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Practical Works

General Chemistry Experiments

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Foreword

The Practical work is an integral aspect of chemistry education, bridging the gap between theoretical knowledge and real-world application. This manuscript is designed to guide students through a series of carefully curated experiments that illustrate fundamental concepts in general chemistry. Targeted primarily at first-year university students in material sciences, sciences and technologies. It provides a structured approach to understanding essential laboratory techniques, safety protocols, and the application of the scientific method.

The experiments included in this manuscript aim to equip students with critical skills, such as precise measurement, accurate data collection, and systematic error analysis. By performing hands-on activities, students gain practical experience that reinforces their theoretical understanding, enabling them to observe chemical phenomena, analyze outcomes, and draw meaningful conclusions.

This document includes seven experimental activities, each presented with a clear, step-by-step structure. It introduces students to essential safety guidelines and laboratory procedures, along with fundamental techniques for preparing solutions of solid and liquid chemicals. The experiments cover key topics such as basic acid titration using colorimetric indicators, pH measurement, and conductivity analysis. They also involve oxidation with potassium permanganate, as well as determining chlorine ion concentration in water using Mohr's method. Each activity provides the objective, required equipment, and detailed protocols to guide students through the experiment.

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Laboratory Report

To achieve the desired goals of the practical works, students are provided with several detailed of the experimental protocols utilized in the practical work. A lab report is required from students to assess their level of understanding and analysis, its goal is not to simply copy what is written in the lab manual, but to present high-quality results a strong analysis and interpretation of the findings. In general, a lab report is typically composed of the following parts.

1- **Cover Page:** It include the title of experiment and the name(s) of the authors.

2-**Introduction:** Providing a concise explanation of the principle and objective of the experiment.

3-**Objetif of the practical work:** The student to clearly define the purpose or objective of the experiment in concise terms, typically within one or two sentences.

4-**Presentation:** It is beneficial to include some definitions and theoretical frameworks relevant to the experimental work, supported by appropriate references for clarity and credibility ;

5-**Materials and Equipments:** Compile a comprehensive a list of all the chemicals and equipments required for the experiment.

6-**Methodology:** Povides a detailed and organized explanation of the procedures, ensuring clarity and enabling replication of the experiment by anyone.

7-**Results:** To include the procedures used in the lab manual. Also to present obtained results (tables, graphs).

8-**Discussion:** Discuss the obtained results using a theoretical approach.

9-**Conclusion:** Summarize the results with emphasizing the key findings.

Abbreviations List

A

AgCl : Silver chloride
Ag_2CrO_4 : Silver chromate
$AgNO_3$: Silver Nitrate

B

$BaOH_2$: Barium hydroxide

C

°C : Degree celsius
Cm : Molality
C_N : Normality
C_M : Molarity
CH_3COOH : Acetic acid
$CaCO_3$: Calcium carbonate
$CaSO_4$: Calcium sulfate

D

D : Density

E

EDTA : Ethylenediaminetetraacetic acid
E.B.T : Eriochrome Black-T

F

$FeSO_4$: Ferrous sulfate

G

g : Gramme

H

HCl : Hydrochloric acid
$H_2C_2O_4$: Oxalic acid
H_2SO_4 : Sulfuric acid

I

J

K

$K_4[Fe(CN)_6]$: Potassium cyanide
KOH : Potassium hydroxide
$KMnO_4$: Potassium permanganate

L

M

M : Mass molar
$MgCO_3$: Magnesium carbonate
$MgCl_2$: Magnesium chloride

N

n : number of moles
NaCl : Sodium chloride
NH_4OH+NH_4Cl : Buffer solution (pH ≈10)
$Na_2C_2O_4$: Sodium oxalate
$Na_2C_2O_4$: Sodium oxalate

O

P

pH : Potential of hydrogen
ppm : Parts-per-million
pk_a : Acid dissociation constant

Q

R

S

T

T : Mass concentration
°TH: Water Hardness

U

V

W

X

Y

Z

Δ

Δx : Absolute uncertainty
$\frac{\Delta x}{x^\circ}$: Relative uncertainty

Δ

Δx : Absolute uncertainty
$\frac{\Delta x}{x^\circ}$: Relative uncertainty
σ : Conductivity
ρ : Resistance

Practical Work N° 1. Laboratory Safety Rules and Equipments

1. Introduction

Laboratory safety is a fundamental priority in any scientific environment to prevent accidents, protect individuals, and ensure the reliability of experimental results. Chemistry laboratories, in particular, can be hazardous if safety rules are not strictly followed, as they involve the handling of flammable, explosive, toxic, and carcinogenic substances, along with equipment that may cause severe burns or cuts if used improperly. Many laboratory accidents result from carelessness, impatience, unauthorized experimentation, or disregard for safety protocols. Therefore, it is essential for students and researchers to adhere to established safety guidelines at all times.

2. Objective

This practical work aims to familiarize students with essential safety rules, protective equipment, and emergency procedures necessary for laboratory work. By understanding and applying these regulations, students will develop responsible lab practices that minimize risks and promote a safe working environment.

3. Laboratory Safety Rules

3.1. Laboratory Attire and Behavior

- Use of lab coats, gloves, and safety goggles.
- Hair must be tied back.
- Do not eat, drink, or store food in the laboratory to prevent contamination and accidental ingestion of chemicals.
- Laboratory experiments must always be conducted under the supervision.
- Never attempt to identify chemical compound by touch, smell or taste.

3.2. Handling Chemical Substances

- Carefully read labels and Safety Data Sheets (SDS).
- Explanation of hazard pictograms.
- Safe handling techniques for hazardous substances.
- Never pipette a chemical solution by mouth use a pipette rubber bulb.
- All experiments generating gases or vapors must be carried out under an extraction hood.
- Always add acid to water, never the reverse, to avoid any undesirable reaction and dangerous accidents.
- Never return unused chemicals to their original container to prevent contamination.

- After the experiment, it's important to clean all the equipment used, return them to their designated places in the laboratory, and make sure to wash your hands.
- Before leaving the laboratory, make sure to turn off the electricity and gas, and close the water supply to ensure safety and maintain order in the laboratory.

3.3. Emergency Procedures

In case of an accident in the laboratory, it is essential to remain calm and act cautiously.

- **Exposure to a chemical substance** : Rinse immediately and thoroughly with plenty of water for at least 15 minutes.
- **Cut** : Clean the wound carefully and apply a sterile dressing.
- **Gas leak** : Ventilate the room immediately and shut off the gas supply if possible.
- **Fire** : Stay calm and follow the laboratory evacuation procedures.

4. Chemical storage conditions

Proper storage of chemicals is essential to ensure safety, prevent contamination, and reduce the risk of accidents. The following guidelines must be followed :

- All chemicals must be clearly labeled with their name, concentration, hazard symbols, and expiration date.
- Store chemicals according to their hazard classification (flammable, corrosive, toxic, reactive, etc.).
- Separate acids and bases to prevent dangerous reactions.
- Flammable liquids must be stored in fire-resistant cabinets.
- Oxidizing agents should be kept away from organic materials and reducing agents.
- Store chemicals in well-ventilated areas, away from direct sunlight and heat sources.
- Maintain appropriate temperature and humidity levels.
- Avoid storing volatile substances in non-ventilated spaces.
- Use chemical-resistant shelves and spill containment trays to prevent leaks or spills.
- Keep fire extinguishers and first-aid equipment near storage areas.

5. Hazard symbols and their meanings

Hazard pictograms on chemical product labels are standardized symbols designed to inform users of the potential risks associated with a substance. These pictograms help quickly identify dangers such as flammability, toxicity, corrosivity, and harmful effects on health and the environment. Their presence on a label assists in taking the necessary precautions during the handling, storage, and disposal of chemical products.

 **Explosive** When handling these substances, avoid friction, impacts, electrical sparks, and heat sources (butane gas-propane gas).	 **Corrosive** Avoid exposure to its fumes and prevent direct contact with skin or clothing. Most acids, such as hydrochloric acid and phosphoric acid, are corrosive.	 **Toxic** A highly toxic and deadly substance. Avoid skin contact, inhaling its fumes, tasting it, or using mouth pipetting.In case of accidental ingestion, seek medical attention immediately.
 **Environmental hazard** These substances pose a threat to living organisms and the environment. Store them properly, use them responsibly, and do not dispose of them in nature.	 **Oxidizing** These substances can fuel combustion and intensify fires, making them harder to extinguish. Store them away from flammable materials, heat sources, and open flames.	 **Flammable** Materials that can ignite quickly when exposed to a heat source or an open flame. Their combustion can occur at relatively low temperatures. Therefore, it is essential to keep them away from flames, direct sunlight, and any heat source.
 **Biological hazard** These substances can endanger the health of living organisms, particularly humans, and may also be harmful to animals. They include medical waste, microbiological samples, viruses, and toxins of biological origin.	 **Carcinogen** These hazardous substances have the potential to cause cancer in humans. They must be handled with great caution, avoiding direct skin contact or inhalation of their vapors.	 **Radiation hazard** These materials emit radioactive radiation and pose potential health risks. Safety measures include wearing protective equipment such as respirators with filters, protective footwear, gloves, and specialized clothing.

6. List of Equipments and tools used in chemical Laboratory


Boiling flasks


Beakers


Erlenmeyer flask
Conical flask


Volumetric flask


Graduated Cylinder


Adjustable Volumetric Pipette


Volumetric pipet


Gratuated pipet


Burette


stand and clamp


Watch Glass


Funnel


Separatory funnel


Thermometer


Spatula


Tongs


Filter paper


Reagent bottles


Wash bottle


Rubber bulb

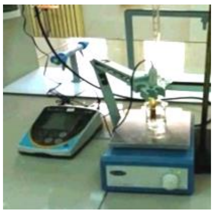

Test tubes


Test tube stand


Crystallising Dish


Petri Dishes


Weighing machine


pH Meter


Conductometer


Calorimeter


Hotplate magnetic stirrer


Magnetic stirrer

7. Uncertainty in measurement

In scientific experiments, particularly in chemistry, measurement plays a crucial role. However, no measurement is perfectly accurate. The difference between the measured value and the true value is known as uncertainty. Understanding uncertainty is essential because it helps assess the reliability of measurements and ensures meaningful conclusions from experimental data.

7.1. Types of Measurement

a) **Direct Measurement:** Obtained directly using measuring instruments (Measuring pressure, conductivity, or pH using devices).

b) **Indirect Measurement:** Derived through calculations based on other measured values (Calculating energy, mass, or concentration).

7.2. Types of Uncertainty

a) **Absolute Uncertainty ($\Delta\, x$):** Represents the maximum possible error in a measurement. Expressed as an absolute value without considering the magnitude of the measurement. It is determined by repeating the experiment multiple times to estimate the variation, then calculating the average of measured values.

$$x_0 = \frac{x_0 + \ x_1 + x_2 + \cdots x_n}{n}$$

$$\Delta x = \frac{|x_0 - x_{max}| + |x_0 - x_{min}|}{2}$$

Where : $x = x_0 \pm \Delta x$

With: $x_0 - \Delta x \leq x \leq x_0 + \Delta x$

b) **Relative Uncertainty:** The ratio of absolute uncertainty to the measured value, it is a dimensionless quantity (has no unit). Mathematically, relative uncertainty is given by $\frac{\Delta x}{x_0}$:

c) **Measurement Accuracy:** Expressed as a percentage to indicate how precise the measurement is and a smaller uncertainty percentage indicates a more accurate measurement $\frac{\Delta x}{x_0}.100\ (\%)$.

Practical Work N° 2. Preparation of Aqueous Solutions in Chemistry

1. Introduction

A solution is a homogeneous mixture of two or more pure substances that do not chemically interact. It is formed when a solute, the substance that dissolves, is dispersed within a solvent, the substance that facilitates dissolution, such as water.

Solution = Solute + Solvent

2. Ways of expressing concentration

Expressing concentration is essential for understanding and controlling chemical reactions, as well as for preparing accurate solutions in laboratories or industrial applications. There are several ways to quantify concentration, suited to various contexts, such as molarity, normality, mole fraction, and mass or volume percentage.

1. **Molarity (M) :** The Molarity is defined as the number of moles of solute dissolved in one liter of solution.

$$\mathrm{M} = \mathrm{C_M} = \frac{moles\ of\ solute}{volume\ of\ solution} = \frac{n_{solute}}{V_{solution}}\ (\frac{mol}{L})$$

2. **Mass concentration (M) :** It refers to the amount of solute present in a given volume of solution, typically expressed in terms of mass per unit volume, denoted by the symbol C or T.

$$T = \frac{masse\ of\ solute}{volume\ of\ solution} = \frac{m_{solute}}{V_{solution}}\ (\frac{g}{L})$$

3. **Normality (N) :** It is defined as the number of equivalents of solute per liter of solution.

$$\mathrm{N} = \mathrm{C_N} = \frac{equivalents\ of\ solute}{volume\ of\ solution} = \frac{n_{eq}}{V_{solution}}\ (\frac{eq}{L})$$

4. **Molality (m) :** Molality is defined as the number of moles of solute per kilogram of solvent.

$$\mathrm{m} = \mathrm{C_m} = \frac{moles\ of\ solute}{mass\ of\ solvent\ in\ kilograms} = \frac{n_{solute}}{m_{solvent}}\ (\frac{mol}{Kg})$$

3. Relation between Normality and Molarity

Normality (**N**) and molarity (**M**) are two important and commonly used concentration units in chemistry. Both are used to express the quantitative amount of a substance in a solution. However, what is the relationship between molarity and normality? The general formula expressing this relationship is : $\mathbf{C_N = Z.\ C_M}$

Where **Z** is a number which depends on the type of reaction.

- **For acid:** Z is the number of H^+ that the acid can lose (HCl : Z = 1), (H_2SO_4 : Z = 2).
- **For base :** Z is the number of OH^- that the base can lose ($NaOH$: Z=1, $BaOH_2$: Z = 2)
- **For oxidation and reduction:** Z is the number of transferred electrons.

  $[MnO_4^- + 5e^- + 8OH^- \longrightarrow Mn^{2+} + 4H_2O] : Z = 5$
- **For salts:** Z is the number of metal atoms in its valence.

  $[Al_2(SO_4)_3 \longrightarrow 2Al^{3+} + 3SO_4^{2-}] : Z = 2 \times (+3) = 6$

4. Objective of the experiment

1. Recognising and using the equipment and tools for preparing solutions.
2. How to prepare a solution from sodium hydroxide (NaOH) by dissolving.
3. How to dilute Hydrochloric acid (HCI) solution.

5. Materials

- Graduated pipette or graduated cylinder
- Volumetric flask
- Spatula - Watch Glass – Funnel
- Analytical balance

6. Chemicals

- Hydrochloric acid (HCl)
- Sodium hydroxide (NaOH)
- Distilled water

7. Experimental Procedure

7.1. Preparation of an aqueous solution from a solid:

This section describes how to prepare a chemical solution using a solid dissolved in a liquid (Figure 1). We need to calculate the mass of the solid required using the equation:

$$\mathbf{m = C_M . \mathit{M} . \mathit{V}}$$

Question. Calculate and describe how to prepare 100 ml of sodium hydroxide (NaOH) solution with a molar concentration of 0.1 mol/L (M_{NaOH} =40 g/mol) ?

1. Calculation of the required mass of NaOH needed to prepare 100 ml of 0.1 mol/L NaOH solution.

$$C_M = \frac{n}{V} = \frac{m}{M.V} \rightarrow m = C_M . M . V$$

$$m_{NaOH}(g) = C_{solution}(\frac{mol}{L}) . M_{NaOH}(\frac{g}{mol}) . V_{solution}\,(L)$$

$$m_{NaOH}(g) = 0{,}1 \times 40 \times 0.1 = 0.4\text{ g}$$

2. Weight the mass of 0.4 g of NaOH.

3. Fill a 100 mL volumetric flask at third full with distilled water.

4. Add 0.4g of NaOH to this volumetric flask using a funnel.

5. Stir the mixture until the NaOH is completely dissolved.

6. Completely fill the volumetric flask with distilled water to the measuring line.

7. Close the volumetric flask, then mix to obtain homogeneous solution of NaOH.

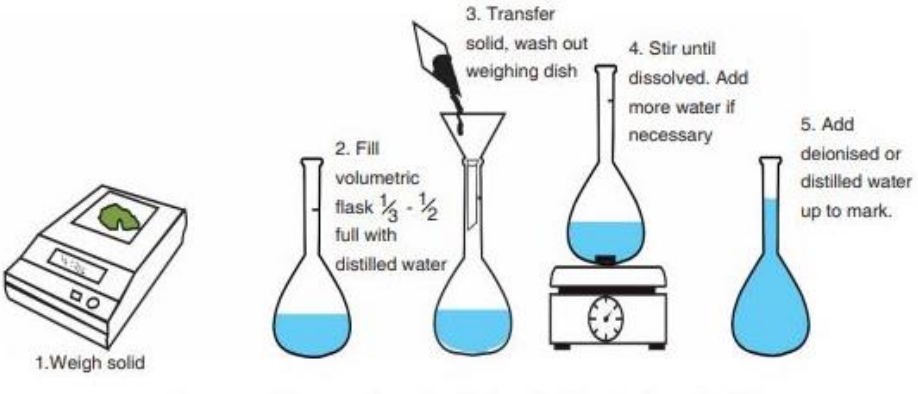


Figure 1. Preparation of an aqueous solution by dissolution of solid

7.2. Preparation of an aqueous solution from a liquid:

This section explains how to prepare a chemical solution through dilution method (figure 2). We need to calculate the volume of the starting solution required using with the equation:

$$C_i \,.\, V_i = C_f \,.\, V_f \rightarrow V_i = \frac{C_f \,.\, V_f}{C_i}$$

Question. Calculate and describe how to prepare 100 ml of 0.1 mol/L hydrochloric acid HCl from concentarted HCl solution ?

1. Read the information on the concentrated HCl(commercial); the density is 1.18, the purity rate is 37%, and the molar mass is 36.5 g/mol.

2. Calculate the mass of the concentrated HCl.

We have a density of 1.18, so the mass is 1.18 kg/L, meaning that one liter of HCl weighs 1180 grams.

3. Determine the mass of pure concentrated HCl in grammes.

The using 37% HCl → the required mass is :

$$m_{HCl}(\text{Concentrated}) = \frac{1180 \times 37}{100} = 436.6 \text{ g}$$

4. Determining the concentration of concentrated HCl.

$$C_{\text{HCl}} = \frac{n_{HCl}}{V} = \frac{\text{m}_{\text{HCl}}}{M_{HCl} . V} = \frac{436.6}{36.6 \times 1} = 11.96 \text{ mol/L}$$

5. Calculating the volume required of concentrated HCl to prepre the needed solution

$$V_{HCl} = \frac{\mathrm{C_f}\,.\,\mathrm{V_f}}{\mathrm{C_{HCl}}} = \frac{0.1 \times 100}{11.96} = 0.83\ \mathrm{mL}$$

6. Fill a clean 100 mL volumetric flask at third of it with water.
7. Take out 0.83 mL of concentrated HCl using the graduated pipette.
8. Transfer it to the volumetric flask.
9. Completely fill the volumetric flask with distilled water to the measuring line.
10. Close the volumetric flask, then mix to obtain homogeneous solution of HCl.

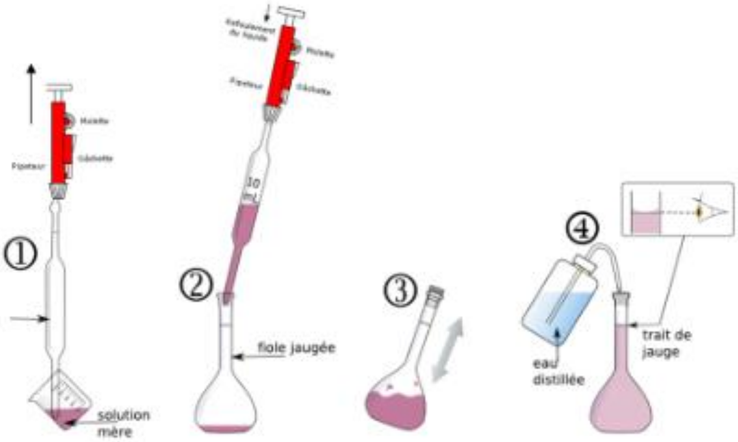


Figure 2. Preparation of solution by diluting

8. Questions

Calculate and describe how to prepare the following solutions :

1. 100 mL of 0.5 M KOH using solid KOH.
2. 200 ml of 0.25N (eq.g/L) H_2SO_4 using concentrated H_2SO_4
3. 250 ml of a 0.5 M CH_3COOH using concentrated CH_3COOH.
4. From the previous solution prepare 150 mL of a 0.1 M solution

Given :

- KOH : Molar mass = 56.11 g/mol, Purity = 85%
- H_2SO_4 : Molar mass = 98 g/mol, Density = 1.84 g/mL, Purity = 96%
- CH_3COOH : Molar mass = 60 g/mol, Density = 1.05 g/mL, Purity = 99%

Calculations

1. Preparation of 100 mL of 0.5 M KOH solution using solid KOH.

The required mass of KOH is calculated as follows :

$$\mathrm{C}_{solution} = \frac{n_{KOH}}{V} = \frac{\mathrm{m_{KOH}}}{M_{KOH} \times V_{solution}} \rightarrow \mathrm{m_{KOH}} = \mathrm{C}_{solution} \times M_{KOH} \times V_{solution}$$

$$\mathrm{m}_{KOH} = 0{,}5 \times 56{,}1 \times 0{,}1 = 2{,}80\ g$$

The using 85% KOH, the mass required is :

$$V_{KOH} = \frac{2.80 \times 100}{85} = 2.29\ g$$

Procedure

To prepare 100 mL of a 0.5 M KOH solution, precisely weigh 2.29 g of KOH using an analytical balance and transfer it into a 100 mL volumetric flask. Add a small amount of distilled water and stir the mixture until the KOH completely dissolves. Then, gradually add more distilled water until the total volume reaches 100 mL. Finally, secure the flask and shake gently to ensure the solution is homogeneous.

2. Preparation of 200 ml of 0.25N (eq.g/L) H_2SO_4 using concentrated H_2SO_4

We first determine the mass of commercial H_2SO_4.

Given that the density is 1.84, so the mass is 1. 84 kg/L, meaning that one liter of H_2SO_4 weighs 1840 grams .

The using 96% The using 37% HCl → the required mass is :

$$m_{\mathrm{H2SO4}}(\mathrm{concentrated}) = \frac{1840 \times 96}{100} = 1766.4\ \mathrm{g}$$

The molar concentration of concentrated H_2SO_4 is calculated as :

$$\mathrm{C_{H2SO4}} = \frac{n_{\mathrm{H2SO4}}}{V} = \frac{\mathrm{m_{H2SO4}}}{M_{\mathrm{H2SO4}}.V} = \frac{1766.4}{98 \times 1} = 18.02\ \mathrm{mol/L}$$

Since H_2SO_4 is diprotic acide

$$\mathrm{Z} = 2 \rightarrow C_M = \frac{C_N}{2}$$

So

$$V_{\mathbf{H2SO4}} = \frac{(\frac{\mathrm{C_N}}{2}).\mathrm{V_f}}{\mathrm{C}_{\mathbf{H2SO4}}} = \frac{(\frac{0.25}{2}) \times 200}{18.02} = 1.38\ \mathrm{mL}$$

Procedure

Using a graduated pipette, measure 1.38 mL of concentrated H_2SO_4 and carefully transfer it into a 200 mL volumetric flask fitted with a funnel. Gradually add distilled water while stirring to ensure proper mixing. Continue adding distilled water until the final volume reaches 200 mL, shaking the flask gently multiple times to ensure homogeneity.

3. Preparation of 250 ml of a 0.5 M CH_3COOH using concentrated CH_3COOH.

First, we determine the mass of commercial CH_3COOH

Given that the density is 1.05, so the mass is 1. 05 kg/L, meaning that one liter of CH_3COOH weighs 1050 grams .

The using 99% CH_3COOH l → the required mass is :

$$m_{\mathrm{CH3COOH}} = \frac{1050 \times 99}{100} = 1039.5 \text{ g}$$

The molar concentration of concentrated CH_3COOH is :

$$\mathrm{C}_{\mathrm{CH3COOH}} = \frac{n_{\mathrm{CH3COOH}}}{V} = \frac{\mathrm{m}_{\mathrm{CH3COOH}}}{M_{\mathrm{CH3COOH}}.V} = \frac{1039.5}{60 \times 1} = 17.32 \text{ mol/L}$$

Since CH_3COOH is a monoprotic acid.

$$\mathrm{Z} = 1 \rightarrow C_N = C_M$$

So

$$V_{\mathrm{CH3COOH}} = \frac{\mathrm{C_f} \, . \, \mathrm{V_f}}{\mathrm{C}_{\mathrm{CH3COOH}}} = \frac{0.5 \times 250}{17.32} = 7.21 \text{ ml}$$

Procedure

To prepare 250 ml of a 0.5 M CH_3COOH, measure 7.21 mL of CH_3COOH using a graduated pipette, and carefully transfer it into a 250 mL volumetric flask. Add distilled water while stirring the solution to ensure proper mixing. Continue adding distilled water until the final volume reaches 250 mL, then shake the flask gently multiple times to achieve a homogeneous solution.

4. Preparation of 150 mL of a 0.1 M solution from the previous solution

$$V_{\mathrm{CH3COOH}} = \frac{\mathrm{C_f} \, . \, \mathrm{V_f}}{\mathrm{C}_{\mathrm{CH3COOH}}} = \frac{0.1 \times 150}{0.5} = 3 \text{ mL}$$

Procedure

To prepare 150 mL of a 0.1 M CH_3COOH solution from the previous solution (solution of 0.5 M of CH_3COOH), measure 3 mL of CH_3COOH using a graduated pipette and carefully transfer it into a 150 mL volumetric flask. Add distilled water while stirring to ensure proper mixing. Continue adding distilled water until the final volume reaches 150 mL, then shake the flask gently multiple times to achieve a homogeneous solution.

Practical Work N° 3. Acid/Basic Titration Using Colorimetric Reagents

1. Introduction

Titration is a quantitative analytical technique used to determine the concentration of an unknown solution by reacting it with a solution of known concentration (titrant). The reaction continues until the equivalence point is reached, which is indicated by a color change, conductivity shift, or precipitate formation.

2. Types of titration

- **Acid-Base Titration :** A method used to determine the concentration of an acid or base through a neutralization reaction.
- **Redox Titration :** A technique based on oxidation-reduction reactions, involving electron transfer, such as the titration of Fe^{2+} with $KMnO_4$.
- **Precipitation Titration :** A method that determines an unknown concentration by forming an insoluble precipitate during the reaction, such as using silver nitrate ($AgNO_3$) to quantify chloride ions in water.
- **Complexometric Titration :** A technique for quantifying metal ions in solution by forming stable complexes, such as determining calcium and magnesium in water using Eriochrome Black T as an indicator.

3. Acid/basic titration using colorimetric reagents

Acid-base titration using colorimetric reagents employs indicators that change color at specific pH levels, providing a visual signal for the equivalence point.

Color indicators are compounds that display a distinct color change at specific pH levels, serving as visual markers to assess a solution's acidity or alkalinity. Usually consisting of weak acids or bases, these indicators undergo reversible chemical reactions in response to pH variations.

$$HInd_{(aq)} + H_2O \rightleftharpoons Ind^{-}_{(aq)} + H_3O^{+}_{(aq)}$$

Where : HInd represents the acidic form of the color indicator, while Ind^- represents its basic form.

4. Principle

The principle of acid-base titration in aqueous solutions is based on the neutralization reaction between an acid and a base. During the titration process, an acid (HA) donates protons (H^+) while a base (BOH) provides hydroxide ions (OH^-). The resulting reaction produces water (H_2O) and a salt (AB).

$$\mathrm{HA} + \mathrm{BOH} \rightarrow \mathrm{AB} + \mathrm{H_2O}$$

The equivalence point occurs when the color changes at the end of the titration, indicating that the moles of acid are equals to the moles of base.

$$n_{Acide} = n_{Base} \Leftrightarrow C_{acide} \cdot V_{acide} = C_{base} \cdot V_{base}$$

$$C_{acide} = \frac{(C_{base} \cdot V_{base})}{V_{acide}}$$

Table 1. Some indicators and their color change range

Indicator	Color change range	Color in acidic state	Color in basic state
Thymol Blue	1.2 - 2.8	Red	Yellow
Methyl Orange	3.1 - 4.4	Orange	Yellow
Phenol Red	6.6 - 8.0	Yellow	Red
Bromothymol Blue	6.0 - 7.6	Yellow	Blue
Phenolphthalein	8.3 - 10	Colorless	Pink

5. Objective of the experiment

1. To familiarize with the tools and equipment used in titration and how to operate them.
2. To learn the procedure for determining the concentration of both a strong acid (HCl) and a weak acid (CH_3COOH) using a strong base (NaOH).

6. Materials

- Burette with stand and clamp.
- Graduated cylinder
- Erlenmeyer flask
- Funnel

7. Chemicals

- HCl solution of unknown concentration
- CH_3COOH with unknown concentration
- 0.1 mol/L of NaOH solution.
- Color reagents (bromothymol blue and phenolphthalein).

8. Experimental Procedure

Experiment 1: Titration of strong acid **HCl** of unknown concentration with a strong base **NaOH** of 0.1 mol/L.

- Begin by filling the burette with a 0.1 mol/L NaOH solution and calibrate it to zero.
- Measure 10 mL of the HCl solution of unknown concentration using a graduated cylinder.
- Transfer it to a 250 mL Erlenmeyer flask.

- Add a few drops of **Bromothymol Blue** indicator to the flask.
- Position the Erlenmeyer flask under the burette and begin the titration.
- When a color change is observed, close the burette valve and record the volume of NaOH used.

Experiment 2: Titration of a weak acid, acetic acid (CH_3COOH), with a strong base, sodium hydroxide (NaOH) at a concentration of 0.1 mol/L.

- Fill the burette with a 0.1 mol/L NaOH solution and calibrate it to zero.
- Use a graduated cylinder to measure 10 mL of the CH_3COOH solution of unknown concentration and transfer it to a 250 mL Erlenmeyer flask.
- Add a few drops of **Phenolphthalein** indicator to the solution.
- Position the Erlenmeyer flask under the burette and begin the titration process.
- Once a color change occurs, close the burette valve and record the volume of NaOH used.

9. Questions

1. Write the chemical equations for each experiment.
2. Determine the molar concentration of HCl and CH_3COOH acids.
3. Calculate the mass concentration of HCl and CH_3COOH acids.
4. Based on the results, is it possible to use different indicator colors for the two experiments? Justify your answers.

Given: Molar mass of acetic acid (CH_3COOH) = 60 g/mol, Molar mass of hydrochloric acid (HCl) = 36.5 g/mol

Calculations

Results: **Experiment 1:** Veq = 10.5mL, **Experiment 2:** Veq = 15.5mL

1. Chemical equations for each experiment.

For HCl :

$$\mathrm{HCl}_{(aq)} + NaOH_{(aq)} \rightarrow \mathrm{NaCl}_{(aq)} + \mathrm{H_2O}_{(l)}$$

For CH_3COOH :

$$\mathrm{CH_3COOH}_{(aq)} + \mathrm{NaOH}_{(aq)} \leftrightarrow \mathrm{CH_3COONa}_{(aq)} + \mathrm{H_2O}_{(l)}$$

2. The molar concentration of HCl and CH_3COOH acids:

At the equivalence point, $n_{acid} = n_{base}$

Since : $C = \frac{n}{V}$

$$n_{Acide} = n_{Base} \Leftrightarrow C_{acide} = \frac{(C_{base}.V_{base})}{V_{acide}}$$

For HCl :

$$C_{HCl} = \frac{(C_{NaOH} . V_{NaOH})}{V_{HCl}} = \frac{0.1 \times 10.5}{10} = 0.105 \text{ N} = 0.105 \text{ M}$$

For CH_3COOH :

$$C_{CH3COOH} = \frac{(C_{NaOH} . V_{NaOH})}{V_{CH3COOH}} = \frac{0.1 \times 15.5}{10} = 0.155 \text{ N} = 0.155 \text{ M}$$

3. The mass concentration of HCI and CH_3COOH acids

Since :

$$T = \frac{m}{V}$$

$$C_M = \frac{n}{V}$$

The mass concentration(T) is calculated using the formula :

$$T = C_M . M$$

For HCl :

$$T_{HCl} = C_M . M = 0.105 .36.5 = 3.832\ g/L$$

For CH_3COOH :

$$T_{CH3COOH} = C_M . M = 0.155 .60.05 = 9.307\ g/L$$

4. Different indicators can be used if their color change range matches the titration curve's inflection point. For example, in the experiment using bromothymol blue, phenol red could be serve as an appropriate alternative due to their similar color change ranges.

Practical Work N° 4. Acid / Basic Titration Using pH meter

1. Introduction

Titration, also referred to as titrimetry or volumetric titration, is a widely used laboratory technique in quantitative chemical analysis. It enables the determination of an unknown solution's concentration by identifying the equivalence point.

2. pH metric titration

In pH-metric titration, the use of an indicator is unnecessary. Instead, a pH meter continuously measures the pH as small increments of base are added to an acid solution. A graph is then plotted, showing pH against the volume of base added. From this graph, the equivalence point can be identified, allowing for the calculation of the acid's molarity.

3. Principle

pH-metric titration operates based on the following principles:

- The initial pH depends on the concentration of the strong acid present at the start. As a strong base is gradually added, the pH increases steadily.
- Before reaching the equivalence point, the pH rises sharply as the acid is neutralized.
- At the equivalence point, the number of moles of acid equals the number of moles of base in a stoichiometric ratio.
- Plotting pH against the volume of added base allows for the determination of the equivalence point.
- Acids that fully dissociate in solution are classified as strong acids, while those that only partially dissociate are considered weak acids.

- For strong acid : $\text{pH} = -log\,C_{HA}$
- For weak acid : $\text{pH} = 1/2(pka - \log C_{HA})$

Where :

- C_{HA}: Acid concentration
- pk_a: Acid Strength, defined by $pk_a = -log_{10}\,k_a$
- k_a: Acid dissociation constant, given by $k_a = \frac{[H^+].[A^-]}{[HA]}$

4. pH Meter Instrument

A pH meter consists of a highly sensitive voltmeter (measuring millivolt-level signals) and a combination electrode or probe. When immersed in a solution, the electrode functions as an electrochemical cell, generating a voltage that depends on the hydrogen ion concentration [H$^+$] By measuring this voltage, the pH of the solution can be determined. Before use, the pH meter must be calibrated with at least two buffer solutions, though using three (pH = 10, pH = 7, pH = 4) is recommended for greater accuracy.

The pH is given by the equation : $\mathrm{pH} = -\log[\mathrm{H_3O^+}]$

5. Objective of the experiment

1. Conduct a pH-metric titration to determine the molarity of an unknown acidic solution.
2. Plot a graph of pH versus the volume of added base to identify the equivalence point.
3. Calculate the molarity of the acid based on the titration data.

6. Materials

- pH meter
- Magnetic stirrer with a stir bar
- Burette with stand and clamp
- Graduated cylinder
- Beaker 100 mL

7. Chemicals

- Hydrochloric acid (HCl) of unknown concentration
- Sodium hydroxide (NaOH) solution (0.1 mol/L)
- Standard buffer solutions (pH 4.0, pH 7.0, and pH 10.0)
- Distilled water

8. Experimental Procedure

1. Fill the burette with a 0.1 mol/L NaOH solution and adjust it to zero.
2. Measure 20 mL of the HCl solution (unknown concentration) using a graduated cylinder.
3. Transfer the measured HCl solution into a 100 mL beaker.
4. Position the beaker under the burette and begin adding NaOH gradually.
5. Stir the solution and record the pH after each addition, ensuring that the magnetic stirrer does not come into contact with the electrode.
6. Record the pH value after waiting few seconds for a stable reading.

9. Questions

1. Record the results obtained in a table.
2. Write the chemical equation for the titration reaction.
3. Plot the curve pH=f (V_b) on graph paper using an appropriate scale.
4. Describe and explain the different regions of the titration curve.
5. Determine the coordinates of the equivalence point from the graph.
6. Calculate the molar concentration of HCI solution.
7. Calculate the mass concentration of HCI.

Calculations

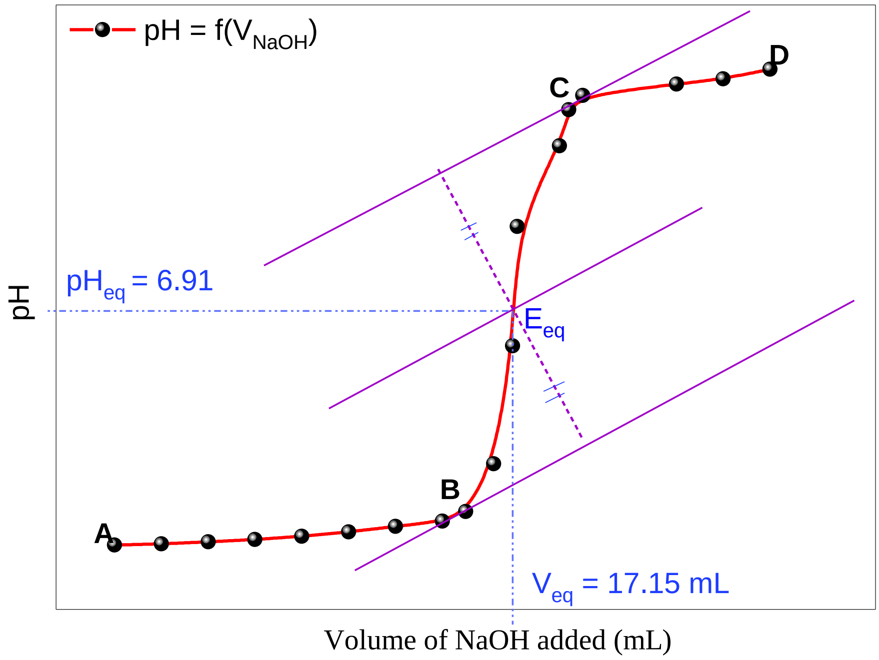
1. The obtained results

V_{NaOH} (*mL*)	0	2	4	6	8	10	12	14	15
pH	2.28	2.30	2.34	2.39	2.45	2.54	2.65	2.75	2.94
V_{NaOH} (*mL*)	16.2	17	17.2	19	19.4	20	24	26	28
pH	3.89	6.23	8.6	10.2	10.92	11.20	11.43	11.53	11.72

2. Equation of titration.

$$HCl + NaOH \rightarrow NaCl + H_2O$$

3. The curve pH=f (V_b)



4. Explanation of the various sections of the graph.

In the titration curve of a strong acid (HCl) with a strong base (NaOH), three distinct regions can be identified.

Before the equivalent point (0 < Vb < 15 ml): The pH decreases rapidly as NaOH is added to HCl. Since HCl fully dissociates into H^+ and Cl^- ions, the pH in this region is primarily controlled by the concentration of H^+ ions. As NaOH neutralizes H^+, the pH drops sharply.

Near the equivalent point (15 < Vb < 20 ml): The pH changes more gradually as stoichiometrically equivalent amounts of acid and base react. At this stage, both conjugate acid (from HCl) and conjugate base (from NaOH) coexist, creating a buffered solution that resists sudden pH changes. The buffer capacity reaches its peak around this point.

After the equivalent point (Vb > 20 ml): Excess NaOH dominates the solution, leading to a sharp increase in pH. The solution becomes increasingly basic as OH^- ions accumulate, making the pH dependent mainly on their concentration.

5. The graph's equivalence point coordinates.

$\mathbf{E_{eq}}$ (pH= 6.91 ; Veq= 17.15)

6. The molar concentration of HCl acid.

At equivalence point we have:

$$n_{acid} = n_{base}$$

$$\frac{n_{Acide}}{1} = \frac{n_{base}}{1} \Leftrightarrow C_{acide} = \frac{(C_{base}.V_{base})}{V_{acide}}$$

$$C_{HCl} = \frac{(C_{NaOH}.V_{NaOH})}{V_{HCl}} = \frac{0.1 \times 17.15}{20} = 0.085 \text{ mol/L}$$

7. The mass concentration of HCl acid.

$$T = \frac{m}{V}$$

$$C_M = \frac{n}{V} \qquad \rightarrow T = C_M.M$$

So

$$T_{HCl} = C_M.M = 0.085\ .36.5 = 3.129\ g/L$$

Practical Work N° 5. Acid / Basic Titration Using Conductometry

1. Introduction

A conductometric titration is an analytical method used to determine the concentration of a solution by measuring changes in its electrical conductivity during the titration process. This technique is based on the gradual replacement of ions in the solution by those from the added reagent, leading to a measurable change in conductivity. The equivalence point is identified by a significant shift in the slope of the curve representing conductivity as a function of the volume of reagent added.

2. Ionic Conductivity

Ionic conductivity refers to the ability of ions to transport an electric current through a solution. It depends on the concentration, mobility, and charge of the ions present. In an electrolytic solution, ions move under the influence of an electric field, contributing to the overall conductivity. Strong electrolytes, such as salts, acids, and bases that fully dissociate in water, exhibit high ionic conductivity, while weak electrolytes, which only partially dissociate, have lower conductivity. The total conductivity of a solution is the sum of the contributions of all present ions and changes during titration based on neutralization or ion replacement reactions.

The unit of conductivity is Siemens per meter (**S/m**) and is commonly represented by the Greek letter sigma (**σ**). It is the inverse of resistance and is described by the following relationship :

$$\boldsymbol{\sigma = \frac{1}{\rho}}$$

Where :

- **σ** : Conductivity (S/m)
- **ρ** = Resistivity of the solution (Ω· m)

3. Principle

The conductometric method is based on measuring the electrical conductivity of a solution during a titration. The conductivity (**σ**) of a solution depends on the concentration and mobility of the ions present. As a reagent is gradually added, it replaces or neutralizes certain ions in the solution, leading to changes in conductivity.

4. Objective of the experiment

1. Perform a conductometric titration to determine the molarity of an unknown solution.
2. Plot the conductivity $\sigma = f\ (V_b)$ to identify the equivalence point.
3. Calculate the molarity of the acid using the titration results.

5. Materials

- Burette with burette stand and clamp
- Graduated cylinder, Cronical flask, Funnel
- Magnetic mixer
- Conductivity measuring device

6. Chemicals

- Hydrochloric acid (HCl) of unknown concentration
- 0.1 mol/L of NaOH solution.
- Distilled water

7. Procedure of the experiment

1. Ensure all equipment is clean before starting.
2. Fill the burette with a 0.1 mol/L sodium hydroxide (NaOH) solution and adjust it to zero.
3. Measure 20 mL of hydrochloric acid (HCl) solution of unknown concentration.
4. Transfer the measured HCl solution into a beaker.
5. Place the beaker on a magnetic stirrer for continuous mixing.
6. Insert the conductivity probe into the solution.
7. Slowly add NaOH from the burette, drop by drop.
8. After each addition, record the conductivity value of the solution (wait a few seconds for the value to stabilize).

8. Questions

1. Record the obtained results in a table.
2. Write the chemical equation for the titration reaction.
3. Plot the graph $\sigma = f\ (V_b)$ on graph paper, selecting an appropriate scale.
4. Determine the coordinates of the equivalence point from the graph.
5. Describe and explain the different regions of the titration curve.
6. Calculate the molar concentration of hydrochloric acid (HCl).
7. Determine the mass concentration of hydrochloric acid.

Given Data: Density (d): 1.18 g/mL, Purity: 37%, Molar mass (M): 36.5 g/mol

Calculations

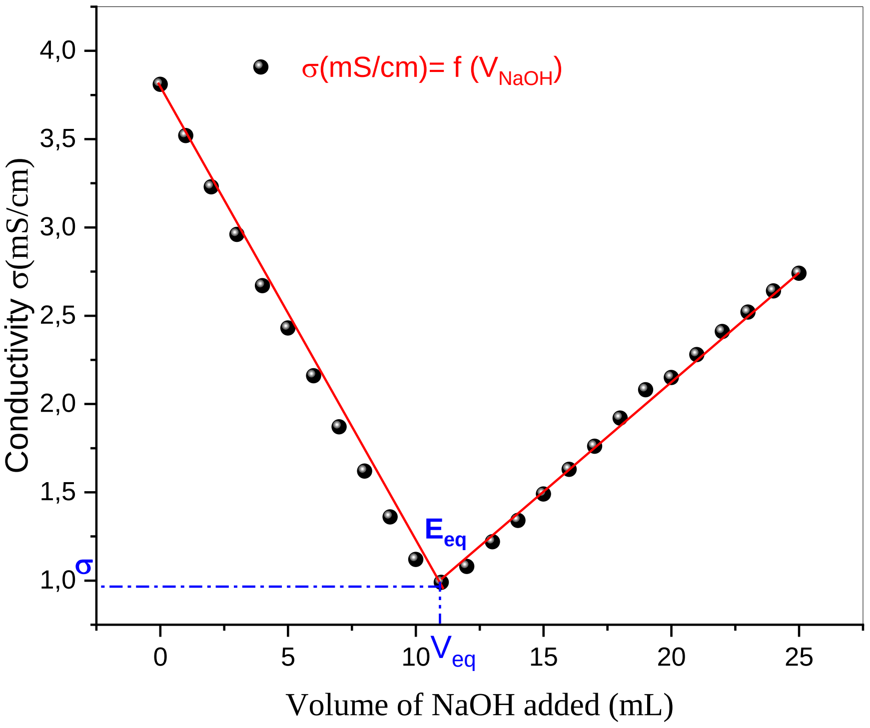
1. The obtained results

V_{NaOH} (*mL*)	0	1	2	3	4	5	6	7	8
σ (m S/cm)	3.81	3.52	3.23	2.96	2.67	2.43	2.16	1.87	1.62
V_{NaOH} (*mL*)	9	10	11	12	13	14	15	16	17
σ (m S/cm)	1.36	1.12	0.99	1.08	1.22	1.34	1.49	1.63	1.76
σ (m S/cm)	18	19	20	21	22	23	24	25	
V_{NaOH} (*mL*	1.92	2.02	2.15	2.28	2.41	2.52	2.64	2.74	

2. The chemical equation for the titration reaction :

$$\mathrm{HCl} + \mathrm{NaOH} \rightarrow \mathrm{NaCl} + \mathrm{H_2O}$$

3. The graph $\sigma = f\ (V_b)$



4. The graph's equivalence point coordinates.

$\mathbf{E_{eq}}$ ($\boldsymbol{\sigma}$ = 0.99 mS/cm, V_{eq} =V_{NaOH} = 11 mL)

5. Explanation of the various sections of the graph.

The titration curve has three main parts.

Before the equivalent point (0 -~ 11 mL): The solution contains more HCl, which is a strong acid. HCl fully dissociates into H^+ and Cl^- ions, making the solution highly conductive. As NaOH is added, its OH⁻ ions react with the H⁺ ions to form water. This reduces the number of free ions, so the conductivity **gradually decreases**.

At the equivalent point (~ 11 ml): All the H⁺ ions have been neutralized by OH⁻ ions. The solution now contains only water and NaCl, which does not contribute much to conductivity. This is the **lowest point** on the graph, indicating that the reaction is complete.

After the equivalent point (Vb > 11 ml): More NaOH is added, increasing the number of OH⁻ ions in the solution. Since OH⁻ ions are highly conductive, the conductivity **increases sharply**. This part of the curve confirms that the acid has been fully neutralized and that excess base is now present in the solution.

6. The molar concentration of hydrochloric acid.

At the equivalence point, the number of moles of acid is equal to the number of moles base added.

$$\frac{n_{Acide}}{1} = \frac{n_{base}}{1}$$

Thus, the molar concentration of HCl is given by :

$$C_{acide} = \frac{(C_{base}.V_{base})}{V_{acide}}$$

So

$$C_{HCl} = \frac{0.1 \times 11}{20} = 0.055 \text{ N}$$

7. The mass concentration of HCl acid.

$$T = \frac{m}{V}$$

$$C_M = \frac{n}{V}$$

So

$$T = C_M.M$$

$$T_{HCl} = C_M.M = 0.055\,.36.5 = 2.007\ g/L$$

Practical Work N° 6. Redox Titration Using Potassium Permanganate

1. Introduction

The redox titration is also known as an oxidation-reduction reaction. In this type of titration, the chemical reaction takes place with a transfer of electrons in the reacting ions of aqueous solutions. The titrations are named after the oxydant that is used in are as follows ;

- Permanganate Titrations.
- Dichromate Titrations.
- Iodometric Titrations.

2. Permanganate Titrations

Potassium permanganate is employed as an oxidizing agent, and this titration must be performed in a strong acid solution, typically sulfuric acid. It is not suitable to conduct this titration in the presence of acids like nitric acid or hydrochloric acid, as they are also oxidizing agents. For example, hydrochloric acid reacts chemically with $KMnO_4$, producing chlorine, which is another oxidizing agent.

The solution containing MnO_4^- ions is purple in color, whereas the solution with Mn^{2+} ions is colorless. As a result, the permanganate solution is decolorized when added to a reducing agent. When $KMnO_4$ reacts with a reducing agent, it acts as its own indicator.

Commercial $KMnO_4$ is considered impure due to contamination with MnO_2, making potassium permanganate solutions unsuitable for use as standard materials. Therefore, it is essential to calibrate these solutions to accurately determine their concentration before use. This can be done by employing substances such as oxalic acid ($H_2C_2O_4$), sodium oxalate ($Na_2C_2O_4$), or potassium cyanide ($K_4[Fe(CN)_6]$). Potassium permanganate solutions are always standardized before being utilized.

3. Objective of the experiment

Experiment 1 : To determine the concentration of the potassium permanganate ($KMnO_4$) solution through titration using the oxalic acid ($H_2C_2O_4$) solution.

Experiment 2 : Use a pre-standardized $KMnO_4$ solution to determine the concentration of iron

4. Materials

- Burette with burette stand and clamp
- Graduated cylinder
- Cronical flask
- Funnel

5. Chemicals

- $KMnO_4$ solution with unknown concentration
- $FeSO_4$ solution with unknown concentration
- 0.1 mol/L $H_2C_2O_4$ solution
- 0.1 mol/L H_2SO_4 solution
- Distilled water

6. Experiment procedure

Experiment 1: Titration of $KMnO_4$ solution with $H_2C_2O_4$ solution

1. Always rinse the materials with distilled water before use.
2. Fill the burette with the potassium permanganate ($KMnO_4$) solution of unknown concentration and set it to zero.
3. Using a graduated cylinder, mesure 10 mL of a 0.1 M oxalic acid ($H_2C_2O_4$) solution.
4. Transfer it into a 250 mL conical flask.
5. Add 20 mL of a 0.1 M sulfuric acid (H_2SO_4) solution to the flask.
6. Incorporate 10 mL of heated distilled water (60 – 90°C).
7. Transfer 0.5 mL of the $KMnO_4$ solution from the burette into the volumetric flask.
8. Heat the mixture until the violet color disappears, indicating the formation of Mn^{2+} ions, et this point, further heating is required.
9. Continue the titration by adding $KMnO_4$ drop by drop until neutralization is reached, indicated by the appearance of a persistent brown color.
10. Record the volume of $KMnO_4$ that was used (V_{KMnO4}).
11. Repeat the experience twice.

Experiment 2: Titration of $FeSO_4$ using the previously titrated $KMnO_4$ solution

1. Refill the burette with a solution of $KMnO_4$ and adjust it to a zero.
2. Using a graduated cylinder, take 10 mL of a 0.1 M solution of $FeSO_4$.
3. Put it into a 250 mL conical flask.
4. Add 20 ml of a 0.1 M sulfuric acid (H_2SO_4) solution.
5. Incorporate 10 mL of distilled water.
6. Continue the titration by adding $KMnO_4$ drop by drop until neutralization is reached, indicated by the appearance of a red color.
7. Record the volume of $KMnO_4$ that was used (V_{KMnO4}).
8. Repeat the experience twice.

7. Questions

Experiment 1

1. Determine the average volume of $KMnO_4$ solution used in the titration.
2. Write the half-reactions involved in the redox process.
3. Give the overall chemical equation for the reaction.
4. Give the number of electrons exchanged during the reaction.
5. Calculate the molar concentration of the potassium permanganate ($KMnO_4$) solution.

Experiment 2

1. Determine the average volume of $KMnO_4$ solution used in the titration.
2. Write the half-reactions involved in the redox process.
3. Write the overall chemical equation for the reaction.
4. Calculate the molar concentration of the iron sulfate ($FeSO_4$) solution.
5. Explain the adding sulfuric acid in the reaction.
6. Justify, why a color indicator was not required in this experiment.

Calculations

Experiment 1

1. The average volume

V_1 = 7.2 mL, V_2 = 6.9 mL → V_{eq} = 7.05 mL

2. The half-reactions involved in the redox process.

$$H_2C_2O_4 \rightarrow 2\mathrm{CO_2} + 2\mathrm{H^+} + 2\mathrm{e^-} \qquad \text{Oxidation}$$

$$MnO_4^- + 8\mathrm{H^+} + 5\mathrm{e^-} \rightarrow \mathrm{Mn^{2+}} + 4\mathrm{H_2O} \qquad \text{Reduction}$$

3. The overall chemical equation for the reaction.

$$5H_2C_2O_4 + 2MnO_4^- + 6\mathrm{H^+} \rightarrow 10\mathrm{CO_2} + 2\mathrm{Mn^{2+}} + 8\mathrm{H_2O}$$

4. The number of exchanged electrons are : **10 e^-**

5. The molar concentration the potassium permanganate ($KMnO_4$) solution.

At the equivalence point

$$\frac{n_{H2C2O4}}{5} = \frac{n_{KMnO4}}{2} \Leftrightarrow C_{KMnO4} = \frac{2}{5} \cdot \frac{(C_{H2C2O4} \cdot V_{H2C2O4})}{V_{KMnO4}}$$

$$C_{KMnO4} = \frac{2}{5} \cdot \frac{(0.1 \times 7.05)}{10} = 0.028 \ \mathrm{mol/L}$$

Experiment 2

1. The average volume

V_1 = 4 mL, V_2 = 4.3 mL → V_{eq} = 4.15 mL

2. The half-reactions involved in the redox process.

$$\mathrm{Fe^{2+}} \rightarrow \mathrm{Fe^{3+}} + \mathrm{e^-} \qquad \text{Oxidation}$$

$$\mathrm{MnO_4^-} + \mathrm{8H^+} + \mathrm{5e^-} \rightarrow \mathrm{Mn^{2+}} + \mathrm{4H_2O} \qquad \text{Reduction}$$

3. The overall chemical equation for the reaction.

$$\mathrm{MnO_4^-} + \mathrm{8H^+} + \mathrm{5Fe^{2+}} \rightarrow \mathrm{Mn^{2+}} + \mathrm{5Fe^{3+}} + \mathrm{4H_2O}$$

4. The molar concentration of the iron sulfate solution

At the equivalence point

$$\frac{n_{FeSO4}}{5} = \frac{n_{KMnO4}}{1} \Leftrightarrow C_{FeSO4} = 5\,.\frac{(C_{KMnO4}.V_{KMnO4})}{V_{FeSO4}}$$

$$C_{FeSO4} = 5\,.\frac{(0.028 \text{ x } 4.15)}{10} = 0.01\; mol/L$$

5. Sulfuric acid is added to provide hydronium ions, which help acidify the medium and accelerate the reduction of potassium permanganate.

6. A color indicator was not needed because potassium permanganate has natural violet color. The endpoint of the titration was identified by the persistence of the violet color, indicating the completion of the reaction.

Practical Work N° 7. Estimation of Hardness of Water

1. Introduction

Water hardness, often represented in degrees of hardness (°TH), measures the concentration of dissolved minerals, primarily calcium (Ca^{2+}) and magnesium (Mg^{2+}) ions, in water. The hardness of water is typically measured in degrees *French* (°TH). Water hardness in °TH is classified into four categories: soft water (0 to 7 °TH) with low levels of calcium and magnesium, moderately hard water (7 to 15 °TH) with moderate amounts, hard water (15 to 30 °TH) with high concentrations, and very hard water (above 30 °TH) with very high levels of calcium and magnesium. Water hardness is classified into two types, temporary and permanent.

- Temporary hardness

This hardness is called "temporary" because it can be removed by boiling the water and then filtering out the resulting precipitate. It is caused by the presence of bicarbonate salts, mainly calcium carbonate **($CaCO_3$)** and magnesium carbonate **($MgCO_3$)**, which are dissolved in the water.

- Permanent hardness

This type of hardness results from the presence of non-bicarbonate minerals, such as calcium sulfate (**$CaSO_4$**) and magnesium chloride (**$MgCl_2$**), which cannot be eliminated by boiling.

2. Measuring water hardness

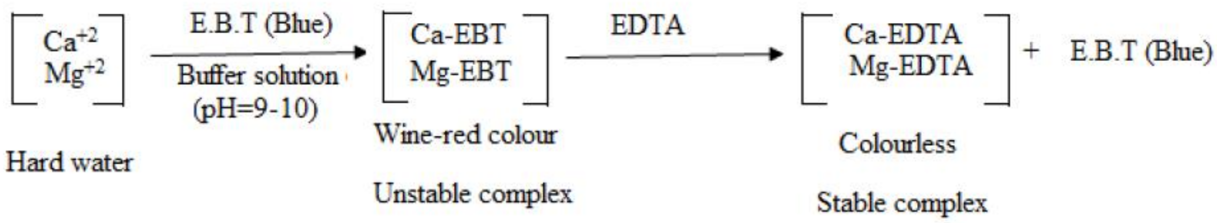
Measuring water hardness involves determining the total hardness, which is the combined concentration of temporary hardness and permanent hardness. The total hardness is typically expressed in milligrams per liter (mg/L) as an equivalent of calcium carbonate ($CaCO_3$)**.**

3. Principle

EDTA **(**Ethylenediamine Tetra Acetic Acid) [H_4Y] forms colorless stable complexes with $Ca^{2+}$and Mg^{2+} ions present in water at pH = 9-10. To maintain the pH of the solution at 9-10, buffer solution ($NH_4Cl + NH_4OH$) is used. Eriochrome Black-T (E.B.T) is used as an indicator. The sample of hard water must be treated with buffer solution and EBT indicator which forms unstable, wine-red colored complexes with Ca^{2+} and Mg^{2+} present in water as follow :

$$Ca^{2+} + \; + Y^{4-} \rightarrow [CaY]^{2-} + 2H^{+} \qquad (1)$$

$$Mg^{2+} + Y^{4-} \rightarrow [MgY]^{2-} + 2H^{+} \qquad (2)$$



4. Objective of the experiment

To estimate the amount of total hardness present in the given sample of water by EDTA titration method.

$$ppm\ (\frac{mg}{L}) = \frac{C_{\mathrm{EDTA}} \cdot V_{\mathrm{eq}}}{V_{\mathrm{H_2O}}}$$

5. Materials

- Burette with burette stand and clamp
- 250 ml Conical flask
- Beaker 250 ml
- Graduated cylinder
- Glass funnel

6. Chemicals

- 0.01mol/L EDTA solution
- Buffer solution (NH_4OH+NH_4CI), pH ≈10
- Eriochrome Black –T

7. Experimental Procedure

Experiment 1 :

1. Clean all equipments.
2. Measure 100 mL of water using a graduated cylinder.
3. Transfer the measured water into a 250 mL beaker.
4. Heat the water until a white precipitate appears.

Experiment 2 :

1. Fill the burette with a 0.01 mol/L EDTA solution and set it to zero.
2. Measure 50 mL of water using a graduated cylinder.
3. Transfer the measured water into an Erlenmeyer flask.
4. Add 5 mL of buffer solution ($NH_4OH + NH_4Cl$) to the flask.
5. Introduce a small amount of solid Aerochrome Black colored reagent.

6. Place the Erlenmeyer flask under the burette and start the titration by adding EDTA drop by drop while stirring.
7. Stop the titration when the color changes from red to blue.
8. Record the volume of EDTA solution added as Veq.

Experiment 3 :

1. Fill the burette with a 0.01 mol/L EDTA solution and calibrate it to zero.
2. Filter the white precipitate to separate it.
3. Measure 50 mL of the filtered water.
4. Transfer the filtered water into an Erlenmeyer flask.
5. Add 5 mL of buffer solution ($NH_4OH + NH_4Cl$) to the flask.
9. Add a small amount of Aerochrome Black colored reagent.
10. Place the Erlenmeyer flask under the burette and the titration by adding EDTA solution drop by drop by drop while stirring.
6. Stop the titration when the color changes from red to blue.
7. Record the volume of EDTA solution added as V'eq.

8. Questions

1. Write the equations for the reaction of EDTA with Ca^{2+} and Mg^{2+}ions :

2. Calculate the total hardness.

3. Calculate the permanent hardness.

4. Deduce the temporary hardness.

Calculations

1. The obtained volumes

Experiment	V_1 (mL)	V_2 (mL)	Veq (mL)
1	8.8	9.2	9
2	4.6	4.1	4.35

2. The equations for the reaction of EDTA with Ca^{2+} and Mg^{2+}ions

$$\mathrm{Ca^{2+} + H_2Y^{2-} \rightarrow CaY^{2-} + 2H^+}$$

$$\mathrm{Mg^{2+} + H_2Y^{4-} \rightarrow MgY^{2-} + 2H^+}$$

3. The total hardness

The total hardness is given by the formula

$$C \,.\, V_{\mathrm{H2O}} = C_{\mathrm{EDTA}} \,.\, V_{\mathrm{EDTA}} \rightarrow C = \frac{C_{\mathrm{EDTA}} \,.\, V_{\mathrm{EDTA}}}{V_{\mathrm{H2O}}}$$

Where :

$$C = [Ca^{2+}] + [Mg^{2+}]$$

$$C = \frac{0.1 \times 9}{50} = 0.018\, mol/L$$

Thus, the total concentration of Ca^{2+} and Mg^{2+}ions is :

$$[Ca^{2+}] + [Mg^{2+}] = 0.018\, mol/L$$

4. The permanent hardness.

At the equivalence point , the permanent hardness is calculated using the formula :

$$C = \frac{C_{\mathrm{EDTA}} \,.\, V_{\mathrm{EDTA}}}{V_{\mathrm{H2O}}}$$

Where :

$$C = [Ca^{2+}]$$

$$C = \frac{0.1 \times 4.35}{50} = 8.7.10^{-3}\, mol/L$$

5. The temporary hardness

The temporary hardness is given by:

$$C = [Ca^{2+}] + [Mg^{2+}]$$

Thus :

$$[Mg^{2+}] = C - [Ca^{2+}]$$

$$[Mg^{2+}] = 0.018 - 8.7.10^{-3} = 9.3\,.\,10^{-3}\, mol/L$$

Practical Work N° 8. Determination of Chloride by the Mohr method

1. Introduction

Chloride ions are significant anions commonly found in natural water sources. When combined with sodium ions, they form sodium chloride, imparting a salty taste to water. Chloride salts are highly soluble in water. Elevated concentrations of chloride ions can have toxic effects. Wastewater typically contains higher chloride levels compared to distributed water, as the human body excretes ingested chlorides from food. Therefore, an increase in chlorine content in drinking water can sometimes indicate sewage leakage. The recommended concentration of chlorides in water intended for human consumption is 25 mg/L.

2. Mohr's Method

Developed by the German chemist Karl Friedrich Mohr, this technique is used to determine the concentration of ions such as Cl^-, Br^-, and CN^- through precipitation titrations, specifically using silver measurement methods. It is an important method for quality control and detecting commercial adulteration in drinking water.

3. Principle

The Mohr method uses chromate ions as an indicator in the titration of chloride ions with a silver nitrate standard solution. After all the chloride has been precipitated as white silver chloride, the first excess of titrant results in the formation of a silver chromate precipitate, which signals the end point (1). The reactions are :

$$Ag^+ + \mathrm{CI}^- \leftrightarrow \mathrm{AgCl(s)}$$

$$2Ag^+ + CrO_4^{2-} \leftrightarrow \mathrm{Ag_2CrO_4\ (s)}$$

4. Objective of the experiment

The objective of this titration is to determine the concentration of chloride ions in water using a silver nitrate solution through Mohr's method.

$$n_{\mathrm{AgNO_3}} = n_{\mathrm{NaCl}} \rightarrow C_{\mathrm{AgNO_3}} \cdot V_{\mathrm{AgNO_3}} = C_{\mathrm{NaCl}} \cdot V_{\mathrm{NaCl}}$$

$$C_{\mathrm{NaCl}} = \frac{C_{\mathrm{AgNO_3}} \cdot V_{\mathrm{AgNO_3}}}{V_{\mathrm{NaCl}}}$$

5. Materials

- Burette with stand and clamp
- 250 ml Conical flask
- Graduated cylinder
- Glass funnel

6. Chemicals

- 0.01mol/L $AgNO_3$ solution
- 10 % K_2CrO_4 solution
- Water

7. Experimental Procedure

1. Fill the burette with a 0.01 mol/L silver nitrate ($AgNO_3$) solution and set it to zero.
2. Measure 50 ml of tap water using graduated cylinder.
3. Transfer the measured water into an Erlenmeyer flask.
4. Add 2 to 3 drops of a 10% potassium chromate (K_2CrO_4) solution as an indicator.
5. Position the Erlenmeyer flask under the burette and start the titration by adding $AgNO_3$ solution drop by drop while continuously shaking.
6. When a permanent red precipitate appears that does not disappear with shaking, close the burette valve and record the volume of silver nitrate used, let it be V_{AgNO3}.
7. Repeat the experiment and calculate the average volume of $AgNO_3$ used.

8. Questions

1. Determine the average volume of $AgNO_3$.
2. Give chemical reactions.
3. Determine the molar concentration of chloride ions in the water.
4. Calculate the mass concentration of chloride ions in the water in mg/L.
5. Is this water suitable for human consumption and provide reasons.

Calculations

1. The average volume : $V_1 = 6.5$ mL, $V_2 = 6.7$ mL → $V_{eq} = 6.6$ mL

2. The chemical reactions

$$\mathrm{AgNO_3} + \mathrm{NaCl} \rightarrow \mathrm{NaNO_3} + \mathrm{AgCl} \quad (\text{White precipitate})$$

$$2\mathrm{AgNO_3} + K_2\mathrm{CrO_4} \rightarrow \mathrm{Ag_2CrO_4} + 2\mathrm{KNO_3} \quad (\text{Red precipitate})$$

3. The molar concentration of chloride ions in the water.

At the equivalence point

$$n_{Cl^-} = n_{Ag^+} \rightarrow C_{Cl^-} \times V_{Cl^-} = C_{Ag^+} \times V_{Ag^+} \rightarrow C_{Cl^-} = \frac{(C_{Ag^+} \times V_{Ag^+})}{V_{Cl^-}}$$

$$C_{Cl^-} = \frac{(C_{Ag^+} \times V_{Ag^+})}{V_{Cl^-}} = C_{Cl^-} = \frac{(0.01 \times 6.6)}{50} = 0.00132\, mol/L$$

4. The mass concentration of chloride ions in the water in mg/L.

$$T_{Cl^-} = M.C_{Cl^-} = 35.5 \times 0.00132 = 0.04686\frac{g}{L} = 46.86\ mg/L$$

5. This water is not safe for drinking as the chlorine concentration exceeds 25 mg/L.

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