

## Experiment (9)

### Oxidation-Reduction Reactions

- ❖ **Oxidation-reduction reactions:** are all the reactions involving change in oxidation number or transfer of electrons among the reacting substances.
- ❖ **Reduction agent:** is one that loses electrons and is oxidized to a higher valiancy condition.

#### Examples of this reaction:

As an instance, oxygen from the atmosphere is reduced during wood combustion, acquiring the electrons of carbon which is being oxidized. This reaction is a faster process.



The iron reacts to the oxygen in the air and produces rust. This reaction is a slow process.



The reaction may happen comparatively slowly, as with rust, or faster, as with fire.

Moreover, Ferrous, reaction sodium thiosulphate and oxalate ion.



❖ **Oxidation agent:** is one that gains electrons and is reduced to a lower valiancy condition.

Ex: Potassium dichromate, potassium permanganate.

The permanganate ion in acid solution is a strong oxidizing agent, because the standard potential in acid solution  $E^0$  has been calculated to be 1.51V.



### **Preparation of solutions:**

#### **A: Preparation of 0.1N potassium permanganate.**

- 1- Weigh out about 3.2gm of  $\text{KMnO}_4$  on a watch glass.
- 2- Transfer it to a 1500mL beaker and add 1L distilled water, cover the beaker with a clock glass then heat the solution to boiling.
- 3- Boil gently for 1hr and allow the solution to cool to the laboratory temperature.
- 4- Filter the solution through a funnel containing a plug of purified glass wool, collect the filtrate in a vessel.
- 5- The solution should be stored in a clean, glass stoppered bottle of a dark-brown-colored glass and kept in the dark.

#### **B: Preparation of 0.1N sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ).**

- 1- Weigh out about 1.675gm of  $\text{Na}_2\text{C}_2\text{O}_4$ .
- 2- dissolved it in distilled water in a 250mL volumetric flask.

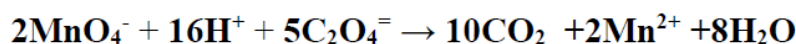
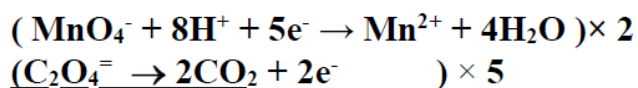
$$N = \frac{\text{Wt}}{\text{eq.wt}} * \frac{1000}{V(\text{mL})}$$

$$0.1 = \frac{\text{Wt}}{67} * \frac{1000}{250} \implies \text{Wt} = \mathbf{1.675 \text{ gm}}$$

### Standardization of permanganate solution with oxalate ion:

- 1-Pipet 10mL of 0.1N of sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ) in a 250mL conical flask.
- 2-Add by a graduated cylinder 5mL of  $\text{H}_2\text{SO}_4$  [1:8].
- 3-Add some drops of potassium permanganate and heat to 55-60°C and complete the titration by adding permanganate solution until a faint pink color persists for 30sec.
- 4-Calculate the normality of permanganate ion.

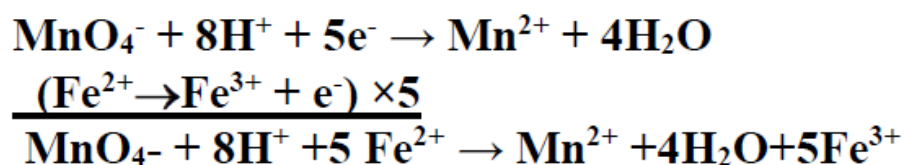
$$N_1 \times V_1 = N_2 \times V_2$$
$$\text{KMnO}_4 \quad \text{KMnO}_4 = \text{Na}_2\text{C}_2\text{O}_4 \quad \text{Na}_2\text{C}_2\text{O}_4$$



### Determination the concentration of ferrous ion:

- 1-Pipet 10mL of ferrous ion solution in a 250mL conical flask.
- 2-Add 5mL of Sulfuric acid  $\text{H}_2\text{SO}_4$ [1:8].
- 3-Titrate the solution with standard (0.1N)  $\text{KMnO}_4$  until the pink color persists.
- 4-Repeat the titration and then calculate the normality of ferrous ion

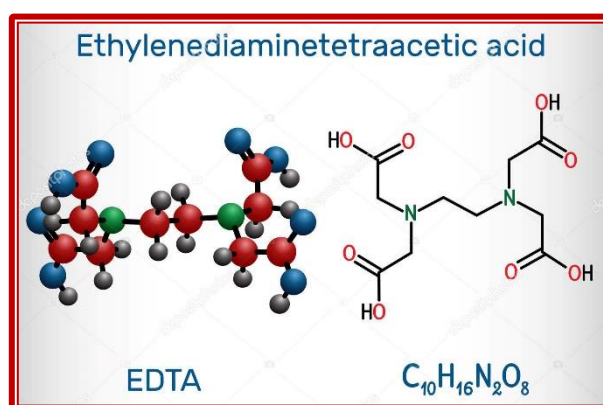
$$V_{\text{KMnO}_4} \times N_{\text{KMnO}_4} = \frac{\text{Wt Fe}^{2+}}{\frac{55.85}{1000}}$$



## Experiment (10)

### Complexometric titration

This kind of titration is called complexometric because it involved formation of complex especially by using the compound EDTA (Ethylene Diamine Tetra Acetic acid) which forms a stable complex with metal ions especially with calcium and magnesium ions. In addition, the user's indicator with EDTA have also capable to form a complex with the same ions ( $Mg^{2+}$  and  $Ca^{2+}$ ) but less stable than the complex formed between the metal ions and EDTA.



### Hardness of Water

The total hardness of water is generally due to dissolved calcium and magnesium salts. Water hardness was defined in terms of the capacity of cations in the water to replace the sodium and potassium ions in soaps and form sparingly soluble products.

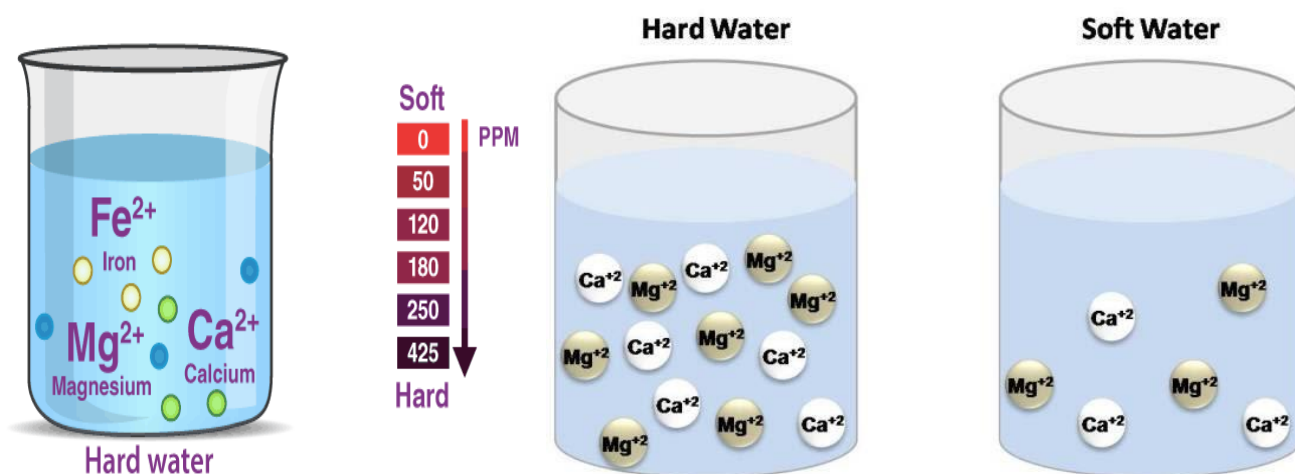
**There are two types of water hardness, temporary and permanent.**

- 1- **Temporary hardness** is due to the bicarbonate ion,  $HCO_3^-$ , (sodium and magnesium bicarbonate) being present in the water. This type of hardness can be removed by boiling the water to expel the  $CO_2$ .

2- **Permanent hardness** is due to the presence of the ions  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{SO}_4^{2-}$ .

This type of hardness cannot be eliminated by boiling.

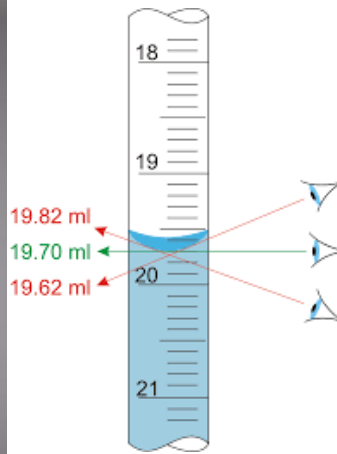
Hardness is now expressed in terms of the concentration of calcium carbonate that is equivalent to the total concentration of all the multivalent cations in the sample. The determination of hardness is a useful analytical test that provides a measure of water quality for household and industries uses.



### Determination of total hardness (permanent and temporary) of water

#### A: Determination of Total Hardness of Water.

- 1-Prepare 250mL of 0.02N of EDTA solution.
- 2-Clean the burette and rinse with EDTA solution.
- 3-Pipet 10mL of hardness water and add 2mL of ammonia buffer solution (PH=10) [prepared from ammonium chloride +conc. ammonia].
- 4-Add 3-4 drops of Eriochrome black-T indicator and titrate with standard EDTA solution until the color changes from wine red to pure blue color.
- 5- Repeat the titration a few times and calculate the total hardness (VA) of the sample of water ( $\text{Mg}^{2+} + \text{Ca}^{2+}$ ).



### Calculations

$V_{EDTA} \equiv$  Volume of EDTA which equivalent to  $(Mg^{2+} + Ca^{2+}) \Rightarrow V \square_A$

$$N_1 \times V_1 = N_2 \times V_2$$

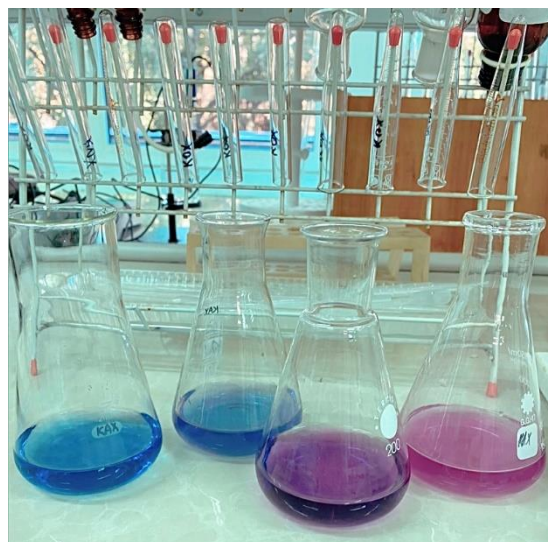
$\downarrow$       $\downarrow$       $\downarrow$       $\downarrow$   
 EDTA   EDTA   Sample(?)   Sample  
 (0.02N)

Total hardness (ppm) =  $N \times eq.wt \times 1000$   
 (Concentration of  $Mg^{2+} + Ca^{2+}$ )    $CaCO_3$

Or Total hardness (ppm) =  $(V_{EDTA}/V_{solution}) \times 2000$   
 (Concentration of  $Mg^{2+} + Ca^{2+}$ )

## B: Determination of the Permanent Hardness

- 1-Pipet 10mL of the hardness water and add 2mL of sodium hydroxide solution NaOH (4N / (PH=12)) which is sufficiently high to precipitate the magnesium quantitatively as the hydroxide.
- 2-Add some crystals of Murexide indicator.
- 3-Stirr the solution and titrate it with standard EDTA solution until the color changes from pink to a purple.
- 4-Repeat the titration a few times and calculate the total hardness (VB), which is the volume of EDTA equivalent to  $\text{Ca}^{2+}$  only.



### Calculations

$V_{\text{EDTA}}$  = Volume of EDTA which equivalent to  $\text{Ca}^{2+}$  only  $\Rightarrow V_{\square B}$

$$\begin{array}{cccc} N_1 \times V_1 & = & N_2 \times V_2 \\ \downarrow & & \downarrow & \downarrow \end{array}$$

EDTA   EDTA   Sample(?)   Sample  
(0.02N)

Permanent hardness (ppm) =  $N \times \text{eq.wt} \times 1000$

(Concentration of  $\text{Ca}^{2+}$ )       $\text{CaCO}_3$

Or Permanent hardness (ppm) =  $(V_{\text{EDTA}}/V_{\text{solution}}) \times 2000$   
(Concentration of  $\text{Ca}^{2+}$ )

### C: Determination of Temporary Hardness

Calculate the temporary hardness of water by subtracting the permanent hardness from the total hardness.

$$\text{Temporary hardness (Mg}^{2+}\text{)} = \text{Total hardness (Mg}^{2+} + \text{Ca}^{2+}\text{)} - \text{Permanent hardness (Ca}^{2+}\text{)} = V_A - V_B$$

$$\begin{array}{ccccccc} N_1 \times (V_A - V_B) & = & N_2 \times V_2 \\ \downarrow & & \downarrow & & \downarrow & & \downarrow \\ \text{EDTA} & & \text{EDTA} & & \text{Sample(?)} & & \text{Sample} \\ (0.02\text{N}) & & & & & & \end{array}$$

$$\text{Temporary hardness (ppm) (Concentration of Mg}^{2+}\text{)} = N \times \text{eq.wt} \times 1000$$

$\text{CaCO}_3$

Or  $\text{Temporary hardness (ppm) (Concentration of Mg}^{2+}\text{)} = (V_{\text{EDTA}}/V_{\text{solution}}) \times 2000$