

Common for All Branches)

Name..... Anmol Barahwal

Class..... B.Tech CSE Roll No..... 282020 8

College..... PIET

Experiments Performed

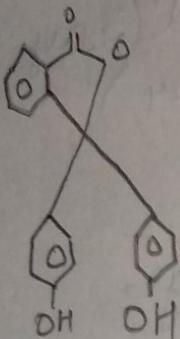
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Experiments Performed

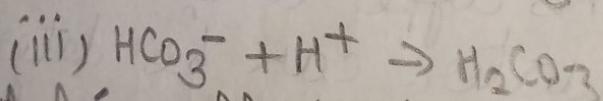
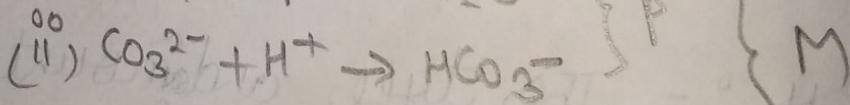
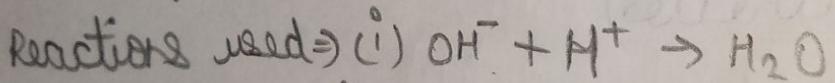
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9.	To determine the surface tension of a given liquid by drop number method using stalagmometer.	22/7/21	29-33	

Experiment - 1

Aim :- To determine alkalinity of water sample.
 Indicators \Rightarrow (i) Phenolphthalein
 (ii) Methyl orange



(i) End point \Rightarrow (i) Pink to colourless with phenolphthalein
 (acid in burette)
 (ii) colour change from light yellow to reddish
 orange with methyl orange



P \rightarrow phenolphthalein alkalinity

M \rightarrow total or methyl orange alkalinity

$P = \text{OH}^- + \frac{1}{2}\text{CO}_3^{2-}$ $M = \text{OH}^- + \text{CO}_3^{2-} + \text{HCO}_3^-$ (all ions)

$M - 2P =$ alkalinity due to HCO_3^- ions

Experiment -1

Aim :- To determine alkalinity of water sample

Reagents Required :- Standard acid (say N/50 HCl or H₂SO₄)

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).

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

S.N.B	case	OH^-	CO_3^{2-}	HCO_3^-
1.	$P=0$ $M=P$	$P \text{ or } M$	0	M
2.	$P=1/2 M$	0	$2P \text{ or } M$	0
3.	$P > 1/2 M$	$2(P-M)$	$2(M-P)$	0
4.	$P < 1/2 M$	0	$2P$	$M-2P$

observation table \Rightarrow

S.N.B.	Initial reading (x)	Final reading		vol ^m of acid used	
		P	M	P-x	M-x
1.	0	2	6.1	2	6.1
2.	0	2	6.1	2	6.1
3.	0	2	6.4	2	6.4

concordant volume of acid used upto phenolphthalein point A $\Rightarrow A \text{ ml} = P-x = 2 \text{ ml}$

concordant vol^m of acid used upto methyl orange end point $\Rightarrow T \text{ ml} = M-x = 6.1 \text{ ml}$

General calculations \Rightarrow

I. For finding phenolphthalein alkalinity (P) -

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

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

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 orange indicator (B 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 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

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II. for finding methyl orange alkalinity (M)
As $N_1 V_1 = N_2 V_2 \Rightarrow N_1 = \frac{1}{50 \times 20} \times 7 \Rightarrow \frac{6.1}{1000}$

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

$$\text{III. Alkalinity due to Bicarbonate} = M - 2P \\ = 305 - 2 \times 100 \\ = 105 \text{ ppm}$$

Results:- Phenolphthalein alkalinity (P) = 100 ppm

$$\text{Methyl orange alkalinity} = 305 \text{ ppm}$$
$$\text{Alkalinity due to } \text{HCO}_3^- \text{ ions} = M - 2P \\ = 105 \text{ ppm}$$

$$\text{for carbonate } (\text{CO}_3^{2-}) \text{ ion} \approx 2P \\ \Rightarrow 200 \text{ ppm}$$

burette reading again. Repeat for taking concordant readings.

Result :- phenolphthalein alkalinity (P),

 = 100 ppm

Methyl orange alkalinity (M) = 305 ppm

Alkalinity due to carbonate

$$\Rightarrow 2P = 200 \text{ ppm}$$

Alkalinity due to HCO_3^- i.e.

$$\Rightarrow M - 2P = 305 - 200 \Rightarrow 105 \text{ ppm}$$

Alkalinity due to CO_3^{2-} i.e. $\Rightarrow 2 \times 100$
 $\Rightarrow 200 \text{ ppm}$

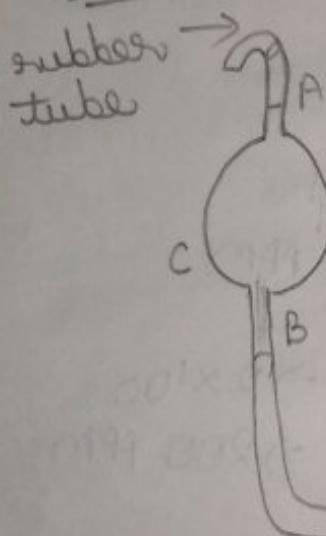
Experiment - 2

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

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

Chemicals Required :- Given liquid, distilled water

Ostwald's viscometer



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

η_1 = viscosity of given liquid at room temperature

η_2 = viscosity of water at room temperature

d_1 = density of liquid, d_2 = density of water

t_1 = time taken by liquid to flow

t_2 = time taken by water to flow.

Experiment - 2

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

Apparatus Required :- 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 a 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

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Observations :-
Room temperature = 22°C

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

calculations \Rightarrow

$$\frac{\tau_1}{\tau_2} = \frac{d_1}{d_2} \times \frac{t_1}{t_2} \Rightarrow \eta_1 = \frac{1.59 \times 457}{356} \times 0.0095 \\ \Rightarrow 0.01881 \text{ Poise}$$

Result :- Absolute viscosity of the given liquid is 0.01881 Poise at 22°C

noted and then using below equation, viscosity can be calculated

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

where, η_1 = viscosity of given liquid at room temperature

η_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 ~~or~~ with distilled water. It is finally washed with alcohol and dried in oven.
2. Attach a clear rubber tube to the end A and hang the viscometer in the stand vertically.
3. Now add 1 ml of given liquid (or sufficient volume) to the arm of the

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sub 1 so that bend portion of U-tube
and more than half of the C is filled
up.

4. Through the rubber tube, suck up the liquid until it rise above the ~~marks~~
mark A. Press the rubber tube with your hand.
5. Now allow the liquid to fall freely through the capillary ~~up~~ upto the mark A.
6. Repeat the experiment thrice.
7. Now empty the viscometer. Clean and dry the viscometer
8. Repeat the experiment by taking some volume of distilled water. Note down the time taken t_2 for the flow of water from A to B. Repeat the experiment thrice.
9. Find the density of liquid using specific gravity bottle by noting down

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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 $22^{\circ}\text{C}.$

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

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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.

Experiment - 3

Aim :- To determine the viscosity of lubricant by redwood viscometers.

Observations :-

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

Mean time of flow of water $\Rightarrow 8.48 \text{ sec}$

Mean time of flow of lubricant $\Rightarrow 11.83 \text{ sec}$

Result :- Relative viscosity of given lubricant w.r.t. distilled water is $3.35 \text{ RW}_2 \text{ sec}$

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

Experiment - 3

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

Apparatus required :- 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 the liquid 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 are 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

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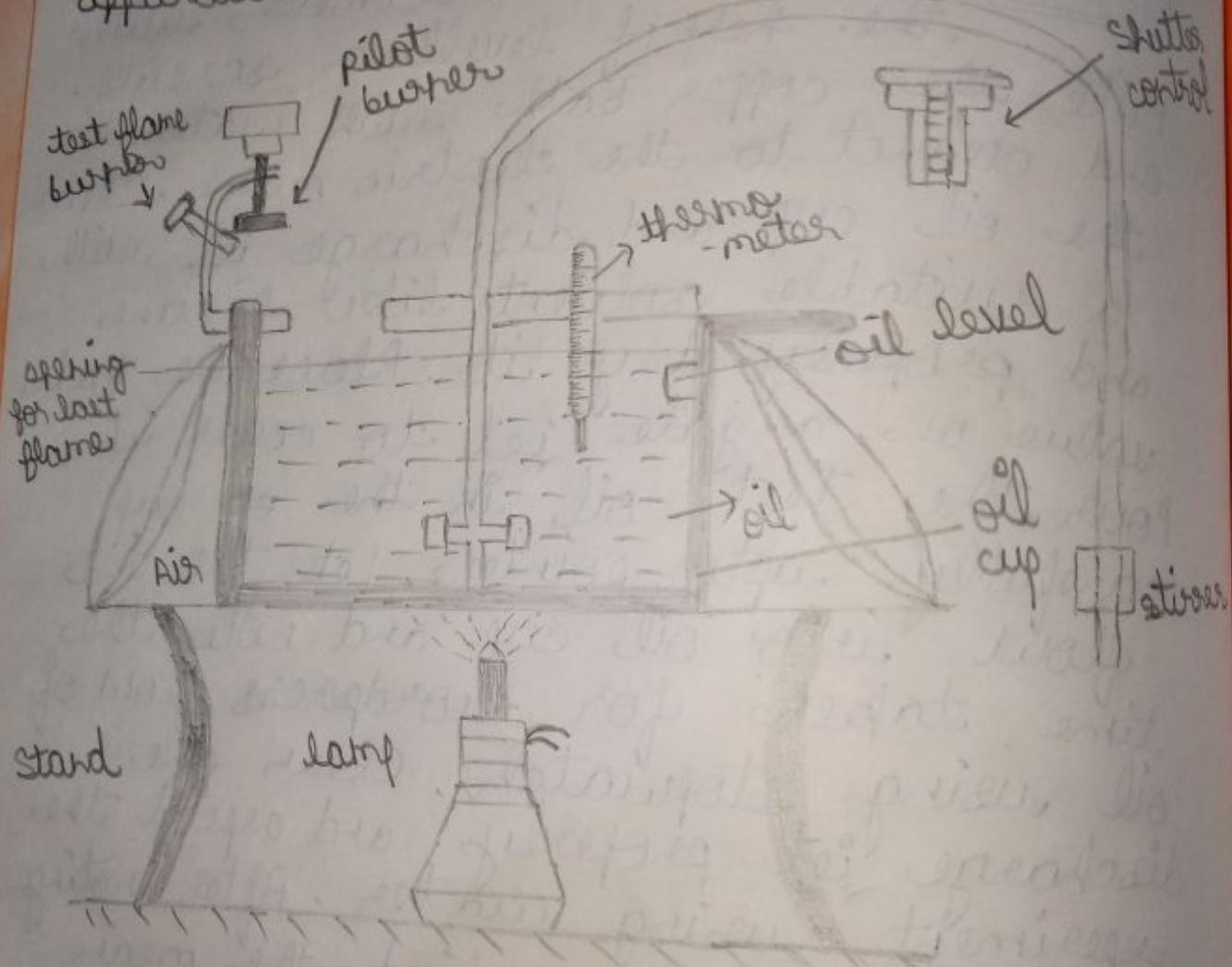
dimensions at uniform temperature.
The viscosity can be determined by
a reduced viscometer.

Procedure :- Level the viscometer with
the help of levelling screws.
Fill the coffee 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 ~~oil~~ cup
carefully upto pointer. Let the oil
flow into oil cup and note the
time taken for ~~flow~~ ~~to~~ 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 RW₁ sec.
Viscosity of given lubricant is 11.83 RW₂ sec

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

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

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

Theory :- Flash point \Rightarrow 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 \Rightarrow 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

Observations :-

For flash point testing temp.	yes/No	for fire point 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

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 heated lighted. The oil is heated in such a way that 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 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 .

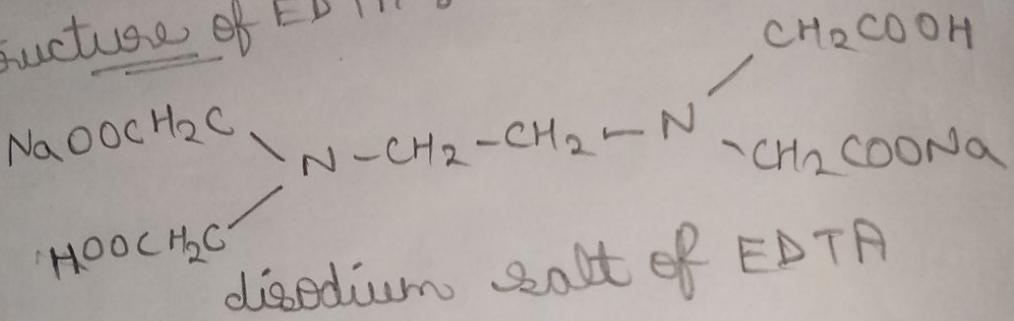
Experiment - 5

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

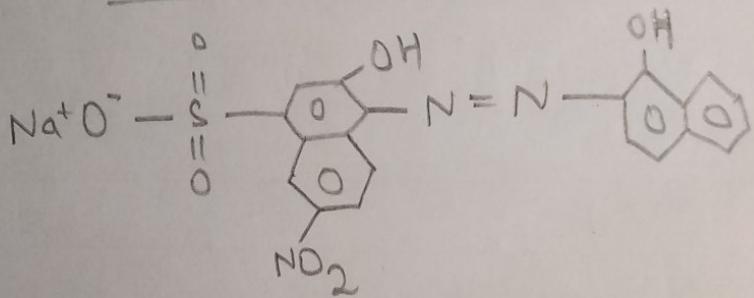
Indicator :- Eriochrome black T

End point :- colour change from wine red to blue

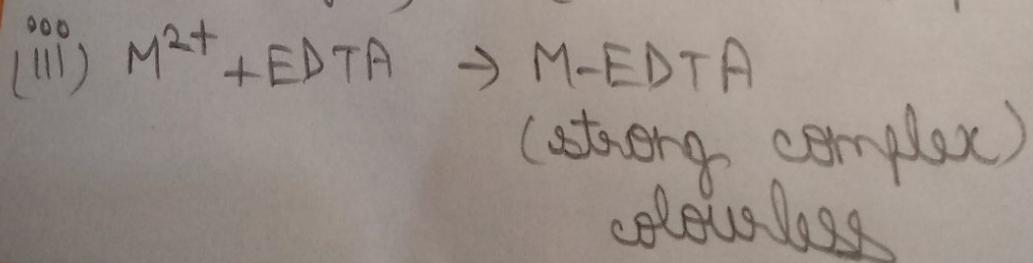
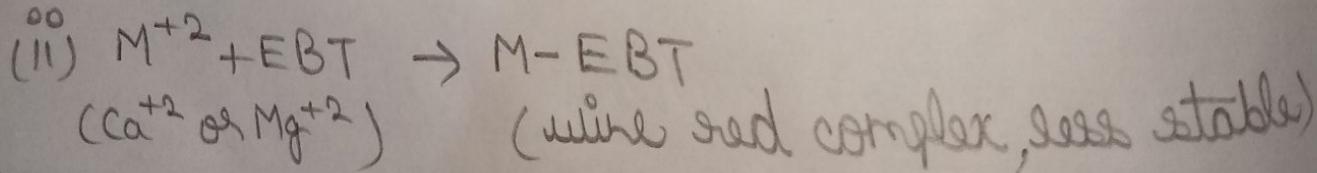
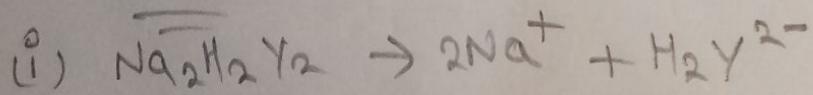
Structure of EDTA :-



Structure of EBT :-



Reactions used :-



Experiment - 5

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

Chemicals Required :- Standard hard water ($1\text{ ml} = 1\text{ mg of } \text{CaCO}_3$), EDTA solution, buffer solution of $\text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$

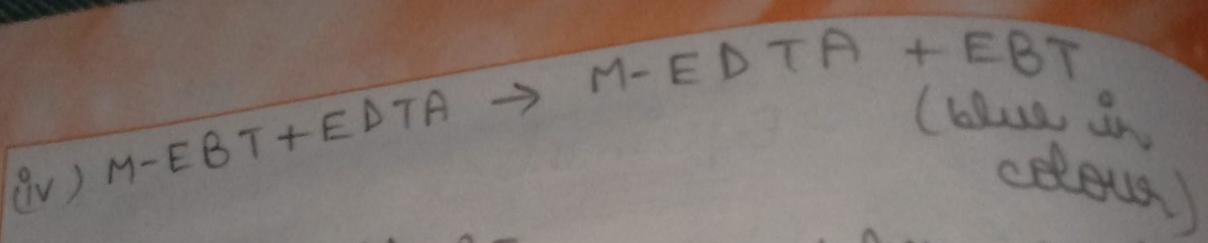
Apparatus Required :- Burette, pipette, titration flask, beakers, test tubes, measuring cylinder

Indicator :- Eriochrome black-T

End point :- colour change from wine 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,

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Observation Table %
 vol^m of S.H.W. taken = 20 ml

I. Standardization of EDTA solution

S.N.B.	Initial burette reading	Final burette reading	vol ^m of EDTA used (ml)
1.	0	10.9	10.9
2.	10.9	22	11.1
3.	22	33.1	11.1

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

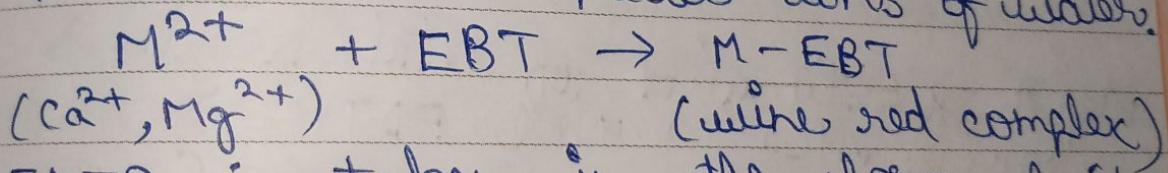
II. For total hardness in water sample

S.No.	Initial burette reading	Final burette reading	vol ^m of EDTA used (ml)
1.	0	7.6	7.6
2.	32.7	40.1	7.4
3.	40.1	47.5	7.4

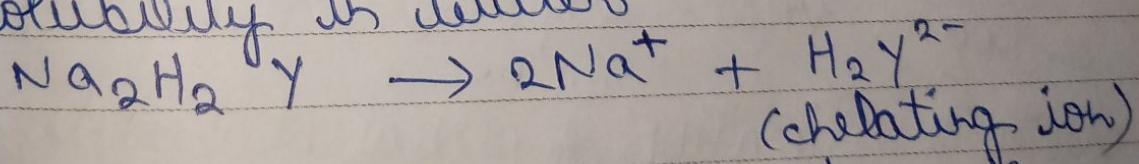
$$\text{concordant reading } (V_2) = 7.4 \text{ ml}$$

buffered to a pH (10) with NH_4OH + NH_4Cl 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 filtrate against EDTA gives permanent hardness. The difference in total 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 ions of hard water to form their respective soluble complexes. These are more stable.

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III. For permanent hardness in water sample

S.N.O.	Initial burette reading	Final burette reading	Vol ^m 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 \Rightarrow standardisation of EDTA
 Step I \Rightarrow standardisation of EDTA solution = 20 ml of S.H.W.
 $11.1 \text{ ml of EDTA solution} = 20 \text{ ml}$
 $1 \text{ ml of EDTA solution} = \frac{20}{11.1} \text{ ml} = \frac{20}{11.1} \times 1 \text{ mg CaCO}_3$
 $\Rightarrow 1.8 \text{ mg CaCO}_3$

Step II \Rightarrow Estimation of total hardness

$$20 \text{ ml of VHW} = 7.4 \text{ ml EDTA}$$

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

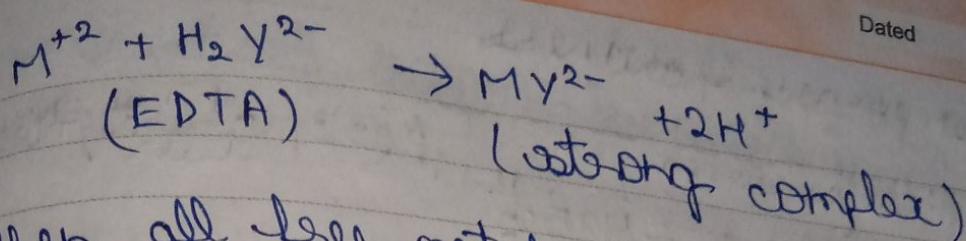
$$1000 \text{ ml VHW} = \frac{7.4}{11.1} \times 1000 = 665.7 \text{ mg/l}$$

Step III \Rightarrow Estimation of permanent hardness

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

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

$$\Rightarrow 495.5 \text{ mg/l}$$



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

$$M-EBT + EDTA \rightarrow M-EDTA + EBT$$

(metal - EDTA (blue)
complex)

Procedure :- The experiment is carried out in three steps :-

1. 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 5ml of buffer solution and 3-4 drops of EBT indicator. Titrate zinc red solution against EDTA solution till colour changes to pure blue. Record the final burette reading. Take concentrated reading.

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IV. Temporary hardness
Temporary hardness \Rightarrow Total hardness -
Permanent hardness

$$\Rightarrow 666.7 - 495.5$$

$$\Rightarrow 171.2 \text{ mg/l}$$

Results :- Permanent hardness = 495.5 mg/l
Temporary hardness = 171.2 mg/l

P.

B.

Let V_1 ml of EDTA is used in this step.

Dated

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

Determination of Permanent hardness in sample \Rightarrow 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 ~~for~~ titrate against EDTA and record the volume of EDTA used as in step 1. Let vol^m of EDTA used be V_3 ml

Results:- Permanent hardness = 495.5 mg/l
 Temporary hardness = 171.2 mg/l

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Experiment - 6

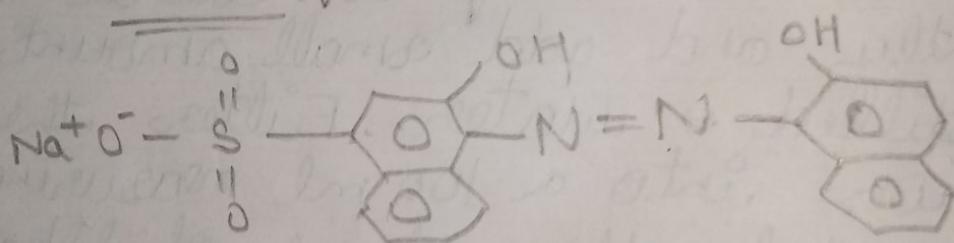
Aim :- To determine Ca^{+2} and Mg^{+2} hardness of water using EDTA solution.

Indicators :- (i) Eriochrome black-T
(ii) calcoph

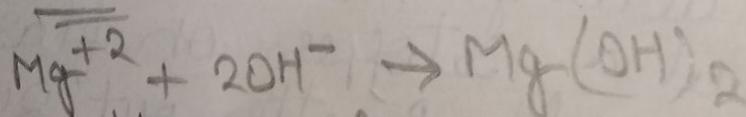
End points \Rightarrow

1. colour changes from wine red to blue with EBT
2. colour changes from pink to purple with calcoph

Structure of calcoph :-



Reactions used :-



Observation table :-

1. volume of standard hard water = 20 ml
For standardization of EDTA solution

\Rightarrow

Experiment - 6

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

Apparatus Required :- Burette, pipette, titration flask, beakers, test tubes, measuring cylinders.

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

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S.N.B.	Initial burette reading	Final burette reading	vol ^m 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

2. For total hardness in water sample.
volume of A.W. sample taken = 20 ml

S.N.B.	Initial burette reading	Final burette reading	vol ^m 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 volume (V_2) = 7.4 ml

3. For permanent hardness in water sample
vol^m of treated HW sample taken = 20 ml

S.N.B.	Initial burette reading	Final burette reading	vol ^m of EDTA used (ml)
1.	0	4.5	4.5
2.	4.5	8.1	4.6
3.	8.1	12.6	4.5

concordant vol^m (V_3) = 4.5 ml

EBT as indicator.

After finding total hardness, Mg^{2+} ions in hard water sample are expressed as magnesium hydroxide by adding diethylamine which makes pH of solution around 12.5

$$Mg^{2+} + 2OH^- \rightarrow Mg(OH)_2$$

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 :-

1. Standardisation 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 with red solution against EDTA till the end point i.e. colour change from blue to red. Note the final burette reading. Repeat for

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calculations :-

1. Standardisation of EDTA solution \Rightarrow
 $20 \text{ ml SHW} = 11.1 \text{ ml of EDTA}$

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

$$= \frac{20}{11.1} \text{ mg of } \text{CaCO}_3 \Rightarrow 1.8 \text{ mg of } \text{CaCO}_3$$

2. Estimation of total hardness in water

sample $\Rightarrow 20 \text{ ml hard water} = 7.4 \text{ ml EDTA}$
 $\Rightarrow 7.4 \times \frac{20}{11.1} \text{ mg of } \text{CaCO}_3$

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

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

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

(total hardness)

3. Estimation of hardness in water sample

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

Ca²⁺ hardness in water $\approx 405.5 \text{ ppm}$

4. Mg²⁺ hardness in water sample

\Rightarrow total hardness - Ca²⁺ hardness

$$\Rightarrow 666.7 - 405.5 \Rightarrow 261.2 \text{ ppm}$$

Results :-

1. Ca²⁺ hardness in water sample = 405.5 ppm

2. Mg²⁺ hardness in water sample = 261.2 ppm

taking concordant readings. Let V_1 ml of EDTA solution be used,

Total hardness in water sample \Rightarrow
titrate 20 ml of given hard water
sample against EDTA as in step 1.
Let volume of EDTA used be V_2 ml.

Ca^{2+} Hardness in water sample \Rightarrow
Pipette out 20 ml 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 change to
purple. Record burette readings.
Take concordant readings. Let V_3
ml of EDTA is used in this step.

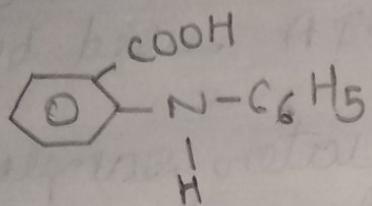
Result :-

-
1. Ca^{2+} hardness in water sample = 405.5 ppm
 2. Mg^{2+} hardness in water sample = 261.2 ppm

Experiment - 7

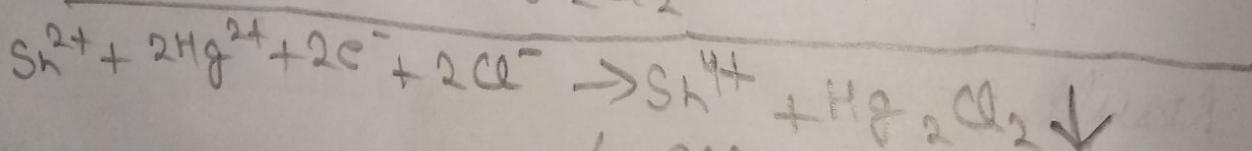
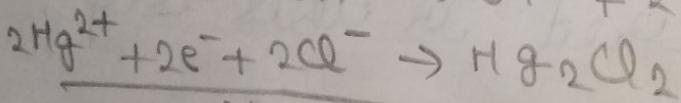
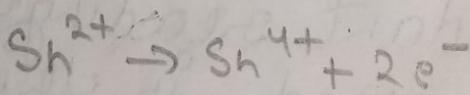
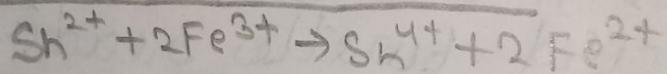
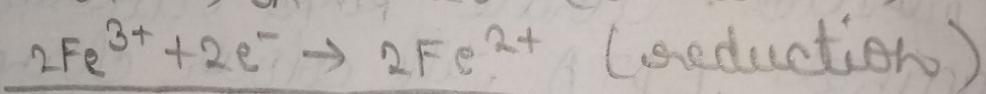
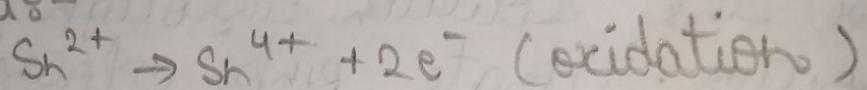
Aim :- To determine amount of total iron in an iron ore solution by internal indicator method.

Indicator :- N-phenylanthranilic acid

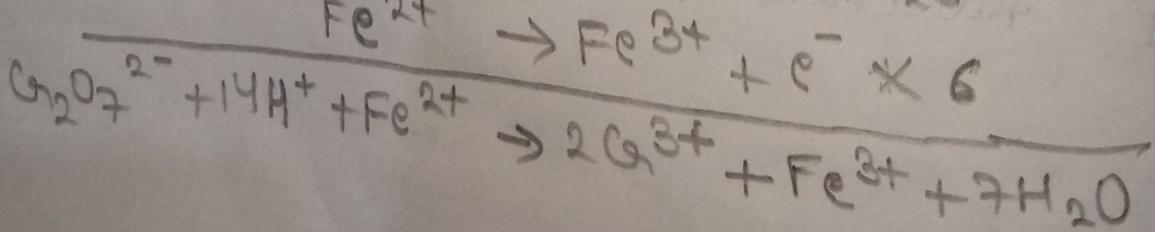
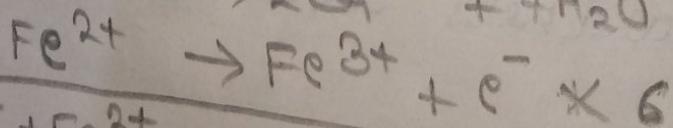
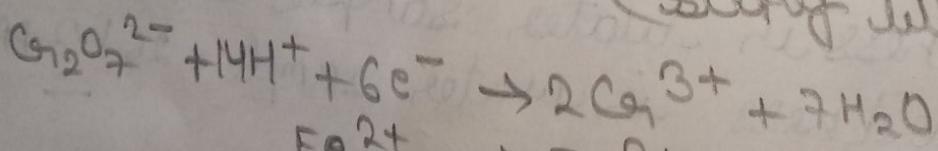


End point :- colour changes from light green to reddish violet

Reagents used :-



(silvery white ppt)



Experiment - 7

Aim :- To determine amount of total iron in an iron ore solution by internal indicators 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 Required :- Burette, pipette, titration flask, 250 ml volumetric flask, 500 ml beaker, test tube, measuring cylinder, filter paper, zinc dust etc

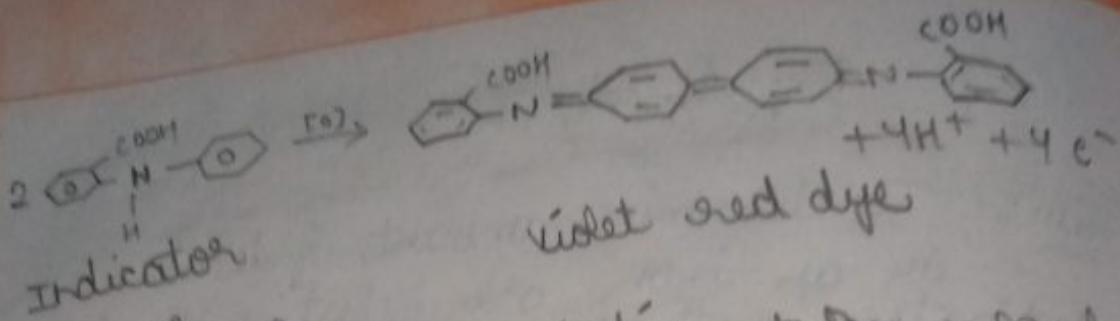
Indicators :- N-Phenylanthranilic acid

End Point :- colour changes from light green to reddish violet

Theory :- Determination of iron content using internal indicator \Rightarrow

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

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Observations :- vol^m of ore solution taken = 20 ml

S.No.	Initial burette reading	Final burette reading	vol ^m of K ₂ C ₂ O ₇ sol ⁿ used
1.	0	13	13
2.	13	26.5	13.5
3.	26.5	39.5	13

concordant volume (V_2) = 13 ml

Calculations :- (i) Normality of ore solution
vol^m of ore solution taken for titration = 20 ml

concordant vol^m of K₂C₂O₇ solⁿ used = 13 ml

Normality of K₂C₂O₇ solution = N/10

Applying Normality equations

$$N_1 V_1 = N_2 V_2$$

(ore) (K₂C₂O₇)

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

$$N_1 = 0.065 \text{ N}$$

(ii) Strength of total iron in ore solⁿ

of 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 agent 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 acidified medium 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 solution in acidic medium using N-phenylanthranilic 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 eye.

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

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Strength of total iron in ore solution

$$= \text{Normality} \times \text{eq^n weight of iron}$$

$$= 0.065 \times 56 \text{ gm/lt}$$

$$= 3.64 \text{ gm/lt}$$

$$= 3.64 \times 10^3 \text{ ppm} \Rightarrow 3640 \text{ ppm}$$

$$= 3.64 \times 10^3 \text{ ppm} \Rightarrow 3640 \text{ ppm}$$

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 sticky 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 point of titration. Note final reading. Repeat the titration to record concordant value.

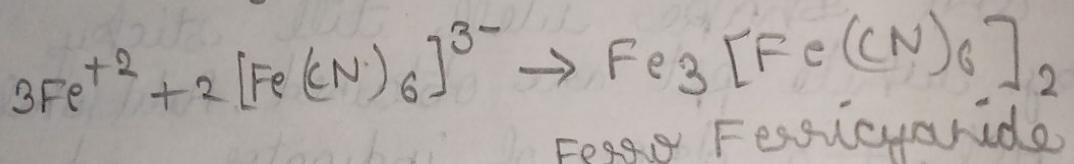
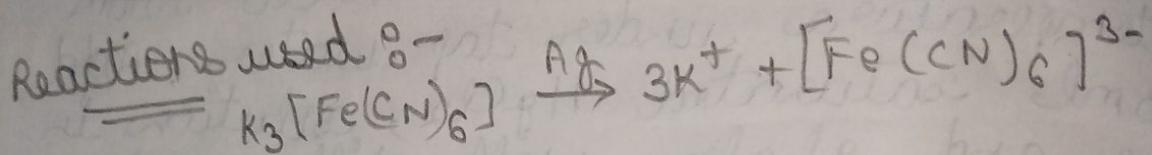
Results - Normality of ore solution = 0.65 N
 Strength of total iron in ore solution
 = 3.64 g/litre
 The given ore solution contain 3.64 gm/l
 litre of total iron.

Experiment - 8

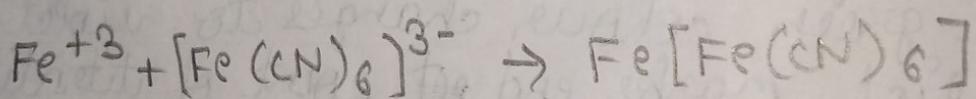
Aim :- To determine the amount of total iron in an iron ore solution by external indicator method.

Indicator :- Potassium ferricyanide

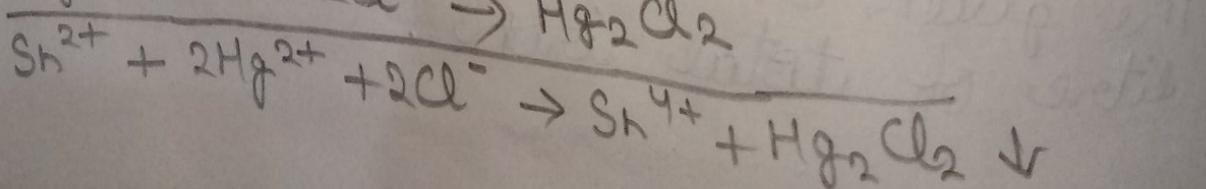
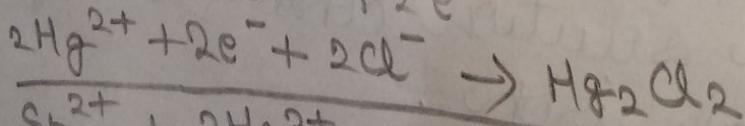
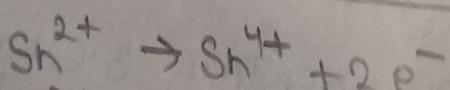
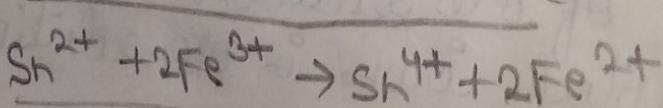
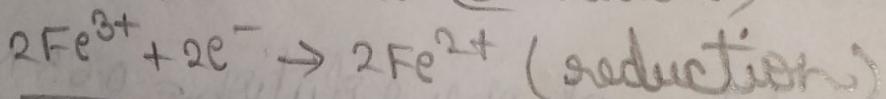
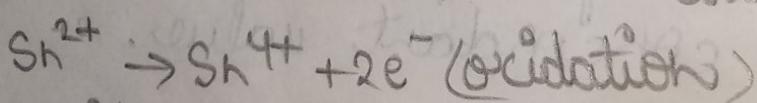
End point :- Non appearance of bluish green colour externally.



(Prussian blue colour)



colourless



Experiment - 8

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

Apparatus Required :- Burette, Pipette, titration flask, 250 ml volumetric flask, 500 ml beaker, test tube, measuring cylinders, filter paper, zinc dust, etc.

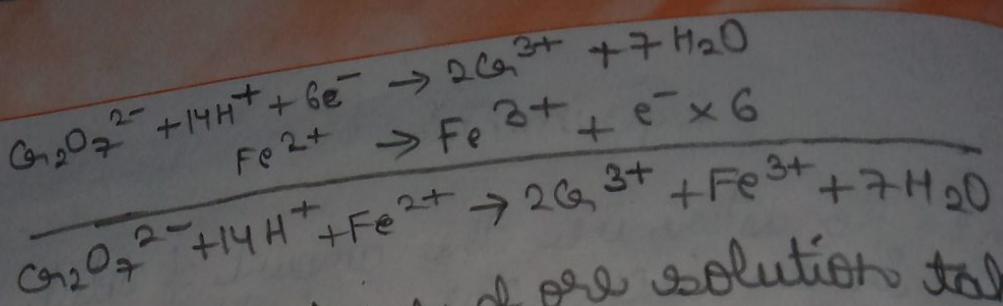
Chemicals Required :- 5% stannous chloride solution, saturated mercuric chloride solution, standard potassium dichromate solution, $\text{Cr}_2\text{O}_7 \text{HCl}$ and dil. HCl.

Indicator :- Potassium ferricyanide

End point :- Non appearance of bluish green colour externally.

Theory :- Determination of iron content using ext. indicator
The ore solution is reduced to

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Observations :- volume of ore solution taken
= 10 ml

S.N.	Initial burette reading	Final burette reading	Vol. of $\text{K}_2\text{Cr}_2\text{O}_7$ used (ml)
1.	0	9.5	9.5
2.	9.5	18.9	9.4
3.	18.9	28.4	9.5

concordant reading (v) = 9.5 ml

Calculations :-

1. Normality of ore solution

volume of ore solution taken for titration
 \Rightarrow 10 ml

concordant volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution used
 \Rightarrow 9.5 ml

Normality of $\text{K}_2\text{Cr}_2\text{O}_7$ solution = $\frac{N}{10}$

Applying Normality equation

$$N_1 V_1 = N_2 V_2$$

(ore) ($\text{K}_2\text{Cr}_2\text{O}_7$)

$$N_1 \times 10 = \frac{1}{10} \times 9.5$$

$$N_1 = 0.095 N$$

slight excess of stannous chloride as in case of internal indicator. The reduced solution of is then titrated against standard dichromate solution in acidic medium using potassium ferricyanide as an external indicator to find out end point of titration. It reacts with Fe^{+2} ions of ore solution to give prussian blue colour. At end point, when all Fe^{+2} ions get oxidised to Fe^{+3} ions, bluish green colour does not appear with indicator.

From the volume of $\text{K}_2\text{Cr}_2\text{O}_7$, used at end point the amount of total iron in ore solution can be calculated in the form of ferrous ions.

Use of external indicator \Rightarrow Place a series of small drops of freshly prepared potassium ferricyanide indicator on a white tile by means of glass rod. Run in dichromate solution from the burette in lots of iron ore solution and shake. Take out a drop of the reaction mixture from the conical flask with the help of glass rod, mix

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2. Strength of total iron in ore solution

\Rightarrow

Strength of total iron in ore solution

= Normality \times eqⁿ weight of ironate

$$= 0.095 \text{ N} \times 56$$

$$= 5.32 \text{ gm/lt}$$

$$= 5.32 \times 10^3 \text{ PPM} \Rightarrow 5320 \text{ PPM}$$

$$= 5.32 \times 10^3 \text{ PPM} \Rightarrow 5320 \text{ PPM}$$

Results :- The amount of total iron in the solution is 5.32 gm/lt

it with one drop of indicator on the tile. If a blue colour is produced, continue the process till a drop of reaction mixture gives no blue colour on the tile. This is the end point.

Procedure :-

Step I \rightarrow Reduction of ore solution

Pipette out 10 ml of iron ore solution into a conical flask. Add 3 ml conc. HCl and heat to boiling to get yellow solution. Add 5% SnCl₂ solution dropwise with shaking till yellow colour just disappears. Add 2-3 drops in excess. Cool the solution to room temperature under tap water.

Add 3 ml saturated HgCl₂ solution in one lot, and shake well till silky white ppt is formed.

Step - II \rightarrow Titration of reduced solution
vs K₂Cr₂O₇ soln

(i) For long range reading \Rightarrow Fill the burette with standard K₂Cr₂O₇ after rinsing. Add 10 ml dil. HCl to reduced

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iron solution. Add 5 ml dichromate solution from burette in lot. Shake contents well. Take out a drop of this solution with a clear tile and check for end point. Add dichromate sol' in 5 ml lots and check for end point till it is obtained. Record the volume of dichromate used as long range reading.

(ii) For short range reading \Rightarrow Reduce the iron solution. Acidify it and add dichromate solution from burette in 1 ml lots starting from lower end of long range reading. After every addition check for the end point externally. Record the volume.

(iii) For decimal reading \Rightarrow Reduce the iron ore. Acidify it with dil. HCl. Add dichromate solution in 0.1 ml lots starting from lower end of short range reading till end point is obtained. Record decimal reading.

Teacher's Signature.....

(iv) For concordant reading \rightarrow take three concordant readings with reduced iron ore solution.

Results :- The amount of total iron in ore solution is 5.32 g/l.t.

Experiment - 9

Aim :- To determine the surface tension of a given liquid by drop number method using stalagmometer.

$$\underline{\text{Formula used :-}} \quad \frac{\sigma_1}{\sigma_2} = \frac{h_2}{h_1} \times \frac{d_1}{d_2}$$

where σ_1 = surface tension of given liquid

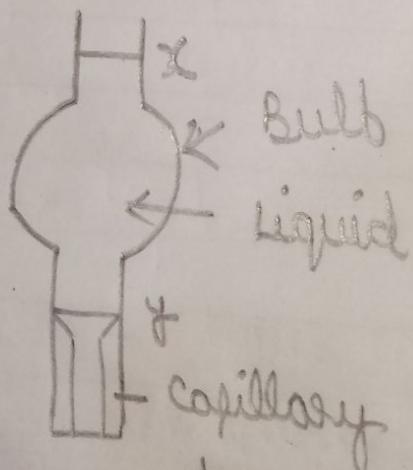
σ_2 = surface tension of water

h_1 = no. of drops counted for given liquid

h_2 = no. of drops counted for water

d_1 = density of given liquid

d_2 = density of water



stalagmometer

Experiment - 9

Aim :- To determine the surface tension of a given liquid by drop number method using stalagmometer.

Apparatus Required :- Stalagmometer, rubber tubing, pinch cock, beakers, relative density bottle, thermometer, stand, weight box, fractional weights etc.

Chemicals Required :- Given liquid, distilled water

Theory :- Surface tension is the property of a liquid due to which its free surface behaves as a stretched membrane and tends to have minimum surface area. It is defined as the force in dynes along the surface of liquid at right direction angles to a line of 1 cm.

When a liquid is allowed to flow through a capillary tube, a drop

Observation table :-

Room temperature = 21°C

S.No.	Water	Liquid	No. of drops	Mean (n ₁)
1.	41	44	38	41
2.	45		41	
3.	46	44	44	

Calculation :-

By applying formula

$$\frac{n_1}{n_2} = \frac{n_2}{n_1} \times \frac{d_1}{d_2}$$

$$\therefore \frac{n_1}{n_2} = \frac{44}{41} \times \frac{1.59}{1} \times 751.64$$

$$\therefore n_1 = 129.067 \text{ dynes/cm}^{-1}$$

\Rightarrow Surface tension of given liquid is 129.067 dynes/cm

Result :-

Surface tension of given liquid is 129.067 dynes/cm

It is equal to 129.067 mN/m

is formed at its lower end. The drop increases to a certain size and then falls off. The size of the chunk drop depends on the radius of capillary and the surface tension of the liquid. The surface acting along the circumference of the capillary tube supports the drop in upward direction. The measurement of the surface tension of a liquid is based on the fact that the drop of the liquid at lower end of the capillary tube falls down when weight of the drop just becomes equal to the surface tension.

Consider two liquids of densities d_1 and d_2 having the surface tension σ_1 and σ_2 respectively. Let the number of drops counted for the same volume V of the two liquids be n_1 and n_2 respectively. Then

$$\frac{\sigma_1}{\sigma_2} = \frac{n_2}{n_1} \times \frac{d_1}{d_2}$$

Procedure :-

1. Clean the stagonometer first with NaOH solution then with chrome

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acid solution and finally a number of times with distilled water. Rinse it with acetone and dry by passing hot air or in an oven.

2. Attach a small piece of clean rubber tubing provided with a screw pinch cock to the top of stalagmometer.
3. Now open the pinch cock and immerse the lower end of stalagmometer in a beaker containing distilled water. Suck up the water about 2 cm above the mark X on stalagmometer and close the pinch cock.
4. Control the rate of ~~loss~~ flow of water by adjusting the pinch cock so that numbers of drops per minute is 10-15.
5. Clamp the stalagmometer in the stand. Refill it with water above the mark X. Start counting the number of drops when water meniscus crosses the upper mark X and stop counting when

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water meniscus passes the lower mark
Y.

6. Repeat the experiments to get three readings.

7. Rinse the stalagmometer with alcohol and dry it again.

8. Fill it in the same way with the given liquid and find out the no. of drops formed in falling of liquid between marks X and Y on stalagmometer. Repeat to get three readings.

9. Fill it with water and weight it again.

10. Remove water, rinse with alcohol and dry the density water. Fill it with the given.

11. Note the room temperature with a thermometer.

Precautions :-

1. Stalagmometer should be cleaned thoroughly as surface tension is greatly effective by minute traces of impurities.
2. Stalagmometer should be held in a vertical position throughout counting period.
3. Drop formation should not exceed 15 drops per minute.
4. The rubber tubing attached to stalagmometer should be perfectly dry and the liquid sucked should not touch the rubber tube.
5. The drops should fall from the tip of the stalagmometer under their own weight and should not be pushed by force.

Result :- Surface tension of the given liquid is $129.067 \text{ dynes cm}^{-1}$.