

CHEMISTRY PRACTICAL MANUAL
ON
DETERMINATION OF ALKALINITY
IN THE GIVEN WATER SAMPLE



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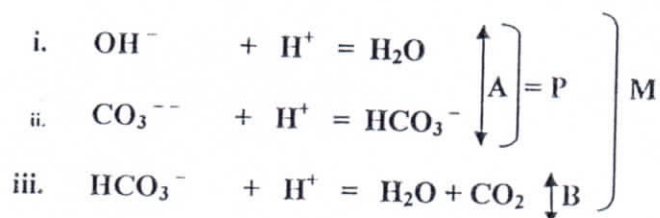
OBJECT: To determine the amount of alkalinity of supplied water sample.

APPARATUS USED: Burette, Pipette, Conical flask, Funnel, Beaker (250ml).

CHEMICALS USED: N/20 HCl, phenolphthalein & methyl orange.

THEORY: "The alkalinity of water may be defined as its capacity to neutralize acids." The alkalinity of water is mainly due to the presence of hydroxide ions (OH^-), carbonate ions (CO_3^{2-}) & bicarbonate ions (HCO_3^-) in water.

These can be estimated separately by the titration against standard acid using phenolphthalein & methyl orange as indicators. The various reaction involved are



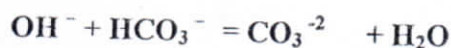
The titration of water samples against standard acid up-to phenolphthalein end point marks the completion of reactions (i) and (ii) only. The volume of acid (A ml.) used up-to this point corresponds to the complete neutralization of OH^- ions and one half of the normal CO_3^{2-} ions present. The alkalinity up-to phenolphthalein end point is known as phenolphthalein alkalinity (P).

The titration of water sample against the standard acid up-to methyl orange end point shows the completion of reactions (i),(ii) and (iii). Therefore the amount of acid (B ml.) used after phenolphthalein end point corresponds to the neutralization of one half of the normal carbonate and all the bicarbonates present. The total amount of acid [(A+B) ml.] in two titrations represent the total alkalinity(M).

The possible combinations of ions causing alkalinity in water are

- i. OH^- only
- ii. CO_3^{2-} only
- iii. HCO_3^- only
- iv. OH^- and CO_3^{2-} only
- v. CO_3^{2-} and HCO_3^- only

The possibility of OH^- and HCO_3^- together is not possible since they combine together to form CO_3^{2-} and H_2O .




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After knowing the values of P and M the nature of alkalinity can be determined by using the table A.

-: Table A:-

Results of titration to phenolphthalein end-point [P] and Methyl Orange end point [M]	OH^-	CO_3^{2-}	HCO_3^-
[P] = 0	Nil	Nil	[M]
[P] = [M]	[P] or [M]	Nil	Nil
[P] = 1/2 [M]	Nil	2[P] or [M]	Nil
[P] > 1/2 [M]	2[P] - [M]	2{[M] - [P]}	Nil
[P] < 1/2 [M]	Nil	2[P]	[M] - 2[P]

Alkalinity is generally expressed as parts per million (ppm) in terms of CaCO_3 .

PROCEDURE: Take 25ml of the sample solution in titration flask and add 2-3 drops of phenolphthalein indicator. Titrate this sample against HCl solution until the pink colour caused by phenolphthalein just disappears. Note the volume of acid used. This is phenolphthalein end point (P). Now add 2-3 drops of methyl orange indicator in the same solution. Continue the titration until yellow colour changes into orange. Note the volume of acid used. This is methyl orange end point (M).


OBSERVATIONS:

S. No.	Vol. of water sample (ml)	Vol. of acid used upto phenolphthalein end point(P) (ml)	Vol. of acid used upto methyl orange end point(M) (ml)
1.	25		
2.	25		
3.	25		

CALCULATIONS:

1. Phenolphthalein alkalinity (P) in terms of CaCO_3 equivalent

$$\begin{aligned}
 N_1 V_1 &= N_2 V_2 \\
 (\text{Acid}) & \quad \quad (\text{Water sample}) \\
 1/20 \times V_1 &= N_2 \times 25 \\
 N_2 &= 1/20 \times 1/25 \times V_1 \\
 &= V_1/500
 \end{aligned}$$


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$$\text{Strength in term of CaCO}_3 \text{ equivalent} = N_2 \times \text{Eq. weight of CaCO}_3$$

$$= [V_1/500] \times 50\text{gm/lit}$$

OR

$$[V_1/500] \times 50 \times 1000\text{mg/lit or ppm}$$

2. Methyl orange alkalinity(M) in terms of CaCO₃ equivalent

$$\begin{aligned} N_3 V_3 &= N_4 V_4 \\ (\text{Acid}) & \quad (\text{Water sample}) \\ 1/20 \times V_3 &= N_4 \times 25 \\ N_4 &= 1/20 \times 1/25 \times V_3 \\ &= V_3/500 \end{aligned}$$

$$\begin{aligned} \text{Strength in term of CaCO}_3 \text{ equivalent} &= N_4 \times \text{Eq. weight of CaCO}_3 \\ &= [V_3/500] \times 50\text{gm/lit} \end{aligned}$$

OR

$$[V_3/500] \times 50 \times 1000\text{mg/lit or ppm}$$

IMPORTANT NOTE:- To calculate the alkalinity in terms of individual ions, find out to which cases the values of P and M falls from table A.

1. If $P = 0$ Alkalinity is only due bicarbonate ions.
Bicarbonate alkalinity in terms of CaCO₃ eq. = M
2. If $P = 1/2 M$, Alkalinity is only due to carbonate ions.
Carbonate alkalinity in term of CaCO₃ eq. = 2P or M
3. $P = M$, Alkalinity is only due to hydroxide ions
Hydroxide alkalinity in terms of CaCO₃ eq. = P or M
4. If $P < 1/2 M$, Alkalinity is due to carbonate and bicarbonate ions.
 - a) Carbonate alkalinity in terms of CaCO₃ eq. = 2P
 - b) Bicarbonate alkalinity in terms of CaCO₃ eq. = [M] - 2[P]
5. If $P > 1/2 M$, Alkalinity is due to hydroxide and carbonate ions.
 - a) Hydroxide alkalinity in terms of CaCO₃ eq. = (2P - M)
 - b) Carbonate alkalinity in terms of CaCO₃ eq. = 2 (M - P)



RESULT: The given water sample has:

$$\text{Alkalinity due to OH}^- = \dots$$

$$\text{Alkalinity due to CO}_3^{--} = \dots$$

$$\text{Alkalinity due to HCO}_3^- = \dots$$

$$\begin{aligned} \text{Total alkalinity} &= \text{Alkalinity due to (CO}_3^{--} + \text{HCO}_3^- + \text{OH}^-) \\ &= \dots \text{ ppm} \end{aligned}$$


 
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PRECAUTIONS:


1. The glass apparatus should be cleaned and rinsed properly before the start of experiment.
2. HCl level in burette must be read carefully.
3. Since phenolphthalein and methyl orange are used simultaneously end point should be noted.
4. Volume of HCl should be same in all titrations.


VIVA VOCE:

1. What is alkalinity?
2. Why alkalinity is not due to the simultaneously presence of OH^- & HCO_3^- .
3. Write the pH range of phenolphthalein and methyl orange indicators.


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CHEMISTRY PRACTICAL MANUAL
ON
DETERMINATION OF TEMPORARY AND
PERMANENT HARDNESS IN WATER
SAMPLE USING EDTA AS STANDARD
SOLUTION


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OBJECT: To determine temporary and permanent hardness of water by using complexometric titration.

APPARATUS USED: Burette, Conical flask, funnel, pipette, beaker etc.

CHEMICALS USED: EDTA solution (0.01 M), Buffer solution ($\text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$), Erichrome Black-T (EBT), water sample.

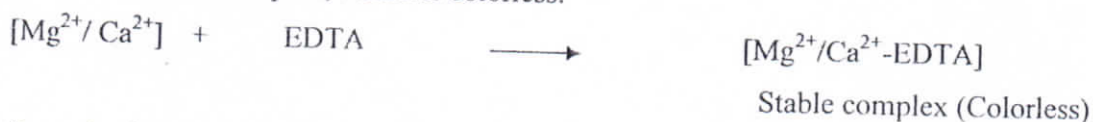
THEORY: The hardness of water can be determined by complexometric titration. EDTA is used as complexing reagent. The Ca^{2+} and Mg^{2+} present in water are titrated with EDTA using Erichrome Black-T as indicator.

Estimation of hardness by EDTA method is based on the following principle.

First, the indicator Erichrome Black - T which is blue colored dye, form an unstable complex with calcium or magnesium ions in hard water at pH of 9 to 10. The complex is wine red in color.



As this solution is titrated against EDTA, the free Ca^{2+} and Mg^{2+} ions in water form stable metal-ion EDTA complex, which is colorless.



Once the free metal ions are complexed, the EDTA replaces Ca^{2+} and Mg^{2+} ions from the unstable indicator complex also. to form a stable complex with the result, the indicator is set free. Since the free indicator is blue in colour at the above mentioned pH. the endpoint is the appearance of blue color.



Thus the amount of EDTA used corresponds to the hardness of water. The temporary hardness of water is removed by boiling and after the removal of precipitate by filtration; the permanent hardness in the filtrate is determined by titration with EDTA as above.

Temporary hardness will be given by the difference of total hardness and permanent hardness.

PROCEDURE: Pipette out 25 ml of hard water sample into 250ml conical flask. Add 2ml of the buffer solution and 3 drops of Erichrome black -T indicator. Titrate against EDTA solution till the colour of the solution changes from wine red to clear blue. This is the end point .Note the



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final reading which corresponds to total hardness. Repeat the titration till at-least two concordant readings are obtained.

Measure out 250ml of the hard water sample in 500ml beaker, boil gently for half an hour, pour the solution into 250 measuring flask. Make the solution up to the mark by adding distilled water and shake thoroughly. Pipette out 25ml of this solution into a 250ml conical flask, add 2ml of the buffer solution and 3 drops of Erichrome black -T indicator. Titrate with EDTA solution until the wine-red color change to clear blue at the end point. Note the final reading which corresponds to the permanent hardness. Repeat the titration till at least two concordant readings are obtained.

OBSERVATIONS:

Total Hardness (Before Boiling)

S.N.	Vol. of water sample taken (ml)	Burette Reading			Actual Vol. of EDTA Used
		Initial	Final	Difference (between initial and final reading)	
1.	25ml				a ml.
2.	25ml				
3.	25ml				

Permanent Hardness (After Boiling)

S.N.	Vol. of water sample taken (ml)	Burette Reading			Actual Vol. of EDTA Used
		Initial	Final	Difference (between initial and final reading)	
1.	25ml				b ml.
2.	25ml				
3.	25ml				

CALCULATIONS:


EDTA forms 1:1 complex with Ca^{+2} and Mg^{+2}

\therefore 1000ml of 1M EDTA = 1 Mole of CaCO_3 = 100 gm. of CaCO_3

\therefore 1ml. of 0.01M EDTA = $100 \times 1/1000 \times 1/100$ gm. of CaCO_3
= 0.001 gm. of CaCO_3 or 1mg. of CaCO_3

Total hardness

a ml of 0.01m EDTA = $1 \times a$
= a mg of CaCO_3


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This hardness is present in 25ml of sample water

So hardness present in 1000ml of sample water = $1000 \times a/25 = 40 a$ gm of CaCO_3

Total hardness = $40 a$ mg/lit. or ppm.

Permanent hardness

b ml of 0.01M EDTA = $1 \times b$

= b mg of CaCO_3

This hardness present in 25ml of sample water

So hardness present in 1000ml of sample water = $1000 \times b/25 = 40 b$ gm. of CaCO_3

Permanent hardness = $40 b$ mg/lit. or ppm.

Temporary hardness

Temporary hardness = Total hardness – Permanent hardness

= $(40 a) - (40 b)$ mg/lit/ or ppm.

RESULT:

Total hardness is = _____ ppm

Permanent hardness is = _____ ppm


Temporary hardness = _____ ppm

PRECAUTIONS:

1. The glass apparatus should be cleaned before starting the experiment.
2. The pH should be maintained during titration.
3. The end point should be noted carefully.
4. Hard water sample should be boiled properly.

VIVA VOCE:

1. How will you define hardness?
2. What is the cause of temporary hardness in water?
3. How will you remove the temporary hardness?
4. What do you understand by permanent hardness of water?
5. What is the effect on the end -point with Eriochrome black -T indicator, if the hard water sample does not contain $\text{Mg}^{2+}/\text{Ca}^{2+}$?


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**DEPARTMENT OF APPLIED SCIENCES &
HUMANITIES**

ENGINEERING CHEMISTRY LAB

ON

**IDENTIFICATION OF FUNCTIONAL GROUPS
IN ORGANIC COMPOUNDS**


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OBJECT : To detect the elements and functional groups in the given organic compound.

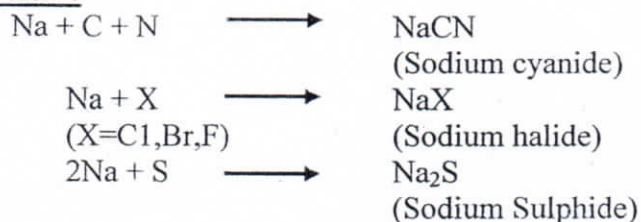
DETECTION OF ELEMENTS : Generally , the organic compounds have C, H and oxygen. In addition to these three elements, halogen , nitrogen, and sulphur may also be present. Firstly, these elements are converted into ionisable inorganic salts by fusion of the organic compound with metallic sodium. Detection of nitrogen, sulphur, and halogens by this method is known as Lassaigne's test.

APPARATUS USED : Test tube, ignition tube , tong, and porcelain dish, beaker, funnel etc.

CHEMICAL USED : Sodium metal, ferrous sulphate solution, dil. H_2SO_4 , $FeCl_3$ solution, sodium nitro nitroprusside solution, dil . acetic acid, lead acetate solution, cone, HNO_3 , $AgNO_3$ solution, dil. HNO_3 , chloroform , carbon tetrachloride, chlorine water, dil. HCl , ethyl alcohol, acetone, cobalt nitrate solution.


LASSAIGNE'S TEST : Take a piece of dry sodium metal into a clean ignition tube and cover it with a piece of organic compound. Heat gently in the flame initially and then strongly untill the bottom of the tube becomes red hot. Plunge the hot tube into a porcelain dish containing 15 ml of cold distilled water. Grind the contents with a pestle, boil the mixture for five minutes and filter. The filtrate is known as sodium extract, which is then used for elemental detection.


REACTIONS INVOLVED :



if N and S are both present together




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Reactions Involved :

Nitrogen



Both N and S



Halogens

Chlorine :



Bromine :




Iodine :



Iodine and Bromine :




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Apparatus Used : Beaker, test tube, boiling tube, funnel, burner, tripod stand, wire gauge etc.

Table: 1



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Table: 1

S.No	Experiment	Observation	Inference
	Phenolic Group		
(i)	Neutral FeCl_3 Test: Take 1 ml of neutral ferric chloride solution in cleaned and dry test tube and add 3-4 drops of a little bit of the organic compound, shake well.	Violet/red/blue/green color	Phenolic group present
(ii)	Litmus Test : put a pinch of the Organic compound on a moist blue litmus paper.	Turns red	Phenolic group present
(iii)	Phthalein Test: Take 0.5 g of organic compound in a cleaned and dry test tube, add 1 g of phthalic anhydride and 2 drops of conc. H_2SO_4 , heat gently for 1 minute. Cool and pour the mixture into a beaker containing 40 ml of water and make it alkaline by adding NaOH.	Red/pink Blue Resorcinol α -naphthol β -naphthol	Phenol, o-cresol Catechol, m-cresol Fluorescent Green Green Faint green
(iv)	Libermann's Nitroso Test: Heat a small amount of the organic compound and few crystals of NaNO_2 , cool and add 1 ml of conc. H_2SO_4 and excess of water and excess of NaOH solution.	A deep green to blue solution is formed which turns red. Deep green to blue color appears.	Phenolic group present
Reactions Involved (i) Litmus Test: Blue \longrightarrow Red (ii) Neutral FeCl_3 test: $6\text{C}_6\text{H}_5\text{OH} + \text{FeCl}_3 \longrightarrow [(\text{C}_6\text{H}_5\text{O})_6\text{Fe}]^{-3} + 3\text{H}^+ + 3\text{HCl}$			

Table: 2


S.No	Experiment	Observation	Inference
	Alcoholic Group (-OH)		
(i)	Ceric Ammonium nitrate test: Take 1 ml of a organic compound in a cleaned and dry test tube, add 3-4 drops of ceric ammonium nitrate solution and shake well.	Red color	Alcoholic group present



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(ii)	Ester Test : Heat 1 ml of organic compound, 1 ml of glacial acetic acid and 3-4 drops of conc. H_2SO_4 in a dry test tube for 10 minutes. Now pour the reactions content into a beaker containing 20 ml of cold water.	A fruity smell	Alcoholic group present
Reactions Involved (i) Na Metla Test : $2ROH + 2Na \longrightarrow 2RONa + H_2$ Brisk effervescence (ii) Ester Test: $ROH + CH_3COOH \xrightarrow{\text{conc. } H_2SO_4} RCOOCH_3 + H_2O$ glacial acetic acid (Ester, fruity smell)			

Table: 3

S.No	Experiment	Observation	Inference
	Aldehyde group (-CHO)		
(i)	Schiff's solution test: Take 0.5 g of solid compound or 4 drops of liquid compound in a test tube, add 1 ml of Schiff's reagent, shake vigorously.	Red or violet color	-CHO group present
(ii)	Tollen's reagent Test : Take a little quantity of the organic compound in a test tube, add 2 ml of tollen's reagent, shake, warm and allow the content to stand for 3-4 minutes.	Silver mirror or grey ppt.	-CHO group present
(iii)	Fehling's Solution Test: Take 1 ml of a mixture of equal amounts of Fehling solution A and B, add 4 drops or 0.2 g of organic compound, boil for about 5 minutes.	Red ppt.	-CHO group present


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Reactions Involved

(i) Tollen's reagent Test:

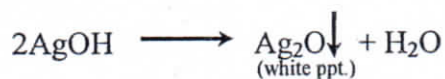



Table: 5

S.No	Experiment	Observation	Inference
	Ketonic group		
(i)	Schiff's solution test: Take 0.5 g of solid compound or 4 drops of liquid compound in a test tube, add 1 ml of Schiff's reagent, shake vigorously.	No color	Ketonic group present
(ii)	2,4 dinitrophenyl hydrazine Test : Take a little quantity of the organic compound in a test tube, add 2 ml of 2,4 dinitrophenyl hydrazine reagent, shake, heat and cool.	A red, yellow or orange coloured ppt.	Ketonic group present

Table: 6

S.No	Experiment	Observation	Inference
	Nitro group (-NO₂)		
(i)	NaOH solution test: Take 0.5 g of solid compound or 4 drops of liquid compound in a test tube, add 1 ml of aq. Solution of NaOH and warm.	Yellow color	Nitro group present


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(ii)	Mulliken's Test : Take a little quantity of the organic compound in a dry test tube, add 2 ml of ethanol, 1 ml CaCl_2 or NH_4Cl solution and a pinch of Zn dust, boil the reaction content for 5 minutes, cool and filter into a test tube containing 2 ml Tollen's reagent.	Grey or black ppt	Nitro group present
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Table: 7

S.No	Experiment	Observation	Inference
	Primary Amines ($-\text{NH}_2$)		
(i)	Litmus Test : put a pinch of the Organic compound on a moist red litmus paper.	Turns blue	Amines group present
(ii)	Nitrous Acid Test : Dissolve a small quantity of the organic compound in dil HCl and cool. Add 10% aq. NaNO_2 solution.	Brisk effervescence	Aliphatic primary amines group present
(iii)	Diazotisation/Dye Test : Dissolve about .2 g of the organic compound in dil HCl and cool. Add 10% aqueous NaNO_2 solution. Pour the reaction content in a beaker containing alkaline β -naphthol solution.	Red or orange dye	Aromatic primary amines group present

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