

6.0 EXPERIMENT ON DETERMINATION OF CALCIUM HARDNESS

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6.0 EXPERIMENT ON DETERMINATION OF CALCIUM HARDNESS

PREAMBLE:

“How to determine calcium hardness in Water and Wastewater”.

Test procedure is in accordance to IS: 3025 (Part 40) - 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 3500 – Ca-D.
- (2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 215.2.

6.1 AIM

To determine the calcium hardness in the given water sample with the stipulations as per **IS: 3025 (Part 40) - Reaffirmed 2003**.

6.2 INTRODUCTION

Water hardness is an expression for the sum of the calcium and magnesium cations concentration in a water sample.

Calcium is usually found in highest concentrations in natural water.

The presence of calcium in water results from deposits of lime stone, gypsum etc.

Calcium is one of the principal cations involved in water hardness.

Calcium hardness is the estimation of hardness due to calcium in water.

These cations form insoluble salts with soap and decrease the cleaning effectiveness of soap.

They also form hard water deposits in hot water heaters.

The calcium content may range from zero to several hundred ppm.

6.2.1 ENVIRONMENTAL SIGNIFICANCE

The relative amounts of Calcium hardness, Carbonate and non-Carbonate hardness present in water are the factors while determining the most economical type of softening process.

Determination of hardness serves as a basis for routine control of softening processes.

Hard water typically contains high concentrations of Ca and Mg cations, which interfere with the use of the water for many applications.

These ions diminish the effectiveness of soaps and detergents for cleansing operations.

They diminish the drinking quality of water and they contribute to the accumulation of insoluble salt deposits in storage vessels or plumbing.

6.3 PRINCIPLE

The quantity of calcium in water will be determined by titrating the water sample with a standard Ethylene Diamine Tetra Acetic acid (EDTA) of known volume and concentration. Based on the stoichiometry of the reactions and the number of moles of EDTA required to reach the end point, the concentration of calcium content in water is calculated.

An indicator, ammonium purpurate which combines only with calcium is used.

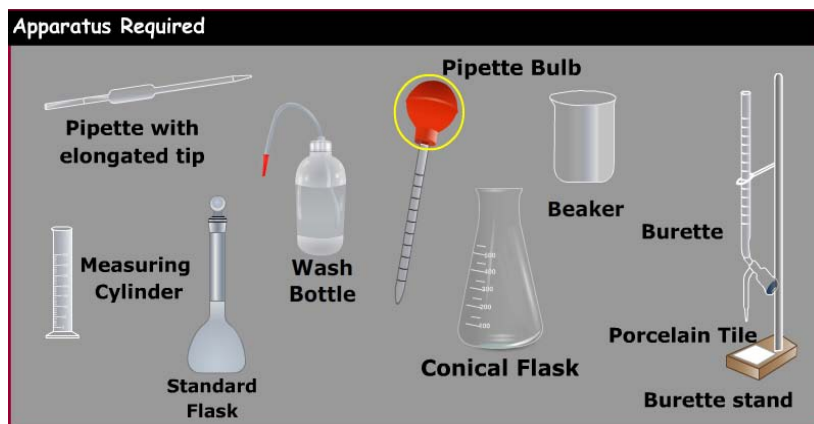
The indicator imparts a pink color to the solution while there are calcium and magnesium ions that have not complexed with EDTA.

Once the endpoint has been reached and there is no more uncomplexed Ca or Mg, the solution will turn to purple color. No hint of pink color will be left.

6.4 MATERIALS REQUIRED

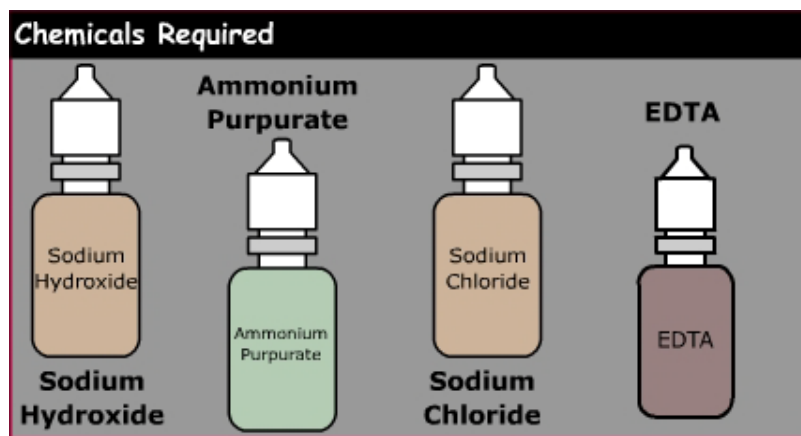
6.4.1 APPARATUS REQUIRED

1. Burette with Stand & Porcelain tile
2. Pipettes with elongated tips
3. Conical flask
4. 250 mL Graduated Cylinder
5. Standard Flask
6. Beakers
7. Wash bottle

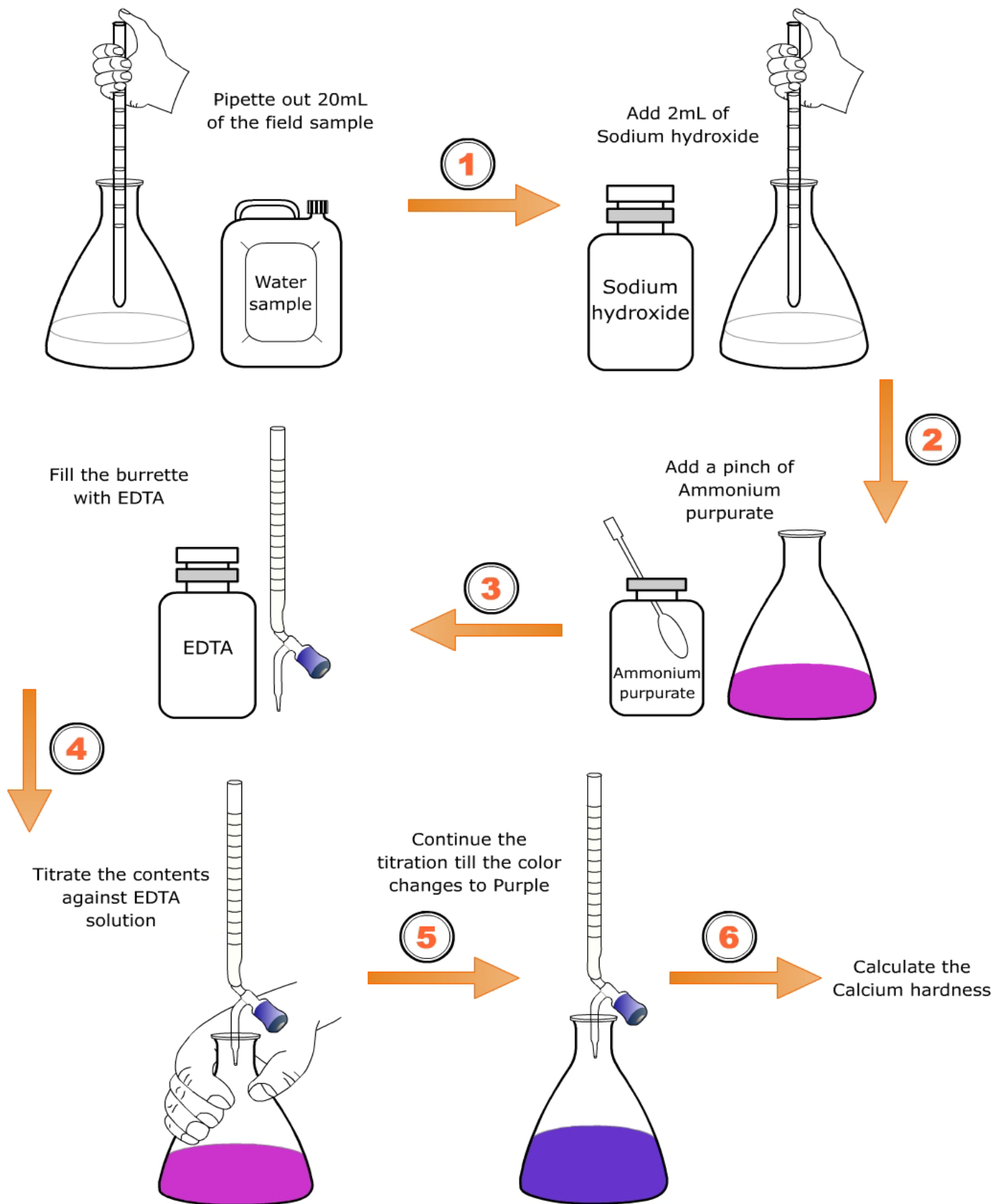


6.4.2 CHEMICALS REQUIRED

1. Ammonium Purpurate
2. Sodium Chloride
3. Sodium Hydroxide
4. EDTA



PROCEDURE CHART



6.5 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.

If Analysis is to be carried out within two hours of collection, cool storage is not necessary. If analysis can not be started within the two hours of sample collection to reduce the change in sample, keep all samples at 4° C.

Do not allow samples to freeze. Do not open sample bottle before analysis.

Begin analysis within six hours of sample collection

6.5.1 PRECAUTIONS

The following precautions should be observed while performing the experiment:

In this experiment, we are handling the indicator in powder form, in contrast with most of all other basic experiments.

Handle the indicator powder carefully to get exact results.

Handling of the alkali, NaOH should be done with utmost care, since it causes irritation.

Since NaOH pellets are hygroscopic in nature, do not expose these pellets to air for a prolonged time period.

6.6 PROCEDURE

6.6.1 PREPARATION OF REAGENTS

Standard EDTA Solution (0.02 M)

1. Switch on the Electronic balance, keep the weighing pan, and set the reading to zero. Take 1000 mL of distilled water in a 1000 mL standard flask.
2. Weigh 3.723g of EDTA sodium salt.
3. Transfer the contents to the water sample. Place the lid and mix the contents thoroughly until all EDTA sodium salt dissolves in water.
4. Make the volume exactly 1000 mL by adding distilled water. Transfer the solution to a clean reagent bottle named EDTA solution.
5. This 0.02 molar EDTA solution is going to be used as a titrant in this experiment. Take this solution in a beaker for easy handling.

Ammonium purpurate

1. Weigh 0.5g of Ammonium Purpurate.
2. Transfer it to the dry beaker.
3. Weigh 100g of sodium chloride.
4. Transfer it to the beaker having ammonium purpurate mix the contents thoroughly. Use it as a dry powder.

Sodium Hydroxide (1N) solution

1. Take 100 mL of distilled water in a beaker.
2. Weigh 4 gm of Sodium hydroxide powder.
3. Transfer it to the distilled water in the beaker and mix it thoroughly.
4. Then transfer the entire content to 100 mL standard flask.
5. Rinse the glass rod and funnel with distilled water.
6. Make the volume 100 mL by adding distilled water up to the mark.
7. Take the sodium hydroxide solution in 100 mL beaker for easy handling.

6.6.2 TESTING OF SAMPLE

- Pipette 20 mL of water sample and transfer it to a clean 250 mL conical flask.
- Measure 2 mL of 1N sodium hydroxide solution using measuring cylinder. Add it to the water sample in conical flask so that the pH will be maintained between 12 and 13.
- Add few amount of Ammonium purpurate indicator to the water sample. Now the sample turns into pink color. This color change is due to the calcium and magnesium contents present in water.
- Before starting the titration rinse the burette with few mL of EDTA solution and discard it. Fill the burette with 0.02M EDTA solution. Adjust the reading to zero, then fix it in burette stand. Ensure that, there is no any air bubble inside the burette.
- Titrate the water sample against the EDTA solution in the burette till all calcium and magnesium ions present in the sample reacts with the EDTA to form a metal EDTA complex by changing the color of the sample to purple. i.e., the end point.
- Note down the burette reading

- The value of titration is 5.7 mL
- Repeat the titration for concordant values

6.7 CALCULATION

To determine Calcium Hardness in the given water sample, the readings are required to be tabulated.

Burette solution: EDTA

Pipette solution: Sample

Indicator: Ammonium Purpurate

End point: Appearance of Purple color.

6.7.1 TABLE

Sample No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of EDTA (mL)
		Initial	Final	
1.				
2.				
3.				

- Here the volume of water sample is 20 mL.
- The EDTA is taken in the Burette.
- For the first titration, the volume of initial reading is 0 mL. The final reading is 5.6 mL.
- The volume of EDTA consumed to get the end point is 5.6 mL.
- For the second titration, the volume of initial reading is 0 mL. The final reading is 5.7 mL.
- The volume of EDTA consumed to get the end point is 5.7 mL.
- For the third titration, the volume of initial reading is 0 mL. The final reading is 5.7 mL.
- The volume of EDTA consumed to get the end point is 5.7 mL.
- For the second and third titration, the burette reading is same so we have achieved concordant value. We can go for the calculations

- Calcium hardness of the given water sample in mg/L as calcium carbonate equivalents is equal to volume of EDTA * normality * 50 *1000 divided by volume of sample taken
- Here the volume of EDTA is 5.7 mL.
- Normality is 0.02Molar
- And volume of sample taken is 20 mL. Substituting the values in the formula and calculating we get the value 285 mg/L
- So the Calcium Hardness in mg/L as CaCO_3 equivalents is 285 mg/L
- So, the calcium present in the sample is equal to Ca hardness in mg/L as CaCO_3 equivalent * Molecular weight of Calcium divided by Molecular weight of Calcium Carbonate.
- Here the molecular weight of calcium is 40.078 and the molecular weight of calcium carbonate is 100.09, substituting the values, and calculating, we get 114 mg/L. So, the result is 114 mg/L.

6.7.2 DATA SHEET

DETERMINATION OF CALCIUM HARDNESS 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 EDTA (mL)
		Initial	Final	
1.	20	0	5.6	5.6
2.	20	0	5.7	5.7
3.	20	0	5.7	5.7

Specimen Calculation:

Volume of EDTA = 5.7 mL
Normality of EDTA = 0.02 N
Volume of Sample = 20.0 mL
Equivalent weight of CaCO_3 = 50

Calcium Hardness = $\frac{\text{Volume of EDTA} \times \text{N} \times 50 \times 1000}{\text{Volume of sample taken}}$

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

Calcium Hardness as CaCO_3 equivalent (mg/L) = $5.7 \times 0.02 \times 50 \times 1000/20$
= 285 mg/L as CaCO_3 equivalent

Calcium Present in the sample = $\frac{\text{Ca hardness in mg/L as CaCO}_3 \times \text{molecular weight of Ca}}{\text{molecular weight of Calcium carbonate}}$

$$= \frac{285 \times 40.078}{100.09}$$
$$= 114 \text{ mg/L}$$

6.8 INTERPRETATION OF RESULTS

The Calcium Hardness of the given sample of water = 285 mg/L and

Calcium ion concentration in the given sample of water = 114 mg/L

6.9 INFERENCE

Calcium and magnesium ions are important contributors to water hardness. When water is heated, they break down and precipitate out of solution, forming scale. Maximum limits have been established. Magnesium concentrations more than 100 mg/L may have a laxative effect on some people.

6.10 EVALUATION

1. In the determination of hardness by titration, EDTA is used as
 - a) Oxidizing Agent
 - b) Chelating Agent
 - c) Neutralizing Agent
 - d) Binding Agent
2. As per drinking water standards in India, the limit of Mg in drinking water is
 - a) 40 ppm
 - b) 20 ppm
 - c) 30 ppm
 - d) 50 ppm
3. As per drinking water standards in India, the limit of Ca in drinking water is
 - a) 75 ppm
 - b) 50 ppm
 - c) 100 ppm
 - d) 150 ppm
4. The other name of Ammonium Purpurate is _____.
 - a) Dioxide
 - b) Murexide
 - c) EBT
 - d) Trioxide

5. The presence of Ca, in the natural water is due to the passage through _____.

- a) Lime Stone
- b) Atmosphere
- c) Stratosphere
- d) Clouds

6. The temporary hardness of water is due to

- a) Sulfate of calcium and magnesium
- b) Chlorides of calcium and magnesium
- c) Carbonate and bicarbonate of calcium and magnesium
- d) Nitrates of calcium and magnesium

7. The permanent hardness of water is due to

- a) Carbonate and bicarbonate of calcium and magnesium
- b) Sulfate and bicarbonates of calcium
- c) Chlorides and carbonates of magnesium
- d) Sulfates, chlorides and nitrates of calcium and magnesium

8. Magnesium hardness with sulphate ions produce.

- a) Cancer
- b) Breathing problem
- c) Laxative effect
- d) Sleepiness

9. The role of NaOH in the titration is

- a) NaOH reacts with Mg^{2+} ions and precipitates it out in the form of $Mg(OH)_2$
- b) NaOH reacts with Ca^{2+} ions and precipitates it out in the form of $Ca(OH)_2$
- c) For pH Adjustment
- d) For reducing the error

10. The calcium salts are useful for the growth of bones in human beings.

- a) True
- b) False

KEY TO ITEMS:

- 1) b
- 2) c
- 3) a
- 4) b
- 5) a
- 6) c
- 7) d
- 8) c
- 9) a
- 10) True