

Academic year 2024-2025 1st year LMD ST/SM 4th Practical work chemistry

4th Practical work: Titration by the Oxidation-Reduction Method

Case: Manganimetry

Terminology

A **redox reaction** is a chemical reaction involving **the transfer of one or more electrons**. It is **the combination** of two oxidation and reduction half-equations.

Oxidation reaction is defined as **the loss of one or more electrons**. The opposite reaction to oxidation is called **reduction**, and it is defined as **the gain of one or more electrons**.

i.e.:

A reducing agent is a chemical species that loses one or more electrons.

An oxidant agent is the chemical species that captures one or more electrons.

So, reduction is the reaction that forms a reducing agent, and oxidation is the reaction that forms an oxidizing agent.

Or, in an oxidation-reduction reaction, the reductant is oxidized and the oxidant is reduced.

 $Red_{1} \longrightarrow Ox_{1} + n_{1} e^{-}$ Oxidation reaction $Ox_{2} + n_{2} e^{-} \longrightarrow Red_{2}$ Reduction reaction $n_{2} Red_{1} + n_{1} Ox_{2} \implies n_{2} Ox_{1} + n_{1} Red_{2}$

So, to determine *the direction of a redox reaction*, we need to quantify the oxidizing and reducing *powers* of the redox couples involved. To do this, we define *the standard potential E*° of a redox couple (**Ox/Red**).

The greater the E°, the stronger the oxidant.

for this, we can follow the gamma rule.



the gamma rule

Examples:

The transition of iron "Fe" into a solution corresponds to an oxidation: Fe \longrightarrow Fe²⁺ + 2 e⁻.

The deposition of copper "Cu" from a solution of Cu^{2+} ions correspond to a reduction: $Cu^{2+} + 2e^{-} \longrightarrow Cu$.



Below are the oxidation-reduction couples and their classification in ascending order:

Major oxidizing agents:	Li⁺	Zn ²⁺	Fe ²⁺	H⁺	I_2	Fe ³⁺	O ₂	MnO4 ⁻ F	2
Corresponding reduced forms:	Li	Zn	Fe	н	I-	Fe ²⁺	H₂O	Mn 2+	F

Objective of the manipulation

The main objective of this practical work is to determine the normality of a solution of **FeSO**₄ by manganimetry titration in an acidic medium.

Principal of practical work

Manganimetry is a dosing (titration) method based on *redox reactions* involving the *permanganate ion* MnO⁴⁻, which is a chemical species capable of *capturing electrons*.

A manganimetry titration therefore involves the "colored" redox couple: (MnO^{4-}/Mn^{2+}) with a standard potential of (1.507 V).

Potassium permanganate KMnO₄ is a strong oxidant for all chemical species, their corresponding reductant can be Mn^{2+} (colorless), Mn^{3+} , or MnO_2 (brown), depending on the reaction medium, either *acidic* or *basic*. Thus, the oxidizing form MnO^{4-} is purple, the reducing form Mn^{2+} is colorless, enabling the equivalent point to be determined without the use of color indicators.

Note: If the medium **is not acidic enough**, the couple involved is **MnO⁴⁻/MnO₂**. As MnO₂ is a brown solid with low solubility, the equivalence point can no longer be determined. It is therefore necessary to acidify the medium by adding concentrated sulfuric acid (hydrochloric acid and nitric acid are not used, as the former is oxidized by the permanganate ion and the latter is also an oxidizing agent).

Operating mode

Used materials:	Used chemicals	
-Burette (25-50 mL).		
-Graduated or volumetric pipettes (10 mL)	-Potassium permanganate solution	
-Pro-pipette	KMnO ₄	
-Erlenmeyer flask (100 mL).	$-H_2SO_4$ sulfuric acid solution (20%).	
-Hot plate	-Oxalic acid ($H_2C_2O_4$, N1= 0.01N)	
-Magnetic strippe	-FeSO ₄ solution	
-Beakers		

1. Determination of normality of KMnO₄ solution:

Note: follow the instructions given in PW N°03 (acid-basic titration) for preparing a dosing device.



since it is unstable, and as it has been previously prepared, so it is preferable to determine its normality. equation is:

This part is carried out using a reducing solution of **oxalic acid** (H₂C₂O₄,2H₂O), its concentration is **0.01 N**, as fellow:

It is an oxidation-reduction reaction, and its balanced:

 Oxidation:
 $C_2O_4 \xrightarrow{--}$ $2 CO_2 + 2 e^- (X 5)$

 Reduction:
 $MnO^{4-} + 5 e^- + 8 H^+$ $Mn^{2+} + 4 H_2O (X 2)$
 $2 MnO_4^- + 16 H^+ + 5 C_2O_4^{--}$ $2 Mn^{2+} + 8 H_2O + 10 CO_2$

a) Fill burette with potassium permanganate solution (KMnO4, oxidizing agent).

Place 10 ml of oxalic acid ($H_2C_2O_4$, N1= 0.01N) and 10 ml of sulfuric acid H_2SO_4 (20%) in the Erlenmeyer flask. Heat the mixture to around **60 °C** as the reaction is slow, and heat **speed up** (catalyse) the reaction, **the color disappears rapidly.**

b) Allow a few drops of KMnO₄ to flow, shake, and wait until they **are decolorized**. Continue adding a few drops until an excess drop produces a persistent pale pink color (does not disappear).

• Note the value of the volume V_e poured (table 1). Repeat the operation three times.

Table 1:

	1 st test	2 nd test	3 rd test
V _e (mL)			
Color			

- 1. Deduce the average equivalence volume (Ve.avg)?
- 2. Calculate the normality and deduce the concentration of the KMnO₄?

2. Determination of normality of FeSO₄:

Introduce into a 100 mL Erlenmeyer flask:

- 10 mL FeSO₄ solution using a pipette.
- 10 mL 20% H₂SO₄ using graduated cylinder.

Place the Erlenmeyer flask under the burette and start dosing **drop by drop** until a pale pink color is obtained due to the addition of a single excess drop of KMnO₄. Note the value of the volume V_e poured (Table 2). Repeat the operation three times.

	1 st test	2 nd test	3 rd test
V _e (mL)			
Color			

1. Deduce the average equivalence volume (**Ve**_{.avr})?

- 2 Give the oxidation-reduction reaction involved?
- 3 Calculate the normality of the titrate solution?