

Object : Determination of Total Alkalinity in the given sample of water using standard Sulphuric acid solution.

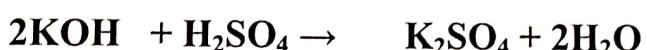
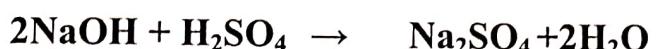
Apparatus: Burette, Pipette, Conical flask, Dropper, Measuring Cylinder, Beaker etc.

Chemicals: Standard N/50 H_2SO_4 solution, Methyl orange indicator, water sample, etc .

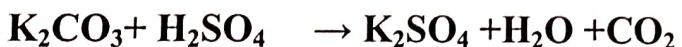
Principle: Alkalinity in water is due to the presence of one or more of the following salts: NaOH , KOH , Na_2CO_3 , K_2CO_3 , NaHCO_3 , $\text{Ca}(\text{HCO}_3)_2$, $\text{Mg}(\text{HCO}_3)_2$, etc. For the estimation of alkalinity of water, a known volume of the given water sample is titrated against standard acid (like N/50 H_2SO_4) solution using methyl orange indicator. The end point is yellow to pink colour.

The chemical equations involved are summarized below:

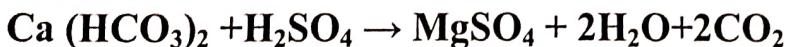
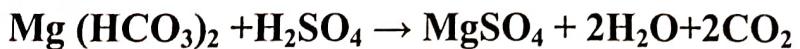
(a) For hydroxides :



(b) For carbonates :



(c) For bicarbonates :



Procedure:

1. After washing, rinse and fill the burette with N/50 H_2SO_4 solution.
2. Wash the titration (or conical) flask with distilled water and add 10 ml of the sample solution (to be analyzed for alkalinity).
3. Add 2-3 drops of methyl orange indicator. The solution will acquire yellow colour.
4. Note the initial reading of the burette and start adding the acid till addition of last drop changes the colour from yellow to pink.
5. Back titration by adding a drop or two of given water sample. If the correct, record the final reading in the observation table.
6. Repeat to get at least three concordant readings.

Observation to be recorded:

Indicator : Methyl Orange

End Point : Yellow to Pink

OBSERVATION TABLE:

S. No.	Volume of sample water taken for titration (ml)	Burette Reading		Volume of H ₂ SO ₄ acid soln. consumed (ml)
		Initial	Final	

Calculation:

Let the concordant volume of H₂SO₄ used = V mL

N₁V₁ of alkaline water = N₂V₂ of standard H₂SO₄

$$N_1 \times 25 = N_2 \times V_2$$

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

Now strength of alkalinity (in ppm in CaCO₃ equivalents)

$$= N_1 \times 50 \text{ gm/L}$$

Results : The total alkalinity of given sample of water was found to be -----
..... ppm.

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Precautions :

1. Use neat and clean apparatus.
2. Before use, rinse burette and pipette properly
3. Find the correct end point by back titration.

Experiment No. 2

Object : Determination of Carbonates, Bicarbonates and Total Alkalinity in water.

Apparatus : Burette, Pipette, Conical flask, Dropper, Measuring Cylinder, Beaker etc.

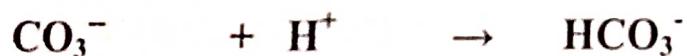
Chemicals: Standard N/50 H_2SO_4 solution, Phenolphthalein, Methyl orange indicator, water sample, etc .

Principle: Alkalinity in water is due to the presence of dissolved carbonates, bicarbonates, hydroxides, and to a lesser extent by borates, silicates, etc. it is measured in terms of titrable base, titrated with standard acid first to a phenolphthalein end point (pH 4.5).Alkalinity refers to the capability of water.

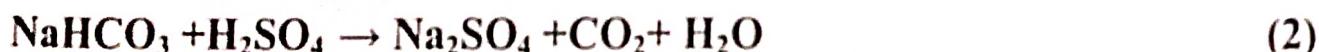
The first titration with phenolphthalein represents the conversion of carbonates to the bicarbonate stage only.



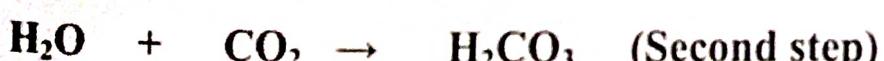
Or



The second titration with methyl orange represents the conversion of bicarbonates to CO_2 and water.



Or



Reagents :

- 1.) Standard Sulphuric acid or hydrochloric acid 0.1N, its alkalinity is very low, use 0.02N solution.
- 2.) Phenolphthalein indicator solution
- 3.) Methyl orange indicator solution

Procedure

If the pH of water sample is above 8.00 then follow the procedure given below-

Pipette out 25 ml (W water) ml of filtered water sample in a conical flask and add 2-4 drops of alcoholic phenolphthalein indicator to it. If the solution turns pink then titrate it with 0.02N HCl until the solution becomes colourless. Let the burette reading be V_1 . To that same solution, which become colourless add 2-4 drops of methyl orange indicator. If the solution becomes yellow then continue titration with 0.02N HCl till orange pink colour is obtained at the end point. Note this burette reading be V_2 as methyl orange end point. Repeat the titration for two more times and find the constant burette reading.

Relation between V_1 & V_2 or P & M	<i>Phenomenal Condition</i>	Alkalinity (ppm)		
		Hydroxide (OH ⁻) alkalinity	Carbonate (CO ₃ ²⁻) alkalinity	Carbonate (CO ₃ ²⁻) Alkalinity
$V_1 = 0$ or $P = 0$	If phenolphthalein end point is zero, then alkalinity is due to bicarbonate.	----	----	M
$V_1 = V_2$ or $P = M$	If methyl orange end point is zero, only there is phenolphthalein end point, then the alkalinity is due to hydroxide alone.	M	---	--

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$V_1 = \frac{1}{2} V_2$ or $P = \frac{1}{2} M$	If phenolphthalein end point is exactly half the total titration, then only carbonate alkalinity is present.	---	2P	---
$V_1 > \frac{1}{2} V_2$ or $P > \frac{1}{2} M$	If phenolphthalein end point is greater than half the total titration, then alkalinity is due to both carbonate & hydroxide.	2P-M	2(M-P)	---
$V_1 < \frac{1}{2} V_2$ or $P < \frac{1}{2} M$	If phenolphthalein end point is less than half the total titration, then alkalinity is due to both carbonate & bicarbonate.	----	2P	M-2P

V_1 are burette readings for Phenolphthalein end point & V_2 burette readings Methyl orange end points respectively. M is total Alkalinity.

Observation to be recorded:

Sample No.	Readings							
	Phenolphthalein end point, $V_1 = P$ (Pink to Colourless)				Methyl orange end point, $V_2 = M$ (Yellow to Orange pink colour)			
	I	II	III	Constant	I	II	III	Constant

Calculations:

All kinds of alkalinity are expressed in terms of CaCO_3 equivalents as parts per million.

$$1000 \text{ ml of } 1\text{N HCl} = 50 \text{ g of } \text{CaCO}_3$$

$$1 \text{ ml of } 1\text{N HCl} = 50 \text{ mg of } \text{CaCO}_3$$

$$V \text{ ml of } 0.02 \text{ N HCl} = 50 \times V \times 0.02 = V \text{ mg of } \text{CaCO}_3$$

V mg of alkalinity is present in W ml (25 ml) of titrated water sample
 The alkalinity is always expressed as **mg/litre or ppm**
 For 1000ml (1 litre) of water sample, alkalinity =

$$= \frac{(V_{HCl} \times N_{HCl} \times 50)1000}{W_{water}}$$

$$(V_{HCl} \times 0.02 \times 50) 1000 / 25 = 40 V_{HCl} \text{ ppm}$$

i.e., 1 part per 10^6 parts or mg / litre

Phenolphthalein alkalinity, P = 40 V_{1HCl} ppm

Methyl orange alkalinity, M = 40 V_{2HCl} ppm

1) Calculation for condition 1: water sample having only bicarbonate alkalinity,

$$V_1 = 0$$

Methyl orange end point, V_{2HCl} = _____ ml

Methyl orange alkalinity, M = 40 V_{2HCl} ppm = _____ ppm

Alkalinity due to HCO₃⁻ = M = _____ ppm

2) Calculation for condition 2:

Water sample having only hydroxide alkalinity, V₁ = V₂

Phenolphthalein end point, V_{1HCl} = _____ ml

Phenolphthalein alkalinity, P = 40 V_{1HCl} ppm = _____ ppm

Alkalinity due to OH⁻ = P = _____ ppm

3) Calculation for condition 3:

water sample having only carbonate alkalinity, V₁ = $\frac{1}{2}$ V₂

Phenolphthalein end point, V_{1HCl} = _____ ml = $\frac{1}{2}$ Methyl orange end point,
 V_{2HCl} = _____ ml

Phenolphthalein alkalinity, P = 40 V_{1HCl} ppm = _____ ppm

Alkalinity due to CO₃²⁻ = 2P = _____ ppm

4) Calculation for condition 4:

water sample having both CO_3^{2-} & OH^- alkalinity,

$$V_1 > \frac{1}{2} V_2$$

Phenolphthalein end point, $V_{1\text{HCl}} = \underline{\hspace{2cm}}$ ml

Phenolphthalein alkalinity, $P = 40 V_{1\text{HCl}}$ ppm = $\underline{\hspace{2cm}}$ ppm

Methyl orange end point, $V_{2\text{HCl}} = \underline{\hspace{2cm}}$ ml

Methyl orange alkalinity, $M = 40 V_{2\text{HCl}}$ ppm = $\underline{\hspace{2cm}}$ ppm

Alkalinity due to $\text{OH}^- = 2P - M = \underline{\hspace{2cm}} = \underline{\hspace{2cm}}$ ppm

Alkalinity due to $\text{CO}_3^{2-} = 2(M - P) = \underline{\hspace{2cm}} = \underline{\hspace{2cm}}$ ppm

5) Calculation for condition 5:

water sample having both CO_3^{2-} & HCO_3^- alkalinity $V_1 < \frac{1}{2} V_2$

Phenolphthalein end point, $V_{1\text{HCl}} = \underline{\hspace{2cm}}$ ml

Phenolphthalein alkalinity, $P = 40 V_{1\text{HCl}}$ ppm = $\underline{\hspace{2cm}}$ ppm

Methyl orange end point, $V_{2\text{HCl}} = \underline{\hspace{2cm}}$ ml

Methyl orange alkalinity, $M = 40 V_{2\text{HCl}}$ ppm = $\underline{\hspace{2cm}}$ ppm

Alkalinity due to $\text{CO}_3^{2-} = 2P = \underline{\hspace{2cm}} = \underline{\hspace{2cm}}$ ppm

Alkalinity due to $\text{HCO}_3^- = M - 2P = \underline{\hspace{2cm}} = \underline{\hspace{2cm}}$ ppm

Results Obtained:

Water sample	Hydroxide (OH^-) alkalinity, ppm	Carbonate (CO_3^{2-}) alkalinity, ppm	Bicarbonate (HCO_3^-) alkalinity, ppm

S.N	volume of acid (CO_3^{2-})	Total HCO_3^- No. of acid	Total hardness $V_b - V_a = v$
1)	22.4	72.4	50
2)	22.4	72.4	50

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Let the concordant volume of H_2SO_4 used = V ml

$N_1 V_1$ of alkaline water = $N_2 V_2$ of standard H_2SO_4

$$N_1 \times 25 = N_2 \times V_2$$

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

Now strength of alkalinity (in ppm in $CaCO_3$ equivalents)

$$= N_1 \times 50 \text{ gm/L}$$

$$= V/25 \times 50 \times 50$$

$$= V/25 \text{ gms/L} = 100 V \text{ mg/L} \quad (1 \text{ gm} = 1000 \text{ mg})$$

Or

$$\text{Total alkalinity of given water sample} = 100V \text{ ppm} \quad (1 \text{ mg/L} = 1 \text{ ppm})$$

Results : The total alkalinity of given sample of water was found to be
 $\text{For } CO_3^{2-}$
.....ppm.

$$N_1 V_1 = N_2 V_2$$

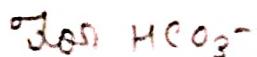
$$N_1 = \frac{N_2 V_2}{V_1}$$

$$= \frac{1}{10} \times \frac{22.4}{25} = 0.896 \text{ ppm}$$

Precautions :

1. Use neat and clean apparatus.
2. Before use, rinse burette and pipette properly
3. Find the correct end point by back titration.

$$\begin{aligned} \text{Strength} &= \frac{22.4}{10 \times 25} \times 60 \text{ ml/litre} \\ &= 5.37 \text{ ml/litre} \end{aligned}$$



$$N_1 V_1 = N_2 V_2$$

$$N_1 = \frac{1}{10} \times \frac{22.4}{25} = 0.896 \text{ ppm}$$

$$= \frac{1}{10} \times \frac{22.4}{25} \times 61 = 54.6$$

$$\text{Total strength} = 5.37 + 3.68 = 40.83 \text{ ppm}$$

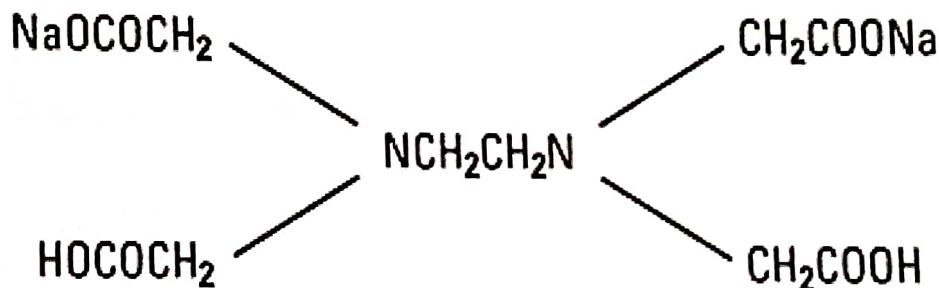
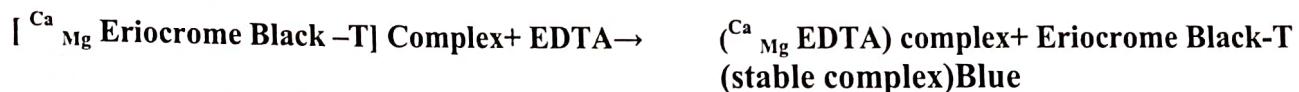
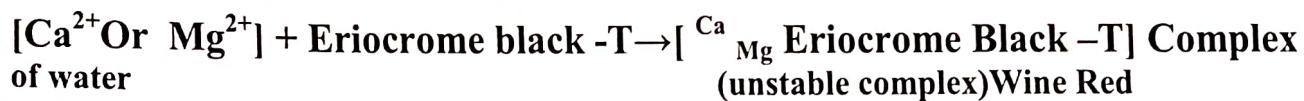
EXPERIMENT NO. 3

Object : Determination of Total hardness of given water sample by Complexometric titration (EDTA method).

Apparatus: Burette, Pipette, Conical flask, Dropper, Beaker etc.

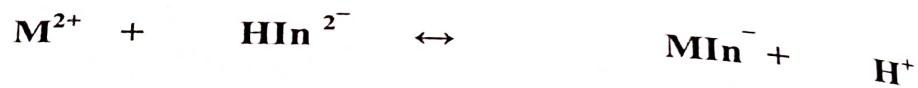
Chemicals: Ethylenediamine Tetra Acetic acid (EDTA) solution, zinc sulphate solution, ammonia buffer of pH 10 & Eriochrome black-T indicator.

Theory: Hardness of water is the soap consuming capacity of water resulting in the formation of white curdy ppt due to the presence of Ca & Mg salts. Disodium EDTA, Na₂EDTA forms 1:1 complex with divalent metal ions like Ca²⁺, Mg²⁺, Fe²⁺, Zn²⁺, etc.

**Disodium-EDTA Structure**

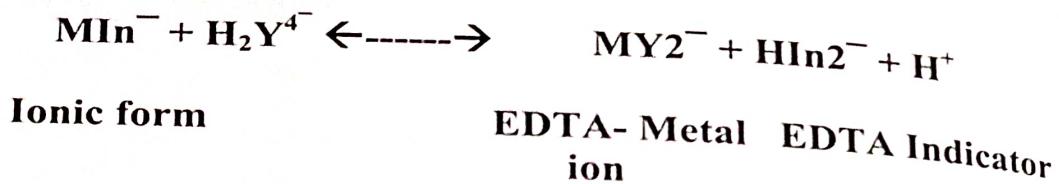
The anion of EDTA (H_2Y^{4-}) is a strong chelating agent, which forms a stable anionic complex with divalent metal ions in basic medium. Hence alkaline buffer of NH₄OH & NH₄Cl of pH -10 is used. In this complexometric titration, Eriochrome black -T is used as an indicator. The indicator forms unstable wine red coloured complex with metal ions, which dissociate on titration with EDTA

solution. On dissociation, a strong metal ion - EDTA complex is formed & indicator is set free which gives blue colour at the end.



Metal ion Indicator Metal-Indicator complex

Blue Wine red



Ionic form Complex Blue

Ca/Mg + EDTA

→ M-EDTA

Where = M = Ca / Mg

The following table gives relation between the type of water sample & the degree of hardness

Nature of water	Hardness in ppm (CaCO ₃ equivalent)
Soft	Below 50 ppm
Moderately hard	50-150 ppm
Hard	150-300 ppm
Very hard	Above 300 ppm

Procedure:

1. Take 25 ml of the given water sample in a clean conical flask.
2. Add 2ml solution of NH₃-NH₄Cl buffer solution .
3. Add 2-3 drops of Eriocrome black -T indicator.
4. Titrate against EDTA solution till the colour of the solution change from wine red to clear blue. Repeat the titration to get concordant values.

Observations

Molarity of EDTA solution prepared = 0.1 M

Volume of hard water sample taken in taken flask = 25ml

Observation:

S.No.	Volume of sample solution taken (ml)	Burette Reading		Volume of standard EDTA solution used (ml)
		Initial	Final	
1.)	25 ml	0	9.5 ml	
2.)	25 ml	9.5 ml	18.3 ml	18.7
3.)	25 ml	18.3 ml	28.4 ml	

Let the concordant volume used = V ml

$$N_1 V_1 = N_2 V_2$$

$$N_1 = \frac{N_2 V_2}{V_1} = \frac{0.1 \times 18.7}{25}$$

$$\text{Strength} = N \times E = \frac{0.1 \times 18.7}{25} \times 20.04 = 1.438 \text{ ppm}$$

Calculations:

1000 ml of 1 M EDTA = 100 gm of CaCO_3 (mol mass of CaCO_3 = 100)

1ml of 0.1M EDTA = 10 mg CaCO_3

V ml of 0.1 M EDTA = 10 V mg CaCO_3

Or 25ml of hard water solution = 10V mg CaCO_3

So, 1000 ml of hard water solution = $\frac{10 \times V \times 1000}{25}$ mg CaCO_3
= 400 V mg CaCO_3

Thus, total hardness is 400Vmg/ L of 400 V ppm of CaCO_3 .

Results:

Total hardness of given water sample is ppm.

Precautions:

1. Use distilled water for washing and rinsing of glass apparatus.
2. Prepare EDTA solution in double distilled water.
3. Add same amount of indicator each time.
4. Maintain pH 10 during the titration by adding buffer.
5. Correctly observe the end point.

Object : Preparation of Urea formaldehyde (UF)resin .

Apparatus : Dropper, Measuring cylinder, Beaker , Test tube , glass rod, separating funnel, waterbath etc.

Chemicals : Urea, 40% formaldehyde & concentrated Sulphuric acid.

Theory: **Urea-formaldehyde**, also known as **urea-methanol**, named so for its common synthesis pathway and overall structure, is a thermosetting resin or polymer, made from urea & formaldehyde. The reaction involves condensation between the nucleophilic nitrogen of urea with the electrophilic carbonyl carbon of formaldehyde. At first dimethylol urea is formed which further reacts with excess of urea to form a water soluble branched copolymer.

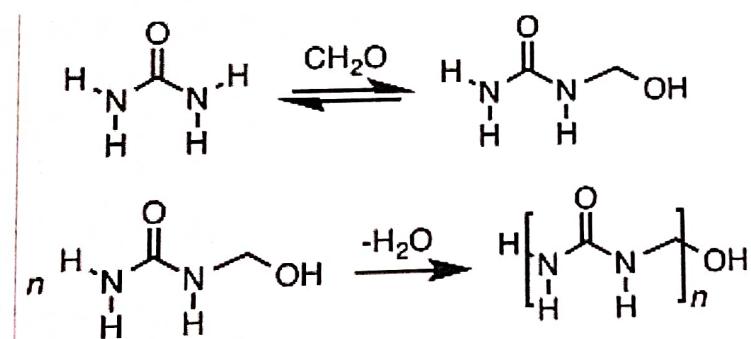


Fig . 4.1

Procedure :

- 1.) Take 20 ml of 40 % formaldehyde solution in 100 ml beaker & add about 10 gm of urea with stirring until a saturated solution is obtained.
- 2.) Then add a few drops of Conc. H_2SO_4 with stirring continuously. A white, hard, solid mass is formed in the beaker.
- 3.) Wash the product with water to remove surface acid and dry it. This dry product is pure urea-formaldehyde resin.
- 4.) Alternatively mild alkalis can also be used instead of strong acid.
- 5.) Take 20 ml of 40 % formaldehyde solution in 100 ml beaker & add 10 gm of urea with stirring until a saturated solution is obtained.
- 6.) Then add few drops of pyridine or ammonia & heat it on a water bath till complete solidification occurs.

Properties:

- 1) UF resins are infusible, insoluble and non-inflammable but soluble in water.
- 2) They are resistant towards heat, scratch, acids, alkalies & many organic solvents.
- 3) They exhibit high thermal stability, excellent electrical insulating & adhesive properties.

Moulding techniques: Compression moulding, die casting (tubes, rods) & extrusion.

Applications:

- 1) Used for making buttons, bottle caps, cosmetic container closures etc.
- 2) As a binder of glass fibres, rock wool & plywood. Also used for electrical insulation (switches, boards, desk lamp casing, etc.)
- 3) Also used in agriculture as a controlled release source of nitrogen fertilizer.

Result: Yield of polymer=-----g

Object : Preparation of resol type Phenol - Formaldehyde (PF) resin:

Apparatus: Conical flask, air condenser, steam bath, thermometer, measuring cylinder, Beaker, etc.

Chemicals: Phenol, 40% formaldehyde (formalin) & conc. aqueous ammonia solution.

Theory: Phenol is reactive towards formaldehyde at the *ortho* and *para* sites (sites 2, 4 and 6) allowing up to 3 units of formaldehyde to attach to the ring. This forms a hydroxymethyl phenol. The hydroxymethyl group is capable of reacting with either another free *ortho* or *para* site, or with another hydroxymethyl group. The first reaction forms a methylene bridge, and the second forms an ether bridge. Phenol formaldehyde resins, are formed by step growth polymerization reaction which may be either acid or base catalysed.

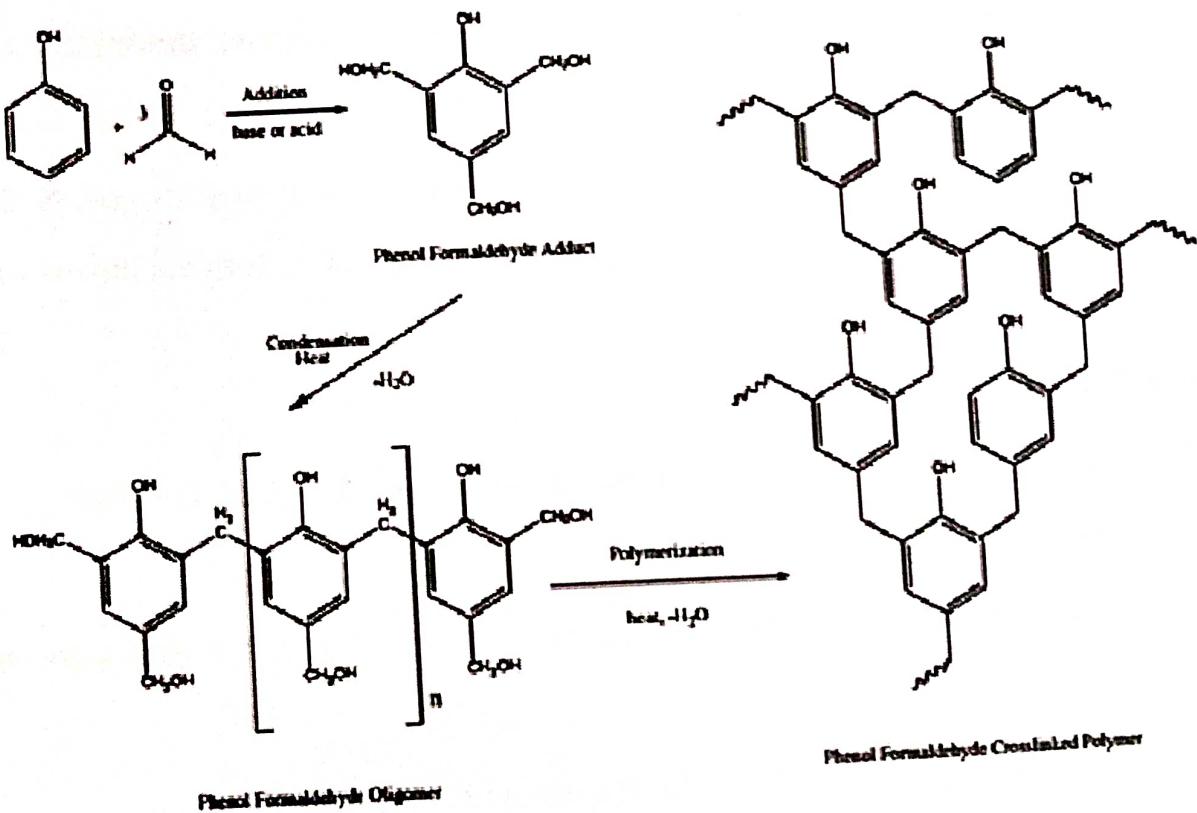


Fig 5.1

Base catalysed phenol formaldehyde resins are made with formaldehyde to phenol ratio of greater than one (around 1.5). Phenol, formaldehyde, water and catalyst are mixed in the desired amount, depending on the resin to be formed, & are then heated. The first part of the reaction, at around 70°C, forms hydroxymethyl phenols. This results in a thick reddish-brown goo, the resin. The negative charge is delocalized over the aromatic ring, activating sites 2, 4 & 6, which then react with formaldehyde to form hydroxymethyl phenols. Hydroxymethyl phenols will crosslink on heating to around 120°C to form methylene and methyl ether bridges. At this point the resin is starting to crosslink, forming highly extended 3-dimensional web of covalent bonds which is typical of polymerized phenolic resins.

Procedure: Take 100 mL conical flask with ground glass joint & transfer 25 mL molten phenol, 30 mL formalin & 5 mL concentrated Hydrochloric acid into it using measuring cylinder. Then heat it on a steam bath for 5 minutes at about 70°C. Frequently shake the reaction mixture during heating to maintain uniformity. After 5 - 7 mins. two layers are formed. Thick Pink gum the resin (resol) is formed. Separate the two layers while hot. Further heating of resol in presence of 8-10 drops of ortho phosphoric acid & 5 ml phenol at about 120°C forms a cross-linked resin (Bakelite).

Properties:

- 1) Resols are soluble in ethanol or acetone but cross-linked product is infusible, insoluble and non-inflammable.
- 2) PF resin is resistant towards heat, scratch, acids, many solvents salts and moisture.
- 3) But it is attacked by alkalis due to the presence of -OH group.
- 4) Are thermally stable upto 250°C & possess excellent electrical insulating & adhesive properties.

Moulding technique: compression moulding, die casting (tubes, rods) & extrusion.

Applications:

- 1) Resol (methylol phenols) is used as varnish & lacquer for making of laminates.
- 2) PF is used in electrical circuits & switches, in automobile parts & for making moulded articles like telephones.
- 3) Also used as an adhesive for grinding wheels & break linings & in making sand paper.

Result: Yield of Polymer is _____ gm

Object : Determination of saponification value of fat or oil

Apparatus : Burette, Pipette, Conical flask, Conical Flask of 200mL ,Beaker, Water bath etc.

Principle: “Saponification number of a fat or oil is defined as the number of milligrams of KOH required to completely saponify one gram of oil or fat”. The saponification reaction may be represented as follows :

Saponification refers to the hydrolysis of ester with NaOH or KOH. For example, triglyceride (derived from glycerol and fatty acid), an ester, undergoes hydrolysis with KOH to produce potassium salt of soap and glycerol. The process of saponification gives salts of the fatty acid.

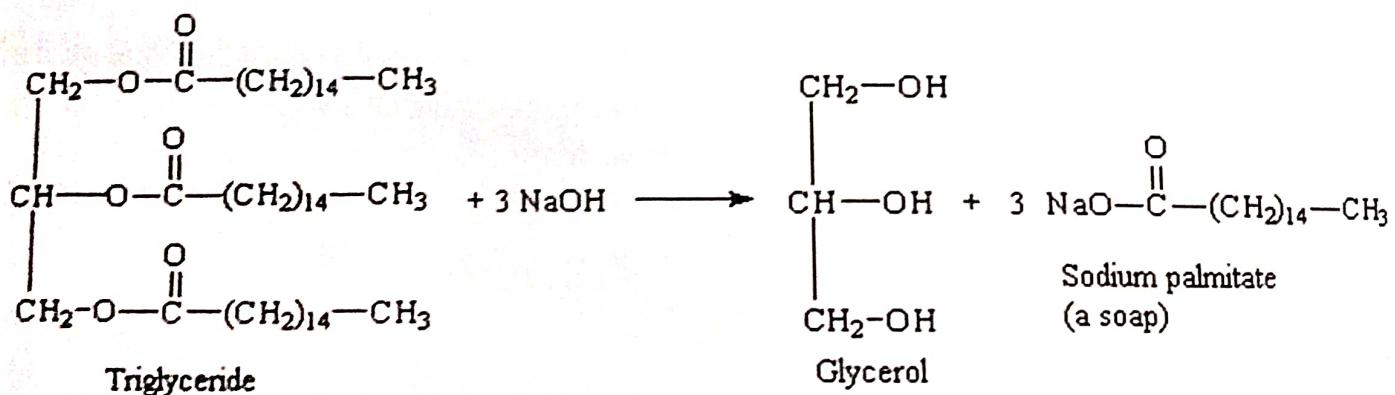


Fig.6.1.Mechanism of saponification reaction

Saponification is alkaline hydrolysis of ester, in which the hydroxyl group of the alkali acts as a nucleophile. At first, the nucleophile attacks the carbonyl carbon of the ester and a negative charge is generated on the carbonyl oxygen atom. Then the negative charge goes back to its original position to produce fatty acid, and the leaving group, alkoxide, is removed. Then the fatty acid gets deprotonated to produce salt of fatty acids or soap and alcohol. Thus, saponification is a hydrolytic

reaction. From the above reaction it is evident that one molecule of the fat requires three molecules of KOH for saponification irrespective of the size of the fat, molecule. Therefore, smaller the molecular weight of the fat, greater the saponification number and vice-versa.

Reagents:

1. Alcoholic KOH : (40g/l) should be aldehyde free . Prepare by refluxing 1 to 2 lit of 95 percent KOH and 6gm of aluminum foil for 30 minutes . Distilled by heating on a steam bath. Discard the first 50 ml of the distillate and collect about 1000 ml and dissolve 40 gm of KOH in it. Preserve the solution in bottle kept in the dark.
2. Standard HCl or H_2SO_4 0.05 N
3. Phenolphthalein indicator solution – one percent in ethyl alcohol.

Procedure

1. Weigh about 2g of the butter sample and add 25 mL of alcoholic KOH solution to it.
2. Heat it on water bath with proper care.
3. Boil the sample for 5min. After boiling allow it to cool down.
4. Fill the burette with standard HCl solution. Titrate the sample against standard HCl solution with phenolphthalein as an indicator.
5. Conduct a blank reading along with it. The end point is colorless or disappearance of pink colour due to the formation of soapy solution.

Calculation:

$$\text{Saponification Value} = \frac{(b-a) \times \text{normality} \times 56}{\text{wt.of oil in sample}}$$

Where a = burette reading of the sample; b = blank reading

(a) = Burette Reading b = blank reading

S.No.	Initial Reading (mL)	Final Reading (mL)	Final Volume consumed(b-a)
1	0	48.6	48.6
2	0	50.4	50.4
3	0	49.2	49.2
4	0	42.2	42.2

Result: The value of saponification of oil/Fat is

Supply not there

Experiment No. 7

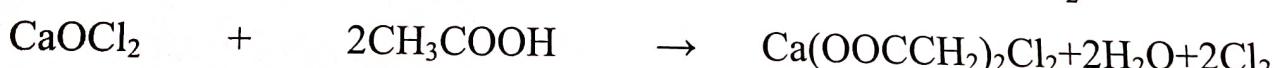
Object : Determination the percentage of available chlorine in a given sample of bleaching powder (Iodometrically).

Apparatus : Burette, Pipette, Measuring flask, Conical Flask of 200ml ,Beaker, weighing bottle etc.

Chemicals: Bleaching Powder, Potassium iodide , Glacial acetic acid , sodium thiosalphate solution, starch solution etc.,

Principle : Bleaching powder is famous for its bleaching action . it is used as disinfectant. The main constituents of bleaching powder are calcium hypo chloride $[\text{CaOCl}_2 \cdot 4\text{H}_2\text{O}]$, basic calcium chloride $[\text{CaCl}_2\text{Ca}(\text{OH})_2\text{H}_2\text{O}]$ and the some free calcium hydroxide $\text{Ca}(\text{OH})_2$. Out of these constituents, the active constituent of bleaching powder is hypochloride . Calcium hypochloride, CaOCl_2 may be represented as $\text{Ca}(\text{OCl})\text{Cl}$

When dilute acid reacts with bleaching powder then free chlorine is liberated, according to one of the following reactions:

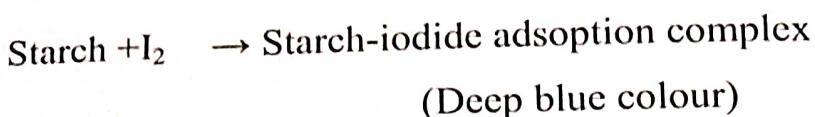
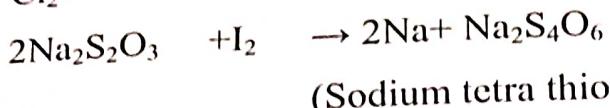


The amount of the chlorine that can be obtained by the action of dilute acid on bleaching powder is known as available chlorine. It is expressed in percentage weight of bleaching powder.

The liberated chlorine in water forms hypochlorous acid (HOCl) which is responsible for bleaching action and disinfection.



The available chlorine is estimated by iodometric method. In this method, a suspension of bleaching power is treated with dilute acid in the presence of KI solution. The liberated chlorine from bleaching powder sets free an equivalent amount of iodine from KI, which is titrated against hypo solution (i.e., sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) solution.



All such titrations in which liberated iodine is titrated with hypo solution are known as iodometric titration . In such titrations, freshly prepared starch solution is used as indicator.

Procedure:

i. For preparation of bleaching powder solution -

1. Weigh about 1gm of bleaching powder in a clean and pre weighed weighing bottle .
2. Transfer it into a mortar, crush with pastle and little water.
3. Make a thin paste of the powdered bleaching powder sample with water.
4. Transfer the paste inti 250 ml volumetric flask using small quantities of water.
5. Wash the mortar and pastle with distilled water. Now transfer the water (used for washing) to volumetric flask.
6. After transferring all the bleaching powder to volumetric flask , add more water to make the solution upto the mark.
7. Shake, until a homogeneous suspension is obtained .

ii. Estimate the available chlorine in bleaching powder by the following method :

1. Fill the clean burette after rinsing with the hypo solution, note down the initial reading of the burette .
2. Similarly first rinse the pipette with the homogeneous solution of bleaching powder . Then transfer 25ml of it, by pipetting out , in conical flask.
3. Add about 20 ml KI crystal or 10 ml of KI solution and about half test tube of glacial acetic acid. Solution turns brown.
4. Titrate the liberated iodine against sodium Thiosulphate (Hypo) solution till a light yellow colour persists.
5. Now add 5-10 drops of freshly prepared starch solution. Solution turns to blue or violet colour.
6. Continue adding hypo solution till blue colour disappears. This is the end point. Note down the total volume of hypo solution added.
7. Repeat the same procedure of titration to get concordant reading.

Observations

- (a) Weight of empty weighing bottle = w_1 gm
(b)Weight of weighing bottle with bleaching powder = w_2 gm

So,

$$(c) \text{Weight of bleaching powder} = (w_2 - w_1) \text{ gm}$$

(b) Titration of bleaching powder sample with standard sodium thiosulphate (Hypo) solution

S.N.	Volume of bleaching powder solu. Taken (ml)	Burette reading		Volume of hypo solu. Consumed (ml)
		Initial	Final	
1)	25	0	37.8	37.8
2)	25	0	41.8	41.8
3)	25	0	40.6	40.6
4)	25	0	39.2	39.2

Let the volume of hypo solution consumed for 25mL bleaching powder solution

$$= V_H \text{ (ml)}$$

Calculation :

(a) Calculation of Normality and strength of bleaching powder :

From the law of chemical equivalents,

$$(N_1 V_1) \text{ of hypo solution} = (N_2 V_2) \text{ of bleaching powder solution}$$

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

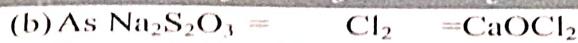
$$N_1 V_1 = N_2 V_2$$

$$N_1 \times 25 = \frac{1}{10} \times 41.8$$

$$N_1 = \frac{41.8}{10 \times 25 \times 10} = 0.1676$$

$$\begin{aligned} \text{Strength of Cl} &= N \times \text{Eq wt} \\ &= 0.167 \times 35.5 \\ &= 5.94 \text{ g P} \end{aligned}$$

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So, available chlorine (amount of chlorine per litre of solution)
 $= N_2 \times 35.5 \text{ gm/lit}$

(c) Percentage of available chlorine

$$= (N_2 \times 35.5) \times 250 / 1000 \times 1 / (w_1 - w_2) \times 100$$

Result : The percentage of available chlorine in a given sample of bleaching powder = 59.49 %.

Precautions

1. Use distilled water in a titration .
2. All the reagents should be freshly prepared .
3. The volume of starch indicator should be same in all the titrations.
4. Use neat and clean glass apparatus.

$$\% = \frac{1}{10} \times \frac{41.9 \times 10}{25 \times 100} \times 35.5$$

$$= 59.49 \%$$

Object: Estimation of pH of acid and base using pH meter

Theory: Sorenson expressed the H^+ concentration in solution in terms of pH which is defined as negative power to which ten must be raised to express H^+ ion concentration.

$$H^+ = 10^{-pH}$$

$$\log H^+ = \log 10^{-pH}$$

$$pH = -\log[H^+]$$

When a solution reacts acidic, its H^+ concentration is more than 10^{-7} moles/lt thus pH less than 7. Similarly, if a solution is alkaline, its pH is more than 7. These different values of pH from 0-14 form a scale commonly called as pH scale.

Chemicals required: Sulphuric acid (0.1N), Sodium Hydroxide (0.1N), Buffer 4, 7.

Procedure: Calibrate the pH meter using the buffer samples of 4 and 7. The buffer is prepared by dissolving the capsule of buffer 4 and 7 in 100 mL distilled water separately in volumetric flask. Firstly the pH meter is calibrated by using buffer 4 and then by buffer 7. Then the sample of Sulphuric acid is taken in the beaker and pH meter is dipped and reading is noted; after that solution of sodium hydroxide is taken in the beaker and pH meter is dipped and reading is noted. The readings are reported and verified using pH paper also.

Result:

Experiment No. 9

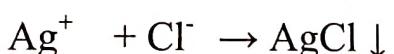
Object: To find out the amount of chloride ions presents in water using silver nitrate (AgNO_3) (Potassium chromate as indicator) .

Apparatus: Burette, Pipette, titration flask, Dropper, Measuring Cylinder, Beaker etc.

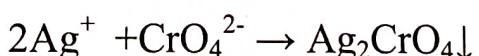
Chemicals: Standard N/50 AgNO_3 solution, K_2CrO_4 , water sample, etc .

Principle: In Mohr's methods, the indicator employed is a solution of potassium chromate. When silver nitrate solution is added to a given solution of chloride containing few drops of indicator ($\text{K}_2\text{Cr}_2\text{O}_4$), when AgCl is precipitated initially. The moment all the chloride ions have been precipitated out, even a drop of silver nitrate (AgNO_3) added in excess gives a brick-red precipitate of silver chromate. This indicates the end point.

Because $K_{sp}(\text{AgCl}) < K_{sp} (\text{Ag}_2\text{CrO}_4)$, hence as long as the chloride ions are available, the soluble silver chloride is precipitated . as soon as all chlorides ions have been precipitated out, even a slight excess of Ag^+ produces insoluble silver chromate which is red in colour.



White ppt.



Brick red ppt.

Procedure

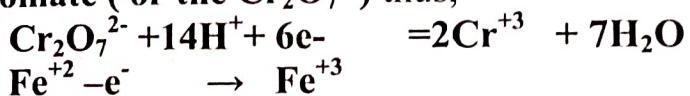
1. Fill the washed and rinsed burette with AgNO_3 (N/50) solution.
2. Wash the pipette with water and then rinse it with chloride solution .
3. Pipette out 25 ml. of chloride solution in the clean titration flask.
4. Add 3-4 drops of K_2CrO_4 in indicator .

Object : Determination of iron by Potassium Dichromate ($K_2Cr_2O_7$)

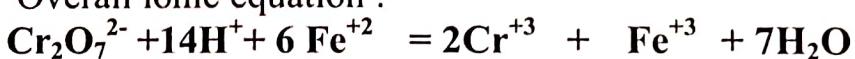
Apparatus: Burette, Pipette, Volumetric flask, Porcelain plate or a glass plate a white paper placed underneath, glass rod preferably taping at one end, Dropper, Measuring Cylinder, Beaker etc.

Chemicals: 0.1N Standard Potassium Dichromate solution, Potassium ferricyanide Indicator- A 5 percent solution prepared after washing crystals with distilled water to remove any ferrocyanide formed , the resulting solution being pale yellow .

Principle: The method is based on the oxidizing action of potassium dichromate (or the $Cr_2O_7^{2-}$) thus,



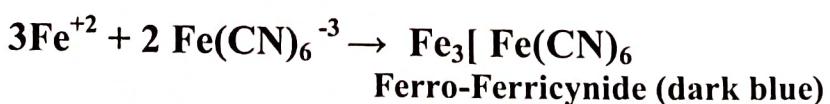
Overall ionic equation :



In the presence of Sulphuric acid the overall reaction being



Since the titration gives rise to Cr^{3+} ions which colour the solution green, the equivalence point is difficult to detect, hence potassium ferricyanide $K_3[Fe(CN)_6]$ is used as indicator for the Fe^{2+} ions with which it forms a dark blue colour of complex ferro-ferricyanide.



The indicator is not added directly to the titration solution but small drop of titration solution withdrawn and is added to indicator drops placed on a white

opaque surface. Here the indicator is called an external indicator since it is not added directly to the titration solution.

Procedure :

1. The unknown iron solution would be usually supplied in a volumetric flask. Dilute it with distilled water upto the mark and mix thoroughly.
2. Fill the burette with the standard $K_2Cr_2O_7$ solution after rinsing it as usual.
3. Take a white, dry porcelain tile or a glass plate with a white paper placed underneath and smear a very little grease over its surface. Places several drops of indicator solution on the plate with the help of a dropper.
4. Take two beaker, in one fill tap water and in other fill distilled water.
5. Pipette out 20 ml aliquot of the unknown diluted solution and transfer it in a beaker.
6. Add 10 ml dilute H_2SO_4 and start the titration.
7. During the first titration, after adding 5ml portion of $K_2Cr_2O_7$ solution, stir the titration solution well with glass rod. Take out the glass rod without taking the drop and bring it in contact with the drop of indicator placed on the plate. If blue colours appears in indicator add more $K_2Cr_2O_7$ until blue colour is no longer formed. After touching the glass rod with indicator drop, wash the rod with tap water and then with distilled water before replacing it in the beaker. Record the reading which gives faintest blue colour.
8. Carry out the second titration with fresh aliquots of solution . add 10 ml Dil H_2SO_4 solution and then add $K_2Cr_2O_7$ solution closure volume to the end point (from the first point reading) and after adding 0.5 ml check the end point (faintest possible blue colour) . The brownish colour in the indicator drop indicates the excess of $K_2Cr_2O_7$

9. Carry out third titration similarly and add $K_2Cr_2O_7$ close to the end point previously reading and after adding one drop of $K_2Cr_2O_7$, check the end point .Confirm the end point with fourth titration.
10. Consider the reading obtained in the forth titration for calculation.

Observation:

S.No.	Volume chloride solution taken (ml)	Burette Reading		Volume of Potassium dichromate soln used (ml)
		Initial	Final	
.....

Observation to be recorded: The volume of Potassium dichromate is ml

Calculations: From the reaction given in the principle , it is clear that one molecule of $K_2Cr_2O_7$, accepts 6 electrons .

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The gm-equivalent of $K_2Cr_2O_7$ is one sixth of its gm molecule.

While the gm equivalent of $FeSO_4$ is the same its gm -molecule weight.

1gm of equivalent of $K_2Cr_2O_7$ = 1gm of equivalent of $FeSO_4$

Since 1ml of N $K_2Cr_2O_7$ = 0.05585 gm Fe^{+2} (factor)

The amount of the iron in given solution = factor $X V_1 X V_2 / A$

Or

$$Amt = \frac{V_1 \times N \times 0.05585 \times 100}{A}$$

$$= \text{-----} \text{gm}$$

Where V_1 = Volume of $K_2Cr_2O_7$ (burette reading)

V_2 = Volume to which the unknown solution diluted in volumetric flask

N = Normality of $K_2Cr_2O_7$ (0.1N)

A = Aliquots of the diluted solution taken for titration.

Results :

Aim: To determine moisture, volatile matter and ash content of a given sample of coal

Apparatus: Silica crucible with vented lid, electric oven, Muffle furnace, spatula, desicator, pair of tongs, weighing balance, long legged tongs, 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 coking, power generation or for iron ore smelting in the manufacture of steel.

Moisture: Moisture held within the coal itself is known as inherent moisture & is analyzed. Moisture reduces the calorific value of coal and considerable amount of heat is wasted in evaporating it during combustion. Moisture content should be as low as possible.

Volatile matter: Volatile matter is usually a mixture of short & long chain hydrocarbons, aromatic hydrocarbons and some sulfur. Volatile matter of the coal is related to the length of the flame, smoke formation & ignition characteristics. High volatile matter coal gives long flame, high smoke & relatively low heating

values. Volatile matter content should be low but minimum 20% is required for the ignition of coal.

Ash: Ash content of coal is the non-combustible residue left after coal is burnt. It consists of inorganic matter like silica, alumina, iron oxide, lime, magnesia, etc. Ash reduces the heating value of coal, reduces air supply in furnaces and also requires labour (extra cost) for its regular disposal. Therefore ash content of coal should be as low as possible.

Fixed carbon: The fixed carbon content of the coal is the carbon found in the material which is left after volatile materials are driven off. More the fixed carbon content, higher will be the calorific value of coal.

Procedure: A. 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 $105 - 110^{\circ}\text{C}$ 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.

Determination of Volatile matter: The dried sample of coal after determining moisture content is closed with a vented lid. The closed crucible is then heated in a Muffle furnace maintained at $925 \pm 20^{\circ}\text{ C}$ for exactly 7 minutes. The crucible is

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taken out from Muffle furnace carefully with the help of long legged tongs. It is first cooled in air and then in a desiccator. When the crucible attains room temperature it is weighed. Calculate the loss in weight.

C. Determination of Ash: Transfer about 1g (known quantity) of powdered air dried coal sample into a previously weighed silica crucible. Place the open crucible with sample in a Muffle furnace maintained at $725 \pm 25^\circ\text{C}$ for about 40 minutes or till constant weight is obtained. Coal burns in open and the residue left is ash. Take out the crucible from Muffle furnace carefully using long legged tongs. Cool the hot crucible first in air and then in a desiccator. Weigh the crucible and find out the amount of unburnt residue left (ash).

D. Determination of Fixed Carbon: The percentage of fixed carbon is determined indirectly by subtracting the sum total percentages of moisture, volatile matter & ash from 100.

Observations and Calculations: (All weights are in grams, g)

A. For Moisture:

1. Weight of empty crucible = W_1 = _____ g.

2. Weight of crucible + Coal sample = W_2 = _____ g.

3. Weight of Coal sample before heating = $W_3 = W_2 - W_1 = \underline{\hspace{2cm}}$ g.

4. Weight of crucible + Sample after heating for 1 hr at $105 - 110^\circ\text{C}$ = $W_4 = \underline{\hspace{2cm}}$ g.

5. Weight of Coal sample after heating $W_5 = W_4 - W_1 = \underline{\hspace{2cm}}$ g.

6. Loss in weight of sample due to moisture $WM = W_3 - W_5 = \underline{\hspace{2cm}}$ g

OR

$$W_2 - W_4 = \underline{\hspace{2cm}} \text{ g.}$$

$$\% \text{ Of Moisture in coal} = \frac{WM}{W_3} \times 100$$

B. For Volatile matter:

1. Weight of empty crucible = W_1 (W_1 from part A) = $\underline{\hspace{2cm}}$ g.

2. Weight of crucible + moisture free coal sample = W_2 (W_4 from part A) = $\underline{\hspace{2cm}}$ g.

3. Weight of moisture free sample (before heating) = $W_2 - W_1 = W_3 = \underline{\hspace{2cm}}$ g.

4. Weight of crucible + Sample after heating for 7 mins. at $925 \pm 20^\circ\text{ C}$ $W_4 = \underline{\hspace{2cm}}$ g.

5. Weight of Sample after heating $W_5 = W_4 - W_1 = \underline{\hspace{2cm}}$ g.

6. Loss in weight of sample due to volatile matter $WVM = W_3 - W_5 = \underline{\hspace{2cm}}$ g.

$$\% \text{ Of Volatile matter in coal} = \frac{WVM}{W3} \times 100$$

C. For Ash:

1. Weight of empty crucible = W1 = _____ g.

2. Weight of crucible + Coal sample = W2 = _____ g.

3. Weight of Coal sample (before heating) W3 = W2 - W1 _____ g.

4. Weight of crucible + Sample after heating for 40 mins. at $725 \pm 25^\circ \text{C}$ W4
= _____ g.

5. Weight of Ash formed WA = W4 - W1 = _____ g.

$$\% \text{ Of Ash in coal} = \frac{WA}{W3} \times 100$$

D. For Fixed carbon (FC):

$$\% \text{ Of Fixed Carbon} = 100 - (\% \text{ Moisture} + \% \text{ Volatile matter} + \% \text{ Ash}) = 100 - ($$

$$_____)$$

$$= 100 - _____ = _____$$

% Results Obtained: The coal sample contains:

Moisture = _____ %

Volatile matter = _____

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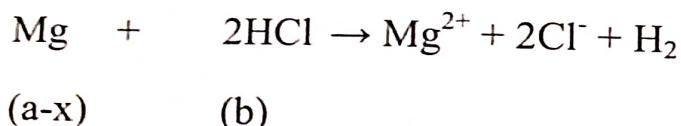
% Ash = _____ %

Fixed Carbon = _____ %

Experiment No. 12

Object: To study the kinetics of dissolution of Mg in dilute HCl

Principle: the reaction between Mg and HCl is represented as follows:-



Where **a** = initial concentration of magnesium for each set of experiment before start of reaction i.e. at $t = 0$

x = amount of magnesium observable at $t = t$ in HCl

(a-x) = the minimum amount of magnesium observable at $t = t$,

b = Concentration of HCl used in excess, which virtually remains constant for each set of experiment

The concentration of HCl is in large excess so that the order determined will be with respect to magnesium, the rate equation for the reaction will be –

$$\frac{dx}{dt} = k(a-x)b^2$$

Where, the integration of above differential equation will lead to the following expression.

$$\ln \left(\frac{a}{a-x} \right) = kb^2t$$

Since, **a** is the constant and **(a-x)** is also assumed to be constant for each set of experiment, it follows that

$$Kb^2t = \text{constant}$$

or

$$B^2t = \text{constant}$$

or

$$B^2a 1/t$$

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Apparatus: five beakers each of 250 ml capacity, 100ml measuring cylinder, stop watch, conc. HCl, 15 inch Mg ribbon in length.

Procedure: Prepare (1:1) Conc. HCl take 30 ml, 25ml, 20ml, 15ml, and 10ml of (1:1) HCl into five different beakers. Make the volume of the acid to 60 ml by adding 30ml of distilled water in the first beaker, 35ml in second beaker, 40ml in third beaker, 45ml in forth beaker and 50ml in fifth beaker to change the concentration of HCl. Cut Mg wire into five equal parts each of 3inch in length. Add one piece of the wire in the first beaker and start the stop watch immediately. Note the time taken to dissolve Mg completely. Similarly note the time to dissolve Mg completely in other sets of experiments.

Observation to be recorded:

S.No	Vol. of HCl before diluting	Vol. of Distill Water	Total Volume	Time (second)	
1	b	b^2	60	10.68	0.083
2	30	900	60	14.53	0.069
3	25	625	60	16.5	0.060
4	20	400	60	17.7	0.056
5	15	225	60	29.57	0.033
	10	100	60		

Result: A graph is plotted between (vol. of HCl)² or b^2 on y-axis and $1/t$ on x-axis. A straight line passing through the origin shows that the dissolution of magnesium in HCl follows the second order kinetics.

Double reciprocal plot

Dimensional analysis

Aman Chaurasiya

22B7CSE073

Scale : X axis = 2 cm = 0.018^{-1}

Y axis = 2 cm = 1.0 cm^{-2}

