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Determination of total hardness of water by EDTA Method.

Aim of the experiment:-

To determine the total hardness of the water by EDTA method.

Requirements:-

Chemicals:- Water sample, standard sodium EDTA solution, buffer solution, Eriochrome Black-T indicator.

Apparatus:- Burette(50ml), Pipette(25ml), Conical flask(250ml), measuring flask(250ml), Test tube(10ml)

Theory:-

The property of water, which restricts or checks the lather formation with soap, is called hardness. Scaling of hot water pipes, boilers & other house hold appliances is due to hard water. Water free from ~~soluble~~ salts of calcium & magnesium is called soft water.

Hard water may have moderate health benefits, but can pose serious problems in industrial settings, where water hardness is monitored to avoid costly breakdowns in boilers, cooling towers & other equipments that handles water. In domestic settings, hard water is often indicated by a lack of suds formation when soap is agitated in water & by the formation of lime scale in kettles & water heaters. Wherever water hardness is a concern, water softening is commonly used to reduce the hard water is adverse effects.

The water which contains large amount of minerals (common calcium containing minerals are calcite & gypsum,

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& magnesium containing minerals is dolomite, which also contains calcium) is called hard water. Hard watere contains bicarbonate (HCO_3^-), carbonate (CO_3^{2-}), halide ($X^- = F^-, Cl^-$ & Br^-), sulphate (SO_4^{2-}) of calcium (Ca^{2+}) & magnesium (Mg^{2+}) as well as trace amount of iron (Fe^{2+}, Fe^{3+}), aluminium (Al^{3+}), heavy metals ($Pb^{2+}, Cd^{2+}, Bi^{3+}, As^{3+}, Hg_2^{2+}$) etc. Basically the hardness of water is due to the soluble salt of Ca^{2+} & Mg^{2+} ions. The following equilibrium reaction describes the dissolving & formation of calcium carbonate.

$$CaCO_3(s) + CO_2(aq) + H_2O(l) \rightleftharpoons Ca^{2+}(aq) + 2HCO_3^-(aq)$$

The hardness is usually expressed in parts of $CaCO_3$ equivalent or calcium & magnesium salts per million parts of water i.e. ppm. Also amount of Ca^{2+} & Mg^{2+} ion present in hard water is significally highere in comparison to other ions.

Temporary or Carbonate hardness: It is due to the Presence of magnesium & calcium bicarbonates [$Ca(HCO_3)_2$ & $[Mg(HCO_3)_2]$], which can be removed by boiling the water.

$$H_2O(l) + CO_2(g) \longrightarrow H_2CO_3(aq)$$
$$CaCO_3(s) + H_2CO_3(aq) \rightarrow Ca(HCO_3)_2(aq)$$
$$MgCO_3(s) + H_2CO_3(aq) \rightarrow Mg(HCO_3)_2(aq)$$

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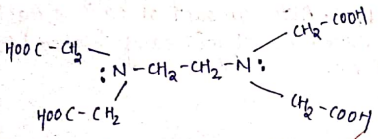
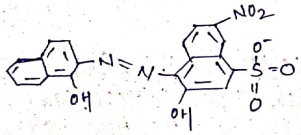


(Structure of EDTA)



(Structure of EBT)

Disodium EDTA

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Temporary hardness can be removed by clarke's method by adding lime water ($Ca(OH)_2$) to the hard water.

$$Ca(HCO_3)_2(aq) + Ca(OH)_2(s) \rightarrow 2CaCO_3 + 2H_2O$$

$$Mg(HCO_3)_2(aq) + Ca(OH)_2(s) \rightarrow MgCO_3 + CaCO_3 + 2H_2O$$

As the magnesium carbonate & calcium carbonate are insoluble in water it could be settled down.

Permanent or non carbonate hardness :- It is due to the presence of soluble salts of magnesium & calcium in the form of chlorides & sulphates in water ($CaCl_2$, $CaSO_4$, $MgCl_2$ & $MgSO_4$), which cannot be removed by boiling.

Principle :-

The hardness of water can be accurately determined by complexometric titration using a chelating agent, ethylene diammine tetra acetic acid (EDTA) usually in the form of disodium salt (H_2Y^{2-}). EDTA in the form of its disodium salt forms complex with Ca^{2+} & Mg^{2+} ions of water sample. When Eriochrome Black-T (EBT) indicator is added to the hard water at PH around 9-10, it gives a wine red coloured unstable complex with Ca^{2+} & Mg^{2+} ions of water sample. To maintain the PH of the solution at 9-10, buffer solution ($NH_4Cl + NH_4OH$) is used. When this wine red coloured complex is titrated against EDTA solution of known strength, the Ca^{2+} & Mg^{2+} ions form stable metal complex with EDTA & colour changes from wine red to blue (colour of free EBT indicator) at the end point titration.

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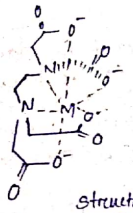

Structure of metal ($M = Ca^{2+}$ and Mg^{2+}) - EDTA complex.

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Chemical Reactions:

$$\left(\begin{matrix}Ca^{2+}, Mg^{2+} \text{ waters} \\ \text{sample colourless}\end{matrix}\right) \xrightarrow{EBT\ (pH = 9\text{-}10)} \left(\begin{matrix}Ca^{2+}, Mg^{2+}\text{-EBT} \\ \text{unstable complex wine red colour}\end{matrix}\right)$$

$$\left(\begin{matrix}Ca^{2+}, Mg^{2+}, EBT \\ \text{unstable complex} \\ \text{wine red colours}\end{matrix}\right) \xrightarrow[(pH = 9\text{-}10)]{EDTA} \left(\begin{matrix}Ca^{2+}, Mg^{2+}\text{-EDTA} \\ \text{stable complex} \\ \text{colourless}\end{matrix}\right) + \left(\begin{matrix}EBT \\ \text{blue} \\ \text{colour}\end{matrix}\right)$$

Procedure (calculation of total hardness)

1. Thoroughly wash burette, pipette & conical flask with supplied waters followed by distilled water to ensure that no contaminant is present.
2. Rinse & fill the burette with given standard EDTA solution. Remove air if any inside the bottom of the burette & take the initial burette reading (IBR).
3. Pipette out exactly 50ml of the supplied waters sample into a conical flask & add 10-15ml of buffer solution & also add 1-2 drops of EBT indicator.
4. Titrate it with the EDTA solution taken in the burette with continous shaking / stirring till the wine red colour solution changes to blue.
5. Write down the final burette reading (FBR)
6. Repeat the procedure to get three concordant readings (three consecutive readings are having equal values).

Procedure (calculation of Permanent hardness):-

1. Thoroughly wash burette, pipette & conical flask with supplied water followed by distilled waters to ensure that no contaminant is required present.

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No. of obs.	Volume of water sample (ml)	Burette reading (ml)		Difference (ml)	Remarks
		IBR	FBR	FBR - IBR (V_1)	
1.	40	0.1	4.4	4.3	Rough
2.	40	4.4	8.6	4.2	
3.	40	8.6	12.8	4.2	concordant
4.	40	12.8	16.9	4.2	

Avg (V_1) = 4.16 ml

No. of obs.	Volume of water sample (ml)	Burette reading (ml)		Difference (ml)	Remarks
		IBR	FBR	FBR - IBR (V_2)	
1.	40	0.3	3.5	3.2	Rough
2.	40	3.5	6.6	3.1	
3.	40	6.6	9.6	3.0	concordant
4.	40	9.6	12.6	3.0	

Avg (V_2) = 3.03 ml

Calculation :-

V_1 = Volume of EDTA needed to convert metal EBT complex to metal EDTA complex for supplied water sample.

V_2 = Volume of EDTA needed to convert metal EBT complex to metal EDTA complex for supplied boiled water sample.

2. Rinse & Fill the burette with given standard EDTA solution. Remove air if any inside the bottom of the burette & take the initial burette reading (IBR).
3. Pipette out exactly 50 ml of the supplied boiled water sample into a conical flask & add 10-15 ml of buffer solution & also add 1-2 drops of EBT indicator.
4. Titrate it with the EDTA solution taken in the burette with continous shaking/ stirring till the wine red colour solution changes to blue.
5. Write down the final burette reading (FBR).
6. Repeat the procedure to get three concordant readings (three consecutive readings are having equal values).

$V_1 = \frac{4.2 + 4.2 + 4.1}{3} = 4.16$ ml

$V_2 = \frac{3.1 + 3.0 + 3.0}{3} = 3.03$ ml

For total hardness

1L of 1M EDTA $\longrightarrow$ 100gm. of $CaCO_3$

1ml of 1M EDTA = $\frac{100}{1000}$ gm of $CaCO_3$

1ml of 0.02M EDTA = $\frac{0.02}{10}$ gm of $CaCO_3$

4.16ml of 0.02M EDTA = $\frac{0.02}{10} \times 4.16 = 8.32 \times 10^{-3}$ gm of $CaCO_3$

40 ml of sample of cold water contains = 8.32×10^{-3} gm of $CaCO_3$

1000 ml of sample of cold water contains = $\frac{8.32}{40} = 0.208$ gm

= 208 ppm

For Permanet hardness :-

1L of 1M EDTA = 100g of $CaCO_3$

1ml of 0.02M EDTA = $100 \times 0.02 \times 10^{-3}$ g $CaCO_3$

3.03ml of 0.02M EDTA = $3.03 \times 0.02 \times 10^{-1}$ g. $CaCO_3$

$= 6.06 \times 10^{-3}$ g. $CaCO_3$

40ml of sample of boiled water contains = 6.06×10^{-3} gm of $CaCO_3$

1L of sample of boiled water contains = $\frac{6.06}{40} = 0.151$ gm

= 151 ppm

Total hardness = 208 ppm

Permanet hardness = 151 ppm

Temporary hardness = total hardness - permanet hardness

= (208 - 151) ppm

= 57 ppm.

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Conclusion :-

The supplied water sample contains 208ppm of total hardness, 151 PPM of permanet hardness and 57 PPM of temporary hardness.

02/02/2017

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