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APPLIED CHEMISTRY LAB: **Paper Code:BS155/156**

EXPERIMENT. 4

AIM: To standardize EDTA solution and to determine total hardness (temporary + permanent) of the given water sample by complexometric titration.

APPARATUS: Burette, pipette, beakers, titration flask, stand.

THEORY: The hardness of water can be determined by complexometric titration. EDTA (Ethylenediamine tetracetic acid) is used as a complexing agent. In the form of its disodium salt it is used to estimate Ca^{2+} and Mg^{2+} ions, using Eriochrome black-T as an indicator.

Estimation of Ca^{2+} or Mg^{2+} by EDTA method is based on the following principal

- First, the indicator Eriochrome Black T, which is a blue colored dye, forms an unstable complex with Ca^{2+} or Mg^{2+} ions of hard water at a pH of 9 to 10. The complex is wine red in colour.

  Mg^{2+}/ Ca^{2+} + EBT → [Mg/Ca-EBT]
  Unstable Complex (wine red)

- As this solution is titrated against EDTA, free Ca^{2+} or Mg^{2+} ions in water from stable metal-ion EDTA complex.

  Mg^{2+}/ Ca^{2+} + EDTA → [Mg/Ca-EDTA]
  Stable complex (colorless)

- Once the free metal ions are complexed, the EDTA replaces Ca or Mg ions from the unstable indicator and the indicator is set free. Since the indicator is blue at above pH, the end point is appearance of blue colour

  [Mg/Ca-EBT] + EDTA → [Mg/Ca-EDTA] + EBT
  Wine red colourless blue

Thus, the amount of EDTA used corresponds to the hardness of water.

The temporary hardness is removed by boiling and after the removal of precipitate by filtration, the permanent hardness of filtrate is determined by titration with EDTA as above. The temporary hardness will be given by the difference of total hardness and permanent hardness.

I. Titration of EDTA with standard hard water (SHW) to determine the strength of given EDTA solution

PROCEDURE:

1. Rinse and fill the burette with EDTA solution.
2. Pipette out 20 mL of the standard hard water sample into a conical flask.
3. Add 2 mL of ammonia buffer solution and 2-3 drops of EBT indicator.
4. Titrate the solution till the wine red color changes to deep blue.
5. Repeat the titration to get 3 concordant readings.

OBSERVATIONS

Observation Table

S.No.	Volume of the solution taken in the titration flask (mL)	Burette Reading		Volume of the titrant used (Final- Initial reading)(mL)
		Initial Reading	Final Reading	
1.				
2.				
3.				

CALCULATIONS:

1 ml of SHW contains 1 mg of $CaCO_3$

So, 20 ml of SHW contains 20 mg of $CaCO_3$

Volume of EDTA consumed for 20 ml SHW = V_1 ml

Thus 1 mL of EDTA is used for = $20/V_1$ mg of $CaCO_3$

Strength of EDTA solution = $(20/V_1)$ mg/mL of $CaCO_3$

II. Titration of EDTA with given water sample

For determination of total hardness of water sample

1. Rinse and fill the burette with EDTA solution.
2. Pipette out 20 mL of the given hard water sample into a conical flask.
3. Add 2 mL of ammonia buffer solution and 2-3 drops of EBT indicator.
4. Titrate the solution till the wine red color changes to deep blue.
5. Repeat the titration to get 3 concordant readings.

OBSERVATIONS

Observation Table

S.No.	Volume of the solution taken in the titration flask (mL)	Burette Reading		Volume of the titrant used (Final- Initial reading)(mL)
		Initial Reading	Final Reading	
1.				
2.				
3.				

CALCULATIONS:

Concordant volume of EDTA consumed = V_2 mL

For 20 ml of given hard water sample V_2 mL of EDTA is used

For 1 ml of given hard water sample $V_2/20$ mL of EDTA is used

∴ 1 ml of given hard water sample = $(V_2/20) \times (20/V_1)$ mg of $CaCO_3$ equivalent hardness

1000 ml of given hard water sample = $(V_2/V_1) \times 1000$ mg of $CaCO_3$ equivalent hardness

Or total hardness = $(V_2/V_1) \times 1000$ ppm

III. Titration of EDTA with boiled water sample

Procedure:

1. Take 200 ml of given hard water sample in a beaker.
2. Boil the sample for about two hours.
3. Cool the sample and filter it.
4. From the filtrate pipette out 20 ml of the water sample and titrate with EDTA as per the previous procedure.

Observation Table

S.No.	Volume of the solution taken in the titration flask (mL)	Burette Reading		Volume of the titrant used (Final- Initial reading)(mL)
		Initial Reading	Final Reading	
1.				
2.				
3.				

Let the concordant volume be V_3 mL

Calculations

Concordant volume of EDTA consumed = V_3 mL

For 20 ml of boiled hard water sample V_3 mL of EDTA is used

For 1 ml of boiled hard water sample $V_3/20$ mL of EDTA is used

$\therefore$ 1 ml of boiled hard water sample = $(V_3/20) \times (20/V_1)$ mg of $CaCO_3$ equivalent hardness

1000 ml of given boiled water sample = $(V_3/V_1) \times 1000$ mg of $CaCO_3$ equivalent hardness

Or permanent hardness = $(V_3/V_1) \times 1000$ ppm

Now, Temporary hardness = Total hardness – Permanent hardness

Temporary hardness = $[(V_2/V_1) \times 1000 \text{ ppm}] - [(V_3/V_1) \times 1000 \text{ ppm}]$

$= [(V_2 - V_3)/ V_1] \times 1000$ ppm

RESULT: 1. Total Hardness of given water sample = ... ppm

2. Permanent Hardness = ... ppm

3. Temporary Hardness = ... ppm

PRECAUTIONS

1. The burette should be rinsed with EDTA solution.
2. Conical flask should be rinsed with distilled water.
3. Re-distilled water should be employed for preparing the EDTA solution.
4. The colour change near the end point is very slow and thus should be observed carefully.

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EXPERIMENT. 5

AIM: Determination of alkalinity in a given sample of Water.

APPARATUS: Burette, Pipette, conical flask, measuring flask.

CHEMICALS: Standard HCl solution, phenolphthalein, methyl orange.

THEORY: In water sample alkalinity may be present due to the following three ions i.e, OH^-, CO_3^{2-} and HCO_3^-

Thus on the basis of above three ions, possible combination of ions causing alkalinity of water are-

1. OH^-
2. CO_3^{2-}
3. HCO_3^-
4. OH^- and CO_3^{2-}
5. CO_3^{2-} and HCO_3^-

The presence of OH^- and HCO_3^- ions together is not possible since they combine together and form CO_3^{2-} ions.

$OH^- + HCO_3^- \rightarrow H_2O + CO_3^{2-}$

Thus, the extent of alkalinity in the given water sample may be determined by titrating the water sample with a standard acid using phenolphthalein and methyl orange indicator according to the pH of the water sample.

a) $OH^- + H^+ \rightarrow H_2O$
b) $CO_3^{2-} + H^+ \rightarrow HCO_3^-$ P
c) $HCO_3^- + H^+ \rightarrow H_2O + CO_2$ M

The volume of acid used up to phenolphthalein end point corresponds to the reaction (a) and (b) i.e. complete neutralization of OH^- ions and neutralization of CO_3^{2-} ions upto HCO_3^- stage (half neutralization)

The volume of acid used up to ~~phenolphthalein~~ methyl orange end point corresponds to the reaction (a), (b) and (c) i.e. complete neutralization of OH^-, CO_3^{2-} and HCO_3^- ions.

Thus from the respective volume of the acid used, the respective strengths of various ions can be determined.

PROCEDURE:

(a) Pipette out 20ml of water sample in to conical flask and add 2 drops of phenolphthalein indicator. Pink color appears.

(b) Rinse and fill the burette with N/10 HCl.

(c) Titrate the water sample with HCl from burette with constant shaking till the pink color just disappears.

(d) Note this reading this corresponds to phenolphthalein end point (P).

(e) Now add 2-3 drops of methyl orange indicator in the same solution and continue the titration until a sharp color change from yellow to pink takes place.

(f) Note this reading, it corresponds to methyl orange end point (M)

(g) Repeat the same procedure at least three times to get the concordant readings.

OBSERVATIONS

Indicator - phenolphthalein and methyl orange

End point - pink to colorless and yellow to ~~pink~~ Red respectively

Vol. of water sample - 20ml

S. No	Initial Volume	Volume of HCl in burette			
		Final vol. up to phenolphthalein end point	Concordant volume (P) (mL)	Final volume up to methyl orange end point	Concordant volume (M) (mL)
1.					
2.					
3					

CALCULATIONS:

(a) Phenolphthalein alkalinity in terms of $CaCO_3$ equivalents

$N_1V_1 = N_2V_2$

(HCl) (water sample)

$1/10 \times V_1 = N_2 \times 20$

$N_2 = V_1/200$

Strength in g/L in terms of $CaCO_3 = N_2 \times 50$ g/L.

(b) Methyl orange alkalinity

$N'_1V'_1 = N'_2V'_2$

(HCl (water sample)

$1/10 \times V'_1 = N'_2 \times 20$

$N'_2 = V'_1/200$

Strength in g/L in term of $CaCO_3 = N'_2 \times 50$ g/L

RESULT: Water is alkaline due to presence of --------ion/ions.

Alkalinity due to the presence of -----ion is------mg/L

PRECAUTIONS:

1. Apparatus should be thoroughly cleaned.
2. Rinse the burette with the solution to be taken in it.
3. Always read the lower meniscus in case of colorless solution.
4. There should be no burette bubble or bubble in pipette during the measurement.

Applied Chemistry Lab

BS155/156

Aim: To determine alkalinity in given sample of water.

Apparatus: Burette, pipette, beaker, Conical flask.

Theory:

$$OH^- + H^+ \longrightarrow H_2O$$
$$CO_3^{2-} + H^+ \longrightarrow HCO_3^-$$ } P
$$HCO_3^- + H^+ \longrightarrow H_2O + CO_2$$ } M

Procedure:

1. Pipette out 10mL water sample in a conical flask and add 1 drop phenolphthalein, pink colour appears.
2. Titrate water sample with N/10 HCl till pink colour just disappears.
3. Now, add 1 drop methyl Orange in same solution and continue titration till colour changes Yellow to Red.

Calculation:

For phenolphthalein

N_1V_1 (sample) $= N_2V_2$ (HCl)

$N_1 =$ ✓

For Methyl Orange

N_3V_3 (sample) $= N_4V_4$ (HCl)

$N_3 =$ ✓