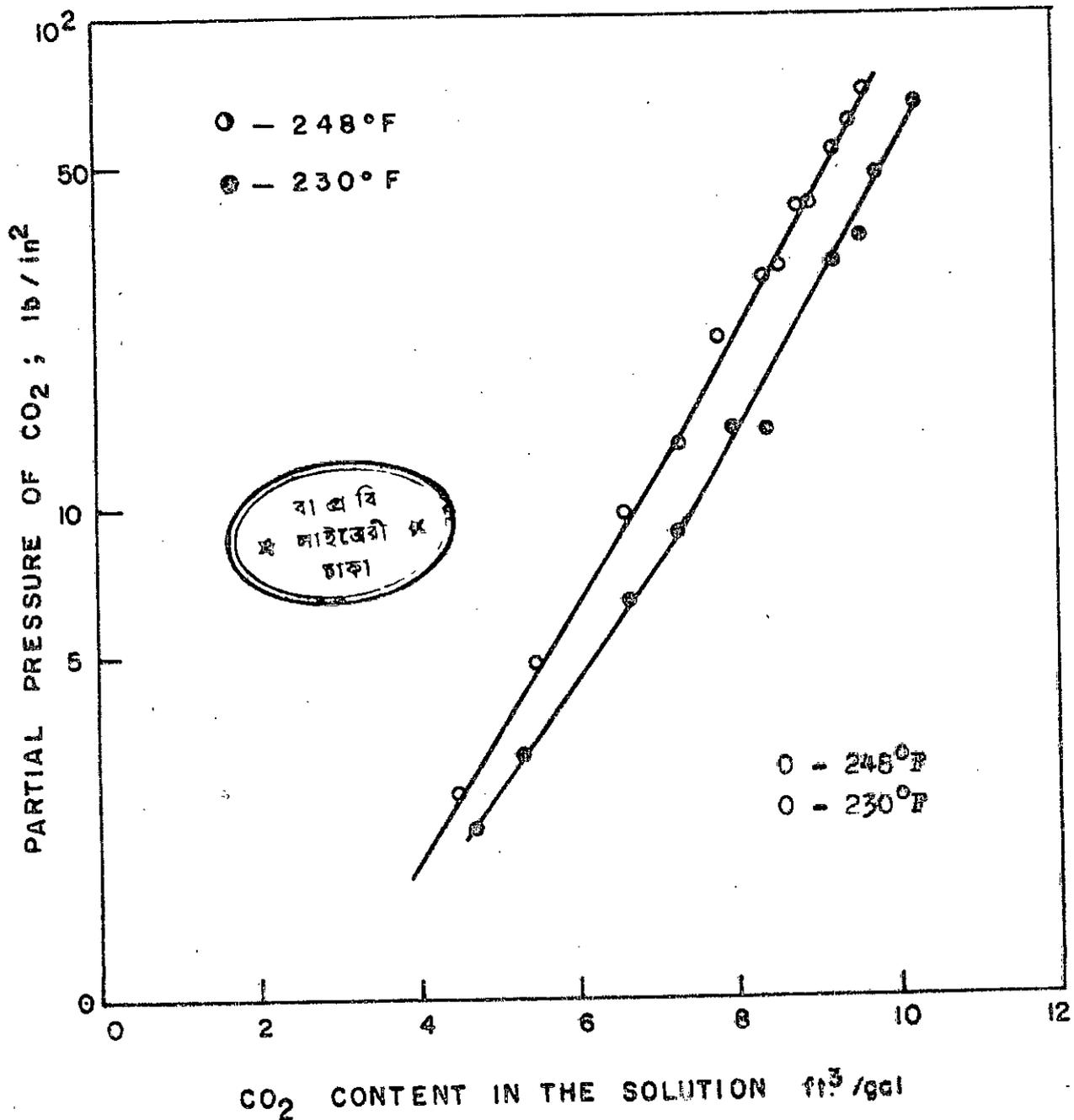


FIG. 2.1 EQUILIBRIUM PRESSURE OF CO₂ OVER 40% K₂CO₃ SOLUTION.

DATA: BENSON, FIELD, HAYNES (3)

rate of absorption increases with the rise of temperature upto a maximum of 70 to 75°C and then decreases as the temperature is increased.

Harte and Baker (12) studied the rate of absorption of CO₂ in sodium carbonate-bicarbonate system with 1-2 normal solution in sodium over a temperature range of 25 - 63°C in a wetted wall column and found that the co-efficient of absorption is independent of sodium normality, decreases approximately 40% with increasing bicarbonate normality from 0.2 to 0.6, and doubles with a temperature rise of approximately 22°C over the same bicarbonate normality range.

Payne and Dodge (13) undertook experiments to determine the rate of absorption of CO₂ by water and solutions of sodium carbonate-sodium bicarbonate and sodium hydroxide in a small tower packed with glass rings and the results reported in the form of an over-all gas absorption coefficient, $K_G a$. The effect of various variables on $K_G a$ is summarized below:

- i. The average over-all absorption coefficient is independent of CO₂ concentration of the gas.
- ii. A four fold increase in the gas rate has no effect on the co-efficient.
- iii. Increase in the liquid rate increases the co-efficient very materially.

- iv. A sixteenfold variation in sodium carbonate concentration has no significant effect on the co-efficient.
- v. Increase in temperature causes a great increase in the co-efficient.
- vi. Substitution of hydrogen for air as gas has little influence, if any, on co-efficient.
- vii. Addition of sodium bicarbonate to the sodium carbonate solution decrease the absorption co-efficient.
- viii. Addition of small percentages of sucrose and formaldehyde to the sodium carbonate solution appears to increase the co-efficient slightly.
- ix. The co-efficient for a dilute NaOH solution is much greater than for the sodium carbonate solutions.

The results of the above authors also show that the rate of absorption of CO_2 by sodium carbonate solution is less than by pure water under comparable conditions. This is contrary to the results of Davis and Crandall (14) who found the initial rate in dilute sodium carbonate to be more than twice as great as in water. Davis (15) repeated the experiments of Davis and Crandall (14); and explained the discrepancy of the results by the fact that

Payne and Dodge (15) performed experiments on continuous counter-current absorption in a packed tower and Davis and Crandall (14) undertook experiment in a batch stirred liquid and hence the difference in results.

Liquid phase mass transfer co-efficient was measured for the absorption of CO_2 into water in co-current flow by Raymond, C; (10) in packed column. The co-efficients were lower by as much as a factor of three with a significant gas rate effect. Liquid flow rates ranged upto 60,000 lbm/ft²hr.

P.V. Danckwerts (17) reviewed useful published information concerning industrial absorption of carbon dioxide; methods which can be used for quantitative design of absorption apparatus; there is very little reliable information about interfacial area and transfer co-efficients in various types of equipments.

2.1.5 Correlation for Mass Transfer Rate.

Ellis, J.E(18) used graphical solution of the problem of absorption accompanied by chemical reaction put forwarded by Van Krevelen (19,20) to correlate absorption rate of carbon dioxide in potassium carbonate solution. Estimates of the effective wetted area of the packing were made but

the insufficient data are available to permit scaling-up of absorption co-efficients with any degree of accuracy. The above author correlated data for 2-inch packed column by the following relation,

$$K_G a = (0.1 - 0.064f) L^{1/3} \dots (2.5)$$

which is valid for f , in the range of 0.25 to 0.70 and $L = .08$ and $0.8 \text{ gm/cm}^2 \text{ sec}$. The result of packed tower having diameter of 8 ft was correlated by the following relationship (18)

$$K_G a = (0.014 - .0087f) L^{1/2} \dots (2.6)$$

with the limitations $f = 0.45 - .77$ and $L = .27$ to $1.20 \text{ gm/cm}^2 \text{ sec}$.

Sherwood and Holloway (21) proposed an equation equivalent to

$$H_G = 1.31 (G''^{0.31}/L''^{0.33}) S^{0.5} \text{ ft} (2.7)$$

$$20 < G'' < 1,830 \text{ lb/in ft}^2; 168 < L < 6,100$$

$$\text{and } H_L = \beta \left(\frac{L''}{\mu_L} \right)^{0.22} S_{cL} \text{ ft} \dots (2.8)$$

on the basis of evaporation of water into air and for systems where the resistance is entirely on the liquid side.

Vivian and Whitney (22) from their work on absorption of sulfur dioxide in water in packed column with 1-in Raschig rings correlated the results by,

$$H_G = 1.24 G''^{0.3}/L''^{0.25} \text{ ft} \dots (2.9)$$

and,

$$H_L = 0.37 L^{0.18} \dots \quad (2.10)$$

Morris and Jackson (23) give the following formula for the height of a gas transfer unit,

$$H_G = 74 \frac{\epsilon_T v_G^{0.25}}{R_G C_a'} \left(1 + \frac{v_L}{v_G}\right)^{-0.75} \left(\frac{293}{T_G}\right) \quad (1.11)$$

A different correlation is proposed by Van Krevelen and Hoftizer (24). They examined critically the existing data on absorption in packed tower and presented them in the following form,

$$Sh_G^1 = 0.2 Re_G^{0.8} Sc_G^{1/3} \dots \quad (2.12)$$

Where Sh_G^1 = Sherwood number

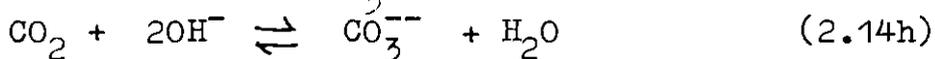
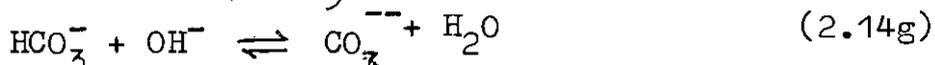
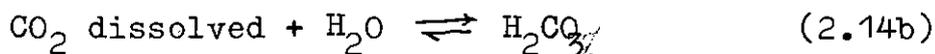
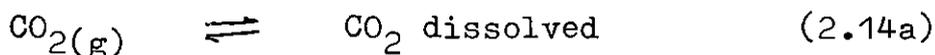
$$= (K_G)_p \frac{d_p}{D_G} \dots \quad (2.13)$$

2.2.1 Mechanism of Absorption in Sodium Hydroxide, Potassium Hydroxide and Sodium Carbonate-Bicarbonate Solution:

The absorption of CO_2 by alkaline solution has been studied by several workers (25). In most of the cases the overall absorption rate is determined by the rate of diffusion in the liquid phase or the rate of chemical reaction

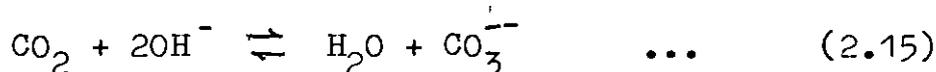
occurring in the liquid phase. The problem of determining the true mechanisms is difficult, because the principal factor which affects the liquid film resistance has a similar effect on the rate of chemical reaction. Temperature, for example, has a large effect on diffusivities and viscosities of liquids, and the combined effect of these variables is quantitatively similar to the effect of temperature on the rate of chemical reaction. The observed effects of temperature consequently are of little value in distinguishing between processes controlled by diffusion and processes controlled by chemical reaction. The determination of the absorption mechanism is further complicated because of the chemical kinetics and, in fact, the actual chemistry of the process is not well understood.

Payne and Dodge (13) list eight probable reactions which may be involved in the absorption of CO_2 in sodium carbonate, sodium bicarbonate and sodium hydroxide solution. They are



The ionic reactions are considered to be rapid, but the rates of the other reactions, which are not well known.

Various assumptions as to which of these reactions may be controlling lead to different pictures of the mechanism of the absorption process. Thus Hatta (25) assumes that the controlling reactions are (2.14a), (2.14b) and (2.14g) and that (2.14g) is much more rapid than (2.14f). Eucken and Grutzner (26) conclude that the overall reaction is



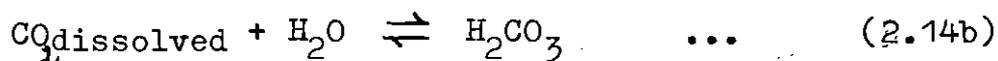
and goes directly in the presence of free hydroxide and the rate of reaction is very rapid. Whether or not the reaction occurs in one or two stages, if it is assumed along with Hatta (25) that the reaction occurs at the stagnant film at the interface, then it may be shown that the rate of absorption follows the following equation,

$$N_A = \frac{C_{Ai} + (DB/D_A) \cdot q}{X_L/D_A} \quad \dots \quad (2.16)$$

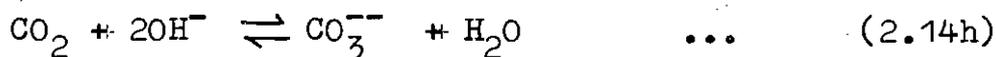
As the reactions between ions are very fast, it is probable that such reaction may be neglected when considering the rate of absorption of CO_2 ; however that still leaves reactions (2.14b), (2.14f) and (2.14a) which may be conceivably slow enough to influence the absorption rate. The evidence on this point is rather meagre and somewhat conflicting. Thus McBain (27) noted that the neutralization of dissolved CO_2 by potassium hydroxide was relatively a

slow reaction.

Eucken and Grutzner (26) have studied the rate of overall reaction represented by the sum of reactions (2.14b) and (2.14c) and found it to be relatively slow, with velocity constant of about $.09 \text{ sec}^{-1}$ at 18°C . This agrees with McBain's observation and indicates that the reaction (2.14b) i.e.,



is the limiting step in the reaction between CO_2 and alkalies. On the other hand, when an excess of alkali was present so that the concentration of HCO_3^- ion was negligible, they considered that reaction (2.14h) i.e.,



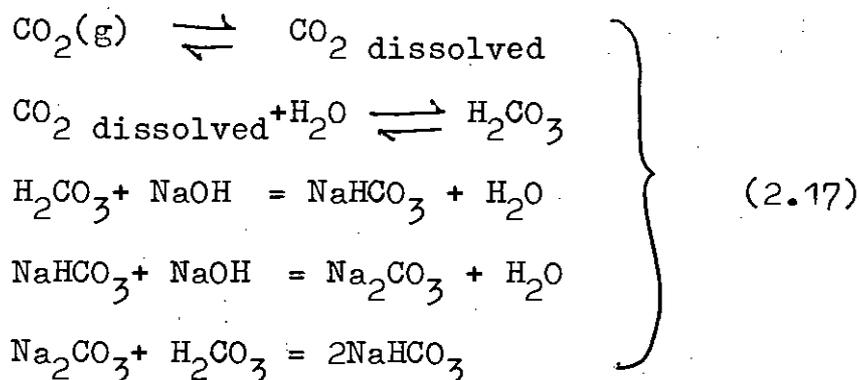
took place directly and that it was quite rapid, Saal (28) studied the velocity of reactions (2.14b) and (2.14f) and found that the reaction (2.14f) went directly without reaction (2.14b) as one of its step because he found it fairly rapid.

In addition to the various chemical reactions involved, one may postulate a liquid film with corresponding diffusion of various ions and of dissolved CO_2 across it. The observed absorption rates are probably the



net result of the combination of various diffusion rates and chemical reaction rates, but in view of the limited knowledge of chemical kinetics and diffusion phenomena, it becomes futile to attempt to postulate any definite mechanism.

An experiment was performed by the School of Chemical Engineering at Cornell University (29) on the absorption of CO_2 by caustic soda solution. The result of their experiment showed that there was a definite decrease in the rate of absorption with increase in the concentration of CO_2 in the gas. They postulated the absorption mechanism as follows,



of the above reactions, the absorption of CO_2 as such in water, and the interaction between CO_2 and water to form H_2CO_3 is reversible.

Unless the carbonic acid is rapidly and completely removed from the interfacial zone by interaction with NaOH

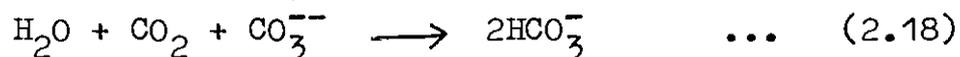
or Na_2CO_3 considerable back pressure of carbondioxide is developed and the rate of absorption decreases.

At very low concentration of CO_2 the sodium hydroxide diffuses, or is carried by convection to the interface fast enough to react with and remove the carbonic acid as fast as it is formed. Thus, under such condition the over-all rate of absorption depends on the rate at which the CO_2 is brought to the interface and the rate at which CO_2 in solution reacts with the water to form carbonic acid. At very low concentrations, this latter factor becomes negligible, because even the CO_2 present as such, in solution will be carried away from the surface as fast as it dissolves.

Again, if the concentration of CO_2 is increased, the rate of access of alkali to the surface becomes of increasing importance. As a result, the liquid film becomes more important and the overall rate of absorption shows a slight but definite decrease.

2.2.2 Absorption in Carbonate-Bicarbonate Buffer Solution:

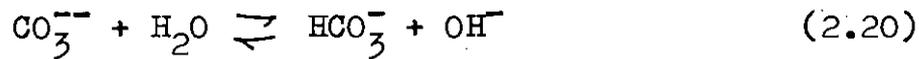
The overall reaction taking place during CO_2 absorption in buffer solution is,



the rate determining step being,



followed by ionic reaction,



The hydroxyl (OH^-) concentration is held almost constant by the buffer action of the carbonate - bicarbonate equilibrium at a level which is much lower than in the case considered for absorption in hydroxide. If the tower is very high, the carbonate to bicarbonate ratio may substantially decrease from top to the bottom of the tower (30).

Different aspects of modeling on mass transfer mechanism put forwarded by various workers (1) may be found elsewhere (2)

2.3.0 MASS TRANSFER EQUIPMENTS

The common characteristic of the mass transfer equipments is that they provide large interfacial area of contact between the two fluid phases, in which mass is interchanged. The following are the different equipments commonly used.

- i. Packed Tower
- ii. Plate Tower
- iii. Spray Tower
- iv. Wetted Wall Column
- v. Agitated Vessel.

2.3.1 Packed Tower

Packed tower is the most widely used forms of continuous contacting tower for the absorption of a pure gas from a mixture in a liquid or for the removal of one of the components from a mixture of gases. It also finds application in liquid-liquid extraction. Various aspects of the design, construction and operation may be found in these references (1,2).

2.3.2 Plate Tower

The most important type of finite stage contactor (Plate tower) is the bubble cap plate unit, although sieve plate , turbogrid trays, cascade towers, and other speciality types of units are also used in industrial operations. Many of the principles that apply to bubble cap plate are also applicable to other types of finite stage contactor. The design and construction of various plate towers are available in these references (1,2).

2.3.3 Spray Tower

In spray tower large interfacial contact area is obtained by nozzle to give a sufficiently subdivision which is largely maintained through the column. The simplest spray tower consists of an empty chamber into

which liquid is sprayed at the top and gas is introduced at the bottom. Such equipment has the advantages of very low pressure drop through the spray chamber and simple inexpensive constructions.

2.3.4 Wetted Wall Column

Wetted wall column have been used extensively on an experimental scale because they offer an opportunity for determining interfacial area of contact. In wetted wall column the liquid passes as film and gases pass over the film transferring masses. These type of towers are commercially unimportant because of their low contacting efficiency.

2.3.5 Agitated Vessels

These are vessels in which the gas is introduced directly into the liquid and is mechanically dispersed by means of a suitable agitator. They are used mainly where the absorption is accompanied by a slow chemical reaction between the dissolved gas and some constituent of the liquid, and sufficient time has to be allowed for this reaction to proceed to the required extent. Alternatively, they may be used where close control of the process is required in order to prevent the formation of undesirable by-products.

Agitated vessels are suitable for batch operation. If used for a continuous process, counter-current flow cannot be obtained within individual vessels, and it is then almost always necessary to use a series of vessels arranged in multiple stages. The pressure-drop through agitated vessels is high compared with that for packed tower, and where this is an important factor the use of a series of packed tower, through which a proportion of the liquid is circulated repeatedly, might be preferable.

CHAPTER - 33.1.0 STATEMENT OF THE OBJECTIVE

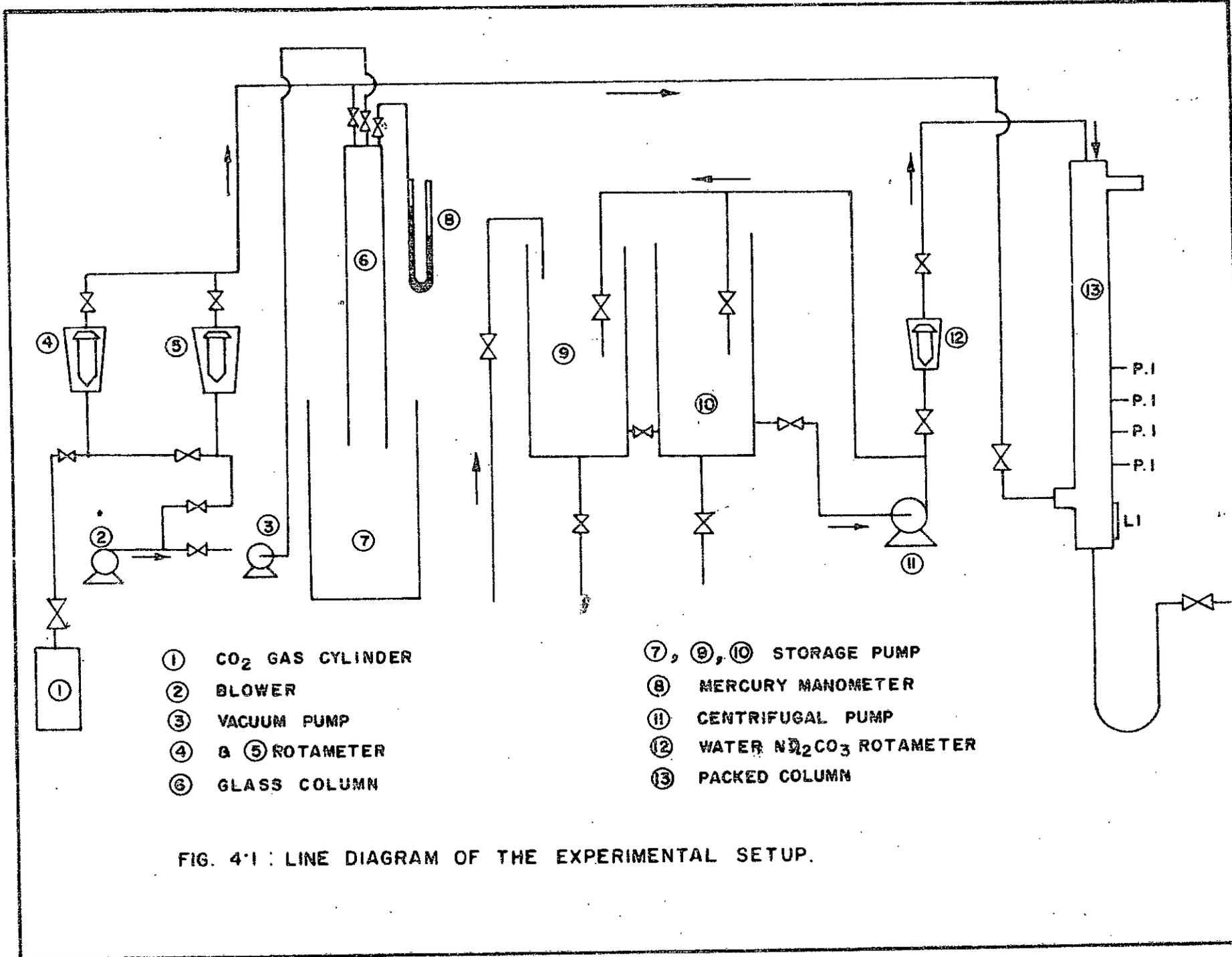
The objective of the present work is to undertake an experimental study of the mass transfer rate in the packed column using $\text{CO}_2 - \text{Na}_2\text{CO}_3$ system. The present work studied the following;

- i. The measurement of the pressure drop in the packed column using air-water system.
- ii. The overall mass transfer co-efficient for $\text{CO}_2 - \text{Na}_2\text{CO}_3$ system and
- iii. Effect of flow rates on the rate of mass transfer for $\text{CO}_2 - \text{Na}_2\text{CO}_3$ system.

The equilibrium partial pressure of CO_2 in solution was determined by the equation given by Harte, Purcell and Baker (6). The solubility data of CO_2 in water which was used in this experiments was that given by Seidell (31). Because a small number of experiments were performed no correlation involving various parameters was attempted. The experimental results of this work are compared with the data available in the literature.

CHAPTER - 44.1.0 EXPERIMENTAL SET UP AND PROCEDURES:

A schematic diagram of the experimental arrangement is shown in Figure 4.1. Sodium carbonate solution from the storage drums having capacity of 116 liters each was pumped by a centrifugal pump to the top of the column through a rotameter. The liquid at the top of the column was distributed by a shower, which was placed just above the packing. The mixture of air and CO_2 was supplied to the bottom of the packing distributor plates. Air and CO_2 were metered separately. The CO_2 was supplied from the carbondioxide cylinder through pressure regulator. Air from air blower at a pressure of 20.32cm of water was supplied. The length of CO_2 and air carrying pipe line was sufficient that the CO_2 and air were thoroughly mixed before it entered into the packed column. The arrangement of pipe mixing is shown in figure 4.2 The detail description of various component of the experimental set up is given below. Solutions of sodium carbonate were prepared and stored in the two drums connected in series. The tanks were covered at the top to prevent the absorption of CO_2 from air.



- ① CO₂ GAS CYLINDER
- ② BLOWER
- ③ VACUUM PUMP
- ④ & ⑤ ROTAMETER
- ⑥ GLASS COLUMN

- ⑦, ⑨, ⑩ STORAGE PUMP
- ⑧ MERCURY MANOMETER
- ⑪ CENTRIFUGAL PUMP
- ⑫ WATER N₂CO₃ ROTAMETER
- ⑬ PACKED COLUMN

FIG. 4.1 : LINE DIAGRAM OF THE EXPERIMENTAL SETUP.

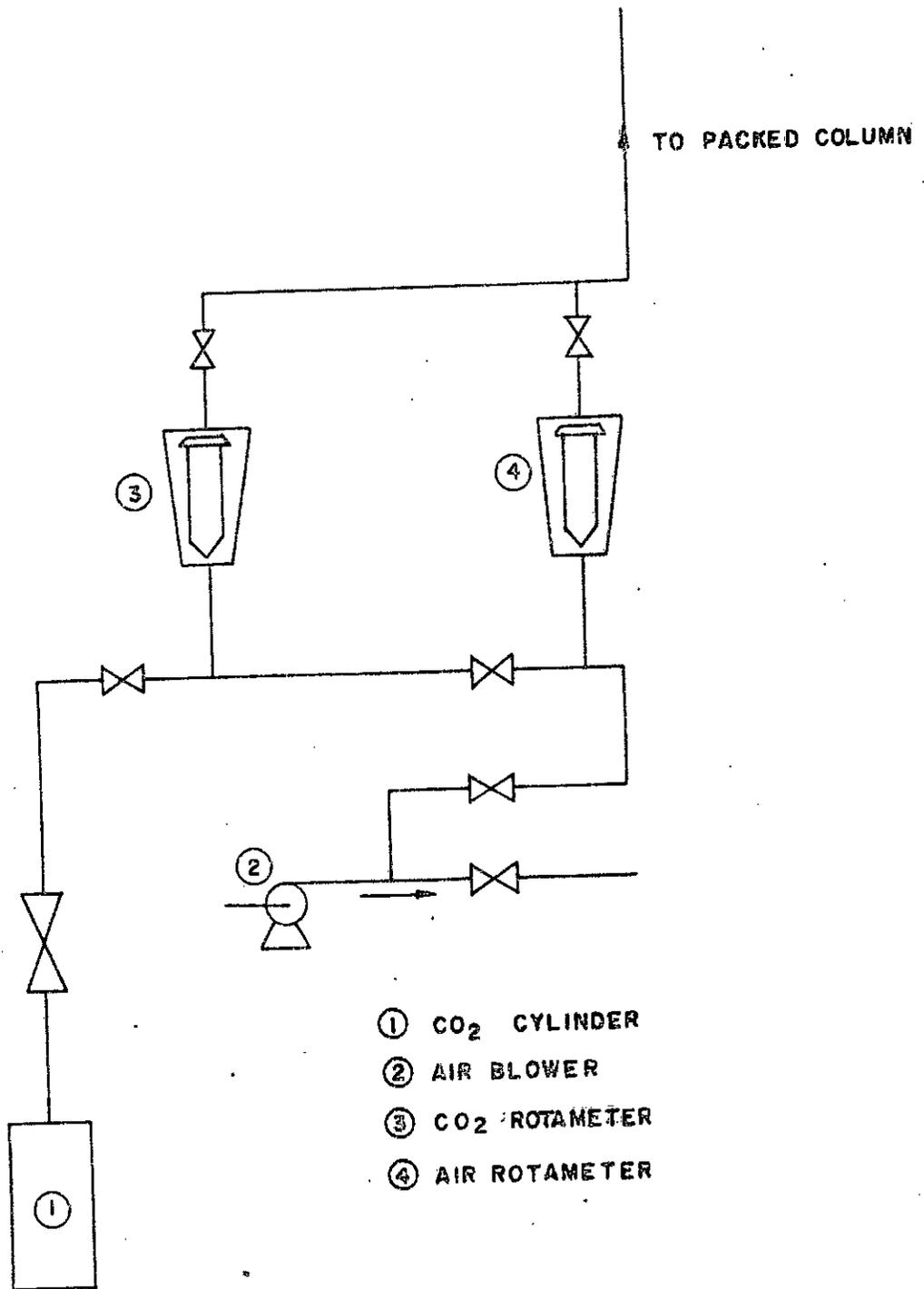


FIG. 4.2 : ARRANGEMENT FOR MIXING CO₂-AIR

The flow rates of CO_2 and air were measured by Fischer Porter rotameters independently. The sodium carbonate flow rate was measured by another Fischer-Porter rotameter. The mixed gases entered at the bottom of the tower and flowed upward through a distributor having the shape of a right circular cone of 5.08 cm height, made of .317 cm mild steel plate and in which .476 cm dia holes were drilled.

There were five pressure tapings on the packed column. These tapings were made of .635 cm dia mild steel pipe, 7.62 cm in length and spaced at 15.24 cm interval from the bottom of the column. The tapings were welded into the wall of the column and connected by PVC tubing to water manometer to measure the pressure drop in the column.

4.1.1 Packed Tower

The 1.524 meter long packed column was made of mild steel pipe with 5.287 cm ID. The cross sectional view of the tower is shown in figure 4.3. The packing material used was glass raschig rings having dimension of .6cm x .4 cm x .6cm and the height of the packed section was 43.565 cm.

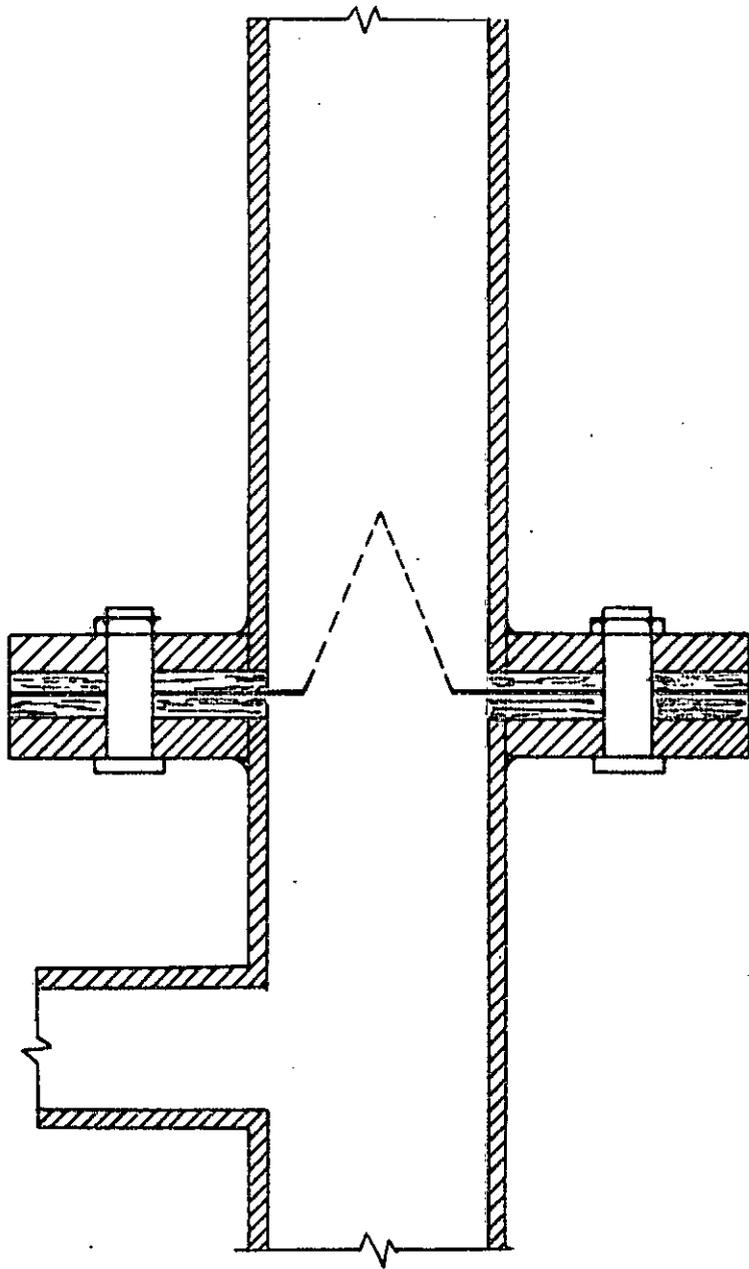


FIG. 4.3 CROSS-SECTIONAL VIEW OF THE PACKED TOWER

4.1.2 Calibration of Rotameter

A special set up was designed for the calibration of rotameters for air and carbondioxide and is shown schematically in figure 4.4. For this purpose a glass column of 10.16 cm. inside diameter and 1.219 meter high was chosen. Rotameters were calibrated by displacing a given volume of water from glass column with the gas entry at the top of the column. The column was filled with water by displacing air with vacuum pump connected at the top. The calibration curves for CO_2 and air are given in figures 4.1.1 - 4.1.2. Relevant temperature and pressure were measured. The calibration curves for water and 1N Na_2CO_3 solutions are shown in figures 4.1.3 and 4.1.4 taken at room temperatures.

4.1.3 Preparation of Na_2CO_3 Solution

Measured amount of sodium carbonate was mixed with measured amount of water in the tank and the solution was thoroughly mixed by continuous pumping, so that the solution became of uniform composition having normality of one approximately.

4.1.4 Experimental Procedure

The experimental studies can be divided into two sections;

- i. to study the pressure drop characteristics in the packed column to ensure that the column follows usual hydrodynamic characteristics.

- 1. BLOWER
- 2. VACUUM PUMP
- 3 & 4. ROTAMETER
- 5. 5" DIAMETER GLASS COLUMN
- 6. DRUM
- 7. MANOMETER
- 8. CO₂ CYLINDER
- 9. GAS REGULATOR

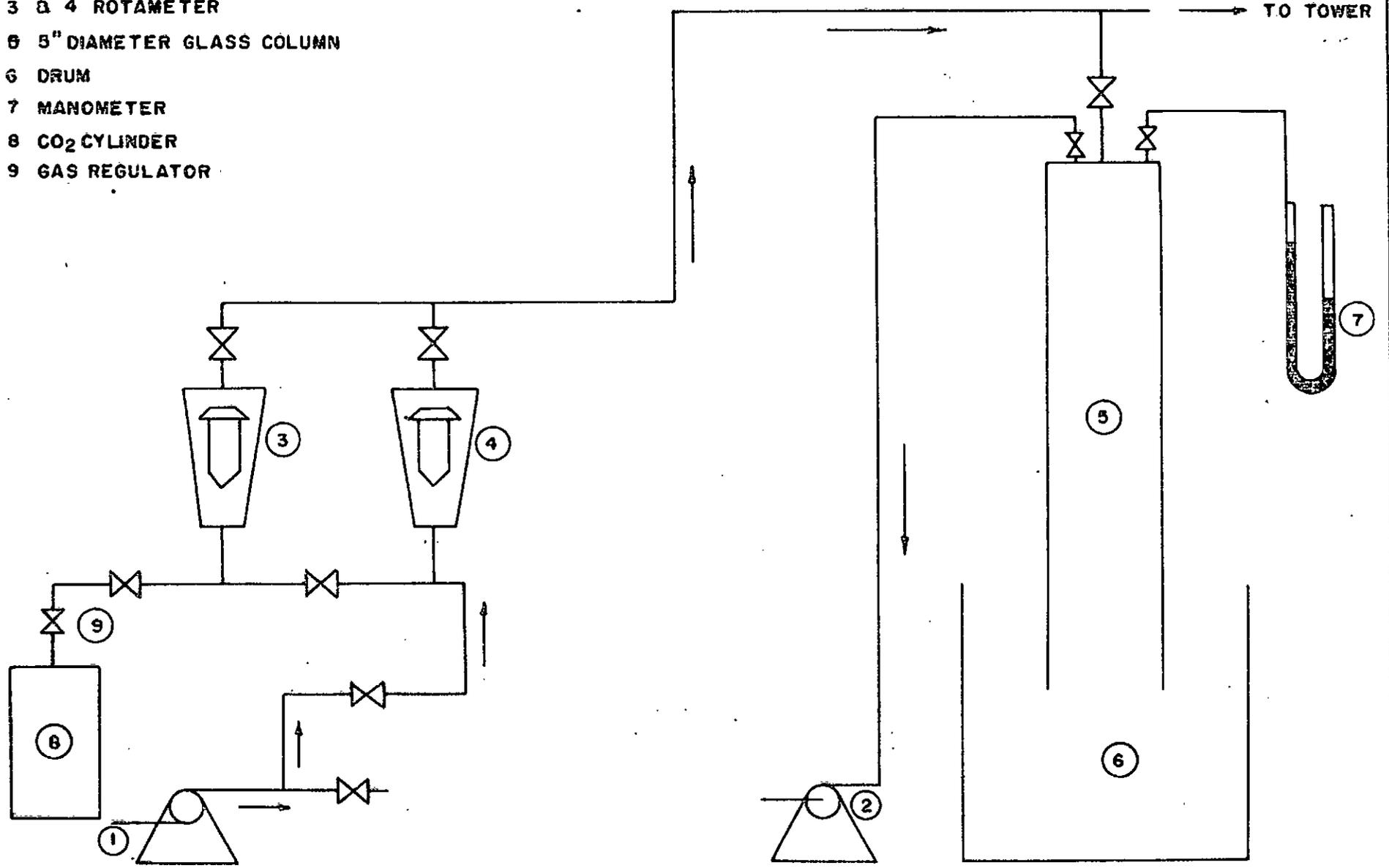


FIG. 4. 4: SCHEMATIC DIAGRAM FOR CALIBRATION OF AIR AND CO₂ ROTAMETERS

- ii. to study the mass transfer rate in the column for carbondioxide-sodium carbonate system.

Initially it was decided that mass transfer rate for the above system would be studied in 5.08 cm ID glass column for flow visualization. But due to lack of facility to drill pressure tapping on the glass column to measure the pressure drop characteristics, the experimental set-up was abandoned and a new-set up with M.S. column was built. The pressure drop was measured by water manometer connected to the tapping as shown in figure 4.5. Some difficulties were encountered in measuring the pressure drop because some water accumulated near the pressure tappings after running the equipment for some time. To ensure correct reading the PVC tube connected to the tapping was cleared of water before each reading of the manometer.

A number of pressure drop measurement were taken for various gas and liquid flow rates. Pressure drop measurements were taken for dry packing also.

Mass transfer studies was begun by pumping a fixed rate of sodium carbonate solution into packed column and by passing required amount of CO_2 and air by two rotameters. At the start of the experiment normality of Na_2CO_3 solution was determined. The atmospheric temperature and

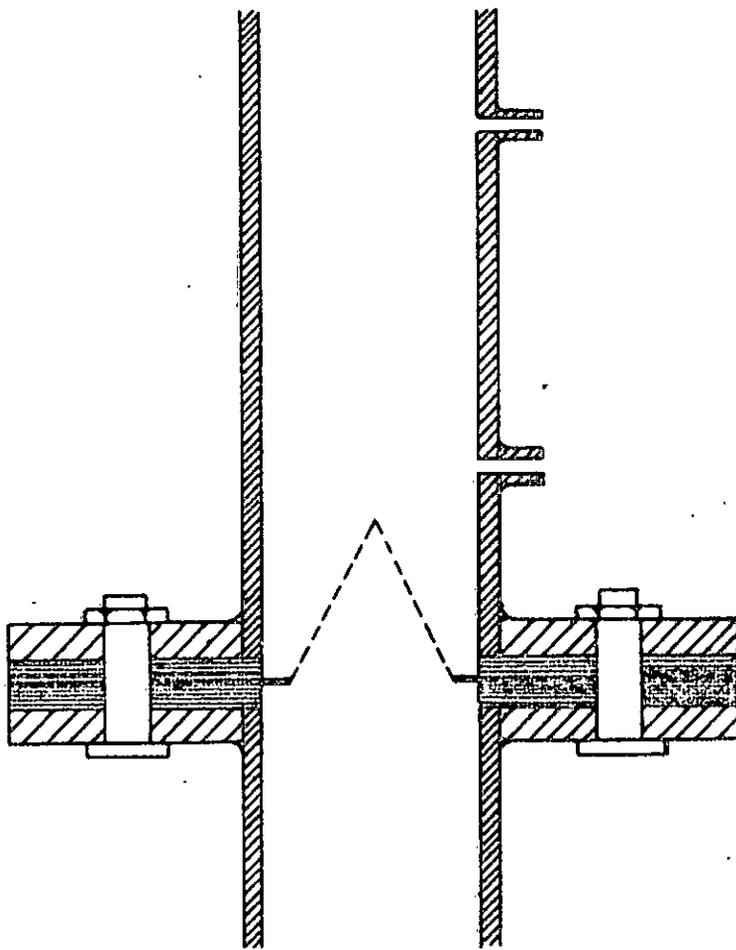


FIGURE 4.5 SCHEMATIC DIAGRAM FOR PRESSURE TAPPING IN THE COLUMN.

pressure were also measured. All the samples were collected at least 15 minutes after the start of the equipment, so that steady state was attained by the system. The steady state condition for the system was determined by constant Na_2CO_3 composition at the bottom. The outlet and inlet gas samples were collected at the top and bottom of the tower for each experiment. The outlet liquid was collected after definite interval of time to determine the amount of carbondioxide absorbed in the liquid. However, for mass transfer calculation, mass balance based on Na_2CO_3 analysis was used.

By keeping gas flow rates fixed, experiments were carried out by varying liquid flow rate, because it was difficult to have the same gas composition with varying flow rates of CO_2 and air seperately.

4.2.0 METHODS OF ANALYSIS

4.2.1 Inlet and Outlet Gas:

The inlet and outlet gases were analyzed by a orsat analyzer for carbon dioxide using saturated KOH solution.

4.2.2 Inlet and Outlet Liquid:

The inlet liquid was analyzed by taking 20 cc of the solution in a flat bottom flask and 1-2 drops of

methyl orange was added as indicator. The sample was then treated with standard hydrochloric acid. The above procedure was repeated three times and the average was taken.

The outlet liquid was first analyzed with methyl orange for total alkalinity by taking 20 c.c. of the sample. And to another 20 cc of the outlet liquid 25cc. of standard sodium hydroxide was added. Then the flask with the above mixture was slightly heated and slight excess of 10% BaCl_2 was added. To this was added one to two drops of phenolphthalein as indicator, this mixture was then titrated with standard hydrochloric acid to determine the amount of CO_2 absorbed.

4.3.1 Measurement of Surface Area of Packing

For the determination of the surface area and hence the inside diameter of the raschig rings, the density of the ring material was calculated by usual procedure. The other dimension of the rings were given by the manufacturer. From these data the total surface area per rashig ring was calculated. This was multiplied by the total number of packing to get the total surface area due to raschig rings only. As the diameter of the column was small the inside wall area of the packed.

section of the column was added to the area due to packing to calculate the total mass transfer area. The number of rings introduced into the column was determined by counting and by taking weight as was done by Lobo, et.al(32).

4.3.2 Measurement of Porosity

The porosity of packed section of the tower was determined by introducing measured volume of water up to the top surface of the packing and using the following equation (4.3.2),

$$\epsilon = \frac{\text{volume of Void}}{\text{Volume of Void} + \text{Volume of Packing}} \quad (4.1)$$

and the average porosity was calculated.

CHAPTER - 55.1.0 RESULTS

Pressure drop measurement were taken for the packed column with air-water system to ensure whether the system fulfills hydrodynamic characteristics usually encountered. Pressure drop measurements were taken over a range of gas and water mass flux and the results are plotted in the figures 1-4 as Δp against G with L as parameter. In figure (5) are shown the plots of $\Delta P-G$ for dry packing.

Experimental measurements on CO_2 absorption by Na_2CO_3 solution of approximately 1N are presented in tables (I-IV). In figures (6) and (7) are plotted as N'_A against L with G as parameter.

For the calculation of $K_G a$, the equilibrium relationship given by Harte, Baker and Purcell (6) based on the work of McCoy (5) was used to calculate the driving force. Over-all mass transfer co-efficient $K_G a$, was calculated using the relationship given below

$$K_G a = \frac{N'_A}{(p_{CO_2} - p_{e,CO_2})} \quad (5.1)$$

The driving force $(p_{CO_2} - p_{e,CO_2})$ used in calculation was that at the bottom of the tower in view of (a) difficultly to analyse accurately the outlet gas

(b) the absorption rate itself was small. The values of $K_G a$ calculated are shown in tables 1-4 and $K_G a$ are plotted against L as shown in figures (8) and (9) with G as parameter.

Similarly the value of K_G was calculated from $K_G a$ by assuming "a" to be the total surface area of packing and wall area of the packed section of the column per unit volume of packing and the results are shown in Tables 1-4 and K_G - L plots are drawn in figures 10-11. The porosity of the packed section of the tower was determined and the results are given in table 5 and the average porosity was found to be 0.387.

To calculate the total surface area of a packing, the density of the packing material was determined, shown in table 6, to calculate the inside diameter of packing. The total surface area per packing was 2.119 cm^2 and the column wall area used was found to be 901.805 cm^2 .

CHAPTER - 66.1.0 DISCUSSION OF THE RESULTS6.1.1 Pressure Drop Data

49442

The experimental data obtained for pressure drop for air-water and dry packing system in the packed tower show that pressure drop characteristics are similar to those observed in literature (33). The nature of $\Delta p-G$ are shown in figures 1-5. According to Chilton and Colburn (34) most of the pressure drop in the turbulent range is due to contraction and expansion losses and changes in direction as the gas flows through the irregular orifices formed by the packing particles, and only a small part approximately 10% is due to skin friction. For dry packing the graph shows that at low gas mass flux the pressure drop increases slowly and for high flow rates the curve bends up steeply indicating that the pressure drop is sensitive at higher gas velocity. But for wet packing the liquid circulated over the packing occupies an appreciable fraction of the voids and reduces the mean free cross section open to passage of the gas. At constant superficial gas velocity, therefore, the actual gas velocity is increased and the pressure drop is appreciably greater than when the packing is dry. Upto the gas velocities at loading approximately same variation of pressure drop is observed, with counter current

liquid flow as in the case of dry packing. Further increase in gas rate changes the slope of pressure drop-gas flux curve abruptly indicating that the flooding has commenced. The different pressure drop-velocity curves show the same trend as described above.

The amount of CO_2 absorbed per minute and $K_G a$ are given in Tables 1-4. The value of $K_G a$, was calculated using the relationship given by equation (5.1), the value of p_{e,CO_2} was calculated using relationship given by Harte, Baker and Purcell (6) and the driving force ($P_{\text{CO}_2} - p_{e,\text{CO}_2}$) used was that at the bottom of the tower in view of (a) difficulty to analyze accurately the outlet gas (b) the absorption rate itself was small. Average driving force whether logarithmic or average would not be much different from the value used because of small amount of actual concentration changes in the system. Nevertheless, an average driving force at the terminal conditions would provide a better prediction of $K_G a$. From all these tables it becomes evident that the amount of carbon dioxide absorbed in the liquid and hence the value of $K_G a$ is sensitive to liquid flow rate i.e. the value of $K_G a$ increases as the value of flow rate is increased upto certain point. At the liquid rate of $1823.27 \text{ gm/hr.cm}^2$ the value of $K_G a$ was found to be $0.284 \text{ gm/atm hr.cc}$. Payne and Dodge (15) measured the value of $K_G a$ for the absorption of CO_2 in 1.4% Na_2CO_3 solution in a packed

column having ring dimension of 10mm x 8mm x 10mm as .128 gm/atm.cc.hr. The discrepancy of the result may be due to the fact that in case of this experiment there were turbulent flow and interfacial area was greater, hence the greater rate of transfer. When the flooding condition is attained in the tower the rate of absorption and consequently the value of $K_G a$ is decreased by an appreciable extent.

The various graphs 6-11 were plotted as $K_G a$ -L, K_G -L, N'_A -L with G as the parameter. It is observed from the figures that the values of $K_G a$ increases with liquid mass flux upto flooding velocity. When the flooding velocity is reached ~~with~~ the value of $K_G a$ is decreased with increase in L indicating the amount of CO_2 absorbed is less when flooding in the column occurs. This is shown figures 8 and 9. In the figures showing the plots of K_G - L, N'_A - L show the same trend as $K_G a$ - L plot.

CHAPTER - 77.1.0 CONCLUSIONS

- i. Pressure drop measurements with dry and wet packing show that the pressure drop characteristics in the column are the same as usually encountered(33)
- ii. Absorption of CO_2 by Na_2CO_3 solution (1H) increases with L upto the flooding point, and beyond flooding point, the rate of absorption is decreased.
- iii. In the absence of large amount of data, no correlation for mass transfer rate has been attempted.

CHAPTER - 88.1.0 RECOMMENDATION FOR FUTURE WORK

More experimental investigation should be carried out with $\text{CO}_2 - \text{Na}_2\text{CO}_3$ system. Effect of different parameter such as concentration, temperature, different types and sizes of packing, packing height, flow rates etc. on the rate of absorption of CO_2 in Na_2CO_3 solution should be studied. Other absorption medium should be tried. The future research programme should be undertaken in such a manner which would enable one to understand the operational characteristics of a packed column and help to collect design data.

CHAPTER - 9

NOMENCLATURE

- a, a' surface area of packing per unit volume of packing, cm^2/cm^3
- C_T, N total sodium concentration, normality.
- C' gas mixture constant.
- C_{Ai} concentration of solute A at the interface, gm moles/ cm^3
- d_p particle diameter, cm
- D_G gas diffusivity, cm^2/sec .
- D_A liquid diffusion co-efficient for component A., cm^2/sec .
- D_B liquid diffusion co-efficient for component B, cm^2/sec .
- f_p fugacity of the pure liquid under its own vapor pressure
- f_{π} fugacity of the pure component as a vapor at the total pressure of the mixture.
- f, X fraction of total base present as bicarbonate.
- G'', G flow rate of gas per unit cross-sectional area of column, $\text{gm}/\text{hr}.\text{cm}^2$.
- H_G height of the gas phase mass transfer unit, cm
- H_L height of the liquid phase transfer unit, cm
- $K_G a$ over all gas film mass transfer co-efficient, $\text{gm}/\text{atm cc}.\text{hr}$.
- K_G over all gas film co-efficient of mass transfer $\text{gm}/\text{atm cm}^2.\text{hr}$.
- L'', L liquid mass flux, $\text{gm}/\text{hr}.\text{cm}^2$
- M numerical co-efficient used in equation (2.4)
- N_A diffusion rate of A, $\text{gm}/\text{hr}.\text{cm}^2$

N'_A	amount of CO_2 absorbed in Na_2CO_3 solution per unit volume of packing, gm/hr cm^3
p	partial pressure
P	total pressure.
P_v	vapor pressure of pure component.
p'	partial pressure in atmosphere.
p_{e,CO_2}	equilibrium partial pressure of CO_2 , mm Hg.
Δp	pressure drop, in of water/ft of packing.
q	in the case of absorption and chemical reaction, the concentration of the substance reacting with the solute, gm moles/ cm^3
R_G	gas phase packing factor
$R_{e,G}$	modified Reynolds number, $G''/a \mu_G$
S	solubility of CO_2 in water, gm-moles/liter
Sc_G	Schmidt number based on gas
Sc_L	liquid phase Schmidt number
T_G	absolute temperature of the gas
t	temperature, $^\circ\text{F}$.
v_G	gas velocity through packing, cm/sec.
v_L	velocity of liquid phase, cm/sec.
x	mole fraction of the solute in the liquid phase.
X_L	effective thickness of the liquid film
y	mole fraction of the solute in gas phase
β	constant for different size of the rings
μ_L	liquid viscosity
μ_G	gas viscosity
ϵ, ϵ_T	porosity.

CHAPTER - 10

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Obs. no.	Reference	Rotameter reading			Time of collection of sample after start (min)	Gas analysis at the inlet		% CO ₂ by vol.	Gas analysis at outlet		% CO ₂ by vol.	Total alkalinity by titration					
		CO ₂	Air	Na ₂ CO ₃ Soln.		initial vol. (ml)	final vol. (ml)		initial vol. (ml)	final vol. (ml)		Vol. of sample taken (ml)	buret reading		Diff.	Average (ml)	Strength of HCl used (N)
													initial (ml)	final (ml)			
1		25	10	8	25	49.8	40.0	19.6787	60.6	50.9	16.0066	20	16.3	49.4	33.1		0.57704
					45	50.6	40.6	19.7628	49.8	41.0	17.67	20	0.0	33.0	33.0	33.066	0.57704
									49.8	41.2	17.69	20	0.0	33.7	33.1		0.57704
									49.8	41.0	17.67	20	14.15	47.35	33.2	33.1	0.57704
													49.8	41.0	17.67	20	0.0
2	Table - I	25	10	10	65	50	37.8	24.4	43.4	42.6	1.843	20	0.0	32.6	32.6	32.7	0.57704
									48.4	40.2	16.942	20	0.0	32.8	32.8		0.57704
					100	49	37.4	23.67	42.6	35.9	15.727	20	0.0	33.6	33.6		0.57704
												20	0.0	33.7	33.7	33.733	0.57704
												20	0.0	33.9	33.9		0.57704
3		25	10	11	120	44.5	34.0	23.595	46.14	37.6	18.9655	20	0.0	40.2	34.2		0.57704
									44.6	43.5	2.46636	20	0.0	33.95	33.95	34.1166	0.57704
					160	45.8	35.6	22.27	45.9	42.2	8.064	20	0.0	34.20	34.20		0.57704
												20	2.0	37.25	35.25	34.9833	0.57704
												20	0.0	34.9	34.9		0.57704
											20	0.0	34.8	34.8		0.57704	

Date: June 18, 1978, temp. 28°C, Pressure: 758 Hg.

Na₂CO₃ and NaHCO₃ Analysis by Titration

Vol. of sample taken (ml)	Buret reading		Diff. (ml)	Average diff. (ml)	Strength of acid used (N)	Volume of NaOH used (ml)	Strength of NaOH (N)	Conversion to bio-carbonate	Bicarbonate fraction	Na ₂ CO ₃ present initially (ml)	% conversion to bicarbonate.	comment
	initial (ml)	final (ml)										
20	0.9	17.5	16.6		0.1137146	25	0.100583	0.0831	0.0327	16.33 ml	3.31694	
19.5	17.5	34.2	16.7	16.6	0.1137146	25	0.100583	ml. of 0.5770 HCl		of 0.7704 HCl.		
20	0.0	16.5	16.5		0.1137146	25	0.100583					
20	0.0	18.0	18.0	17.8	0.1137146	25	0.100583	0.8336	0.0251	16.33	2.55262	
20	1.0	18.6	17.6		0.1137140	25	0.100583	ml of 0.5770 HCl		ml of 0.5770 HCl		
20	0.2	17.55	17.35	17.8	0.1137140	25	0.100583	0.8497	0.0259	16.33	2.60172	
20	17.58	35.85	18.27		0.1137140	25	0.100583	ml of 0.5770 HCl		ml of 0.5770 HCl		
20	0.0	13.35	13.35	13.65	0.1137140	25	0.100583	0.1664	0.0493	16.33	5.09645	
20	0.0	13.95	13.35		0.1137140	25	0.100583	ml of 0.5770 HCl		ml of 0.5770 HCl		
20	0.0	8.15	8.15		0.1137140	25	0.100583	2.9059	0.0851	16.33	8.89771	
20	0.0	15.45	7.45	7.35	0.1137140	25	0.100583	ml. of 0.5770 HCl		ml of 0.5770 HCl		
20	15.4	22.6	7.20		0.1137140	25	0.100583					
20	0.0	20.45	20.45		0.1137140	25	0.100583	3.7284	0.1065	16.33	11.41598	
20	0.0	20.85	20.85	21.0	0.1137140	25	0.100583	ml. of 0.5770 HCl		ml. of 0.5770 HCl		
20	0.0	22.0	22.0		0.1137140	25	0.100583					

Obs. no.	Reference	Meter reading			Time of collection of sample after start (min)	Gas analysis at the inlet		% CO ₂ by vol.	Gas analysis at outlet		% CO ₂ by vol.	Total alkalinity by titration					
		CO ₂	Air	Na ₂ CO ₃ Soln.		initial vol. (ml)	final vol. (ml)		initial vol. (ml)	final vol. (ml)		Vol. of sample taken (ml)	buret reading		Diff.	Average (ml)	Strength of HCl used (N)
												initial (ml)	final (ml)				
1		28	12	8	15	47.6	39.4	17.22689	49.6	39.6	19.556	20	0.0	38.25	38.25		0.5770
									49.2	40.0	18.699	20	0.0	38.1	38.1	38.13	"
2		28	12	9	35	57.4	45.2	21.254	51.0	41.2	19.2156	20	0.0	38.1	38.1		"
									78.6	65.3	16.9211	20	0.0	38.15	38.15	38.083	"
3	Table - III	28	12	$\frac{1.083 \text{ lhm}}{\text{min}}$	15	49.5	39.	19.5959	47.6	38.3	19.5378	20	0.0	39.3	38.3		"
									52.4	42.8	18.3206	20	0.0	38.25	38.25	38.283	"
4		28	12	$\frac{0.3125 \text{ lhm}}{\text{min}}$	35	55.6	44.6	19.7814	58.2	47.6	18.213	20	0.0	19.0	19.0		1.1612
									65.4	52.8	19.266	20	0.0	19.05	19.05	19.0166	"
5		28	12	10	40	51	41	19.6078	61.0	48.2	20.9836	20	0.0	19.0	19.0		"
									13.8	55.9	12.382	20	0.0	19.0	19.0	18.983	"
												20	0.0	18.95	18.95		"

Date: 29 and 30 June 1978; Temp. 28°C, pressure: 757 mm Hg.

Na₂CO₃ and NaHCO₃ Analysis by Titration

Vol. of sample taken (ml)	Buret reading		Diff. (ml)	Average diff. (ml)	Strength of acid used (N)	Volume of NaOH used (ml)	Strength of NaOH (N)	Conversion to bio-carbonate	Bicarbonate fraction	Na ₂ CO ₃ present initially	% conversion to bicarbonate.	com
	initial (ml)	final (ml)										
20	ex discarding				0.11317146	25	0.0995769	0.5353897		38.03 ml of		
20	11.4	30.0	18.95	19.7175	"	25	"	ml of 0.0140411	0.577049N	0.577049N	1.4078	
20	0.0	19.4	19.40		"	25	"	HCl				
20	0.0	20.9	20.9		"	25	"	0.290717			0.7644	
20	3.0	22.6	19.6	20.416	"	25	"	ml of 0.0076337	0.577049N			
20	22.6	43.35	20.75		"	25	"	HCl				
20	0.1	16.55	16.45		"	25	0.10254	1.057073				
20	0.0	16.50	16.50	16.56	"	25	"	ml of 0.0274455	0.577049N		2.76283	
20	0.0	16.75	16.75		"	25	"	HCl				
20	0.0	13.25	13.25		"	25	"	1.82233				
20	0.0	13.55	13.55	13.30	"	25	"	ml of 0.047621	0.577049N		4.7918	
20	0.0	13.10	13.10		"	25	"	HCl				
20	0.0	14.75	14.75		"	25	"	1.5259098				
20	0.0	14.85	14.85	14.80	"	25	"	ml of 0.0399499	0.577049N		4.01238	Flooding started
20	0.0	14.85	14.85	14.80	"	25	"	HCl				

Obs. no.	Reference	Rotameter reading			Time of collection of sample after start (min.)	Gas analysis at the inlet		% CO ₂ by vol.	Gas analysis at outlet		% CO ₂ by vol.	Total alkalinity by titrat				
		CO ₂	Air	Na ₂ CO ₃ Soln.		initial vol. (ml)	final vol. (ml)		initial vol. (ml)	final vol. (ml)		Vol. of sample taken (ml)	buret reading		Diff.	Average (ml)
												initial (ml)	final (ml)			
1		25	14	$\frac{0.3125}{hr}$	20	51.6	44.0	14.72868	49.2	42.0	14.634	20	0.0	39.0	39.0	
									45.7	39.4	14.785	20	0.0	38.95	38.95	38.966
									48.7	42.0	13.7577	20	0.0	38.95	38.95	
2		25	14	$\frac{1.083}{hr}$	42	65.1	55.6	14.5929	61.2	52.4	14.379	20	2.0	40.9	38.9	
									44.8	38.4	14.2857	20	0.0	38.9	38.9	38.916
												20	0.0	38.95	38.95	
3	Table - III	25	14	8	60	60.0	65.2	15.337	61.7	53.0	14.10048	20	0.0	38.9	39.9	
									54.2	46.1	14.94464	20	0.0	38.95	38.95	38.916
									48.9	41.9	14.31	20	0.0	38.90	38.9	
4		25	14	9	90	53.4	45.0	15.73	Flooding occurred,			20	0.0	38.95	38.95	
									gas sample could not be collected			20	0.0	38.9	38.9	38.933
												20	0.0	38.95	38.95	
5		25	14	10	8 mins. after expt.	59.8	49.6	17.056	57.6	50.6	12.1527	20	0.0	21.75	21.75	
									64.7	60.1	7.1097	20	5.1	25.4	20.30	21.025
									58.4	50.4	13.6986	20	-	-	-	-

Date: July 3, 1978, temp: 29°C, press: 29.9 in Hg.

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Na₂CO₃ and NaHCO₃ Analysis by Titration

Vol. of sample taken (ml)	Buret reading		Diff. (ml)	Average diff. (ml)	Strength of acid used (N)	Volume of NaOH used (ml)	Strength of NaOH (N)	Conversion to bio-carbonate	Bicarbonate fraction	Na ₂ CO ₃ present initially	% conversion to bicarbonate.	comment
	initial (ml)	final (ml)										
20	1.0	14.45	14.5		0.1137146	25	0.10254	1.6044152				
20	discarded			14.5				ml of 0/041174		38.8333	4.13154	
20	0.0	14.55	14.55		0.1137146	25	0.10254	0.5700763N		0.5700763N		
20	0.0	17.9	17.9		"	25	"	0.9361823				
20	0.0	17.8	17.8	17.85	"	25	"	ml of 0.024056		"	2.41077	
20	0.0	17.85	17.85		"	25	"	0.5770763N				
20	0.0	18.45	18.45		"	25	"	HCl				
20	0.0	18.30	18.30	18.375	"	25	"	0.8314593				
20	0.0	18.30	18.30	18.375	"	25	"	ml of 0.0213651		"	2.14109	
20	discarded							0.5770763N				
20	0.0	9.7	9.7		"	25	"	HCl				
20	0.0	10.7	10.7	10.2166	"	25	"	2.4588225				
20	0.0	10.25	10.25		"	25	"	ml of 0.0631546		"	6.33173	Flooding started
20	0.0	15.2	15.2		"	25	"	0.5770763N				
20	0.0	15.2	15.2		"	25	"	HCl				
20	0.0	15.2	15.2		"	25	"	1.43486				
20	0.1	15.6	15.5	15.35	"	25	"	ml of 0.0682455		"		Water entered into gas collect syringe.
20	0.1	15.6	15.5	15.35	"	25	"	0.5770763N				
20	0.1	15.6	15.5	15.35	"	25	"	HCl				

Obs. no.	Reference	Rotaneter reading			Time of collection of sample after start (min)	Gas analysis at the inlet		% CO ₂ by vol.	Gas analysis at outlet		% CO ₂ by vol.	Total alkalinity by titration					
		CO ₂	Air	Na ₂ CO ₃ Soln.		initial vol. (ml)	final vol. (ml)		initial vol. (ml)	final vol. (ml)		Vol. of sample taken (ml)	buret reading		Diff.	Average (ml)	Strength of HCl used (N)
		initial (ml)		final (ml)													
1	25	10	8	14	47.8	37.8	20.92	49.6	39.8	19.758	20	0.0	38.0	38.0	38.0	0.577	
								45.0	36.0	20.00	20	0.0	38.0	38.0			
2	20	10	9	35	50.4	40	20.80	47.8	38.5	19.456	20	1.0	39.1	38.1			
								50.0	39.8	20.40	20	0.0	37.95	37.95	38.0166		
								49.0	38.2	22.04	20	0.0	38.0	38.0			
3	25	10	10	90	49.2	37.6	23.577	58.4	47.8	18.15	20	0.0	38.0	38.0	38.033		
								discarded		20	0.0	38.0	38.0				
								discarded		20	0.0	37.95	37.95	37.9833			
								48.2	38.8	19.50	20	0.0	38.0	38.0			
								Flooding occurred no sample collected		20	0.0	38.0	38.0	38.0			
5	25	10	12	-	49.6	37.5	24.395				20	0.0	38.0	38.0			
											20	0.0	38.0	38.0	38.0		
6	25	10	13	7	49.9	25.8	48.29				20	0.0	38.0	38.0			
											20	0.0	38.0	38.0			
7	25	10	14	-	70.7	52.4	25.884				20	0.0	38.0	38.0			
											20	0.0	37.95	37.95	37.95		
											20	0.0	37.95	37.95	37.95		
						ovg.	24.36				20	0.0	37.95	37.95	37.95		

Table IV

Date: 24 June 1978, temp. 28°C, Pressure :757 mm Hg.

Na₂CO₃ and NaHCO₃ Analysis by Titration

Vol. of sample taken (ml)	Buret reading		Diff. (ml)	Average diff. (ml)	Strength of acid used (N)	Volume of NaOH used (ml)	Strength of NaOH (N)	Conversion to bio-carbonate	Bicarbonate fraction	Na ₂ CO ₃ present initially	% conversion to bicarbonate.	comment
	initial (ml)	final (ml)										
20	11.0	30.2	19.2	19.35	0.1137146	25	0.0995769	0.5009038		38.11 ml		
20	5.0	24.5	19.5		"	25	"	ml of 0.0131816 0.577049N HCl		of 0.577049N HCl	1.31415	
20	7.0	26.2	19.2		"	25	"	0.4680732		38.11 ml of		
20	2.0	38.4	18.4	19.5166	"	25	"	ml of 0.0123123 0.577049N HCl		0.577049N HCl	1.22802	
20	2.1	41.95	20.95		"	25	"					
20	3.0	18.5	15.5		"	25	"	1.2037655				
20	9.0	15.85	15.85	15.7833	"	25	"	ml. of 0.01316505 0.577049N HCl		"	3.16642	
20	0.0	16.0	16.0		"	25	"					
20	0.0	11.1	11.1		"	25	"	2.133367				
20	0.0	11.3	11.3	11.066	"	25	"	ml of 0.0561659 0.577049N HCl		"	6.64649	
20	0.0	10.8	10.8		"	25	"					
20	0.0	10.85	10.85	10.875	"	25	"	2.171006				
20	21.0	31.9	10.9		"	25	"	ml of 0.057137 0.577049N HCl		"	5.69578	Flooding occurred, out- gas analysis done.
20	32.1	36.1	4.1		"	25	"	3.47004				
20	46.2	40.5	4.30	4.283	"	25	"	ml of 0.0913168 0.577049N HCl		"	9.1039	"
20	40.5	44.95	4.45		"	25	"					
20	1.0	10.8	9.8	9.95	"	25	"	2.35329				
20	20.03	30.1	10.1		"	25	"	ml of 0.061983 0.577049N of HCl		"	6.1443	"

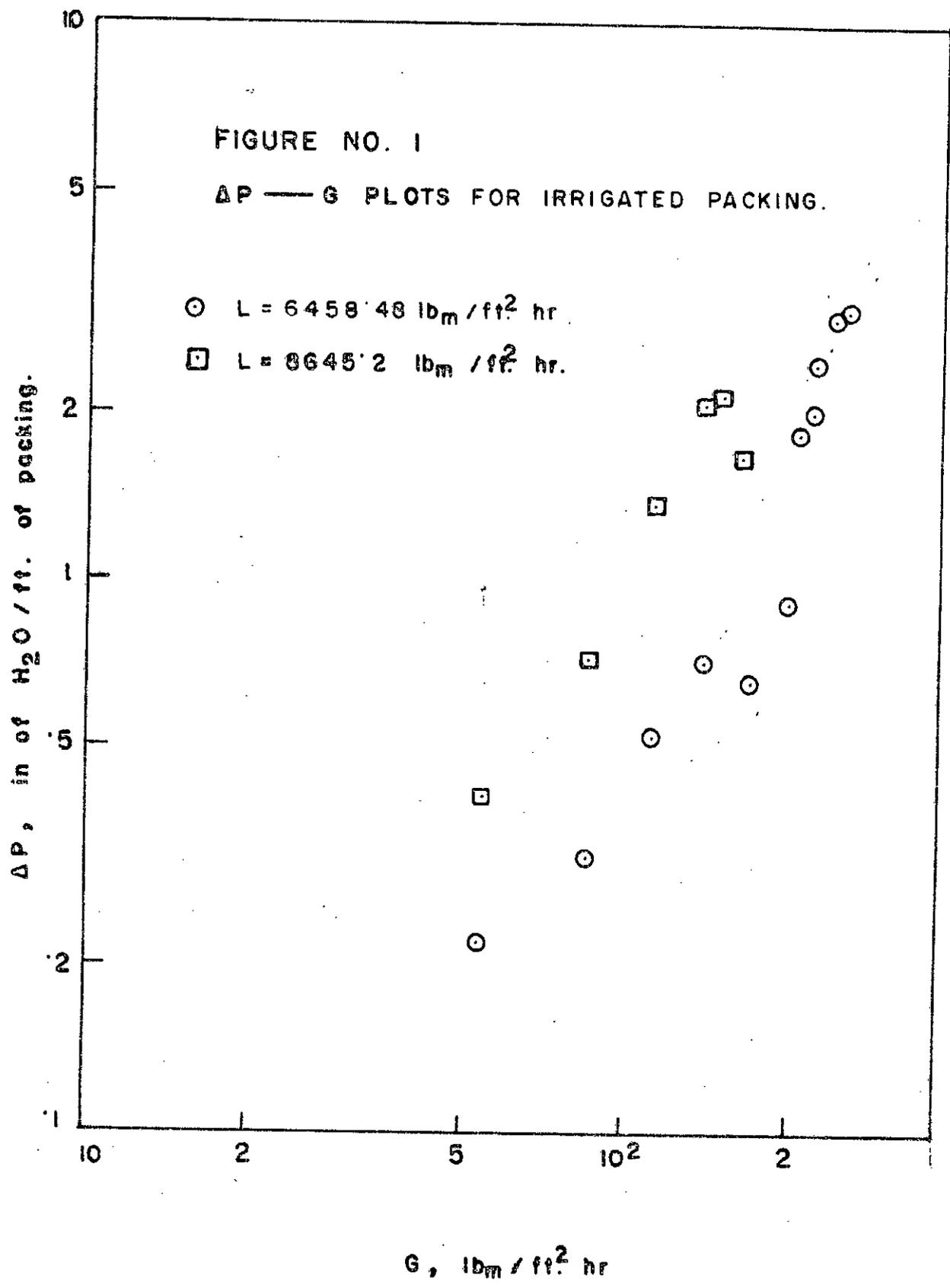


FIGURE NO. 2

$\Delta P - G$ PLOTS FOR IRRIGATED PACKING.

\circ $L = 246.72 \text{ lb}_m / \text{ft}^2 \text{ hr.}$

Δ $L = 9484.31 \text{ lb}_m / \text{ft}^2 \text{ hr.}$

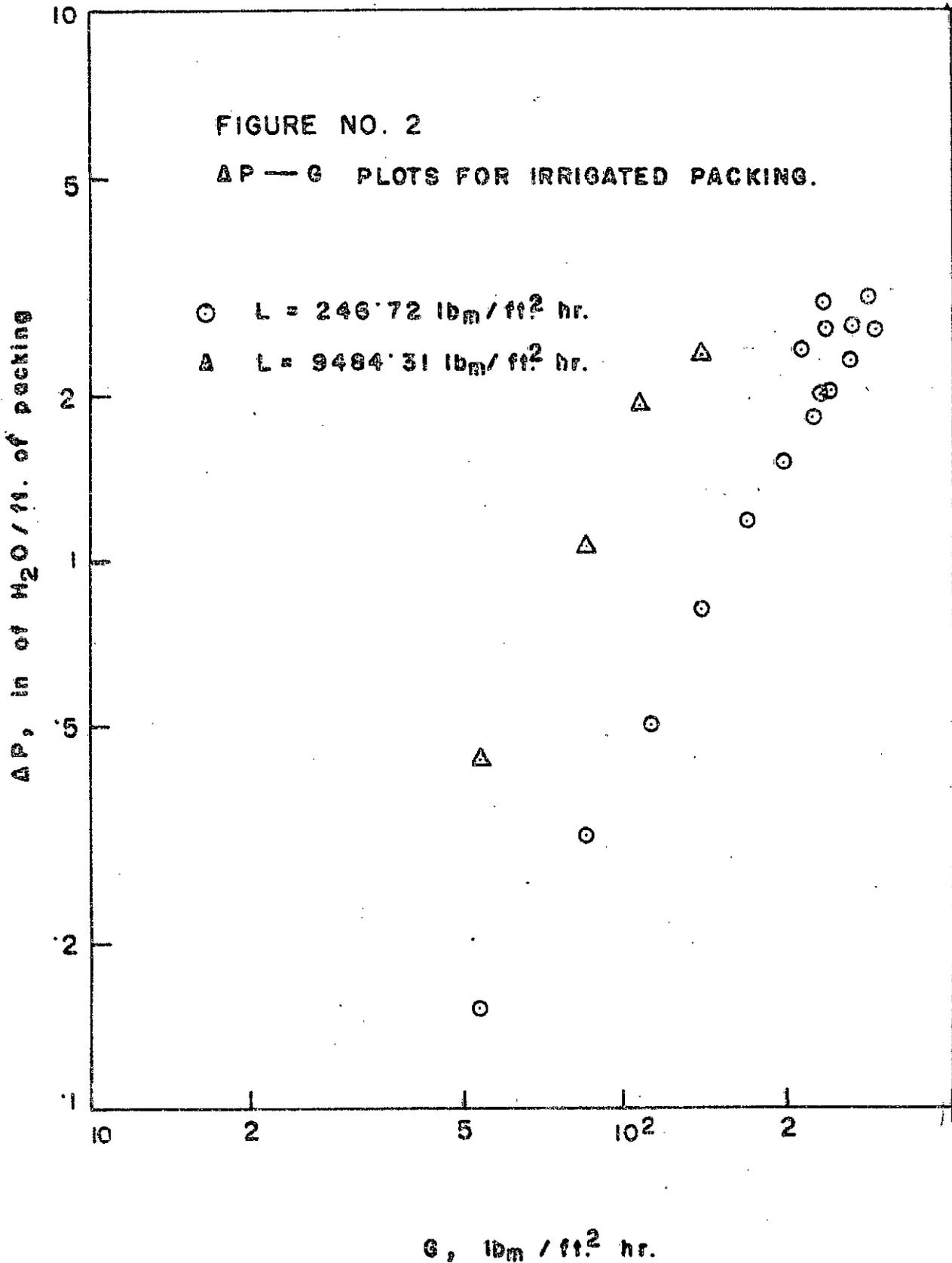


FIGURE NO. 3

$\Delta P - G$ PLOTS FOR IRRIGATED PACKING.

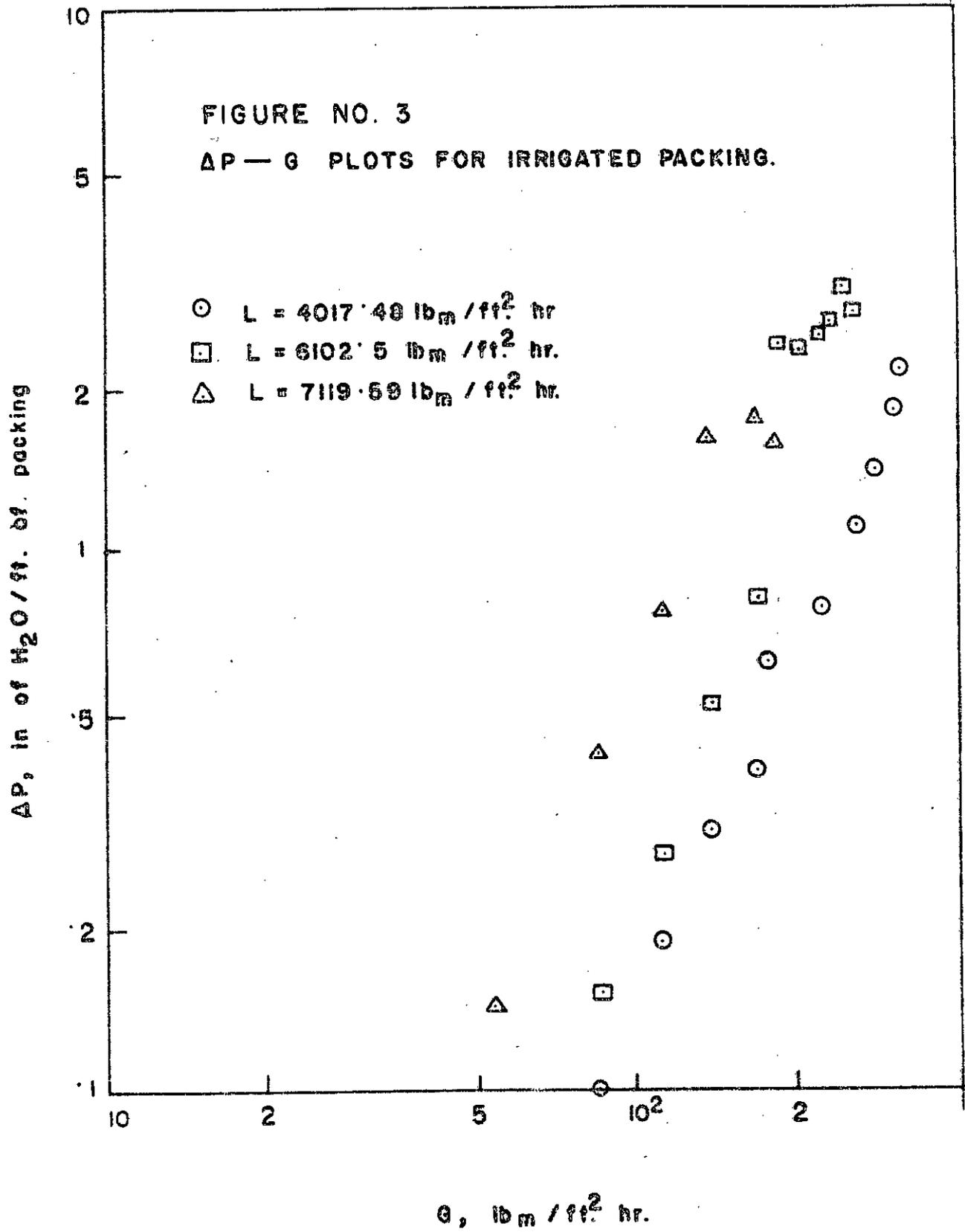


FIGURE NO. 4

ΔP - G PLOTS FOR IRRIGATED PACKING

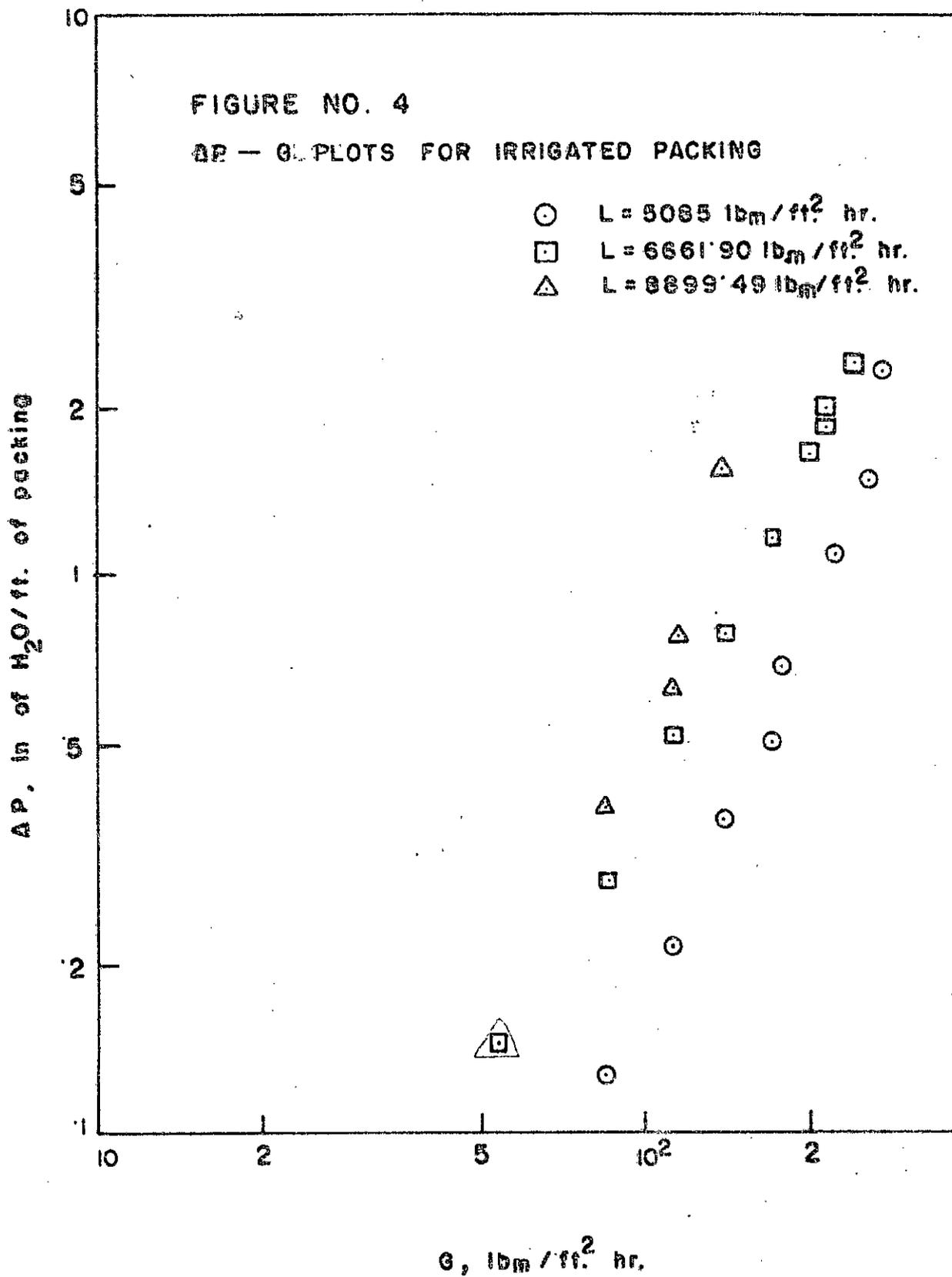
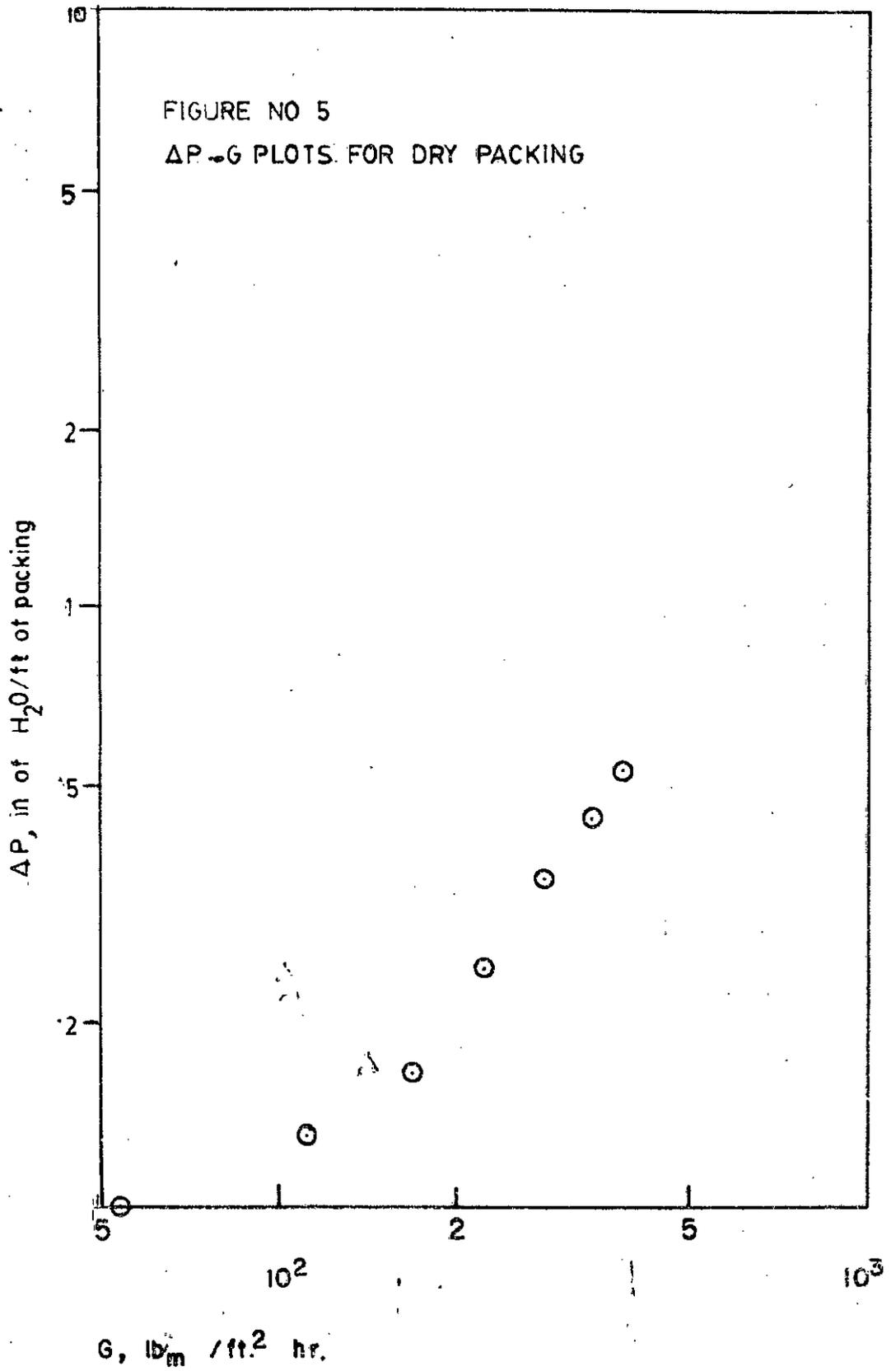
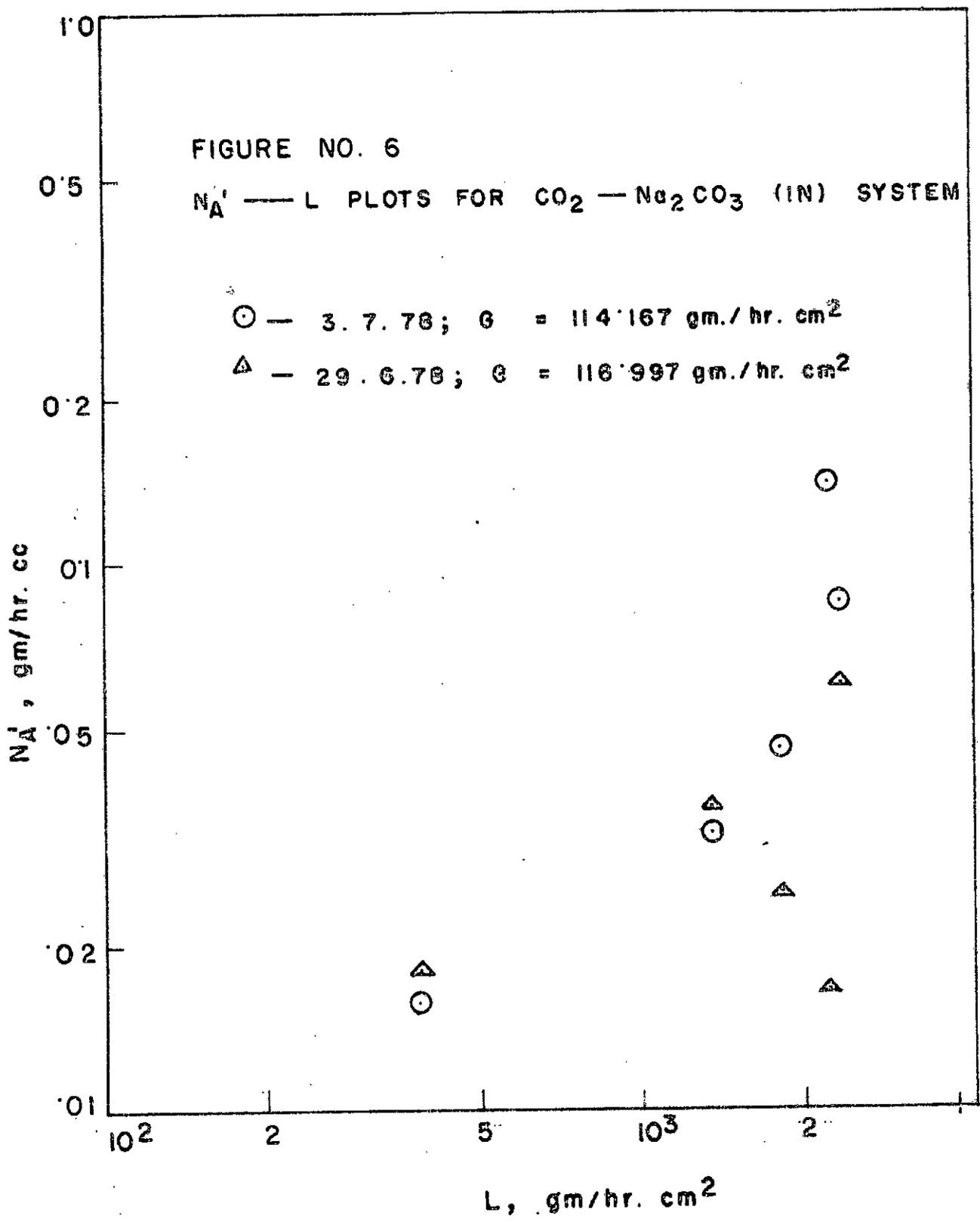


FIGURE NO 5

ΔP - G PLOTS FOR DRY PACKING





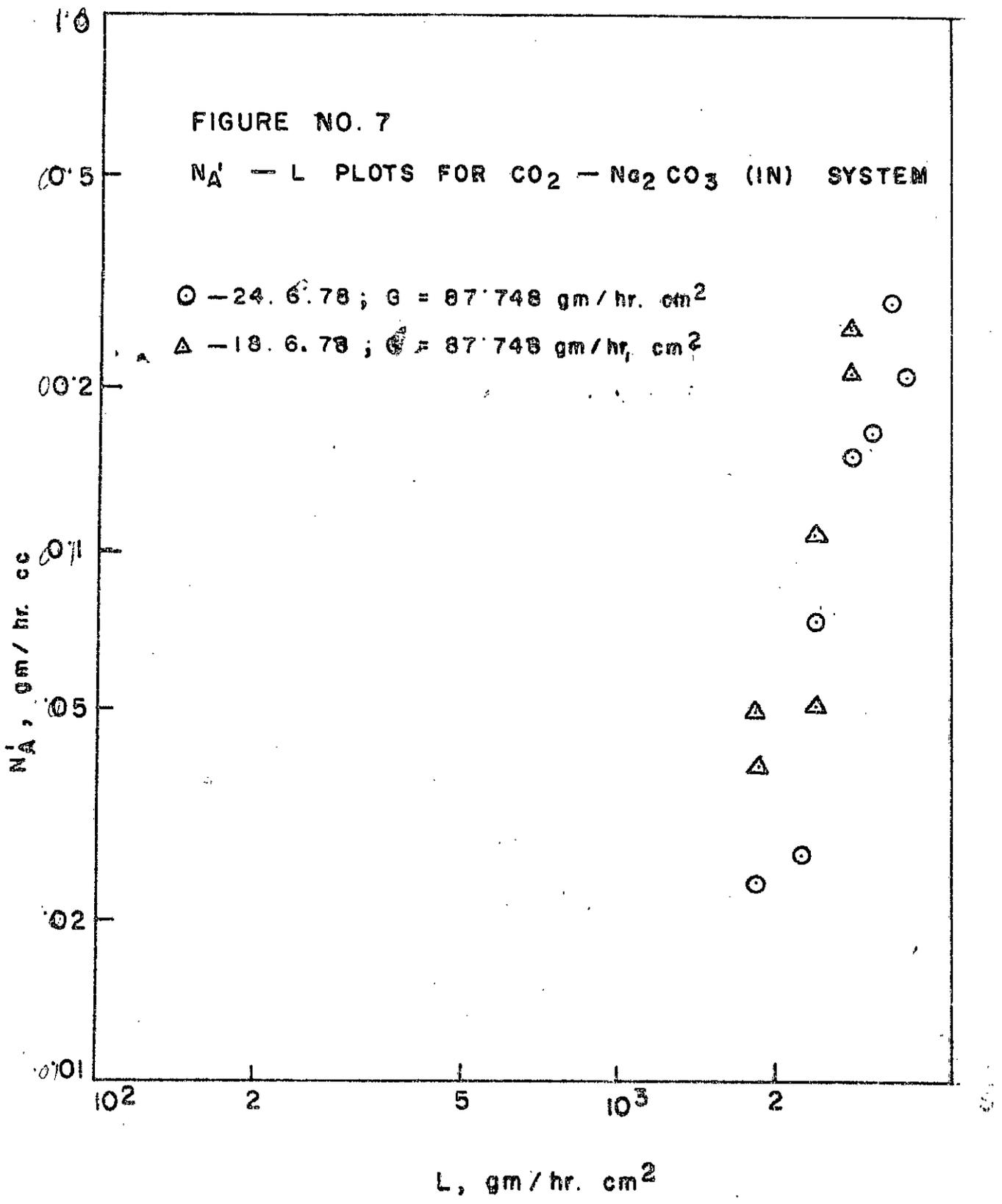


FIGURE NO. 8

K_{Ga} — L PLOTS FOR $CO_2 - Na_2CO_3$ (IN) SYSTEM

○ - 24. 6. 78; $G = 87.748 \text{ gm/hr. cm}^2$

△ - 18. 6. 78; $G = 87.748 \text{ gm/hr. cm}^2$

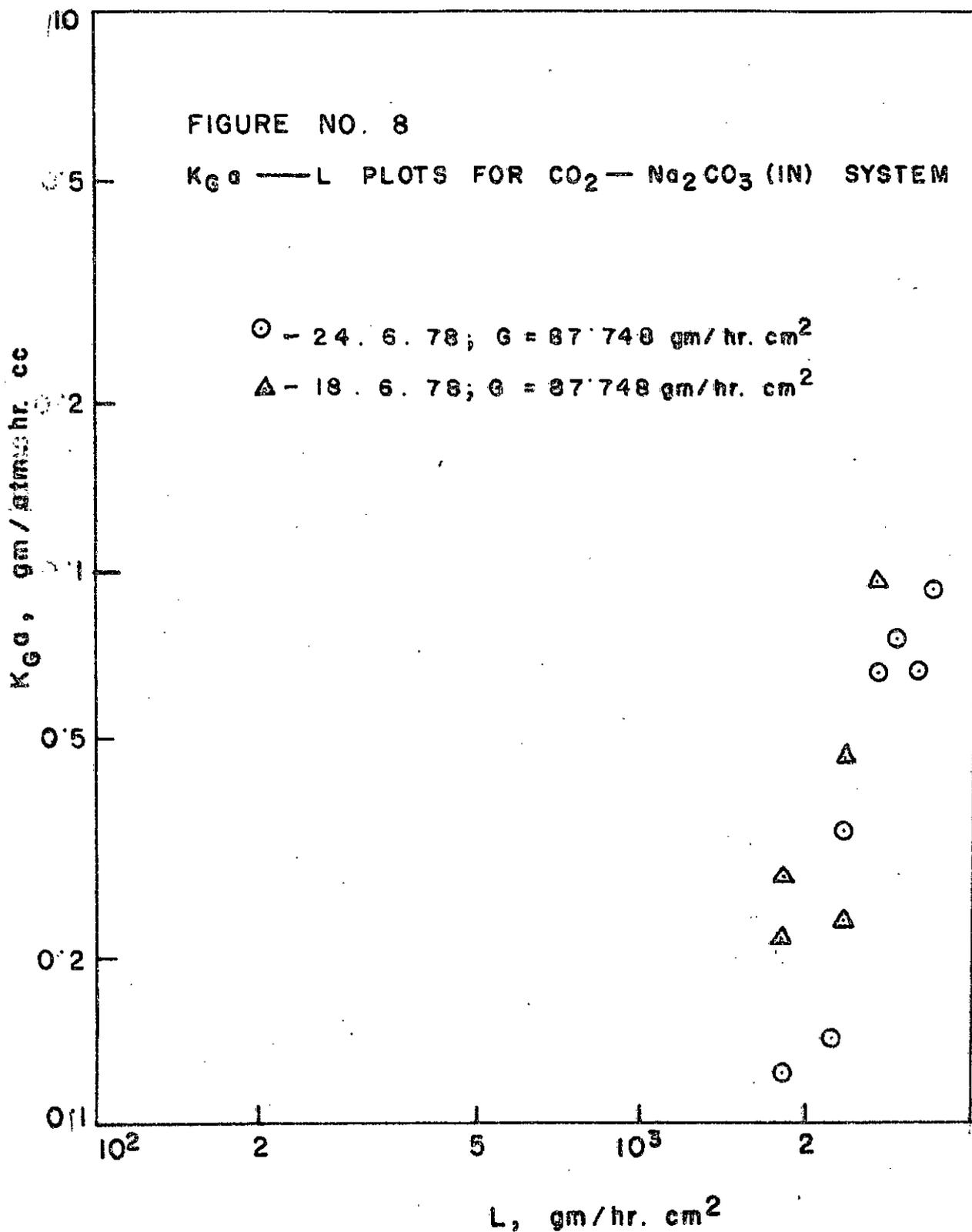


FIGURE NO. 9

$K_G a - L$ PLOTS FOR $\text{CO}_2 - \text{Na}_2\text{CO}_3$ (IN) SYSTEM

○ — 29.6.78; $G_i = 116.997 \text{ gm/hr. cm}^2$

△ — 3.7.78; $G_i = 114.167 \text{ gm/hr. cm}^2$

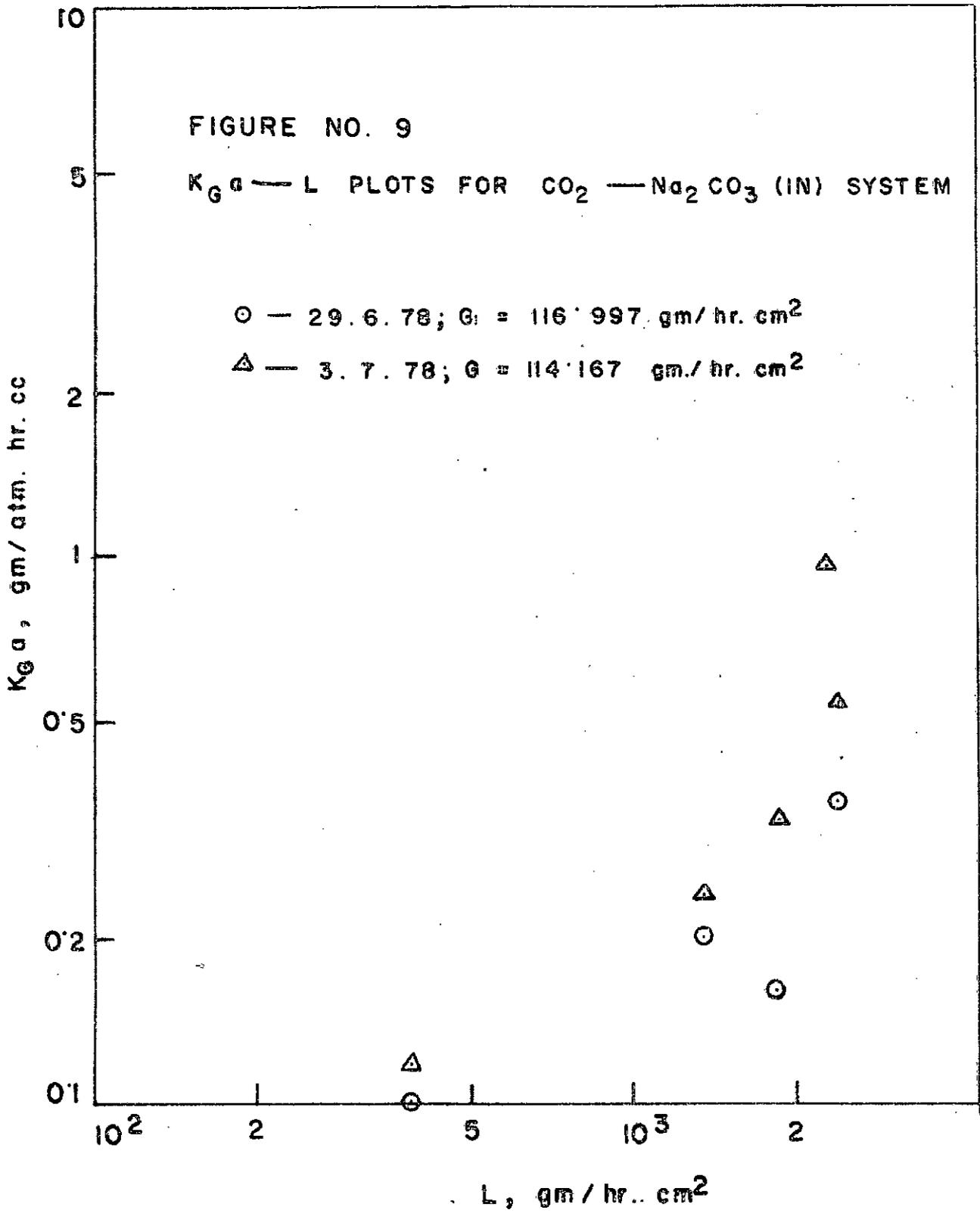


FIGURE NO. 10

K_G — L PLOTS FOR $\text{CO}_2 - \text{Na}_2\text{CO}_3$ (IN) SYSTEM

○ — 3.7.78 ; $G = 114.167 \text{ gm/hr. cm}^2$

△ — 29.6.78 ; $G = 116.997 \text{ gm/hr. cm}^2$

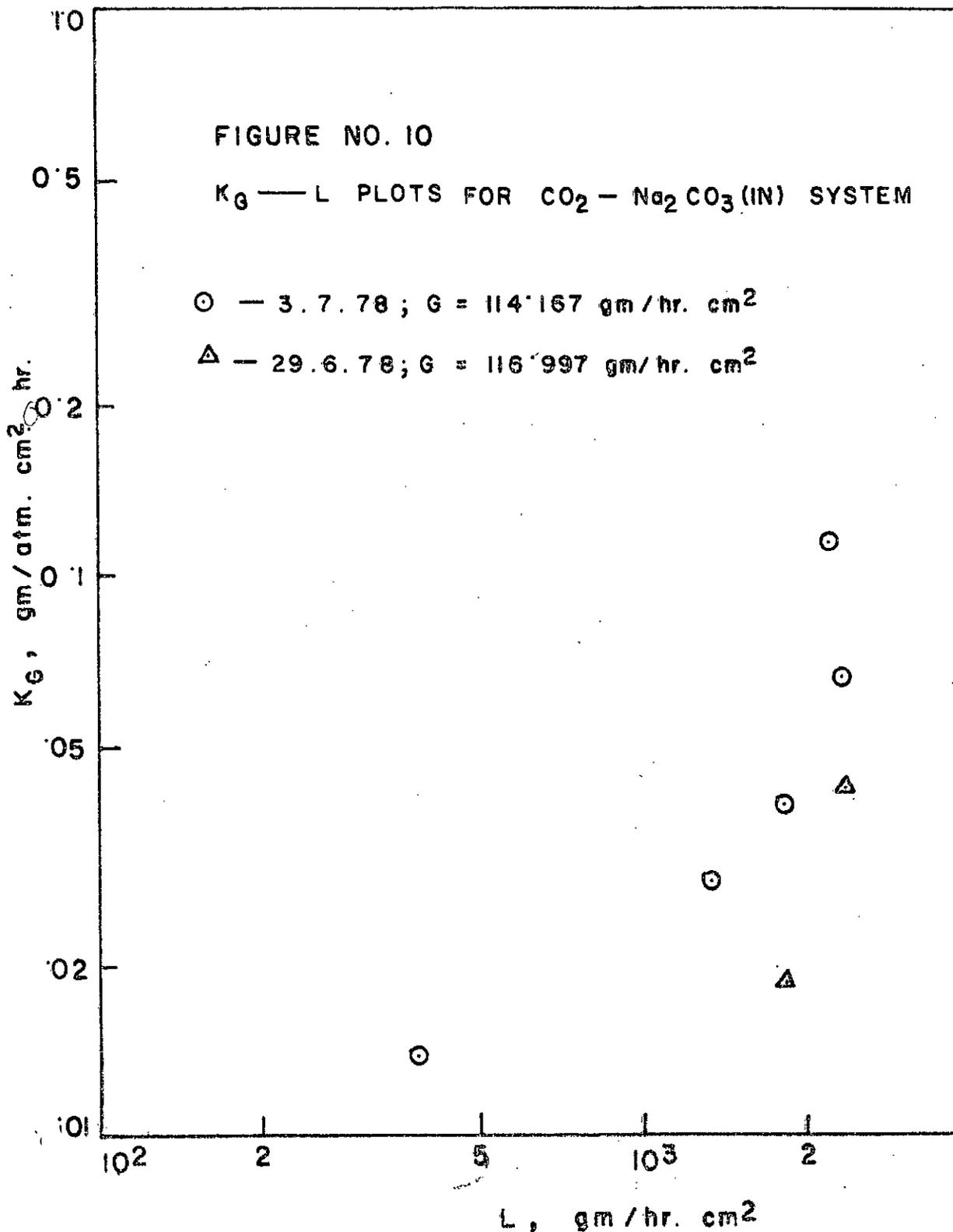
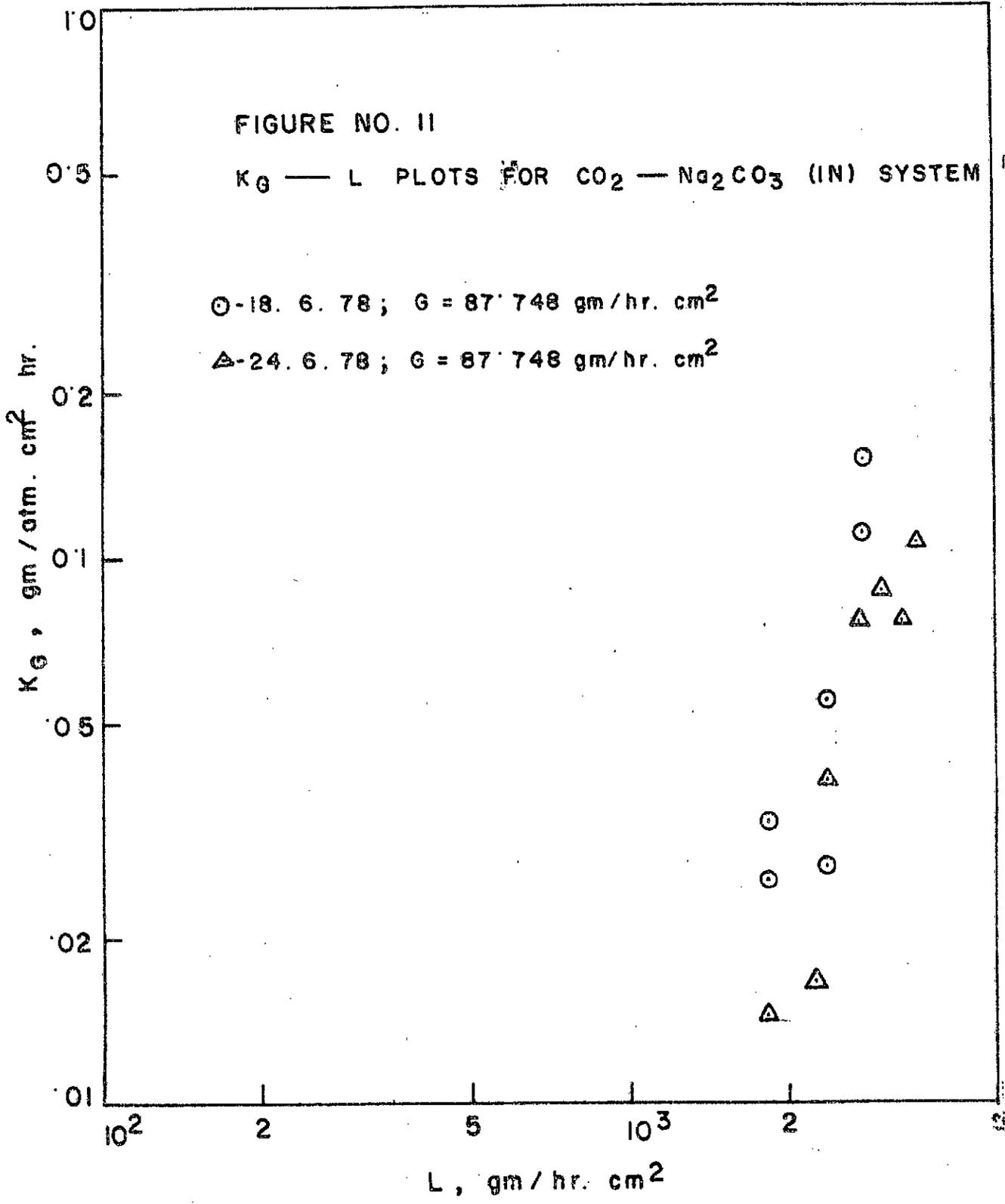


FIGURE NO. 11

K_G — L PLOTS FOR CO_2 — Na_2CO_3 (IN) SYSTEM

○ - 18. 6. 78 ; $G = 87.748 \text{ gm/hr. cm}^2$

△ - 24. 6. 78 ; $G = 87.748 \text{ gm/hr. cm}^2$



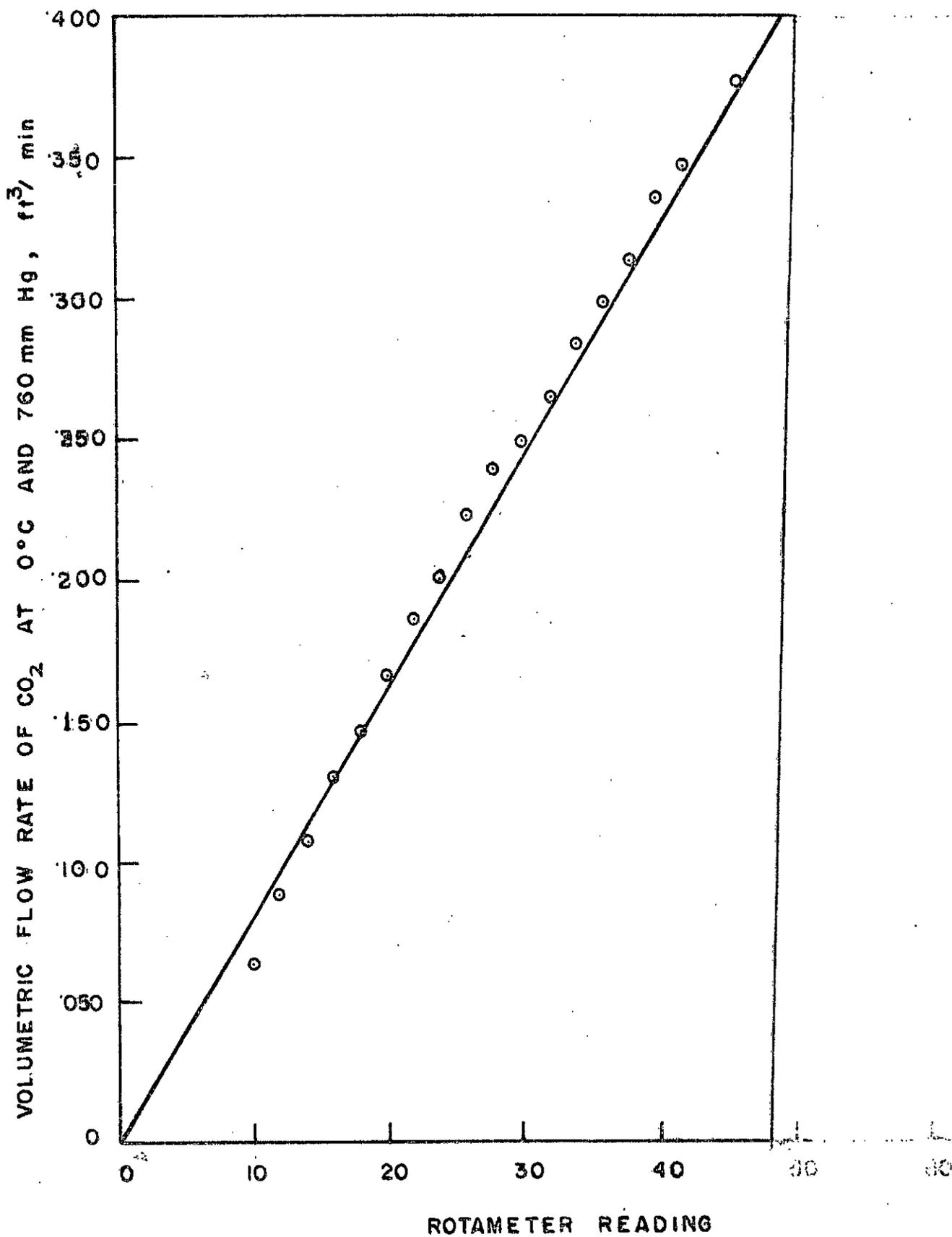
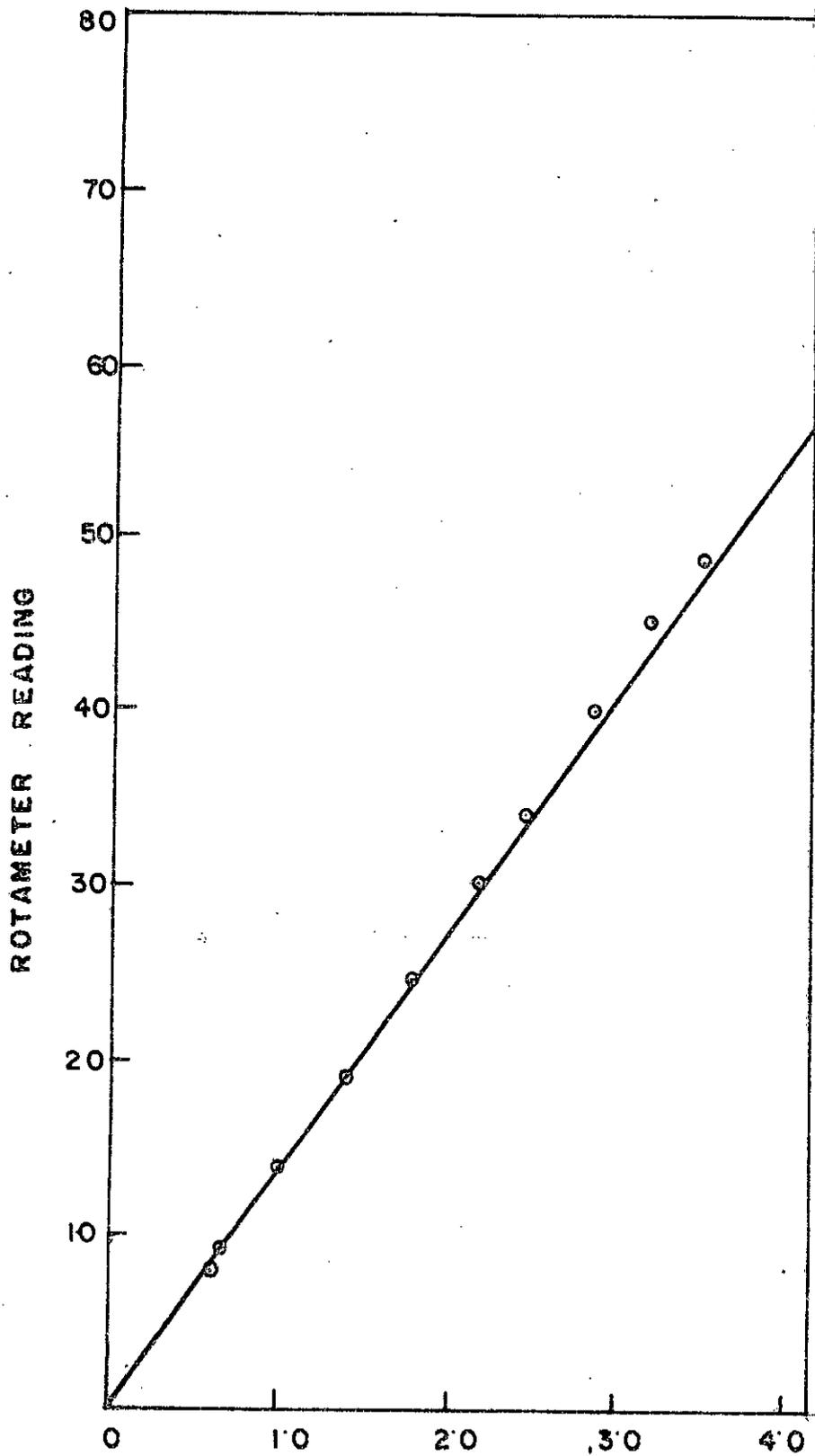


FIG. 4.1.1 CALIBRATION CURVE FOR CO₂ ROTAMETER.



VOLUMETRIC FLOW RATE OF AIR St. ft³/min.

FIG. 4.1.2 CALIBRATION CURVE FOR AIR ROTAMETER.

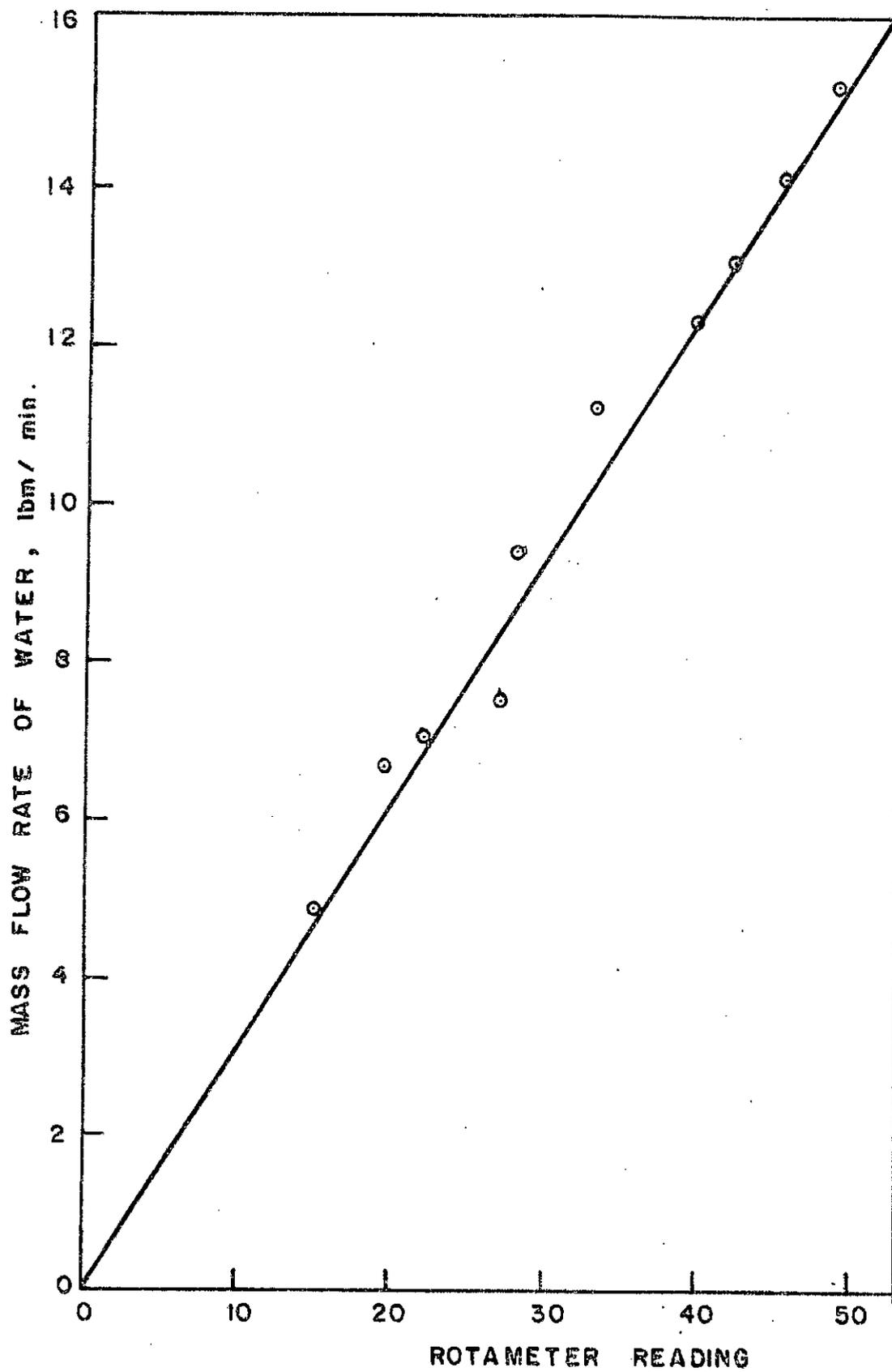


FIG. 4.1.3 CALIBRATION OF WATER ROTAMETER.

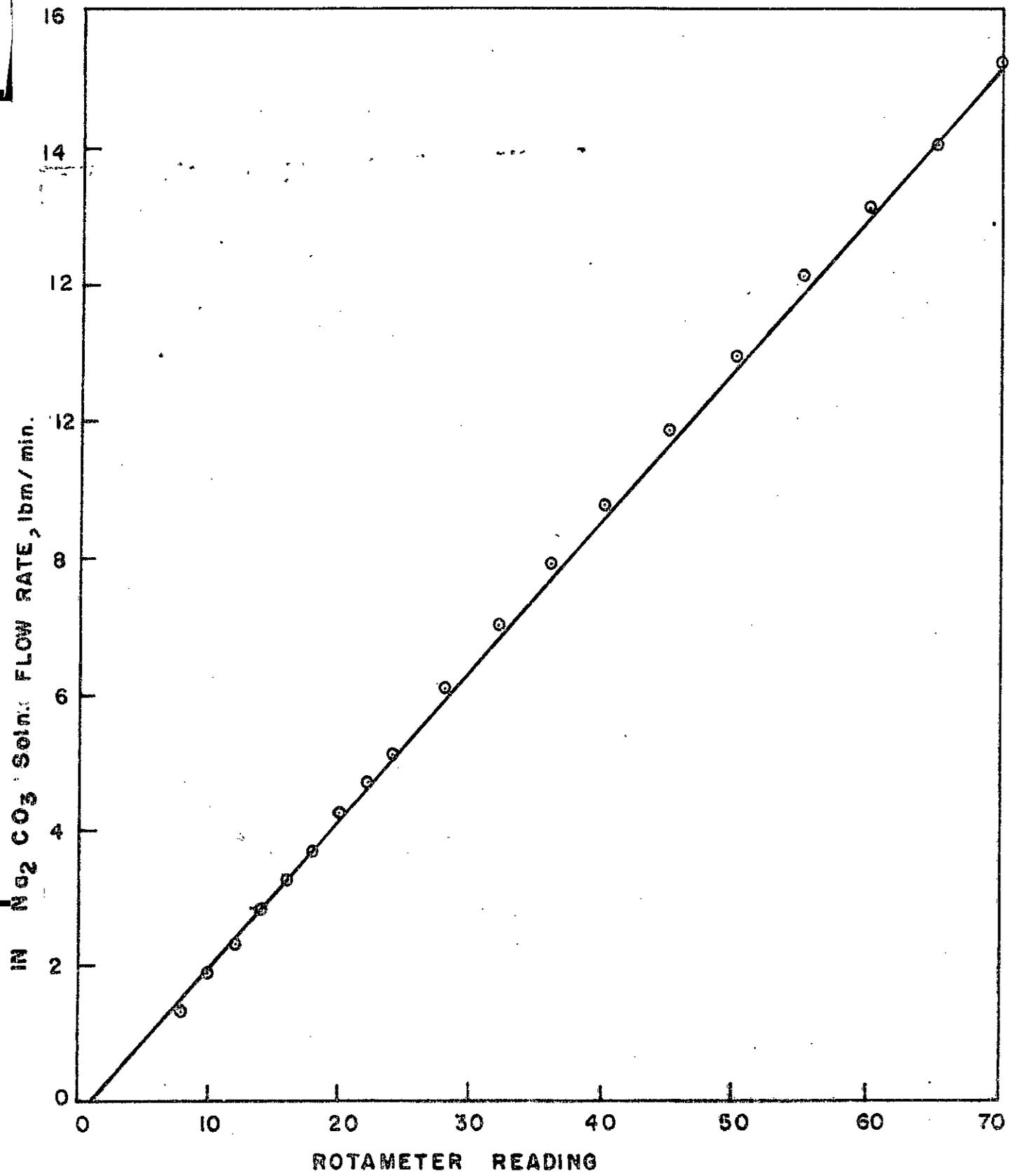


FIG. 4.1.4 CALIBRATION CURVE FOR Na_2CO_3 ROTAMETER.

TABLE - 1

No. of obs.	CO ₂ Flow rate (ft ³ /min)	Airflow rate (ft ³ /min)	Total gas flow rate ft ³ /min.	Na ₂ CO ₃ flow rate, (gm/hr.cm ²)	% of CO ₂ entering	CO ₂ absorbed N _A =(gm/hr.cc.)	K _{Ga} gm/atm. hr cc	K _G gm/atm.hr.cm ²
1A	.21	.72	.93	1823.27	19.678	.051157	.284	.0333
1B	.21	.72	.93	1823.27	19.763	.039376	.2181	.0256
2A	.21	.72	.93	2356.533	24.400	.05187	.2327	.0273
2B	.21	.72	.93	2356.533	23.673	.10707	.4699	.0551
3A	.21	.72	.93	2728.71	23.596	.21647	.9536	.1118
3B	.21	.72	.93	2728.71	22.270	.2636	1.2969	0.1506

Temp 28°C; pressure - 759 mmHg; 18.6.78

TABLE - 2

No. of obs.	CO ₂ flow rate (ft ³ /min)	Air flow rate (ft ³ /min)	Total gas flow rate (ft ³ /min)	Na ₂ CO ₃ flow rate (gm/hr.cm ²)	% of CO ₂ entering	CO ₂ absor- bed N _A ' (gm/hr.cc.)	K _{Ga} gm/atm.hr. cc.	K _G gm/ atm hr.cm ²
1	.2425	1.0	1.2425	1823.27	17.227	.02521	.1604	.0188
2	.2425	1.0	1.2425	2232.58	21.254	.01664	.0858	.0101
3	.2425	1.0	1.2425	2356.53	19.608	.06389	.3573	.0419
4	.2425	1.0	1.2425	307.60	19.781	.01823	.1010	.0118
5	.2425	1.0	1.2425	1343.68	19.596	.03644	.2038	.0239

Temp - 28°C; Pressure - 757 mHg; 29 & 30.6.78

TABLE 3

No. of obs.	CO ₂ flow rate (ft ³ /min)	Air flow rate (ft ³ /min)	Total gas flow rate (ft ³ /min)	Na ₂ CO ₃ flow rate (gm/hr.cm ²)	% of CO ₂ entering	CO ₂ absorbed N' _A (gm/hr.cc)	K _{Ga} gm/atm. cc.hr	K _G gm/atm. cm ² hr.
1	.21	1.0	1.21	387.60	14.7289	.0159	.1175	.0138
2	.21	1.0	1.21	1343.68	14.5959	.0321	.2408	.0282
3	.21	1.0	1.21	1823.27	15.3370	.04665	.3303	.0387
4	.21	1.0	1.21	2232.58	15.7300	.14028	.9774	.1146
5	.21	1.0	1.21	2356.53	17.056 *	.0863	.5546	.065

Temp-29°C; Pressure - 7595 mmHg; 3.7.78

TABLE - 4

No. of obs.	CO ₂ flow rate (ft ³ /min)	Air flow rate (ft ³ /min)	Total Gas flow rate (ft ³ /min)	Na ₂ CO ₃ flow rate (gm/hr cm ²)	% of CO ₂ entering	CO ₂ absor- bed N _A (gm/hr.cc)	K _G a(gm/ atm.cc. hr)	K _G gm/ atm.hr. cm ²
1	.21	.72	.93	1823.27	20.92	.02355	.1235	.0145
2	.21	.72	.93	2232.583	20.80	.02700	.1424	.0167
3	.21	.72	.93	2356.535	23.577	.07321	.3404	.0399
4	.21	.72	.93	2728.71	25.16	.15044	.6556	.0769
5	.21	.72	.93	2976.777	24.395	.16684	.7508	.088
6	.21	.65	.86	3249.5609	48.2966	.291276	.6619	.0776
7	.21	.72	.93	3472.688	24.3600	.21111	.9123	.107

Temp-28°C; Pressure 757 mHg; 24.6.78

TABLE - 5

No. of obs.	Weight of packing material (gm)	Height of packing (in)	Volume of water added cc	Volume occupied by packing (in ³)	Voidage of packing (in ³)	Porosity	Average porosity
1	266	5.45	158.6	14.36248	9.678365	0.40258	
2	226	9.95	145.8	14.137166	8.89726	0.386259	
3	226	14.35	142.6	13.82300	8.701985	0.38632	0.387298
4	226	18.95	141.5	14.451325	8.6485	0.374027	

N.B. Water and packings were added by increments

TABLE - 6

No. of obs.	Weight of raschig rings in air(mg)	Weight of raschig rings in water (mg)	Diff	Density (gm/cc)	Average density
1	737.7	452	285.7	2.582077	
2	673.5	420	253.5	2.6568047	2.61944

TABLE - 7

PRESSURE DROP - FLOW RATE DATA (DRY PACKING)

No. of obs.	Manometer reading difference in height (in)	Airrotameter reading	Air Mass flux lbm/ft ² .hr	ΔP , in of H ₂ O/ft of packing
1	.15	10	54.11	0.094
2	.21	20	112.08	0.132
3	.27	30	170.06	0.169
4	.4	40	224.17	0.2511
5	.57	50	282.14	0.358
6	.72	60	343.98	0.452
7	.86	67	384.56	0.54

TABLE - 8

PRESSURE DROP - FLOW RATE DATA (AIR-WATER SYSTEM)

No. of obs.	Manometer reading (difference) (in)	Air rotameter reading	Water rotameter reading	Air mass flux, lbm/ft ² hr.	Water mass flux, lbm/ft ² hr	ΔP , in of H ₂ O/ft of packing
1	.05	10	8	54.11	4017.48	.0314
2	.16	15	8	85.03	4017.48	.1004
3	.3	20	8	112.08	4017.48	.188
4	.48	25	8	139.14	4017.48	.3013
5	.63	30	8	170.06	4017.48	.3955
6	1.0	35	8	177.79	4017.48	.6277
7	1.25	40	8	224.17	4017.48	.7847
8	1.78	46.5	8	262.0	4017.48	1.1174
9	2.23	50	8	282.81	4017.48	1.4
10	2.9	54	8	307.26	4017.48	1.8205
11	3.43	56	8	317.89	4017.48	2.1532

TABLE - 9

PRESSURE DROP - FLOW RATE DATA

No. of obs.	Manometer reading (difference) (in)	Air rotameter reading	Water rotameter reading	Air mass flux, lbm/ft ² hr.	Water mass flux, lbm/ft ² hr	ΔP , in of H ₂ O/ft of packing
1	.23	10	14	54.11	7119.59	.1444
2	.68	15	14	85.03	7119.59	.4268
3	1.25	20	14	112.08	7119.59	.7847
4	2.6	25	14	139.14	7119.59	1.6321
5	2.8	30	14	170.06	7119.59	1.7577
6	2.5	33	14	185.52	7119.59	1.5693

TABLE - 10

PRESSURE DROP - FLOW RATE DATA

No. of obs.	Manometer reading (difference) (in)	Air rotameter reading	Water rotameter reading	Air mass flux, lbm/ft ² hr.	Water mass flux, lbm/ft ² hr	ΔP , in. of H ₂ O/ft of packing
1	.03	10	10	54.11	5085.42	.0188
2	.2	15	10	85.03	5085.42	.12555
3	.34	20	10	112.08	5085.42	.2134
4	.58	25	10	139.14	5085.42	.3641
5	.8	30	10	170.06	5085.42	.5022
6	1.1	35	10	177.79	5085.42	.6905
7	1.72	40	10	224.79	5085.42	1.08
8	2.35	45	10	255.04	5085.42	1.475
9	3.7	48	10	270.54	5085.42	2.323

TABLE - 11

PRESSURE DROP - FLOW RATE DATA

No. of obs.	Manometer reading (difference) (in)	Air rotameter reading	Water rotameter reading	Air mass flux, lbm/ft ² hr.	Water mass flux, lbm/ft ² hr	ΔP, in. of H ₂ O/ft of packing
1	.23	10	.13	54.11	6661.90	.1444
2	.21	15	.13	85.03	6661.90	.2825
3	.78	20	.13	112.08	6661.90	.521
4	1.2	25	.13	139.14	6661.90	.7847
5	1.8	30	.13	170.06	6661.90	1.1613
6	2.6	35	.13	197.11	6661.90	1.6635
7	2.9	38	.13	212.57	6661.90	1.8518
8	3.2	38	.13	212.57	6661.90	1.8518
9	3.8	43	.13	239.63	6661.90	2.3854

TABLE - 12

PRESSURE DROP - FLOW RATE DATA

No. of obs.	Manometer reading (difference) (in)	Air rotameter reading	Water rotameter reading	Air mass flux, lbm/ft ² hr.	Water mass flux, lbm/ft ² hr	ΔP , in of H ₂ O/ft of packing
1	.23	10	17	54.11	8899.49	.1444
2	.61	15	17	85.03	8899.49	.3829
3	1.0	20	17	112.08	8899.49	.6277
4	1.4	21	17	115.95	8899.49	.8788
5	2.5	25	17	139.14	8899.49	1.5694

TABLE - 13

PRESSURE DROP - FLOW RATE DATA

No. of obs.	Manometer reading (difference) (in)	Air rotameter reading	Water rotameter reading	Air mass flux, lbm/ft ² hr.	Water mass flux, lbm/ft ² hr	ΔP , in of H ₂ O/ft of packing
1	.09	10	12	54.11	6102.5	.0565
2	.24	15	12	85.03	6102.5	.1507
3	.44	20	12	112.08	6102.5	.2762
4	.83	25	12	139.14	6102.5	.521
5	1.31	30	12	170.06	6102.5	.8223
6	3.84	34	12	189.38	6102.5	2.4105
7	3.64	35	12	255.08	6102.5	2.285
8	3.74	36	12	200.96	6102.5	2.348
9	4.04	40	12	224.17	6102.5	2.536
10	4.24	42	12	235.76	6102.5	2.6616
11	4.84	44	12	247.35	6102.5	3.0383

Analysis of Na_2CO_3 Soln. at inlet

Reference	Buret reading		Diff. (ml)	Avg.diff. (ml)	Vol.of sample (ml)	Strength of acid	Normation of Na_2CO_3 Soln. (N)
	Initial (ml)	final (ml)					
Table - <u>I</u>	0.1	16.3	16.2		10	0.577049	
	17.0	33.3	16.5	16.33	10	"	0.942321
	33.3	49.8	16.3		10	"	
Table - <u>II</u>	3.0	41.0	38.0		20	"	
	0.0	38.0	38.0	38.03	20	"	1.0972586
	0.0	38.1	38.1		20	"	
Table - <u>III</u>	0.0	38.85	38.85		20	0.5700763	1.106897
	0.0	38.80	38.80	38.833	20	"	
	0.0	38.83	38.83		20	"	
Table - <u>IV</u>	13.0	51.0	38.0		20	0.577049	
	2.0	40.05	38.05	38.116	20	"	1.0997393
	0.0	38.30	38.3		20	"	

TABLE - 14

Total volume of packing = 65.61244 in³
= 1075.1951 cc.

Total Height (Effective) of packing = 19.12 in

Total weight of packing introduced into the column = 904 gms.

Average weight of each packing = 239.6964 mg

Number of Rasching Rings when counted in 226 gms = 943 piece

Average density of ring material = 2.619441 gm/cc

Average number of rings = $\frac{943 + 937}{2} = 940$

Total number of rasching rings = $\frac{940 \text{ gm} \times 940}{226 \text{ gm}}$
= 3760 piece

Total surface area/rasching ring = 0.3408634 in²
~~8268.67 cm²~~

Surface area of the packed section
of the wall = 901,80561 cm²

Total surface area of packing + wall = 9170.4756 cm²

Value of "a" = $\frac{9170.4756}{1075.1951} = 8.529127 \text{ cm}^2/\text{cm}^3$

SAMPLE CALCULATION

Reading No. 1 of 18.6.78

Sample collected at the outlet after 25 minutes.

i. Total alkalinity of outlet liquid = 33.066 ml of
.577049NHCl

ii 25 ml of .1005083N NaOH is equivalent to

$$N_{\text{NaOH}} \times V_{\text{NaOH}} = N_{\text{HCl}} \times V_{\text{HCl}}$$

$$.1005083 \text{N} \times 25 \text{ ml} = .577049 \text{ N} \times V_{\text{HCl}}$$

$$V_{\text{HCl}} = 4.3544 \text{ ml} = .577049 \text{ N HCl}$$

iii. Volume of HCl required to neutralize excess:

$$\begin{aligned} \text{NaOH} &= N_{\text{HCl}} \times V_{\text{HCl}} = V_{\text{HCl}} \times N_{\text{HCl}} \\ &= .1137146\text{N} \times 16.6 \text{ ml} = .577049\text{N} \times V_{\text{HCl}} \end{aligned}$$

$$V_{\text{HCl}} = 3.2712339 \text{ ml of } .577049\text{N HCl}$$

iv. Conversion to bicarbonate = $4.3544 - 3.2712339$
= 1.0831661 ml of
 $.577049 \text{ N HCl}$

v. Na_2CO_3 rotameter reading 8

vii. mg equivalent of NaHCO_3 = mg eq. of HCl
= $1.0831 \times .57704$

viii. 20 cc. of the outlet solution contains
= $1.0831 \times .577043 \text{ mg.}$
equivalent of CO_2

1000 cc of the outlet solution contains

$$\begin{aligned} &= \frac{1.0831 \times .577049 \times 1000}{20} \text{ mg.eq.} \\ &= \frac{1.0831 \times .57049 \times 1000}{20} \times \frac{1 \text{ gm}}{1000 \text{ mg}} \text{ mg.eq.} \\ &= \frac{1.0831 \times .57049 \text{ gm-eq.}}{20} \times \frac{44 \text{ gms}}{1 \text{ gm-eq.}} \end{aligned}$$

Hence the amount of CO_2 absorbed per cc of the outlet solution

$$\begin{aligned} &= \frac{1.0831 \times .57049 \times 44}{20 \times 1000} \left(\frac{\text{gm}}{\text{cc}} \right) \times 666.79 \text{ cc/min} \\ &= 0.916832 \text{ gm/min.} \\ &= 0.051157 \text{ gm/hr cc.} \end{aligned}$$

ix. Total volumetric flow rate of gas mixture
= 26,334.66 cc/min.

x. Total alkalinity = 33.06 ml of .577049N HCl
25 ml of .1005083N NaOH is equivalent to,

$$N_{\text{NaOH}} \times V_{\text{NaOH}} = N_{\text{HCl}} \times V_{\text{HCl}}$$

$$.1005083 \times 25 = .577049 \times V_{\text{HCl}}$$

$$V_{\text{HCl}} = 4.3344 \text{ ml of } .577049 \text{ HCl}$$

xi. Volume of HCl to neutralize excess NaOH

$$.1137146 \times 16.6 = .577049 \times V_{\text{HCl}}$$

$$V_{\text{HCl}} (.577049) = 3.2712339 \text{ ml of } .577049 \text{N HCl}$$

xii. Conversion to bicarbonate \pm 4.3544 - 3.271 339
= 1.0831661 ml of .57049N HCl

xiii. Bicarbonate fraction = $\frac{\text{NaHCO}_3}{\text{NaHCO}_3 + \text{Na}_2\text{CO}_3}$
= $\frac{.1.0831661}{1.0831661 + 31.9828339}$
 \pm .0327576

xiv. Total alkalinity of outlet liquid

$$30.066 \times .577049 = 20 \text{ N}$$

$$\text{N} = .9540351 \text{N}$$

iv. $P_w, \text{CO}_2 = 137 f^2 N^{1.29} / S(1-f)(365-t)$
= $137 (.0327576)^2 (.9540351)^{1.29} /$
 $.03138 (1 - .0327576) (282.6)$
= .0161311 mm Hg
= .0000212 atm.

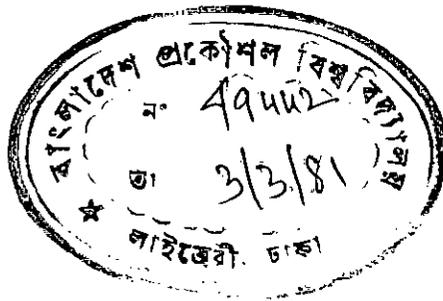
xvi. Vapor pressure of H_2O at 28°C : 28.349 mm Hg

xvii. % of CO_2 entering the tower = 19.67871

$$\text{xviii. } P_{\text{CO}_2} = \frac{.1967871 (758 - 28.349)}{760} \text{ atm.}$$
$$=.1889288 \text{ atm.}$$

$$\text{xix. } K_{\text{Ga}} = \frac{.9168362 \times 60}{(.1889288 - .0000212) \times 1075.15}$$
$$= .284 \text{ gm/hr. atm. cc.}$$

$$\text{xxvi. } K_{\text{G}} = \frac{0.84}{9.529} = 0.0332 \text{ gm/hr. atm. cm}^2$$



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