

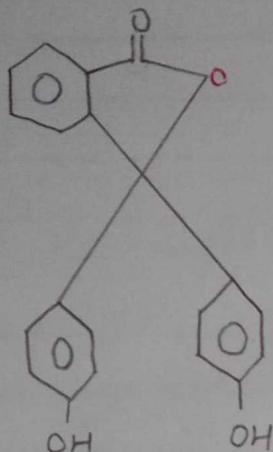
Experiments Performed

S.NO.	PARTICULARS	DATE	PAGE	REMARKS
1.	To determine alkalinity of water sample.	23/11/2020	1-3	Room 7/11/20 (1)
2.	To determine coefficient of viscosity of given liquid by Ostwald's viscometer.	24/11/2020	4-6	Room 7/11/20 (1)
3.	To determine viscosity of lubricant by redwood viscometer.	24/11/2020	7-8	Room 7/11/20 (2)
4.	To determine flash and fire point of an oil by Pensky - Marten's flash point apparatus.	25/11/2020	9-10	Room 7/11/20 (1)
5.	To determine temporary and permanent hardness of given water sample by EDTA method.	3/12/2020	11-14	Room 7/12/20 (2)
6.	To determine Ca^{+2} and Mg^{+2} hardness of water using EDTA solution.	4/12/2020	15-17	Room 7/12/20 (2)
7.	To determine amount of total iron in an iron ore solution by internal indicator method.	14/12/2020	18-20	Room 24/12/20 (2)

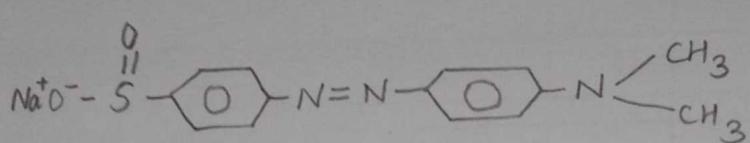
Experiment - I

SIM - To determine alkalinity of water sample.

Indicators - i) Phenolphthalein
ii) Methyl orange



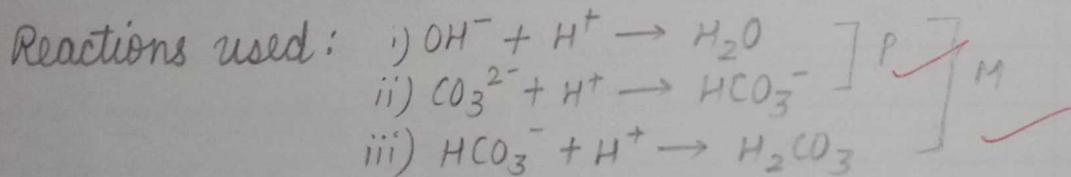
(i)



(ii)

End Point - i) Pink to colourless with Phenolphthalein
(Acid in burette)

ii) Colour change from light yellow to reddish orange
with methyl orange.



P → phenolphthalein alkalinity

M → total or methyl orange alkalinity

$$P = \text{OH}^- + \frac{1}{2} \text{CO}_3^{2-}$$

$$M = \text{OH}^- + \text{CO}_3^{2-} + \text{HCO}_3^- \quad (\text{all ions})$$

$M - P =$ alkalinity due to HCO_3^- ions

Experiment - 1

Aim - To determine alkalinity of water sample.

Reagents Required - Standard acid (say N/50 HCl)
or H_2SO_4

Indicators - 1) Phenolphthalein
2) Methyl Orange

End Point - 1) Pink to colourless with phenolphthalein (Acid in burette)
2) Colour change from light yellow to reddish orange with methyl orange (Acid in burette)

Theory - The type and amount of alkalinity in water can be determined by titrating a known volume of it against standard acid using two indicators namely phenolphthalein and methyl orange respectively.

Volume of acid upto phenolphthalein end point (A ml) neutralizes all OH^- ions and converts carbonate ions into bicarbonate ions (i.e half neutralization of CO_3^{2-}). The end point using disappearance of pink colour at a pH 8.3. The acid used in further titration using methyl

Teacher's Signature.....

From the calculated values of P and M, following correlations can be made:

S.N.O.	case	Alkalinity (in ppm)		
		OH^-	CO_3^{2-}	HCO_3^-
1.	$P=0$	0	0	M
2.	$M=P$	$P \text{ or } M$	0	0
3.	$P=\frac{1}{2}M$	0	$2P \text{ or } M$	0
4.	$P > \frac{1}{2}M$	$2(P-M)$	$2(M-P)$	0
5.	$P < \frac{1}{2}M$	0	$2P$	$M-2P$

Observation table -

S.N.O.	Initial Reading (x)	Final phenolphthalein (P)	Reading Methylorange (M)	Vol. of acid used	
				$P-x$	$M-x$
1.	0	2	6.1	2	6.1
2.	0	2	6.1	2	
3.	0	2	6.4	2	6.4

Concordant volume of acid used upto phenolphthalein end

$$\text{pt. A} = A \text{ ml} = P-x = 2 \text{ ml}$$

Concordant volume of acid used upto methyl orange end

$$\text{pt.} = T \text{ ml} = M-x = 6.1 \text{ ml}$$

General Calculations -

I) For finding phenolphthalein alkalinity - (P) -

$$\text{As } N_1 V_1 = N_2 V_2 \Rightarrow N_1 = \frac{1}{50 \times 20} \times A \Rightarrow N_1 = \frac{2}{1000}$$

Strength in terms of $\text{CaCO}_3 = \frac{2}{1000} \times 50 \times 10^3 = 100 \text{ ppm}$

orange indicator ($B \text{ ml}$) neutralizes a HCO_3^- ions whether present originally or obtained from CO_3^{2-} . The end point is colour change from light yellow to reddish orange and comes at a pH range of 3-4.5. The total acid used in the titration ($A+B$ or $T \text{ ml}$) gives the total alkalinity of water.

Procedure : Rinse and fill the burette with standard acid and note down its initial reading. Pipette out 20 ml of water sample in a conical flask. Add 1-2 drops of phenolphthalein indicator which may make the solution pink. Titrate pink solution against acid till the colour disappears. Note down the final burette readings. To the colourless solution so obtained, add 1-2 drops of solution becomes light yellow. Titrate it further against acid till the colour changes to reddish orange. Note the final burette reading again. Repeat for taking concordant readings.

Results -

$$\text{Phenolphthalein alkalinity (P)} = 100 \text{ ppm}$$

$$\text{Methyl orange alkalinity (M)} = 305 \text{ ppm}$$

II) for finding methyl orange alkalinity (M)

$$A \propto N_1 V_1 = N_2 V_2 \Rightarrow N_1 = \frac{1}{50 \times 20} \times 1 = \frac{6.1}{1000}$$

$$\text{Strength in terms of } \text{CaCO}_3 = \frac{6.1}{1000} \times 50 \times 10^3 = 305 \text{ ppm}$$

III Alkalinity due to bicarbonate = $M - 2P$

$$= 305 - 2 \times 100$$

$$= 105 \text{ ppm}$$

Results - Phenolphthalein alkalinity (P) = 100 ppm

Methyl orange alkalinity = 305 ppm

Alkalinity due to HCO_3^- ion = $M - 2P$

$$= 105 \text{ ppm}$$

and carbonate (CO_3^{2-}) ion $\Rightarrow 2P = 2 \times 100$
 $= 200 \text{ ppm}$

200
31/12/20

$$\text{Alkalinity due to carbonate} = 2P = 2 \times 100 \\ = 200 \text{ ppm}$$

$$\text{Alkalinity due to } \text{HCO}_3^- \text{ ion} = M - 2P \\ = 305 - 200 \\ = 105 \text{ ppm}$$

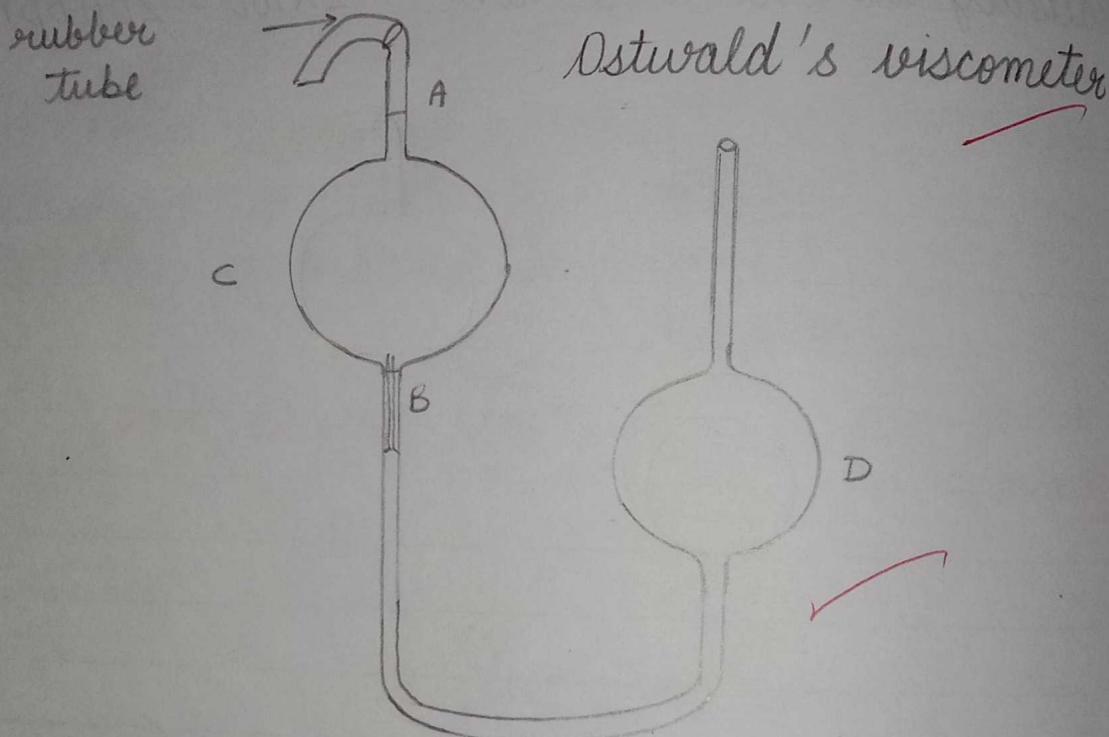
$$\text{Alkalinity due to } \text{CO}_3^{2-} \text{ ion} = 2 \times 100 = 200 \text{ ppm}$$

Experiment - 2

Aim - To determine the coefficient of viscosity of the given liquid by Ostwald's viscometer.

Apparatus - Ostwald viscometer, specific gravity bottle, stop watch, rubber tube, tubing etc.

Chemicals Required - Given liquid, distilled water



Formula used - $\frac{\eta_1}{\eta_2} = \frac{d_1}{d_2} \times \frac{t_1}{t_2}$

η_1 = viscosity of given liquids at room temperature

η_2 = viscosity of water at room temperature

d_1 = density of liquids d_2 = density of water

t_1 = time taken by liquid to flow d_2 = density of water

t_2 = time taken by water to flow

Experiment - 2

SIM - To determine the coefficient of viscosity of the given liquid by Ostwald's viscometer.

Apparatus - Ostwald viscometer, specific gravity bottle, stop watch, rubber tubing etc.

Chemicals Required - Given liquid, distilled water

Theory - The viscosity of a liquid is a measure of its resistance to own flow. The coefficient of viscosity can be defined as the force per unit area required to maintain unit difference of velocity between two layers which are unit distance apart.

Coefficient of viscosity of an unknown liquid can be determined by Ostwald's Viscometer. Same volume of given liquid and distilled water are allowed to flow through it. The times are noted and then using below equation, viscosity can be calculated:-

$$\frac{\eta_1}{\eta_2} = \frac{d_1}{d_2} \times \frac{t_1}{t_2}$$

Observations -
Room temperature = 22°C

S.N.O.	Given liquid	Water	Time of flow	Time of flow
1.	457 sec	356 sec		

Calculations -

$$\frac{\eta_1}{\eta_2} = \frac{d_1}{d_2} \times \frac{t_1}{t_2} \Rightarrow \eta_1 = 1.59 \times \frac{457}{356} \times 0.0095$$

$\therefore \eta_1 = 0.01881 \text{ Poise}$

n_1 = viscosity of given liquids at room temperature

n_2 = viscosity of water at room temperature

d_1 = density of liquid

d_2 = density of water

t_1 = time taken by given liquid to flow

t_2 = time taken by water to flow

Procedure -

1. Clean the viscometer with chromic acid followed by washing with distilled water. It is finally washed with alcohol and dried in oven.
2. Attach a clean rubber tube to the end A and clamp the viscometer ~~in~~ the stand vertically.
3. Now add 10 ml of given liquid (or sufficient volume) to the arm of the bulb D so that bend position of U-tube and more than half of the bulb C is filled up.
4. Through the rubber tube, suck up the liquid until it rise above the mark A. Press the rubber tube with your hand.
5. Now allow the liquid to fall freely through the capillary upto the ~~mark A~~.
6. Repeat the experiment thrice.
7. Now empty the viscometer. Clean and dry the viscometer.

Result - Absolute viscosity of the given liquids
is 0.01881 poise at 22°C

200mL
7/12/20

8. Repeat the experiment by taking some volume of distilled water. Note down the time taken t_2 for the flow of water from mark A to B. Repeat the experiment thrice.
9. Find the density of liquid using specific gravity bottle by noting down weight of empty bottle.
10. Fill it with given liquid and weigh again.
11. Empty the bottle, wash with alcohol and dry it in oven. Fill with distilled water and weigh again.
12. Note the room temperature with thermometer.

**Result - Absolute viscosity of the given liquid is
0.01881 poise at 22°C.**

- Precautions -**
1. The viscometer and specific gravity bottle must be cleaned thoroughly.
 2. The viscometer should be perfectly vertical in position while noting time of flow of liquids.
 3. Exactly same volume of the given liquids and reference liquids should be used.
 4. The viscometer should not be disturbed while measuring the time of flow of liquids.
 5. While sucking the liquids through the rubber tube, air bubbles must be avoided.

Ques - To determine the viscosity of lubricant by redwood viscometer.

Observations -

S.NO.	Time of flow (lubricant)	Time of flow (water)
1.	11.47 sec	8.01 sec
2.	12.86 sec	8.83 sec
3.	11.08 sec	8.60 sec

Mean time of flow of water is 8.48 sec
Mean time of flow of lubricant is 11.83 sec

Experiment - 3

Aim- To determine the viscosity of lubricant by red wood viscometer.

Apparatus- Red wood viscometer, stop watch, thermometer

Chemicals Required- Lubricating oil, distilled water

Theory- Viscosity is defined as the internal friction offered by the layers of liquids to its flow. Viscometer is a measure of flowability of a liquid at a definite temperature. It determines the performance of an oil. Higher is the viscosity of the liquid, lesser will be its flow. The coefficient of viscosity also called absolute viscosity.

The absolute or dynamic viscosity of a lubricant is determined by measuring the time of flow of oil through a capillary of definite dimension at uniform temperature. The viscosity can be determined by a reduced viscometer.

Result - Relative viscosity of given lubricant w.r.t
distilled water is $3.35 \text{ RW}_1 \text{ sec.}$

Viscosity of given lubricant is $11.83 \text{ RW}_2 \text{ sec.}$

Procedure - Level the viscometer with the help of levelling screws. Fill the copper bath with water and connect to the electric mains. Clean the oil cup and discharge jet with a suitable solvent like hexane and properly dry it. Place the ball valve on a gate jet to cover it. Pour the test oil in the cup carefully upto pointer. Let the oil flow into oil cup and note the time taken for flow of oil using stopwatch. Clean the discharge jet properly and repeat the experiment using water. After noting down the values, find the mean for both the values of time taken.

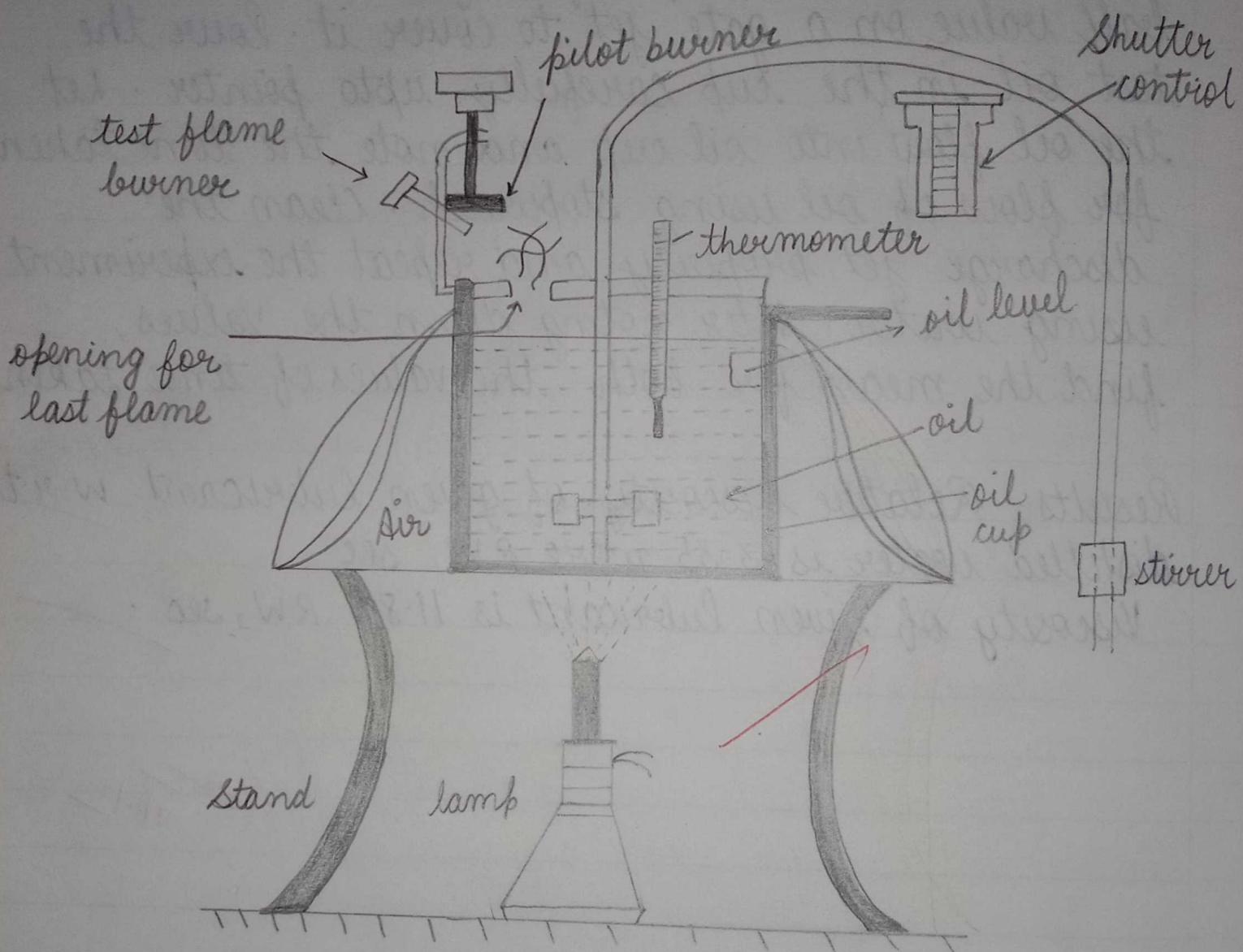
Results - Relative viscosity of given lubricant w.r.t distilled water is 3.35 more RW, sec.

Viscosity of given lubricant is 11.83 RW, sec.

100%
71.21%

Experiment - 4

Aim - To determine flash and fire point of an oil by Pensky - Marten's flash point apparatus.



Pensky - Marten's flash point ~~apparatus~~

Experiment - 4

Sim - To determine flash and fire point of an oil by Pensky - Marten's flash point apparatus.

Apparatus - Pensky - Marten's flash point apparatus, thermometer, lubricating oil (chemicals)

Theory -

Flash Point - It is defined as the lowest temperature at which an oil gives off enough vapours which ignite for a moment, giving a flash. When a flame of specific dimension is brought near the surface of oil being heated under specific conditions.

Fire Point - It is the minimum temperature (above flash point) at which the oil produces sufficient vapours which burn for at least 5 seconds. When a tiny flame is brought near the oil surface being heated under specific conditions.

Procedure - The oil sample under test is filled into oil cup upto the mark for it. It is covered with the lid and placed over the heating device. A thermometer is inserted into the oil through an opening for it in the lid. The test flame is lighted. The oil is lighted in such a way that

Observations -

For flash point		For fire point	
Testing temp.	Yes / No	Testing temp.	Yes / No
45°	Yes	60°	Yes

Results -

The given oil has a flash point = 45°C

The given oil has a fire point = 60°C

its temperature rises by $3\text{-}5^\circ\text{C}$ by minute. During heating the mechanical stirrer is rotated slowly for homogeneous heating of oil. When the temperature rises to about 15°C of anticipated flash point of oil. Checking is started by introducing the test flame into oil cup through opening for it in the lid. The checking is done for every 3°C rise in its temperature. The temperature at which a momentary flash is produced by oil vapours is recorded as flash point. The heating of oil is continued with frequent testing till the oil gives a flame which burns for at least five seconds. This temperature is recorded as fire point. The process is repeated and mean value of two temperature is reported separately for flash and fire point.

Results -

The given oil has a flash point of 45°C and fire point of 60°C .

Boons
7/12/20

its temperature rises by $3-5^{\circ}\text{C}$ by minute. During heating the mechanical stirrer is rotated slowly for homogeneous heating of oil. When the temperature rises to about 15°C of anticipated flash point of oil. Checking is started by introducing the test flame into oil cup through opening for it in the lid. The checking is done for every 3°C rise in its temperature. The temperature at which a momentary flash is produced by oil vapours is recorded as flash point. The heating of oil is continued with frequent testing till the oil gives a flame which burns for at least five seconds. This temperature is recorded as fire point. The process is repeated and mean value of two temperature is reported separately for flash and fire point.

Results -

The given oil has a flash point of 45°C and fire point of 60°C .

Boons
7/12/20

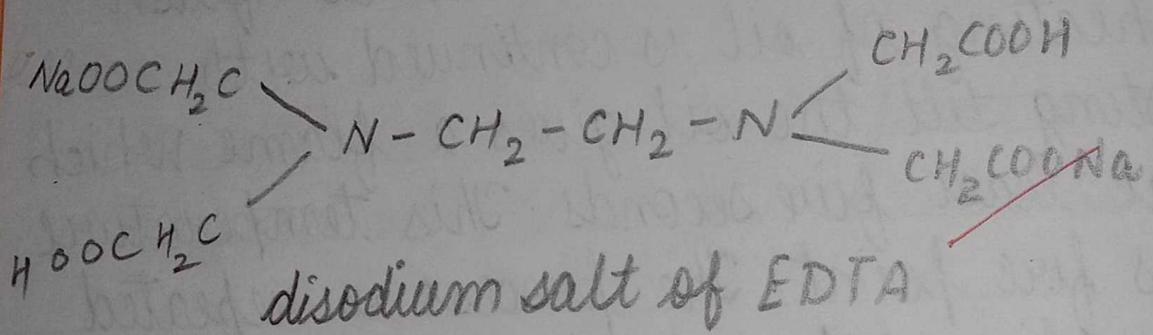
Experiment - 5

Aim - To determine temporary and permanent hardness of given water sample by EDTA method.

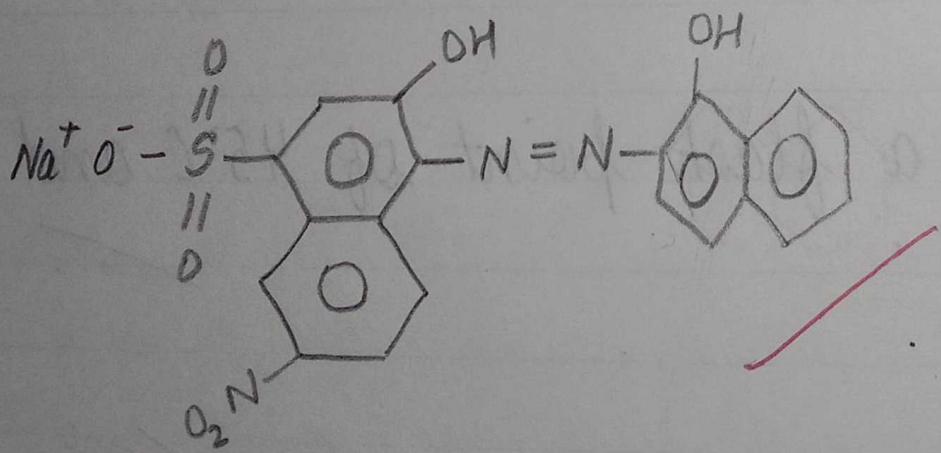
Indicator - Eriochrome - black T

End Point - colour change from wine red to blue.

Structure of EDTA:



Structure of EBT:



Experiment - 5

Aim - To determine temporary and permanent hardness of given water sample by EDTA method.

Chemicals Required : Standard hard water (1 ml = 1 mg of CaCO_3), EDTA solution, buffer solution of $\text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$

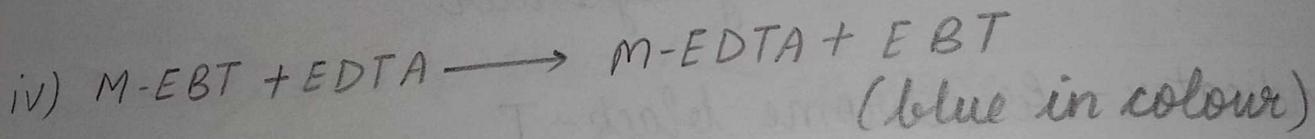
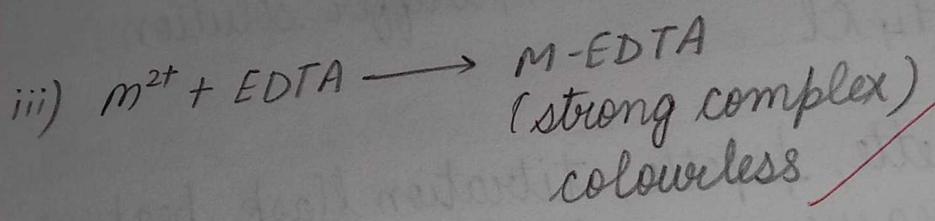
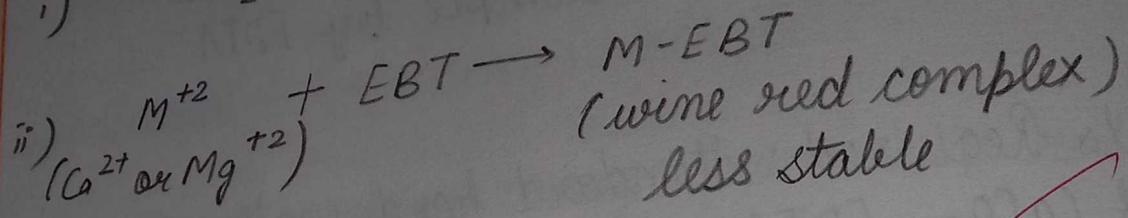
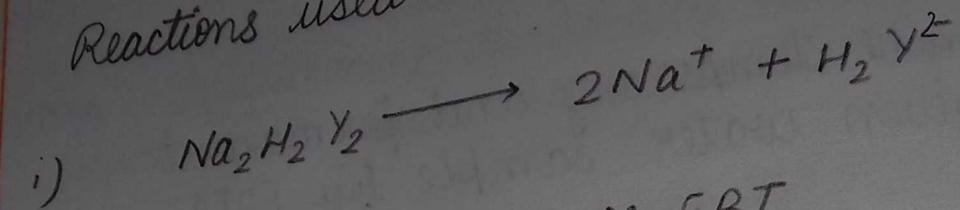
Apparatus - burette, pipette, titration flask, beakers, test tubes, measuring cylinder

Indicator - Eriochrome black-T

End point - colour change from ~~turn~~ red to blue.

Theory - It is a complexometric titration method. In this method, total hardness of water can be determined by estimating divalent metal ions present in water by titrating a known value of it, buffered to a pH (10) with $\text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$ buffer, against standard solution of disodium salt of EDTA in presence of an indicator EBT. Prolonged boiling of hard water followed by filtration and titration of

Reactions used:



Observation Table - Vol. of S.H.W taken = 20ml

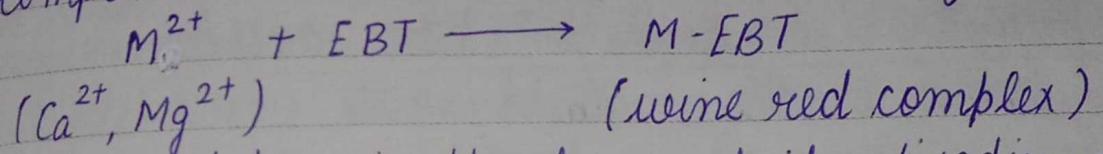
I) For standardization of EDTA solution

S.No.	Initial burette reading	Final burette reading	Vol. of EDTA used (ml)
1.	0	10.9	10.9
2.	10.9	22	11.1
3.	22	33.1	11.1

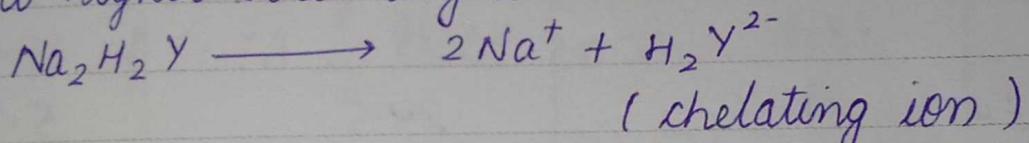
Concordant volume (V_1) = 11.1 ml

filtrate against EDTA gives permanent hardness.
The difference in two hardness values gives the temporary hardness of water.

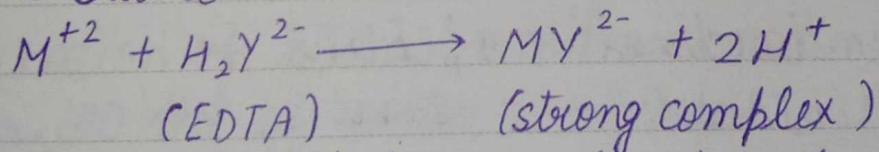
When added to hard water at a pH around 10, Eriochrome Black T forms unstable wine red coloured complexes with bivalent metal ions of water.



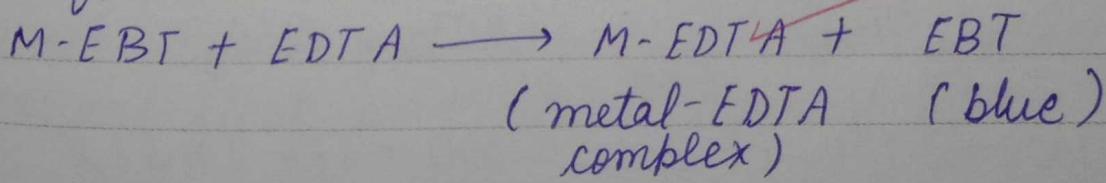
EDTA is taken in the form of its disodium salt due to higher solubility in water.



When added from burette to wine red solution, EDTA combines with free metal ions of hard water to form their respective soluble complexes. These are more stable.



When all free metal ions of hard water have complexed with EDTA, a slight excess of EDTA removes metal ions from weak-indicator complexes to form metal-EDTA which releases the indicator in free form which is blue in colour.



II For total hardness in water sample			
S.N.O.	Initial burette reading	Final burette reading	Vol. of EDTA used (ml)
1.	0	7.6	7.6
2.	32.7	40.1	7.4
3.	40.1	47.5	7.4

Concordant reading (V_2) = 7.4 ml

III For permanent hardness in water sample			
S.N.O.	Initial burette reading	Final burette reading	Vol. of EDTA used (ml)
1.	7.6	13.1	5.5
2.	13.1	19	5.9
3.	19	24.5	5.5

Concordant reading (V_3) = 5.5 ml

Calculations -

Step I - Standardization of EDTA

11.1 ml of EDTA solution = 20 ml of SHW

$$1 \text{ ml of EDTA solution} = \frac{20}{11.1} \text{ ml} = \frac{20}{11.1} \times 1 \text{ mg CaCO}_3 \\ = 1.8 \text{ mg CaCO}_3$$

Step II Estimation of total hardness

20 ml UHW = 7.4 ml EDTA

$$1 \text{ ml UHW} = \frac{7.4}{20} \times \frac{20}{11.1} \text{ mg CaCO}_3$$

$$1000 \text{ ml UHW} = \frac{7.4}{11.1} \times 1000 = 666.7 \text{ mg/l}$$

Procedure - The experiment is carried out in three steps:

I Standardisation of EDTA solution: Rinse and fill the burette with EDTA solution. Note initial burette readings. Transfer 20 ml of standard hard water into a conical flask using pipette. Add 5 ml of buffer solution and 3-4 drops of EBT indicator. Titrate wine red solution against EDTA solution till colour changes to pure blue. Record the final burette reading. Take concordant readings. Let V_1 ml of EDTA is used in this step.

II Determination of total hardness in sample: Similarly titrate 20 ml of hard water sample against EDTA and record the volume of EDTA used as V_2 ml.

III Determination of permanent hardness in sample - Transfer 100 ml of water sample in a beaker. Boil it on burner flame to near dryness. Cool it slightly and add small amount of distilled water. Filter the solution into a 100 ml measuring flask. Make the volume of filtrate 100 ml by adding more distilled water. Put a stopper on flask and shake well. Pipette out 20 ml of this solution in a conical flask and titrate against EDTA.

III Estimation of permanent hardness

$$1 \text{ ml boiled water} = \frac{5.5}{20} \times \frac{20}{11.1} \text{ mg } \text{CaCO}_3$$

$$1000 \text{ ml boiled water} = \frac{5.5}{11.1} \times 1000 \text{ mg/l}$$
$$= 495.5 \text{ mg/l}$$

IV Temporary hardness

$$\text{Temporary hardness} = \text{Total hardness} -$$
$$\text{Permanent hardness}$$
$$= 666.7 - 495.5$$
$$= 171.2 \text{ mg/l}$$

Results-

$$\text{Permanent hardness} = 495.5 \text{ mg/l}$$

$$\text{Temporary hardness} = 171.2 \text{ mg/l}$$

2009/2/12

solution as in step 1. Let volume of EDTA used
be V_3 ml.

Results -

Permanent hardness - 495.5 mg/l

Temporary hardness - 171.2 mg/l

Experiment - 6

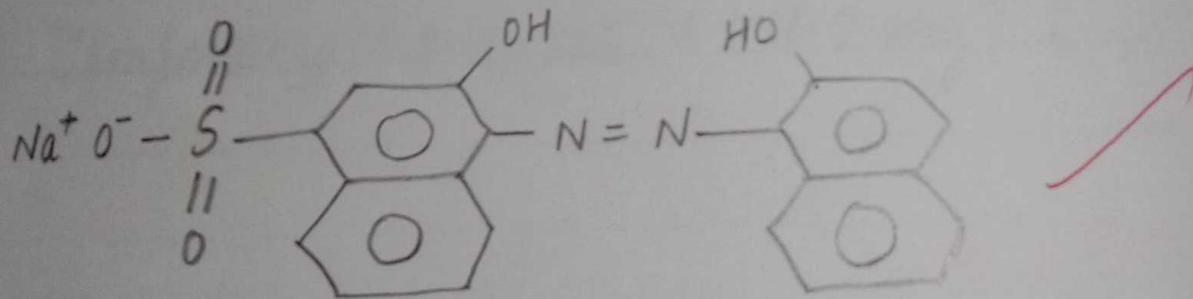
Aim - To determine Ca^{+2} and Mg^{2+} hardness of water using EDTA solution.

Indicator - i) Eriochrome black-T
ii) Calcon

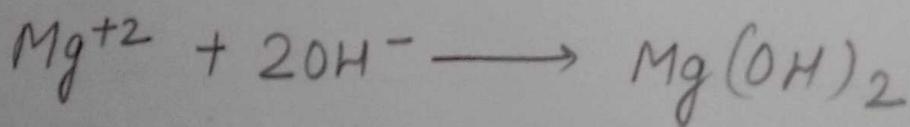
End Points -

- 1) Colour changes from wine red to blue with EBT.
- 2) Colour changes from pink to purple with calcon.

Structure of Calcon -



Reactions used -



Experiment - 6

Ques - To determine Ca^{2+} and Mg^{2+} hardness of water using EDTA solution.

Apparatus - Burette, pipette, titration flask, beakers, test tubes, measuring cylinder

Chemicals Required - Standard hard water, EDTA solution, buffer solution of $\text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$ and diethyl amine

Indicator : i) Eriochrome black-T ✓
ii) calcon ✓

End Points -

1. Colour changes from wine red to blue with EBT.
2. Colour changes from pink to purple with calcon

Theory - For finding total hardness of water a known volume of water sample, buffered to a pH around 10 with ammonical buffer is titrated with standard EDTA solution using EBT as indicator.

After finding total hardness, Mg^{2+} ions in hard water sample are suppressed as magnesium

Observation Table -

1) Volume of standard hard water = 20ml
For standardization of EDTA solution :-

S.NO.	Initial burette reading	Final burette reading	Vol. of EDTA used (ml)
1.	0	10.9	10.9
2.	10.9	22	11.1
3.	22	33.1	11.1

$$\text{concordant volume } (V_1) = 11.1 \text{ ml}$$

2) For total hardness in water sample -

Volume of h.w sample taken = 20 ml

S.NO.	Initial burette reading	Final burette reading	Vol. of EDTA used (ml)
1.	0	7.6	7.6
2.	32.7	40.1	7.4
3.	40.1	47.5	7.4

$$\text{concordant volume } (V_2) = 7.4 \text{ ml}$$

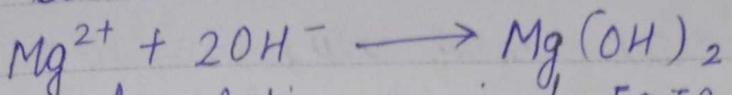
3) For permanent hardness in water sample

Vol. of treated h.w sample taken = 20 ml

S.NO.	Initial burette reading	Final burette reading	Vol. of EDTA used (ml)
1	0	4.5	4.5
2	4.5	7.8.1	4.6
3	8.1	12.5	4.5

$$\text{concordant volume } (V_3) = 4.5 \text{ ml}$$

hydroxide by adding diethyl amine which makes pH of solution around 12.5



Titration of solution against EDTA using calcon indicator gives Ca^{2+} hardness of water. Mg^{2+} hardness is then obtained from difference of total and Ca^{2+} hardness.

Procedure -

I Standardization of EDTA solution - Rinse and fill the burette with EDTA solution. Note initial burette reading. Pipette out 20 ml of standard hard water into a conical flask. To this add 5 ml of buffer solution and 3-4 drops of EBT indicator. Titrate wine red solution against EDTA till the end point i.e. colour change from wine red to blue. Note the final burette reading. Repeat for taking concordant readings. Let V , ml of EDTA solution be used.

II Total hardness in water sample - Titrate 20 ml of given hard water sample against EDTA as in step I. Let volume of EDTA used be V_2 ml.

III Ca^{2+} hardness in water sample - Pipette out 20 ml

Calculations -

1) Standardisation of EDTA solution -

$$20 \text{ ml S.H.W} = 11.1 \text{ ml of EDTA}$$

$$1 \text{ ml of EDTA} = \frac{20}{11.1} \text{ ml}$$

$$= \frac{20}{11.1} \text{ mg of } \text{CaCO}_3$$

$$= 1.8 \text{ mg of } \text{CaCO}_3$$

2) Estimation of total hardness in water sample

$$20 \text{ ml hard water} = 7.4 \text{ ml EDTA}$$

$$= 7.4 \times \frac{20}{11.1} \text{ mg of } \text{CaCO}_3$$

$$1 \text{ ml hard water} = \frac{7.4}{11.1} \times \frac{20}{20} \text{ mg } \text{CaCO}_3$$

$$= \frac{7.4}{11.1} \text{ mg } \text{CaCO}_3$$

$$1000 \text{ ml hard water} = \frac{7.4}{(total \text{ hardness})} \times 1000 = 666.7 \text{ mg/l}$$

3) Estimation of Ca^{+2} hardness in water sample

$$1000 \text{ ml h.w} = 4.5 \times \frac{20}{11.1} \times \frac{1000}{20} = 405.5 \text{ mg/l}$$

$$\text{Ca}^{+2} \text{ hardness in water} = 405.5 \text{ ppm}$$

4) Mg^{+2} hardness in water sample = Total hardness -

$$\text{Ca}^{+2} \text{ hardness}$$
$$= 666.7 - 405.5 \text{ ppm}$$

$$= 261.2 \text{ ppm}$$

Results -

$$1) \text{Ca}^{+2} \text{ hardness in water sample} = 405.5 \text{ ppm}$$

$$2) \text{Mg}^{+2} \text{ hardness in water sample} = 261.2 \text{ ppm}$$

of water sample in conical flask. Add 3-4 ml of diethyl amine and 5-6 drops of calcon indicator. Shake the solution thoroughly for 2 minutes.

Titrate pink coloured solution against EDTA till colour changes to purple. Record burette readings. Take concordant readings. Let V_3 ml of EDTA is used in this step.

Result -

1. Ca^{2+} hardness in water sample = 405.5 ppm

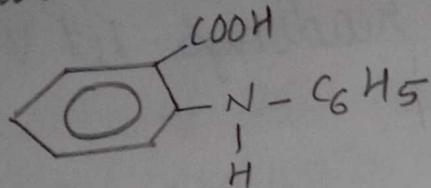
2. Mg^{2+} hardness in water sample = 261.2 ppm

$100 \text{ } \cancel{\text{ml}}$ $\cancel{100 \text{ ml}}$

Experiment - 7

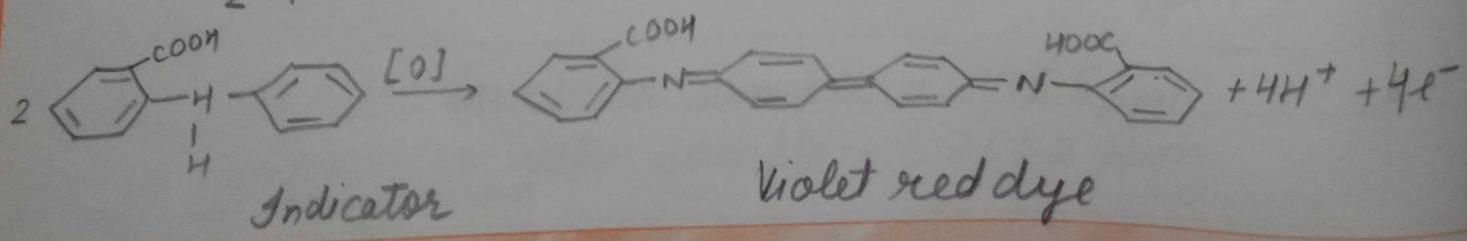
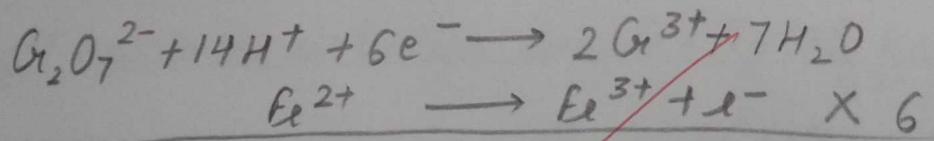
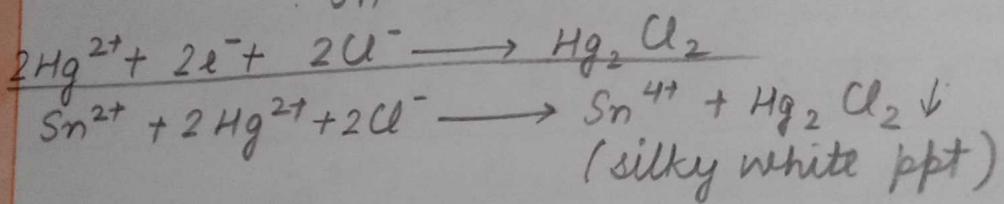
sim - To determine amount of total iron in an iron ore solution by internal indicator method.

Indicator - N-Phenylanthranilic acid



End Point- Colour changes from light green to reddish violet.

$$\text{Reactions used: } \begin{array}{l} \text{Sn}^{2+} \rightarrow \text{Sn}^{4+} + 2e^- \quad (\text{oxidation}) \\ \underline{2\text{Fe}^{3+} + 2e^- \rightarrow 2\text{Fe}^{2+}} \quad (\text{reduction}) \\ \underline{\text{Sn}^{2+} + 2\text{Fe}^{3+} \rightarrow \text{Sn}^{4+} + 2\text{Fe}^{2+}} \\ \text{Sn}^{2+} \rightarrow \text{Sn}^{4+} + 2e^- \end{array}$$



Experiment - 7

Aim - To determine amount of total iron in an iron ore solution by internal indicator method.

Chemicals Required - 5% stannous chloride solution (SnCl_2), saturated mercuric chloride solution (HgCl_2), standard pot. dichromate solution, conc. HCl and dil. HCl

Apparatus - Burette, pipette, titration flask, 250 ml volumetric flask, 500 ml beaker, test tube, measuring cylinder, ~~filter paper~~, zinc dust etc.

Indicator - N-Phenylanthranilic acid

End Point - Colour changes from light green to reddish violet

Theory: Determination of iron content using internal indicator

An iron ore solution is prepared by treating the ore with acid. It contains both ferrous (Fe^{+2}) and ferric (Fe^{+3}) ions. The amount of

Observations :-

Volume of ore solution taken = 20 ml

S.NO.	Initial Burette Reading	Final Burette Reading	Vol. of $K_2Cr_2O_7$ sol ⁿ used (ml)
1.	0	13	13
2.	13	26.5	13.5
3	26.5	39.5	13

Concordant volume (V_2) = 13 ml

Calculations :- 1) Normality of ore solution

Vol. of ore solution taken for titration = 20 ml

Concordant vol. of $K_2Cr_2O_7$ solⁿ used = 13 ml

Normality of $K_2Cr_2O_7$ solⁿ = $\frac{N}{10}$

Applying Normality equation,

$$\frac{N_1 V_1}{(\text{ore})} = \frac{N_2 V_2}{(K_2Cr_2O_7)}$$

$$N_1 \times 20 = \frac{1}{10} \times 13$$

$$N_1 = \frac{13}{10 \times 20} = 0.065 N$$

iron in ore can be estimated by its titration with an oxidising agent like standard pot. dichromate solution in acidic medium. However, direct titration gives only amount of Fe^{+2} ions since Fe^{+3} ions being oxidising in nature do not react with dichromate ions also oxidising in behaviour.

For finding total iron, the ore solution is first reduced with slight excess of stannous chloride in medium acidified with conc. HCl.

Sn^{+2} left in excess is destroyed by adding saturated mercuric chloride solution in one lot. Total iron present as Fe^{+2} in reduced solution is titrated against dichromate solⁿ in acidic medium using *N*-phenylathranilic acid as internal indicator.

At the end point when all Fe^{+2} ions get oxidised by dichromate, an excess drop of dichromate oxidises indicator to form a violet red dye.

From the volume of dichromate solution used in titration, total iron in ore solution can be calculated.

2) Strength of total iron in ore solution.

$$\begin{aligned} \text{Strength of total iron in ore solution} &= \text{Normality} \times \text{Eq. weight of iron} \\ &= 0.065 / \times 56 \text{ g/lt} \\ &= 3.64 \text{ g/lt} \\ &= 3.64 \times 10^3 \text{ ppm} \\ &= 3640 \text{ ppm} \end{aligned}$$

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Procedure - Rinse and fill the burette with dichromate solution. Note initial burette reading. Pipette out 10 ml of ore solution in a conical flask. Add 3 ml conc. HCl into it and boil on flame to yellow solution. Add 5% stannous chloride solution dropwise till yellow colour disappears. Add 2-3 drops in excess. cool the solution to room temperature under tap water. Add 3 ml saturated $HgCl_2$ solution in one and shake well till silky white ppt are obtained. Add 10 ml dil. HCl, 7-8 drops of indicator and titrate against dichromate solution till light green colour changes to violet red which marks end pt. of titration. Note final reading. Repeat the titration to record concordant value.

Result -

Normality of ore solution $\rightarrow 0.65 N$
Strength of total iron in ore solution $\rightarrow 3.64 g/l$
The given ore solution contain $3.64 g/l$
of total iron.