17BITOD28

Water softening through ion exchange method – Estimation of residual hardness

Expt No. 06 **Date:** 01/02/2018

Principle: Hardness of water is due to the presence of dissolved calcium and magnesium salts in water. Ehtylenediaminetetraacetic acid (EDTA) forms complexes with a large number of cations including Ca^{2+} and Mg^{2+} depending upon pH of solution. Hence, it is possible to determine the total hardness of water using EDTA solution. EDTA in the form of its sodium salt (H_2Y^{2-}) is commonly used in complexometric titration for estimation of metal ion because pure EDTA (H_4Y) is sparingly soluble in water.EDTA has six binding sites (the four carboxylate groups and the two amino groups) providing six pairs of electrons. The resulting metal-ligand complex, in which EDTA forms a cage-like structure around the metal ion, is very stable at specific *p*H. All metal-EDTA complexes have a 1:1 stoichiometry. The H_2Y^{2-} form complexes with metal ions as follows.

$$M + H_2Y^{2-} \rightarrow MY^{2-} + 2H^+ \text{ ---------- (1)}$$

Where, M is Ca^{2+} and Mg^{2+} present in water. Reaction (1) can be carried out quantitatively at *p*H 10 usingEriochrome Black T (EBT) as indicator. EBT forms a wine-red complex with M^{2+}ions which is relatively less stable than the M^{2+}-EDTA complex. On titration EDTA first reacts with free M^{2+} ions and then with the metal-EBT indicator complex. The latter gives a colour change from wine-red to steel blue at the equivalence point.

Removal of hardness using ion exchange method: See in page-3.

Requirements

Reagents and solutions: Standard hard water (1mg/mL of $CaCO_3$ equivalents), EDTA solution, EBT indicator, hard water sample, NH_3-NH_4Cl buffer solutionand ion exchange resin.

Apparatus: Burette, pipette, conical flask, standard flask burette stand and ion exchange column.

Procedure

Titration I - Standardisation of EDTA

Pipette out 20mL of the standard hard water containing 1mg/mL of calcium carbonate (1000 ppm)into a clean conical flask. Add one test tube full of ammonia buffer (NH_4OH – NH_4Cl) solution to maintain the pH around 10. Add three drops of Eriochrome Black – T (EBT) indicator and titrate it against the given EDTA solution taken in the burette. The end point is change of colour from wine red to steel blue. Repeat the titration for concordant titre values. Let 'V_1' be the volume of EDTA consumed.

S. No.	Volume of standard hard water (mL)	Burette reading (mL)		Volume of EDTA (V_1, mL)
		Initial	Final	
1	20	0	18.6	18.6
2	20	0	18.6	18.6
3				
Concordant titre value				18.6

Calculations:

20mL of given hard water consumes V_1mL of EDTA

20 mg of $CaCO_3$ requires V_1 mL of EDTA for complexation

$\therefore$1 mL of EDTA requires = 20/V1 mg $CaCO_3$ for complexation

This relation will be used in other two titrations

Titration II - Estimation of Total hardness of hard water sample

Pipette out 20mL of the given sample of hard water into a clean conical flask. Add one test tube full of ammonia buffer (NH_4OH – NH_4Cl) solution and three drops of Eriochrome Black–T (EBT) indicator. Titrate this mixture against standardized EDTA solution taken in the burette. The end point is the change of color from wine red to steel blue. Repeat the titration as carried out earlier for concordant titre value. Let 'V_2' be the volume of EDTA consumed.

S. No.	Volume of standard hard water (mL)	Burette reading (mL)		Volume of EDTA (V_2, mL)
		Initial	Final	
1	20	0	10.2	10.2
2	20	0	10.2	10.2
3				
Concordant titre value				10.2

Calculations:

From Titration 1, we have the following relation:

∴1 mL of EDTA requires = 20/V1 mg $CaCO_3$ for complexation

From this relation, it can be seen that

20 mL of hard water sample consumes = V_2 mL of EDTA.

= V_2 x 20/V1 mg of $CaCO_3$equ

∴ 1000 mL of hardwater sample consumes = V_2 x 20/V1×1000/20

= V_2/V1×1000 ppm

∴Total hardness of the water sample = "X" ppm = 548.3 ppm

Removal of hardness using ion exchange method: (This should be the first step in the lab)

Arrange the ion exchange column on to a burette stand and place a clean funnel on top of the column. Take 50 mL of the hardwater sample in a beaker and slowly pass the water sample through the ion exchange column. Place a clean beaker under the column and collect water passing through the column over a period of 15 minutes. Adjust the valve of the column to match the duration of outflow.

From the water collected through the column, pipette out 20 mL into a clean conical flask and repeat the EDTA titration as carried out above. Note down the volume of EDTA consumed as ' V_3'.

Calculations:

From Titration 1, we have the following relation:

∴1 mL of EDTA requires = 20/V1 mg $CaCO_3$ for complexation

From this relation, it can be seen that

20 mL of water sample after softening through the column consumes = 0.0 V_3mL of EDTA.

$= V_3 \times 20/V_1$ mg of $CaCO_3$ equ

∴ 1000 mL of water sample after softening through the column consumes =

$= V_3 \times 20/V_1 \times 1000/20$

$= V_3/V_1 \times 1000$ ppm

∴Residual hardness of the water sample = "Y" ppm

Result:

1. Total hardness of the water sample = "X" ppm = 548.3 ppm
2. Residual hardness in the water sample = "Y" ppm = 0 ppm
3. Hardness removed through the column = X – Y ppm = 548.3 ppm

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