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076
AIDS - B2

LAB MANUAL: EXPERIMENT 5

Aim: To estimate the amount of total, permanent and temporary hardness in a given sample of water.

Theory:

Hardness in water is due to the presence of dissolved salts of calcium and magnesium. It is unfit for drinking, bathing, washing and it also forms scales in boilers. Hence it is necessary to estimate the amount of hardness producing substances present in the water sample. Once it is estimated, the amount of chemicals required for the treatment of water can be calculated.

The estimation of hardness is based on complexometric titration. Hardness of water is determined by titrating with a standard solution of ethylene diamine tetra acetic acid (EDTA) which is a complexing agent. Since EDTA is insoluble in water, the disodium salt of EDTA is taken for this experiment. EDTA can form four or six coordination bonds with a metal ion.

1. Total hardness

Total hardness is due to the presence of bicarbonates, carbonates, chlorides and sulphates of calcium and magnesium ions.

The total hardness of water is estimated by titrating the water sample against EDTA using Eriochrome Black-T (EBT) indicator. Initially EBT forms a weak EBT-Ca^{2+}/Mg^{2+} wine red coloured complex with Ca^{2+}/Mg^{2+} ions present in the hard water. On addition of EDTA solution, Ca^{2+}/Mg^{2+} ions preferably forms a stable EDTA-Ca^{2+}/Mg^{2+}complex with EDTA leaving the free EBT indicator in solution which is steel blue in colour in the presence of ammonia buffer (mixture of ammonium chloride and ammonium hydroxide, pH 10).

Eriochrome Black-T + Ca^{2+}/Mg^{2+} ⟶ Eriochrome Black-T-Ca^{2+}/Mg^{2+}
(Wine red)

Eriochrome Black-T-Ca^{2+}/Mg^{2+} + EDTA ⟶ EDTA-Ca^{2+}/Mg^{2+} + Eriochrome Black-T
(Wine red) (Steel blue)

2. Temporary hardness

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

When water is boiled, temporary hardness producing substances (bicarbonates) are precipitated as insoluble carbonates or hydroxides. This precipitate can be removed by filtration. (The filtrate is used in the next step)

Eriochrome Black-T + Ca^{2+}/Mg^{2+} ⟶ Eriochrome Black-T-Ca^{2+}/Mg^{2+}
(Wine red)

Eriochrome Black-T-Ca^{2+}/Mg^{2+} + EDTA ⟶ EDTA-Ca^{2+}/Mg^{2+} + Eriochrome Black-T
(Wine red) (Steel blue)

3. Permanent hardness

Permanent hardness is due to the presence of chlorides and sulphates of calcium and magnesium ions. This type of hardness cannot be removed by boiling. The filtrate obtained from the above step contains permanent hardness producing substances and is estimated against EDTA using EBT indicator.

Requirements:

EDTA ($Na_2H_2Y.2H_2O$), pH 10 buffer (2000 mL has been prepared by dissolving 140.0 g of NH_4Cl in 650 mL of deionized water, adding 1136 mL of conc. ammonia and diluting to 2000 mL), Eriochrome Black T, Burette, Pipette, conical flask (100 mL), Beaker (200 mL).

Procedure:

a) The burette was filled with standard EDTA solution to the zero level.

1. Estimation of Total Hardness

..... ml of the given water sample is pipetted out into a clean conical flask. -..... ml ammonia buffer and 2 drops of EBT indicator are added and titrated against EDTA from the burette. The end point is the change of colour from wine red to steel blue. The titration is repeated to get concordant titre value.

Titration-1 Estimation of Total Hardness

Volume of hard water sample (ml)	Burette Reading		Volume of EDTA solution (ml)	Indicator
	Initial	Final		
10	0	4.6	4.6	EBT (2drop)
10	4.6	9.4	4.8	"
10	9.4	14.0	4.6	"

2. Estimation of Permanent Hardness

100 ml of the given sample of water is pipetted out into a clean beaker and boiled for 20 minutes. It is then filtered to remove the precipitate formed due to the decomposition of temporary hardness producing salts. The filtrate is made up to 100 ml in standard measuring flask (SMF) using distilled water.

..... ml of the made-up solution is pipetted out into a conical flask, ... ml ammonia buffer and 2 drops of EBT indicator are added and titrated against the EDTA. The end point is the change of color from wine red to steel blue. The titration is repeated to get concordant titer value.

Volume of boiled water sample (ml)	Burette Reading		Volume of EDTA solution (ml)	Indicator
	Initial	Final		
10	0	2.8	2.8	EBT (2drop)
10	2.8	5.7	2.9	"
10	5.7	8.9	2.8	"

3. Temporary Hardness

The temporary hardness is calculated from the total and permanent hardness.

Temporary Hardness = Total Hardness – Permanent Hardness

Calculation:

1 ml of 0.01 M EDTA ≡ 1 mg of $CaCO_3$

V1 ml of EDTA ≡ V1 mg of $CaCO_3$

Calculation of total hardness

Volume of EDTA solution consumed = ...4.66.... ml

Volume of hard water taken = ...10..... ml

Total hardness = $\frac{\text{Volume of EDTA solution consumed X1000 ppm}}{\text{Volume of the hard water taken}}$

= .....466..... ppm

Calculation of permanent hardness

Volume of EDTA solution consumed = ...2.83..... ml

Volume of boiled water taken = ...10..... ml

Permanent Hardness = $\frac{\text{Volume of EDTA solution consumed X1000 ppm}}{\text{Volume of the boiled water taken}}$

= .....283..... ppm

Calculation of temporary hardness

Temporary hardness of the given sample of water

= Total hardness - Permanent hardness

= .....183..... ppm

Result:

The water sample contains

Total hardness =.....466.....ppm

Permanent hardness =.....283.....ppm

Temporary hardness =.....183.....ppm

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