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AIDS-B2
076

LAB MANUAL: EXPERIMENT 6

Aim: Estimation of Ca^{2+} and Mg^{2+} in a given sample of water by complexometric titration with a standard solution of EDTA.

Theory:

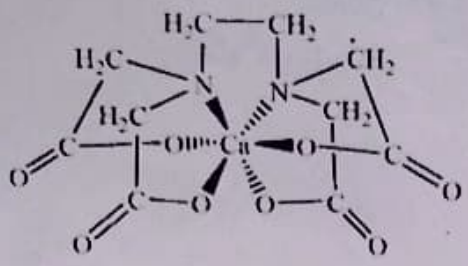
Hardness in water is due to the presence of dissolved salts of calcium and magnesium. It is unfit for drinking, bathing, and washing. It also forms scales in boilers. Hence it is necessary to estimate the amount of hardness-producing substances present in the water sample. Once it is estimated, the amount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

Estimation of the total amount of Ca^{2+} & Mg^{2+}

The total amount of Ca^{2+} & Mg^{2+} in a given sample 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-colored complex with Ca^{2+}/Mg^{2+} ions present in the hard water. Upon addition of EDTA solution, Ca^{2+}/Mg^{2+} ions preferably form a stable EDTA- Ca^{2+}/Mg^{2+} complex with EDTA leaving the free EBT indicator in solution which is steel blue in color 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
(Wine red) ---→ EDTA-Ca^{2+}/Mg^{2+} + Eriochrome Black-T
(Steel blue)



Estimation of the total amount of Ca^{2+}

At any pH beyond 10, Mg^{2+} gets precipitated as $Mg(OH)_2$. So, the solution contains only Ca^{2+} after making the pH over 10 by the addition of NaOH. Then the Ca^{2+} present in the resultant solution can be estimated by complexometric titration with EDTA solution.

Requirements:

Water sample, EDTA solution (0.01M), Eriochrome Black T, Buffer solution, NaOH solution, Burette, Pipette, conical flask (100 mL), Beaker (200 mL).

Procedure:

a) Standard 0.01 M EDTA Solution was prepared by weighing about 3.8 g of the disodium EDTA salt (Na2H2Y 2H2O) into a 1-liter volumetric flask followed by its dissolution and dilution to the mark with deionized water.

c) **Estimation of the total amount of Ca^{2+} & Mg^{2+}**

The burette was filled with standard EDTA solution to the zero level, following usual precautions. 10 mL of the given water sample is pipetted out into a clean conical flask. 2-3 mL ammonia buffer and 2 drops of EBT indicator are added and titrated against EDTA from the burette. The endpoint was the change of color from wine red to steel blue. The titration is repeated to get three concordant titer values.

d) **Estimation of the total amount of Ca^{2+}**

In a conical flask or beaker 10 mL water sample was taken and pH was made beyond 10 by the dropwise addition of NaOH solution. The solution was shaken and filtered. The filtrate was titrated by EDTA solution using EBT as the indicator.

Results and calculation:

Titration-1 Estimation of Ca^{2+} & Mg^{2+} by titration with EDTA

The volume of the given water sample (mL)	Burette Reading (mL)			The volume of EDTA solution (mL)
	Initial	Final	Use volume	
10	0	0	12	12
10	12	24.1	12.1	12.1
10	24.1	36.1	12	12

Mean = $\frac{12 + 12.1 + 12}{3}$ = 12.033

Titration-2 Estimation of Ca^{2+} by titration with EDTA

The volume of the given water sample (mL)	Burette Reading (mL)			The volume of EDTA solution (mL)
	Initial	Final	Use volume	
10	0	8	9	9
10	9	18	9	9
10	18	27.1	9.1	9.1

Mean = $\frac{9 + 9 + 9.1}{3}$ = 9.033

Calculation:

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

9.03 ml of EDTA ≡ 9.03 mg of $CaCO_3$

Calculation of the amount of Ca^{2+} & Mg^{2+}

Volume of sample water taken = 10 ml

Volume of EDTA solution consumed = 12.033 ml

$$\text{Total amount of } Ca^{2+} \text{ \& } Mg^{2+} = \frac{\text{Volume of EDTA solution consumed X1000}}{\text{The volume of the hard water taken}} \text{ ppm}$$

$$= \frac{12.033 \times 1000}{10} \text{ ppm} = 1203.3 \text{ ppm}$$

$$\text{Total amount of } Ca^{2+} = \frac{\text{Volume of EDTA solution consumed X1000}}{\text{The volume of the hard water taken}} \text{ ppm}$$

$$= \frac{9.033 \times 1000}{10} \text{ ppm} = 903.3 \text{ ppm}.$$

Total amount of Mg^{2+} = (Total amount of Ca^{2+} & Mg^{2+} - Total amount of Ca^{2+})

= 300 ppm ⇒ 1203.3 − 903.3

Result:

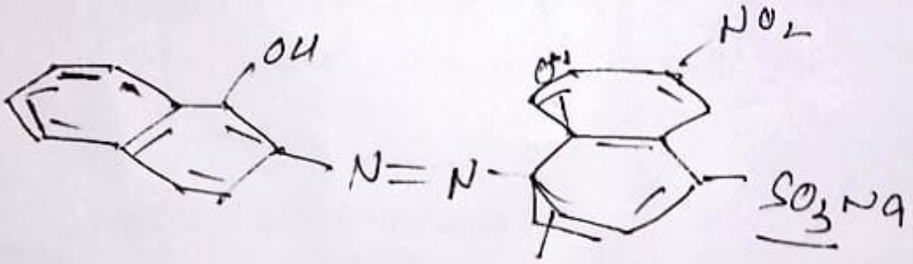
The collected water sample contains

Total amount of Ca^{2+} & Mg^{2+} = 1203.3 ppm

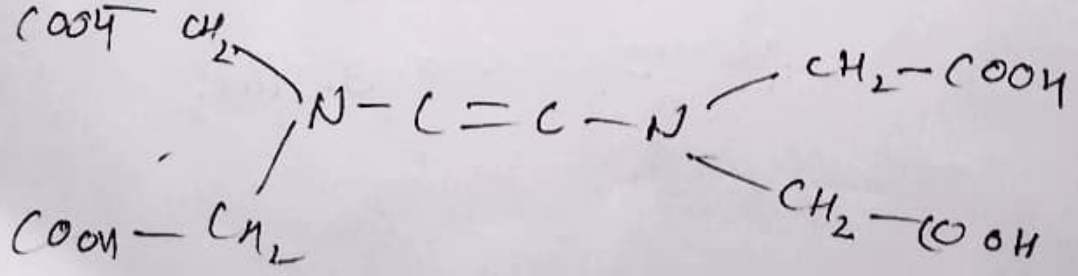
The total amount of Ca^{2+} = 903.3 ppm

The total amount of Mg^{2+} = 300 ppm

19/06/23



EBT



EDTA