

5.0 EXPERIMENT ON DETERMINATION OF TOTAL HARDNESS

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5.0 EXPERIMENT ON DETERMINATION OF TOTAL HARDNESS

PREAMBLE:

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

Test procedure is in accordance to IS: 3025 (Part 21) - Reaffirmed 2002.

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 2340 - C.
- (2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 130.2.

5.1 AIM

To determine the total hardness of given water sample with the stipulations as per **IS: 3025 (Part 21) - Reaffirmed 2002**.

5.2 INTRODUCTION

Water that has high mineral content is known as **Hard water**. Hard water contains bicarbonate, chlorides and sulphates of calcium and magnesium.

When treated hard water with soap, it gets precipitated in the form of insoluble salts of calcium and magnesium. Hardness of water is a measure of the total concentration of the calcium and magnesium ions expressed as calcium carbonate. There are two types of hardness

1. Temporary hardness
2. Permanent hardness

Temporary Hardness is due to the presence of bicarbonates of calcium and magnesium. It can be easily removed by boiling.

Permanent Hardness is due to the presence of chlorides and sulphates of calcium and magnesium. This type of hardness cannot be removed by boiling.

5.2.1 Environmental Significance

- Scales are formed as inner coating of the pipelines prevents corrosion.
- Absolutely soft waters are corrosive and dissolve the metals.
- More cases of cardio vascular diseases are reported in soft water areas.
- Hard water is useful to growth of children due to the presence of calcium.
- Hard waters cause excessive consumption of soap used for cleaning purpose. Sodium soaps react with multivalent metallic cations to form a

precipitate, thereby lose their surfactant properties. Lathering doesn't take place until all hardness ions precipitate out.

- This precipitate adheres to surfaces of tubes, sinks, dish washer and may stain clothing.
- Scales formed mainly due to carbonate hardness act as insulations and cause enormous loss of fuel in boiler.
- Scales deposited mainly due to increase in pH to 9 at which bicarbonates are converted as carbonates are formed in distribution mains reducing their carrying capacity.

5.3 PRINCIPLE

A water sample is buffered to pH 10.1 and taken in to a conical flask. If an indicator dye like EBT, when added to a solution containing Calcium and Magnesium ions, the color of the solution turns to wine red.

EDTA, the titrant, complexes with Magnesium and Calcium ions, removing them from association with the indicator.

When all the Mg and Ca are complexed with EDTA, the indicator will turn blue. This is the end point of the titration.

5.4 MATERIALS REQUIRED

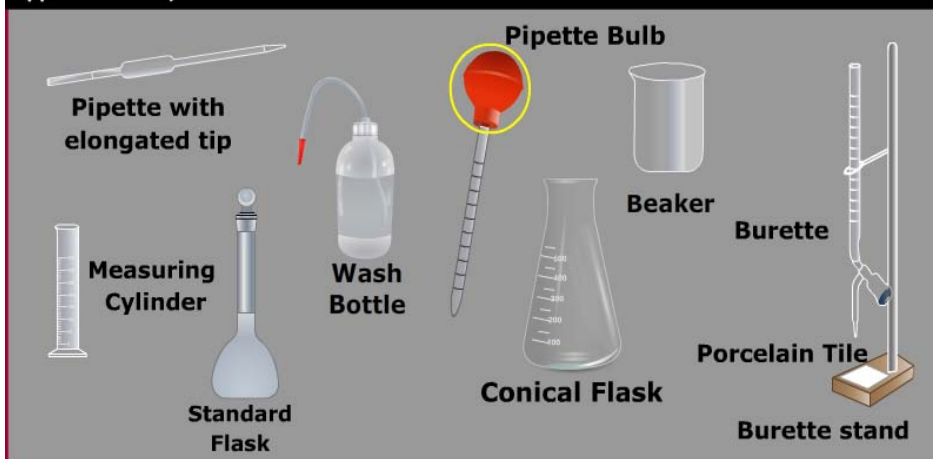
5.4.1 APPARATUS REQUIRED

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

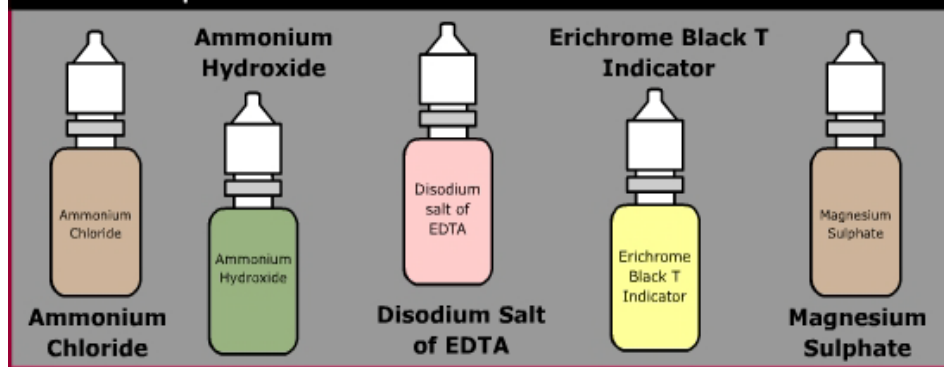
5.4.2 CHEMICALS REQUIRED

1. Ammonium Chloride
2. Ammonium Hydroxide
3. EDTA (Disodium Salt of EDTA)
4. Erichrome Black T
5. Magnesium sulphate

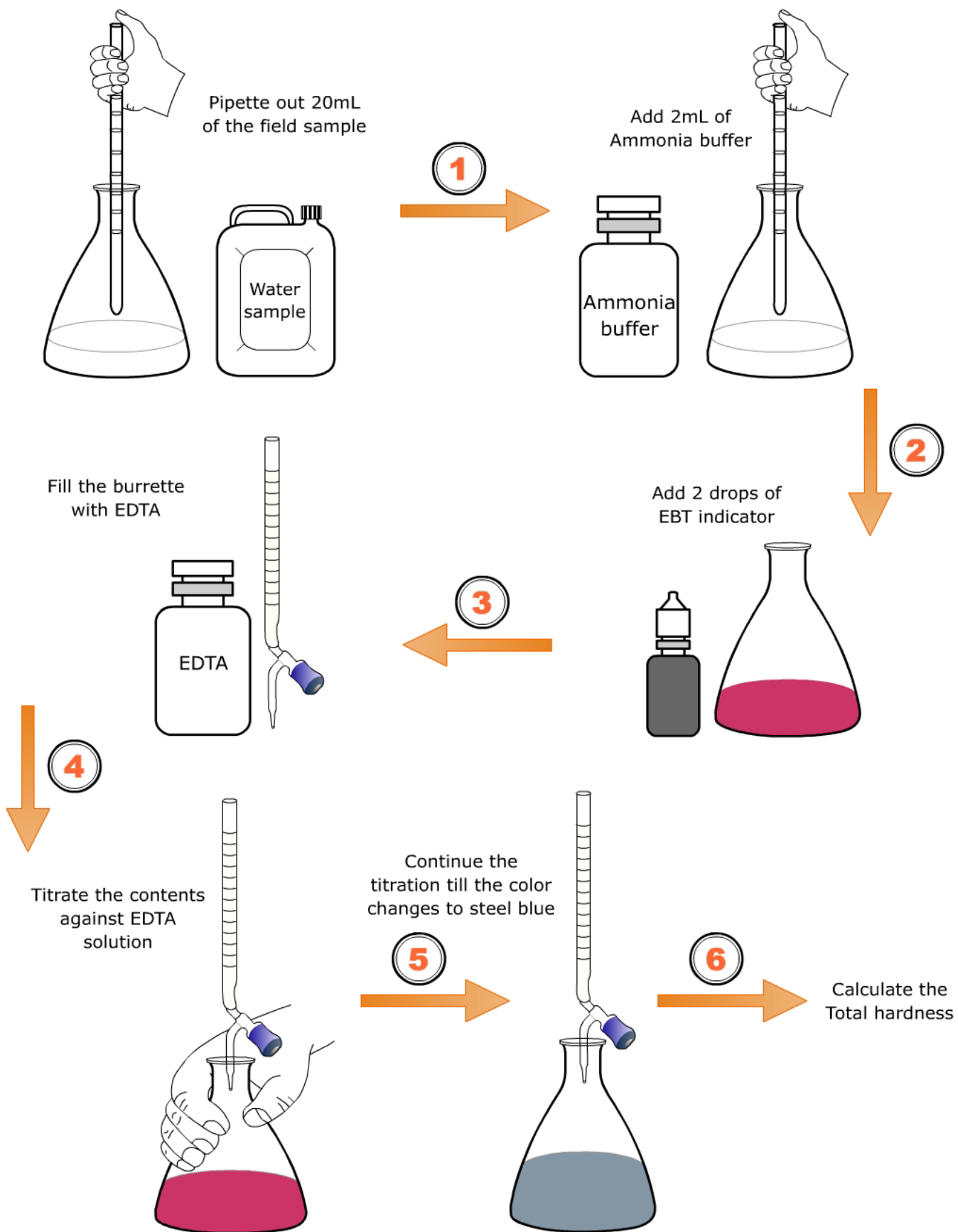
Apparatus Required



Chemicals Required



PROCEDURE CHART



5.5 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

5.5.1 PRECAUTIONS

- Here we are handling ammonia solution so necessary precaution should be taken for preventing the inhalation.
- It causes irritation if inhaled.
- Do not pipette out the buffer solution using either measuring cylinder, automatic pipette (or) pipette with a sucker.
- Always store EDTA solution and buffer solution in a plastic or resistant glass container.
- Discard the buffer solution if it is turbid or if it is stored for a very long period of time.

5.6 PROCEDURE

5.6.1 PREPARATION OF REAGENTS

Buffer Solution preparation

- Switch on the Electronic balance, keep the weighing pan, set the reading to zero.
- Measure 50 mL of distilled water and transfer it to the beaker
- Weigh 1.179g of EDTA
- Now the weight is 1.179gms
- Transfer the contents to the beaker having 50 mL of distilled water and dissolve it thoroughly.
- Weigh 16.9g of ammonium chloride.
- Add it to the contents in the beaker. And dissolve it thoroughly.
- Weigh 780mg of magnesium sulphates and transfer it to the beaker.
- Measure 143 mL of Ammonium hydroxide solution using measuring cylinder and add it to the contents in the beaker.

- Place the funnel over the 250mL standard flask and transfer the dissolved contents from beaker
- Make the volume upto 250mL mark by adding distilled water. Transfer the buffer solution to a clean reagent bottle labelled as buffer solution. This buffer solution is used to maintain the pH of water sample between 9 and 10.

Erichrome Black T

- Weigh 0.5g of Erichrome black T
- Transfer it to 100mL standard flask using funnel
- Add distilled water in the standard flask and make the volume exactly upto 100 mL mark.
- Put the lid and shake the contents well.
- Transfer the solution to a clean reagent bottle named EBT

Standard EDTA Solution (0.02 M)

- Switch on the Electronic balance, keep the weighing pan, and set the reading to zero.
- Weigh 3.723g of EDTA sodium salt
- Transfer the entire content to 1000 mL standard flask
- Fill with distilled water up to 1000 mL mark
- Put the lid and shake the contents well.
- For easy handling take the EDTA solution in a 250 mL beaker.

5.6.2 TESTING OF WATER SAMPLE

- Pipette 20mL of water sample and transfer it to a clean 250mL conical flask.
- Add 2mL of Ammonia buffer solution to the water sample so that the pH will be maintained between 9 and 10.
- Add few drops of EBT indicator to the conical flask and the sample turns to wine red in color.
- Before starting the titration rinse the burette with few mL of EDTA. Fill the burette with 0.02M EDTA solution and adjust to zero then fix it in burette stand.
- Titrate the sample against the EDTA solution in the burette till all calcium and magnesium ions present in the sample reacts with the EDTA. The appearance of blue colour indicates that all Ca & Mg ions

are complexed with EDTA and forms a metal EDTA complex i.e., the end point of the titration.

- Note down the burette reading
- The value of titration is 29.8mL
- Repeat the titration for concordant values

5.7 CALCULATION

5.7.1 TABLE

Burette solution: EDTA

Pipette solution: Sample

Indicator: EBT

End point: Appearance of blue color

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 29.3 mL.
- The volume of EDTA consumed to get the end point is 29.3 mL.
- For the second titration, the volume of initial reading is 0 mL. The final reading is 29.8 mL.
- The volume of EDTA consumed to get the end point is 29.8 mL.
- For the third titration, the volume of initial reading is 0. The final reading is 29.8 mL.
- The volume of EDTA consumed to get the end point is 29.8 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 in to normality in to 50 in to 1000 divided by volume of sample taken
- Here the volume of EDTA is 29.8 mL
- Normality is 0.02Molar
- And volume of sample taken is 20mL. Substituting the values in the formula and calculating we get the value 1490 mg/L

5.7.2 DATA SHEET

DETERMINATION OF TOTAL 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	29.3	29.3
2.	20	0	29.8	29.8
3.	20	0	29.8	29.8

Specimen Calculation:

Volume of EDTA = 29.8 mL

Normality of EDTA = 0.02 N

Volume of Sample = 20.0 mL

Equivalent weight of CaCO_3 = 50

Total 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) = $29.8 \times 0.02 \times 50 \times 1000 / 20$

= 1490 mg/L as CaCO_3 equivalent

5.8 INTERPRETATION OF RESULTS

The Total Hardness of the given sample of water = **1490 mg/L**.

5.9 INFERENCE

Hardness is the property which makes water to form an insoluble precipitate with soap and is primarily due to the presence of calcium and magnesium ions. Hard waters have no known adverse health effects and may be more palatable than soft waters. Hard water is primarily of concern because it requires more soap for effective cleaning, causes yellowing of fabrics, toughens vegetables cooked in the water and forms scales in boilers, water heaters, pipes and cooking utensils. The hardness of good quality water should not exceed 250 mg/L measured as calcium carbonate equivalents. Waters softer than 30 to 50 mg/L may be corrosive to piping depending on pH, alkalinity and dissolved oxygen.

5.10 EVALUATION

1. The cation that cannot cause hardness is _____.
 - a) Ca
 - b) Mg
 - c) Fe
 - d) H

2. Addition of complexometrically neutral Mg to buffer is to
 - a) get a clear precise end point
 - b) neutralize sodium present
 - c) neutralize calcium present
 - d) maintain pH of the buffer

3. The hard water does not lather with soap because Soap is precipitated
 - a) by Ca and Mg ions
 - b) only by Mg ions
 - c) only by Ca ions
 - d) by Na ions

4. The buffers can be stored

- a) for a month
- b) no time limit
- c) for an year
- d) as long as the solution is clear without any turbidity

5. The need for buffer in the titration is ____.

- a) for maintaining volume
- b) for maintaining the pH
- c) for maintaining the temperature
- d) to nullify the error

6. The hard water consumes more soap for cleaning purposes.

- a) True
- b) False

7. E.D.T.A. means

- a) Ethylene diamine tetra acetic acid
- b) Erichrome diamine tetra acetic acid
- c) Ethylene dye toluene acid
- d) Erichrome dye toluene acid

8. The hard water

- a) is corrosive
- b) forms scales
- c) is tasteless
- d) is costly

9. Buffer solution is the solution which resists change in its

- a) pH
- b) colour
- c) turbidity
- d) conductivity

10. The Erichrome Black T is used as a catalyst.

- a) True
- b) False

KEY TO ITEMS:

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