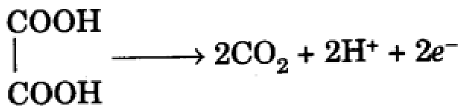
EXPERIMENT No. 1

AIM: Prepare 250 ml of a N/10 Solution of Oxalic Acid from Crystalline Oxalic Acid

Theory

Crystalline oxalic acid is a primary standard, its standard solution can be prepared directly.

The formula for crystalline oxalic acid is $\begin{array}{l}\text{COOH}\\ |\\ \text{COOH}\end{array}\ 2H_2O$. The ionic equation for the oxidation of oxalic acid is



$$\begin{array}{l}\text{COOH}\\ |\\ \text{COOH}\end{array} \longrightarrow 2CO_2 + 2H^+ + 2e^-$$

It is clear from the above equation that two electrons are given out during oxidation of one molecule of oxalic acid.

$$\therefore \text{ Eq. mass of oxalic acid} = \frac{\text{Molecular mass of oxalic acid}}{\text{No. of electrons lost by one molecule of it}}$$

$$= \frac{126}{2} = 63$$

$$\text{Strength } (g/l) = \text{Normality} \times \text{Eq. mass}$$

$$= \frac{1}{10} \times 63 = 6.3 \text{ g/l}$$

$\therefore$ For preparing 1 litre of $\frac{N}{10}$ oxalic acid solution 6.3 g of it have to be dissolved.

$\therefore$ For preparing 250 ml of $\frac{N}{10}$ oxalic acid, oxalic acid crystals required

$$= \frac{6.3}{1000} \times 250 = 1.575 \text{ g.}$$

Apparatus

Watch glass, analytical balance, weight box, fractional weight box, 250 ml beaker, glass rod, 250 ml measuring flask and wash bottle.

Chemical Required

Oxalic acid crystals and distilled water.

Procedure

1. Take a watch glass, wash it with distilled water and then dry it.
2. Weigh the clean and dried watch glass accurately and record its weight in the note book.

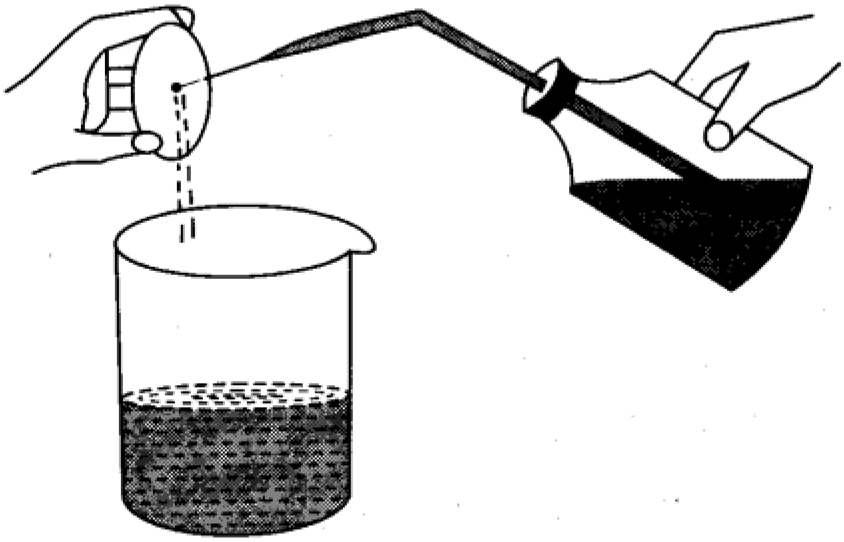


Fig. Washing of watch glass to transfer sticking particles to beaker.

3. Weigh 3.150 g oxalic acid on the watch glass accurately and record this weight in the note-book.
4. Transfer gently and carefully the oxalic acid from the watch glass into a clean 250 ml beaker. Wash the watch glass with distilled water with the help of a wash bottle to transfer the particles sticking to it into the beaker [as shown in the Figure above].
   The volume of distilled water for this purpose should not be more than 50 ml.
5. Dissolve oxalic acid crystals in the beaker by gentle stirring with a clean glass rod.
6. When the oxalic acid in the beaker is completely dissolved, transfer carefully the entire solution from

the beaker into a 250 ml measuring flask (volumetric flask) with the help of a funnel [as shown in figure below].

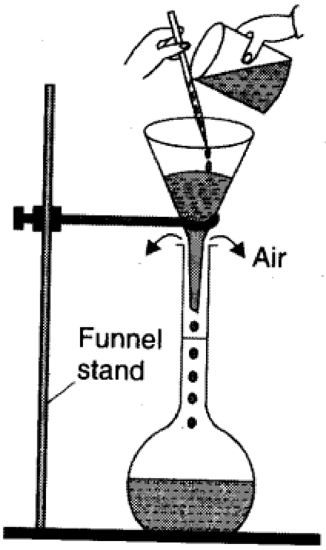


Fig. Transferring solution to measuring flask.

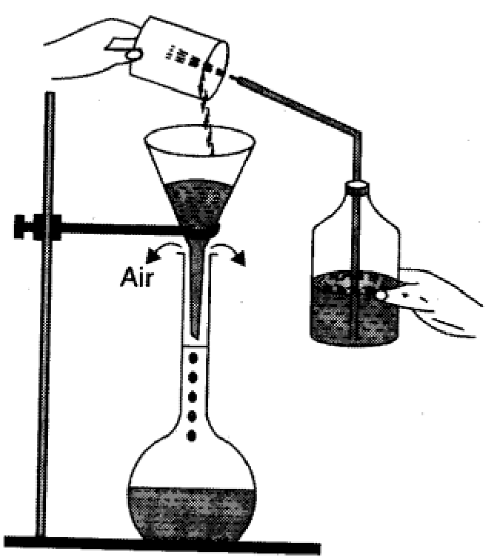


Fig. Transferring last traces of solution to measuring flask.

7. Wash the beaker with distilled water. Transfer the washings into the measuring flask [as shown in figure below].
8. Finally wash the funnel well with distilled water with the help of a wash bottle to transfer the solution sticking to the funnel into the measuring flask [as shown in figure below].
9. Add enough distilled water to the measuring flask carefully, up to just below the etched mark on it, with the help of a wash bottle.
10. Add the last few drops of distilled water with a pipette until the lower level of the meniscus just touches the mark on the measuring flask.
11. Stopper the measuring flask and shake gently to make the solution uniform through-out. Label it as

oxalic acid solution.

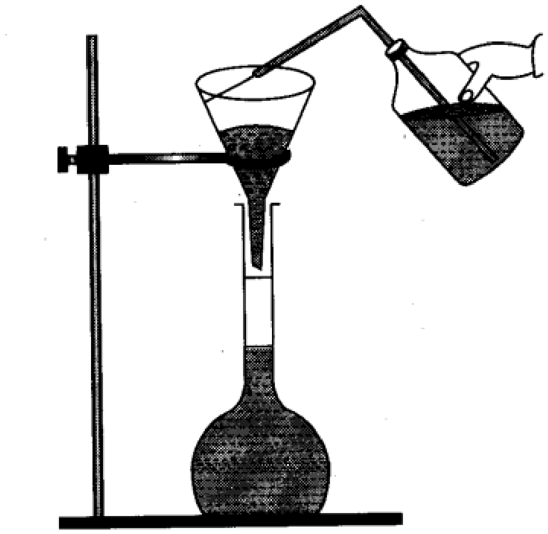


Fig. Washing last traces of solution from funnel to the measuring flask.

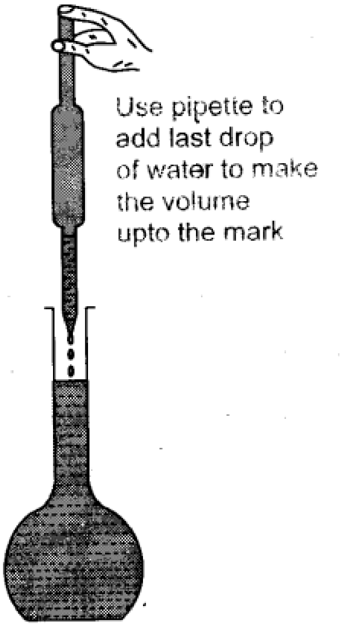


Fig. Using pipette to add last drop of water to make the volume upto the mark.

EXPERIMENT NO. 2

AIM: Prepare 250ml N/10 sodium carbonate solution.

Theory

Sodium carbonate is a primary standard. Its molecular mass is 106. To prepare N/10 Na_2CO_3 solution,

$$\text{Eq. mass of } Na_2CO_3 = \frac{Mol.mass}{2} = \frac{106}{2} = 53$$

1000mL of sodium carbonate solution contains= 53 g (1N Na_2CO_3 solution)
1mL of sodium carbonate solution contains= 53/1000 g (1N Na_2CO_3 solution)
250mL of sodium carbonate solution contains= (53x250)/1000g (1N Na_2CO_3 solution)
250mL of sodium carbonate solution contains= (53x250)/(1000x10) g =1.325g (N /10 Na_2CO_3 solution)

53 g of sodium carbonate should be dissolved per litre of the solution.
Therefore, to prepare 250 ml of N/10 Na_2CO_3 solution, 1.325 g of sodium carbonate are dissolved in lesser quantity of water and the solution diluted to exactly 250 ml.

Apparatus

Weight box, chemical balance, watch glass, 250 ml beaker, glass rod, 250 ml measuring flask, wash bottle.

Procedure

1. Take a watch glass, wash it with distilled water and then dry it.

2. Weigh the clean and dried watch glass accurately and record its weight in the note-book.

3. Weigh 1.325 g sodium carbonate on the watch glass accurately and record this weight in the note-book.

4. Transfer gently and carefully sodium carbonate from the watch glass into a clean and dry measuring flask using a funnel. Wash the watch glass with distilled water with the help of a wash bottle to transfer the particles sticking to it into funnel. The volume of distilled water for this purpose should not be more than 50 ml.

5. Wash funnel several times with distilled water by using a wash bottle to transfer the sticking particles into the measuring particles into the measuring flask. While washing the funnel, add water in small amounts. The volume of distilled water used for this purpose should not be more than 50 mL.

6. Finally wash the funnel thoroughly with distilled water with the help of a wash bottle to transfer the solution sticking to the funnel into the measuring flask (Figure a).

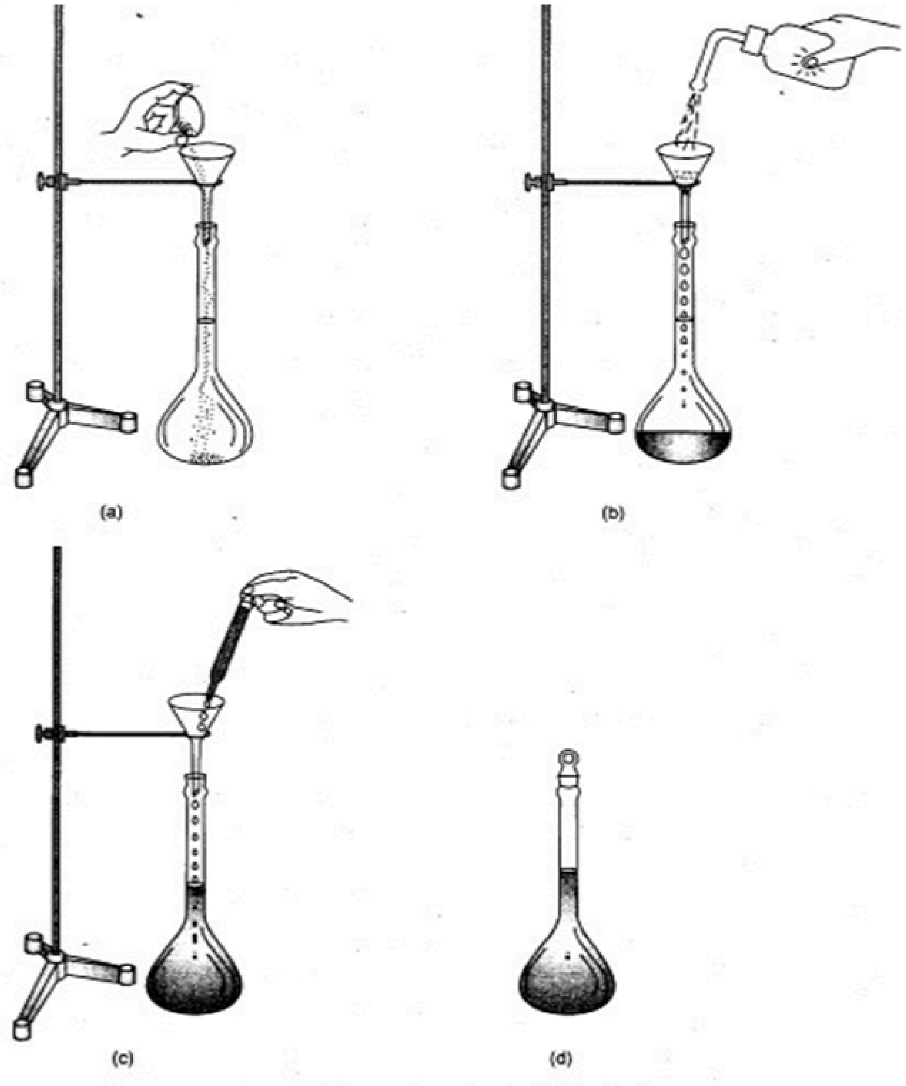
7. Swirl the measuring flask till solid sodium carbonate dissolves.

8. Add enough distilled water to the measuring flask carefully upto just below the etched mark on it, with the help of wash bottle (Figure b).

9. Add the last few mL of distilled water dropwise until the

lower level of the meniscus just touches the mark on the measuring flask (Figure c).

10. Stopper the measuring flask and shake gently to make the solution uniform throughout. Label it as N/10 sodium carbonate (Figure d) solution.



. Preparation of a standard solution.

(*a*) Transferring sodium carbonate
(*b*) Addition of water
(*c*) Adding last few mL drop wise
(*d*) Standard solution of sodium carbonate.

EXPERIMENT NO. 3

AIM

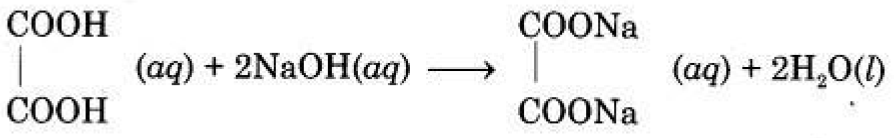
Determine the strength of the given sodium hydroxide solution by titrating it against a standard oxalic acid solution.

Materials Required

1. Burette
2. Pipette
3. Conical flask
4. Burette stand
5. Funnel
6. White glazed tile
7. Measuring flask
8. Oxalic acid (as per needed)
9. Sodium hydroxide solution (as per needed)
10. Phenolphthalein indicator (as per needed)

Theory

This estimation involves titration of a weak acid that is oxalic acid against a strong base is sodium hydroxide and phenolphthalein is the indicator of choice. The reaction between oxalic acid and sodium hydroxide is



$$\begin{array}{l}\mathrm{COOH}\\ \vert\\ \mathrm{COOH}\end{array}(aq) + 2\mathrm{NaOH}(aq) \longrightarrow \begin{array}{l}\mathrm{COONa}\\ \vert\\ \mathrm{COONa}\end{array}(aq) + 2\mathrm{H_2O}(l)$$

Since sodium hydroxide is not a primary standard a standard solution of oxalic acid is prepared and used for standardisation of sodium hydroxide.

In acid base titration at the end point the amount of acid becomes chemically equivalent to the amount of base present. In case of strong acid and strong base titration at the end point of solution the solution become neutral.

Indicator. Phenolphthalein.

End Point. Colourless to pink (Alkali in burette)

Procedure

1. Prepare standard solution of Oxalic acid of concentration M/20.
2. Take a burette and wash it with water.
3. Rinse and then fill the burette with the given sodium hydroxide solution. Clamp it vertically in burette stand.
4. Rinse the pipette with the given oxalic acid solution.
5. Pipette out 20 ml of the oxalic acid solution in a washed titration flask.
6. Add 1-2 drops of phenolphthalein indicator into it and place it just below the nozzle of the burette over a white glazed tile.
7. Note down the lower meniscus of the solution in the burette and record it as the initial burette reading.
8. Now run the sodium hydroxide solution slowly and dropwise into the flask till a very faint permanent pink colour is just obtained. Read

the lower meniscus of the solution again in the burette and record it as final burette reading.

9. Repeat the procedure until three concordant readings are obtained.

Observations

Molarity of the given oxalic acid solution = M/20

Volume of oxalic acid solution taken for each titration = 20 ml.

S.No.	Initial reading of burette (ml)	Final reading of burette (ml)	Volume of sodium hydroxide used (ml)
1			
2			
3			
4			

Concordant volume = y ml (say)

Calculations

According to the equation, one mole of oxalic acid reacts with two moles of sodium hydroxide.

$M_{O.A} \times V_{O.A} = M_{NaOH} \times V_{NaOH}$

$(20 \times 1/20) = (y \times M_{NaOH})$

$M_{NaOH} = 1/y$

Strength of NaOH = Molarity x Molar mass= (1/y)x80=80/y g/L

Result

The strength of the given sodium hydroxide solution is 80/y g/L

EXPERIMENT NO.: 4

Aim: To estimate the amount of total, permanent and temporary hardness in the collected sample of water. A standard solution of ……………. EDTA is provided.

Principle:

Hardness in water is due to the presence of dissolved salts of calcium and magnesium. It is unfit for drinking, bathing, washing and it also forms scales in boilers. Hence it is necessary to estimate the amount of hardness producing substances present in the water sample. Once it is estimated, the amount of chemicals required for the treatment of water can be calculated.

The estimation of hardness is based on complexometric titration. Hardness of water is determined by titrating with a standard solution of ethylene diamine tetra acetic acid (EDTA) which is a complexing agent. Since EDTA is insoluble in water, the disodium salt of EDTA is taken for this experiment. EDTA can form four or six coordination bonds with a metal ion.

1. Total hardness

Total hardness is due to the presence of bicarbonates, chlorides and sulphates of calcium and magnesium ions. The total hardness of water is estimated by titrating the water sample against EDTA using

Eriochrome Black-T (EBT) indicator. Initially EBT forms a weak EBTCa2+/Mg2+ wine red coloured complex with Ca2+/Mg2+ ions present in the hard water. On addition of EDTA solution, Ca2+/Mg2+ ions preferably forms a stable EDTACa2+/Mg2+ complex with EDTA leaving the free EBT indicator in solution which is steel blue in colour in the presence of ammonia buffer (mixture of ammonium chloride and ammonium hydroxide, pH 10).

$$\text{Eriochrome Black-T} + Ca^{2+}/Mg^{2+} \longrightarrow \underset{\text{(Wine red)}}{\text{Eriochrome Black-T-}Ca^{2+}/Mg^{2+}}$$

$$\underset{\text{(Wine red)}}{\text{Eriochrome Black-T-}Ca^{2+}/Mg^{2+}} + \text{EDTA} \longrightarrow \text{EDTA-}Ca^{2+}/Mg^{2+} + \underset{\text{(Steel blue)}}{\text{Eriochrome Black-T}}$$

2.Temporary hardness

Temporary hardness is due to the presence of bicarbonates of calcium and magnesium ions. It can be easily removed by boiling.

When water is boiled, temporary hardness producing substances (bicarbonates) are precipitated as insoluble carbonates or hydroxides. This precipitate can be removed by filtration. (The filtrate is used in the next step)

$$\text{Eriochrome Black-T} + Ca^{2+}/Mg^{2+} \longrightarrow \underset{\text{(Wine red)}}{\text{Eriochrome Black-T-}Ca^{2+}/Mg^{2+}}$$

$$\underset{\text{(Wine red)}}{\text{Eriochrome Black-T-}Ca^{2+}/Mg^{2+}} + \text{EDTA} \longrightarrow \text{EDTA-}Ca^{2+}/Mg^{2+} + \underset{\text{(Steel blue)}}{\text{Eriochrome Black-T}}$$

3. Permanent hardness

Permanent hardness is due to the presence of chlorides and sulphates of calcium and magnesium ions. This type of hardness cannot be removed by boiling. The filtrate obtained from the above step contains permanent hardness producing substances and is estimated against EDTA using EBT indicator.

Procedure:

The burette is filled with standard EDTA solution to the zero level, following usual precautions.

1. Estimation of Total Hardness 20 ml of the given water sample is pipetted out into a clean conical flask. 5 ml ammonia buffer and 2 drops of EBT indicator are added and titrated against EDTA from the burette. The end point is the change of colour from wine red to steel blue. The titration is repeated to get concordant titre value.

2. Estimation of Permanent Hardness

100 ml of the given sample of water is pipetted out into a clean beaker and boiled for 20 minutes. It is then filtered to remove the precipitate formed due to the decomposition of temporary hardness producing salts. The filtrate is made up to 100 ml in standard measuring flask (SMF) using distilled water.

20 ml of the made up solution is pipetted out into a conical flask, 5 ml ammonia buffer and 2 drops of EBT indicator are added and titrated against the EDTA. The end point is the change of colour from wine red to steel blue. The titration is repeated to get concordant titre value.

3. Temporary Hardness

The temporary hardness is calculated from the total and permanent hardness. Temporary Hardness = Total Hardness − Permanent Hardness

Result:

The collected water sample contains

Total hardness = …………ppm

Permanent hardness = …………ppm

Temporary hardness =…………… ppm

Titration-1 Estimation of Total Hardness

Standard EDTA vs Water sample

Volume of hard water sample (ml)	Burette Reading		Volume of EDTA solution (ml)	indicator	
	Initial	Final			

Titration-2 Estimation of Permanent Hardness

Standard EDTA X Boiled water sample				
Volume of boiled water sample (ml)	Burette Reading		Volume of EDTA solution (ml)	Indicator
	Initial	Final		

Calculation

1 ml of 0.01 M EDTA ≡ 1 mg of CaCO3

V1 ml of EDTA ≡ V1 mg of CaCO3

1) Calculation of total hardness

Volume of EDTA solution consumed = ………… ml

Volume of hard water taken = …………. ml

Total hardness = (Volume of EDTA solution consumed X1000)/(Volume of the hard water taken) ppm = ………………….. ppm

2) Calculation of permanent hardness

Volume of EDTA solution consumed = ………… ml

Volume of boiled water taken = …………. ml

Permanent Hardness = (Volume of EDTA solution consumed X1000)/ (Volume of the boiled water taken) ppm = ………………….. ppm

3) Calculation of temporary hardness

Temporary hardness of the given sample of water

= Total hardness - Permanent hardness

= ……………….. ppm

AIM: - To prepare phenol formaldehyde resin (Bakelite).

CHEMICALS USED: - Glacial acetic acid, 40% formaldehyde solution, Phenol, conc. H_2SO_4.

APPARATUS REQUIRED: -Glass rod, beakers, funnel, measuring cylinder, dropper and filter paper.

PRINCIPLE: - Phenol formaldehyde resins (PFs) are condensation polymers and are obtained by condensing phenol with formaldehyde in the presence of an acidic or alkaline catalyst. They were first prepared by ***Backeland***, an American Chemist who gave them the name as ***Bakelite.*** These are thermosetting polymers.

Thermosets:- The polymers which on heating change irreversibly into hard rigid and infusible materials are called thermosetting polymers. These polymers are usually prepared by heating relatively low molecular mass, semi fluid polymers, which becomes infusible and form an insoluble hard mass on heating. The hardening on heating is due to the formation of extensive cross-linking between different polymeric chains. This leads to the formation of a three dimensional network of bonds connecting the polymer chains. Since 3D network structure is rigid and does not soften on heating, the thermosetting polymers can not be reprocessed. Some important examples of thermosetting polymers are Urea-Formaldehyde resin and Melamine-Formaldehyde resins.

Properties:-

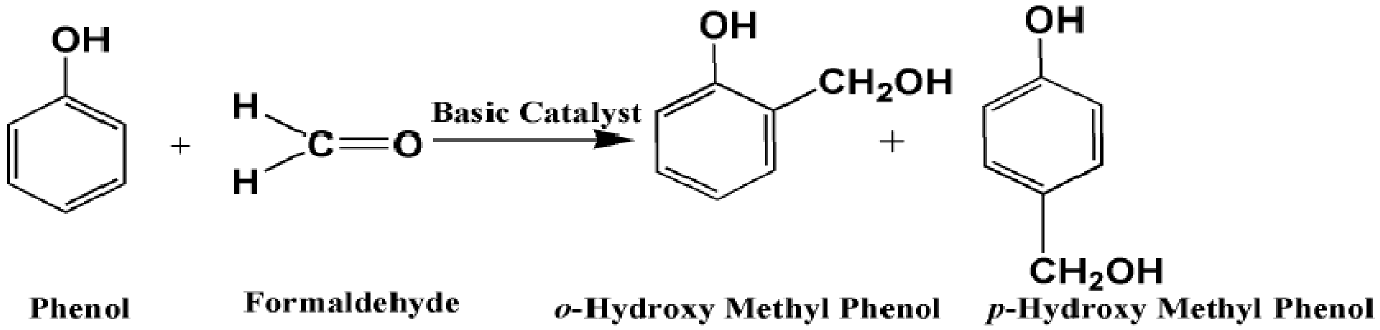
- Phenol- formaldehyde resins having low degree of polymerization are soft. They possess excellent adhesive properties and are usually used as bonding glue for laminated wooden planks and in varnishes and lacquers.
- Phenol- formaldehyde resins having high degree of polymerization are hard, rigid, scratch-resistant and infusible. They are resistant to non-oxidizing acids, salts and many organic solvents. They can withstand very high temperatures. They act as excellent electrical insulators also.

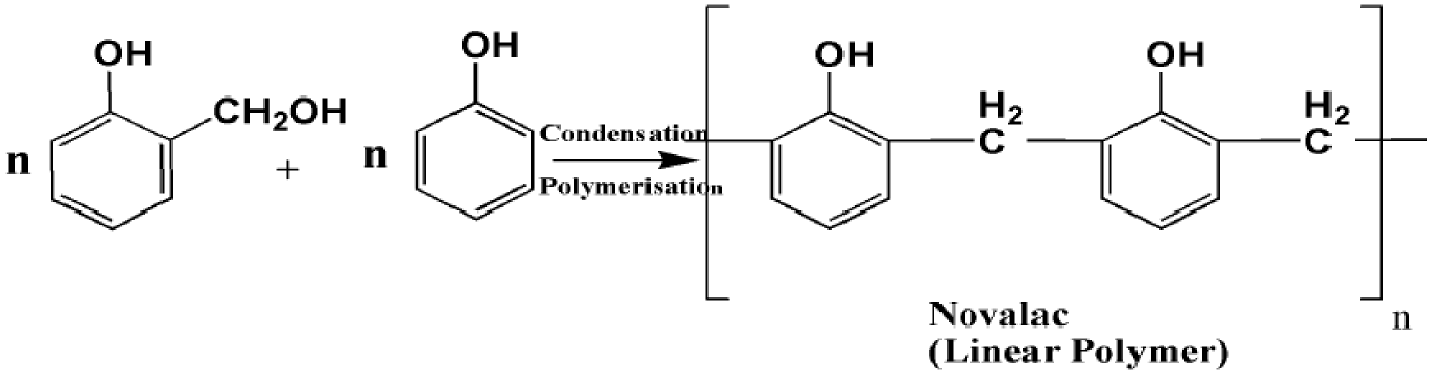
Uses:-

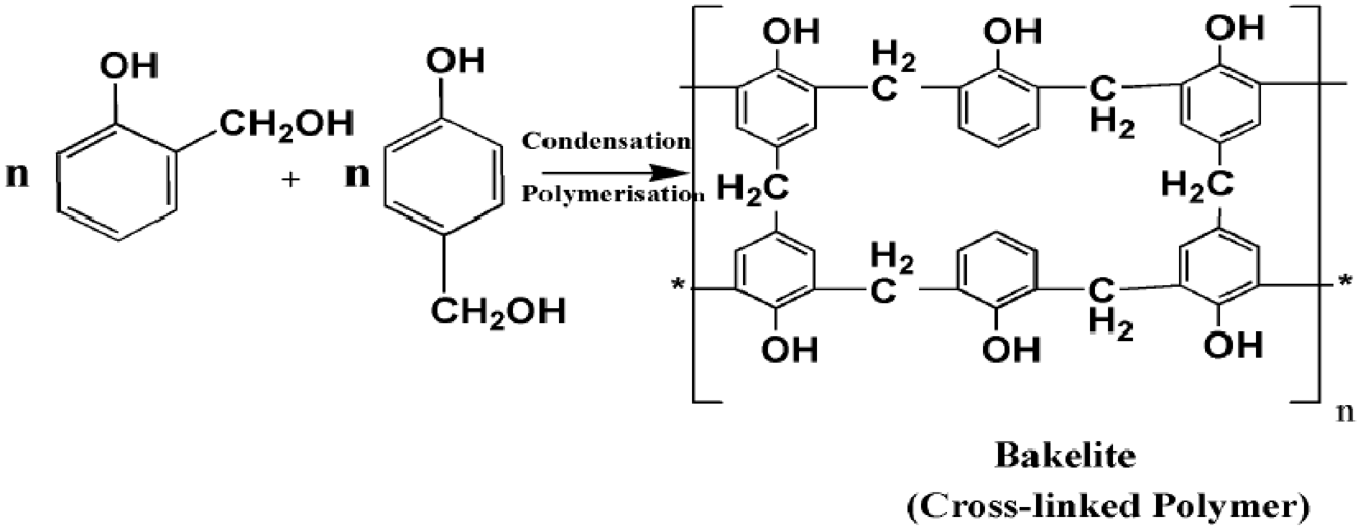
- They are used for making moulded articles such as radio and TV parts, combs, fountain pen barrels, phonograph records etc.
- They are used for making decorative laminates, wall coverings etc.
- They are used for making electrical goods such as switches, plugs etc.
- They are used for impregnating fabrics wood and paper.
- They are used as bonding glue for laminated wooden planks and in varnishes and lacquers.
- Sulphonated phenol-formaldehyde resins are use as ion-exchange resins.

Preparation:- PFs are prepared by reaction of phenol with formaldehyde in the presence of acidic or basic catalyst. The process may be carried out as follows:

A mixture of phenol and formaldehyde are allowed to react in the presence of a catalyst. The process involves formation of methylene bridges in *ortho*, *para* or *both ortho* and *para* positions. These results first the formation of linear polymer (called *NOVALAC*) and then in to cross-linked polymer called phenol-formaldehyde resin or *bakelite*.







PROCEDURE: -

1. Place 5ml of glacial acetic acid and 2.5 ml of 40% formaldehyde solution in a 500ml beaker and add 2 grams of phenol.
2. Add few ml of conc. Sulphuric acid into the mixture carefully. Within 5 min a large mass of plastic is formed.
3. The residue obtained is washed several times with distilled water, and filtered product is dried and yield is calculated.

RESULT: - The weight of the phenol formaldehyde resin is ___ g.

PRECAUTIONS: -

1. The reaction is sometimes vigorous and it is better to be a few feet away from the beaker while adding the H_2SO_4 and until the reaction is complete.
2. The experiment should be preferably carried out in fume cupboard.

EXPERIMENT NO. 5

Aim: To determine moisture content of a given sample of coal.

Apparatus: Silica crucible with vented lid, electric oven, desiccator, weighing balance, etc.

Chemicals: Powdered coal sample.

Theory:
Coal is a primary, solid, fossil fuel. Coal sample has to be analysed before using it in any field/industry to find out its quality & suitability. Moisture, volatile matter & ash content of coal are determined under proximate analysis. This method is simple & quick and is used primarily to determine the suitability of coal for cooking, power generation or for iron ores melting in the manufacture of steel. Coal comes in following main types or ranks Peat, lignite or brown coal, bituminous coal, anthracite & graphite. Each type of coal has a certain set of physical parameters which are mostly controlled by moisture, volatile content (in terms of aliphatic or aromatic hydrocarbons' & carbon content. The carbon content is lowest in peat and highest in anthracite.

Moisture
Moisture is an important property of coal, as all coals are mined wet. Groundwater and other extraneous moisture is known as adventitious moisture and is readily evaporated. Moisture held within the coal itself is known as inherent moisture & is analysed. Moisture reduces the calorific value of coal and considerable amount of heat is wasted in evaporating it during combustion.
Moisture mayoccur in four possible forms within coal: Surface moisture, hydroscopic moisture, Decomposition moisture & Mineral moisture. Total moisture is analysed by loss of mass of sample by drying in air at 100 to 105 ℃ (210-220 F) and relative loss of mass is determined. Total moisture content of the coal should be as low as possible.

Procedure: Determination of Inherent Moisture:
Transfer about 1g known quantity' of powdered air dried coal sample into a previously weighed silica crucible. Place the open crucible with sample in an electric oven and heat it at about 100 to 105 ℃ for an hour. Take out the crucible after one hour from the oven and cool it in a desiccator (containing moisture absorbing anhydrous calcium chloride). Then weigh the crucible with sample and repeat the process of heating, cooling & weighing till constant weight is obtained. Calculate the loss in weight.

Observations and Calculations :For Moisture:

Weight of empty crucible= $w_{1=}$ ....g.

Weight of crucible + Coal sample = w_2 =…………………g.

Weight of Crucible + Coal sample after heating = w_3= ………………g.

Percentage of moisture in given sample of coal

= w_2-w_3 / w_2-w_1 x 100 =……………..g.

EXPERIMENT—14

AIM: DETERMINATION OF pH OF A SAMPLE OF WATER

THEORY: The term “pH” refers to the measurement of hydrogen ion activity in the solution. Since the direct measurement of the pH is very difficult, specific electrodes are needed for quick and accurate pH determination. pH is measured on a scale of 0 to 14, with lower values indicating high H^+ (more acidic) and higher values indicating low H^+ ion activity (less acidic). A pH of 7 is considered as neutral. Every whole unit in pH represents a ten-fold increase in or decrease in hydrogen ion concentration.

Most natural waters possess the pH values ranging from 5.0 to 8.5. Rain water have a pH value of 5.4 to 6.0 which then reacts with the soils and minerals causing the reduction in H^+ ion concentration and thus the water may become alkaline with a pH 0f 8.0-8.5. More acid water (pH 9) and other immediate changes in the hydrogen ion concentration (pH) suggest that the quality of the water is adversely affected due to the introduction of some toxic contaminants in water bodies.

pH is measured using pH meter, which comprises a detecting unit consisting of a glass electrode, reference electrode, usually a calomel electrode connected by KCl Bridge to the pH sensitive glass electrode and an indicating unit which indicates the pH corresponding to the electromotive force is then detected. Before measurement, pH meter should be calibrated by using at least two buffers.

EQUIPMENT REQUIRED:-

1. pH meter
2. pH electrode filled with KCL solution
3. Buffer solutions of pH4 and pH 7
4. Clean beakers
5. Tissue papers
6. Distilled water
7. Thermometer

PROCEDURE:-

* Plug in the pH meter to power source and let it warm up for 5 to 10 minutes

* Wash the glass electrode with distilled water and clean slowly with a soft tissue. * Note the temperature of water and set the same on the pH meter

* Place the electrode in pH 7 buffer solution and set the value of 7 on the pH meter turning the Calibrate knob on the meter.

* Take out the electrode, wash with DW and clean.

* Dip the electrode in the pH 4 buffer solution. Adjust the value on the pH readout meter by the Slope switch . Repeat with pH 7 and pH4 buffers till a correct and stable reading is displaced.

* While moving and cleaning the electrode, put the selector switch on standby mode. Turn to pH mode for recording the pH.

* Now place the electrode in the water sample whose pH is to be determined.

* You can take a number of simultaneous readings for different samples until the power is on

PRECAUTIONS 1. The pH meter can be standardized by measuring the 7-pH buffer solution or any other solution of standard pH. Sometimes, the manufacturer of the pH meter may suggest other methods of standardizing, which too have to be followed.

2. The electrodes have to be inserted into the water so that it does not touch the bottom of the beaker. Bottom contact with damage may cause damage to the electrodes.

3. Any cause of slow response due to the polarization can be solved by washing the electrodes thoroughly.

4. Periodic check has to be conducted to check the electrodes

5. During the electrode storage, they have to be kept moist. And also follow the instructions of the manufacturer.