

# Gas Absorption Column

## INTRODUCTION

The packed tower, in which two fluids flowing in opposite directions enable a chemical component to be transferred from one fluids phase to the other, occurs in almost all chemical plants. The process may be gas absorption, distillation, solvent extraction or chemical reaction. Knowledge of the characteristics of both fluid flow and of mass transfer in such towers is necessary for both plant operators and designers.

The modified Armfield Gas Absorption Apparatus has been designed to allow for these studies to be made and the instrumentation and layout enables students to follow both the hydrodynamic characteristics in the absence of mass transfer, and also, separately, to advise the performance of the mass transfer process involved in gas absorption. The size of the equipment has been chosen so that experiments may be completed in a typical laboratory class period, while at the same time being capable of demonstrating full scale plant behavior. Considerable attention is directed towards matters of safety which is of crucial importance in the process industries.

This instruction manual gives details of all the laboratory services required for operating the column, together with instructions for the designed experiments.

## EQUIPMENT DIAGRAMS

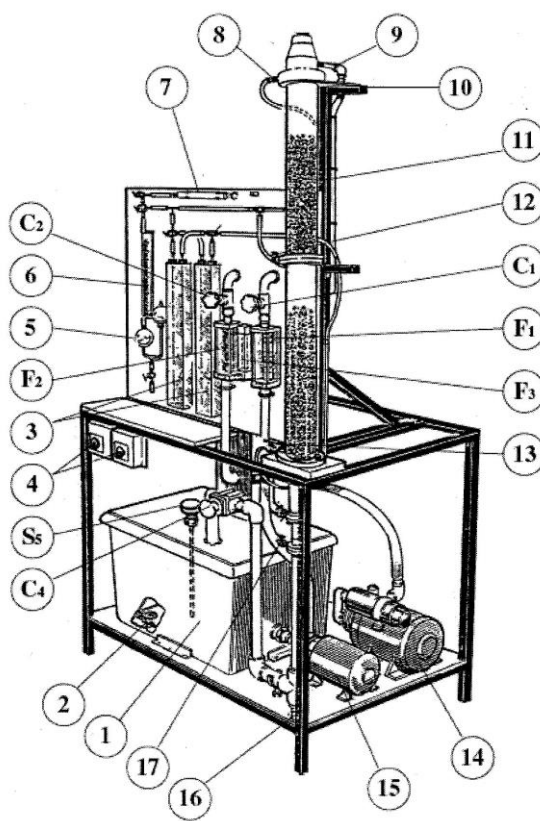


Figure 1: UOP 7 Gas Absorption Column

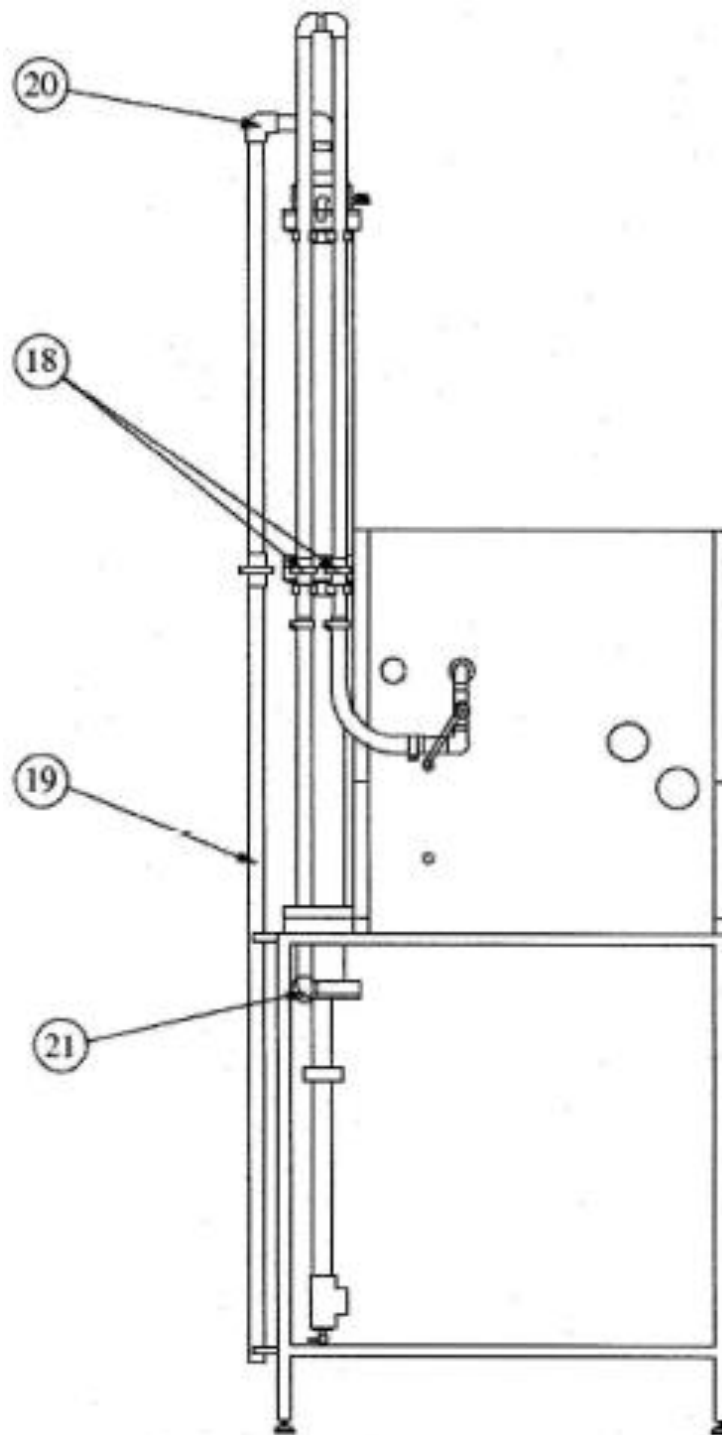


Figure 2: Diagram showing details of the overflow arrangement and air pipework.

## OVERVIEW

Where necessary, refer to the drawings in the Equipment Diagrams section above.

The packed absorption column is made of two 80 mm inside diameter clear acrylic sections joined end to end to give a total column length of 1.4 m and is installed vertically on a mild steel floor standing framework. The column is filled with 10 mm x 10 mm glass Raschig rings (11) which is representative of the type of packing used in gas absorption.

The liquid for the process is normally water and is stored in a 50 liter rectangular sump tank (1). A valve (2) on the underside of the tank allows it to be drained for cleaning. A float valve on the side of the tank supplies fresh water as required. A centrifugal pump (15) is used to deliver the liquid to the top of the column (9) where it falls by gravity through the packing before returning to the sump tank via a 'U' trap that forms a liquid seal. A control valve  $C_4$  downstream of the 'U' trap allows the head of water in the column to be varied. This valve is usually fully open when the column is in use but can be partially closed to restrict the flow of water returning back to the sump tank. Water can be drained from the 'U' trap after use by opening the drain cock (16) at the bottom. A variable area flowmeter  $F_1$  gives a direct reading of the liquid flow rate entering the top of the column. The flow of liquid can be varied using the control valve  $C_1$  above the liquid flowmeter.

The gas to be absorbed is normally carbon dioxide and is taken from a cylinder of compressed gas, located in a suitable storage rack adjacent to the column. The gas is connected to a low pressure regulator that is located at the rear of the vertical backboard. The gas passes through a calibrated variable area flow meter  $F_3$  and is mixed with an air stream, also via a calibrated variable area flowmeter  $F_2$  from a rotary compressor (14). The flow of air can be varied using the control valve  $C_2$  above the air flowmeter. The ratio of gas to air in the mixture entering the column is therefore known and easily varied. Entering the base of the column, the gas mixture rises up through the packed bed of Raschig rings (11) and is counter-currently contacted with the liquid flowing down the column. Gas is prevented from escaping at the base of the column by the liquid in the 'U' trap.

Tappings are provided at the base (position 13 with isolating valve), centre (position 12 with isolating valve) and top (8) of the column to allow indication of pressure drop in the two sections of column using a pair of 'U' tube manometers (3). These tappings also provide a means of extracting samples of gas from the column for analysis using the sampling syringe (7). The carbon dioxide content of the gas samples is determined using a Hempel apparatus (5) and readings are indicated on the scale (6). A sampling point (17) below the column allows analysis of the liquid outlet stream. A manual sump pump,  $S_5$ , can be used to withdraw a sample of liquid from the sump tank

for analysis. The flow meters, manometers and gas analysis equipment are all mounted on a vertical backboard at a convenient height for operation.

The effluent gas leaves the top of the column via a cap with an overflow arrangement (19 & 20). This arrangement ensures that any liquid is safely returned to the sump tank if the column becomes flooded. When using a solution of caustic soda (sodium hydroxide) in the column it is suggested that a suitable container is located below the overflow to intercept any liquid that spills from the top of the column. For safe operation the flooding characteristics of the column should be evaluated using clean tap water. Caustic soda should not be used for this series of tests.

## INDEX TO EXERCISES

**Exercise A** - To determine the air pressure differential across the dry column as a function of the air flow rate.

**Exercise B** - To examine the air pressure differential across the column as a function of air flow rate for different water flow rates down the column.

**Exercise C** - To measure the absorption of carbon dioxide into water flowing down the packed column, using the gas analysis equipment provided.

**Exercise D** - To calculate rate of absorption of carbon dioxide into water from analysis of liquid solutions flowing down the packed column.

**Exercise E** - To calculate rate of absorption of carbon dioxide into caustic soda solutions from analysis of liquid solutions flowing down the packed column.

**Exercise F** - To show that the amount of carbon dioxide removed from the air stream equals the amount taken up by a liquid stream of caustic soda solution.

**Exercise G** - Determination of Overall Mass Transfer Coefficient ( $K_{og}$ ).

## NOMENCLATURE

a = Effective interfacial area per unit packed column ( $\text{cm}^{-1}$ )

A = Cross sectional area of the tower ( $\text{m}^2$ )

F = Flow (volume/time, (liters/sec or liters/min))

G	=	Gas flow rate (moles/second)
$K_{og}$	=	Gas-side mass transfer coefficient (moles/second*cm <sup>2</sup> *atm)
L	=	Liquid flow (liters/second)
M	=	molecular weight (g/mol)
P	=	Pressure (atm)
R	=	Mass transfer rate (moles/cm <sup>2</sup> *second)
X	=	Mole fraction of component in liquid phase
Y	=	Mole fraction of component in gas phase

#### Subscripts

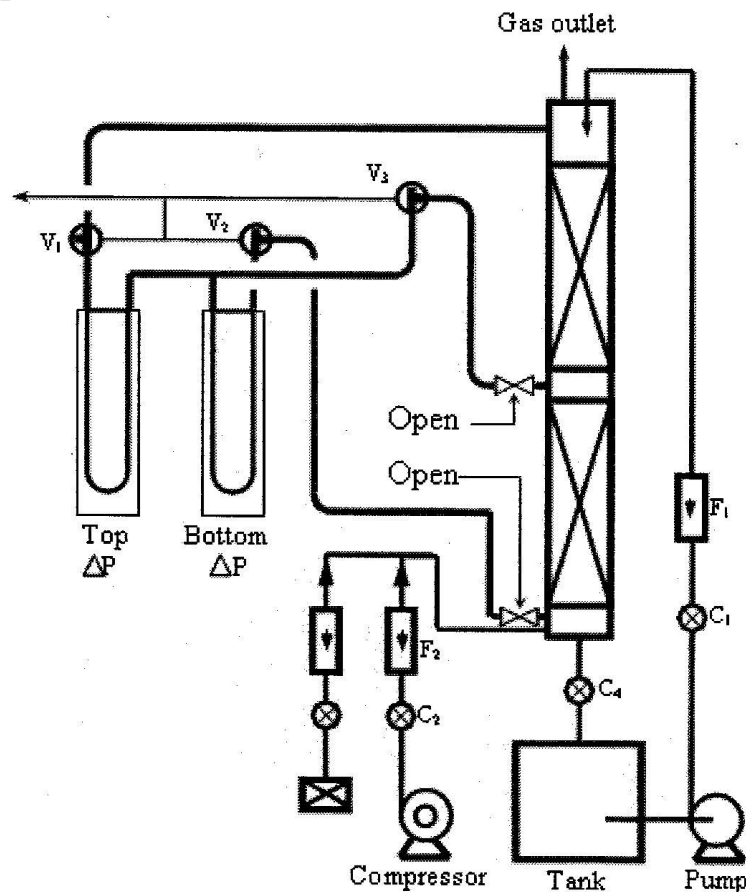
T	=	Total
i	=	Inlet conditions to column
o	=	Outlet conditions to column
N	=	Rate of absorption (g*moles/second)

## EXERCISE A

### Objective

To determine the air pressure differential across the dry column as a function of the air flow rate.

### Equipment Set up:



No additional equipment is required for this experiment.

### Procedure

**Note:** It is important to carry out this exercise at the beginning of the lab since it requires a dry column.

1. Do not use the water pump in this experiment. Do not use the CO<sub>2</sub> tank in this experiment. Only the air compressor is needed.
2. The column must be dried by passing the maximum air flow until all evidence of moisture in the packing has disappeared.

3. Set the valves  $V_1$ ,  $V_2$  and  $V_3$  as shown in the diagram so that the pressure differential in the top and bottom sections of the column are indicated on the two water manometers.
4. Take manometer readings of pressure differential across the column for a range of air flow rates. The total pressure differential is the sum of the differential across the top and bottom halves of the column, i.e. add the readings from the two water manometers.
5. Collect enough data to produce decent trends and graphs.

### **Results and Calculations**

Plot the pressure differential as a function of air flow rate on log-log scale and establish the relationship between these variables.

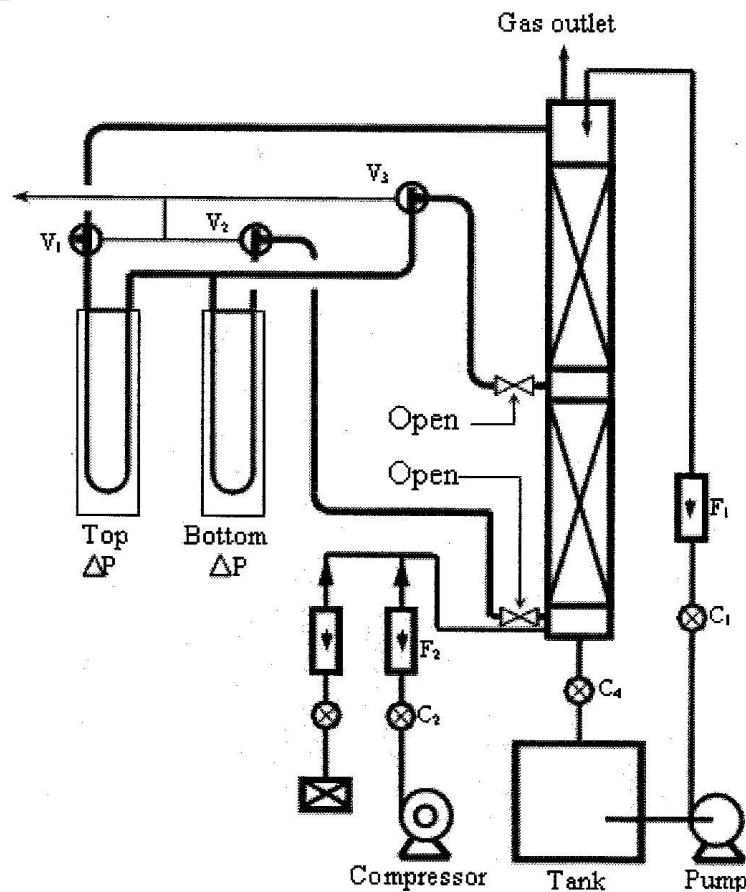


## EXERCISE B

### Objective

To examine the air pressure differential across the column as a function of the air flow rate for different water flow rates down the column.

### Equipment Set up:



No additional equipment is required for this experiment.

### Procedure

1. Fill the water reservoir tank to three-quarters full with tap water. Set valves  $V_1$ ,  $V_2$  and  $V_3$  as shown on the diagram so that differential pressures in the top and bottom sections of the column are indicated on the two water manometers.
2. Please make sure that you are firstly introducing water into the column, waiting for water stabilization and then actuating the incoming air. Follow the general steps below for each water flow rate.

For example, after running the unit with a drained column (0 L/min water flow), i.e. EXERCISE A:

- Turn off the air line,
  - Set up water flow to 1 L/min,
  - Wait until the water flow stabilizes,
  - Set up air flow to 30 L/min. Take measurements of pressure differential,
  - Re-set the air flow to 40 L/min. Take measurements of pressure differential,
  - Continue progressively changing the air flow rate until the column floods.
- 
- Turn off the air line,
  - Set up water flow to 2 L/min,
  - Wait until the water flow stabilizes,
  - Set up air flow to 30 L/min, Take measurements of pressure differential,
  - Re-set the air flow to 40 L/min. Take measurements of pressure differential,
  - Continue progressively changing the air flow rate until the column floods.

And continue this procedure with increasing flow of water up to 10 L/min until the column floods with minimum air flow. Note the appearance of the column for each experiment.

The range of possible air flow rates will decrease with increasing water flow rate due to onset of ‘flooding’ of the column, which should be noted.

## **Results and Calculations**

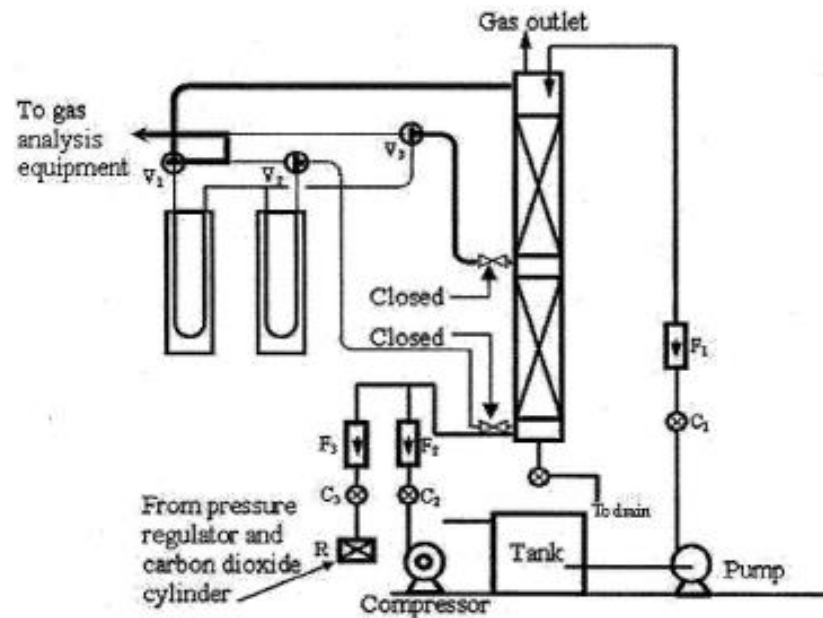
Pressure differential should be plotted as a function of air flow rate on log-log scale for each water flow rate. It may be necessary then, to take more readings in order to define precisely the transition points on the resulting graphs.

## EXERCISE C

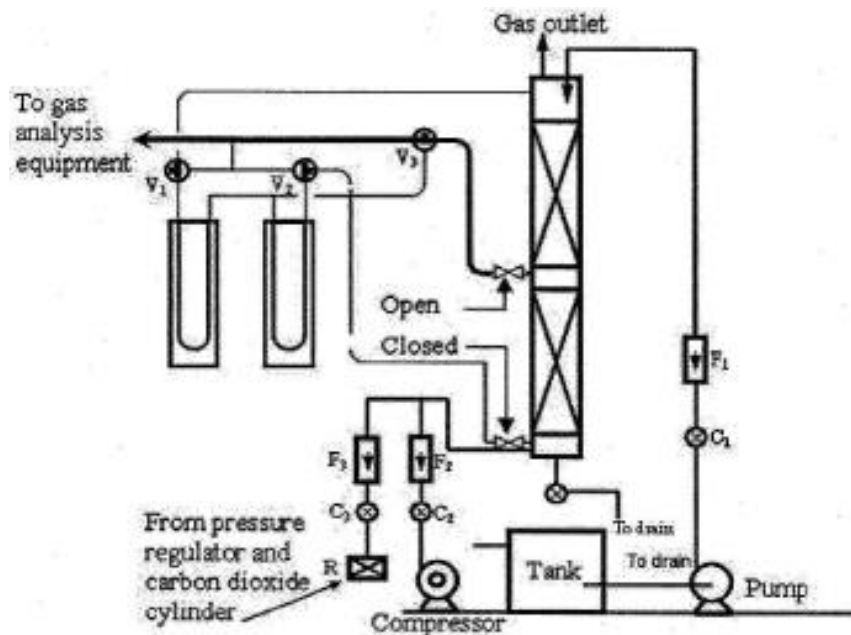
### Objective

To measure the absorption of carbon dioxide into water flowing down the tower, using the gas analysis equipment provided.

### Equipment Set Up



Obtaining a gas sample from the Top of the Column.



Obtaining a gas sample from the Middle of the Column.

## Equipment Required

1. Carbon dioxide cylinder with integral pressure regulator, connected to regulator R on gas inlet on the apparatus.
2. Approximately 300ml of 1.0 M sodium hydroxide solution. Small funnel and tubing for filling analysis equipment.

## Procedure

**Throughout this lab, be ready to turn off main water supply if tank fills too full.**

1. Turn on the main water supply to the sump tank. Fit the drain pipe to the water return from the column so that water is not returned to the sump tank.
2. First fill the two globes of the absorption analysis equipment on the left of the panel with 1.0 M NaOH and use a dropper to fill when the solution level in the globes are close to zero. If over-fill, adjust the level in the globes to the '0' mark on the sight tube, using the drain valve below the two globes and drain the NaOH into a flask to do this. (See Step A in the figures below).
3. With gas flow control valves  $C_2$  and  $C_3$  closed, start the liquid pump and adjust the water flow through the column to approximately 4 litres/minute on flowmeter  $F_1$  by adjusting control valve  $C_1$ .
4. Start the compressor and adjust control valve  $C_2$  to give an airflow of approximately 30 litres/minute in flowmeter  $F_2$ .
5. Carefully open the pressure regulating valve on the carbon dioxide cylinder, and adjust valve  $C_3$  to give a value on the flowmeter  $F_3$  approximately one half of the air flow  $F_2$ . Ensure the liquid seal at the base of the absorption column is maintained by, if necessary, adjustment of control valve  $C_4$ .
6. After 5 minutes or so of steady operation, take samples of gas from the top and middle sample points. (Refer to the appropriate diagram above for details of the valve positions.) Analyze these consecutively for carbon dioxide content in these gas samples as shown in the figures below and following notes. This means, carry out steps 7 to 13 for the analysis of the top sample and then repeat all the steps for the analysis of the middle sample.

**When carrying out the analysis of the carbon dioxide content make sure that you do not rush the procedure as this will most likely result in poor data and spills and splashes of caustic soda (NaOH) which will need to be cleaned up. Please be advised that caustic soda can cause damage to clothes and it will dissolve your skin (and other tissue).**

See below for Step A to F.

7. Flush the sample lines by repeated sucking from the line, using the gas piston and expelling the contents of the cylinder to the atmosphere. Note that the volume of the cylinder is about 100 mL. Three full volumes of the gas piston is sufficient. (Steps B and C).
8. With the absorption globe isolated and the vent to atmosphere closed, fill the cylinder from the selected line by drawing the piston out slowly (Step B). Note the volume taken into the cylinder, which should be approximately 20 ml for this particular exercise (see WARNING note below). Wait at least two minutes to allow the gas to come to the temperature of the cylinder.
9. Isolate the cylinder from the column and the absorption globe and vent the cylinder to atmospheric pressure. Close after about 10 seconds (Step D).
10. Connect cylinder to absorption globe. The liquid level should not change. If it does change, briefly open to atmosphere again.
11. Wait until the level in the indicator tube is on zero showing that the pressure in the cylinder is atmospheric.
12. Slowly close the piston to empty the cylinder into the absorption globe. Slowly draw the piston out again (Step E and F).

Note the level in the indicator tube.

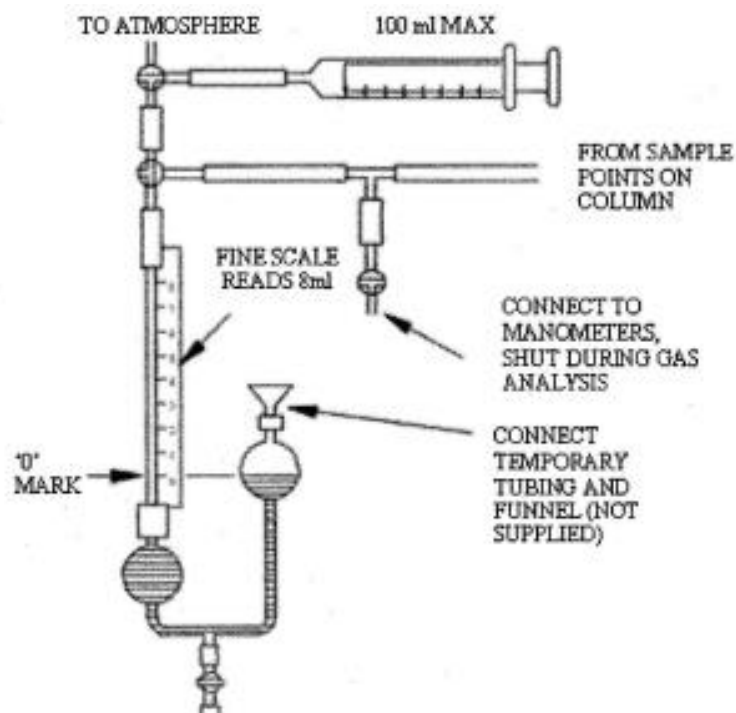
Repeat steps E and F until no significant change in level occurs. Read the indicator tube marking and record the value. This represents the volume of the gas sampled.

**WARNING:** If the concentration of CO<sub>2</sub> in the gas sampled is greater than 8%, it is possible to suck liquid into the cylinder. This will ruin your exercise and takes time to correct. Under these circumstances, do not pull the piston out to the end of its travel. Stop it at a particular mark, e.g. 20 mL on the coarse scale, and read the fine scale.

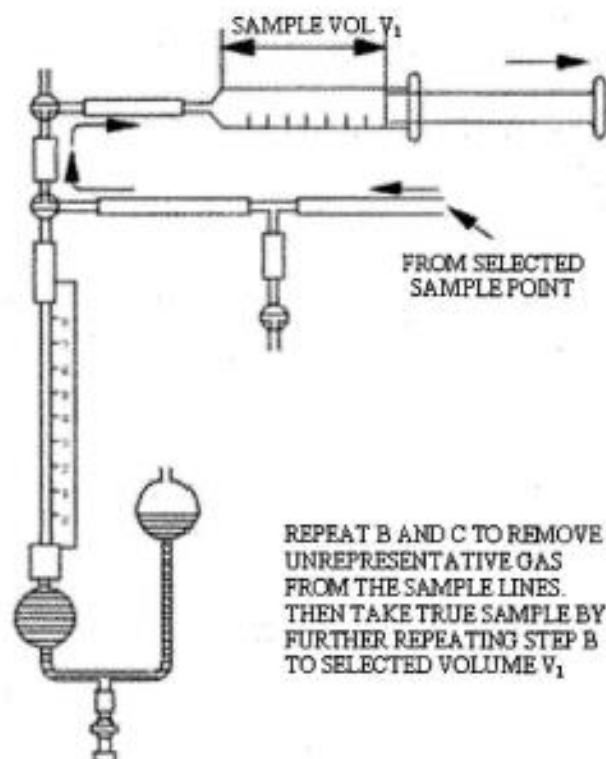
13. Once the final value has been read from the scale, slowly close the piston to empty the cylinder into the absorption globe. Reposition the valves as shown in Step G to allow the globe to equilibrate with atmosphere.

**WARNING:** It is important to follow the above steps closely in order to take representative gas samples and to achieve reproducible results. Deviation from the above can lead to erroneous results.

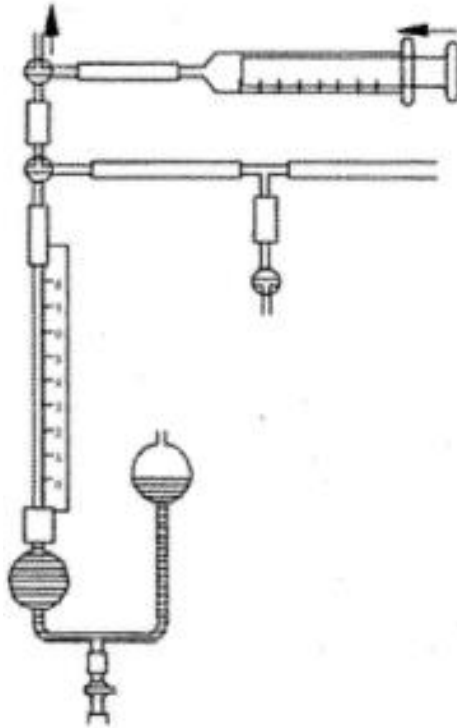
Using the Hempel apparatus for gas sampling, step A to G:



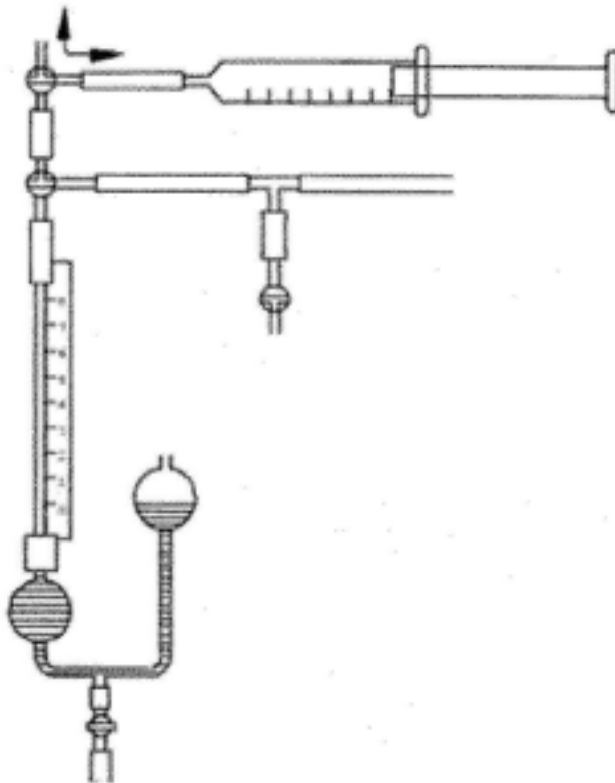
Step A: Fill with standard reagent to '0' on the scale.



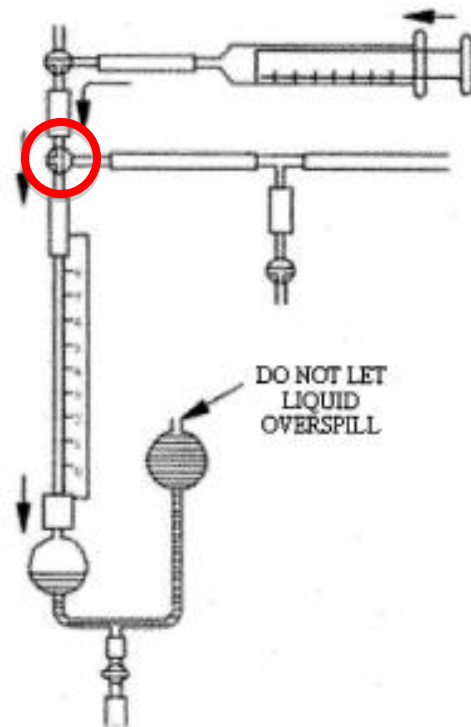
Step B: Taking gas sample



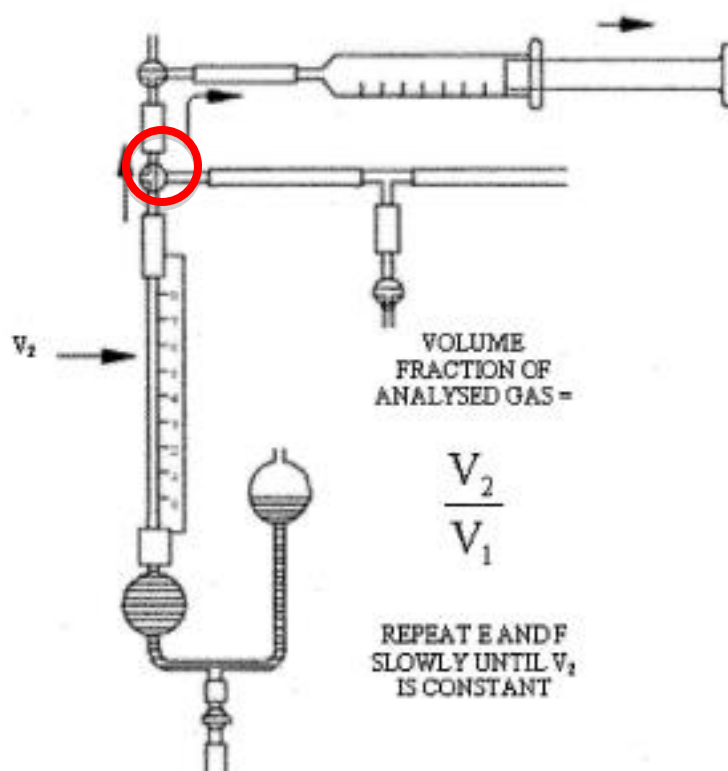
Step C: Discharging to atmosphere



Step D: Venting to atmosphere (balancing pressures)

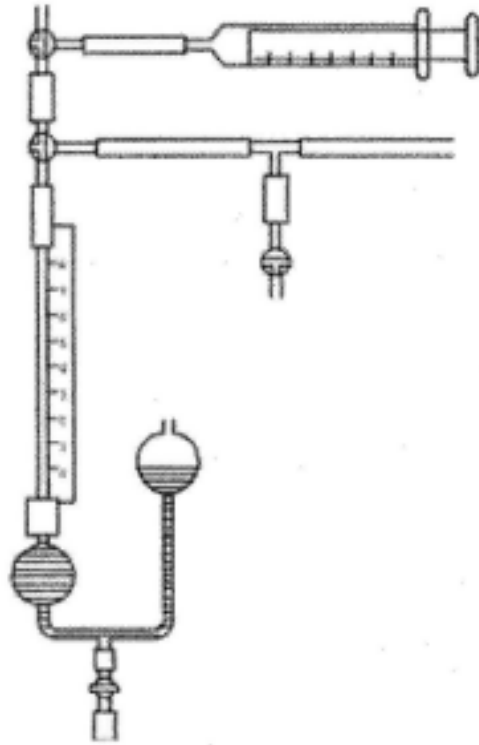


Step E: Charging the Hempel apparatus. Ensure that the valves are EXACTLY as shown above, especially valves circled in Red.



Step F: Withdrawing gas sample and taking reading  $V_2$





Step G: Resetting scale prior to next sample

## Results and Calculations

A. CO<sub>2</sub> content of gas samples:

From use of Hempel apparatus, volume fraction of

$$\text{CO}_2 = \frac{V_2}{V_1}$$

Where  $V_1$  is the volume of the gas sample taken, e.g. 20 mL, and  $V_2$  is the volume reading from the indicator tube marking.

For ideal gases, volume fraction = mole fraction =  $Y$ . Check that the sample taken from the inlet to the absorption column should give the same value of CO<sub>2</sub> fraction as that indicated by the inlet flowmeters.

i.e.

$$\text{CO}_2 = \frac{V_2}{V_1} = Y = \frac{F_3}{F_2 + F_3}$$

B. Calculation of amount of CO<sub>2</sub> absorbed in column from analysis of samples at inlet and outlet. From analysis with Hempel apparatus, volume fraction of CO<sub>2</sub> in gas stream at inlet is given by:

$$= \left( \frac{V_2}{V_1} \right)_i = Y_i$$

and at outlet:

$$Y_o = \left( \frac{V_2}{V_1} \right)_o$$

F<sub>a</sub> can be denoted as liters/second of CO<sub>2</sub> absorbed between top and bottom, this can be found by first calculating the amount of CO<sub>2</sub> going in and coming out of the column and then taking the difference:

$$CO_{2, in} = [F_2 + F_3] Y_i$$

$$CO_{2, out} = [F_2 + (F_3 - F_a)] Y_o$$

$$F_a = CO_{2, in} - CO_{2, out} = [F_2 + F_3] Y_i - [F_2 + (F_3 - F_a)] Y_o$$

$$\therefore F_a = \frac{(Y_i - Y_o)(F_2 + F_3)}{1 - Y_o} = \frac{(Y_i - Y_o)}{(1 - Y_o)} \times (\text{total gas inlet flow})$$

**Note:** Liters/second can be converted to moles/second as follows:

G can be calculated from the fact that one gram-mole occupies 22.42 liters at 273K and 760mm Hg pressure.

$$G_a = \frac{F_a}{22.42} \times \left( \frac{\text{ave. column pressure mmHg}}{760} \right) \times \left( \frac{273}{\text{ave. column temp K}} \right)$$

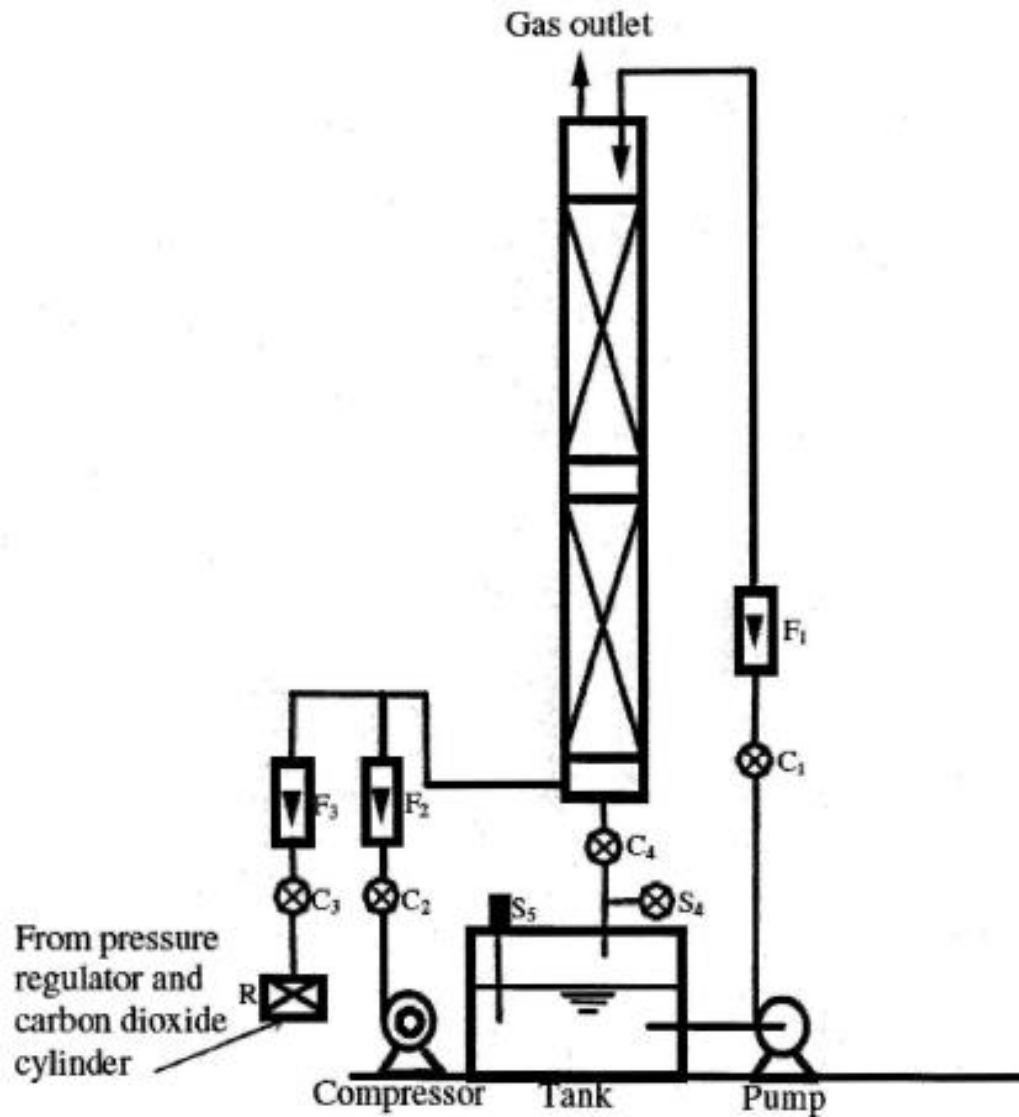
The assumption implicitly made here is that the volume flow is not affected by the pressure drop through the column as this drop should be small in comparison with atmospheric pressure. In Exercise F, a correct solution to this problem is made.

## EXERCISE D

### Objective

To calculate rate of absorption of carbon dioxide into water from analysis of liquid solutions flowing down absorption column.

### Equipment Set Up



### Procedure

1. Turn on the main water supply to the sump tank and allow the tank to fill. Ensure that the water outlet from the column is fitted to the pipe that returns water to the tank.

2. With gas flow control valves  $C_2$  and  $C_3$  closed, start the liquid pump and adjust the water flow through the column to approx. 6 liters/minute on flowmeter  $F_1$  by adjusting flow control valve  $C_1$ .
3. Start the compressor and adjust control valve  $C_2$  to give an air flow of approx. 15% of full scale on flowmeter  $F_2$ .
4. Carefully open the pressure regulating valve on the carbon dioxide cylinder, and adjust valve  $C_3$  to give a value on the flowmeter  $F_3$  approx. one half of the air flow  $F_2$ . Ensure the liquid seal at the base of the absorption column is maintained by, if necessary, adjustment of control valve  $C_4$ .
5. After 5 minutes of steady operation, take samples at 10 minute intervals from the main water inlet and from  $S_4$ . Take 150ml samples at known times in each case. Analyze the samples according to the procedure detailed below. Continue sampling until 60 minutes have passed.

#### *Analysis of Carbon Dioxide Dissolved in Water*

**Note:** Water used for absorption should be deionized as presence of dissolved salts affect the analysis described below. If tap water is used, no metal ions should be present in greater quantities than 1.0 mg/liter and pH should be just alkaline: 7.1 to 7.8.

#### *Chemical Solutions Needed*

- a. Phenolphthalein indicator prepared from carbon dioxide-free distilled water i.e. A. R. grade.
- b. Standard 0.0277 M sodium hydroxide solution, prepared by diluting 27.70 mL 1 M caustic soda standard solution to 1 liter with carbon dioxide free distilled water. Prepare daily and protect from carbon dioxide in the atmosphere by keeping in a capped Pyrex bottle.
- c. Standard 0.01 M sodium bicarbonate solution, prepared by dissolving approximately 0.1 gram of anhydrous sodium bicarbonate in carbon dioxide free distilled water to 100 mL.

#### *Procedure*

1. Take a water sample from the main water inlet, approximate volume of 150 mL, or from liquid outflow point  $S_4$ .
2. Discharge the sample at the base of a 100 mL graduated cylinder, flicking the cylinder to throw off excess liquid above the 100 mL mark.

3. Add 5-10 drops of phenolphthalein indicator solution a) above; if the sample turns red immediately, no free CO<sub>2</sub> is present. If the sample remains colorless, titrate with standard alkali solution b) above. Stir gently with a glass rod until a definite pink color persists for about 30 seconds. This color change is the end point volume (denoted V<sub>B</sub> in the equations below) of alkali solution added.

For best results, use a color comparison standard, prepared by adding the identical volume of phenolphthalein solution a) to 100 mL of sodium bicarbonate solution c) in a similar graduated cylinder.

#### *Calculation*

The amount of free CO<sub>2</sub> in the water sample (C<sub>d</sub>) is calculated from:

$$\text{mole/liter of free CO}_2 = \frac{V_B \times 0.0277}{\text{mL of sample}} = C_d$$

**Note:** Solubility of CO<sub>2</sub> in water is a strong function of temperature. The accuracy of this titrimetric method is approximately  $\pm 10\%$ .

#### **Calculations:**

The amount of CO<sub>2</sub> absorbed across the column at any particular time can be calculated and the absorption rate can be found:

$$\text{Inlet flow of dissolved CO}_2 = F_1 \cdot C_{d, \text{in}}$$

$$\text{Outlet flow of dissolved CO}_2 = F_1 \cdot C_{d, \text{out}}$$

$$\text{Absorption rate} = F_1 \cdot [C_{d, \text{in}} - C_{d, \text{out}}]$$

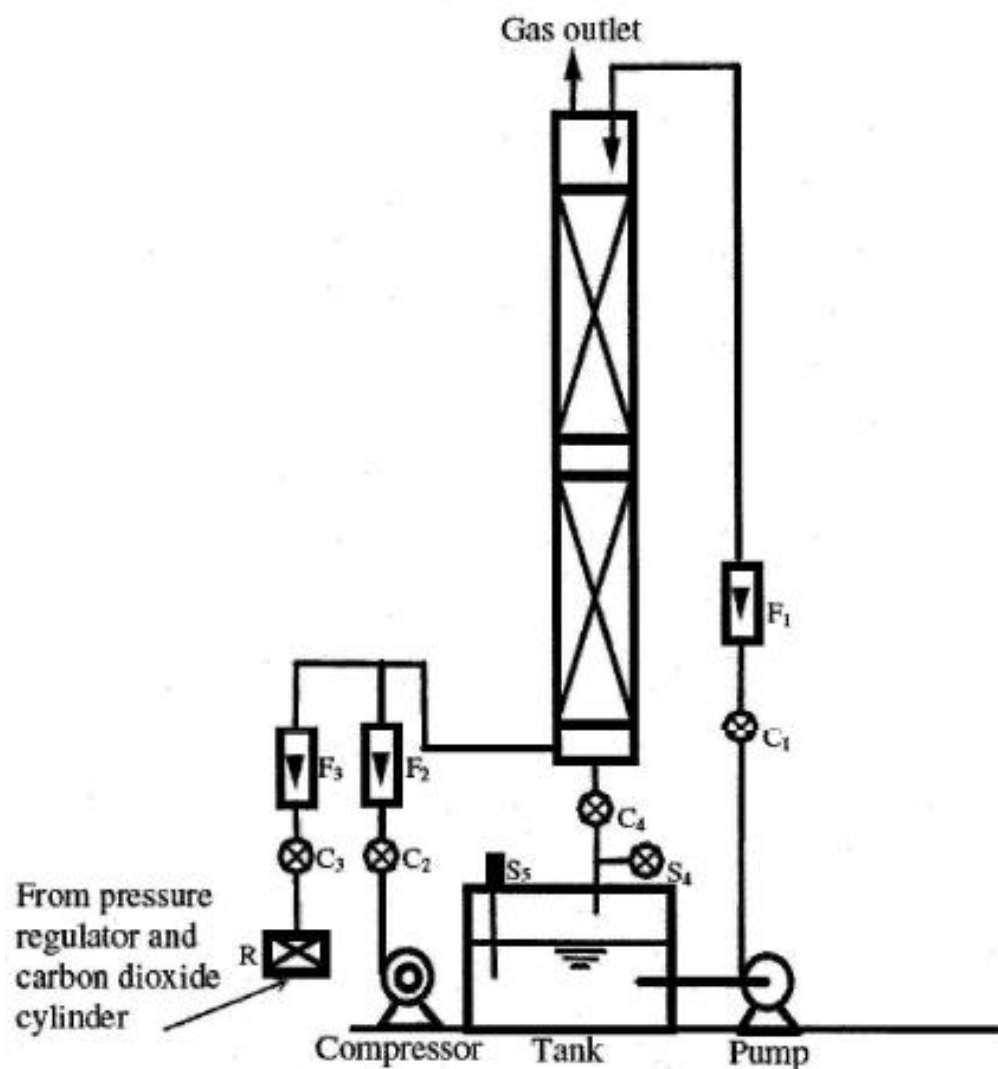
## EXERCISE E

### Objective

To calculate rate of absorption of carbon dioxide into caustic soda solutions from analysis of liquid solutions flowing down absorption column.

**Before beginning this experiment, contact the TA for instruction.**

### Equipment Set Up



### Procedure

1. Exchange the water in the sump tank at base of the column and fill to approximately ~39.6 L clean tap water.

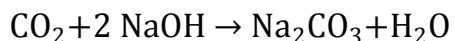
2. Ensure that the water outlet from the column is fitted to the pipe that returns water to the tank.
3. Calculate the number of mL of 50/50 wt. % NaOH solution to add to produce 40L 0.2 caustic soda solution (Density = 1.5g/mL @ 25° C). Once calculation is complete, call over TA to prepare the solution.
4. TA will add the required volume of 50/50 wt% NaOH solution in small aliquots, stirring often. Note to TA: The preparation of this requires great care and GLOVES, GOGGLES and FACE SHIELD MUST BE WORN.
5. With gas flow control valves C<sub>2</sub> and C<sub>3</sub> closed, start the liquid pump and adjust the flow of caustic soda through the column to approximately 3 liters/min on flowmeter F<sub>1</sub> by adjusting control valve C<sub>1</sub>.
6. Start the compressor and adjust valve C<sub>2</sub> to give an air flow of approximately 30 liters/minute on flowmeter F<sub>2</sub>.

**CAUTION:** As the solution used in this exercise is caustic it is essential that the column is not allowed to flood. The air supply should be turned off immediately if any liquid is present in the clear tube above the top of the column.

1. Carefully open the pressure regulating valve on the carbon dioxide cylinder and adjust valve C<sub>3</sub> to give a flow of 3 liters/minute on flowmeter F<sub>3</sub>. Ensure the liquid seal at the base of the absorption column is maintained by, if necessary, adjustment of control valve C<sub>4</sub>.
2. After 15 minutes of steady operation, take samples at 20 minute intervals simultaneously from S<sub>4</sub> and S<sub>5</sub>. Be very careful in handling the caustic solution. Continue sampling until 60 minutes have passed. It is recommended that a 250ml sample is taken so analysis can be done in duplicates. Note the time the samples were taken in each case. Analyze both samples according to the procedure detailed below. Make sure you start the analysis immediately so that you do not accumulate samples as the procedure is time consuming.

*Analysis of Carbon Dioxide Dissolved in Caustic Soda/Sodium Carbonate Solutions Theory:*

The absorption of carbon dioxide from mixtures with air into caustic solutions is characterized by the overall reaction (for most conditions) as:



Under the conditions chosen for the absorption exercises, the amount of CO<sub>2</sub> removed from the air stream can be estimated from the amounts of NaOH and Na<sub>2</sub>CO<sub>3</sub> in the liquid samples, as virtually no 'free' CO<sub>2</sub> will remain unreacted in the liquid.

In using titration techniques of analysis, acid is first used to neutralize the caustic soda and at the same time converts all sodium carbonate to bicarbonate. Continuation of the titration with acid then neutralizes all bicarbonate. The total concentration of carbonate can thus be determined, and hence the amount of CO<sub>2</sub> absorbed deduced.

*Solutions Needed:*

1. Phenolphthalein indicator prepared from carbon dioxide-free distilled water.
2. Methyl orange indicator similarly prepared.
3. One liter of standardized 0.20 M hydrochloric acid.
4. One liter of 5% by weight of barium chloride solution.

*Analysis Procedure:*

- a. Take a 250 mL sample of liquid from the absorption column liquid outlet or sump tank (as directed in the exercise) into a conical flask. **Be very careful!** Accurately measure 100 mL into two separate conical flasks for each sample.
- b. Flask 1 - Add a drop of phenolphthalein solution 1) to the contents and titrate until the pink color just disappears with the standard hydrochloric acid 3) Note the volume of acid added (denote this as T<sub>1</sub>), which is that needed to neutralize all hydroxide and convert carbonate to bicarbonate. Then add a drop of methyl orange 2) to the flask and continue to titrate with standard acid 3) until the end point is found. Note the total acid added to this second end point (denote this as T<sub>2</sub>), which represents neutralization of all bicarbonate as (T<sub>2</sub> - T<sub>1</sub>).
- c. Flask 2 - Add about 10 % more than the value of (T<sub>2</sub> - T<sub>1</sub>) of the barium chloride solution 4 ), to the flask contents and shake well. This precipitates out all the original carbonate in the sample as barium carbonate. Now add two drops of phenolphthalein solution 1) and titrate against the standard acid solution 3) to the end point. Note the volume of acid added (denote this as T<sub>3</sub>), which represents that needed to neutralize only the original caustic soda. (T<sub>2</sub> - T<sub>3</sub>) represents the difference between total acid required for carbonate and hydroxide, and that required for hydroxide alone.



It is advisable to repeat all of the above to check reproducibility.

*Calculations of Sample Composition*

- a. Concentration of NaOH in original sample (denoted C<sub>C</sub>):



$$C_C = \frac{T_3}{50} \times 0.2 \text{ M}$$

b. Concentration of  $\text{Na}_2\text{CO}_3$  in original sample (denoted  $C_N$ ):

$$C_N = \frac{(T_2 - T_3)}{50} \times 0.2 \text{ M} \times 0.5$$

c. Amount of  $\text{CO}_2$  removed from air mixture:

During a time interval, or between the top and bottom of the column,  $C_N$  will increase as  $\text{CO}_2$  is absorbed in equi-molar proportions, while  $C_C$  should decrease in twice-molar proportions.

### Results and Calculations:

Make sure you make notes of all titration volumes, flow rates and total volume of the solution in the system.

#### *Calculations:*

The amount of  $\text{CO}_2$  absorbed across the column as measured from samples taken simultaneously from the sump tank feeding the column top and at the bottom outlet, is given by:

$$\text{CO}_2 \text{ absorbed} = \text{Liquid flow rate} \times [C_{N, \text{out}} - C_{N, \text{in}}]$$

and:

$$\text{CO}_2 \text{ absorbed} = \text{Liquid flow rate} \times \frac{1}{2} [C_{C, \text{in}} - C_{C, \text{out}}]$$

Similarly, over a time period  $\Theta$  seconds after a first sample is taken from the sump  $S_5$ :

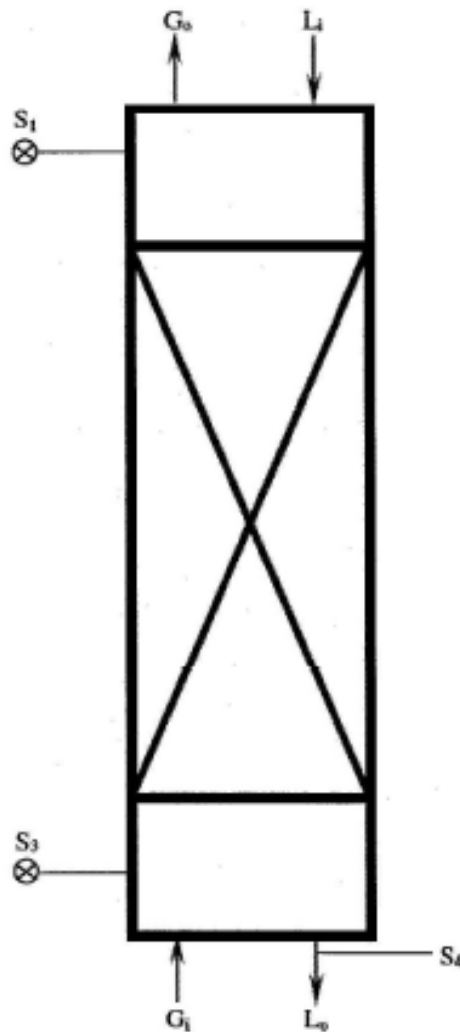
$$\text{CO}_2 \text{ absorbed} = \text{Volume of liquid in the whole system} \times [(C_N)_{t=\Theta} - (C_N)_{t=0}]$$

## EXERCISE F

### Objective

To show that the amount of carbon dioxide removed from the air stream equals the amount taken up by a liquid stream of caustic soda solution.

### Theory



At steady state, the transfer of absorbing gas from the gas stream should equal that transferred to the liquid.

Let:  $L_i$  and  $L_o$  be the volume flow of liquid entering and leaving the column respectively.

Let:  $G_i$  and  $G_o$  be the total gas molal flow entering and leaving the column respectively.

Let:  $Y_i$  and  $Y_o$  be the mole fractions of  $\text{CO}_2$  entering and leaving the column in the gas stream.

Amount of CO<sub>2</sub> removed from the gas stream:

$$G_i - G_o \text{ (moles/sec)} \quad (1)$$

as no air dissolved in the solution.

But from Exercise D, the amount of CO<sub>2</sub> removed from the liquid stream equals the amount of carbonate ions produced:

$$L_o * C_{No} - L_i * C_{Ni} \text{ (moles/sec)} \quad (2)$$

The object is to check that (1) equals (2). Note that, while the liquid flows in and out are the same in this exercise ( $L_o = L_i$ ), the gas flows are not equal because of the removal of CO<sub>2</sub>, and also because of the pressure drop across the column.

G<sub>o</sub> can be calculated from a molar balance on the air stream, none of which is absorbed:

$$G_o * (1 - Y_o) = G_i * (1 - Y_i)$$

G can be calculated from the fact that one gram mole occupies 22.42 liters at 273 K and 760mm Hg pressure:

$$G = \frac{F_2 + F_3}{22.42} \times \left( \frac{760 + \text{column pressure drop}}{760} \right) \times \left( \frac{273}{\text{ave. column temp K}} \right)$$

And Y<sub>i</sub> and Y<sub>o</sub> are estimated by sampling as in Exercise C.

## Procedure

The same procedure should be followed as for Exercise E, except that gas samples at the inlet and outlet, as described in Exercise C, are to be taken as well.

As the liquid composition is slowly changing as progressive absorption of CO<sub>2</sub> takes place, the steady state can only be approximated by taking samples as close to each other in time as possible.

Thus, after 5 minutes of circulating liquid and gases at the pre-set rates, take

1. a gas outlet sample, to give Y<sub>o</sub>,
2. a liquid outlet sample from the point S<sub>4</sub> as soon after step 1 as possible,
3. a liquid sample from the sump tank at S<sub>5</sub> at the same time,
4. inlet gas sample, which should be constant and therefore can be taken last.

Below is an example of the data that needs to be collected and the necessary calculations:

Readings:	Comments:
$F_2 = 0.40$ litres/second	Flowmeter readings divided by 60
$F_3 = 0.052$ litres/second	
$L = 0.051$ litres/second	
$Y_o = 0.032$	From Hempl apparatus readings (Use 0.111 for $Y_i$ )
$Y_i = 0.111$	
$\left[ \text{and } Y = \frac{F_3}{F_2 + F_3} = \frac{0.052}{0.40 + 0.052} = 0.115 \right]$	
$T_1 (o) = 33.2$ ml	As in exercise C, 0.20M HCl used in titrations of 50ml sample.
$T_2 (o) = 50.9$ ml	
$T_3 (o) = 35.0$ ml	
$T_1 (i) = 48.4$ ml	As in exercise C, 0.20M HCl used in titrations of 50ml sample.
$T_2 (i) = 47.2$ ml	
$T_3 (i) = 45.0$ ml	
$P = 759$ mm Hg	Lab. Barometer
$\Delta P = 100$ mm $H_2O$	$\frac{100}{13.6} = 7.35 \approx 7$ mm Hg
$\theta_i = 19^\circ C$	292 K

*Calculations:*

1. Gas Flows:

$G_i$  (moles/second of gas mixture entering column)

$$G_i = \frac{0.40 + 0.052}{22.42} \times \left( \frac{760 + 7}{759} \right) \times \left( \frac{273}{292} \right) = 0.019 \text{ moles/second}$$

and

$$G_o = \frac{G_i(1 - Y_i)}{(1 - Y_o)} = 0.019 \times \frac{1 - 0.111}{1 - 0.032} = 0.0175 \text{ moles/second}$$

$$\therefore \text{Amount of CO}_2 \text{ removed} = 0.0015 \text{ g. moles/second} \quad (1)$$

## 2. Liquid Flows:

a. NaOH: Concentration of CO<sub>2</sub> related to hydroxide consumption in liquid:

$$\text{At inlet} = \frac{T_{3,i}}{50} \times 0.20 = \left(\frac{45}{50}\right) \times 0.20 = 0.18 \text{ M} = C_{ci}$$

$$\text{At outlet} = \frac{T_{3,o}}{50} \times 0.20 = \left(\frac{30}{50}\right) \times 0.20 = 0.12 \text{ M} = C_{co}$$

Caustic soda used is equivalent to CO<sub>2</sub> absorbed:

$$= L \times \frac{1}{2} (C_{ci} - C_{co}) = \frac{0.051}{2} (0.18 - 0.12) = 0.0015 \text{ moles/second} \quad (2)$$

b. Na<sub>2</sub>CO<sub>3</sub>

As from Exercise E, concentration of Na<sub>2</sub>CO<sub>3</sub>:

$$C_N = \frac{(T_2 - T_3)}{50} \times 0.2 \text{ M} \times 0.5$$

$$\therefore C_{N, \text{inlet}} = \frac{(47.2 - 45.0)}{50} \times 0.2 \times 0.5 = 0.0044 \text{ M}$$

and

$$C_{N, \text{outlet}} = \frac{(50.9 - 35.0)}{50} \times 0.2 \times 0.5 = 0.0318 \text{ M}$$

Hence, the carbonate produced (by CO<sub>2</sub> absorption)

$$= L (C_{N,o} - C_{N,i}) = 0.051 (0.0318 - 0.0044)$$

$$= 0.0014 \text{ moles/second} \quad (3)$$

The results of both (2) and (3) agree well with (1).

## EXERCISE G

### Objective

Determination of Overall Mass Transfer Coefficient ( $K_{og}$ ).

### Theory

The familiar equation for packed tower is:

$$H = \int_{Y_i}^{Y_o} \frac{d[G \cdot Y]}{K_{og} \cdot a \cdot A (Y^* - Y)}$$

Where  $Y^*$  is the mole fraction of gas in equilibrium with the liquid at any point in the tower and where  $Y$  is the bulk mole fraction;  $A$  is the cross sectional area of the tower;  $H$  is the packing height and  $a$  is the specific area of packing/unit volume of packing.

For dilute gases in an otherwise inert gas stream, the above equation can be simplified:

$$\frac{H \cdot a \cdot A \cdot K_{og}}{G} = \int_{Y_i}^{Y_o} \frac{dY}{Y^* - Y}$$

The right hand side of this equation is difficult to integrate and  $K_{og}$  is more simply (but less accurately) evaluated from the definition of  $K_{og}$  as:

$$N = K_{og} \cdot a \cdot AH \cdot \Delta P_m$$

Where

$N$	=	Rate of absorption (moles/second)
$K_{og}$	=	Mass transfer coefficient (mol/atm.m <sup>2</sup> .s)
$a$	=	specific area of packing /unit volume of tower (m <sup>2</sup> /m <sup>3</sup> )
$AH$	=	volume of column (m <sup>3</sup> )
$\Delta P_m$	=	log mean driving force (atm, mm Hg)

The log mean driving force is expressed as:

$$\Delta P_m = \frac{p_i - p_o}{\ln \frac{p_i}{p_o}}$$

where  $p_i$  is pressure at the column inlet and  $p_o$  is the pressure at column outlet.

## Procedure

The exercise is conducted as for Exercise F, except that the liquid analyses may be omitted as the gas analysis alone can determine the rate of absorption [as in Exercise C].

## Results and Calculations

*Below is an example of a typical calculation:*

1. N is calculated as in Exercises A and D.
2. a is specific area of packing/unit volume of tower, which for 9mm Raschig rings is  $440 \text{ m}^2/\text{m}^3$ .
3. AH is volume of the column.  
= volume of tower = (area \* height).
4. Partial pressures (of  $\text{CO}_2$ ):

Partial pressure = mole fraction \* total pressure

Partial pressure = volume fraction \* total pressure

Using results of Exercise F:

$$\text{Inlet } p_i = 0.111 \times \left( \frac{759.17}{760} \right) = 0.111 \text{ atm}$$

$$\text{Outlet } p_o = 0.032 \times \left( \frac{759}{760} \right) = 0.032 \text{ atm}$$

$$\text{Log mean driving force} = \Delta P_m = \frac{p_i - p_o}{\ln \frac{p_i}{p_o}} = \frac{0.111 - 0.032}{\ln \left( \frac{0.111}{0.032} \right)} = 0.064 \text{ atm}$$

5. Mass transfer coefficient:

$$K_{og} = \frac{N}{a \cdot AH \cdot \Delta P_m} = \frac{0.0015 \left( \frac{\text{moles}}{\text{sec}} \right)}{0.062 \text{ m}^3 \times 440 \text{ m}^2/\text{m}^3 \times 0.064 \text{ atm}} = 0.0086 \text{ moles}/\text{m}^2 \cdot \text{atm} \cdot \text{sec}$$