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Page 1

Objective: To determine total hardness, temporary and permanent hardness of water by EDTA complexometric titration method.

Apparatus and reagents required:

i) Burette (50 ml)
ii) pipette (20 ml)
iii) conical flask
iv) Volumetric flask (100 ml)
v) $CaCO_3$ (1 ppm)
vi) EDTA solution
vii) Ammonia buffer solution
viii) Eriochrome Black-T indicator

Theory :-

Hardness in water is that characteristic which prevents the lathering of soap. Hardness is categorised as temporary or carbonate hardness and permanent hardness. Temporary hardness is caused by carbonates or bicarbonates of calcium and magnesium. Permanent hardness is caused due to chlorides, ~~sulphates~~ sulphates of calcium, magnesium, ferrous, ferric etc.

Hardness in water is generally expressed in terms of $CaCO_3$ equivalent because it is most insoluble salt and molecular weight 100 g makes the calculations easy.

It is expressed in Parts per million (PPM) or mg/L.

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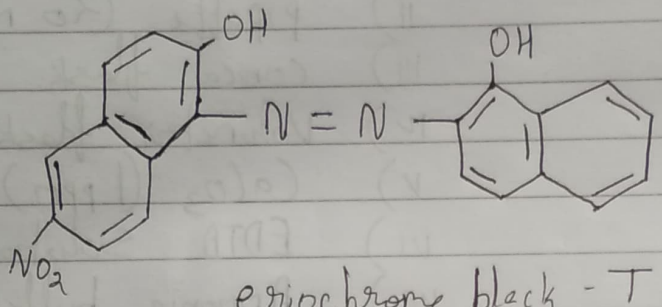
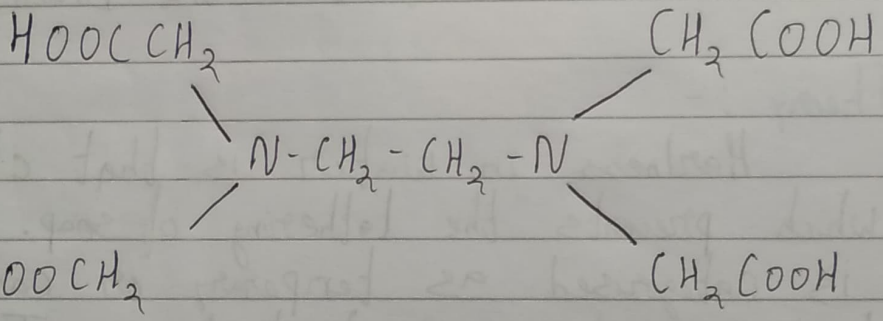
The complexometric method is supposed to be a most accurate method for determining hardness of water. In this method Ethylene diamine ~~tetraacetat~~ tetraacetic acid (EDTA) and Eriochrome Black-T (EBT) are used where EDTA is used as a complexing agent.

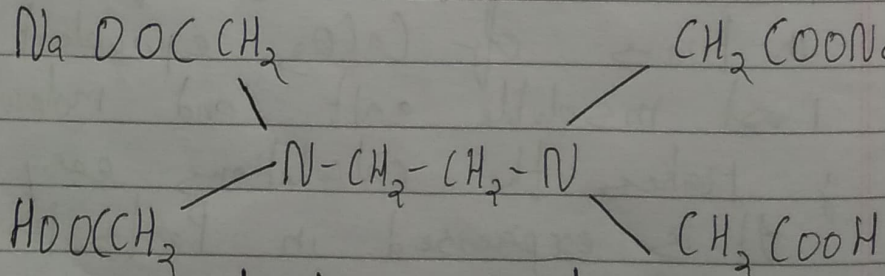


eriochrome black-T



Ethylene diamine tetraacetic acid

As it is not very soluble in water and hence EDTA in the form of its disodium salt is normally used in all complexometric titrations.



disodium salt of EDTA

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EBT indicator is effective at pH of about 10. When EBT is added to hard water buffered to pH of about 10 by employing NH_4OH buffer, a wine red unstable complex is formed. Thus

$[Ca^{2+}/Mg^{2+}]$ + EBT $\xrightarrow{pH\ 10}$ [M - EBT] complex
hardness wine red

So, initially a wine red colour is obtained. During the course of titration against EDTA solution, EDTA combines with M^{2+} $[Ca^{2+}/Mg^{2+}]$ ions from stable complex, M-EDTA and releasing free EBT, which instantaneously combines with M^{2+} ions still present in the solution, thereby wine red colour is retained. Thus,

[M-EBT] complex + EDTA $\xrightarrow{Titration}$ [M-EDTA] Complex + EBT (blue)

M^{2+} + EBT → [M-EBT] Complex
$[Ca^{2+}/Mg^{2+}]$ ~~still present~~ (blue) wine red
still present

However when nearly all M^{2+} (Ca^{2+}/Mg^{2+}) ions have been used, next drop of EDTA will displace the EBT indicator and the wine red colour changes to blue colour.

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Thus end point is marked by change of colour from wine red to blue. Thus this is the equivalence point.

[M·EBT] complex + EDTA → [M-EDTA] complex + EBT (blue)

Procedure:

1) Preparation of standard hard water: 1.0 g of pure dry $CaCO_3$ is dissolved in ~~dissolved~~ dilute HCl of minimum quantity and then evaporated to dryness on a water bath. The residue is dissolved in distilled water to make 1 litre solution. Each ml of this solution thus contains 1mg of $CaCO_3$ equ hardness.

2) Preparation of EDTA solution: 3.78 g of disodium salt of EDTA is dissolved in distilled water and made upto 1 litre.

3) Preparation of EBT indicator: 0.5 g of EBT is dissolved in 100 ml of ethanol.

4) Buffer solution: Ammonia buffer of pH ~ 10 is obtained by mixing 70 g of NH_4Cl and 565 ml of ammonia solution and then dilution upto 1 litre.

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Step I Standardization of EDTA solution :-

(i) Rinse the burette with EDTA solution.
(ii) ~~20 ml of~~ 20 ml of standard hard water is pipetted out in a conical flask

iii) 3-4 ml of ammonia buffer and 2 drops of EBT indicator is added.

iv) The colour of the solution turns into wine red.

v) The solution is then titrated ~~against~~ against standard EDTA solution until the colour changes from wine red to distinct blue.
This is the equivalence point. Let the EDTA used be V_1 ml.

Step II Determination of total hardness of sample water:

- 20 ml of sample hard water is titrated against EDTA solution as per the process discussed above.
Let the volume consumed be V_2 ml.

Step III Determination of permanent hardness.
- 20 ml of boiled water sample and titrated it against EDTA as per procedure discussed above. Let the volume be V_3 ml.

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Observation: Volume of water sample taken = 20 ml

Table-1 : SHW vs EDTA

S.No	Vol of SHW (ml)	Initial Reading	Final Reading	Vol. of EDTA (ml)
1	20	0.0	14.5	14.5
2	20	14.5	28.9	14.4
3	20	28.9	43.3	14.4

$\therefore V_1 = 14.4$ ml

Table-2 : Hard water Sample vs EDTA

S.No	Vol of Sample HW (ml)	Initial Reading	Final Reading	Vol of EDTA (ml)
1	↑	15	17.5	2.5
2	20	17.5	19.8	2.3
3	↓	19.8	22.1	2.3

$V_2 = 2.3$ ml

Table 3 : Boiled water sample vs EDTA

S.No	Vol of Boiled HW (ml)	Initial Reading	Final Reading	Vol of EDTA (ml)
1	↑	22.1	23.2	1.1
2	20	23.2	24.2	1.0
3	↓	24.2	25.2	1.0

$V_3 = 1.0$ ml

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Calculations:

Standardization of EDTA:

1 ml of SHW = 1 mg $CaCO_3$
14.4 ml of EDTA = 20 ml of SHW
14.4 ml of EDTA = 20 mg of $CaCO_3$ eqv
1 ml of EDTA = $\frac{20}{14.4}$ mg of $CaCO_3$ eqv

- - - (1)

Total hardness of water sample:

20 ml of Sample hard water : 2.3 ml of EDTA

20 ml of sample hard water = $\left(\frac{2.3 \times 20}{14.4}\right)$ mg of $CaCO_3$ eq

1 ml of sample hard water = $\left(\frac{2.3 \times 20}{14.4 \times 20}\right)$ mg of $CaCO_3$ eq

1000 ml of sample hard water = $\left(\frac{2.3}{14.4} \times 1000\right)$ mg of $CaCO_3$ eq

= 159.72 mg of $CaCO_3$ eq

Hence Total hardness = 159.72 mg of $CaCO_3$ eqv
= 159.73 ppm

Similarly Permanent hardness
= ~~Total hardness~~ = $\frac{1}{14.4} \times 1000$

= 69.44 ppm

Permanent hardness = 69.44 ppm

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Temporary hardness = Total hardness - permanent hardness

= (159.72 - 69.44) ppm

= 90.28 ppm

Conclusion:

The total hardness of the given water sample was found out to be 159.72 ppm.

The permanent hardness was found out to be 69.44 ppm.

The temporary hardness was found out to be 90.28 ppm.

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