

First year LMD 2023-2024

Manipulation N°4

A redox titration

Redox titration

<u>1-Introduction</u>:

The titration consists in determining the normality of a reducing solution knowing that of the oxidizing solution. It is proposed to study the oxidation of the Fe^{2+} ion by the permanganate ion MnO^{4-} in an acid medium.

This assay is called manganimetry. The oxidizing properties of the permanganate ion are at the origin of manganimetry. The oxidizing form MnO^{4-} is purple, the reducing form Mn^{2+} is colorless, which makes it possible to determine the equivalent point without using colored indicators.

2- Goal of the Practical Work:

This involves determining the normality of a solution (FeSO4), using a solution of Potassium Permanganate (KMnO₄) prepared in the laboratory.

<u>3- Definitions:</u>

3-1. Oxidation: An oxidation is a reaction during which a reactant yields (loses) one or more electrons.

$$Fe \rightarrow Fe^{2+} + 2e^{-}$$

In this reaction, the Fe^{2+} ion is the oxidized form and Fe is the reduced form of the oxidation-reduction couple (or redox couple) (Fe^{2+}/Fe).

3-2. Reduction: A reduction is a reaction in which a reactant captures (takes) one or more electrons.

$$Cu^{2+} + 2e^- \rightarrow Cu$$

In this reaction, the metallic copper is the reduced form and the Cu^{2+} ion is the oxidized form of the redox couple (Cu^{2+}/Cu).

3-3. The oxidant: is the reactant capable of causing an oxidation, that is to say that it captures one or more electrons from another reactant.

$$Cu^{2+}$$
 : $Cu^{2+} + 2e^- \rightarrow Cu$

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$$Fe^{2+}$$
: $Fe \rightarrow Fe^{2+} + 2e^{-1}$

3-4. The reducer: is the reagent capable of causing a reduction, that is to say that it yields one or more electrons to another reagent.

$$Cu: Cu^{2+} + 2e^{-} \rightarrow Cu$$

Fe: Fe \rightarrow Fe²⁺ + 2e⁻

3-5. Oxidation-reduction couple: Oxidation-reduction couple or redox couple is a couple formed by an oxidant and its conjugated reducing agent.

$oxydant + ne^- \leftrightarrow reducer$

Examples of redox couple:

redox couple	oxidant	$+ n e^{-}$	\leftrightarrow	reducing
Cu ²⁺ /Cu	<i>Cu</i> ²⁺	+ 2 e ⁻	\leftrightarrow	Си
Fe ²⁺ /Fe	Fe^{2+}	+ 2 e ⁻	\leftrightarrow	Fe
H^+/H_2	2H ⁺	+ 2 e ⁻	\leftrightarrow	H_2
Ag ⁺ /Ag	Ag^+	+ 1 e ⁻	\leftrightarrow	Ag

3-6. Potassium permanganate:

Potassium permanganate (KMnO₄) is a particularly powerful oxidant. It comes in the form of purple crystals composed of potassium ions, K^+ , and permanganate ions, [MnO4]⁻ It is odorless and has a bitter taste.

In the laboratory, potassium permanganate is used to perform titrations. At equivalence, the solution indeed changes color, going from purple to pink. Potassium permanganate is also used in water treatment since it oxidizes the iron and manganese contained in groundwater. In everyday life, it can be used, in dilute solution, to eliminate black traces left by fungi between bathroom tiles.

4- Experimental part:

4-1. Dosage of oxalic acid by potassium permanganate:

Equipment: Burette, Erlenmeyer, test tube, wash bottle, KMnO₄ solution (0.1N), $H_2C_2O_4.2H_2O$ solution, Distilled water, H_2SO_4 (10%).

- 1- Rinse the material. Burette, Erlenmeyer.
- 2- Fill the burette with the Normality $KMnO_4$ solution ($N_A = 0.1N$).
- 3- Take a 250 mL Erlenmeyer flask and put 50 mL of distilled water in it.
- 4- Add ($V_B=10$ ml) solution ($H_2C_2O_4$. 2 H_2O).
- 5- Add about 5ml of H_2SO_4 at (10%), and heat to 60-70°C,



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- 6- Perform a rapid assay to estimate the equivalence volume.
- 7- Note the volume V_A of (KMnO₄) poured.
- 8- Carry out 2 dosing tests.

4-2. Determination of iron in ferrous sulphate by potassium permanganate:

Equipment: Burette, Erlenmeyer, test tube, wash bottle, KMnO₄ solution (0.1N), *FeSO*₄.7 H_2O solution, Distilled water, H₂SO₄ (10%).

- 1- Rinse the material. Burette, Erlenmeyer.
- 2- Fill the burette with the Normality $KMnO_4$ solution ($N_A = 0.1N$).
- 3- Take a 250 mL Erlenmeyer flask and put 50 mL of distilled water in it.
- 4- Add (V_B=10 ml) solution ($FeSO_4.7H_2O$).
- 5- Add about 5ml of H_2SO_4 at (10%), and heat to 60-70°C,
- 6- Perform a rapid assay to estimate the equivalence volume.

5- Results and calculations:

5-1. Dosage of oxalic acid by potassium permanganate:

- 1- Purpose of the practical work.
- 2- Write the oxidation-reduction half-reactions, specify the redox couples.
- 3- Write the overall reaction.
- 4- Note the volume (V_A) of (KMnO₄) poured and calculate the Normality (N_B) of ($H_2C_2O_4$. $2H_2O$).
- 5- Calculate the Concentration (C_B) of the solution ($H_2C_2O_4$. $2H_2O$).
- 6- What to conclude when the role of sulfuric acid H_2SO_4 ?
- 7- For what reason in heating.
- 8- Determine the limiting reagent of this reaction.
- 9- Can sulfuric acid be replaced by HCl or H₃PO₄. Explain.

5-2. Determination of iron in ferrous sulphate by potassium permanganate:

- 1- Purpose of the practical work.
- 2- Write the oxidation-reduction half-reactions, specify the redox couples.
- 3- Write the overall reaction.
- 4- Note the volume (V_A) of (KMnO₄) poured and calculate the Normality (N_B) of (FeSO₄.7H₂O).
- 5- Calculate the Concentration (C_B) of the solution (FeSO₄.7H₂O).
- 6- Determine the limiting reagent of this reaction.
- 7- Can sulfuric acid be replaced by HCl or H₃PO₄. Explain.