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Section: 1

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Manvendra Singh, section 1,
Chemistry practical file,

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1. To prepare and standardize the solution of NaOH (N/10) against standard solution of Oxalic acid (N/5).
2. To determine the constituent and amount of alkalinity in the given water sample by titrating it against standard HCl solution (N/20) using phenolphthalein and methyl orange as an internal indicators.
3. 6 To determine the temporary, permanent and total hardness of water in a given sample of water by titrating it against standard EDTA solution (N/20) using Eriochrome Black -T as an internal indicator.
4. To determine chloride ion content in a given water sample by Argentometric method (Mohr's method) using K_2CrO_2 as an internal indicator



Practical ---->1

Object::

To prepare and standardize the solution of NaOH (N/10) against standard solution of Oxalic acid (N/5).

Theory:

Standard solution is one in which exact amount of a substance is present in a definite volume of the solution, or a solution whose concentration (strength) is known to us is also called as standard solution.

Volumetric solutions are classified into following two types:

1. Primary Standard Solution.
2. Secondary Standard solution

Primary Standard Solution: The substance whose standard solution is prepared by dissolving directly its known amount in a definite volume of solvent or solution is known as a primary standard substances & the solution is called as primary standard solution. Commonly used primary standard substances are anhydrous Na_2CO_3 , Oxalic acid etc.

Secondary Standard Solution: The substance whose solution cannot be prepared directly by weighing its definite amount and then dissolving in definite volume of solvent is called secondary standard substance & the solution is called as secondary standard solution. The solution of this type of substance firstly prepared is of approximate strength which is then standardized with a standard solution of a primary standard substance. The common secondary standard substances are alkali hydroxides, inorganic acids and KMnO_4 etc.

Classification of Methods of Volumetric Analysis:

Volumetric analyses are of following types

1. Neutralization titrations or Acid Base titrations
2. Oxidation-reduction titrations
3. Precipitation titrations

4. Complexometric titrations

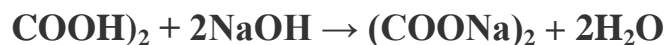
Normality (N): The normality of a solution is the number of gram-equivalents of the solute per litre of the solution.

$$N = \frac{\text{no. Of gram equivalent of solute}}{\text{volume of solution in 100 ml}}$$

Molarity of the solution can be calculated by using the formula:

$$a_1 M_1 V_1 = a_2 M_2 V_2 \dots$$

where a_1 , M_1 , V_1 are respectively basicity, molarity and volume of acid used and a_2 , M_2 and V_2 are acidity, molarity and volume respectively of base used in the titration.



Material Required:

- Burette
- Pipette
- Conical flask
- Burette stand
- Funnel
- Stirrer
- White glazed tile
- Measuring flask
- Oxalic acid (solid)
- Oxalic acid (as per needed)
- Sodium hydroxide solution (as per needed)

- Phenolphthalein indicator (as per needed)

Titration of Sodium Hydroxide and Oxalic Acid Solution

1. Rinse the burette with the standard oxalic acid solution.
2. Take 10cm³ of oxalic acid solution in a titration flask. Fill the burette with sodium hydroxide solution.
3. Remove the air gap if any, from the burette by running the solution forcefully from the burette nozzle and note the initial reading
4. Pipette out 20ml of NaOH solution in a conical flask. Add 2-3 drops of phenolphthalein indicator to it.
5. Titrate the base with oxalic acid solution until pink colour disappears.
6. Repeat the titration till three concordant readings are obtained.

Observation table:

S.No	Volume of the NaOH soln. taken for titration (ml)(V ₁)	Burette Initial	Reading Final	Volume of std. Solution of oxalic acid consumed(ml) (v ₂)
1	10	0	11.2	11.2
2	10	11.2	22.4	11.2
3	10	22.4	33.6	11.2
4	10	33.6	44.8	11.2

Calculation of the normality of the solution prepare

$$\begin{array}{ccc}
 N_1 V_1 & = & N_2 V_2 \\
 \text{(NaOH)} & & \text{(Oxalic acid)}
 \end{array}$$

$$N_1 \times 10 = 11.2/5$$

$$N_1 = 0.224 \text{ N}$$

- If the strength of the solution is less than the desired strength then repeat the experiment with the fresh preparation of the sample.
- If the strength of the solution is more than the desired strength follow the method below.

$$N_1 V_1 = N_2 V_2$$

(For NaOH soln. prepared) (For NaOH soln. of Required Strength)

$$0.224 \times V_1 = 0.1 \times 100$$

$$V_1 = 44.64 \text{ ml}$$

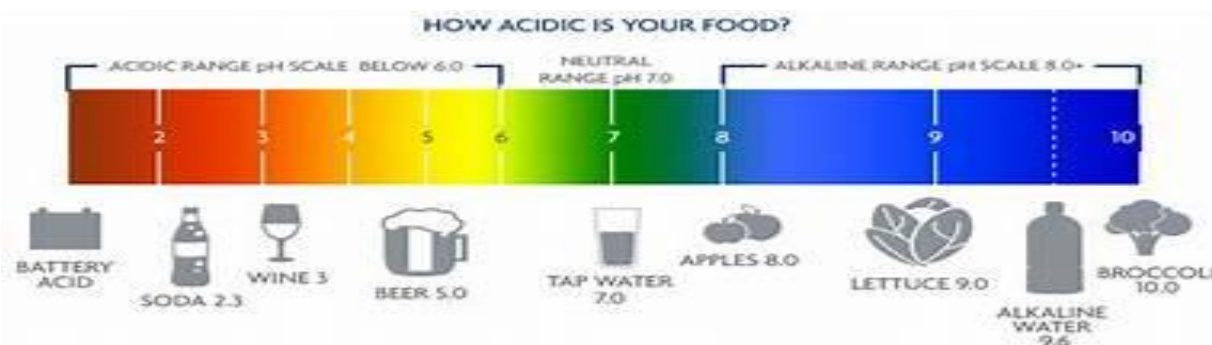
Volume of distilled water required for dilution = $(100 - V_1)$ ml

$$100 - 44.64 = 55.36 \text{ ml}$$

Result: Standard solution of N/10 NaOH solution is prepared.

Precautions:

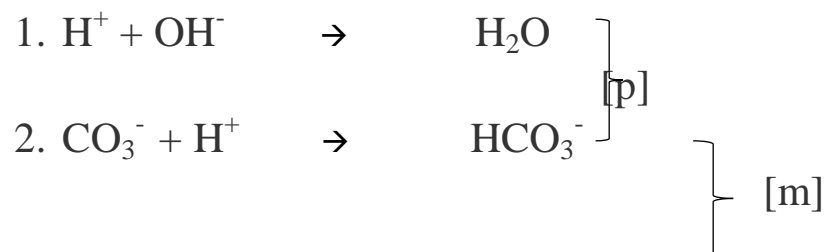
1. Solution should be making up to desired volume the solute.
2. During titration the solution should be stirred thoroughly
3. Do not take mean of burette readings.

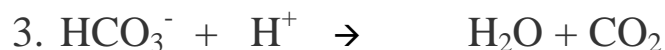


Practical ---->2

Object: To determine the constituent and amount of alkalinity in the given water sample by titrating it against standard HCl solution (N/20) using phenolphthalein and methyl orange as an internal indicators.

Materials/Chemicals/Apparatus required: N/20 HCl solution, phenolphthalein and methyl orange indicators, sample solution, burette, pipette, conical flask, beaker, funnel, etc. Theory: The alkalinity in water is due to the presence of hydroxyl ion (OH^-), carbonate ion (CO_3^{2-}) and bicarbonate ion (HCO_3^-) present in the given sample of water. These can be estimated separately by titrating against standard acid (N/20 HCl) using phenolphthalein and methyl orange as indicators. The chemical reaction involved can be shown by the equations given below:





[p]- stands for phenolphthalein

[m] - stands for methyle orange indicator

Procedure:

1. Rinse and fill the burette with N/20 HCl.
2. Pipette out 10 ml of water sample into a conical flask. Add 1-2 drops of phenolphthalein indicator. A pink color will appear.
3. Add HCl from burette drop by drop till the pink color just disappears. Note this reading.
4. Now add 1-2 drops of methyl orange indicator to above solution. A yellow color will appear.
5. Add N/20 HCl drop by drop to it till the yellow color just changes to red. Note this reading.
6. Repeat this titration until two concordant readings are obtained.

Observation Table:

S.No	Volume Of sample Solution (ml)	Burette reading				Volume Of HCL with phenolphthalein	Volume Of HCl with methyle orange
		Using phenolphthalein		Using methyle orange			
		Initial	Final	Initial	Final		
1.	10	0	0.6	0.6	2.2	1.2	1.6

Calculations:

2(1) For CO_3^{2-}

$$N_1 V_1 = N_2 V_2 \rightarrow N_1 * 10 = \frac{1}{20}(2p) = \frac{1}{200}(1.2) * 50$$

(For water sample) (for HCl) $N_1 = 0.3$

Volume of HCl used (V_2) = $2P = 1.2 \text{ ml}$

Strength = $N_1 * \text{equivalent wt. of } \text{CO}_3^{2-} \times 1000 \text{ ppm} = 9 \text{ kppm}$

Alkalinity = $N_1 * \text{equivalent wt. of } \text{CaCO}_3 \times 1000 \text{ ppm} = 15.013 \text{ kppm}$

(2) For HCO_3^-

$$N_1 V_1 = N_2 V_2 \rightarrow N_1 * 10 = \frac{1}{20}(m-p) = \frac{1}{200}(2.2-0.6) * 50$$

$N_1 = 0.4$

(For water sample) (for HCl)

Volume of HCl used (V_2) = $M - P = 1.6 \text{ ml}$

Strength = $N_1 \times \text{equivalent wt. of } \text{HCO}_3^- \times 1000 \text{ ppm} = 24.4 \text{ kppm}$

Alkalinity = $N_1 \times \text{equivalent wt. of } \text{CaCO}_3 \times 1000 \text{ ppm} = 20.01 \text{ kppm}$

Results:

1. The alkalinity due to presence of CO_3^{2-} is = 9 kppm
2. The alkalinity due to presence of HCO_3^- is = 24.4 kppm

Precautions:

1. Phenolphthalein indicator should be added first and then methyl orange.
2. The volume of indicator should same in all the titrations.

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3. The reaction mixture should be shaken properly.
4. Do not take mean of burette readings



PRACTICAL → 3

Object:

To determine the temporary, permanent and total hardness of water in a given sample of water by titrating it against standard EDTA solution (N/20) using Eriochrome Black-T as an internal indicator.

Materials/chemicals/apparatus required:-

Burette, pipette, conical flask, beaker, measuring cylinder, tripod stand, wire gauze, funnel, filter paper, dropper, standard EDTA Solution (N 20), ammonium buffer solution, Eriochrome Black-T Indicator, Hard water

Principle:

When Eriochrome Black-T (indicator) is added to hard water solution at around (given water sample). 10.0 pH. it gives wine red colored unstable complex with Ca^{2+} and Mg^{2+} ions of the sample water. When this wine red colored complex is

titrated against EDTA solution. The color of the complex changes from wine red to original blue color showing the end point. EDTA (Ethylene Diamine Tetra Acetic acid) is a well-known complexing agent, which is widely used in analytical work on account of its powerful complexing action and commercial availability.

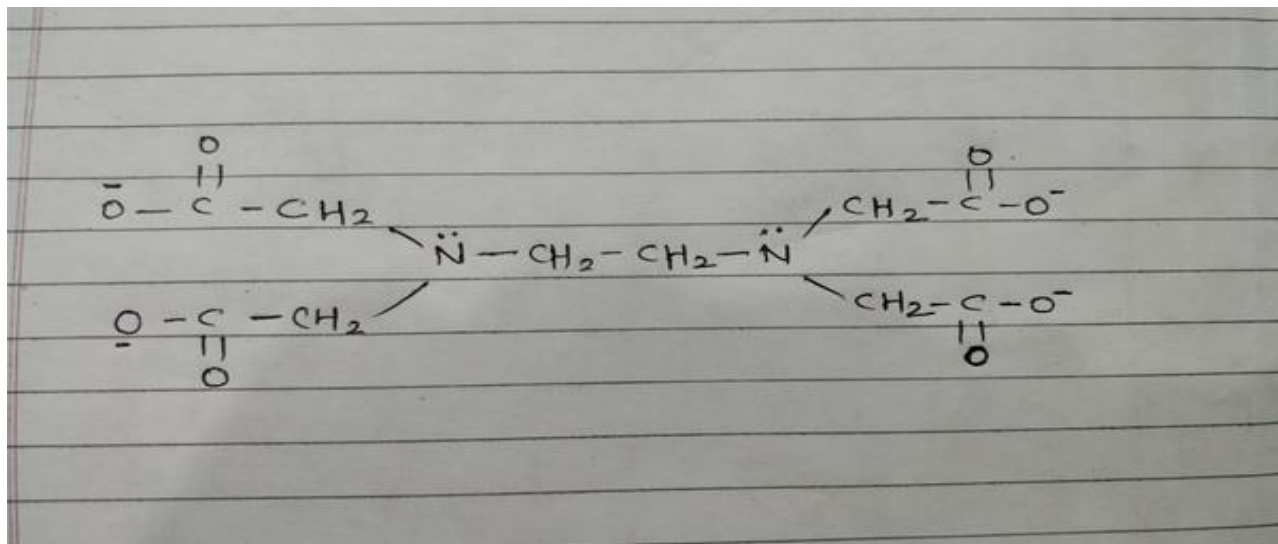


Figure 1: Structure of EDTA

EDTA complex with divalent metal cation ($M=Ca^{2+}$, Mg^{2+}) In aqueous solution EDTA ionizes to give two Na ions and a strong chelating agent. The indicator used is a complex organic compound (sodium-1-(hydroxy-2-naphthylazo)-6 nitro-2 naphthol-4-saiphonate) commercially known as Eriochrome Black-T. It has two ionisable phenolic hydrogen atoms and for simplicity it is represented as $Na+H_2$ Ion-.

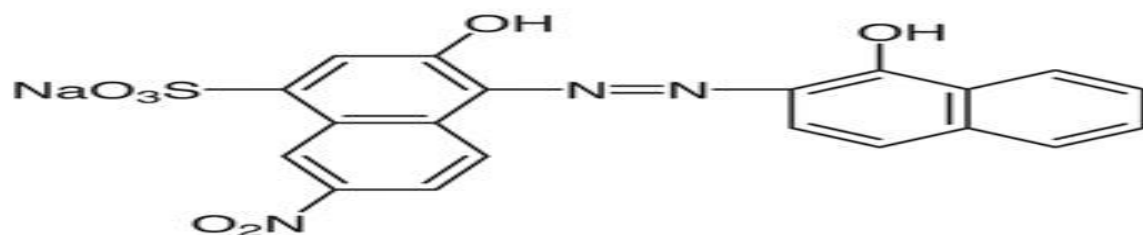


Figure 2: Structure of Eriochrome black-T

Procedure:

1. Rinse the burette with N/20 EDTA soln.
2. Pipette out 10 ml of hard water into a conical flask and add 3 ml of basic buffer solution.
3. Add 2-3 drops indicator Eriochrome Black The color of the soln. becomes wine red.
4. Add drop by drop EDTA soln. from burette till the wine red color just changes to blue.
5. Repeat this titration until two concordant readings are obtained.
6. Take 50 ml of hard water in a beaker and boil it until the volume is reduces to nearly half.
7. Filter it and make it to 50 ml by adding distilled water.
8. Pipette out 10 ml of this water and titrate it with EDTA as given above.
9. Repeat this titration until two concordant readings are obtained.

Observation table:(hard water):

S.No	Vol. of Hard Water(ml)	Burette reading		Vol of EDTA solt.(ml)
		Initial	Final	
1	10	0	3.8	3.8

Observation table:(boiled water):

S.No	Vol. of boiled Water(ml)	Burette reading		Vol of EDTA solt.(ml)
		Initial	Final	
1	10	0	2.7	2.7

Calculations:

1ml 1M EDTA=100 mg of CaCO₃

1ml 1N EDTA=50 mg of CaCO₃

Total hardness →

$$N_1 V_1 = N_2 V_2 \rightarrow N_1 * 10 = \frac{1}{20} * 3.8$$

$$N_1 = 0.019 \text{ N or gm/eq.wt}$$

$$\text{Strength} = N_1 * \text{eq.wt} = 0.019 * 50 \rightarrow 0.95 \text{ gm/ltr} = 950 \text{ mg/L}$$

Permanent hardness →

$$N_1 V_1 = N_2 V_2$$

$$N_1 * 10 = \frac{1}{20} * 2.7 \rightarrow N_1 = 0.0135 \text{ N}$$

$$\text{Strength} = N_1 * \text{eq.wt} = 0.135 * 50 \rightarrow 0.675 \text{ gm/ltr} = 675 \text{ mg/L}$$

Temporary hardness → Total hardness - Permanent hardness = 950 - 675 = 275

Results:

The given water sample contains

- (i) Total hardness= 950 mg/litre or ppm.
- (ii) Permanent hardness=675mg/litre or ppm.
- (iii) Temporary hardness=275mg/litre or ppm.

the result can also be reported in Clark.by dividing all results by 14.3

Precautions:

- 1. The glassware's should be properly rinsed with distilled water.
- 2. The reaction mixture should be shaken properly.
- 3. The end point should be noted correctly.
- 4. The pH should be maintained during titration.
- 5. The amount indicator should be same in all titrations.
- 6. Do not take mean of burette readings



Practical → 4

Object: To determine chloride ion content in a given water sample by Argentometric method (Mohr's method) using K_2CrO_4 as an internal indicator.

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Material/Chemical Apparatus required: Burette, pipette, conical flask, measuring flask, standard silver nitrate solution (N/40), potassium chromate (K_2CrO_4), etc.

Theory: Chloride ions are present in water usually as NaCl, MgCl and $CaCl_2$, Although chloride ions are not harmful as such but their concentration over 250 ppm imparts a peculiar taste to the water thus rendering the water unacceptable for drinking purposes. By Argentometric method chloride ions in a water sample, which is neutral or slightly alkaline. can be determined by titrating it against standard silver nitrate solution using potassium chromate as an internal indicator.



Brick red colour formed due to formation of silver chromate disappears as the solution contains high concentration of Cl



When the concentration of chloride ions has decreased, the red colour starts disappearing slowly on shaking and a stage is reached when all the chloride ions have formed AgCl ppt., then one extra drop of $AgNO_3$ at this point reacts with potassium chromate and forms a reddish coloured ppt of silver chromate.



Procedure:

(A) Blank Correction:

1. Pipette out 10 ml distilled water in a conical flask and add 2-3 drops of K_2CrO_4 indicator.
2. Titrate it against N/40 $AgNO_3$ soln. till the brick red color appears in the soln.
3. Note the reading and repeat it until two concordant readings are obtained.

(3) Sample titration :

1. Pipette out 10 ml sample water in a conical flask and add 2-3 drops of K_2CrO_4 indicator.
2. Titrate it against N/40 AgNO_3 soln. till the brick red color appears in the soln. 3. Note the reading and repeat it until two concordant readings are obtained.

Observations:

Table for blank correction:-

S.No	Vol. of distilled Water(ml) v_1	Burette reading		Vol of AgNO_3 soln.(ml) V
		Initial	Final	
1	10	17.6	18.6	0.8

Table for sample water:-

S.No	Vol. of sample Water(ml) v_1	Burette reading		Vol of AgNO_3 soln.(ml) V'
		Initial	Final	
1	10	0	17.6	17.6

Calculations:

Volume of water sample taken for titration=10ml

Volume of AgNO_3 (N/20) used $= (V' - V) = V_2 \text{ml}$

$$N_1 V_1 = N_2 V_2$$

$$N_1 * 10 = (V' - V) N/40 \rightarrow \frac{1}{40} (17.6 - 0.8) = \frac{16.8}{400} = 0.042N$$

$$\text{Strength} = N_1 * \text{eqv. wt} = 0.042 * 35.5 = 1.491 \text{ g/l} = 1491 \text{ ppm}$$



Strength of Cl^- ions in water sample = Normality * Equivalent weight of Cl^- gm /litre = Normality x Equivalent weight of CF x1000 ppm

Result:

Chloride content present in une given water sample is 1.491 gm/litre or 1491 ppm

Precautions;

- 1 The whole apparatus should be washed with distilled water before the start of the experiment.
- 2 The reaction mixture should be briskly shaken during the titration.
3. The end point of the reaction should be carefully observed.
4. The volume of the indicator should be same in all the titrations.
5. The pH of the sample solution should be adjusted to 7-8 ranges by adding acidic/basic solution.
6. Do not take mean of burette readings.