

# LABORATORY MANUAL ON CHE 310 (CHEMICAL ENGINEERING LAB 2)



## DEPARTMENT OF CHEMICAL ENGINEERING

FEDERAL UNIVERSITY OF TECHNOLOGY OWERRI  
P.M.B 1526 OWERRI, IMO STATE, NIGERIA

NAME:.....

REG. NO:.....

GROUP:..... DATE:.....

ADDRESS:.....



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**(CHEMICAL ENGINEERING LAB 2)**

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## PREFACE

The Chemical Engineering Department in the Federal University of Technology has four laboratories which are as follows:

- The Unit Operations Laboratory
- The Transport Phenomena Laboratory
- The Reaction Engineering Laboratory, and
- The Analysis Laboratory

The Chemical Engineering programme is structured such that the practical components of all our courses are extracted and packaged into 3 laboratory courses namely:

- CHE 305 (Chemical Engineering Laboratory 1)
- CHE 310 (Chemical Engineering Laboratory 2) , and
- CHE 411 (Chemical Engineering Laboratory 3)

These 3 laboratory courses cover the gamut of the Chemical Engineering knowledge such as Reaction Kinetics & Engineering, Transport Phenomena, Unit Operations & Unit Processes, Particulate Fluid Systems, Control Engineering, Corrosion Studies and other fundamental aspects.

As a student of the Department, your primary concern as you participate in these laboratory courses should be to establish the nexus between these stand-alone laboratory modules and the corresponding courses/theoretical principles being demonstrated. This should not be difficult if you have been attending lectures and if you pay close attention to the introductory comments made by the instructors before each laboratory practical.

It is hoped by this delicate balancing of the theoretical and practical aspects of our courses that our graduates will emerge fully poised to proffer solutions to the ever increasing societal engineering challenges which only Chemical Engineers are equipped to solve.

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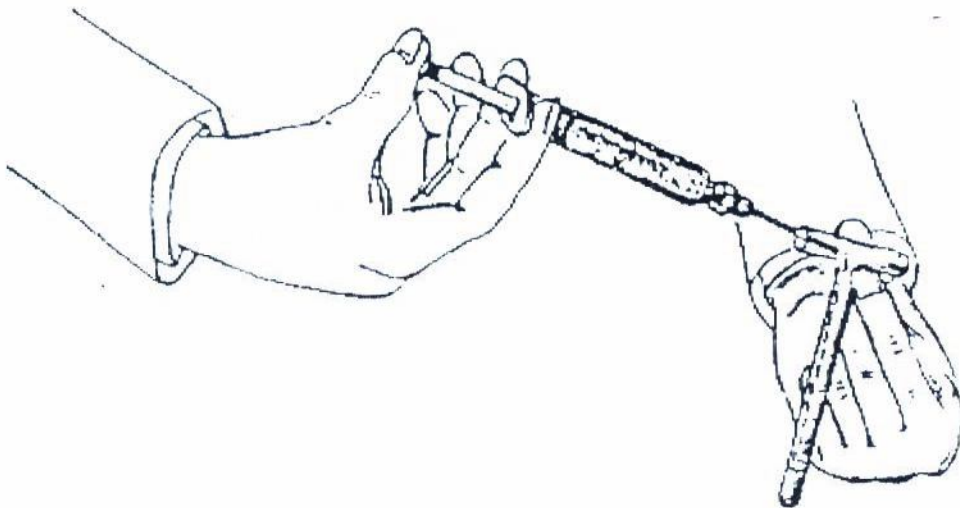
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## EXPERIMENT 1

### DIFFUSION OF A GAS

#### PRIMING PROCEDURE FOR CAPILLARY TUBE

Before using the capillary tube in an experiment using Acetone, it will be necessary to clean the inside of the tube by using a detergent such as washing-up liquid. A weak solution of the detergent should be injected into the tube slowly as shown below.



With the point of the hypodermic needle over the edge of the capillary tube, gently press the syringe, so that droplets of solution fall down onto the inside wall of the capillary tube. If the solution does not flow down into the bottom of the tube, but instead forms a meniscus with air trapped beneath it, gently tap the outside wall of the tube near the meniscus with your finger. Repeat this procedure until the capillary tube is full. To empty the tube, simply shake the tube whilst it is upside down until all the solution has gone.

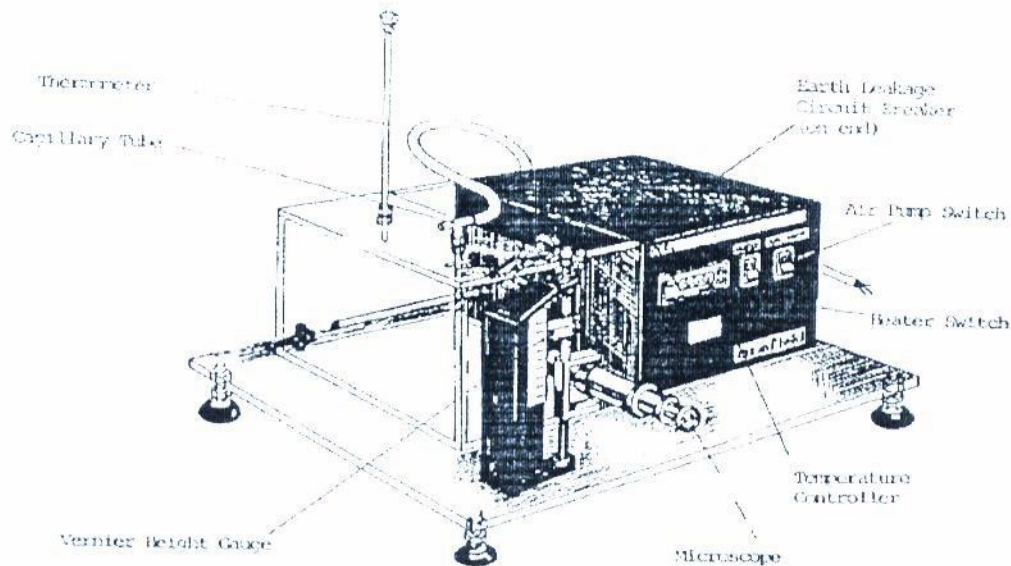
The capillary tube can now be primed with Acetone using the same procedure, but this time there should be no need to tap the tube as it should fill easily. The depth of Acetone should be approximately 35mm when filled.



## EXERCISE A – DIFFUSION OF A GAS

**OBJECTIVE:** To determine the diffusion coefficient of a gas by evaporation from a liquid surface.

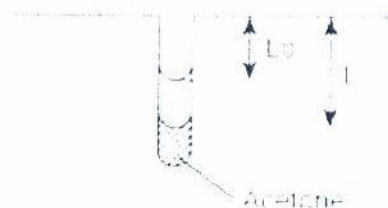
### Equipment Set-Up



### Theory

The diffusivity of the vapour of a volatile liquid in air can be conveniently determined by Winkelman's method in which liquid is contained in a narrow diameter vertical tube, maintained at a constant temperature, and an air stream is passed over the top of the tube to ensure that the partial pressure of the vapour is transferred from the surface of the liquid to the air stream by molecular diffusion.

The rate of mass transfer is given by:



$$N'_n = \frac{N_n}{L} \frac{N_n}{N_{mm}}$$

Where:

$D$	=	Diffusivity ( $\text{m}^2/\text{s}$ )
$C_A$	=	Saturation concentration at interface ( $\text{kmol}/\text{m}^3$ )
$L$	=	Effective distance of mass transfer (mm)
$C_{Bm}$	=	Logarithmic mean molecular concentration of vapour ( $\text{kmol}/\text{m}^3$ )

$$C_T = \text{Total molar concentration} = C_A + C_{am} \text{ (kmol/m}^3\text{)}$$

Considering the evaporation of the liquid.

$$N'_A = \left(\frac{\rho_L}{M}\right) \frac{dL}{dt}$$

Where  $\rho_L$  is the density of the liquid

$$\left(\frac{\rho_L}{M}\right) \frac{dL}{dx} = D \left(\frac{C_A}{L}\right) \left(\frac{C_T}{C_{Bm}}\right)$$

Thus

Integrating and putting  $L = L_0$  at  $t = 0$

$$L^2 - L_0^2 = \left(\frac{2MD}{\rho_L}\right) \left(\frac{C_A C_T}{C_{Bm}}\right) t$$

**Note:**  $L_0$  and  $L$  cannot be measured accurately but  $L - L_0$  can be measured accurately using the veneeron the microscope.

$$(L - L_0) (L - L_0 + 2L_0) = \left(\frac{2MD}{\rho_L}\right) \left(\frac{C_A C_T}{C_{Bm}}\right) t$$

$$\text{Or } \frac{t}{(L - L_0)} = \left(\frac{\rho_L}{2MD}\right) \left(\frac{C_{Bm}}{C_A C_T}\right) (L - L_0) + \left(\frac{\rho_L C_{Bm}}{MDC_A C_T}\right) L_0$$

Where  $M$  = molecular weight kg (mol),  $t$  = time (s)

If  $s$  is the slope of a graph of  $\frac{t}{(L - L_0)}$  against  $(L - L_0)$  then:

$$S = \frac{(\rho_L C_{Bm})}{2MDC_A C_T} \text{ or } D = \frac{(\rho_L C_{Bm})}{s(2MDC_A C_T)}$$

Where

$$C_T = \left(\frac{1}{K_{molVol_T}}\right) \left(\frac{T_{shs}}{T_a}\right) \text{ (Kmol Volume} = 22.214\text{m}^3\text{/kmol)}$$

$$C_{B1} = C_T$$

$$C_{B2} = \left(\frac{P_a - P_u}{P_a}\right) C_T$$

$$C_{Bm} = \frac{(C_{b1} - C_{b2})}{\ln\left(\frac{C_{b1}}{C_{b2}}\right)}$$

$$C_A = \left(\frac{P_v}{P_a}\right) C_T$$

### Readings to be taken:

**Note:** To prevent the acetone from boiling do not set the temperature controller above 50°C.

Partially fill the capillary tube with Acetone to a depth of approximately 35mm (see Priming Procedure for Capillary Tube). Remove top nut from metal fitting. Carefully insert capillary tube through the rubber ring, inside the metal nut until the top of the tube rests on the top of the nut. Gently screw this assembly onto the top plate with the 'T' piece normal to the microscope. Connect flexible air tub to one end of the 'T' piece. With the microscope set up as shown, adjust the object lens to within 20-30mm from the tank.

Adjust the vertical height of the microscope until the capillary tube is visible; if the capillary tube is not visible adjust the distance from the object lens to the tank until it is. For a clearer and well defined view of the meniscus inside the capillary tube, adjust the position of the viewing lens in or out of the microscope body as necessary. Note that when viewing the capillary tube the image will be upside down, so that the bottom of the tube is at the top of the image. When the meniscus has been determined, the sliding vernier scale should be aligned with a suitable graduation on the fixed scale. Switch on air pump. (Airflow should only be low velocity across the capillary tube and can be adjusted using the Hoffman clip on the flexible tube.) Record the level inside the capillary tube. Switch on temperature controlled water bath (adjust set point on controller to 40°C) and obtain a steady temperature. After approximately 60 minutes, switch off water bath (to prevent air bubbles from obscuring the reading) and record the change in level inside the capillary tube. Switch on bath and repeat the procedure approximately every 60 minutes.

$(L-L_0)$  at time  $t$  = Initial reading on vernier – reading on vernier of time  $t$ .

## Results

Time from commencement of Experiment	Liquid Level $(L-L_0)$	$\frac{t}{(L-L_0)}$
Ks	mm	Ks/mm

Plot  $t / (L - L_0)$  against  $(L-L_0)$  and determine the gradients from the graph.

Armfield Instruction Manual

Calculate the diffusivity  $D$  using your results.

Note: The vapour pressure of acetone changes with temperature. At 313K (40°C) the vapour pressure  $P_v$  is 56kN/m<sup>2</sup>. If the experiment is performed with the water bath set to different temperatures it will be necessary to obtain suitable values for  $P_v$ .

The density of acetone is 790kg/m<sup>3</sup>. Take the kmol volume as 22.4m<sup>3</sup>. The molecular weight is 58.08 kg/mol.

Repeat the experiment at different temperatures and comment on the effect of temperature on the diffusivity  $D$ .

A set of typical results is presented overleaf for information.



### Typical Results

Diffusivity of Acetone in air at 313K (40°C) and atmospheric pressure (Pa = 101.3) from the following experimental data.

Time from commencement of Experiment	Liquid Level (L-L <sub>0</sub> )	T (L-L <sub>0</sub> )
Ks	mm	Ks/mm
0.000	0.00	0.000
3.60	2.20	1.636
7.20	4.20	1.714
11.160	6.30	1.771
15.900	8.80	1.807
19.980	10.80	1.850
23.400	12.40	1.887
78.780	34.50	2.233
83.520	36.10	2.313
87.240	37.30	2.339
91.800	38.90	2.360
97.320	40.80	2.385
101.100	42.00	2.407

From resulting graph

$$\begin{aligned}
 S &= 0.0175 \text{ ks/mm}^2 \text{ or } 1.75 \times 10^7 \text{ s/m}^2 \\
 CT &= (1/22.4) (273/313) = 0.0389 \text{ kmol/m}^3 \\
 M &= 58.08 \text{ kg/mol} \\
 C_A &= (56/101.3) 0.0389 = 0.0215 \text{ kmol/m}^3 \\
 P_L &= 790 \text{ kg/m}^3 \\
 C_{B1} &= 0.0389 \\
 C_{B2} &= \{(101.3 - 56) / 101.3\} \times 0.0389 = 0.0174 \text{ kmol/m}^3 \\
 C_{Bm} &= (0.0389 - 0.0174) / \ln (0.0389/0.0174) = 0.0267 \text{ kmol/m}^3
 \end{aligned}$$

$$\begin{aligned}
 \text{Therefore } D &= (790 \times 0.0267) / (2 \times 58 \times 0.215 \times 0.0389 \times 1.75 \times 10^7) \\
 D &= 21.09 / 1.700 \times 10^6 \\
 D &= 12.4 \times 10^{-6} \text{ m}^2/\text{s}
 \end{aligned}$$

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## EXPERIMENT 2

### DIFFUSION OF A LIQUID

#### Nomenclature

Name	Symbol	Units	Definition
Time	t	secs	Measured variable
Diffusion Constant	D	cm <sup>2</sup> /sec	Calculated variable
Diffusion Flux	J	Mol/cm <sup>2</sup> sec	Calculated variable (obtained from slop of graph)
Volume in vessel	V	litres	Constant (typically 1.01)
Diameter of capillaries	d	cm	Constant (typically 0.1)
Height of capillaries	x	cm	Constant (typically 0.45)
Molarity of salt solution in diffusion cell	M	Mol/litre	Constant for a given salt solution
Number of capillaries	N		Constant (typically 121)
Electrical conductivity change for unit Molarity change	Cm	$\Omega^{-1}M^{-1}$	Constant for a given Molarity of a particular salt (typically 0.112 for 1MNaCl)
Concentration in vessel	C	Mol/cm <sup>3</sup>	Constant for "a"

### EXERCISE A – DIFFUSION OF A LIQUID

#### OBJECTIVE

To determine the Diffusion Coefficient of 1M NaCl solution.

#### METHOD

A small volume of concentrated solution is placed on one side of the honeycomb of capillaries, inside the glass diffusion cell, whilst the other side consists initially of a large volume of pure solvent (water). As diffusion of the solute occurs, the concentration within the larger volume increases and is monitored with a conductivity concentration within the bulk liquid. Whilst the apparatus may be used with any required aqueous system, for introductory studies, 1.0 M or 0.1M solutions of Sodium Chloride are recommended, for which conductivity data is provided.

#### EQUIPMENT REQUIRED

Armfield CERB comprising

- Magnetic stirrer
- Acrylic diffusion vessel incorporating conductivity electrode
- Lid for vessel incorporating glass diffusion cell
- Conductivity meter
- 1 Litre measuring beaker
- Plastic syringe
- Filter paper (not supplied)



- Deionised / Demineralised water (Not supplied)
- 1 M solution of NaCl (Not supplied)
- Volumetric glassware and balance for making up solutions of known concentration (not supplied)
- Stopwatch (if PC with CERB data logging software is not available).

### Optional Equipment

Compatible PC (Not supplied) for logging conductivity readings; requires RS232 serial Port.

Armfield software for CERB (Supplied with CERB)

### Theory

The rate of diffusion is expressed by the equation:

$$J = -D \frac{\partial C}{\partial x}$$

Where J is the diffusion flux across the unit right angles to the x – direction

D is the diffusion constant

C is the Concentration

and  $\frac{\partial C}{\partial x}$  is the concentration gradient in the x direction.

(the negative sign indicates that flow is from high to low concentration)

If J is in mole/cm<sup>2</sup> sec

C is in mole / cm<sup>3</sup>

x is in cm

and t is in seconds

then D will be in cm<sup>2</sup>/Sec

The apparatus uses 121 vertical capillaries, 4.5mm long and 1 mm bore to restrict diffusion to one dimension. The concentration at the lower ends is chosen and taken to be constant. The concentration at the top ends is effectively zero during the experiment.

$$\text{thus } \frac{V}{C_M} \frac{dk}{dt} = -D \frac{\pi d^2}{4} N \frac{M}{x}$$

$$\text{Therefore } D = - \frac{AVx}{\pi d^2 N M C_M} \frac{dk}{dt}$$

Nominal dimensions

Where

V = Volume of water in outer vessel in litres 1 litre

x = Length of capillaries in cm 0.45 cm

d = Diameter of capillaries in cm 0.1cm

N = Number of capillaries 121

M = Molarity of salt solution in diffusion cell

C<sub>m</sub> = Electrical conductivity change for unit

Molarity change (dilute solution) Ω<sup>-1</sup>M<sup>-1</sup>

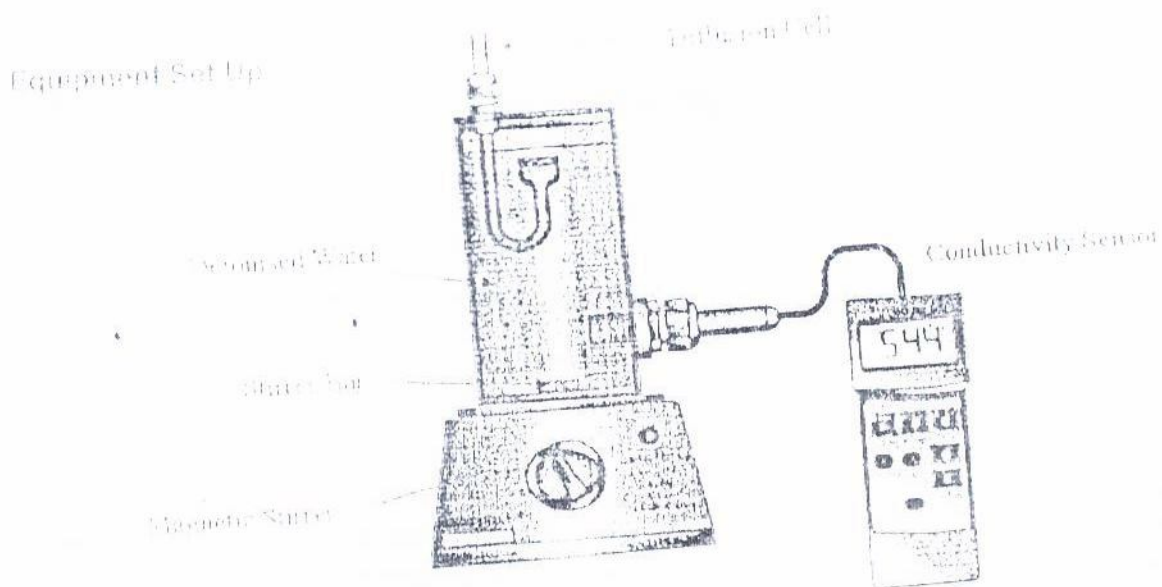
$\frac{dk}{dt}$  = Rate of change of conductivity with time Ω<sup>-1</sup>sec<sup>-1</sup>

By plotting conductivity as a function of time the diffusion can be calculated from the slope of the graph.

$$\eta = \frac{4\pi m}{m^2 n n_2} \approx 10^3$$

Ignoring the sign and correcting for units

## Equipment Set Up



Before using the equipment wash the glass diffusion cell with warm soapy water then rinse with clean water to remove any grease or contamination from the cell/

Check that the conductivity electrode is located centrally in the vessel with the holes in the shield aligned vertically. Make up a 1M solution of NaCl by dissolving 58.44 gm of the salt into 1.0 litres of water (see Making salt solutions for further details).

Fill the acrylic vessel with 1 litre of distilled or deionised water using the measuring jug supplied. Locate the lid (with the glass diffusion cell fitted) on top of the vessel but locate it eccentrically so that it does not locate in the recess and the lid is 5mm above its normal operating position.

Steady the lid on top of the vessel and check that the top of the honeycomb of capillaries is flush with the surface of the water. If necessary adjust the height of the glass diffusion less by unscrewing the gland nut that secure it. After making any



adjustments ensure that the gland nut is tightened to clamp the glass diffusion cell at the correct height.

Remove the lid / diffusion cell and dry the cell to remove any excess water. Check that the collar on the vertical shaft is located 5mm above the tops of the capillaries to assist when filling the cell.

Place the stirrer bar into the bottom of the acrylic vessel and locate the vessel on top of the battery operated stirrer.

Connect the conductivity electrode to the socket at the top of the conductivity meter. If using the CERB data logging software connect the lead supplied from the jack socket on the meter (under a sealing grommet) to the serial port connector on the PC.

Set the range switch to 199.9  $\mu\text{S}$  on the conductivity meter and switch the meter on by pressing the POWER button.

Press the REC button to leave the meter permanently powered (if REC is not indicated on the display the meter will automatically switch off after 10 minutes to save the battery).

Fill the glass diffusion cell with 1M NaCl using the syringe supplied. Ensure that the glass hook and plastic honeycomb are filled with the solution with no air bubbles trapped. This can be achieved by immersing the hook into the solution; while forcing liquid through the capillaries using the syringe filled the same solution, the syringe is fitted with a flexible collar to fit the top of the cell. Wipe off any excess solution from the outside of the cell and the tops of the capillaries using filter paper or similar absorbent material but take care not to suck the salt solution through the capillaries. It is important to adjust the level inside the cell so that the free surface is adjacent to the collar on the vertical limb of the cell i.e. 5mm above the tops of the capillaries. This ensures that the salt solution is not disturbed when the cell is immersed 5mm below the surface of the water in the vessel. Do not place the cell into the vessel until instructed.

## **Procedure**

Ensure that the timer or CERB data logging software is ready for use as appropriate.

Switch on the magnetic stirrer and adjust the speed control until the contents of the diffusion vessel is gently agitated without excessive motion at the surface (The speed must be sufficient to give good mixing within the vessel but not to allow disturbance such as a vortex to form).

Carefully install the lid / diffusion cell on top of the vessel ensuring that it is located squarely into the recess. Take care not to tilt the cell as it is immersed in the water to prevent loss of the salt solution.

Start the stopwatch and record the conductivity reading or start the data logger as appropriate.

If using a stopwatch record the conductivity reading at typically 200 second interval. The experiment should continue for at least 2000 seconds.



If required, the value for the constant  $C_m$  (typically 0.112 when using 1M NaCl) can be confirmed in a separate exercise by measuring the change of conductivity of very dilute solutions of NaCl, typically 0.001M, 0.002M etc. It will be necessary to determine the value of  $C_m$  using this technique if testing different salts.

## Results

Plot a graph of conductivity readings (S) obtained as a function of time (Secs) then determine the slope of the best fit straight line (ignore any non-linearity immediately after immersing the cell as the system settles down). Note that readings recorded by the data logger are displayed in mS and the readings on the conductivity meter are displayed in  $\mu$ S.

Determine the diffusion coefficient using the equation.

$$D = \frac{4Vx}{\mu d^2 NMC_m} \times \text{Slope} \times 10^3$$

When using 1M NaCl the value of  $C_m$  should be taken as 0.112.

If the experiment is performed using a different salt then the value of  $C_m$  must be determined by calibration the apparatus using diluted solutions of known concentration.

## CONCLUSION

The value of the Diffusion Coefficient obtained using the experiment should be compared with the value quoted below. Comment on any difference between the values and the likely cause of any difference.

For 1M NaCl	$D = 1.484 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$
For 0.1M NaCl	$D = 1.483 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$
For 0.01M NaCl	$D = 1.545 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$

Using different salts in the CERB

For 1M NaCl	$D = 1.892 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$
For 0.1M NaCl	$D = 1.844 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$
For 0.01M NaCl	$D = 1.917 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$

For 1M NaCl <sub>2</sub>	$D = 1.203 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$
For 0.1M NaCl <sub>2</sub>	$D = 1.110 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$
For 0.01M NaCl <sub>2</sub>	$D = 1.188 \times 10^{-5} \text{ cm}^2 \text{ sec}^{-1}$

## PERSONAL NOTES/OBSERVATIONS:

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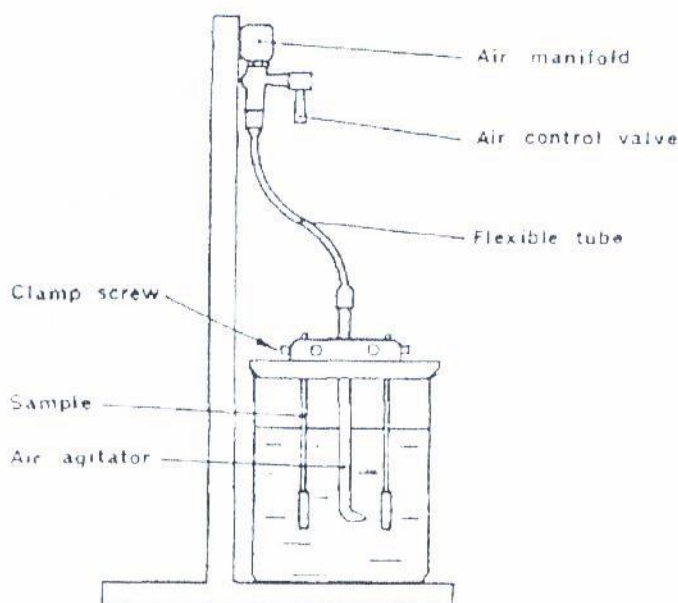
## EXPERIMENT 3

### DETERMINATION OF THE INFLUENCE OF pH ON CORROSION

**Objective:** To examine the effect of pH on the rate of corrosion of steel.

#### Equipment Set-up

Equipment Set-up



#### Theory

Iron and steel corrode in acid environments but very little corrosion occurs in alkaline situations. This behavior can be explained by reference to the electromotive series in which elements are arranged in order of the decreasing negative electrode potential when the element is immersed in an acid solution. Metals such as iron above hydrogen (more electro-negative) will displace it from solution and hence corrode, metals below hydrogen will not. An increase in the hydrogen ion concentration (more acid) will tend to move hydrogen down the series and hence increase the voltage in the metal –  $H^+$  system which will lead to higher corrosion rates.

#### Procedure

Weigh the steel samples and clamp three symmetrically around the bubbler in each of three lids of test vessels. Fill the test vessels with water about two thirds full and make the pH values 4, 7 and 9 with the buffer powders checking each with the pH meter.



After 3 – 7 days of continuous running, switch off the compressor, remove and completely dry the samples then weigh them.

## Results

PERSONAL NOTES/OBSERVATIONS:

[illegible]

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## EXPERIMENT 4

### DISTRIBUTION COEFFICIENT

**AIM:** To determine the distribution coefficient for the system trichloroethylene –propionic Acid water and to show its dependence on concentration.

#### EQUIPMENT SET-UP

The following apparatus is required:

- 250ml Conical stoppered flask
- 250ml Measuring cylinder
- 250ml Separating funnel
- Burette
- Sodium Hydroxide Solution (M/2)
- Phenolphthalein

#### SUMMARY OF THEORY

The solvent (water) and solution (trichloroethylene /propionic acid) are mixed together and then allowed to separate into the extract phase and the raffinate phase. The extract phase will be water and propionic acid and the raffinate, trichloroethylene with a trace of propionic acid.

The distribution coefficient,  $K$ , is defined as the ratio

$$\frac{(\text{Concentration of solute in the extract phase}, Y)}{(\text{Concentration of solute in the raffinate to phase}, X)}$$

It is assumed that equilibrium exists between the two phases. At low concentrations, the distribution coefficient is dependent on the concentration and thus  $Y = kX$ .

#### READINGS TO BE TAKEN

1. Make up in a conical flask a mixture of 50ml trichloroethylene and 50ml of demineralised water.
2. Add 5g of propionic acid. 5ml can be pipette into the flask using a pipette with a rubber bulb ( $\rho = 0.994 \text{ g/ml}$ ).
3. Place a stopper into the flask and shake for a minimum of 5 minutes.
4. Pour into a separating funnel and remove the lower aqueous layer.
5. Take a 10ml sample of this layer and titrate against M/2 sodium hydroxide solution using phenolphthalein as indicator.
6. Repeat the experiment for two further concentrations of propionic acid i.e. for initial additions of 2ml and 1ml of propionic acid.



**RESULTS:**

PROPIONIC ACID ADDED (ML)	TITRE OF M/2NAOH	PROPIONIC ACID CONCENTRATION IN AQUEOUS LAYER, Y	PROPIONIC ACID CONCENTRATION IN ORGANIC LAYER, X	$k = \frac{Y}{X}$
4				
2				
1				

PERSONAL NOTES/OBSERVATIONS:

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## EXPERIMENT 5

### APPLICATION OF HALF – LIFE TIME METHOD FOR ANALYSIS OF KINETIC LABORATORY DATA

#### INTRODUCTION

Half-life time method is one of the integral method of analysis of laboratory data. This method is applied for irreversible reaction and for such reactions in which reactants are present and remain in stoichiometric ratio throughout the reaction. The half-life method requires making a series of runs, each at a different initial concentration.

For the irreversible reaction of the type:



we may write:

$$-r_A = -\frac{dc_A}{dt} = K C_A C_B$$

if the reactants are present in their stoichiometric ratios

$$\left( \frac{C_{B0}}{C_{A0}} \right) \neq \frac{\beta}{\alpha}$$

They will remain at that ratio throughout the reaction

$$\left( \frac{C_B}{C_A} \right) \neq \frac{\beta}{\alpha}$$

Thus for reactants A and B we may write at any time:

$$-r_A = -\frac{dc_A}{dt} = K C_A^\alpha \left( \frac{\beta}{\alpha} C_A \right)^b = \dots$$

$$K \left( \frac{\beta}{\alpha} \right)^b \dots C_A^{\alpha + \beta} \dots$$

$$\text{Or } -\frac{dc_A}{dt} = K' C_A^n \quad (\text{for } \alpha = \beta, K' = K)$$

Integrating for  $n > 1$  gives

$$C_A^{1-n} - C_A^{1-n} = K'(n-1)t$$

Defining the half-life of the reaction,  $t_{1/2}$  as the time needed for the concentration of reactants to drop to one-half the original value we obtain:

$$t_{1/2} = \frac{2^{n-1} - 1}{K'(n-1)} C_{A0}^{1-n}$$

This expression shows that a plot of  $\log t_{1/2}$  versus  $\log C_{A0}$  gives a straight line of slope  $(1 - n)$ .

**Apparatus:** Magnetic stirrer, conical flask  $1\text{dm}^3$ , conical flask  $0.2\text{dm}^3$

## PROCEDURE

Prepare  $0.25\text{dm}^3$  of  $0.04\text{M}$  NaOH and pour it into  $1\text{dm}^3$  conical flask. Take  $5\text{cm}^3$  of solution from the flask and check concentration of NaOH in the solution using  $0.02\text{N}$   $\text{H}_2\text{SO}_4$  or  $0.01\text{M}$  HCl for titration.

Put the conical flask on a plate of magnetic stirrer and start low speed mixing. Prepare  $0.25\text{dm}^3$  of  $0.04\text{M}$  ester ( $\text{CH}_3\text{COOC}_2\text{H}_5$ ) and add it to the reactor. Start measuring time since the moment of adding ester and take  $10\text{cm}^3$  sample from the reactor after 2 minutes ( $t_2$ ) and then at  $t_5$ ,  $t_{10}$ ,  $t_{15}$ ,  $t_{20}$ ,  $t_{25}$ ,  $t_{30}$ , dilute sample with a  $100\text{cm}^3$  of distilled water and titrate immediately against  $0.01\text{M}$  HCl or  $0.02\text{N}$   $\text{H}_2\text{SO}_4$  (with phenolphthalein as indicator) to get concentration of NaOH in the reactor at  $t_2$ ,  $t_5$ ,  $t_{10}$ , .....

Measure temperature of the solution in the reactor at the beginning and at the end of the reaction. Repeat the experiment with the following concentrations of NaOH and ester in the reactor.

$C_{A0} = B_0 = 0.3; 0.4; 0.5; 0.6\text{mol}/\text{dm}^3$ .

From the obtained results calculate rate constant and order of the reaction by means of half-life method.

$C_{A0} = B_0 = \dots\dots\dots\text{mol}/\text{dm}^3$ .

Time (min)	$t_2$	$t_5$	$t_{10}$	$t_{15}$	$t_{20}$	$t_{25}$	$t_{30}$
$C_A = C_B\text{mol}/\text{dm}^3$							
$t_{1/2} =$							

## PERSONAL NOTES/OBSERVATIONS:

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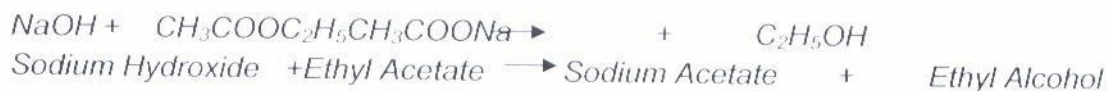
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## EXPERIMENT 6

### EXERCISE E – DEMONSTRATION OF THE PROGRESS OF A SECOND ORDER CHEMICAL REACTION THROUGH THREE CONTINUOUS STIRRED TANK REACTORS CONNECTED IN SERIES.

#### THEORY:

The reaction:



Can be considered equi-molar and first order with respect to both Sodium Hydroxide and Ethyl Acetate i.e. second order overall, within the limits of concentration (0- 0.1M) and temperature (20 – 40<sup>0</sup>C) studied.

The reaction carried out in a continuous stirred tank reactor eventually reaches steady state when a certain amount of conversion of the starting reagents has taken place.

The steady state conditions will vary depending on concentration of reagents, flowrate, volume of reactor and temperature of reaction.

#### Method:

Make up 5.0 litre batches of 0.05M Sodium Hydroxide and 0.05M Ethyl Acetate.

**Important :**It is essential when handling these chemicals to wear protective clothing, gloves and safety spectacles.

Remove the lids of the reagent vessels and carefully fill with the reagents to a level approximately 50mm from the top. Refit the lids.

Collection of conductivity data will be until a steady state condition is reached in the reactor and this takes approximately 45 minutes. It is advisable to set the data collection period to, say, 60 minutes.

Using the calibration graph for each of the feed pumps, set the pump speed controls to give 60 ml/min flowrate.

Set the agitator speed controller to 7.0

Switch on both feed pumps and agitator motor, and instigate the data logger program (or begin taking readings if no computer is being used).

It has been determined that the degree of conversion of the reagents affects the conductivity of the reactor contents so that recording the conductivity with respect to time using the Armfield data logger can be used to calculate the amount of conversion.

## INTERPRETATION OF RESULTS

Having used the Armfield data logger CEX-304IFD to record the conductivity of the contents of the reactors over the period of the reaction, the conductivity measurements are automatically translated into degree of conversion of the constituents.

Both sodium hydroxide and sodium acetate contribute conductance to the reaction solution whilst ethyl acetate and ethyl alcohol do not. The conductivity of a sodium hydroxide solution at a given concentration and temperature however, is not the same as that of a sodium acetate solution at the same molarity and temperature and a relationship has been established allowing conversion to be inferred from conductivity.

### Comment upon the results obtained

If readings have been obtained manually then the necessary calculations are best carried out using a spreadsheet such as EXCEL so that the results can be displayed in tabular and graphical form. On conclusion of the experiment the recorded data can be transferred into the spreadsheet. Enter the following known constants from the experiment using the nomenclature below. Ensure use of correct units.

$F_a$  =

$F_b$  =

$a_\mu$  =

$b_\mu$  =

$C_\mu$  =

$T$  =

$V$  =

Using the spreadsheet, calculate the values of

$\Lambda_0$ ,  $b_0$ ,  $c_\infty$ ,  $a_\infty$ ,  $\Lambda_{C^\infty}$ ,  $\Lambda_{a0}$ ,  $\Lambda_{a^\infty}$ ,  $\Lambda_0$ , and  $\Lambda_\infty$ , from the following formulae:

$$a_0 = \frac{F_b}{F_a + F_b} a_\mu$$

$$b_0 = \frac{F_b}{F_a + F_b} b_\mu$$

$$c_\infty = b_0$$

$$c_\infty = a_0$$

$$\Lambda_{C^\infty} = 0.070[1 + 0.0284 (T - 294)] c_\infty$$

$$\Lambda_{a0} = 0.195[1 + 0.0184 (T - 294)] a_0$$

$$\Lambda_0 = \Lambda_{a0}$$

$$a_\infty = 0$$

$$a_\infty = (a_0 - b_0)$$

$$\Lambda_{a^\infty} = 0.195[1 + 0.0184 (T - 294)] a_\infty$$

$$\Lambda_\infty = \Lambda_{C^\infty} + \Lambda_{a^\infty}$$

for  $b_0 < a_0$

for  $b_0 \geq a_0$

assumes  $c_0 = 0$

for  $a_0 < b_0$

for  $a_0 \geq b_0$

if  $a_\infty \neq 0$



These can be calculated and listed in columns (use spreadsheet COPY facility) alongside the readings of conductivity using the following equations:

$$\begin{aligned} a_1 &= (a_\infty - a_o) \left[ \frac{\Lambda_o - \Lambda_1}{\Lambda_o - \Lambda_\infty} \right] + a_o \\ C_1 &= C_\infty \left[ \frac{\Lambda_o - \Lambda_1}{\Lambda_o - \Lambda_\infty} \right] \quad \text{for } C_o = 0 \\ X_o &= \frac{a_o - a_1}{a_o} \\ X_c &= \frac{c_1}{C_\infty} \text{ for } C_o = 0 \end{aligned}$$

The overall mass balance at steady-state condition may be written as:

i.e. for a reactant "a" in a reactor of volume V

$$\frac{d(Va_1)}{dt} = F - a_0 F - a_1 - V \cdot K \cdot a_1^2$$

$$k = \frac{F}{V} \cdot \frac{a_o - a_1}{a_1^2} = \frac{(F_a + F_b) (a_o - a_1)}{V a_1^2} \text{mol/dm}^3 \text{ sec}$$

**PERSONAL NOTES/OBSERVATIONS:**

[illegible]

[illegible]

## EXPERIMENT 7

### HEAT OF SOLUTION

**OBJECTIVE:** Determine the heat of solution of benzoic acid in water from solubility measurements

**THEORY:** The solubility of a solid which forms an ideal solution with liquid related to its heat of solution by the Van't – Hoff isochore equation;

$$\frac{dX}{dT} = \frac{\Delta H}{RT^2}$$

Where

X	=	solubility of solid in liquid mole fraction
$\Delta H$	=	heat of solution of solid in liquid Cal/g mole
R	=	Gas constant, Cal/(g mole K)
T	=	Temperature K

If the heat of solution can be assumed constant over a temperature range, the equation can be integrated to give,

$$\ln \frac{X_1}{X_2} = \frac{-\Delta H}{R} \left[ \frac{1}{T_2} - \frac{1}{T_1} \right]$$

Hence, the heat of solution can be obtained by determining the solubility of the solid at different temperatures.

**Experiment:** Heat 200ml distilled water with about 2g benzoic acid to obtain a saturated solution. Place this solution in the thermostat water bath to bring it to a desired temperature. Withdraw three 25ml sample and titrate each with 0.1N NaOH using phenolphthalein as indicator.

In withdrawing samples, care is needed to avoid either drawing up particles of solid or allowing crystallization of the acid in the cold pipette. A warm pipette with a cotton wool filter should be used. Repeat the whole procedure at another temperature, suitable temperatures should be chosen between 25°C and 40°C. All alternative method is to decant some of the benzoic acid solution into a conical flask and determine the quantity before titrating by weighing. Try this and compare the results with the first method.

#### PERSONAL NOTES/OBSERVATIONS:

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Lined area for student response, consisting of multiple horizontal dotted lines.

