

7.0 EXPERIMENT ON DETERMINATION OF ALKALINITY OF WATER

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7.0 EXPERIMENT ON DETERMINATION OF ALKALINITY OF WATER

PREAMBLE:

“How to determine alkalinity in Water and Wastewater”.

Test procedure is in accordance to IS: 3025 (Part 23) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

- (1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 2320.
- (2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 310.1.

7.1 AIM

To determine the alkalinity of given water sample with the stipulations as per **IS: 3025 (Part 23) - Reaffirmed 2003.**

7.2 INTRODUCTION

Alkalinity is primarily a way of measuring the acid neutralizing capacity of water.

In other words, its ability to maintain a relatively constant pH.

The possibility to maintain constant pH is due to the hydroxyl, carbonate and bicarbonate ions present in water.

The ability of natural water to act as a buffer is controlled in part by the amount of calcium and carbonate ions in solution.

Carbonate ion and calcium ion both come from calcium carbonate or limestone.

So water that comes in contact with limestone will contain high levels of both Ca^{++} and CO_3^{2-} ions and have elevated hardness and alkalinity.

7.2.1 ENVIRONMENTAL SIGNIFICANCE

Alkalinity is important for fish and aquatic life because it protects or buffers against rapid pH changes. Higher alkalinity levels in surface waters will buffer acid rain and other acid wastes and prevent pH changes that are harmful to aquatic life.

Large amount of alkalinity imparts bitter taste in water.

The principal objection of alkaline water is the reactions that can occur between alkalinity and certain cations in waters. The resultant precipitate can corrode pipes and other accessories of water distribution systems.

Wastewaters containing excess caustic (hydroxide) alkalinity are not to be discharged into natural water bodies or sewers.

Alkalinity as carbonate and bicarbonate of saline water is very important in tertiary recovery processes for recovering petroleum. Alkaline water offers better wetting to the formation rock and improve oil release. As an additional benefit, ions that provide alkalinity absorb on rock surfaces occupying adsorption sites and decrease the loss of recovery chemical by adsorption.

The alkalinity value is necessary in the calculation of carbonate scaling tendencies of saline waters.

The alkalinity acts as a pH buffer in coagulation and lime-soda softening of water.

In wastewater treatment, alkalinity is an important parameter in determining the amenability of wastes to the treatment process and control of processes such as anaerobic digestion, where bicarbonate alkalinity, total alkalinity, and any fraction contributed by volatile acid salts become considerations.

7.3 PRINCIPLE

The alkalinity of water can be determined by titrating the water sample with Sulphuric acid of known values of pH, volume and concentrations. Based on stoichiometry of the reaction and number of moles of Sulphuric acid needed to reach the end point, the concentration of alkalinity in water is calculated.

When a water sample that has a pH of greater than 4.5 is titrated with acid to a pH 4.5 end point, all OH^- , CO_3^{2-} , and HCO_3^- will be neutralized.

For the pH more than 8.3, add phenolphthalein indicator, the colour changes to pink colour. This pink colour is due to presence of hydroxyl ions.

If sulphuric acid is added to it, the pink colour disappears i.e. OH^- ions are neutralized.

Then add mixed indicator, the presence of CO_3^{2-} and HCO_3^- ions in the solution changes the colour to blue. While adding sulphuric acid, the color changes to red, this color change indicates that all the CO_3^{2-} and HCO_3^- ions has been neutralized. This is the end point.

7.4 MATERIALS REQUIRED

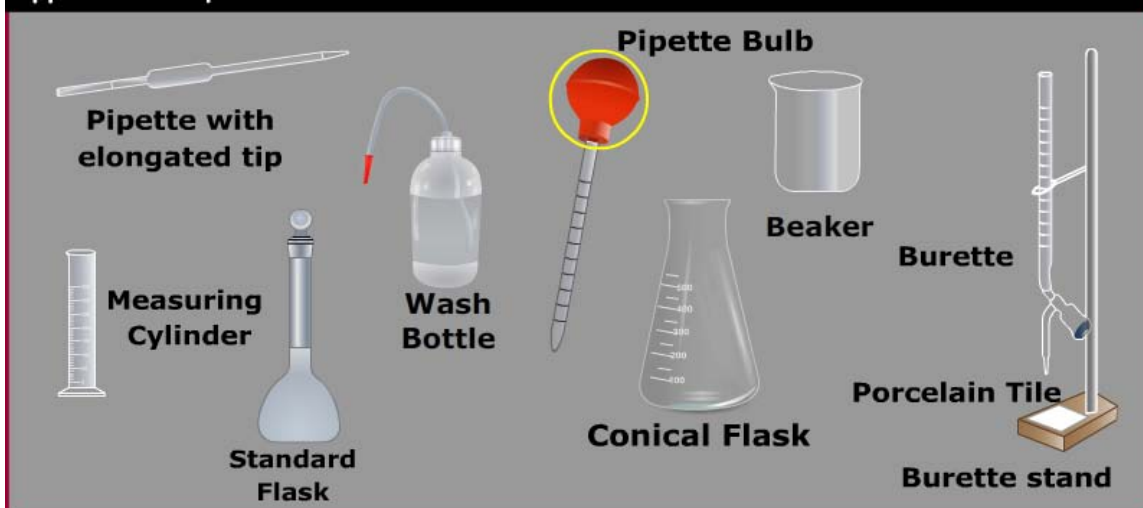
7.4.1 APPARATUS REQUIRED

1. Burette with Burette stand and porcelain tile
2. Pipettes with elongated tips
3. Pipette bulb
4. Conical flask (Erlenmeyer Flask)
5. 250 mL Measuring cylinders
6. Standard flask
7. Wash Bottle
8. Beakers

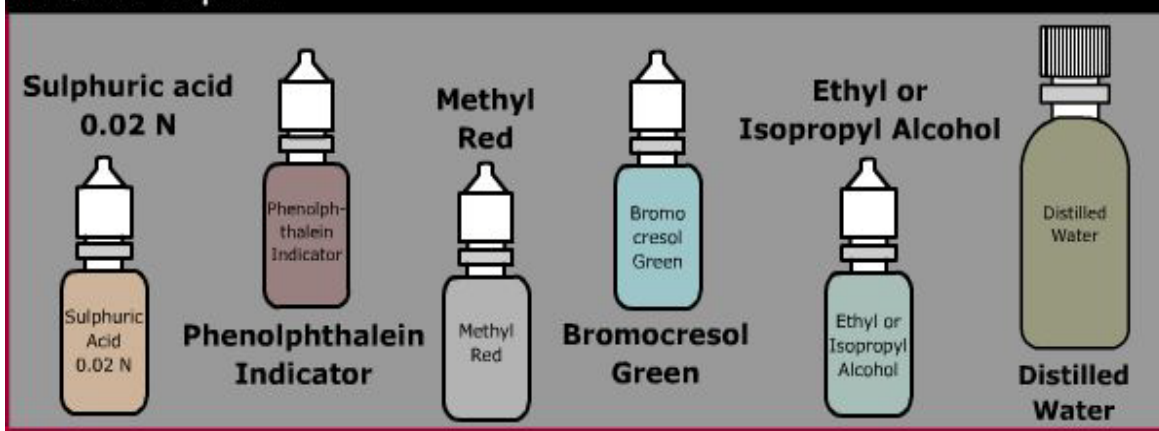
7.4.2 CHEMICALS REQUIRED

1. Standard sulphuric acid
2. Phenolphthalein
3. Mixed Indicator
4. Bromocresol Green
5. Methyl Red
6. Ethyl alcohol
7. Distilled Water

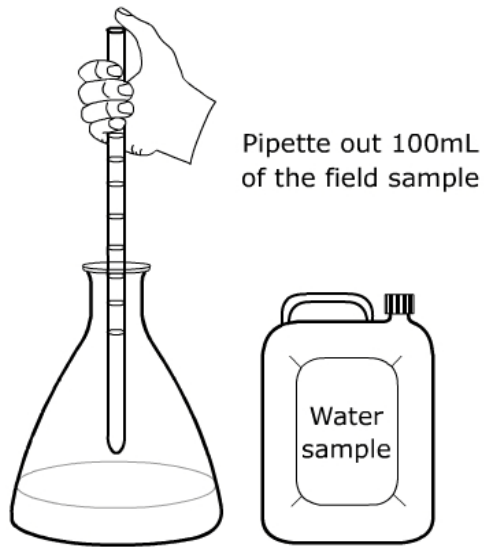
Apparatus Required



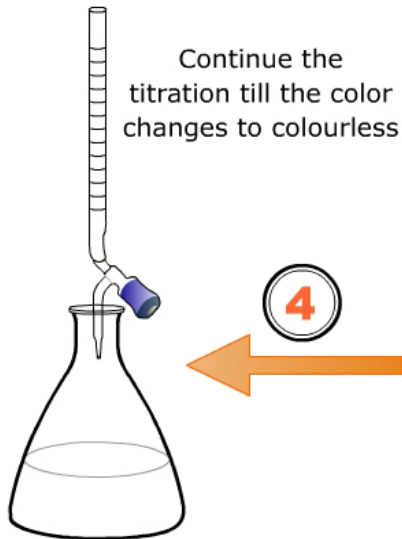
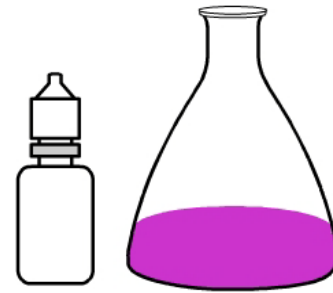
Chemicals Required



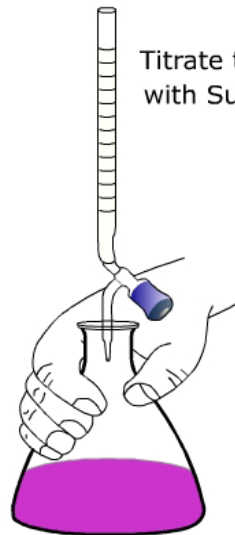
PROCEDURE CHART



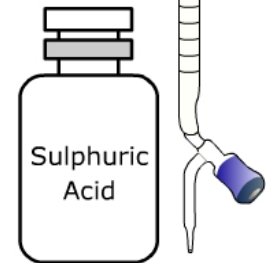
Add 2 drops of Phenolphthalein indicator



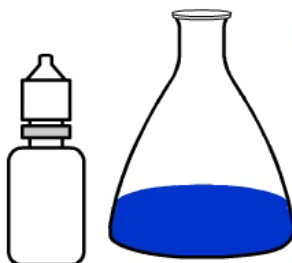
Titrate the contents with Sulphuric Acid



Fill the burette with Sulphuric Acid



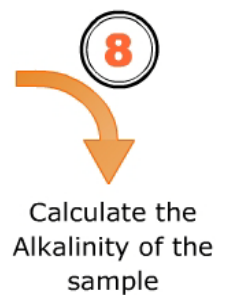
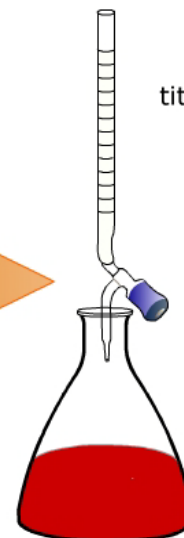
Add 2 drops of mixed indicator



Continue the titration with sulphuric acid



Continue the titration till the color changes to red



7.5 SAMPLE HANDLING AND PRESERVATION

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.

To reduce the change in samples, keep all samples at 4°C. Do not allow samples to freeze.

Analysis should begin as soon as possible.

Do not open sample bottle before analysis.

7.5.1 PRECAUTIONS

The following precautions should be observed while performing the experiment:

1. Do not keep the indicator solution open since it contains the alcohol which tends to evaporate.
2. The mixed indicator solution is containing dye in it; care should be taken so that it is not spilled to your skin.
3. If it spills on your skin, the scar will remain at least for two to three days.

7.6 PROCEDURE

7.6.1 PREPARATION OF REAGENTS

For testing the given sample, first the reagents are required to be prepared.

Sulphuric Acid Solution (0.02N):

- Take approximately 500 mL of distilled water in a 1000 mL standard flask.
- Pipette 20 mL of concentrated 0.1 Normality Sulphuric acid and add slowly along the sides of the standard flask.
- Then make up the volume up to 1000 mL mark. Now the strength of this solution is 0.02 N.

Phenolphthalein Indicator Preparation:

- Weigh 1g of phenolphthalein and add to 100 mL of 95% ethyl alcohol or to 100 mL of distilled water. Use the readymade Phenolphthalein indicator available in the market.

Mixed Indicator Preparation:

- Dissolve 100 mg Bromocresol green and 20 mg of methyl red in 100 mL of 95% ethyl alcohol or use 100 mL of distilled water. Mixed indicator also readily available in the market. So it can be used as indicator in this experiment.

7.6.2 TESTING OF WATER SAMPLE

- Rinse the burette with 0.02N Sulphuric acid and discard the solution.
- Fill the burette with 0.02N sulphuric acid and adjust it to zero.
- Fix the burette in the stand.
- Using a measuring cylinder exactly measure 100 mL of sample and pour it into a 250 mL of conical flask.
- Add few drops of phenolphthalein indicator to the contents of conical flask. The colour of the solution will turn to pink. This colour change is due to alkalinity of hydroxyl ions in the water sample.
- Titrate it against 0.02N sulphuric acid till the pink color disappears. This indicates that all the hydroxyl ions are removed from the water sample. Note down the titter value (V1). The value of titration is 0.5mL .This value is used in calculating the phenolphthalein alkalinity.
- To the same solution in the conical flask add few drops of mixed indicator. The colour of the solution turns to blue. This colour change is due to CO_3^{2-} & HCO_3^- ions in water sample.
- Continue the titration from the point where stopped for the phenolphthalein alkalinity. Titrate till the solution becomes red. The entire volume (V2) of sulphuric acid is noted down and it is accountable in calculating the total alkalinity.
- The value of titration is 8.3mL.
- Repeat the titration for concordant values.

7.7 CALCULATION

7.7.1 TABLE

Table -1 Phenolphthalein Alkalinity:

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)
		Initial	Final	
1.				
2.				
3.				

Burette solution: Sulphuric Acid Solution

Pipette solution: Sample.

Indicator: Phenolphthalein Indicator.

End point: Disappearance of pink color.

- For the calculation of Phenolphthalein Alkalinity
- The Sulphuric acid is taken in the Burette.
- For the first titration, the volume of water sample taken is 100 mL. The initial reading is 0. The final reading is 0.6.
- The volume of sulphuric acid consumed to get the end point is 0.6 mL.
- For the second titration, the volume of water sample taken is 100 mL. The initial reading is 0. The final reading is 0.5.
- The volume of sulphuric acid consumed to get the end point is 0.5 mL.
- For the third titration, the volume of water sample taken is 100 mL. The initial reading is 0. The final reading is 0.5.
- The volume of Sulphuric Acid consumed to get the end point is 0.5 mL.
- For the second and third titration, the burette reading is same so we have achieved concordant value. We can go for the calculations
- Phenolphthalein Alkalinity of the given water sample is equal to volume of H_2SO_4 (V_1) * normality * 50 * 1000 divided by volume of sample taken
- Here the volume of H_2SO_4 (V_1) is 0.5mL
- Normality is 0.02Molar
- And volume of sample taken is 100mL
- Substituting the values in the formula and calculating we get the value 5 mg/L
- So the Phenolphthalein Alkalinity mg/L is 5 mg/L

Table - 2 Total Alkalinity:

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)
		Initial	Final	
1.				
2.				
3.				

Burette solution: Sulphuric Acid Solution

Pipette solution: Sample.

Indicator: Mixed Indicator.

End point: Appearance of Red color.

- For Calculation of Total Alkalinity the volume of water sample taken is 100 mL.
- The Sulphuric acid is taken in the Burette.
- For the first titration, the volume of water sample taken is 100 mL. The initial reading is 0. The final reading is 8.5.
- The volume of sulphuric acid consumed to get the end point is 8.5 mL.
- For the second titration, the volume of water sample taken is 100 mL. The initial reading is 0. The final reading is 8.3
- The volume of sulphuric acid consumed to get the end point is 8.3 mL.
- For the third titration, the volume of water sample taken is 100 mL. The initial reading is 0. The final reading is 8.3
- The volume of H_2SO_4 (V_2) consumed to get the end point is 8.3 mL.
- For the second and third titration, the burette reading is same so we have achieved concordant value. We can go for the calculations
- Total Alkalinity of the given water sample is equal to volume of H_2SO_4 (V_2) * normality * 50 * 1000 divided by volume of sample taken
- Here the volume of H_2SO_4 (V_2) is 8.3 mL.

Normality is 0.02Molar and volume of sample taken is 100mL.

Substituting the values in the formula and calculating we get the value 83 mg/L.

So the Total Alkalinity is 83 mg/L.

7.7.2 DATA SHEET

DETERMINATION OF ALKALINITY DATA SHEET

Date Tested : August 30, 2010
Tested By : CEM Class, Group A
Project Name : CEM, NITTTR Lab
Sample Number : BH1
Sample Location : Perungudi (Lat 12' 57" 31.74 S Long 80'14" 8.82)
Sample Description : Surface water

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)
		Initial	Final	
4.	100	0	0.6	0.6
5.	100	0	0.5	0.5
6.	100	0	0.5	0.5

Specimen Calculation:

Volume of Sulphuric Acid = 0.5 mL
Normality of Sulphuric Acid = 0.02 N
Volume of Sample = 50 mL
Equivalent weight of CaCO_3 = 1000

$$\text{Phenolphthalein Alkalinity} = \frac{\text{volume of } \text{H}_2\text{SO}_4 (\text{V}_1) * \text{Normality} * 50 * 1000}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L to convert the sample size from mL to L

$$\begin{aligned}\text{Alkalinity as } \text{CaCO}_3 \text{ equivalent (mg/L)} &= 0.5 \times 0.02 \times 50 \times 1000 / 100 \\ &= 5 \text{ mg/L as } \text{CaCO}_3 \text{ equivalent}\end{aligned}$$

Total Alkalinity:

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)
		Initial	Final	
1	100	0	8.5	8.5
2	100	0	8.3	8.3
3	100	0	8.3	8.3

Specimen Calculation:

Volume of Sulphuric Acid = 8.3 mL

Normality of Sulphuric Acid = 0.02 N

Volume of Sample = 50 mL

Equivalent weight of CaCO_3 = 1000

$$\text{Total Alkalinity} = \frac{(\text{volume of } \text{H}_2\text{SO}_4 (V_1) * \text{Normality} * 50 * 1000)}{\text{volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L to convert the sample size from mL to L

$$\text{Alkalinity as } \text{CaCO}_3 \text{ equivalent (mg/L)} = 8.3 \times 0.02 \times 50 \times 1000/100$$

$$= 83 \text{ mg/L as } \text{CaCO}_3 \text{ equivalent}$$

7.8 INTERPRETATION OF RESULTS

Value of P and T	Alkalinity due to		
	OH^-	CO_3^{2-}	HCO_3^-
$P=0$	0	0	$T = 83$
$P < \frac{1}{2} T$	0	$2P = 10$	$T - 2P = 73$
$P = \frac{1}{2} T$	0	$2P = 10$	0
$P > \frac{1}{2} T$	$2P - T = -73$	$2P - T = -73$	0
$P = T$	$T = 83$	0	0

- To find the different values of Alkalinity due to Hydroxyl, Carbonate and Bicarbonate ions take P as Phenolphthalein Alkalinity and T as Total Alkalinity.
- If $P=0$, The Alkalinity due to Hydroxyl and carbonate ions is 0. Alkalinity due to Bicarbonate ion is equal to the Total Alkalinity i.e. $T = 83 \text{ mg/L}$.
- If $P < \frac{1}{2} T$, then the Alkalinity due to Hydroxyl ion is 0. The Alkalinity due to carbonate ion is $2P$. i.e. $2P = 10 \text{ mg/L}$. Alkalinity due to Bicarbonate ion is equal to the Total Alkalinity minus 2 times Phenolphthalein Alkalinity i.e. $T - 2P = 73 \text{ mg/L}$.
- If $P = \frac{1}{2} T$, then the Alkalinity due to Hydroxyl ion is 0. The Alkalinity due to carbonate ion is $2P$. i.e. $2P = 10 \text{ mg/L}$. Alkalinity due to Bicarbonate ion is equal to 0
- If $P > \frac{1}{2} T$, then the Alkalinity due to Hydroxyl and carbonate ions is $2P - T$. i.e. $2P - T = -73 \text{ mg/L}$. Alkalinity due to Bicarbonate ion is 0
- If $P = T$, The Alkalinity due to Hydroxyl is equal to the Total Alkalinity i.e. $T = 83 \text{ mg/L}$. Alkalinity due to carbonate and Bicarbonate ions is 0.
- If $P > \frac{1}{2} T$, then the Alkalinity due to Hydroxyl and carbonate ions is $2P - T$. i.e. $2P - T = -73 \text{ mg/L}$. Alkalinity due to Bicarbonate ion is 0. If $P = T$, The Alkalinity due to Hydroxyl is equal to the Total Alkalinity. i.e. $T = 83 \text{ mg/L}$. Alkalinity due to carbonate and Bicarbonate ions is 0.

7.9 INFERENCE

Alkalinity is a measure of the capacity of water to neutralize acids. The predominant chemical system present in natural waters is one where carbonates, bicarbonates and hydroxides are present. The bicarbonate ion is usually prevalent. However, the ratio of these ions is a function of pH, mineral composition, temperature and ionic strength. Water may have a low alkalinity rating but a relatively high pH or vice versa, so alkalinity alone is not of major importance as a measure of water quality. Alkalinity is not considered detrimental to humans but is generally associated with high pH values, hardness and excess dissolved solids. High alkalinity waters may also have a distinctly flat, unpleasant taste. Based on the testing, it is found that the alkalinity of the sample is 83 mg/L. As per the provisional code, alkalinity should not exceed 200 mg/L for potable water. For the fresh water alkalinity ranges between 20 – 100 mg/L. Alkalinity of tested sample is within the limits specified in the standards. Hence the water sample is fit for drinking.

7.10 EVALUATION

1. Alkalinity of water is an indication of
 - a) Base neutralizing capacity
 - b) Acid neutralizing capacity
 - c) Quantity of base present
 - d) Quality of base present

2. Mixed indicator is a combination of
 - a) Bromcresol Blue and Methyl Orange
 - b) Bromcresol Green and Methyl Red
 - c) Bromcresol Blue and Methyl Red
 - d) Bromcresol Green and Methyl Orange

3. Alkalinity is present due to all except _____.
 - a) Bromates
 - b) Phosphates
 - c) Silicates
 - d) Chlorides

4. Alkalinity is not caused by
 - a) Carbonates ions
 - b) Bicarbonates ions
 - c) Hydroxyl ions
 - d) Chloride ions

5. The phenolphthalein alkalinity is present then the pH of that water will be More than

- a) 8.3
- b) 9.3
- c) 7.3
- d) 6.3

6. Alkalinity of natural water is mainly due to the presence of _____.

- a) Bicarbonates
- b) Bromates
- c) Phosphates
- d) Silicates

7. The bicarbonate equivalence point normally occur at pH

- a) 2.5
- b) 3.5
- c) 4.5
- d) 5.5

8. What is ppm?

- a) Parts per meter square
- b) Parts per meter
- c) Parts per million
- d) Parts per millimeter

9. The normality of the acid used in the titration is ____.

- a) 0.2 N
- b) 0.02 N
- c) 0.002 N
- d) 2.0 N

10. A standard solution is a

- a) Solution of accurately known strength
- b) Solution of accurately known pH
- c) Coloured solution
- d) Colourless solution

KEY TO ITEMS:

1) b

2) b

3) d

4) d

5) a

6) a

7) c

8) c

9) b

10) a