**BATNA2 University**
**Faculty of Natural and Life Sciences (L1)**
**Academic year 2023-2024**

# Lab Practical 2 (S2): Redox titration of $Fe^{2+}$ ($FeSO_4$) using $KMnO_4$ (Manganimetry titration)

## ❖ Objectives

At the end of this lab practical, the student will be able to:

- **Understand** the concept of the oxidation-reduction titration.

**- Carry out** a Redox Titration of $FeSO_4$ solution with $KMnO_4$ solution.

- **Identify** the Equivalence point.

-**Calculate** the unknown $FeSO_4$ concentration.

## ❖ Principle (primary objective)

The principle of this experiment is to determine the mass of $Fe^{2+}$ ions in the $FeSO_4$ solution.

## I- Theoretical part

### I-1-Definitions

➢ **Oxidation state (O.S)**

Oxidation number or oxidation state (O.S): Usually a positive, zero, or negative number (an integer).

The oxidation state (O.S) of an element corresponds to the number of electrons, that an atom loses, gains, or appears to use when joining with other atoms in compounds.

- **An atom of a free element** has an oxidation number of **0**.

  **Example**: **O.S of each Cl** atom in $Cl_2$ = **0**.

- **A monatomic ion** has an oxidation number equal to its charge.

  **Example: O.S ($Cu^{2+}$) = +2, O.S ($Br^-$) = -1.**

- **Hydrogen** generally has an oxidation state of **+1** in most compounds.

  **Example: O.S of Hydrogen** in the molecule of HCl = **+1.**

- **Oxygen** generally has an oxidation state of **-2** in compounds.

  **Example: O.S of Oxygen** in the molecule of $H_2O$ = **-2.**

- **Neutral compounds** have a total oxidation state of zero **(Σ O.S = 0),** whereas **polyatomic ions** have a total oxidation state equal to their charge **(Σ O.S = charge).**

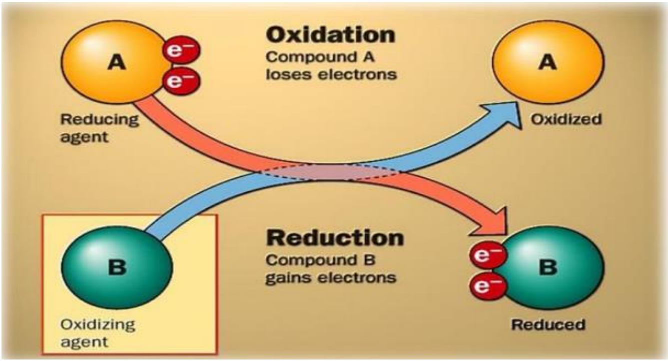
  **Example:** O.S of **$KMnO_4$ =** O.S **(K)** + O.S **(Mn)** + **[4 x** O.S **(O)] = 0.**

  O.S of **$SO_4^{2-}$** = O.S **(S)** + **[4 x** O.S **(O)] = -2.**

**Note:** To determine the oxidation state of the Mn atom in the neutral compound $KMNO_4$.

O.S **(Mn)** = O.S of **$KMnO_4$** – O.S **(K) - [4 x** O.S **(O)] = 0 - (+1) -[4 x (-2)]** = **+7.**

- **Oxidation**: The loss of one or more electrons during a reaction by a molecule, atom, or ion.
  - The oxidation number (O.S) of an element **increases** when it undergoes oxidation.
- **Reduction:** The gain of one or more electrons during a reaction by a molecule, atom, or ion.
  - The oxidation number (O.S) of an element **decreases** when it undergoes reduction.
- **Oxidizing agent:** Is one, which accepts electrons in reduction reaction.
- **Reducing agent:** Is one, which loses the electrons in the oxidation reaction.




- **Redox reaction:** Is a type of titration that examines the oxidation and reduction of certain chemical species.

- When there is an atom that donates electrons, there is always an atom that accepts electrons
- Electron transfer happens from one atom to another
- Redox titration is based on the redox reaction (oxidation-reduction) between the **analyte** and **titrant.**

- **The redox potential:** Is an empirical measurement stated in volts and symbolized as ***E*.**

This measurement is used for redox couples to anticipate the reactivity of chemical species towards each other. The standard potential $E°$ is conventionally measured relative to the water/hydrogen couple **($H^{+}/H_2$),** which has a potential of **zero**.

In general, the half-equation with the **more positive redox potential** will be **the reduction** reaction and the **other** will be **the oxidation**.

**Example:** $F_2(g) + 2e^- \rightleftharpoons 2F^- \quad E^o = +2.87\ V$

$Cl_2(g) + 2e^- \rightleftharpoons 2Cl^- \quad E^o = +1.36\ V$

$+2.87 > +1.36$

⟹ $F_2(g)$ will be reduced (reduction reaction) and $Cl^-$ oxidized (oxidation reaction).

## I-2- Titration reaction

To determine the mass of $Fe^{2+}$ ions in the $FeSO_4$ solution, we used a colorimetric titration.

The $FeSO_4$ solution serves as the **analyte,** and the $KMnO_4$ solution is used as the **titrant.**

- **The two half-reactions**
  - **Oxidation reaction**:

$$\textbf{(Reducing agent) } Fe^{2+}_{(aq)} \longrightarrow Fe^{3+}_{(aq)} + 1\,e^{-}$$

  - **Reduction reaction:**

$$\textbf{(Oxidizing agent) } MnO_4^{-}{}_{(aq)} + 8H^{+} + 5e^{-} \longrightarrow Mn^{2+}{}_{(aq)} + 4H_2O$$

**The reduction reaction was obtained after the following procedures:**

1- Balancing oxygens: $MnO_4^{-}{}_{(aq)} \longrightarrow Mn^{2+}{}_{(aq)} + 4H_2O$
2- Balancing hydrogens: $MnO_4^{-}{}_{(aq)} + 8H^{+} \longrightarrow Mn^{2+}{}_{(aq)} + 4H_2O$
3- Balancing electrons: $MnO_4^{-}{}_{(aq)} + 8H^{+} + 5e^{-} \longrightarrow Mn^{2+}{}_{(aq)} + 4H_2O$

O.S of $MnO_4^{-}$ = -1= O.S (Mn) + (4 x O.S (O)) $\longrightarrow$ **O.S of Mn** in $MnO_4^{-}$ = **+7**

  - **Final balancing of the half-reactions:**

$$[\ Fe^{2+}_{(aq)} \longrightarrow Fe^{3+}_{(aq)} + 1\,e^{-}\ ] \times 5$$
$$[\ MnO_4^{-}{}_{(aq)} + 8H^{+} + 5e^{-} \longrightarrow Mn^{2+}{}_{(aq)} + 4H_2O\ ] \times 1$$

- **The total reaction:**

$$\boxed{\mathbf{5Fe^{2+}{}_{(aq)} + MnO_4^{-}{}_{(aq)} + 8H^{+} \longrightarrow 5Fe^{3+} + Mn^{2+}{}_{(aq)} + 4H_2O}}$$

## I-3- Equivalence point

The oxidized and reduced forms of $MnO4^{-}$ have different colors. A solution of **$MnO4^{-}$** is intensely **purple**. In an **acidic solution** (**$8H^{+}$**), however, permanganate's reduced form, **$Mn2^{+}$,** is nearly **colorless.** When using $MnO4^{-}$ as a titrant, the analyte's solution remains colorless until the **equivalence point**. The first drop of excess $MnO4^{-}$ produces a **persistent pale pink color (Self-indicator).**

**Note:**

- The choice and amount of acid are important in this redox titration because, in the half-reaction of reduction **8 moles of acid** are needed for **each mole of manganate** (**$MnO_4^{-}$**).
- **Insufficient volume** of acid will mean that the solution will not be acidic enough and a **brown solid**, $MnO_2$, will be produced instead of $Mn^{2+}$ ions. The brown solid will **mask** the color change and lead to larger volumes of manganate ions used than required.

- **At the equivalence point:** $N_{Ox} \times V_{Ox} = N_{Red} \times V_{Red} \rightarrow N_{MnO_4^-} \times V_{MnO_4^-} = N_{Fe^{2+}} \times V_{Fe^{2+}}$

$N_{Red}$: unknown normality of reducing agent.

$N_{Ox}$: known normality of oxidizing agent in the burette.

- **Calculation of normality of $Fe^{2+}$ ions (<u>the analyte</u>):** $N_{Fe^{2+}} = N_{MnO_4^-} \times V_{MnO_4} / V_{Fe^{2+}}$
- **Calculation of molar concentration of $Fe^{2+}$ ions:**

$N_{Fe^{2+}} = x \times C_{Fe^{2+}}$ (**x: is the number of** $e^-$ lost by **$Fe^{2+}$ ions**) $\rightarrow N_{Fe^{2+}} = 1 \times C_{Fe^{2+}}$

$$Fe^{2+}_{(aq)} \longrightarrow Fe^{3+}_{(aq)} + 1\,e^-$$

- **Calculation of mass of $Fe^{2+}$ ions present in $FeSO_4$ Solution:**

$$n_{Fe^{2+}} = C_{Fe^{2+}} \times V_{Fe^{2+}}$$

$$m_{Fe^{2+}} = n_{Fe^{2+}} \times M_{Fe^{2+}} \qquad (M_{Fe^{2+}} = 55.85 g/mol)$$

## I- Practical part (Experimental protocol)

| Material | Products |
|---|---|
| - Erlenmeyer flask, - graduated cylinder<br>- Graduated burette, -Funnel. | - $FeSO_4$ solution, sulfuric acid $H_2SO_4$ (2N)<br>- $KMnO_4$ solution (0.05N). |

- To ensure the success of the experiment, follow these steps:
  - Measure 5 ml of $FeSO_4$ solution using a graduated cylinder.
  - Transfer the measured volume to an Erlenmeyer flask.
  - Add 2 ml of Sulfuric acid $H_2SO_4$ (2 N).
  - Fill the burette with $KMnO_4$ solution (0.05 N).
  - Titrate the $FeSO_4$ solution by adding the $KMnO_4$ solution drop by drop until you achieve the persistent pale pink color.
  - Notate the $KMnO_4$ volume at the equivalency point.

**<u>Report</u>**

**The report must contain:**

-A cover page according to the model below.

- A detailed response to the questions at the end of the Lab Practical session.

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