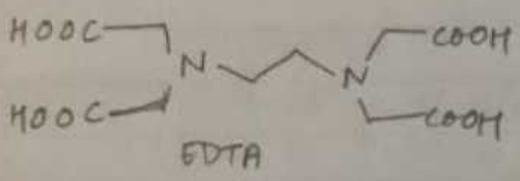
Experiment 2

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

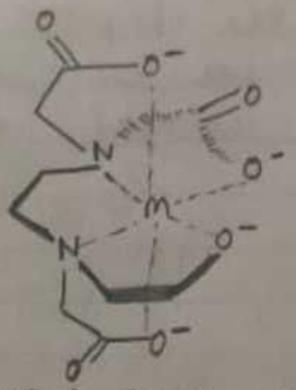
Apparatus :- Pipette, burette, beakers, conical flask, funnel, burette stand and clamp.

Chemicals :- water sample, ethylenediaminetetraacetic acid (EDTA) Erichrome black-T (EBT) indicator, ammonium hydroxide - ammonium chloride buffer of pH 10.

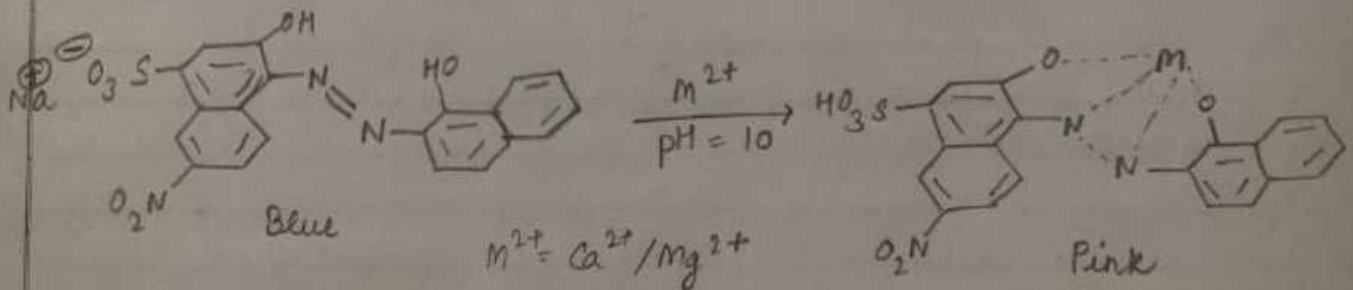
Chemical structures



EDTA



Metal EDTA complex.



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

Erichrome Black T

Chemical Equations

Ca^{2+}/Mg^{2+} + EBT (Blue dye) $\xrightarrow{pH\,10}$ Ca^{2+}/Mg^{2+} EBT complex (wine red) (unstable complex)

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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 samples, ethylenediaminetetraacetic (EDTA) Erichrome 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 by EDTA titration is a very accurate method based on the fact that when Erichrome black-T (EBT, blue dye) is added to the hard water (at about pH 10), it gives a wine red coloured unstable complex with Ca^{2+}/Mg^{2+} ion.

Equation : 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 bicarbonates of Hardness producing ions (Ca^{2+} and Mg^{2+}) this can be removed by prolonged boiling due to decomposition of bicarbonates with the evaluation of CO_2 and simultaneous precipitation of the respective carbonates, when EDTA solution is added to the hard water (with permanent and temporary

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Ca^{2+}/Mg^{2+} EBT complex + EDTA $\xrightarrow{pH\ 10}$ Ca^{2+}/Mg^{2+} EDTA complex.
wine red (unstable complex) Colourless + EBT blue dye.

Indicator :- EBT (Eriochrome Black T) Blue dye.
End point :- wine red to blue colour

OBSERVATION : i) Standardisation of EDTA soln.
Volume of 0.01 M standard hard water solution taken for each titration = 10 ml

Sr. No.	Burette Reading (ml)		Vol. of EDTA used (ml)
	Initial	Final	
1	0	9.9	9.9
2	0	9.9	9.9
3	0	9.9	9.9
4	0	9.9	9.9

Mean volume of EDTA used (V_0) = 9.9 ml.

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	7.7	7.7
2	0	7.7	7.7
3	0	7.7	7.7
4	0	7.7	7.7

Mean volume of EDTA used (V_1) = 7.7 (ml)

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hardness), the unstable wine red complex of 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 colour (unstable) — Colorless complex — blue dye

PROCEDURE: i) Preparation of Standard Hard water:
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 solution contains 1 mg of $CaCO_3$ i.e hardness of this solution is 1000 ppm (0.01M). This solution is used to standardize the EDTA solution.

ii) Standardization of EDTA

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

b.) Add about 2-3 ml of ammonia/ammonium chloride buffer solution and 2-3 drops of the EBT indicator. The color of solution becomes wine red.

c.) Titrate the hard water against the EDTA solution, till the wine red color changes to blue. Note readings (V_0 ml)

d.) Repeat the procedure until three concordant reading are obtained.

Determination of total hardness of water sample.
Rinse the titration flask with distilled water and

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iii) Determination of permanent hardness.

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

Sr. No	Burette reading (ml) Initial	Final	Vol. of EDTA used (ml)
1	0	6.3	6.3
2	0	6.3	6.3
3	0	6.3	6.3
4	0	6.3	6.3

Mean Volume of EDTA used (V_2) = 6.3 ml.

Calculations :- i) Determination of molarity of EDTA solution

Applying the molarity eq.

(Standard Hard water) = (EDTA)

$0.01 \times 10 = M_1 \times V_0$

Molarity of the EDTA $M_1 = \frac{0.01 \times 10}{V_0} \Rightarrow \frac{0.01 \times 10}{9.9}$

$M_1 = 0.0101\ M$

ii) Determination of total hardness.

Molarity of EDTA (M_1) =

Applying the molarity equation

(Hard water) = (EDTA)

$M_2 \times 10 = M_1 \times V_1$

$M_2 = \frac{M_1 V_1}{10} \Rightarrow \frac{0.0101 \times 7.7}{10} \rightarrow 0.007777\ M$

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

$= \frac{M_1 V_1}{10} \times 100 \times 1000$ mg/L = 777.7 mg/L

Total hardness (Y) = 777.7 ppm (mg/L)

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transfer 10ml of the give sample into this using pipette (Follow the steps 2-4 given above for standardisation of EDTA) Let the titre value corresponding to total hardness of water sample be V_1

Determination of permanent hardness

Make 100ml of hard water sample into 500ml beakers boil gently for 30-35 min. Filter the solution into a 100ml measuring flask. Make up the solution upto the mark with de-ionised water and mix thoroughly. Rinse the titration flask with distilled water and transfer 10ml of this (boiled water) sample into it using a pipette. Follow the step 2-4 as given above (for standardisation of EDTA) Let us titre values corresponding to total hardness of water sample be V_2.

Determination of temporary hardness.

Difference between the two values $(V_1 - V_2)$ corresponds to temporary hardness.

General calculations

i) Determining the molarity of EDTA soln.

Applying the molarity equation

(Standard Hard water) (EDTA)

$0.01 \times 10 = M_1 \times V_0$

$M_1 = (0.01 \times 10) / V_0$

ii) Determination of total hardness

Molarity of EDTA = M_1

Applying molarity equation (Hard) (EDTA)

$M_2 \times 10 = M_1 \times V_1$

Molarity of hard water, $M_2 = (M_1 \times V_1) / 10$

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

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iii) Determination of permanent hardness.

Apply the molarity equation

(Boiled Hard water) (EDTA)

$M_3 \times V_1 = M_1 \times V_2$

M_3 = (Molarity of hard water due to permanent hardness)

$$M_3 = \frac{M_1 V_2}{10} = \frac{0.0101 \times 6.3}{10} = 0.006363\ M$$

Permanent hardness of water sample (Z) =

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

$$= \frac{M_1 V_2}{10} \times 100 \times 1000\ mg/L = 636.3.$$

Total hardness (Z) = 636.3 ppm (mg/L).

iv) Temporary hardness = (Y-Z) ppm ⇒ 141.4 ppm.

Results → Total hardness (Y) = 777.7 ppm.

Permanent hardness (Z) = 636.3 ppm

Temporary hardness (Y-Z)

= 141.4 ppm.

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molecular weight of $CaCO_3$.

Hardness of water sample, $Y = M_1V_1/10 \times 100$ (m.w of $CaCO_3$) gm/L

$= (M_1V_1)/10 \times 100$ (MW of $CaCO_3$) $\times$ 1000 mg/L

Total hardness = Y ppm (mg/L)

Determination of permanent hardness:

(Hard Water) EDTA

$M_2 \times V_1 = M_1 \times V_2$ (M_2 = Molarity due to perm-anent hardness)

Molarity of hard water = $\frac{M_1 \times V_2}{V_1} = \frac{M_1 \times V_2}{10}$

Permanent hardness of water sample, Z = Molarity x M.W of $CaCO_3$

Permanent hardness of water sample.

$Z = (M_1V_2)/10 \times 100$ (Mol. Wt of $CaCO_3$) gm/L

$= (M_1V_2)/10 \times 100 \times 1000$ mg/L

Permanent hardness = Z ppm (mg/L)

(iv) Temporary Hardness: Total - permanent = (Y-Z) ppm.

Results: Total Hardness = Y ppm = 777.7 ppm

Permanent = Z ppm = 636.3 ppm.

Temporary hardness = (Y-Z) ppm = 141.4 ppm.

PRECAUTIONS → 1.) wash Titration flask with distilled water each time, before transferring hard/sample water soln.

Expected CLOs/Daily life Application → Determination of hardness of water can help in industrial settings, where water hardness is monitored to avoid costly breakdown in boilers, cooling towers and other equipment. High calcium levels also cause irritation in eyes. Hence, qualification of ions in potable water is necessary

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