

Practical Work Chemistry II

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Objectives



This lab is intended for first-year undergraduate students in science and technology. At the end of this course, the learner will be able to:

In terms of knowledge:

- Become familiar with laboratory equipment.
- Know how to carry out a series of measurements in an organized and rigorous manner.
- Know how to use the information provided in various materials to write literal and numerical expressions, as well as equations modeling the situations proposed in the problems.

In terms of practical skills:

- Know how to use the calorimeter and the different laboratory equipment to measure the various parameters required in the problem.
- Know how to perform calculations to determine different parameters (heat capacities, specific heat capacity, concentration, pH, etc.).
- Write reports with rigor and clarity.

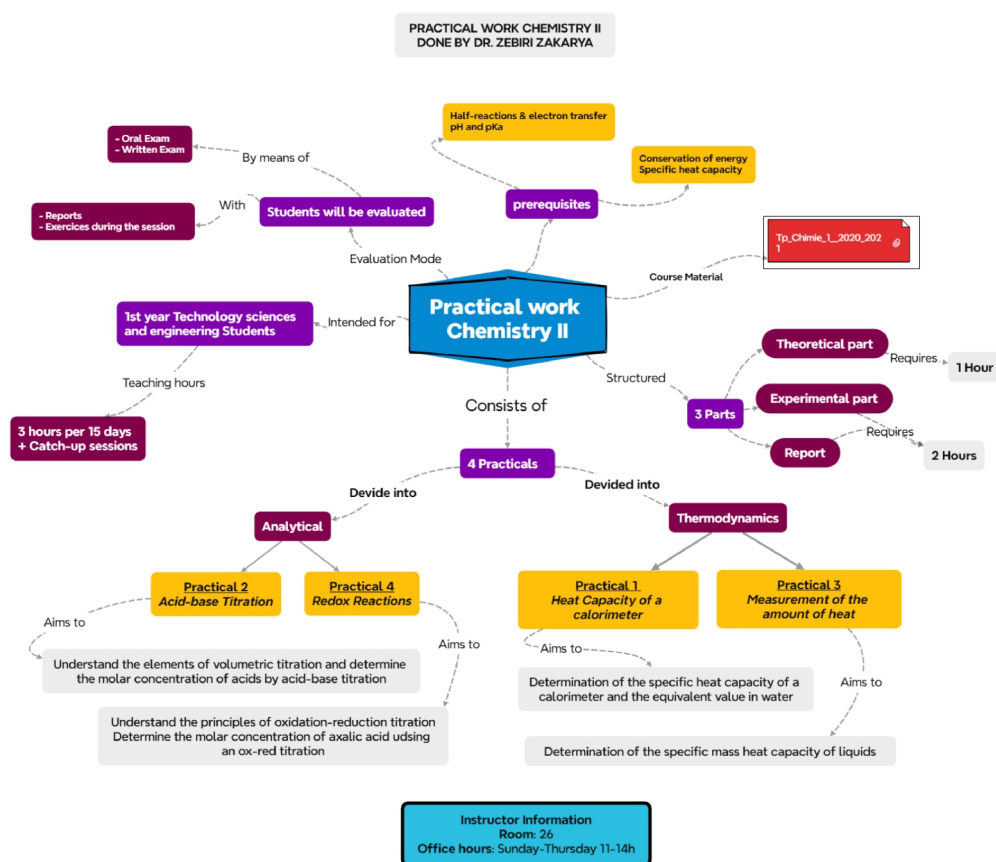
In terms of attitudes (professional behavior):

- Develop awareness of the two fundamental principles of calorimetry: the principle of equality of heat exchange (what is lost by one system is gained by another) and the principle of reverse transformations.
- Develop awareness of the principles of acid–base colorimetric titration and redox titration.

Introduction



This module introduces key experimental techniques in thermochemistry and analytical chemistry through calorimetry and acid–base titration. It covers the determination of a calorimeter’s specific heat capacity, highlighting heat transfer and energy conservation principles. It also develops skills in quantitative analysis via titration, focusing on neutralization, equivalence points, and accurate measurement.



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Mind map.

Practical N°4: Redox titration



1. Objectives

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

- Identify and interpret oxidation–reduction (redox) reactions.
- Perform a redox titration using KMnO_4 as an oxidizing agent.
- Determine the concentration of an aqueous oxalic acid solution by volumetric analysis.

2. Introduction

In nature, electrons are continuously transferred between chemical species (ions, atoms, and molecules). They are never found free because their negative charge causes them to interact with electron donors, known as **reducing agents**, and electron acceptors, known as **oxidizing agents**.

Oxidation–reduction (redox) reactions have numerous applications in chemistry, including volumetric analysis, the manufacture of rechargeable cells and alkaline batteries, and the study of corrosion processes.

Redox titration is a quantitative analytical technique used to determine the concentration of a reducing agent using a standard oxidizing solution of known concentration. In this lab work, the oxidation of oxalate ions ($\text{C}_2\text{O}_4^{2-}$) by permanganate ions (MnO_4^-) in an acidic medium will be investigated.

This titration method is known as **permanganometry**. It is based on the strong oxidizing properties of the permanganate ion. The oxidized form, MnO_4^- , is purple, whereas the reduced form, Mn^{2+} , is pale pink, nearly colorless. This distinct color change enables the equivalence point to be detected without the use of an external indicator.

3. Quiz

In a redox half-equation:

- ☐ Electrons always appear on the **left-hand side**.
- ☐ Electrons always appear on the **right-hand side**.
- ☐ Electrons always appear on the side containing the **oxidizing agent** of the redox couple.
- ☐ Electrons always appear on the side containing the **reducing agent** of the redox couple.

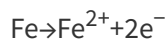
4. Theoretical Background



Definition

- **Oxidation**

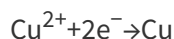
Oxidation is a reaction in which a chemical species loses one or more electrons.



In this reaction, **Fe²⁺** is the oxidized form, while **Fe** is the reduced form of the **redox couple** (**Fe²⁺/Fe**).

- **Reduction**

Reduction is a reaction in which a chemical species gains one or more electrons.



In this reaction, metallic **Cu** is the reduced form, whereas **Cu²⁺** is the oxidized form of the redox couple (**Cu²⁺/Cu**).

- **Oxidizing Agent**

An oxidizing agent (oxidant) is a species that causes oxidation by accepting one or more electrons from another species. As a result, the oxidizing agent is itself reduced.

In the previous reactions, **Cu²⁺** is the oxidizing agent.

- **Reducing Agent**

A reducing agent (reductant) is a species that causes reduction by donating one or more electrons to another species. As a result, the reducing agent is itself oxidized.

In this reaction, **Fe** is the reducing agent.

- **Redox Couple**

A redox couple consists of an oxidizing agent and its corresponding reduced form (conjugate reducing agent). It is conventionally written as:

Oxidized form/Reduced form

Examples include:

- **Fe²⁺/Fe**
- **Cu²⁺/Cu**
- **MnO₄⁻/Mn²⁺**

- **Permanganometry**

Permanganometry is a redox titration technique that uses the permanganate ion (**MnO₄⁻**) as the titrant. The redox couple involved is **MnO₄⁻/Mn²⁺**, which has a standard reduction potential of **+1.507 V** in acidic medium.

The permanganate ion is a powerful oxidizing agent. During the reaction, the purple **MnO₄⁻** ion is reduced to the nearly colorless **Mn²⁺** ion:



Because the permanganate ion is intensely colored while its reduced form is essentially colorless, it acts as a **self-indicator**. Consequently, the endpoint of the titration can be detected without the addition of an external indicator. The first persistent pale pink color indicates that a slight excess of permanganate has been added, marking the equivalence point.



A balanced half-reaction must satisfy both mass and charge conservation. In acidic medium, oxygen atoms are balanced by adding H₂O molecules, and hydrogen atoms are balanced by adding H⁺ ions. Electrons are added last to balance the charges.

5. Experimental Section

5.1. Materials and Reagents

Apparatus

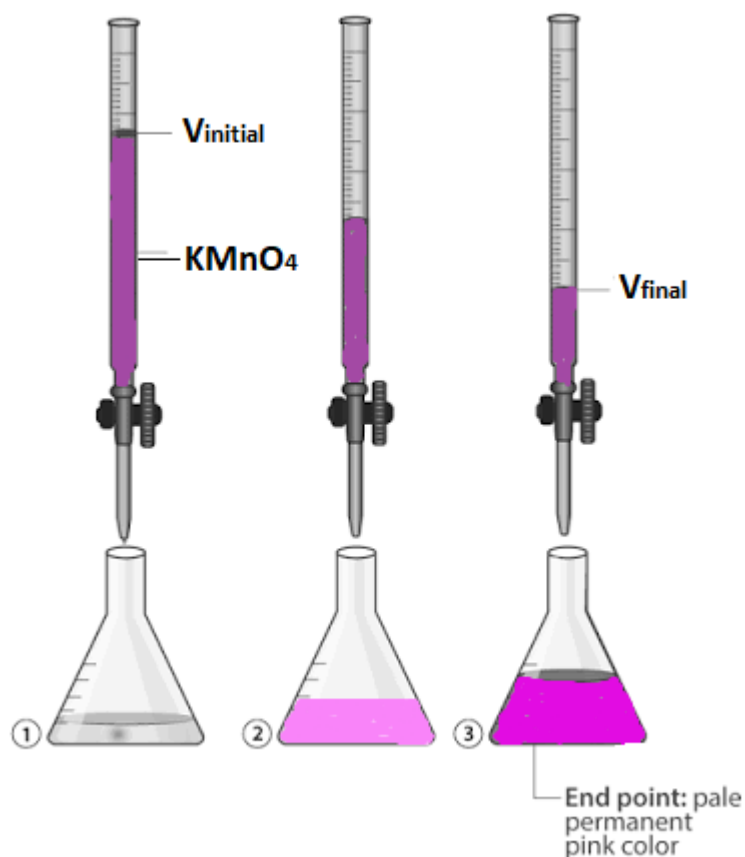
- Burette
- Volumetric pipette
- Erlenmeyer flask
- Hot plate

Reagents

- 0.1 M oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$)
- 10% sulfuric acid (H_2SO_4)
- Potassium permanganate (KMnO_4) solution

5.2. Experimental Procedure

1. Fill the burette with the **potassium permanganate (KMnO_4) solution**.
2. Using a volumetric pipette, transfer **10.0 mL of 0.1 N oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$)** into an **Erlenmeyer flask**, then add approximately **10 mL of 10% sulfuric acid (H_2SO_4)**.
3. Heat the mixture to approximately **60°C** using a hot plate.
4. Add the **KMnO_4** solution dropwise while continuously swirling the flask until each drop is completely decolorized.
5. Continue adding the KMnO_4 solution slowly with constant swirling until a **faint pink color persists for about 30 seconds**, indicating that the endpoint has been reached.
6. Repeat the titration **three times** and record the volume of KMnO_4 used for each trial.



Titration of $\text{H}_2\text{C}_2\text{O}_4$ using KMnO_4

5.3. Results and Data Analysis

1. Write the oxidation and reduction half-reactions, then derive the overall redox reaction.
2. Explain why no external indicator was used during the titration.
3. Determine the concentration of the potassium permanganate (KMnO_4) solution.

5.4. Quiz

Tick the correct statement

- ☐ Water is the reducing agent of the $\text{O}_2/\text{H}_2\text{O}$ couple
- ☐ A redox reaction takes place between the oxidizing agent and the reducing agent of the same couple.
- ☐ During a redox reaction, the number of electrons donated by the reducing agent is equal to the number of electrons captured by the oxidizing agent.
- ☐ The couple made up of the iron(III) ion and the iron(II) ion is written $\text{Fe}^{3+}/\text{Fe}^{2+}$

5.5. Quiz

Given that the permanganate ion is deep purple and all other species involved in the titration are colorless, how is the equivalence point detected in a permanganometric titration?

- ☐ By the disappearance of the purple color in the solution.
- ☐ By the appearance of a persistent pale pink color in the solution.
- ☐ By adding an external acid-base indicator such as phenolphthalein.

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