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Level: 1st Year- Licence-ST  
Subject: Laboratory Work in  
Chemistry1



***PRACTICAL WORK HANDOUT***

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## "PW N°1: Introduction to Chemistry Lab Work

### I. Safety rules

Laboratory safety protocols are an essential component of an effective chemistry education, particularly when utilizing fragile glassware, heating equipment, and various chemical substances (such as toxic organic solvents, flammable liquids, and irritants). Implementing and adhering to these protocols from the outset of the academic year is crucial to ensure the safety of both individuals and equipment.

#### 1. General Hygiene and Conduct:

- ☐ Food and Drink: For hygienic reasons, eating and drinking are strictly prohibited in the laboratory.
- ☐ Personal Attire:
  - Lab Coat: Wearing a properly fitting cotton lab coat is mandatory. Ensure the lab coat is of reasonable length with long sleeves and kept buttoned up throughout the session.
  - Hair and Clothing: Long hair must be tied back securely. Clothing should not be placed on or near the workbench. Keep personal belongings to a minimum on the workbench. During experiments, store belongings under the workbench. Ensure aisles remain unobstructed at all times.
- ☐ Workstation: Always work while standing. Stools or chairs should be stored under the workbench to avoid obstructing aisles. Movement should be kept to a minimum.
- ☐ Safety Awareness: Familiarize yourself with all safety pictograms displayed in the laboratory.

#### 2. Personal Protective Equipment (PPE) and Safety Procedures:

- ☐ Gloves and Goggles: Wear gloves and protective goggles during any manipulations with potential risks as indicated by the instructor.
- ☐ Fume Hoods: Manipulations involving hazardous chemicals must be performed under a ventilated fume hood with protective glass panels. Follow the instructor's instructions for proper use of the fume hood.
- ☐ Pipetting: Pipetting by mouth is strictly prohibited, even for products considered mildly hazardous.
- ☐ Workstation Cleanliness: Maintain a clean and organized workspace throughout the session. Thoroughly clean and dry the workbench at the end of the session.

#### 3. General Safety and Responsibility:

- ☐ Personal Safety and Awareness: Always prioritize your own safety and the safety of your peers.

- ☐ Hand Hygiene: Wash your hands thoroughly with soap and water before leaving the laboratory.

## II. Reading a product label

Information on the Hazards of Chemical Products: The label attached to containers of commercially available products provides crucial information about their dangerous properties. It must include:

- ☐ Name of the substance: This identifies the chemical and allows users to reference its safety data sheet (SDS).
- ☐ One, two, or three hazard symbols (pictograms): These standardized symbols instantly convey the primary dangers associated with the chemical.
- ☐ One or more risk phrases (R phrases): These phrases describe the specific hazards of the substance and its potential effects on human health and the environment.
- ☐ One or more safety phrases (S phrases): These phrases provide instructions on how to safely handle the chemical, including precautions to take and first aid measures if necessary.








Risk and safety phrases supplement the information provided by the pictograms, which often only highlight the main danger. This comprehensive labelling system ensures users are aware of the potential hazards and can handle the chemicals safely.

### ➤ Labelling During Repackaging:

When repackaging any chemical, it is critical to reproduce the original labelling accurately and completely. This ensures that the new container carries all essential safety information.

- ☐ Additionally, ensure the correct labelling of all bottles, flasks, and containers containing:
- ☐ Reactants: These are the starting materials used in a chemical reaction.
- ☐ Ongoing preparations: These include any intermediate products or mixtures formed during a reaction.
- ☐ Date for solutions: This is especially important for solutions that degrade over time and may become unstable.
- ☐ Replace labels systematically when they are damaged or difficult to read. Clear and legible labels are crucial for ensuring safety and preventing accidents.

### III. Symbols used on chemical product labels and associated risks

Pictogram	Signification	Risks	Safety precautions
	Oxidizing Substance	May cause or worsen a fire.	Avoid all contact with combustible materials
	Easily Flammable Substance F	<ul style="list-style-type: none"> <li>✓ Self-igniting</li> <li>✓ Easily flammable gas</li> <li>✓ Flammable liquid</li> </ul>	<ul style="list-style-type: none"> <li>• Avoid all contact with air.</li> <li>• Avoid the formation of flammable vapor-air mixtures and contact with any source of ignition.</li> <li>• Avoid contact with water.</li> <li>• Keep away from flames, sparks, and any source of heat.</li> </ul>
	Harmful Substance Xn Irritant Xi	Its absorption can cause minor injuries or this product can irritate the skin, eyes, or respiratory tract.	Avoid contact with the skin and eyes, and inhalation of vapours.
	Toxic Substance T Very Toxic T+	Causes severe injuries or even death through inhalation, ingestion, or skin contact.	Avoid all contact with the body
	Explosive Substance E	Under certain conditions, presents a specific explosion hazard.	Avoid shocks, friction, sparks, and fire
	Corrosive Substance C	Contact leads to the destruction of living tissues and materials	Avoid inhaling vapours and contact with the skin, eyes, and clothing
	Harmful to the environment N	Products that can cause harm to wildlife, flora, or lead to water pollution	Do not pour down the sink (use waste containers).

#### IV. Laboratory Equipment and Glassware:

Chemistry laboratories commonly share a core set of equipment, glassware, and characterization tools, including:

- ☐ Equipment: Stirrers, balances, hotplates, centrifuges, etc.
- ☐ General-use glassware: Beakers, test tubes, Erlenmeyer flasks, watch glasses, etc.
- ☐ Volumetric glassware: Pipettes (graduated and volumetric), burettes, volumetric flasks, etc.
- ☐ Accessories: Wash bottles, funnels, spatulas, brushes, etc.

Laboratory equipment serves two primary functions: facilitating manipulations and experiments, and taking measurements and gathering data. The appropriate choice of glassware depends on the desired level of precision:

- ☐ Volumetric flasks and pipettes: Used for highly precise measurements.
- ☐ Graduated cylinders and pipettes: Used for moderately precise measurements.
- ☐ Beakers, Erlenmeyer flasks, etc.: Used for approximate measurements.



Wash bottle



Spoonbill



Electronic scale



Wooden clamp



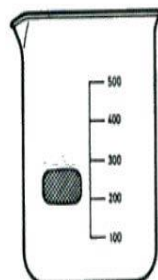
Bunsen burner



Erlenmeyer flask



Test tube



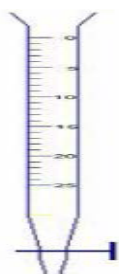
Beaker



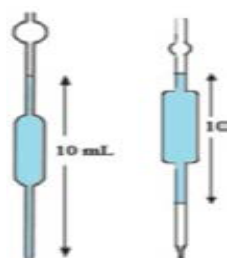
Graduated cylinder



Funnel



Burette



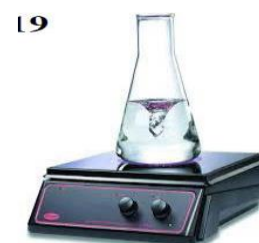
Volumetric pipette



Graduated pipette



Watch glass



Magnetic stirrer



### V. Accurately Reading volume of liquid in a Burette

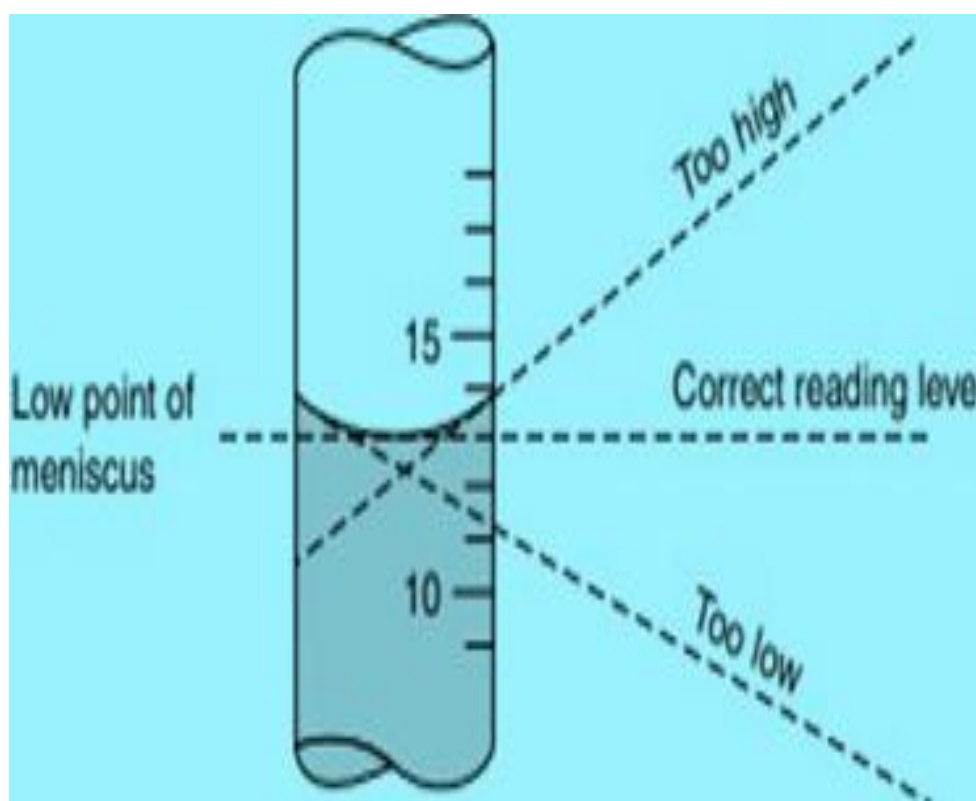
When reading the graduations in the burette, the eye of the observer must be level with the liquid surface to minimize parallax error. Even the thickness of the graduations printed on the burette can affect the reading. For accurate measurement, the bottom of the meniscus (the concave curve of the liquid surface) should touch the top of the desired graduation line. This is illustrated in the accompanying image for transparent solutions.

For highly coloured solutions where the meniscus is difficult to discern, the reading should be taken at the upper surface of the liquid. To ensure accurate reading, the burette should be held vertically in a stand placed on a flat, stable surface. A white sheet of paper placed behind the scale can also enhance visibility.

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#### Reading the volume of liquid

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## PW N°2: Preparation of Solutions

### I. Objectives:

- ☐ Develop a dissolution protocol.
- ☐ Implement a dilution protocol.

### II. Materials:

- |  |   |
|--|---|
| <input type="checkbox"/> balance                 | <input type="checkbox"/> Funnel                       |
| <input type="checkbox"/> Beakers, watch glass    | <input type="checkbox"/> Distilled water spray bottle |
| <input type="checkbox"/> 100 mL volumetric flask | <input type="checkbox"/> Salt (NaCl)                  |
| <input type="checkbox"/> 5 mL graduated pipette  | <input type="checkbox"/> Hydrochloric acid (HCl) :1N  |

### III. Basic Concepts:

1. **Solvent:** A solvent is a liquid that has the property of dissolving and diluting other substances without chemically altering them or itself undergoing modification.
2. **Solute:** A substance dissolved in a solvent.
3. **Solution:** A homogeneous mixture formed when a solid, liquid, or gas is dissolved in a liquid.
4. **Molar Mass (M):** The mass of one mole of a compound, expressed in grams per mole (g/mol).
5. **Amount of Substance (n):** The number of moles in a sample. It can be calculated using the following equation:.

$$n = \frac{m}{M} \text{ (mol)}$$

where:

m is the mass of the sample in grams.

M is the molar mass of the compound in g/mol.

6. **Density (ρ):** The ratio of the mass (m) of a substance to its volume (V). It can be calculated using the following equation:

$$\rho = \frac{m_{\text{solution}}}{V_{\text{solution}}} \text{ (in kg/m}^3 \text{ or g/cm}^3\text{)}$$

where:

m<sub>solution</sub> is the mass of the solution in gram.

V<sub>solution</sub> is the volume of the solution in liter.

Example: The density of water is 1000 kg.m<sup>3</sup> (1 m<sup>3</sup> of water has a mass of 1000 kg).

7. **Density (d):** The ratio of the density of a substance to the density of a reference substance. For liquids and solids, the reference substance is pure water at 4 °C. The following equation applies:

$$d = \rho \text{ (sample)} / \rho \text{ (reference)}$$

For gases and vapors, the reference substance is air at the same temperature and pressure. The following equation applies:

$$d = \frac{\rho(\text{sample})}{\rho(\text{air})} = \frac{M(\text{gaz})}{M(\text{air})} = \frac{M(\text{gaz})}{29}$$

### 8. Molecular Concentration (Molarity, M):

The amount of solute present per liter of solution. It can be calculated using the following equation:

$$C = \frac{n_{\text{solute}}}{V_{\text{solution}}} = \frac{m}{M \cdot V} \text{ (mol/L)}$$

where:

n solute is the amount of solute in moles

V<sub>solution</sub> is the volume of the solution in liters

m is the mass of the solute in grams

M is the molar mass of the solute in g/mol

9. **Mass Concentration (C<sub>m</sub>):** The mass of solute per liter of solution. It can be calculated using the following equation:

$$C_m = \frac{m_{\text{solute}}}{V_{\text{solution}}} = \text{(g/L)}$$

$$C_m = C * M \text{ (g/L)}$$

10. **Normal Concentration (N):** The number of gram-equivalents or the number of moles of equivalents of solute in one liter of solution. It can be calculated using the following equation:

$$N = C * Z \text{ (eq. g /L)}$$

In the case of an acid-base reaction, Z is the number of H<sup>+</sup> ions involved in the reaction.

For a redox reaction: Z is the number of electrons involved in the reaction.

11. **Mass Fraction (x<sub>m</sub>):** The ratio between the mass of solute and the mass of solution. It can be calculated using the following equation:

$$x_m = \frac{m_{\text{solute}}}{m_{\text{solution}}} * 100$$

12. **Molar Fraction (x):** The ratio between the number of moles of solute and the total number of moles in the solution. It can be calculated using the following equation:

$$x = \frac{n_{\text{solute}}}{n_{\text{solution}}} * 100$$

## IV. Dilution:

The addition of a solvent to a solution to reduce its concentration. If the initial solution concentration (C<sub>m</sub>), desired solution concentration (C<sub>d</sub>), and final solution volume (V<sub>d</sub>) are known, the volume of the initial solution to be taken (V<sub>m</sub>) can be determined using the following equation:

$$C_m * V_m = C_d * V_d$$

## V. Preparation of a Solution by Dissolution of a Solid Compound:

We aim to prepare 100 mL of an aqueous sodium chloride solution with a concentration of  $C = 0.1 \text{ mol/L}$ .

- ☐ Determine the molar mass of the sodium chloride: This requires consulting a chemical reference table or using the periodic table to find the atomic masses of sodium and chlorine, and then summing their masses.
- ☐ Calculate the mass 'm' of the product required to make the solution.
- ☐ Weigh the determined mass of sodium chloride: Use a clean, dry spatula and watch glass, and weigh the required mass accurately on a balance.
- ☐ Transfer the solid to a 100 mL volumetric flask: Use a funnel to avoid spills.
- ☐ Rinse the watch glass and funnel with distilled water: Transfer the rinsing water into the flask.
- ☐ Fill the volumetric flask to three-quarters with distilled water: Use a spray bottle to dispense the water gently.
- ☐ Dissolve all NaCl crystals: After sealing the flask, shake it vigorously until all solid dissolves.
- ☐ Adjust the level to the calibration mark: Hold the flask vertically, aligning your eye with the bottom of the meniscus to avoid parallax error.
- ☐ Seal the volumetric flask securely and invert it several times: This completes the preparation of the "stock" solution of NaCl.

## VI. Preparation of a Solution by Dilution

We aim to prepare an aqueous solution (F) of sodium chloride with a molar concentration of  $C_f = 0.02 \text{ mol/L}$ . However, we only have the previously prepared 100 mL aqueous solution with a concentration of  $C = 0.1 \text{ mol/L}$  and a 250 mL volumetric flask.

Determine the volume (V) to be taken from the initial solution.

## PWN03: Acid-Base Titration

### I. Objective:

To determine the unknown amount (mass) of the base present in the sample.

### II. Materials:

- |   |  |
|---|--|
| <input type="checkbox"/> Graduated burette on its stand | <input type="checkbox"/> 100 ml volumetric flask         |
| <input type="checkbox"/> Magnetic stirrer               | <input type="checkbox"/> Spray bottle of distilled water |
| <input type="checkbox"/> Magnetic stirring bar          | <input type="checkbox"/> Coloured indicator.             |
| <input type="checkbox"/> Beaker                         | <input type="checkbox"/> HCl (0,1N)                      |
| <input type="checkbox"/> Erlenmeyer flask               | <input type="checkbox"/> NaOH                            |
| <input type="checkbox"/> Pipette 20ml                   |  |

### III. General Concepts:

#### 1. Acid-base titration

Acid-base titration is a quantitative analytical technique used to determine the unknown concentration of an acidic or basic solution. This technique involves the neutralization reaction between a titrant (solution of known concentration) and an analyte (solution of unknown concentration).

#### 2. Acids:

An acid is a chemical compound that releases hydrogen ions ( $H^+$ ) when dissolved in water. It is characterized by its ability to donate protons ( $H^+$ ) or accept electrons in a chemical reaction.

- a. **Monoprotic Acid:** Hydrochloric Acid (HCl)

Dissociation Equation in Water:  $HCl \rightarrow H^+ + Cl^-$

- b. **Diprotic Acid:** Sulfuric Acid ( $H_2SO_4$ )

Dissociation Equation in Water:  $H_2SO_4 \rightarrow 2H^+ + SO_4^{2-}$

- c. **Triprotic Acid:** Phosphoric Acid ( $H_3PO_4$ )

Dissociation Equation in Water:  $H_3PO_4 \rightarrow 3H^+ + PO_4^{3-}$

#### 3. Bases:

A base is a chemical compound that releases hydroxide ions ( $OH^-$ ) when dissolved in water. Bases are characterized by their ability to accept protons or donate electron pairs in a chemical reaction.

a. **Monofunctional Base:** Sodium Hydroxide (NaOH)

. Dissociation Equation in Water:  $\text{NaOH} \rightarrow \text{Na}^+ + \text{OH}^-$

b. **Difunctional Base:** Calcium Hydroxide (Ca(OH)<sub>2</sub>)

Dissociation Equation in Water:  $\text{Ca(OH)}_2 \rightarrow \text{Ca}^{2+} + 2\text{OH}^-$

c. **Trifunctional Base:** Aluminium Hydroxide (Al(OH)<sub>3</sub>)

Dissociation Equation in Water:  $\text{Al(OH)}_3 \rightarrow \text{Al}^{3+} + 3\text{OH}^-$

#### IV. Principle

At equivalence, the number of moles of H<sub>3</sub>O<sup>+</sup> provided by the acid must be equal to the number of moles of OH<sup>-</sup> provided by the base. This leads to the following equation:

$$N_A \cdot V_A = N_B \cdot V_B$$

where:

N<sub>A</sub> is the normality of the acid

V<sub>A</sub> is the volume of the acid

N<sub>B</sub> is the normality of the base

V<sub>B</sub> is the volume of the base

An acid-base titration can be monitored by:

- ☐ pH-metry: This involves measuring the pH change during the reaction.
- ☐ Colorimetry using a coloured indicator: This method relies on a reagent whose colour changes depending on the pH. It can be used to detect the endpoint of a titration if the equivalence point falls within the indicator's colour change range.

#### V. Experimental procedure

- ☐ A sample of an unknown concentration basic solution in a labelled flask with a capacity of 100 millilitres.
- ☐ Fill the flask to the calibration mark with distilled water.
- ☐ Shake the solution to ensure homogeneity.
- ☐ Transfer a 20 ml aliquot of the solution to an Erlenmeyer flask.
- ☐ Add two to three drops of the coloured indicator to the Erlenmeyer flask.
- ☐ Fill the burette with hydrochloric acid solution.
- ☐ Place the Erlenmeyer flask under the burette's tip.
- ☐ Initiate the titration.
- ☐ Record the volume of acid used for the titration.

## PWN° 4: pH-Metric Titration

### I. Objective

Determination of the concentration and acidity constant ( $K_a$ ) of acetic acid using a pH-metric titration.

### II. Principle

A titration involves determining the unknown concentration of an acid (or base) in a solution by reacting it with a known concentration of a base (or acid) until the reaction reaches equivalence. During a pH-metric titration, the pH of the titrated solution is continuously measured as the titrant is added. To accurately represent the titration curve, equal volume increments of titrant are added initially, followed by smaller increments closer to the equivalence point where the pH changes rapidly.

The resulting pH-metric titration curve ( $\text{pH} = f(V_{\text{added}})$ ) shows a significant increase in pH at the equivalence point, signifying the complete reaction of the acid with the base.

### III. Experimental Part

#### 1) Materials and Products

- |  |  |
|--|--|
| <input type="checkbox"/> Graduated burette                             | <input type="checkbox"/> Magnetic stirrer                                  |
| <input type="checkbox"/> pH meter                                      | <input type="checkbox"/> Colored indicator                                 |
| <input type="checkbox"/> Beaker 150 mL                                 | <input type="checkbox"/> Magnetic stirrer bar                              |
| <input type="checkbox"/> Hydroxide Sodium (NaOH)<br>solution 0.1 mol/L | <input type="checkbox"/> Acetic acid solution ( $\text{CH}_3\text{COOH}$ ) |

#### 2) Procedure

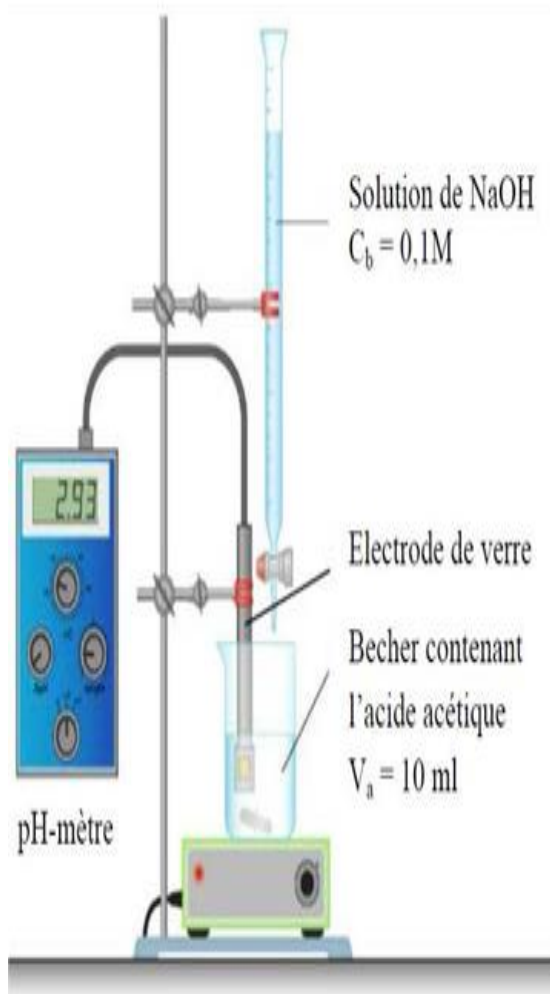
##### 1. Preparation:

- ☐ Prior to titration, dilute the acetic acid solution.
- ☐ Fill the burette with the known concentration ( $C_b = 0.1 \text{ mol/L}$ ) aqueous sodium hydroxide (NaOH) solution.
- ☐ Measure 10.0 mL of the diluted acetic acid solution with a calibrated pipette and transfer it to a 150 mL beaker.
- ☐ Add approximately 25.0 mL of distilled water to the beaker.

##### 2. Titration:

- ☐ Position the beaker under the burette and immerse the pH meter probe.

- ☐ Set up and activate the magnetic stirrer with a magnetic stirring bar.
- ☐ Slowly add the titrant (0.1 M NaOH) dropwise into the beaker, continuously stirring the solution.
- ☐ After each addition, record the volume of titrant dispensed and the corresponding pH of the solution in a data table.





## PW N° 05: Redox Titration (Iodometry)

### I) Basic concepts

#### 1. Oxidation-Reduction Reactions:

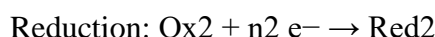
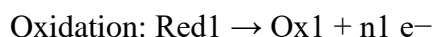
An oxidation-reduction (redox) reaction involves the transfer of electrons between chemical species. These reactions have diverse applications in numerous fields, including combustion, metallurgy, electrochemistry, and even within the human body, such as the respiratory system. Redox reactions are fundamentally composed of two half-reactions: oxidation and reduction.

#### 2. Understanding Oxidation and Reduction:

Oxidation is precisely defined as the loss of one or more electrons, while the reverse reaction, the gain of electrons, is termed reduction. The species that loses electrons is known as the reducing agent, while the species that gains electrons is the oxidizing agent.

Relationship Between Oxidation and Reduction:

Reduction is directly associated with the generation of a reducing agent, while oxidation corresponds to the formation of an oxidizing agent. In a redox reaction, the reducer is oxidized, and the oxidizer is reduced. This relationship can be represented by the following half-reactions:



where Red1 and Red2 represent the reduced species, Ox1 and Ox2 represent the oxidized species, and  $n1$  and  $n2$  represent the number of electrons transferred.

#### 3. Balancing Redox Reactions:

To ensure the conservation of electrons in a redox reaction, the half-reactions must be balanced. This involves adjusting the coefficients of the species in each half-reaction such that the total number of electrons lost in the oxidation half-reaction equals the total number of electrons gained in the reduction half-reaction. The balanced overall reaction can be obtained by summing the balanced half-reactions.

#### 4. Assessing Redox Activity:

To determine the directionality of a redox reaction, it is crucial to compare the oxidizing and reducing capacities of the participating species. This can be achieved by analysing the standard electrode potential ( $E^\circ$ ). A higher  $E^\circ$  value indicates a stronger oxidizing agent, whereas a lower  $E^\circ$  value indicates a stronger reducing agent. The direction of the reaction will favour the formation of the stronger oxidizing agent and the stronger reducing agent.

## II) Principle:

Iodometry is a redox titration method used to titrate iodine ( $I_2$ ) present in a solution using sodium thiosulfate ( $Na_2S_2O_3$ ).

Oxidation/reduction couples:  $I_2/I^-$ ;  $S_4O_6^{2-}/S_2O_3^{2-}$ .

## III) Experimental Part:

### 1) Materials and Products

- |  |  |
|--|--|
| <input type="checkbox"/> Graduated burette on its stand. | <input type="checkbox"/> Erlenmeyer flask.             |
| <input type="checkbox"/> Magnetic stirrer.               | <input type="checkbox"/> Distilled water spray bottle. |
| <input type="checkbox"/> Magnetic stirring bar.          | <input type="checkbox"/> Coloured indicator.           |
| <input type="checkbox"/> Calibrated pipette              | <input type="checkbox"/> White paper.                  |
| <input type="checkbox"/> Beaker.                         |  |

### 2) Procedure:

- ☐ Fill the burette with an aqueous solution of sodium thiosulfate having a concentration of  $C_1 = 0.1 \text{ mol/L}$ .
- ☐ Pipette 10.0 mL of the iodine solution to be titrated using a calibrated pipette into an Erlenmeyer flask positioned on a magnetic stirrer.
- ☐ Add a few drops of starch indicator solution.
- ☐ Perform an initial rapid titration to estimate the equivalence point volume, followed by a second precise titration. Record the equivalence point volume as  $V_E$ .

## PW N° 06: Redox Titration (Manganimetry).

### I) Manganimetry

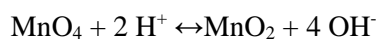
Manganimetry is a quantitative analytical technique based on redox reactions involving the permanganate ion ( $\text{MnO}_4^-$ ). This ion readily accepts electrons, acting as an oxidizing agent. The quantification process relies on the redox couple  $\text{MnO}_4^-/\text{Mn}^{2+}$ , which has a standard potential of 1.507 V. This potential difference serves as a measure of the amount of analyte oxidized by the permanganate ion, allowing for its determination.

### II) Principle of Manganimetry

Potassium permanganate ( $\text{KMnO}_4$ ) is a strong oxidizing agent for a wide range of chemical species. Its corresponding reduction products include  $\text{Mn}^{2+}$  (colourless),  $\text{Mn}^{3+}$  (slightly coloured), or  $\text{MnO}_2$  (brown), depending on the reaction medium (acidic or basic). Importantly, the oxidizing form,  $\text{MnO}_4^-$ , is intensely violet, while the reduced form,  $\text{Mn}^{2+}$ , is colourless. This distinct colour change allows for the determination of the equivalence point without relying on additional coloured indicators.

However, if the medium is not sufficiently acidic, the involved redox couple becomes  $\text{MnO}_4^-/\text{MnO}_2$ . Since manganese dioxide is a sparingly soluble brown solid, the equivalence point is no longer visually detectable. To overcome this issue, the medium must be acidified by adding concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ). Hydrochloric acid ( $\text{HCl}$ ) and nitric acid ( $\text{HNO}_3$ ) are not suitable alternatives.  $\text{HCl}$  is itself oxidized by permanganate ions, and  $\text{HNO}_3$  acts as an additional oxidizing agent, interfering with the desired reaction.

The addition of  $\text{H}^+$  ions from  $\text{H}_2\text{SO}_4$  effectively prevents the reaction from occurring in a neutral medium:



The equivalence point in a redox titration can be determined in several ways:

- ☐ Direct observation of colour change: If one of the reactants or products is coloured (e.g.,  $\text{MnO}_4^-$ ), the appearance or disappearance of the colour can indicate equivalence.
- ☐ Monitoring the potential: The potential of the involved redox couples changes throughout the titration. A sharp jump in potential signifies the equivalence point.
- ☐ Utilizing a coloured redox indicator: Some indicators, such as starch, exhibit a distinct colour change in response to the presence of excess  $\text{MnO}_4^-$ . This can also be used to identify the equivalence point.

### III) Objective:

This experiment aims to calibrate a potassium permanganate ( $\text{KMnO}_4$ ) solution using a standardized oxalic acid solution and subsequently determine the exact concentration of the  $\text{KMnO}_4$ . Due to the initially slow reaction rate between these two chemicals, gentle heating (not exceeding  $50^\circ\text{C}$ ) will be applied at the beginning of the titration to accelerate the reaction.

### IV) Preparation of Oxalic Acid Solution:

Prepare a 100 mL aqueous solution of oxalic acid with a normality (Na) of 0.1 N. To achieve this, utilize dehydrated oxalic acid crystals with the formula ( $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ), which possess a molar mass of 126.07 g/mol.

#### Procedure:

1. Calculate the mass of oxalic acid required:

- ☐ First, determine the moles of oxalic acid needed:  $\text{moles oxalic acid} = \text{Na (N)} \times \text{volume (L)}$
- ☐ Then, calculate the mass of oxalic acid:  $\text{mass oxalic acid} = \text{moles oxalic acid} \times \text{molar mass oxalic acid}$

Prepare the aqueous oxalic acid solution:

- ☐ Accurately weigh the calculated m (g) of dehydrated oxalic acid crystals.
- ☐ Transfer the weighed crystals to a 100 mL volumetric flask.
- ☐ Add a small amount of deionized water to the flask and swirl gently to dissolve the crystals.
- ☐ Fill the flask to the calibration mark with deionized water, ensuring the solution is well-mixed.

**Caution:** Always pre-rinse the volumetric flask with deionized water before adding the oxalic acid crystals. Slowly add the crystals while swirling the flask continuously to ensure complete dissolution.

## V) Experimental Part

### 1. Materials and Chemicals

- |  |  |
|--|--|
| <input type="checkbox"/> Graduated burette on its stand. | <input type="checkbox"/> Pro-pipette.                  |
| <input type="checkbox"/> Magnetic stirrer.               | <input type="checkbox"/> Beaker.                       |
| <input type="checkbox"/> Magnetic stirring bar.          | <input type="checkbox"/> Erlenmeyer flask.             |
| <input type="checkbox"/> Hotplate.                       | <input type="checkbox"/> Graduated cylinder.           |
| <input type="checkbox"/> Balance.                        | <input type="checkbox"/> Distilled water spray bottle. |
| <input type="checkbox"/> Watch glass + spatula.          | <input type="checkbox"/> White paper.                  |
| <input type="checkbox"/> Funnel.                         | <input type="checkbox"/> 100 mL volumetric flask       |

## 2. Operating procedure:

### 1. Preparation:

- ☐ Fill the burette with the potassium permanganate solution.
- ☐ Using a micropipette, accurately transfer 10.00 mL of the oxalic acid standard solution into an Erlenmeyer flask.
- ☐ Add 20.00 mL of 1.00 mol/L sulfuric acid solution to the Erlenmeyer flask (do not use a pipette).

### 2. Titration:

- ☐ Allow approximately 2 mL of potassium permanganate solution to flow into the Erlenmeyer flask. Do not reset the burette to zero.
- ☐ Heat the Erlenmeyer flask on the hot plate until the solution becomes colourless.
- ☐ Continue the titration carefully, adding the potassium permanganate solution dropwise, until a persistent pink colour appears.

### 3. Repeat:

- ☐ Perform a second titration, adding the potassium permanganate solution dropwise until a persistent pink colour is observed.

## VI) Experiment II: Back Titration of Potassium Dichromate Solution Using an Aqueous Solution of Known Ferrous Salt concentration

### VII) Objective

This experiment aims to determine the concentration of a potassium dichromate  $K_2Cr_2O_7$  solution using a back titration technique with an aqueous solution of known titer ferrous salt.

### I) Principle

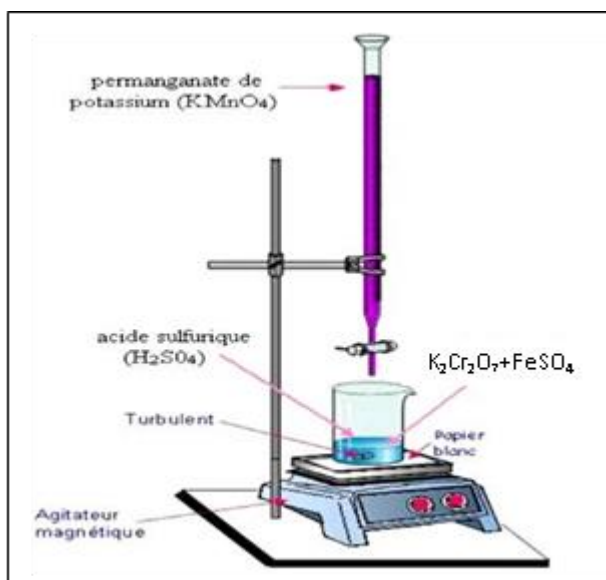
Direct titration of chromium with potassium permanganate is not feasible due to the masking of color changes at the equivalence point. The interfering colours include yellow-orange  $CrO_4^{2-}$  ions, green  $Cr^{3+}$  ions, and rust-coloured  $Fe^{3+}$  ions. Therefore, an indirect or back titration is employed.

In this method, excess ferrous ions ( $Fe^{2+}$ ) from a solution of known concentration are used to reduce  $Cr_2O_7^{2-}$  ions. This excess ensures complete reaction of the chromium species.

The remaining  $Fe^{2+}$  ions are then titrated with a standard solution of potassium permanganate ( $KMnO_4$ ) in the presence of sulfuric acid ( $H_2SO_4$ ). The involved redox couples are  $Cr_2O_7^{2-}/Cr^{3+}$ ,  $Fe^{3+}/Fe^{2+}$ ,  $MnO_4^-/Mn^{2+}$ .

## II) Operating procedure:

- ☐ Fill the burette with a standardized potassium permanganate solution of concentration  $C_2 = 0.010 \text{ mol/L}$ .
- ☐ Transfer a volume  $V_1 = 50 \text{ mL}$  of the iron(II) sulfate solution ( $C_1 = 0.020 \text{ mol/L}$ ) into an Erlenmeyer flask. In addition, add a volume  $V_2 = 10 \text{ mL}$  of the potassium dichromate solution and a volume  $V_3 = 10 \text{ mL}$  of concentrated sulfuric acid.
- ☐ Place the Erlenmeyer flask under the burette and stir the mixture to ensure homogeneity.
- ☐ Gradually add the potassium permanganate solution from the burette to the Erlenmeyer flask until a persistent faint pink colour is observed. Record the volume of potassium permanganate used (VE).
- ☐ Perform two replicate titrations, adding the potassium permanganate dropwise near the endpoint for increased accuracy.



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