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General Laboratory instructions / precautions

1. Student should bring the following items before coming :

(a) Laboratory coat (b) Chemistry pratical note book
(c) Rough notebook (d) Pen, pencil and Eraser

2. It should be compulsory to wear laboratory coat while working in chemistry laboratory.
3. For real understanding of principle, techniques and procedure, the students should plan his/her work in advance & work purposefully during lab. period.
4. All chemicals in the laboratory are hazardous in some way or other. They should be handled carefully.
5. To prevent accidents, suitable precautions should be taken while working in lab. Always follow the instructions exactly & in sequence given by instructor.
6. Do not throw waste material into sink. Throw them only in the wastebasket.
7. Pieces of Na metal should not be thrown into the sink or waste jar. It may be destroyed by reacting it with alcohol. Na must be kept in toulene or kerosene.
8. Experiments should be done only in the presence of the instructor. Never work alone in the lab.
9. All the doors, windows must remains open while working in lab.

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10. Equipments, glassware, reagents and bottle and other items should be placed in a schematic manner.
11. Check all the glassware before use. Never use unclean, ordinary, cracked glassware for any exp.
12. If a student has broken any apparatus bring this fact immediately to the notice of lab. staff.
13. The experiment work should be done systematically.
14. Start noting down the readings of the experiments, get it checked by the teacher concerned.
15. While heating a test tube never point its mouth towards yourself & any other else.
16. Clean all ~~the~~ glassware after completing experiment
17. Do not take the chemical, glassware out of the lab.
18. Follow all the instructions given by supervisor.

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Instructions of Lab.

DO's:

1. Please switch off the mobile before entering lab.
2. Enter in the lab with complete lab uniform.
3. Intimate the lab in charge whenever you are incompatible of doing experiment.
4. Arrange all the apparatus, seats before leaving the lab.
5. Keep the bag in the racks.
6. Enter the lab on time., leave at proper time.
7. Maintain the decorum of the lab.
8. Utilize lab hours ~~in performing~~ the alloted experiment
9. Switch off the equipment immediately after completing the experiment.
10. Handle the equipments carefully in the lab.

Dont's:

1. Don't mishandle the equipments.
2. Don't make noise in the lab.
3. Don't enter in the lab without permission of lab manual files.
4. Don't litter in the lab.
5. Don't delete or make any modification in lab files
6. Don't carry chemicals outside the lab.

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List of Practical

Name of Practical

1. Determination of Hardness by HCl method.
2. Determination of Hardness by Edta method
3. Determine conductivity of diff. water sample.
4. Proximate analysis of coal sample.
5. Determination of flash & fire point.
6. Determination of cloud pt. & pour pt & Redwood Viscometer.
7. Determination of viscosity of Redwood Viscometer.
8. Determination of alkalinity of water
9. Determination of strength of $CuSO_4$
10. Determination of water sample by pH meter.
11. Determination of surface tension by Stalgmometer.

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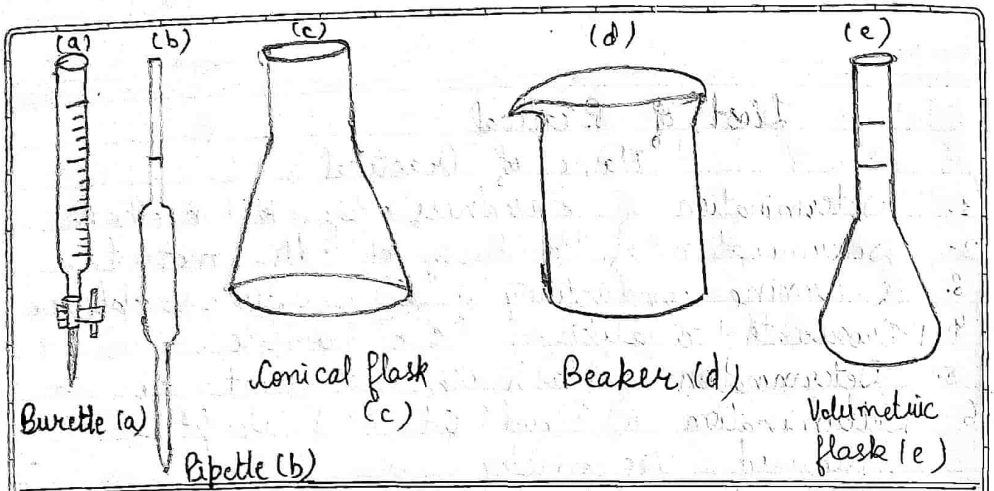

Reactions :

$Mg(HCO_3)_2 \xrightarrow{\text{Boiling}} Mg(OH)_2 + 2CO_2$

$Ca(HCO_3)_2 \xrightarrow{\text{Boiling}} CaCO_3 + CO_2 + H_2O$

$Ca(HCO_3)_2 + 2HCl \rightarrow CaCl_2 + 2CO_2 + 2H_2O$

$Mg(HCO_3)_2 + 2HCl \rightarrow MgCl_2 + 2CO_2 + 2H_2O$

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Experiment No = 1

Object : To determine the hardness of water by HCl.

Apparatus : Burette, Pipette, Conical flask, beaker, Volumetric flask, water sample, Std. N/40 HCl, Methyl Orange (indicator)

Theory : Temporary hardness in water is due to bicarbonates of hardness producing metal (Ca^{2+}, Mg^{2+}). The temporary hardness is removed by prolonged boiling because of the evolution of CO_2 and the ppt of corresponding carbonates :

Since, these bicarbonates also contributes towards the alkalinity of the water, the temp. hardness can be determined by estimating the alkalinity of the water sample before & after boiling by titrating with Std. N/40 HCl.

Alkalinity of water may be caused due to :

1. NaOH & KOH
2. Na_2CO_3 & K_2CO_3.
3. $NaHCO_3$ & $KHCO_3$.
4. $Ca(HCO_3)_2$ & $Mg(HCO_3)_2$, contributes towards temporary hardness.

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Observation :

S.No.	Vol of water-Sample taken (ml)	Vol of N/40 HCl consumed (ml)				A = a - b
		Before boiling	Concordant reading (a)	After boiling	Concordant reading (b)	
1.	10	2.0	2.0	0.4	0.4	2.0 - 0.4
2.	10	2.0	2.0	0.4		= 1.6
3.	10	2.0	2.0	0.4		

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Procedure :

1. First wash all the apparatus with dist water.
2. Rinse & fill burette with N/40 Std. HCl and note the intial reading.
3. Pipette out of 10 ml of water sample into a conical flask, add 2-3 drops of methyl orange indicator.
4. Now add HCl acid from the burette with constant string the titration mixture. When a sharp colour change is noticed from yellow to cherry red. Note down the burette reading.
5. Repeat the titration till the two concordant reading are obtained.
6. Gently boil 50 ml of freshly water sample for about half an hour, bicarbonates are completely decomposed.
7. Titrate the boiled water against HCl acid acid of burette using methyl orange as an indicator & note down the reading when colour change from yellow to cherry red.
8. Repeat the procedure till the two concordant readings are obtained.

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Calculation:

The volume of N/40 HCl consumed in removing the temporary hardness in 10 ml of water = A ml

Volume of the N/40 HCl required to remove the temporary hardness present in 1000 ml of the water sample = A × 1000/10

As we know that:

1 ml of 1 N HCl = 50 mg of $CaCO_3$

A × 1000/10 of N/40 HCl = (A × 1000/10)50 × 1/40 mg/L

Hardness = $10 \times \frac{1000}{10} \times \frac{50}{40}$ mg/L

= 200 mg/L

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Expected Outcomes:

The temporary hardness of the water sample = 200 ppm

Achieved Outcomes:

The temporary hardness of the water sample = 200 ppm

Precautions:

1. Carefully observe the colour change at end point
2. Use only 1-2 drops of indicator.
3. All the solution should be freshly prepared.

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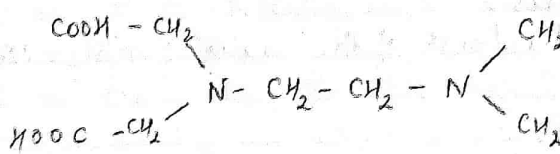
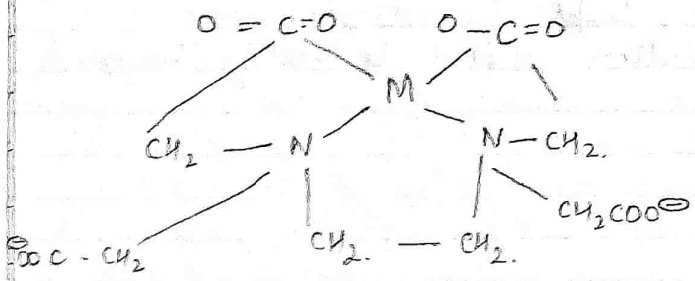
① EDTA Structure :



② EDTA form complex with Metal (Ca^{2+} or Mg^{2+})



[M = Ca^{2+} or Mg^{2+}]

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Experiment No = 2

Object = To determine the hardness of given water sample by complexometric method using EDTA.

Apparatus : Burette, pipette, conical flask, beaker, burner, Std. M/100 EDTA solution, Eriochrome black T indicator, NH_4Cl- NH_4OH buffer solution of pH 10, hard ~~sample~~.

Preparation of $NH_4Cl - NH_4OH$ of pH - 10 :
70 gm of A.R NH_4Cl, 568 ml concentrated ammonia solution stir, dil sol^n to 1 l with deionised water.

Preparation of M/100 EDTA Sol^n :
Dry 5 gm of commercially available A.R. EDTA in an air oven at 80°C for 1/2 hour. Cool and accurately weigh 3.7224 gm and dissolve it in distilled water, makeup to 1 l. This may be used as std. sol^n.

Prep^n of Eriochrome Black T- indicator :
Dissolve 0.5gm of Eriochrome Black-T indicator in 100 ml alcohol.

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Theory:

Ethylene diamine tetra acetic acid (EDTA) is a well known powerful complexing agent.

EDTA forms complexes with Ca^{2+} and Mg^{2+} ions as well as with many other metal cations.

When hard water is titrated against EDTA sol^{n}, then EDTA or its sodium salt forms complexes with Ca^{2+} or Mg^{2+} ions in water.

$M + EDTA \longrightarrow M-EDTA$

(Stable Complex)

The titration is carried out in presence of an indicator Erichrome black T. When indicator is added to hard water which is buffered at pH 10, then it combines with Ca^{2+}, Mg^{2+} ions to form weak complex of wine red colour. When an excess of EDTA is added then colour changes to blue due to Ca^{2+} / Mg^{2+}. EDTA complex. Thus change of wine Red to distinct blue marks the end point of titration.

$M + EBT \rightarrow M-EBT$ (Unstable wine Red)

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

Blue. Stable.

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Observation :

① Titration : EDTA V/S Std. hard water (V_1 ml)

S.No.	Vol of water sample (ml)	Burette reading		Concordant reading
		Initial	final	
1.	10	0	7	7
2.	10	0	7	
3.	10			

② Titration : EDTA V/S ~~Sample~~ hard water (V_2 ml)

S.No	Vol of water	Burette reading		Concordant reading
		Initial	final	
1.	10	0	1.5	1.5
2.	10	0	1.5	
3.	10			

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Procedure :

1.) Pipette out 10 ml of std. hard water in conical flask.
2.) Add 2 ml of buffer sol^n, 2 drops of EBT as
3.) Titrate the sol^n against std. M/100 EDTA from burette till colour changes from red to blue.
4.) Repeat it to get concordant titre.
5.) Repeat the process with sample hard two concordant readings.
6.) Pipette out 10 ml of boiled conical flask, add 2ml of drops of Eriochrome Black
7.) Titrate it with M/100 appears, at end point
8.) Repeat the procedure This corresponds

③ Titration : EDTA VIS Boiled Water (hard) [V_3 ml]

S.No.	Vol of boiled water sample (ml)	Burette reading		Concordant reading (ml)
		Initial	final	
1.	10	0	0.5	0.5
2.	10	0	0.5	
3.	10			

Calculation :

Total hardness = $V_2 / V_1 \times 1000$ ppm.

Permanent hardness = $V_3 / V_1 \times 1000$ ppm.

Temporary hardness = Total − Permanent hardness

Total hard = $\frac{1.5}{7} \times 1000$.

Permanent = $\frac{0.5}{7} \times 1000$

Temp = $1000 \times \left(\frac{1.5 - 0.5}{7}\right) \Rightarrow 142.85$ ppm

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Expected outcome :

The temporary, permanent and total hardness of water sample comes out to be 142.85 ppm.

Achieved outcome :

The temporary, permanent and total hardness of water sample is 142.85 ppm.

Precautions :

1) Titration should be performed slowly near end point.
2) There should not be any tinge or reddish blue colour at end point
3) pH of the solution maintained properly

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Experiment No. 13

Object:
To determine the conductivity of given water samples by conductivity water meter

Apparatus and reagent required:
Conductivity meter, water samples, distilled water, 0.01 N KCl solution, Beaker etc.

Theory =
Conductivity is a good indication of water quality. It is the ability of a material to transfer an electric charge from one point to another. If a sol^{n} is a good conductor, it consists primarily of ions. If a solution is a poor conductor, it primarily consists of molecule. The conductivity of a sol^{n} is measured with the help of a conductivity cell using conductivity meter.

Procedure -
* Switch on the conductivity meter for about 15 to 20 min for its warm up.
* Wash the conductivity cell in distilled water.
* Set the function switch to check position.
* Display must read 1.000 otherwise set it with CAL control knob of conductivity meter.

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Observation-

S.No.	Water sample sol^n	Conductivity
1.	A	0.50 x 0.984
	B	
2.		2.94 x 0.984
3.	C	6.23 x 0.984

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★ Adjust the temp. knob to room temp.
★ Dip the washed and dried conductivity cell in the beaker containing sample sol^n.
★ After completion of experiment switch off the apparatus and put the cell in distilled water.

~~Observation~~ -

Result -

Conductivity of the given water sample are as follows

Sample	Conductivity
A	0.49
B	2.88
C	6.10

Precautions -

★ Always calibrate the cell constant of conductivity cell.
★ Temp. of the sample should be carefully noted as conductivity varies with the temp.
★ The conductivity cell should be handled carefully.
★ Conductivity cell should be completely dipped in the solution.

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