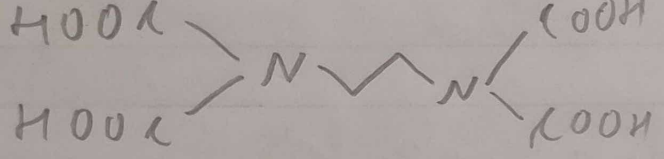
Experiment-2

Experiment- To find the temporary and permanent hardness of water sample by complexometric titration using standard EDTA solution.

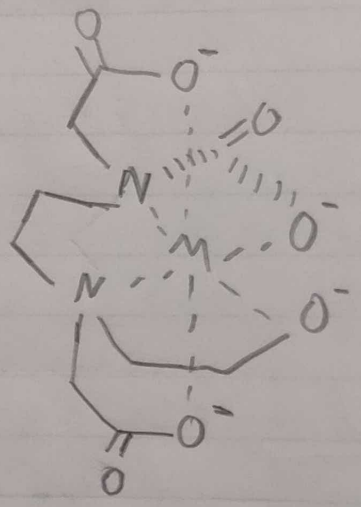
Apparatus - Pipette, Burette, beakers, conical flask, funnel, burette stand and clamp

Chemicals - water sample, EDTA, EBT, indicator, ammonium hydroxide-ammonium chloride buffer of pH 10.

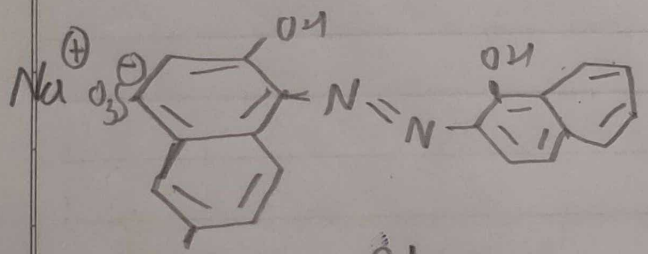
Chemical Structures-



EDTA



Metal EDTA complex.

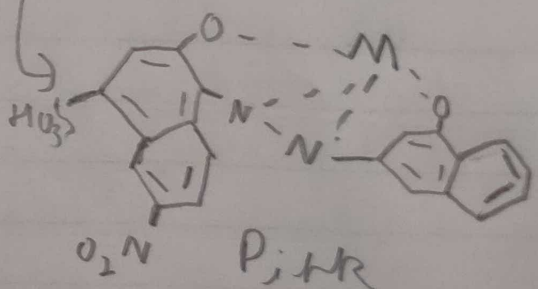


Blue

M^{2+}

pH=10

$M^{2+} = Ca^{2+} / Mg^{2+}$



Pink

Eriochrom Black T

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Experiment - To find the temporary and permanent hardness of water sample by complexometric titration using standard EDTA solution.

Apparatus - Pipette, burette, beaker, conical flask, funnel, burette stand and clamp

Chemicals - Water sample, EDTA, Eriochrome Black-T (EBT) indicator, ammonium hydroxide - ammonium chloride buffer of pH 10.

Theory - Hardness of water is due to the presence of solute salts of Ca and Mg. It is an important parameter to judge the quality of water. Determination of hardness of water EDTA titration is a very accurate method based on the fact that when EBT (blue dye) is added to the hard water (pH 10), it gives a wine red coloured unstable complex with Ca^{2+}/Mg^{2+} ion.

Equations :- $Ca^{2+}/Mg^{2+} + EBT \xrightarrow{pH\ 10} Ca^{2+}/Mg^{2+}$ EBT complex

Blue dye — wine red (unstable)

Temporary hardness in water sample is caused by bicarbonate of hardness producing ions (Ca^{2+} & Mg^{2+}) this can be removed by prolonged boiling due to decomposition of bicarbonates with the

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Chemical Equations -

$Ca^{2+}/Mg^{2+} + EBT \xrightarrow{pH\,10}$ Ca^{2+}/Mg^{2+} EBT complex

Blue Dye — Wine red (unstable complex)

Ca^{2+}/Mg^{2+} EBT complex + EDTA $\xrightarrow{pH\,10}$ Ca^{2+}/Mg^{2+} EDTA complex + EDT Blue dye

Wine red (unstable complex)

Indicator - EBT Blue dye.

End point - Wine red to blue colour.

Observation - Standardisation of EDTA sol^n vol. of 0.01 M standard hard water sol^n taken for each titration = 10 ml.

Sr No.	Burette Reading (ml)		Vol. of EDTA used (ml)
	Initial	final	
1	0	9.7	9.7
2	0	9.7	9.7
3	0	9.7	9.7
4	0	9.7	9.7

Mean volume of EDTA used

V_0 = 9.7 ml.

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evaluation of Ca & simultaneous precipitation of the respective carbonates when EDTA solution is added to the hard water (with permanent and temporary hardness), the unstable wine red complex Ca^{2+} / Mg^{2+} EBT breaks and a stable complex of Ca^{2+} / Mg^{2+} with EDTA is formed resulting in change of color of the solution from wine red to blue at the end point.

Equation:- Ca^{2+} / Mg^{2+} EBT complex + EDTA $\xrightarrow{pH\,10}$ Ca^{2+} / Mg^{2+} + EDTA + EBT
wine red (unstable) colorless complex blue dyl.

Procedure - (1) Preparation of standard hard water.

(i) Dissolve 1 gm of pure dry $CaCO_3$ in minimum quantity of dilute HCl. Evaporate the solution to dryness on a water bath to remove excess of acid. dilute the contents with distilled water to make 1L. Each ml of the sol^n contains 1 mg of $CaCO_3$ i.e hardness of this sol^n is 1000 ppm (0.01M). This sol^n is used to standardize the EDTA sol^n

(ii) (2) Standardization of EDTA

(i) Rinse the titration flask with distilled

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(ii) Determination of total hardness

Volume of hard water sample (unknown) taken for each titration = 10 ml.

Sr No	Burette Reading (ml)		Volume of EDTA used (ml)
	Initial	Final	
1	0	7.5	7.5
2	0	7.5	7.5
3	0	7.5	7.5
4	0	7.5	7.5

Mean volume of EDTA used (V_1) = 7.5 ml

(iii) Determination of permanent hardness.

Volume of boiled hard water sample taken for each titration = 10 ml.

Sr No	Burette Reading (ml)		Volume of EDTA used (ml)
	Initial	Final	
1	0	6.1	6.1
2	0	6.1	6.1
3	0	6.1	6.1
4	0	6.1	6.1

Mean volume of EDTA used (V_2) = 6.1 ml

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water and transfer 10 ml of the standard hard water sample (0.01M) into it using a pipette.

(ii) Add about 2-3 ml of ammonia/ammonium chloride buffer sol^n and 2-3 drops of the EBT indicator. The color of sol^n becomes wine red.

(iii) Titrate the hard water against the EDTA sol^n, till the wine red color changes to blue. Note readings (V_0 ml).

(iv) Repeat the procedure until concordant readings are obtained.

Determination of total hardness of water sample.

Rinse the titration flask with distilled water and transfer 10 ml of the given sample into this using pipette (follow the step 2-4 given above for standardisation of EDTA) Let the titrate value corresponding to total hardness of water sample be V_1

Determination of permanent hardness

Add 100 ml of hard water sample into 500 ml beaker, boil gently for 30-35 min. Filter the

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Calculations :- 1. Determination of molarity of EDTA solution Applying the molarity eqn :

(Standard hard water) = EDTA

$$0.01 \times 10 = M_1 \times V_0$$

$$\text{Molarity of EDTA} = M_1 = \frac{0.01 \times 10}{V_0} = \frac{0.01 \times 10}{9.7}$$

$$M_1 = 0.0103\ M$$

(ii) Determination of Total hardness

Molarity of EDTA (M_1) = Applying the molarity eqn.

Hard water = EDTA

$$M_2 \times 10 = M_1 V_1$$

$$M_2 = \frac{M_1 V_1}{10} = \frac{0.0103 \times 7.5}{10} = 0.00773\ M$$

Hardness of water sample $x = \frac{M_1 V_1}{10} \times 100$ (mol wt of $CaCO_3$) gm/L

$$= \frac{M_1 V_1}{10} \times 100 \times 1000\ mg/L = 773.1\ mg/L$$

Total hardness (y) = 773.1 ppm (mg/L).

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Solution upto the mark with de-ionised water and mix thoroughly. Rinse the titration flask with distilled water and transfer 10 cm of this (boiled water) sample into it using a pipette. Follow the step 2-4 given above (for standardisation of EDTA). Let us ~~fit into~~ the titre values corresponding to total hardness of water sample be V_2

Determination of temporary hardness -

Difference b/w the 2 values ($V_1 - V_2$) corresponds to temporary hardness

General calculations

i) Determining the molarity of EDTA soln

Applying the molarity eq.

Standard hard water $\quad = \quad$ EDTA

$0.01 \times 10 \quad = \quad M_1 \times V_0$

$$M_1 = \frac{0.01 \times 10}{V_0}$$

ii) Determination of total hardness

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(iii) Determination of permanent hardness

Apply the molarity eq.
(Boiled Hard water) = EDTA

$$M_3 \times V_1 = M_1 \times V_2$$

$$M_3 = \frac{M_1 V_2}{10} = \frac{0.0103 \times 6.1}{10} = 0.006283\ M$$

Permanent hardness of water sample (z)

$$\frac{M_1 V_2}{10} \times 100 \text{ (mol wt of } CaCO_3\text{) gm/L}$$

$$= \frac{M_1 V_2}{10} \times 100 \times 1000 = 628.3$$

Total hardness (z) = 636.3 ppm (mg/L)

(iv) Temporary hardness = y − z = 136.8 ppm

Result → Total hardness (y) = 773.1 ppm

Permanent hardness (z) = 628.3 ppm

Temporary hardness (y−z) = 144.8 ppm

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Molarity of EDTA = M_1

Apply molarity eq Hard EDTA

$M_2 \times 10 = M_1 \times V_1$

Molarity of hard water $M_2 = \frac{M_1 \times V_1}{10}$

Hardness of water sample, y = molarity $\times 100$

Molecular weight of $CaCO_3$

Hardness of water sample $y = M_1 V_1 / 10 \times 100$ (M.W of $CaCO_3$) gm/L

$= (M_1 V_1) / 10 \times 100$ (M wt of $Ca(O_3)$ $\times 1000$ mg/L

Total hardness = y ppm (mg/L)

Determination of permanent hardness:

Hard water - EDTA

$M_3 \times V_1 = M_1 V_2$ (M_3 = molarity due to permanent hardness)

Molarity of hard water = $\frac{M_1 \times V_2}{V_1} = \frac{M_2 \times V_2}{100}$

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Permanent hardness = Z ppm (mg/L)

(v) Temporary Hardness: Total - permanent = (y - z) ppm

Result - Total hardness = y ppm = 773.1 ppm

Permanent = Z ppm = 628.3 ppm

Temporary hardness = y - z = 144.8 ppm

Precautions - 1. Wash titration flask with distilled water each time, before transferring hard/sample water sol^n.

Expected CLOs & Daily life application - Determination of hardness of water can help in industrial settings, where water hardness is monitored to avoid scaling precipitation in boilers, cooling towers and other equipment. High calcium levels also caused irritation in eyes. Hence, quantification of ions in potable water is necessary.

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