



**UNIVERSITY OF PETROLEUM & ENERGY STUDIES**

**PRACTICAL MANUAL**

**CHEMISTRY LAB**

**B.TECH. (I Year)**

**DEPARTMENT OF CHEMISTRY**

**SCHOOL OF ENGINEERING**

### **LIST OF EXPERIMENTS**

1. To determine the strength of given solution of NaOH by titrating it against standard oxalic acid solution using phenolphthalein
2. To determine the percentage of moisture, volatile matter, ash content and fixed carbon in a given coal sample by proximate analysis
3. To estimate sulphur as barium sulphate in a given solution of sodium sulphate
4. To determine the rate constant and order of the reaction of the hydrolysis of an ester (methyl acetate) at 25<sup>o</sup> C in the presence of 0.5N hydrochloric acid
5. To determine the strength of given HCl solution by titrating it against standard NaOH solution conductometrically
6. To determine the strength of the given solution pH-metrically
7. To determine the Total hardness of the given hard water sample by EDTA method
8. To determine the alkalinity of a given water sample
9. To prepare Urea-Formaldehyde resin
10. To prepare of Phenol-Formaldehyde resin
11. To prepare N/30 ferrous ammonium sulphate solution and to determine the strength of given ferrous ammonium sulphate solution using potassium dichromate as intermediate solution and diphenyl amine as internal indicator
12. To study the adsorption of acetic acid on activated charcoal
13. To determine the molecular weight of the given non-volatile compound cryoscopically
14. To prepare aniline- $\beta$ -naphthol azo dye from aniline
15. Virtual Lab – I. <http://vlab.amrita.edu/?sub=2&brch=193&sim=352&cnt=4>
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## EXPERIMENT-01

### OBJECTIVE:

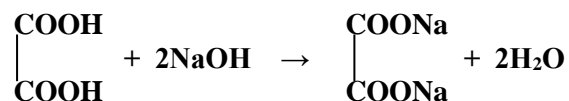
To determine the strength of given solution of NaOH by titrating it against standard oxalic acid solution using phenolphthalein.

### APPARATUS/REAGENTS REQUIRED:

Burette, pipette, conical flask, given NaOH solution, standard oxalic acid solution and phenolphthalein.

### THEORY:

NaOH is titrated against a standard solution of oxalic acid leading to the following reaction:



Phenolphthalein is used as indicator giving a colorless solution at the end point.

### PROCEDURE:

Pipette out 10ml of given NaOH solution into the conical (titration) flask. To this add 1 – 2 drops of phenolphthalein indicator. A pink color is developed in the flask. Now titrate it against standard oxalic acid solution (filled in burette). Continue adding of oxalic acid solution drop by drop till the color of the solution just disappears permanently. Note down the volume of oxalic acid used ( $V_1$ ml). Repeat the titration to get at least two concordant readings.

### OBSERVATIONS:

Volume of oxalic acid used =  $V_1$  ml

Volume of NaOH solution in each titration = 10 ml

Normality of oxalic acid solution = N/20

### CALCULATIONS:

Applying normality equation:  $N_1V_1 = N_2V_2$

Strength of NaOH = Normality  $\times$  Equivalent weight

### RESULT:

Strength of NaOH solution = ----- g/l.

### PRECAUTIONS:

1. Titration is carried out at room temperature.
2. All the volumetric apparatus should be washed with distilled water before use.
3. Rinse the burette with a solution of oxalic acid to be taken in the burette.
4. Wash the conical flask with distilled water after every titration.
5. Oxalic acid solution should be added to the conical flask drop wise with constant stirring.

## EXPERIMENT-02

### OBJECTIVE:

To determine the percentage of moisture, volatile matter, ash content and fixed carbon in a given coal sample by proximate analysis.

### REQUIREMENTS:

Powdered coal, analytical balance, electric oven, desiccator, silica crucible, tong, Muffle furnace and given coal sample.

### THEORY:

Proximate analysis is a type of assay used in the determination of different constituents present in the coal sample.

1. **Moisture:** In coal sample, moisture may be of two types:
  - (i) Free or surface moisture, which is lost on air drying.
  - (ii) Internal moisture, which is retained by air dried coal.
2. **Volatile matter:** It consists of a complex mixture of gaseous and liquid products resulting from the thermal decomposition of coal substances.
3. **Ash content:** It is the incombustible content left after burning of organic matter from the coal. It may again be of two types:
  - (i) Fixed or inherent ash, intimately dispersed within the mass of the coal.
  - (ii) Free or extraneous ash, occurring in different layers of coal.

### PROCEDURE:

1. **Determination of inherent moisture:** 1gm of air dried coal sample is taken in the preweighed silica crucible. It is kept inside the electric oven at 105 - 110°C for an hour. It is cooled in the dessicator and weighed. The difference in weight is reported in percentage as the amount of inherent moisture in coal.
2. **Volatile matter:** The dried coal sample after determining moisture content is heated in a muffle furnace at about 925°C for exactly 7 min. The coal sample is taken in a crucible and heated with a lid. It is cooled in the dessicator and weighed. Loss in weight gives the percentage volatile matter.
3. **Ash:** The residual coal in the crucible after the determination of volatile matter in it, is heated without lid in the muffle furnace at about 725°C for half an hour for constant weight. It is cooled in dessicator. The amount of unburnt carbon residue in crucible is ash which can be reported in percentage.

### OBSERVATIONS AND CALCULATIONS:

#### 1. Moisture:

Weight of empty crucible =  $w_1$  g

Weight of crucible + sample =  $w_2$  g

Weight of sample =  $(w_2 - w_1)$  g

Weight of (crucible + sample) after heating at 110°C =  $w_3$  g

Moisture contents =  $(w_2 - w_3)$  g

$$\% \text{ moisture} = \frac{w_2 - w_3}{w_2 - w_1} \times 100$$

#### 2. Volatile matter:

Weight of (crucible + sample) after heating at 925°C for 7 min =  $w_4$  g

$$\text{Volatile matter} = (w_3 - w_4) \text{ g}$$

$$\% \text{ of volatile matter} = \frac{w_3 - w_4}{w_2 - w_1} \times 100$$

### 3.Ash:

$$\text{Weight of (crucible + ash)} = w_5 \text{ g}$$

$$\text{Ash contents} = (w_5 - w_1) \text{ g}$$

$$\% \text{ ash} = \frac{w_5 - w_1}{w_2 - w_1} \times 100$$

### RESULT:

The given coal sample contains:

- (i) Moisture.....%
- (ii) Volatile matter.....%
- (iii) Ash.....%

### PRECAUTIONS:

1. To avoid mechanical loss due to rapid repulsion of volatile matter, the temperature should be raised at a slow rate.
2. After or before weighing the crucible, it should not be held with fingers. The tong should be used for putting the crucible inside the oven / furnace.
3. The weighings should be made up to fourth decimal places.

## EXPERIMENT-03

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### OBJECTIVE:

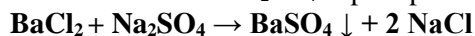
To estimate sulphur as barium sulphate in a given solution of sodium sulphate.

### REQUIREMENTS:

BaCl<sub>2</sub> solution, Na<sub>2</sub>SO<sub>4</sub> solution, beaker, silica crucible, desiccator, weight box.

### THEORY:

The solution of BaCl<sub>2</sub> on treatment with hot Dil. H<sub>2</sub>SO<sub>4</sub> is precipitated in the form of BaSO<sub>4</sub>.



### PROCEDURE:

Dilute the given solution of Na<sub>2</sub>SO<sub>4</sub> by adding about 100 ml of distilled water. Heat the solution to about 60 - 70°C and precipitate it by adding hot BaCl<sub>2</sub> solution to it. Heat the solution using a wire gauze. Now, replace the beaker from the burner and let it settle down. Check for complete precipitation. Now filter the solution by decantation method. Check the filtrate for further precipitation. Wash the precipitates with hot water. Dry the precipitates placed in the funnel over an iron cone or in oven. After complete drying, ignite the precipitate along with the filter paper in a pre-constantly weighed silica crucible. Heat the crucible to red heat. Cool it in desiccator and weigh it. Proceed for constant weighing.

### OBSERVATIONS:

Weight of empty crucible = x gm

Weight of crucible + precipitate = y gm

Weight of precipitate = (y - x) gm

### RESULT:

The given solution of Na<sub>2</sub>SO<sub>4</sub> contains ..... grams of BaSO<sub>4</sub>.

### PRECAUTIONS:

1. Precipitation should be carried out in hot and dilute solution.
2. The precipitated solution should be digested properly.
3. During digestion:
  - (i) glass rod must be inside the beaker.
  - (ii) beaker must be covered with beaker.
  - (iii) there must be no bumping in the solution.
4. Filtration must be done by decantation method.
5. During ignition there must be no loss of precipitates.
6. Red - hot crucible must not be placed directly in the desiccator.
7. Only Whatmann filter paper (No. 40, 41 or 42) should be used for filtration.

## EXPERIMENT-04

### OBJECTIVE:

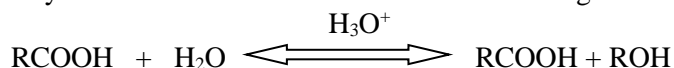
To determine the rate constant and order of the reaction of the hydrolysis of an ester (methyl acetate) at 25° C in the presence of 0.5N hydrochloric acid.

### REQUIREMENT:

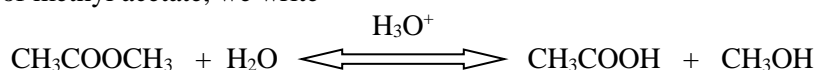
Pipette, conical flask, ice, stop watch, burette, water bath, methyl acetate, HCl, NaOH, phenolphthalein indicator.

### THEORY:

The hydrolysis of an ester in acidic medium follows the general equation:



In case of methyl acetate, we write



Since water is to be taken in large excess, its concentration remains practically constant during the hydrolysis. The rate equation for the above reaction has been found to be:

$$R = K [\text{CH}_3\text{COOCH}_3]$$

It is a first order reaction but its molecularity is 2, and it is therefore, is called pseudo unimolecular reaction. Since hydrolysis produces acetic acid, the rate of the reaction can be studied by titrating the acid formed against a standard solution of an alkali, say NaOH. The reaction can be stopped at any instance by lowering temperature of the reaction zone, using the ice. Its rate constant, K is calculated using integrated rate equation of the first order

$$K = \frac{2.303}{t} \log_{10} \frac{V_{\infty} - V_0}{V_{\infty} - V_t}$$

$V_0$  = Titration reading in the beginning

$V_t$  = Titration reading after time 't'

$V_{\infty}$  = Titration reading after infinite time (~ 48 hrs)

### PROCEDURE:

- (i) Transfer 100 ml of 0.5 N HCl into one conical flask and 15 ml of methyl acetate into another and place these in a water bath.
- (ii) Now fill the burette with 0.1 N NaOH and keep phenolphthalein indicator nearby for ready use.
- (iii) When the acid and the ester have attained a steady temperature of the bath, pipette 5 ml of the ester and transfer nearly half of it to the acid kept in water bath and start the stop watch. Now transfer remaining as well and shake well the contents and then immediately, pipette 5 ml of the reaction mixture and transfer into a flask containing ice cold water. This will stop the reaction due to low temperature. Add few drops of phenolphthalein in it and titrate it quickly against NaOH solution. Note the end point (Shown by appearance of pink colour). This reading is called  $V_0$  as the time is zero. Titration should be completed in five minutes.
- (iv) Now when the stop watch shows 10 minutes, take out again 5 ml from the reaction mixture to another ice cooled conical flask and titrate again quickly. Note the burette reading. It is  $V_t$  as  $t=10$  minutes. Now this is to be repeated after 20, 30 and 40 minutes. These readings will give the values of  $V_t$  at different intervals. Now heat the water bath to ~ 60° C for about an hour. This will heat



the reaction mixture to complete the hydrolysis. Take 5ml of it and titrate this also against the NaOH solution n. This reading is  $V_{\infty}$ . Heating has reduced the time for completion of the reaction.

### OBSERVATIONS & CALCULATIONS:

Temperature during the experiments: .....

Time in mins	Volume of NaOH used in titration (ml)	$(V_{\infty}-V_t)$ ml	K
0	$V_0$	$V_{\infty}-V_0$	
10	$V_{t1}$	$V_{\infty}-V_{t1}$	
20	$V_{t2}$	$V_{\infty}-V_{t2}$	
30	$V_{t3}$	$V_{\infty}-V_{t3}$	
40	$V_{t4}$	$V_{\infty}-V_{t4}$	
$\infty$	$V_{\infty}$	$V_{\infty}-V_{t4}$	

### RESULT:

- i) Order of the reaction .....
- ii) Rate constant .....

### PRECAUTIONS:

- i) All the glass apparatus must be clean.
- ii) All solution must be prepared in the deionized water.
- iii) Temperature of the bath must not vary beyond  $\pm 0.5^{\circ}\text{C}$
- iv) The reaction must be immediately arrested by adding ice cold water in the part of reaction mixture taken out.

## EXPERIMENT-05

### OBJECTIVE:

To determine the strength of given HCl solution by titrating it against standard NaOH solution conductometrically.

### REAGENTS AND APPARATUS USED:

Conductometer, conductivity cell, conductivity bridge, standard NaOH solution, unknown acid solution.

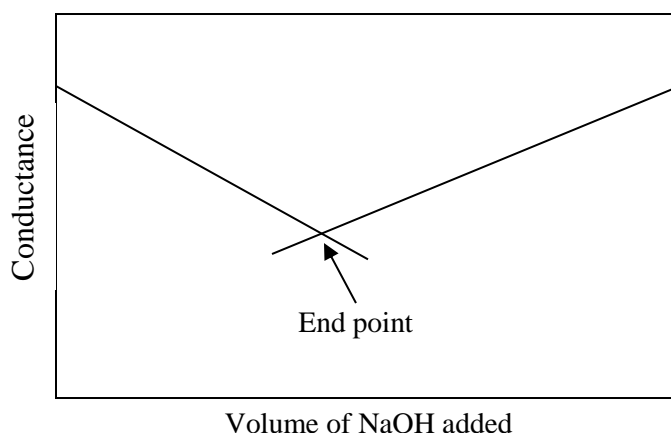
### THEORY:

During ionic reactions, the conductance may either increase or decrease depending upon the nature of the electrolyte involved.

In the titration of unknown HCl against standard NaOH solution, HCl is taken in the conductivity vessel and NaOH is dropwise added from the burette into this solution. Initially, the conductance of the solution is due to the presence of  $\text{H}^+$  ions and  $\text{Cl}^-$  ions. As NaOH is gradually added,  $\text{H}^+$  ions by combining with  $\text{OH}^-$  will form unionized water.



Therefore, on adding more of NaOH, the conductance will go on decrease until whole of the acid has been neutralized by the base. Further addition of NaOH will increase the conductance value.



**Figure 1. Conductometric titration of HCl vs NaOH**

### PROCEDURE:

The calibration of the conductometer is done by using standard KCl solution. Afterwards, 50 ml of the given acid solution is taken in a beaker and diluted with 50 ml water so that the electrodes of the cell are completely dipped in the solution. Now, connect the conductivity bridge and note the conductance of the solution. Run NaOH from burette in the acid solution and note the conductivity of the resulting solution after each addition of 0.5 ml of NaOH till almost a constant value of conductance is not obtained.

Plot the graph of conductance vs amount of NaOH added gradually.

**OBSERVATIONS:**

Volume of HCl used = 50 ml.

S.No.	Volume of NaOH added (ml)	Conductance of the solution (ohm <sup>-1</sup> )	Conductance with correction factor
1.	0.0		
2.	0.5		
3.	1.0		
4.	1.5		
5.	2.0		
6.	2.5		
7.	3.0		
8.	3.5		
9.	4.0		
10.	4.5		
11.	etc.		

**CALCULATION:**

Applying normality equation:

$$N_1 V_1 = N_2 V_2$$

Where,  $N_1 \rightarrow$  Normality of acid

$V_1 \rightarrow$  Volume of acid used

$N_2 \rightarrow$  Normality of NaOH

$V_2 \rightarrow$  Volume of NaOH required to reach the end point.

$$\text{Strength of HCl} = \text{Normality} \times \text{Equivalent weight}$$

**RESULT:**

Strength of HCl solution = ----- g/lit.

**PRECAUTIONS:**

1. Stirring should be done after each addition of titrant.
2. All precautions regarding handling of the instrument should be observed.
3. Cell must properly be washed before using.

## EXPERIMENT-06

### OBJECTIVE:

To determine the strength of the given solution pH-metrically.

### REQUIREMENTS:

pH-meter, glass rod, reference electrode, beaker, burette, stirrer, given acid solution, standard alkali solution, buffers of pH 4.0 and 9.2.

### THEORY:

When an alkali is added to an acid solution, the pH of the solution increases slowly, but at the vicinity of the equivalence point, the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find the equivalence point, from which the strength can be calculated.

### PROCEDURE:

The standardization of the pH-meter is done by using a buffer of known pH.

Afterwards, wash the glass electrode with distilled water and then with the given acid solution. Take 50 ml of given acid solution in a 400 ml beaker. Add 50 ml of water so that ions are separated and the glass electrode is completely dipped. Note the pH of pure acid. Now add 1 ml of standard NaOH from the burette in the beaker. Stir the contents well. Note the pH of the solution. Now go on adding NaOH solution from the burette and note the pH of the solution till a constant value of pH is not obtained.

### OBSERVATIONS:

Table for added NaOH:

Volume of alkali added (ml)	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0	9.0	10.0
pH										

### CALCULATIONS:

A curve is plotted in pH vs amount of alkali added. Find out the end point.

Let, it comes to be equivalent to x ml standard alkali.

$$N_1 V_1 = N_2 V_2$$

(Acid) (Alkali)

or,

$$N_1 = \frac{N_2 V_2}{V_1}$$

$$\text{Strength of acid solution} = N_1 \times \text{Equivalent Weight}$$

### RESULT:

The strength of given acid solution is .....g/l.

### PRECAUTIONS:

1. The temperature knob of the pH-meter should be adjusted at room temperature.
2. After the addition of the alkali, the solution should be thoroughly stirred.
3. The pH-meter should be first standardized by taking a buffer of known pH.

## EXPERIMENT-07

### OBJECTIVE:

To determine the Total hardness of the given hard water sample by EDTA method. You are provided with Standard Hard Water (1 ml of SHW = 1 mg of  $\text{CaCO}_3$ ).

### EQUIPMENTS AND APPARATUES REQUIRED:

Burette, Burette stand, Titration flask, Pipette, Beakers, Funnel etc.

### MATERIALS REQUIRED:

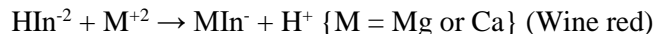
Standard hard water, EDTA solution, Eriochrome Black-T indicator (EBT), Ammonia buffer solution, Sample hard water.

### THEORY:

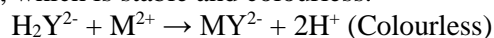
Water hardness is expressed in mg/l or ppm (parts per million). Complexometric titrations are mainly used to determine the concentration of divalent cations such as calcium, magnesium, zinc, copper, lead etc. Hard water is the water that has high concentration of calcium and magnesium ions. The Ethylenediaminetetraacetic acid (EDTA) is the most commonly used complexant. EDTA is a chelating agent. It is able to form coordination complex with metal ions present in hard water. It is necessary to maintain well defined pH to perform complexometric reaction. The buffer solution has the composition of  $\text{NH}_4\text{Cl}$  and  $\text{NH}_4\text{OH}$  is used to maintain pH value of about 10.5. This pH value is crucial for two reasons; (a) Below pH 10.5, the principal species responsible for divalent cation binding of EDTA is not  $\text{Y}^{4-}$  and therefore no complete complexation takes place and (b) the eriochrome black T indicator requires a pH of 9 to 10.5 for the desired color change.

How the indicator works:

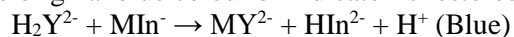
(i) When indicator is added to hard water it combines with free metal ions present in water.



(ii) When EDTA solution is added to the titration flask it combines with the free metal ions giving metal EDTA complex, which is stable and colourless.



(iii) When all the free metal ions are exhausted, next drop of EDTA removes the metal ion engaged with indicator and the original blue colour of indicator is restored.



### PROCEDURE:

(a) Standardisation of EDTA solution:

1. Pipette out 10ml of standard hard water in the titration flask. Add to it 2-3ml of buffer solution and two drops of Eriochrome Black-T indicator. A wine red color appears.
2. Titrate this solution against EDTA solution taken in a burette till wine red colour changes to blue color. This is the end point.
3. Recovered the volume of EDTA consumed as A ml. Repeated the procedure to get at least three concordant readings

(b) Determination of Total Hardness:-

1. Pipette out 10ml of sample hard water in the titration flask. Add to it 2-3ml of buffer solution and two drops of Eriochrome Black-T indicator.
2. A wine red colour appears. Titrate this solution against EDTA solution taken in a burette till wine red colour changes to blue color. This is the end point.
3. Recovered the volume of EDTA consumed as B ml. Repeated the procedure to get at least three concordant readings

**CALCULATIONS:** (a) Calculate normality of standard hard water (N<sub>2</sub>) Strength = Normality × equivalent weight of CaCO<sub>3</sub> (b) Standardization of EDTA solution:

Find out the exact normality of EDTA solution (N<sub>1</sub>) by applying normality equation using standard hard water:  $N_1V_1 = N_2V_2$  (EDTA) (Standard Hard Water)

(b) Calculation of total hardness:

Volume of EDTA (for sample hard water) = B ml Normality of EDTA = N<sub>1</sub>

Equivalent weight of CaCO<sub>3</sub> = 50 Volume of sample water = 10ml

**RESULT:** The total hardness of water is=\_\_\_\_\_mg/L as CaCO<sub>3</sub> equivalent.

The determination of water hardness is a useful test that provides a measure of quality of water for households and industrial uses. Originally, water hardness was defined as the measure of the capacity of the water to precipitate soap.

**PRECAUTIONS:**

- i. The burette, pipette and conical flask should be washed and then rinsed with distilled water.
- ii. Redistilled water should be employed for preparing the EDTA solution.
- iii. The colour change near the end point is very slow and thus should be observed very carefully.

## EXPERIMENT-08

### OBJECTIVE:

To determine the alkalinity of a given water sample

### EQUIPMENT AND APPARATUS REQUIRED:

Burette, Burette stand, Titration flask, Pipette, Beakers, Funnel etc.

### MATERIAL REQUIRED:

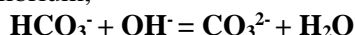
Phenolphthalein, Methyl Orange indicator,  $\text{H}_2\text{SO}_4$ , alkaline water sample

### THEORY:

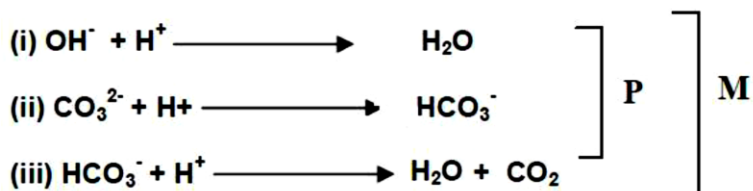
Alkalinity is the measure of ability of water to neutralize added acid. The alkalinity of a water sample is due to carbonates ( $\text{CO}_3^{2-}$ ), bicarbonates ( $\text{HCO}_3^-$ ) and hydroxide ( $\text{OH}^-$ ) ions. Thus in a given water sample the possible combination of ions causing alkalinity are as follows.

- (i)  $\text{OH}^-$  alone
- (ii)  $\text{CO}_3^{2-}$  alone
- (iii)  $\text{HCO}_3^-$  alone
- (iv)  $\text{OH}^-$  and  $\text{CO}_3^{2-}$  together
- (v)  $\text{CO}_3^{2-}$  and  $\text{HCO}_3^-$  together

The possibility of  $\text{OH}^-$  and  $\text{HCO}_3^-$  ions together in the same solution is ruled out as they react to establish the following neutralization equilibrium;



To understand the basis of two consecutive titrations at different pH range:  $\text{OH}^-$ ,  $\text{CO}_3^{2-}$  (caustic alkalinity) and  $\text{HCO}_3^-$  can be estimated separately by titration against standard acid using phenolphthalein and methyl orange as indicators. The determination is based on the following reactions:



Thus titration of a given water sample in presence of phenolphthalein (pink color above pH 8.5 and below it become colourless) as an indicator indicates completion of reaction 1 and 2 whereas the same water sample, if titrated in presence of Methyl orange (yellow color above pH 4.5 and sharply changes to orange-red below pH 3.1) as an indicator indicates the completion of reaction 1, 2 and 3. v. The water sample, when titrated with an acid solution using phenolphthalein indicator gives End point = P; as ml volume of titrant for phenolphthalein indicator and with methyl orange indicator gives End point = M; as ml volume of titrant for methyl orange indicator. The relation between P and M points gives the type and extent of alkalinity is established as follows:

Sr No	Result of titration	Hydroxide ion	carbonate ion	bicarbonate ion
1	P = 0	Nil	Nil	M
2	P = M	P or M	Nil	Nil
3	P=1/2M(V1 = V2)	Nil	2P	Nil
4	P>1/2M(V1 > V2)	2P-M	2(M-P)	Nil
5	P<1/2M(V1 < V2)	nil	2P	M-2P

**PROCEDURE:**

Determination of  $\text{OH}^-$  and  $\text{CO}_3^{2-}$  alkalinity (in mg/L as  $\text{CaCO}_3$  eq.) types:

Fill burette with  $\text{H}_2\text{SO}_4$  solution. Pipette out 10 ml water sample in a 250 ml conical flask. Add 2-3 drops of phenolphthalein as an indicator. The solution becomes pink. Titrate this solution with acid solution taken in burette till the color of the solution changes to colorless. Note this end point  $V_1$  ml. Now at this point, add 2 drops of methyl orange indicator into the same solution. Solution becomes yellow. Continue the titration without refilling the burette till the color of solution turns to orange-red. Note this end point as  $V_2$  ml of the titration. Repeat the same procedure to get successive constant readings of P and M point.

The relationship between P point and M point denotes the type and extent of alkalinity present in the given water samples as shown in earlier table.

Table1: Determination of  $\text{OH}^-$  and  $\text{CO}_3^{2-}$  alkalinity types

S. No.	Burette Reading			Vol $\text{H}_2\text{SO}_4$ (ml)	
	Initial (I)	$V_1$	$V_2$	$P = V_1 - I$	$M = V_2 - I$

**CALCULATIONS:**

We consider here the amount of alkalinity  $\text{OH}^-$  and  $\text{CO}_3^{2-}$  present in the sample corresponds to the type P > 1/2M, then, the types of alkalinity present in given water sample is  $\text{OH}^-$  and  $\text{CO}_3^{2-}$  ( use the correlation table given above for types of alkalinity). Volume of acid corresponding to  $\text{OH}^- = 2P - M$  Volume of acid corresponding to  $\text{CO}_3^{2-} = 2(M - P)$

**Calculation of carbonate alkalinity (in mg/L as  $\text{CaCO}_3$  eq.):** Normality of  $\text{H}_2\text{SO}_4 = N = 0.05 \text{ N}$

Volume of  $\text{H}_2\text{SO}_4$  consumed =  $2(M - P)$  ml

Volume of sample water = 10 ml Equivalent weight of  $\text{CaCO}_3 = 50$

**Calculation of hydroxide alkalinity (in mg/L as  $\text{CaCO}_3$  eq.):** Normality of  $\text{H}_2\text{SO}_4 = N = 0.05 \text{ N}$  Volume of  $\text{H}_2\text{SO}_4$  consumed =  $2P - M$  ml

Volume of sample water = 10 ml Equivalent weight of  $\text{CaCO}_3 = 50$

Conc. Of  $\text{CO}_3^{2-}$  as  $\text{CaCO}_3$  equivalent (mg/l) =  $2(M - P) \times N \times 50 \times 1000 / \text{Vol. of sample water}$

**RESULT:**

(i) The carbonate alkalinity of water sample is = \_\_\_\_\_ mg/L as  $\text{CaCO}_3$  equivalent.

(ii) The hydroxyl alkalinity of water sample is = \_\_\_\_\_ mg/L as  $\text{CaCO}_3$  equivalent.

**PRECAUTIONS:**

- The burette, pipette and conical flask should be washed properly and then rinsed with distilled water.
- Redistilled water should be employed for preparing the alkaline solution.
- The colour change near the end point is very slow and thus should be observed very carefully.



## EXPERIMENT-09

### OBJECTIVE:

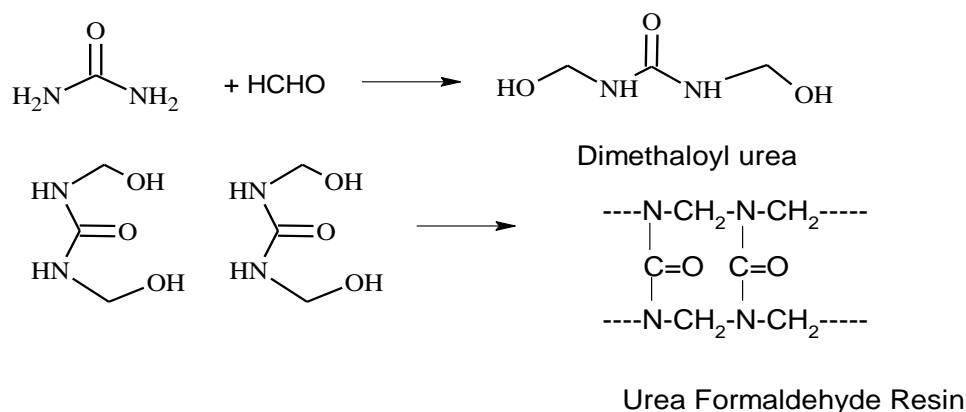
To prepare urea-formaldehyde (UF) resin

### APPARATUS/REAGENT REQUIRED:

Beaker, formaldehyde (40%), urea, conc.  $\text{H}_2\text{SO}_4$ , beaker, glass funnel, filter paper

### THEORY:

Amino resins are the condensation products obtained by the reaction of urea with formaldehyde.



### PROCEDURE:

Place 20ml of 40% formaldehyde solution in a 100ml beaker. Add about 10g of urea while stirring until a saturated solution is obtained. Add a few drops of conc.  $\text{H}_2\text{SO}_4$  with constant stirring. Continue stirring till a white coloured solid mass is not obtained. Wash the resin with water, dry and weigh it.

### RESULT:

- (i) **Physical appearance:** .....
- (ii) **Yield:** .....g

**Note:** Attach a small amount of sample in polybag in your practical copies.

### PRECAUTIONS:

1. Reaction is sometimes vigorous; hence, addition of conc.  $\text{H}_2\text{SO}_4$  is done very slowly.
2. The reaction should be carried conc.  $\text{H}_2\text{SO}_4$  must be added only when urea is completely dissolved in formaldehyde.

## EXPERIMENT-10

### OBJECTIVE:

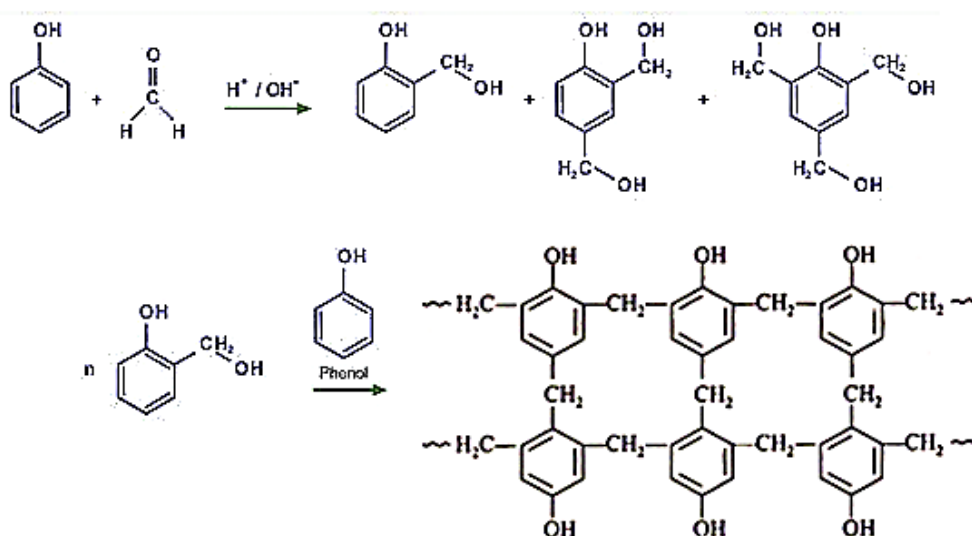
To prepare phenol-formaldehyde resin (Bakelite)

### APPARATUS/REAGENTS REQUIRED:

Glacial acetic acid, 40% formaldehyde solution, phenol, conc. HCl, beaker, glass rod, glass funnel, filter paper

### THEORY:

Phenolic resins are condensation polymerization products of phenolic derivatives (like phenol) and aldehydes (like formaldehyde). Bakelite is an important resin of this type and is prepared by condensing phenol with formaldehyde in the presence of acidic/alkaline catalyst.



### PROCEDURE:

Place 5ml of glacial acetic acid and 2.5ml of 40% formaldehyde solution in a beaker and add 2g of phenol. When whole of phenol is dissolved, add 2-3 drops of conc. HCl to it with constant stirring. Heat the content for about 5 minutes in the hot air oven. A pinkish solid mass is obtained. Wash the product, dry and weigh it.

### RESULT:

- (i) Physical appearance:.....
- (ii) Yield:.....g

**Note:** Attach a small amount of sample in polybag in your practical copies.

### PRECAUTIONS:

1. Reaction is sometimes vigorous, hence addition of conc. HCl is done very slowly.
2. The reaction should be carried conc. HCl must be added only when phenol is completely dissolved in formaldehyde.

## EXPERIMENT –11

### OBJECTIVE:

To prepare N/30 ferrous ammonium sulphate solution and to determine the strength of given ferrous ammonium sulphate solution using potassium dichromate as intermediate solution and diphenyl amine as internal indicator

### REQUIREMENTS:

Ferrous ammonium sulphate (Mohr's salt), unknown ferrous ammonium sulphate solution, standard potassium dichromate solution, diphenyl amine, dilute sulphuric acid, burette, pipette, conical flask.

### INDICATOR:

Diphenyl amine, (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>NH.

### END POINT:

Change of colourless, greenish solution to bluish-violet.

### THEORY:

Potassium dichromate oxidizes ferrous sulphate present in Mohr's salt into ferric sulphate in the presence of dilute sulphuric acid.



Diphenyl amine, in the presence of potassium dichromate, is converted from benzenoid to quinonoid form which is of bluish-violet colour. As long as single ferrous ion is present in the reaction mixture, no change in colour of the solution is there but as all the ferrous ions have been converted to ferric ions, addition of single drop of potassium dichromate converts benzenoid form of diphenyl amine (colourless) to its quinonoid form (bluish-violet) marking the end point of the reaction.

### PROCEDURE:

It completes in three stages:

#### 1. Preparation of known (standard) ferrous ammonium sulphate solution:

Weigh out exactly 1.3066 g of Mohr's salt.

$$W = \frac{NVE}{1000} \text{ g}$$

Where, N → Normality of solution required (N/30)

V → Volume of solution to be prepared (100 ml)

E → Equivalent weight of ferrous ammonium sulphate (392)

Transfer it into a 100ml measuring flask and dissolve it in about 1ml of dilute sulphuric acid. Now make up the solution upto the mark with distilled water. This is N/30 ferrous ammonium sulphate solution.

#### 2. Titration of known ferrous ammonium sulphate solution against standard potassium dichromate solution:

Pipette out 10ml of N/30 ferrous ammonium sulphate solution in a conical flask and add about 5ml of dilute sulphuric acid to it. Now add 4-5 drops of indicator to the reaction vessel and start titrating ferrous ammonium sulphate solution in a conical flask against standard potassium dichromate solution filled in the burette. Continue titration till the greenish solution of the reaction vessel does not change to bluish-violet. This is the end point of the reaction. Repeat the same titration for concordant readings.

3. **Titration of unknown ferrous ammonium sulphate solution against standard potassium dichromate solution:** Repeat exactly the same titration with unknown (supplied, whose strength is to find out) ferrous ammonium sulphate solution and take concordant readings.

**OBSERVATIONS:**

Weight of empty watch glass = x g

Weight of watch glass + Mohr's salt = y g

Weight of Mohr's salt = (y – x) g

**Titration of known ferrous ammonium sulphate (FAS) solution against standard  $K_2Cr_2O_7$ :**

S. No.	Volume of FAS (ml)	Reading of burette		Volume of $K_2Cr_2O_7$
		Initial	Final	
1.	10	0		
2.	10	0		
3.	10	0		

Let, volume of  $K_2Cr_2O_7$  used with known FAS solution =  $V_1$  ml

**Titration of unknown ferrous ammonium sulphate (FAS) solution against standard  $K_2Cr_2O_7$ :**

S. No.	Volume of FAS (ml)	Reading of burette		Volume of $K_2Cr_2O_7$
		Initial	Final	
1.	10	0		
2.	10	0		
3.	10	0		

Let, volume of  $K_2Cr_2O_7$  used with unknown FAS solution =  $V_2$  ml

**CALCULATIONS:**

$$\text{Strength of given ferrous ammonium sulphate solution} = \frac{10.w.V_2}{V_1} \text{ g/l}$$

**RESULT:**

The strength of given ferrous ammonium sulphate solution is .....g/l.

**PRECAUTIONS:**

1. While preparing known ferrous ammonium sulphate solution, never dissolve the salt directly in water.
2. Properly rinse the burette and pipette with the respective solutions.
3. Add the titrant from the burette drop wise with constant stirring.
4. After every titration, wash the conical flask well with water.

## EXPERIMENT-12

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### OBJECTIVE:

To study the adsorption of acetic acid on activated charcoal.

### APPARATUS/REAGENTS REQUIRED:

5-6 conical flasks or glass stoppered bottles, pipette, burette, beakers, glacial acetic and activated charcoal, 0.1N NaOH, thermostat or a large trough etc.

### THEORY:

The phenomenon of accumulation of solutes (solids dissolved in liquids and liquids like acetic acid miscible with water), gases (vapours) and liquids at the surface of a solid or liquid (or at the interface of two phases, solid-liquid, solid-gas, liquid-liquid) is referred to as adsorption. In adsorption, particles adhere to the surface of solids/liquids and in case of a solid, the greater the area of its surface (by a given weight), the greater will be its adsorptive capacity. The substance which is adsorbed on a particular surface is called adsorbate and the surface which has adsorbed is called adsorbent. If a solid is colloiddally dispersed, the surface area presented by it for adsorption of acetic acid on activated charcoal is to be measured. In fact charcoal does not produce a true colloidal solution, but it is used because it is capable of being obtained in sufficiently divided state. The results will be interpreted in terms of the Freundlich adsorption isotherm which is expressed as:

$$\frac{x}{m} = kc^{\frac{1}{n}}$$

Where, x = amount of solute adsorbed (adsorbate) per unit mass of the adsorbent at concentration c.

m = amount of adsorbent.

c = equilibrium concentration of adsorbate in the solution.

k and n are the constants and depend upon the nature of adsorbate and adsorbent.

Taking log of the above equation.

$$\log \frac{x}{m} = \log k + \frac{1}{n} \log c$$

If a graph is plotted between the values of x/m and c, a parabolic curve is obtained. If log x/m is plotted against log c, a straight line graph is obtained, whose slope is 1/n and the intercept is log k.

### PROCEDURE:

Place 50, 40, 30, 20 and 10cm<sup>3</sup> of a standard 0.1N solution of acetic acid in five glass stoppered bottles (flasks). Add 0,10,20,30,40 cm<sup>3</sup> of distilled water and 1g of activated charcoal to each bottle respectively. Shake vigorously the bottles for about an hour.

Filter the solution from each bottle and titrate each filtrate with a standard NaOH solution. Calculate the amount of the acid adsorbed per gram of the adsorbent and the equilibrium concentration of the acid.

### OBSERVATIONS:

Room temperature = ...°C

Amount of charcoal added to each bottle = m g

Volume of filtrate taken each time for titration (after adsorption) = 10 cm<sup>3</sup>

Bottle No.	Vol. of 0.1N acetic acid (ml)	Vol. of distilled water added(ml)	Initial conc. of acid in g.eq./dm <sup>3</sup>	Volume of N/10 NaOH used in titration			
				(i)	(ii)	(iii)	Correct value
1	10	40	a	b			
2	20						
3	30						
4	40						
5	50						

### CALCULATIONS:

For initial conc. of acid in g.eq./dm<sup>3</sup>,

$$N_1 V_1 = N_2 V_2$$

$$0.1 \times 10 = N_2 \times 50$$

$$N_2 = \text{say 'a'}$$

Conc. of acid in g.eq./dm<sup>3</sup> after adsorption,

$$N_1 V_1 = N_2 V_2$$

$$(\text{CH}_3\text{COOH}) \quad (\text{NaOH})$$

$$N_1 \times 10 = 0.1 \times b$$

$$N_1 = \text{say 'd'}$$

∴ The amount of acetic acid adsorbed in g.eq./dm<sup>3</sup> by the charcoal = (a – d)

$$\text{Conc. of acid adsorbed in g (x)} = \frac{NVE}{1000} = \frac{(a-d) \times 50 \times 60}{1000}$$

Then, calculate the amount of acid adsorbed per g. of adsorbent (x/m). Calculate x/m in a similar manner for other sets and record the results as follows:

Plot a graph between x/m and c and between log x/m and log c.

Bottle No.	Initial conc. of acid in gm. eq./dm <sup>3</sup>	Conc. of acid after adsorption gm.eq./dm <sup>3</sup>	Conc. of acid adsorbed in gm.eq./dm <sup>3</sup> (c)	Conc. of acid adsorbed in gm.(x)	x/m	log x/m	log c
1	a	d	(a-d)				
2							
3							
4							
5							

### RESULT:

The two curves plotted discussed above should be attached. The nature of these curves is in line with the velocity of Freundlich's isotherm. The value of the constants k and n as calculated from these curves come out to be ... and ... respectively.

### PRECAUTIONS:

1. Steady and vigorous shaking should be given to all
2. Fresh activated charcoal should be used

## EXPERIMENT-13

### OBJECTIVE:

To determine the molecular weight of the given non-volatile compound cryoscopically.

### REAGENTS AND APPARATUS USED:

Unknown substance, water, Beckmann's thermometer, outer and inner freezing tubes, ice bath, stirrer, ice, salt.

### THEORY:

Every liquid freezes at fixed temperature and the effect of addition of a solute to it is the lowering of the melting point. The depression of freezing point is found to be proportional to the amount of solute added to a known weight of the solvent.

The molecular weight of the solute is calculated by the formula.

$$M = \frac{100KW_2}{W_1\Delta T}$$

Where,  $W_2 \rightarrow$  weight of solute (g)

$W_1 \rightarrow$  weight of solvent (g)

$\Delta T \rightarrow$  depression in freezing point

$K \rightarrow$  molar depression constant (18.5)

$M \rightarrow$  molecular weight of the solute.

### PROCEDURE:

Set the assembly as shown in the figure. The inner freezing tube (B) is fitted into the outer jacket (A). A large metal stirrer (L) is used to stir the freezing mixture in the ice bath (I) in order to have a uniform cooling. A fine glass stirrer (F) and Beckmann thermometer are placed carefully inside the inner tube (B).

A known amount of pure solvent is placed in the inner tube (B) and the ice bath is filled with the freezing mixture. The liquid solvent is stirred uniformly and gently and the temperature is noted down when it becomes constant. This is the freezing point of the pure solvent.

Now, a weighed amount of solute is added through the side arm (D) in the inner tube and again the temperature is noted on constancy. This is the freezing point of the solution.

The difference between the two readings gives the depression in the freezing point.

### OBSERVATIONS:

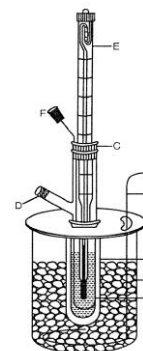
Freezing point of the given solvent = ..... °C

The amount of substance taken = ..... gm.

Freezing point of given solution = ..... °C

Depression in freezing point ( $\Delta T$ ) = ..... °C

Volume of the solvent taken = V ml (say)



**Fig2. Cryoscopic determination of M. Wt.**

Hence, weight of solvent ( $W_1$ ) =  $V \times$  Density of solvent

**CALCULATION:**

$$M = \frac{100KW_2}{W_1\Delta T}$$

**RESULT:**

The molecular weight of the given solute is.....

**PRECAUTIONS:**

1. The temperature of the freezing mixture must not be more than 3°C or 4°C below the freezing point of the solvent.
2. The solvent should be given a slow and steady cooling in order to prevent an excessive super-cooling.
3. The stirring should be steady, slow and smooth to avoid the production of heat due to friction.
4. The thermometer should be gently tapped before a reading to avoid the effect due to sticking of mercury in the capillary.



## EXPERIMENT-14

### OBJECTIVE:

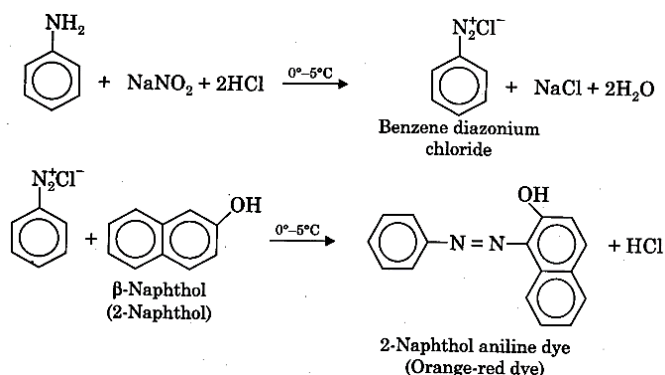
To prepare aniline- $\beta$ -naphthol azo dye from aniline.

### REQUIREMENTS:

Aniline, dilute HCl, sodium nitrite,  $\beta$ -naphthol, sodium hydroxide.

### THEORY:

Aniline on treatment with sodium nitrite in acidic medium undergoes diazotization to give anilinium chloride.



### PROCEDURE:

Take given amount of aniline and add to it dilute HCl in a dropwise manner with constant shaking till whole of the aniline is not converted to its salt. Cool the reaction mixture under ice cold water. Now add a dilute solution of sodium nitrite to this solution to neutralize it completely. Again cool the reaction mixture under tap water. Then add a very dilute alkaline  $\beta$ -naphthol solution to it. A bright orange dye is obtained which is filtered, dried and weighed.

### RESULT:

- (i) **Physical appearance:** ...
- (ii) **Yield:** ...g

**Note:** Attach a small amount of sample in poly bag in your practical copies.

### PRECAUTIONS:

1. Convert whole of aniline to its salt.
2. Neutralization must be complete.
3. The reaction mixture must be cooled properly at each step.
4. Dilute solutions must be used.

**Virtual Lab 1:** <http://vlab.amrita.edu/?sub=2&brch=193&sim=352&cnt=4>

**Virtual Lab 2:** <http://csc-iiiit.vlabs.ac.in/exp2/index.html#>