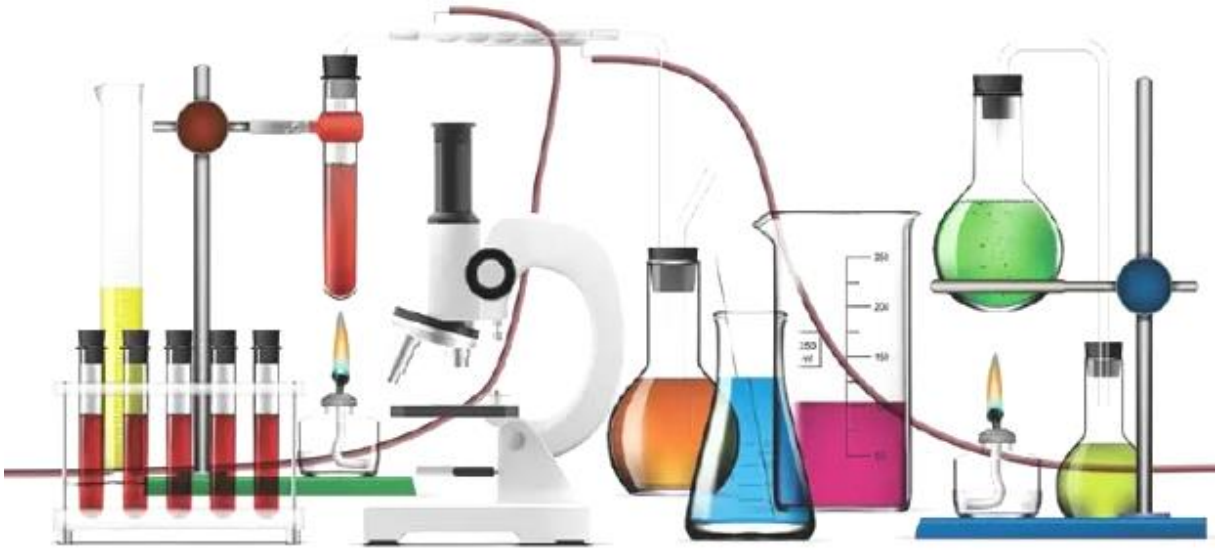
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Faculty of Sciences
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Chemistry 1

Practical Manual



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Introduction

This laboratory manual is designed for first-year undergraduate students enrolled in the common core program and constitutes an essential pedagogical resource for the execution of fundamental experiments in the chemistry laboratory. It provides a structured and progressive introduction to laboratory practices and experimental methodologies, ensuring a coherent integration of theoretical chemistry concepts with hands-on laboratory experience.

A primary objective of this manual is to establish rigorous laboratory safety practices in accordance with chemical laboratory standards. Students are trained in the identification and evaluation of chemical, physical, and procedural hazards, as well as in the proper handling, storage, and disposal of chemical reagents. Emphasis is placed on the correct use of personal protective equipment, safe manipulation of laboratory apparatus, and the adoption of responsible behavior to minimize risks during experimental work.

The manual further aims to develop a solid understanding of basic quantitative concepts in chemistry, including molar concentration, mass concentration, density, and volume, by integrating theoretical principles with hands-on experimentation.. These concepts are explored through calculations, solution preparation, and quantitative measurements using standard laboratory equipment such as volumetric flasks, pipettes, burettes, and analytical balances.

In addition, this manual introduces students to standard analytical techniques commonly employed in chemical laboratories, particularly volumetric analysis. Special attention is given to titration methods, including the identification of reaction endpoints through the observation of color changes and other visual indicators. Accurate reading of volumetric graduations during liquid sampling, transfer, and titration procedures is emphasized to ensure consistency and compatibility of experimental results.

Within this pedagogical framework, practical laboratory sessions serve as an indispensable complement to lectures and tutorial classes, allowing students to apply theoretical knowledge and develop essential technical skills. Each laboratory session spans three hours and requires students to perform experiments, record observations, analyze data, and interpret results using appropriate scientific reasoning. Student performance is evaluated through a written laboratory report completed during a 30-minute assessment period at the end of each session.

Practical Work N°1: Safety Rules and Introduction to Chemistry Lab Work

1. Introduction

Chemistry laboratories can be hazardous if the safety protocols are not adhered to. During a chemistry course a student may handle substances which are toxic, flammable, explosive, carcinogenic, and also some of these materials and equipment may also cause severe burns and cuts if handled improperly or carelessly. Most accidents that occur in the chemistry laboratory are a result of negligence, impatience, unauthorized experimentation, and a lack of adherence to safety rules or proper operating procedures. In order to minimize the likelihood of accidents occurring in the laboratory, it is essential to follow specific rules and regulations at all times when working in the chemistry laboratory.

2. Outcomes

At the end of this practical work, the student will be able to:

- **Recognize** the safety instructions and protocols designed to reduce the risks while working in the laboratory.
- **Identify** the hazards associated with chemical products and conduct chemical operations in the safest possible environment.
- **Understand** the proper uses of various types of glassware and their respective functions.
- **Develop** practical skills and gain hands-on experience in handling glassware and measuring instruments safely and effectively.

3. Laboratory Rules

1. Do not perform unauthorized experiments or work in a laboratory alone.

2. Approved eye protection must be worn at all times in the laboratory. Eye protection must be splash proof chemical goggles and be approved by your instructor. If you do get a chemical in your eye rinse immediately with large quantities of water using the eye-wash stations.

3. Long hair and loose clothing must be confined while in a laboratory.

4. All students must wear lab coats at all times. Your legs must be completely covered below the knee by your lab coat.

5. Closed shoes with socks must be worn at all times – open-toed shoes, backless shoes, sling backs, clogs, and sandals are not permitted.

6. Know the location and proper use of fire extinguishers, fire blankets, safety showers, eye wash devices, and first aid kits.

7. Before obtaining any chemicals carefully read the label on the reagent bottles.

8. Eating, smoking, chewing and drinking are not allowed in a chemistry laboratory.

9. Thoroughly wash your hands after leaving the laboratory.

10. Use the fume hoods when toxic or irritating vapors are involved.

11. Mouth suction is never used to fill a pipette.

12. Never force glass tubing through cork or rubber stoppers without proper lubrication.

13. Never direct the open end of test tube toward yourself or anyone else.

14. Never pour water into concentrated acid.

15. Learn the proper procedure for igniting and operating a laboratory burner. Always extinguish the flame when the burner is not being used. Make sure that all flammable reagents are well removed before lighting the burner.

16. Liquid and solid waste containers must be properly used at all times.

17. Never place chemicals directly on balance pan. Always use a proper weighing container when using a balance to weigh a chemical. Never pour chemicals directly over the balance.

18. Never return unused chemicals to their original container.

19. Securely replace lids, caps, and stoppers after removing reagents from containers.

20. Always wipe spatulas clean before and after inserting into reagent bottles.

21. Report any accident and/or injury, however minor, to your instructor immediately.

22. Never place anything that is not directly required for the experiment on laboratory desks; other items may interfere with the experiment.

23. All personal belongings should be placed in the bookcases as you enter chemistry laboratory.

24. Clean up any spill immediately.

25. Before leaving the laboratory, make sure your work area is clean and dry. Ensure that all gas, water, vacuum, and air valves are completely turned off.

26. Your instructor is available for any assistance you may need. Never hesitate to ask questions especially if there is any question concerning proper operating procedure. Be sure that you understand every instruction before proceeding.

27. In any emergency, to get the lab supervisor attention is to SCREAM!

28. If an acid is to be diluted, pour acid slowly into the water with constant stirring. Never add water to acid.

29. Students should use their own calculator. Mobile phones cannot be used as calculator.

30. Be familiar with the following terms and their effects.

4. Collective and Personal Protective Laboratory Equipments

Laboratory safety requires a combination of Collective Protective Equipment (CPE) to secure the environment and Personal Protective Equipment (PPE) to protect individuals. Key PPE includes lab coats, gloves, and eye protection (goggles/shields), while crucial CPE involves fume hoods, biosafety cabinets, and emergency showers/eyewash stations, collectively forming a mandatory defense against chemical, biological, or physical hazards.

4.1. Collective Protective Equipment (CPE)

CPE protects all personnel in the lab by isolating hazards at the source.

- **Chemical Fume Hoods:** Ventilation systems that remove toxic fumes and vapors.
- **Fire Extinguishers & Fire Blankets:** Strategically placed for immediate fire control.
- **Radiation Shields:** Used for protection in labs working with radioactive materials.
- **Biological Safety Cabinets (BSCs):** Essential for handling infectious agents to prevent aerosol release.

- **Emergency Showers and Eyewash Stations:** Immediate decontaminating devices for accidental exposure.

4.2. Personal Protective Equipment (PPE)

PPE is the last line of defense worn by individuals to minimize exposure to hazards.

- **Lab Coats**: Must be worn, preferably 100% cotton coat for protection against the fire and the spills.
- **Leg Coverage**: Long pants or ankle-length skirts that cover the leg to the top of the shoe, avoiding exposed ankles.
- **Footwear**: Closed-toe, closed-heel shoes (no sandals, clogs, or mesh tops).
- **Eye Protection**: Safety glasses with side shields or chemical splash goggles are mandatory for protecting against spills and flying particles.
- **Hair & Accessories**: Long hair must be tied back; dangling jewelry, loose clothing, or scarves are forbidden.
- **Gloves**: Appropriate gloves should be worn when handling hazardous materials and removed before leaving the lab.
- **Respiratory Protection:** Masks or respirators for protection against hazardous vapors or airborne particles.

5. General Laboratory Procedures

The following procedures will help you use your time efficiently and will help minimize the waste of chemicals and other supplies. Other techniques will be described to you as needed in later experiments.

- **Cleaning glassware**: Scrub inside and out with a brush, detergent, and tap water. Rinse away all suds with tap water. Rinse the inside of the glassware two or three times with small amounts of distilled water. Shake out as much rinse water as possible and dry the outside with a towel. If dry glassware is immediately needed, rinse it twice with small amounts of acetone. The residual acetone in the equipment will vaporize quickly leaving no residue.

- **Disposal of used materials**: Used chemicals and other materials must be disposed of appropriately. Some used chemicals can be flushed down the sink drain with water.

Most used chemicals should be collected in labeled containers located in the lab. Your instructor or other qualified individuals will then ensure the proper disposal of the collected materials (never return excess chemical to the original container).

6. Chemical Hazards & Key Information

Laboratory chemical hazards involve substances that cause injury, illness, or damage through inherent properties like toxicity, flammability, or corrosivity. Key safety protocols include consulting Safety Data Sheets (SDS), using personal protective equipment (PPE), proper labeling, and understanding hazard symbols. Hazards range from acute toxins and carcinogens to reactive materials.

Health Hazards: Include toxins, carcinogens, reproductive toxins, sensitizers, and irritants that can enter the body via inhalation, ingestion, or skin absorption.

Physical Hazards: Flammable liquids/gases, oxidizers, combustibles, explosives, and water-reactive substances.

Corrosives: Acids and bases that cause severe skin burns and eye damage.

Information Resources: Safety Data Sheets (SDS) affixed to chemical products containers provide crucial details on toxicity, safe handling, storage, and emergency rules for chemical products. It has the role of informing users about the hazardous properties and must include:

- The name of the substance as well as its formula

- The dangers symbols (**pictograms**)

- One or more risk phrases and safety tips

- Some physicochemical properties and other indications.

7. Symbols used on Labels (Pictograms)

The handling of chemical species is not always safe for users but also for environment. The manufacturers therefore indicate on each chemical product bottle drawings which are called pictograms to indicate the different dangers.

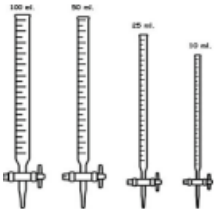
Symbol	Pictogram	Name General Meaning
	**Explosive**	These chemicals can explode.
	**Flammable**	These chemicals burn or can release gases that burn.
	**Oxidizing**	These chemicals give off oxygen and can make a fire spread.
	**Corrosive**	These chemicals cause permanent damage to skin or eyes. These chemicals destroy metals.
	**Acute toxicity or toxic**	These chemicals are poisons that quickly cause sickness or death. A toxin may attack one or more parts of the body, such as the liver, kidneys, nerves, lungs, skin, eyes, or bone.
	**Hazardous to the environment**	These chemicals are dangerous if they get into rivers, lakes or oceans
	**Health hazard/Hazardous to the ozone layer**	These chemicals cause health problems. Usually less toxic than Acute toxicity pictogram. This pictogram is also used for chemicals that can destroy the ozone layer.
	**Serious health hazard**	These chemicals cause serious health problems. Some problems show up immediately, but some may show up much later.

8. Product Risk Phrases, Safety Phrases and Hazard Statements

Product risk (R), safety (S), and hazard statements are standardized codes used to label hazardous chemicals, informing users of dangers and necessary precautions.

- **R-phrases (Risk):** Indicated dangers like flammability, toxicity, or environmental risks (e.g., R11: Highly flammable; R50: Very toxic to aquatic organisms).
- **S-phrases (Safety):** Provided safety advice for handling, storage, and first aid (e.g., S2: Keep out of reach of children; S36: Wear suitable protective clothing).
- **H-phrases (Hazard Statements):** these describe the nature of hazards, including physical (H2xx), health (H3xx), and environmental (H4xx) risks (e.g., H222: Extremely flammable aerosol; H314: Causes severe skin burns; H410: Very toxic to aquatic life with long-lasting effects).

9. List of Glassware and Equipments commonly used in Chemical Laboratory

1-Test tube 	**2- Test tube holder** 	**3- Test tube stand** 	**4- Beaker** 
5- Erlenmeyer flask 	**6- Volumetric flask** 	**7- Graduated cylinder** 	**8- Graduated pipette** 
9- Burette 	**10- Burette clamp** 	**11- Funnel** 	**12- Petri dish** 

13- Glass rod 	**14- Wash Bottle** 	**15- Spatula** 	**16-Round-bottom flasks** 
17- Glass Condenser 	**18- Filter paper** 	**19- Separatory funnel** 	**20- Watch glass** 
21- Mortar and pestle 	**22-Analytical balance** 	**23- Magnetic Stirrer** 	**24- Pipette bulbs** 
25-A rotary evaporator 	**26- Oven** 	**27- pH meter** 	**28- fume hood** 

1- A test tube is a common piece of laboratory glassware consisting of glass or clear plastic tubing, open at the top and closed at the bottom, used for chemical test.

2- A test tube holder is used to hold hot test tubes.

3- Test tube stand is used to hold several test tubes together.

4- Beakers are used as containers. They are available in a variety of sizes.

5- Erlenmeyer flasks are often used in chemistry reactions, particularly in titrations. As with beakers, the volume markings should not be considered accurate.

6- Volumetric flasks are used to measure and store solutions with a high degree of accuracy. These flasks generally possess a marking near the top that indicates the level at which the volume of the liquid is equal to the volume written on the flask.

7- Graduated cylinders are used to measure and transfer liquids with a moderate level of accuracy.

8- A graduated pipette, it is used for accurately measure and transfer a volume of liquid from one container to another.

9- Burettes are devices used typically in analytical, quantitative chemistry applications for measuring liquid solution. Burettes are used heavily in titration experiments.

10- Burette clamp is scientific equipment which used specifically to hold and secure a burette on a stand to be more convenient for the experiment.

11- The funnel is a tube that is wide at the top and narrow at the bottom. It is used to direct liquid or powder into a small opening. It can be used for filtration by adding filter paper.

12- A petri dish is a small dish shaped like a cylinder, used for cultivating microorganisms.

13- A glass rod is a piece of laboratory equipment used to mix chemicals and liquids for laboratory purposes. They are usually made of solid glass, thick and long, with rounded ends.

14- A wash bottle is a squeeze bottle with a nozzle used to rinse various laboratory glassware, such as test tubes and round-bottom flasks. Wash bottles are sealed with a screw-top lid.

15- Laboratory spatula made of stainless steel, used for measuring solids, moving objects and scraping materials from beaker

16- Round-bottom flasks are types of flasks having spherical bottoms used as laboratory glassware, mostly for chemical or biochemical work.

17- Condenser is a glass device used for condensation (changing the physical state of a substance from its gaseous state to its liquid state). In chemistry laboratory, condensers are used in the extraction of essential oils.

18- Filter paper is a semi-permeable paper used in science chemical laboratory to remove solids from liquids.

19- A separation funnel is a piece of laboratory glassware used in liquid-liquid extractions to separate the components of a mixture into two immiscible solvent phases of different densities.

20- A watch glass is a circular concave piece of glass used in chemistry to hold solids while being weighed and as a cover for a beaker.

21- Mortar and pestle are used since ancient times to prepare ingredients or substances by crushing them into a fine paste or powder in the laboratory.

22- Analytical balance is a highly sensitive laboratory instrument used in chemical analysis to measure the mass of chemicals and objects accurately and precisely up to 0.0001 grams.

23- A magnetic stirrer or a magnetic mixer is a laboratory device that employs a rotating magnetic field to cause a stir bar immersed in a liquid to spin very quickly, thus stirring it. It is used in chemistry and biology as a convenient way to stir small volumes of solutions.

24- Pipette bulbs or rubber bulbs, are used in chemistry laboratories, by placing them on top of a pipette. It serves as a vacuum source for filling reagents.

25- A rotary evaporator is a piece of equipment primarily used to remove solvent from a sample through “evaporation under reduced pressure”. The presence of reduced pressure in the apparatus causes the solvent to boil at a lower temperature than normal.

26. An oven is an essential laboratory equipment that *uses to dry heat (hot air*) to sterilize laboratory objects and samples.

27. A pH meter is an essential, highly accurate electronic instrument used in chemistry to measure the hydrogen ion activity in water-based solutions, determining their acidity or alkalinity on a 0-14 pH scale.

28. A fume hood is an essential safety enclosure that protects laboratory personnel from hazardous vapors, gases, dust, and aerosols by using continuous airflow to extract contaminants, exhausting them outside the building.

10. Laboratory Glassware Classification

Laboratory glassware is primarily classified by accuracy (Class A/B), function (volumetric, container, specialized), and material, with high-quality borosilicate glass used for thermal/chemical resistance. Class A is for precise, certified work (e.g., volumetric flasks, volumetric pipettes, and graduated pipettes), while Class B is for general-purpose, lower-accuracy tasks. Key types include beakers, Erlenmeyer flasks, graduated cylinders, burettes, and pipettes.

Classification by Accuracy (Tolerance)

Class A: Highest accuracy and precision, often serialized for traceability, used in analytical labs.

Class B: General-purpose, lower accuracy (tolerance is usually twice that of Class A). Ideal where precise volume is not critical.

Classification by Function

Volumetric (Measuring) Glassware: Used for precise measurements of liquids. Examples: Volumetric flasks, pipettes, burettes, and graduated cylinders.

Containers/General Purpose: Used for mixing, heating, and storing. Examples: Beakers, Erlenmeyer flasks, test tubes, round-bottom flasks.

Specialized/Support: Used for specific processes like distillation, filtration, or storage. Examples: Condensers, funnels, Petri dishes, desiccators, separating funnels.

Classification by Material

Borosilicate Glass: Highly resistant to thermal shock and chemical corrosion (e.g., Pyrex).

Soda-Lime Glass: Less resistant to heat, typically used for disposable or low-cost items.

11. How to take Measurements with a Meniscus:

To take accurate measurements with a meniscus, place the measuring container on a stable, flat surface and position your eyes directly level with the liquid surface to avoid parallax error. Read concave, downward-curving liquids (like water) at the lowest point, and convex, upward-curving liquids (like mercury) at the highest point.

Steps for Taking Measurements:

1. Level Your Eyes: Ensure your eyes are at the same level as the meniscus. Looking from above or below causes distortion, leading to inaccurate readings.

2. Identify the Meniscus Type:

- Concave (Downward curve): For most liquids (water), read the volume at the bottom of the curve.
- Convex (Upward curve): For liquids like mercury, read the volume at the top of the curve.



3. Read the Graduation Lines: Determine the value of the intervals on the graduated cylinder (e.g., if there are 10 marks between 50 and 60, each line is 1 mL).

4. Estimate: Estimate the measurement to 1/5 or 1/10 of the smallest graduation mark for higher precision.

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Test of the Practical Work N°1: Safety Rules and Introduction to Chemistry Lab Work

Multiple Choice Questions

1. **What should you do if a chemical splashes into your eyes?**
   a) Rub your eyes vigorously
   b) Rinse your eyes with water for at least 15 minutes
   c) Close your eyes and wait for the pain to subside
   d) Apply a cold compress
2. **Which of the following is NOT appropriate lab attire?**
   a) Closed-toe shoes
   b) Long pants
   c) Loose, flowing clothing
   d) Safety goggles
3. **What is the first thing you should do if you spill a chemical on your skin?**
   a) Wipe it off with a cloth
   b) Rinse the affected area with water immediately
   c) Apply a bandage
   d) Ignore it if it doesn't hurt
4. **Where should you dispose of broken glassware?**
   a) Regular trash bin
   b) Recycling bin
   c) Designated glass disposal container
   d) Sink
5. **Which of the following statements about lab safety is TRUE?**
   a) Eating and drinking are allowed in the lab
   b) You should always work alone in the lab
   c) Always read labels and safety data sheets before using chemicals
   d) It's okay to leave a Bunsen burner unattended
6. **What is forbidden in the lab or experimental work area?**
   a) Talking
   b) Eating or drinking
   c) Wearing a lab coat
   d) Using a calculator
7. **How should chipped or cracked glassware be handled?**

a) Ignore and use it
b) Dispose of immediately
c) Repair with tape
d) Store for later use

8. What is the recommended technique for mixing solutions in glass containers?

a) Rapid stirring
b) Using metal stirrers
c) Gentle stirring with appropriate tools
d) Maximum speed

9. How should glassware be cleaned after use?

a) Rapidly with hot water
b) Using abrasive materials
c) Gentle washing with appropriate solvents
d) Ignoring cleaning

10. Graduated cylinders are mainly used for:

a) Heating liquids
b) Measuring approximate volumes
c) Storing samples
d) Mixing solids

11. To get an accurate reading from a graduated cylinder you must:

a) Tilt it slightly
b) Read meniscus at eye level
c) Read from the top edge
d) Add an extra drop

12. Volumetric pipettes are designed to measure:

a) Approximate volumes
b) A single fixed volume accurately
c) Multiple variable volumes
d) Gas volumes

13. Which instrument requires reading the meniscus at eye level for accuracy?

a) Burette
b) Beaker
c) Petri dish
d) Funnel

14. To get an accurate reading from a graduated cylinder you must:

e) Tilt it slightly

f) Read meniscus at eye level

g) Read from the top edge

h) Add an extra drop

15. What is the purpose of using a fume hood when working with volatile substances?

a) Aesthetic purposes

b) Increase efficiency

c) Enhance safety by venting fumes

d) Save time

True/False Questions

1. **You should always wear safety goggles when handling chemicals.**

   ☐ True

   ☐ False

2. **It's safe to taste chemicals in the lab to identify them.**

   ☐ True

   ☐ False

3. **You should know the location of safety equipment such as fire extinguishers, eyewash stations, and safety showers.**

   ☐ True

   ☐ False

4. **Long hair should be tied back when working in the lab.**

   ☐ True

   ☐ False

5. **It's acceptable to use your phone in the lab as long as you're careful.**

   ☐ True

   ☐ False

Practical Work N°2: Preparation of Aqueous Solutions from Solid and Liquid Matter

1. Introduction

Solutions play a crucial role in the field of chemistry. A solution is defined as a homogeneous mixture formed by dissolving one or more solutes in a solvent. The chemical present in the greatest quantity within the mixture is termed the solvent, and the other components are called solutes. Solutions with known concentrations can be referred to as standard (stock) solutions.

Laboratory experiments and various types of research often necessitate the preparation of chemical solutions as a part of their procedure. Many experiments involving chemicals require their application in solution form. This entails mixing two or more substances in specified quantities, which may involve weighing a precise amount of a dry material or measuring a precise amount of a liquid.

In this laboratory session, you will practice preparing solutions of different concentrations. The amount of solute that is dissolved in a given quantity of solvent is expressed as the concentration of the solution. A dilute solution is characterized by containing a small amount of solute in a given amount of solution. Chemists often use various units to describe the concentration of solutions such as molarity, molality, normality, and percentage.

During this laboratory session, you will experiment 2 ways of a solution preparation:

A - Preparing a solution by weighing a chemical substance (solid or liquid).

B - Preparing a solution by diluting a stock solution of known concentration.

2. Outcomes

- **Learn** the proper techniques for preparing a solution by dissolving solutes in solvents, ensuring complete dissolution and accurate concentrations.
- **Understand** the process of diluting solutions with a known concentration, including the calculations required to obtain the desired concentrations from stock solutions
- **Comprehend** different units of concentration, such as molarity, molality, and percentage...etc.
- **Recognize** and utilize the equipment and laboratory tool for preparing aqueous solutions.

3. Reminders:

a) Definition of a Solution: In chemistry, a solution is a homogeneous mixture of two or more constituents. The major constituent is called **solvent**. The minority compound(s) are called **solute(s)**. Solutions can be categorized as liquid, gaseous, or solid.

-When the solvent is water, the solution is called an "aqueous solution".

- When the solvent can no longer hold any more solute, the solution is said to be "saturated".

-A solution can be prepared by:

- Dissolving a solid.
- Diluting a more concentrated liquid.

b) Dissolution of a Solid: A solid substance dissolves in a liquid upon contact. It disappears into the liquid to form a homogeneous solution. Dissolution is a physical process, without any chemical reaction. However, it may be accompanied by a change in temperature.

c) Dilution: it's a process that involves obtaining a solution with a concentration lower than that of the starting one. Diluting a solution means decreasing its concentration by adding distilled water. During dilution, the amount of solute material is retained.

The starting solution is called the '**mother solution'** and the diluted solution is called the '**daughter solution'**.

d) Dilution Factor: The dilution is characterized by its dilution rate, also called dilution factor. We call the coefficient dilution factor $\boldsymbol{k = C_i/C_f = V_f/V_i}$

4. Different Expressions of Concentration

We call the concentration of the solution the amount of solute contained in the unit of volume or mass of the solution. If a compound X is present in a solution, we can define the composition of the solution at X using:

a) The Molar Concentration (Molarity [mol.L $^{-1}$]): It is the number of moles of solute X dissolved in one liter of the solution. The molar concentration (**M**) is expressed in mol.L^{-1}.

$$\mathbf{M} = \frac{\text{Moles of solute}}{\text{Liters of solution}}$$

Example: Find the molarity of a solution with 5.85 g of NaCl in 500 mL of total solution.

- Calculate moles of solute (NaCl): Moles of NaCl=5.85 g/58.5 g/mol =0.101 mol
- Convert volume to litters: 500 g/1000=0.5 L
- Calculate molarity: M = Molarity (M) = 0.101 mol/0.5 L = 0.2002 M

b) The Mass Concentration (Mass Title [g.L^{-1}]): It is given by moles of solute X dissolved per kg of the solvent. Mass is expressed in kg or g and volume often expressed in L and sometimes in m^3.

$$\text{m} = \frac{\text{Moles of solute}}{\text{kilograms of solution}}$$

Example: Find the molality of a solution containing 10 g of NaCl dissolved in 500 g of H_2O?

- Calculate moles of NaCl (solute): Moles of NaCl=10 g/58.5 g/mol =0.171 mol
- Convert water (solvent) to kg: 500 g/1000=0.5 kg
- Calculate molality: m = Molality (m) = 0.171 mol/0.5 kg = 0.342 mol/Kg

c) The Normal Concentration (Normality N [eq.L $^{-1}$]): It is the number of gram equivalent of solute contained in one liter of solution (V).

$$\text{N} = \frac{\text{Equivalents of solute}}{\text{Liters of solution}}$$

Relation between Normality and Molarity : $N = Z \times M$

Where **Z** is a constant with a number of states

For acid: Z is the number of H^+ protons that the acid can lose (Example: for HCI and H_2SO_4, Z = 1 and Z = 2, respectively).

For base : Z is the number of hydroxides OH that the base can lose (Example: for NaOH and $BaOH_2$, Z=1 and Z=2, respectively).

For oxidation and reduction : Z is the number of transferred electrons (Example: for the reaction $MnO_4^- + 5e^- + 8OH^- \rightarrow Mn^{2+} + 4H_2O$, Z = 5).

For salts : Z is the number of metal atoms in its valence (Example: $Al_2(SO_4)_3$ $2Al^{3+} + 3SO_4^{2-}$, $Z = 2 \times (+3) = 6$).

Example: Find the normality of a solution containing 4.9g of Sulfuric Acid (H_2SO_4) in 500mL of solution.

- Find equivalent weight of H_2SO_4 (M = 98g/mol, Z = 2): Equivalent Weight = 98/2 = 49g/eq
- Calculate equivalents: Equivalents = 4.9g /49 g/eq = 0.1eq
- Convert volume to Liters: 500mL = 0.5L
- Calculate Normality: N = 0.1eq/0.5L = 0.2N

d) Mole Fraction (X): is a way of expressing the concentration of a component in a mixture, defined as the ratio of the number of moles of a specific component to the total number of moles of all components in the mixture. It is a dimensionless quantity, meaning it has no units.

$$X = \frac{n_i}{n_{total}}$$

Example: calculate the mole fraction of a substance A and B in a mixture containing 2 moles of component A and 3 moles of component B

- The mole fraction of the component A would be: $X_A = 2/ 2+3 = 0,4$
- The mole fraction of the component B: $X_B = 3/2+3 = 0,6$

e) Mass Percentage (w/w): When the concentration is expressed as the percent of one component in the solution by mass it is called mass percentage (w/w).

Suppose we have a solution containing component A as the solute and B as the solvent, then its mass percentage is expressed as:

$$Mass\ \%\ of\ A\ = \frac{Mass\ of\ component\ A\ in\ the\ solution}{Total\ mass\ of\ the\ solution\ \times\ 100}$$

For example, if a solution of NaOH in water is said to be 50 % by mass that means a 1Kg solution will contain 500 g NaOH.

f) Volume Percentage (V/V): Sometimes we express the concentration as a percent of one component in the solution by volume, it is then called as volume percentage and is given as:

$$\text{Volume } \%\, of\ A\ = \frac{\text{Volume } of\ component\ A\ in\ the\ solution}{Total \text{ volume } of\ the\ solution\ \times\ 100}$$

For example, if a solution of HCl in water is said to be 10 % by volume that means a 100 ml solution will contain 10 ml HCl.

5. Experimental Procedure

A. Preparing a Solution by Weighing a Chemical Substance

A solution with known concentration can be prepared from solids using two similar methods. In the first method, the solid solute is weighed out on weighing paper or in a small container and then transferred directly to a volumetric flask. A funnel might be helpful when transferring the solid into the narrow neck of the volumetric flask. A small amount of solvent is then added to the flask and the contents are swirled gently until the substance is completely dissolved. More solvent is then added until the meniscus of the liquid reaches the calibration mark on the neck of the volumetric flask (a process called "diluting to volume"). The flask is then capped and inverted several times until the contents are mixed and completely dissolved.

In the second method, the solid is initially weighed in a small beaker. A small amount of solvent is added to the beaker and the solution is stirred until the solid is dissolved. The solution is then transferred to the volumetric flask. Again, a funnel may need to be inserted into the narrow neck of the flask. Before adding more solvent to the flask, the beaker, stirring rod, and funnel must be rinsed carefully and the washings added to the flask making sure all remaining traces of the solution have been transferred. Finally, the solvent is added to the flask until the liquid level reaches the calibration mark.

You will prepare a solution using the second method described. To prepare a solution with a desired concentration you will weigh out an appropriate mass of the reagent, dissolve it in a portion of solvent, and adjust to the desired volume. The mass of the solute is calculated using known concentration and volume of the solution to be prepared:

$$\boldsymbol{n_{solute}\ =\ C_{solution}\ \times\ V_{solution}}$$

$$\boldsymbol{m_{solute}\ =\ n_{solute}\ \times\ M_{solute}}$$

B. Preparing a Solution by diluting a Stock Solution of Known Concentration

Solutions are often prepared by diluting a more concentrated stock solution. A known volume of the stock solution is transferred to a new container and brought to a new volume. Since the total amount (number of moles) of solute is the same before and after dilution, we know that

$$C_i \times V_i = C_f \times V_f$$

Where C_i is the stock solution's concentration, V_i is the volume of stock solution being diluted, C_f is the dilute solution's concentration, and V_f is the volume of the dilute new solution.

6. Manipulation

Laboratory Tools: Graduated pipette, pipette bulb, beakers, funnel, graduated cylinder, watch glass, spatula, volumetric flasks of 100 ml, wash bottle, analytical balance.

Laboratory Reagents: NaOH (powder), distilled water, HCl.

6.1. Preparing Aqueous Solution by Weighting a Chemical Substance



(a) An amount of the solute is weighed out on an analytical balance

(b) A portion of the solvent is added to the volumetric flask.

(c) The mixture is swirled until all of the solute is dissolved.

(d) Additional solvent is added up to the mark on the volumetric flask.

Task: Prepare 100 mL of sodium hydroxide NaOH solution at concentration C_1 = 0.5 mol/L. Calculate the mass needed to prepare this solution.

Procedure: Take a watch glass and put it into the balance. Press the zero/tare (0/T) button. When a balance is tarred with an object on the balance pan, the weight of the object will be automatically subtracted from every reading until the balance is re-tarred or zeroed. Using spatula, take a small quantity of sodium hydroxide and put it into the watch glass carefully.

During weighing, avoid spilling chemicals on the working table, particularly on the balance. Any remaining of chemicals on the balance will lead to the error during measurements. Repeat adding the substance until you reach the needed mass.

Dissolve the solid compound NaOH in a beaker of 50 mL of distilled water (DW) using stirring rode. With the help of a funnel, pour the solution into a 100 mL volumetric flask. Rinse the funnel with a small amount of DW, and add the washings to the volumetric flask making sure all remaining traces of the solution have been transferred. Complete the volumetric flask with DW at one finger from the calibration mark (meniscus). Cap the flask and shake the solution. Bring the level of solution to the meniscus by adding the last drops of DW using dropper. Shake it again and check the meniscus. Clean the beaker, stirring rode and the funnel. Label the flask with compound name (NaOH) and concentration (0.5M).

6.2. Preparing Aqueous Solutions by Dilution of a Stock Solution more Concentrated Mother Solution.



(a) A volume (Vi) containing the desired moles of solute is measured from a stock solution of known concentration.

(b) The measured volume of stock solution is transferred to a second volumetric flask.

(c) The measured volume in the second flask is then diluted with solvent up to the volumetric mark.

Task: Prepare 100 mL of Hydrochloric acid (HCl) solution of 0,4M. Calculate the volume of the HCl you would mix with DW.

Procedure: Fill 2/3 of 100 mL volumetric flask with distilled water. Using volumetric pipette transfer needed volume of HCl into a the volumetric flask. Cap the flask and invert it several times to mix solution. Next, add water until the liquid is just below the etched line on the neck of the flask. Complete carefully with DW until the calibration mark and label it with the compound name (HCl) and concentration (0.05M).

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Test of the Practical Work N°2: Preparation of Aqueous Solutions from Solid and Liquid Matter

1. What are the different methods of preparing solutions?

2. Units commonly used for amount concentration?

3. Explain the meanings of the following pictograms?






4. Name the following glassware:






5. What lab equipment is used to make solutions and perform dissolution and dilution of NaOH and HCl solutions, respectively?

6. Determine the concentration and the molar mass of NaOH and HCl we worked with for each experiment?

7. Calculate the mass of NaOH weighed for the preparation of 100 ml of C = 0.5 mol/L?

8. Determine the mass concentration of the prepared NaOH solution?

9. Determine the concentrations of the stock solution of HCl used?

10. Calculate the volume of HCl to be taken from the stock solution required to prepare the 100 ml of 0.4 M HCl?

11. Determine the dilution factor for the prepared HCl solution?

12. Explain why it's necessary to add the acid to the water during the procedure instead of adding water to the acid?

Practical Work N°3: Acid-Base Titrations

1. Introduction

An acid base titration is a method of quantitative analysis used in chemistry for determining the concentration of acid or base by neutralizing it using a solution of a known concentration. The significance of acid base titration extends beyond the laboratory as it play a crucial role in various scientific fields, including pharmaceuticals, environmental monitoring, and quality control in industries. This method's accuracy and simplicity makes it an important tool in quantitative analysis, greatly contributing to the overall understanding of solution chemistry.

2. Outcomes:

- Apply volumetric analysis to determine the concentration of solutions and Understand the principle of neutralization and indicator color selection in the acid base titration.
- Develop skills in identifying the endpoint (equivalent point) between acid and base and computing sample pH at important stages of a titration
- Enhance laboratory skills in accurate measurement and the use of titration equipment.

3. Reminders:

3.1. Key Acid & Base Definitions

Lewis definition: Acids are electron-pair acceptors, and bases are electron-pair donors.

Arrhenius definition: Acids produce hydronium ions (H_3O^+) ions in water, while bases produce hydroxides **(**OH^-) ions in water.

Brønsted-Lowry definition: Acids are proton donors, and bases are proton acceptors.

3.2. Strong Acids & Bases

1. **Definition of Strong Acids:** Strong acids are substances that fully ionize/dissociate in aqueous solution, producing a high concentration of protons (H^+).
   Examples: HCl (Hydrochloric), H_2SO_4 (Sulfuric), HNO_3 (Nitric),
2. **Definition of Strong Bases:** Strong babses are substances that fully ionize/dissociate in aqueous solution, producing a high concentration of hydroxides (OH^-).
   Examples: NaOH (Sodium Hydroxide), KOH (Potassium Hydroxide).

3.3. Weak Acids & Bases

3. **Definition:** Partially ionize/dissociate in aqueous solution, establishing equilibrium between ions and molecules.
   Examples: CH_3COOH (Acetic acid), HF (Hydrofluoric acid), NH_3 (Ammonia base).

3.4. Buffer Solutions: a buffer solution is an aqueous system that resists changes in pH upon the addition of small amounts of acid or base. It typically composed of a weak acid and its conjugate base or a weak base and its conjugate acid.

4. Acid and Base Titration

An acid-base titration is a quantitative analysis technique used to determine the unknown concentration of an acid or base (**analyte** or **titrand**) by neutralizing it with a solution of base or acid (**titrant**) having known concentration. The neutralization process is based on stoichiometric reaction between H^+ ions from acids and OH^- ions from bases to form water:

$$\mathbf{H^+ (acid) + OH^- (base) <====> 2H_2O}$$

At the equivalence point, the number of moles of acid equals to the number moles of base, meaning neutralization is complete, which allows the unknown concentration to be deduced.

$$\mathbf{C_{Acid} \times V_{Acid} = C_{Base} \times V_{Base}}$$

The equivalence point of acid-base titration can be indicated by either identifying the steepest point on the titration curve measured by a pH meter, or a color change of a pH indicator.

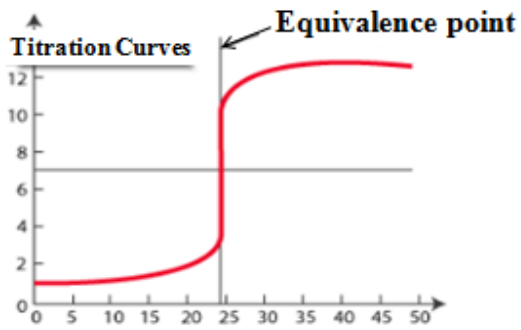
5. Types of Acid-Base Titration

The types and examples of strong/weak acids and bases are tabulated below.

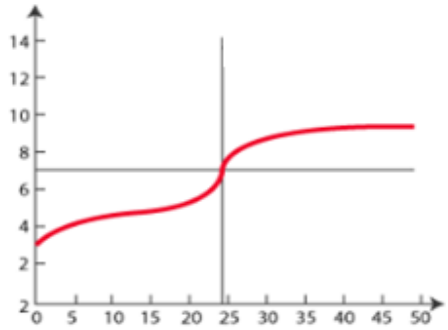
Types	**Examples**
Strong acid-strong base	Hydrochloric acid and sodium hydroxide
Weak acid-strong base	Ethanoic acid and sodium hydroxide
Strong acid-weak base	Hydrochloric acid and ammonia
Weak acid-weak base	Ethanoic and ammonia

6. Titration Curve & Equivalence Point

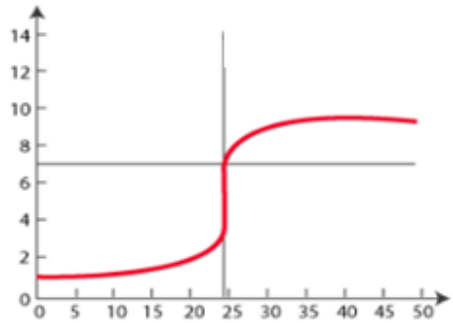
In acid base titration, the equivalence point is the point at which exactly the same number of moles of hydroxide ions (OH^-) has been added as there are moles of hydrogen ions (H^+). In an acid base titration, if the base is added from the burette and the acid has been accurately measured into a flask. The shape of each titration curve is typical for the type of acid-base titration. The pH does not change in a regular manner as the acid is added.



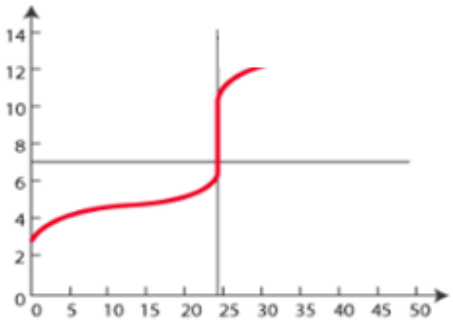
Strong acid and strong base



Strong acid and weak base



Weak acid and weak base



Weak acid and strong base

7. Acid-base Indicators

Acid-base indicators, also known as pH indicators, are chemical substances that demonstrate acid-base characteristics. Their acidic form "**HIn**" and basic form "**In**" display distinct colors. The color change of these indicators based on the pH level of the environment in which they are present and occurs due to the changes in the molecular structure of the indicator as it either gains or losses protons (H^+ ions). The transition range over which the pH indicators change color, allows us to detect the equivalence point. Each indicator possesses a specific pH transition range, making it suitable for different pH measurements.

$$HIn\ (Acid\ Conjugate) + H_2O \leftrightarrow H_3O^+ + In^-(base)$$

8. Choosing a Colored Indicator:

Acid-base titrations can be grouped according to the nature of the species present, thus predicting the pH transition range.

- Reaction between a **strong acid** and a **strong base**: the pH transition range can be predicted. In this case, the equivalence point will be at **pH 7**.
- Between a **strong acid** and a **weak base**: the equivalence point will be at **pH < 7**.
- Between a **weak acid** and a **strong base**: the equivalence point will be at **pH > 7**.
- Between a **weak acid** and a **weak base**: for this last case, we cannot conclude a priori; a calculation using the **pKa** values of the acid-base pairs involved is essential. Therefore, depending on the pH, the indicator should be chosen; its color change range must correspond to the equivalence point during the titration.

Example: Methyl orange is red in its acidic form and yellow in its basic form. When both forms coexist, we are in the color change range, and the color is orange. This indicator is suitable if the expected pH at the equivalence point is acidic (between **3.2** and **4.5**).

A list of colored indicators used in chemistry is provided:

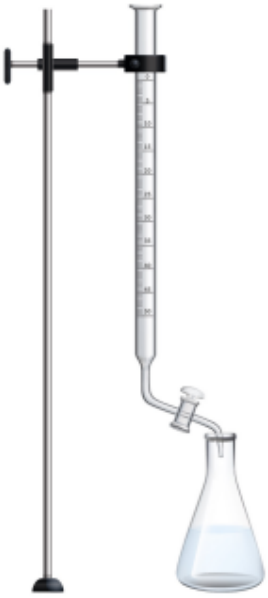
Indicator	Color change interval (pH)	Acid	Base
Thymol Blue	**1.2 - 2.8**	Red	Yellow
Methyl Orange	**3.1 - 4.4**	orange	Yellow
Methyl Red	**4.4 - 6.2**	Red	Yellow
Chlorophenol Red	**5.4 - 6.8**	Yellow	Red
Bromothymol Blue	**6.2 -7.6**	Yellow	Blue
Phenol Red	**6.4 - 8.0**	Yellow	Red
Thymol Blue	**8.0 - 9.6**	Yellow	Blue
Phenolphthalein	**8.0 - 10.0**	colorless	Magenta
Alizarin Yellow	**10.0 -12.0**	Yellow	green

9. Experimental Procedure

Laboratory Tools: Erlenmeyer flask, graduated cylinder, ring clamp, ring stand, pipettes, beakers, wash bottle, funnel, pipette bulb, pH-meter, magnetic stirrer, magnetic stir bar. **Laboratory Reagents:** Standard solution of NaOH, distilled water, HCl (unknown concentration), Phenolphthalein indicator.

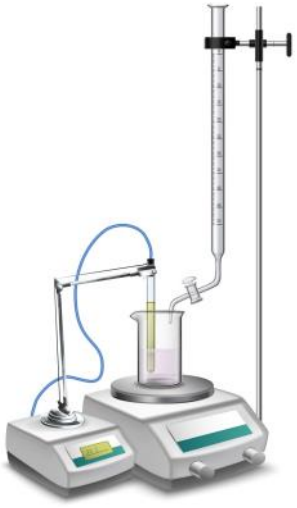
10. Manipulation

a. Colorimetric Titration:



1. Check that the burette tap is properly closed.
2. Rinse the burette with distilled water and then with NaOH solution (0,1M) and adjust the liquid level to the zero level of the burette, ensuring no air bubbles are present.
3. Pour 10mL of HCl solution into a clean Erlenmeyer flask using a graduated cylinder.
4. Add 2-3 drops of phenolphthalein indicator to the Erlenmeyer flask and insert the magnetic stir bar.
5. Place the Erlenmeyer flask on a white sheet to enhance the visibility of color changes (on the magnetic stirrer).
6. Open the burette tap slowly to allow the NaOH solution to flow into the HCl solution and carry out the dosage drop by drop until the color change.
7. Note the Volume Vb of the NaOH base poured and calculate Ca of HCl acid
8. Repeat the operation two to three times.

b. pH-metric Titration:



1. Adjust the liquid level to the zero level of the burette by adding the NaOH solution.
2. Pour 10mL of HCl solution into a clean beaker using a clean graduated cylinder and insert the pH probe into the beaker.
3. By using a magnetic stirrer, place a magnetic stir bar in the beaker and start stirring gently.
4. Do the titration ml per ml, note the pH every 1ml between 1 and 8 ml, then every 0,5 ml between 8 and 10 ml, and finally every 1ml between 10 ml and 15 ml.

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Test of the Practical Work N°3: Acid-Base Titrations

1. What is the purpose of acid base titration?

2. List the differences between strong and weak acids and give 2 examples of each.

3. How do the buffer solutions resist the change in pH?

4. How is an indicator selected for detecting the pH change in acid-base titrations?

5. What methods are used to carry out this practical work?

6. How can you identify the equivalent of acid-base titrations by using a pH-meter?

7. Give the names of the glassware below:






8. Give the names of these Pictograms:





9. Write the chemical equation for the neutralization reaction of hydrochloric acid and sodium hydroxide?

10. Calculate the number moles of hydrochloric acid used in the reaction for experiment 1?

11. Plot the pH curve as a function of base volume for experiment 2. What trend of pH is observed?

12. Provide the coordinates of the equivalence point and calculate the molarity of the HCl solution?

Practical Work N°4: Titration of Milk

1. Introduction

Acid-base reactions involve the exchange of protons (H^+) between acids and bases. In this mechanism, acids donate protons, while bases accept them, establishing equilibrium. This fundamental principle drives titrations, where a strong base neutralizes acidic solutions. Lactic acid is produced during milk spoilage by bacteria like Lactobacillus, indicating **freshness** loss. In titration, **lactic acid** reacts with a strong base such as **NaOH**, allowing measurement of acidity levels. The increase in lactic acid means increased milk spoilage, measurable through titration outcomes. In acid-base titration, a known volume of milk is mixed with a few drops of an indicator like **phenolphthalein**, which changes color upon neutralization. Titrating with NaOH until a permanent color change indicates that the amount of lactic acid can then be quantified, revealing the milk's freshness. Fresh milk not only offers better taste but also contains vital nutrients. Spoiled milk can contain harmful bacteria, posing health risks to consumers. The presence of lactic acid is a key indicator of spoilage in milk. When milk spoils, bacteria like Lactobacillus convert lactose into lactic acid, affecting taste and safety.

2. Outcomes:

- **Understand** how to manage product quality and assess the acidity level of milk
- **Gain** knowledge on how to identify the equivalence point in a colorimetric acid-base titration with the help of a color indicator.
- **Apply** acid base titration analysis and stoichiometric calculations, linking theoretical concepts to hands-on experimentation with lactic acid quantification in milk.

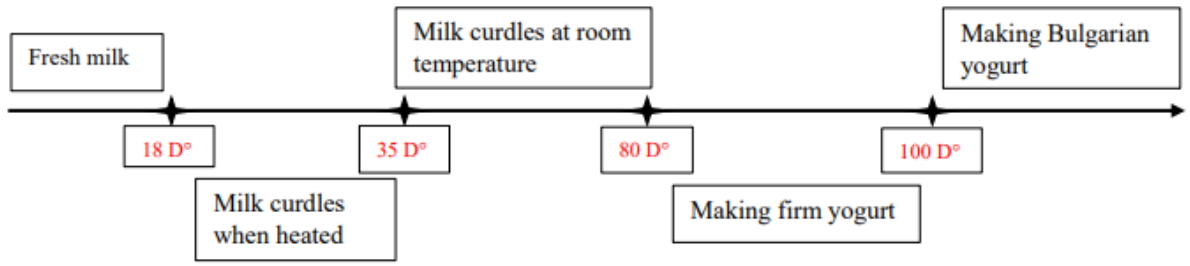
3. Definitions

3.1. Lactic acid: Lactic acid (**$C_3H_6O_3$**) or mono-hydroxyl acid (**2-hydroxypropanoic acid**) is the main constituent of milk. The bacteria in milk, when subjected to heat, convert lactose to glucose and galactose, which are then further converted to lactic acid. Lactic acid is widely used in the food and pharmaceutical industry for its acidifying and bacteriostatic properties.

3.2. Dornic degree: The acidity measure (expressed in Dornic degree) makes it possible to control the quality of raw milk: 1 Dornic degree (1°**D**) is equal to **0.1g** of lactic acid per liter.

$$D = \frac{C_m}{0.1}$$

Where **D** is the dornic degree and **Cm** is mass concentration in g/L



3.3. Lactic acid Titration: the amount of **lactic acid** in milk will be measured using colorimetric titration. The measurement principle is unique, and is based on adding, to a given volume of milk, the necessary volume of alkaline solution (**sodium hydroxide**) of an exact concentration until the neutralization point is reached, which is determined by the presence of an indicator, generally **phenolphthalein**, which turns from colorless to pink at pH 8.4.

4. Acid-base Titration

4.1. Titration Reaction

$$CH_3CH(OH)COOH\ (aq) + NaOH\ (aq) \rightarrow CH_3CH(OH)COONa\ (aq) + H_2O\ (l)$$

$$AH(aq)\ (\textbf{acid}) + OH^-\ (\textbf{base}) \rightarrow A^-\ (aq)\ (\textbf{salt}) + H_2O(l)\ (\textbf{water})$$

Lactic acid (**$C_3H_6O_3$**) classified as a weak acid, only partially dissociates in water. Therefore, the titration process allows us to determine the amount of sodium hydroxide (NaOH) needed to neutralize the lactic acid present in the sample. The volume of NaOH required to reach the neutralization point is used to calculate the concentration of lactic acid in the milk sample.

To calculate the concentration of milk we use the formula:

$$\mathbf{C_{Acid} \times V_{Acid} = C_{Base} \times V_{Base}}$$

Where $\mathbf{C_{Acid}}$ is the unknown concentration of acid $CH_3CH(OH)COOH$ solution in the Erlenmeyer flask and $\mathbf{C_{Base}}$ is the known concentration of NaOH in the burette.

To calculate the mass concentration, we use the formula:

$$\mathbf{C_m = M_{CH_3CH(OH)COOH} \times C_{Acid}}$$

Where: $\mathbf{M_{CH3CH(OH)COOH}} = 3M_C + 3M_O + 6M_H = (3\times12) + (3\times16) + (6\times1) = 90$ g/mol.

5. Experimental Procedure

Laboratory Tools: 25 mL graduated burette and its stand ; 100 mL graduated cylinder ; Funnel and wash bottle ; 20 mL or 10 mL volumetric pipette and a pipette bulb ; 250 mL Erlenmeyer flask or beaker ; Magnetic Stirrer and magnetic bar.

Laboratory Reagents: Sodium hydroxide solution (NaOH) with a concentration of 0.1 mol/L ; Milk ; Distilled water ; Phenolphthalein solution (0.2% in ethanol).

6. Manipulation

- Fill the burette with sodium hydroxide solution of molar concentration C_B = 0,1mol/L.
- Adjust the level of the solution to the zero graduation of the burette.
- Using a graduated cylinder, take V_A = 20 mL of milk and pour it into a 250 mL Erlenmeyer flask.
- Add 100 mL of distilled water and a few drops of phenolphthalein.
- Introduce the magnetic stir bar.
- Place the Erlenmeyer flask on a white sheet under the burette to enhance the visibility of color changes (on the magnetic stirrer).
- Adjust the stirrer to homogenize the mixture in the Erlenmeyer flask.
- Open the burette tap slowly to allow the NaOH solution to flow into the milk solution and carry out the dosage drop by drop until the solution color becomes persistent pink.
- Note the value of the volume of the poured NaOH solution (V_B = …. mL).

Bibliography

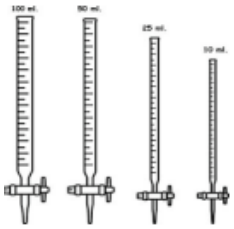
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Test of the Practical Work N°4: Titration of Milk

1. What's the objective of the practical Work?

2. Give the names of the following glassware:




3. Give the names of these Pictograms:





4. How to check acidity of milk?

5. Describe the procedure used for the titration of milk?

6. Write the balanced equation for the neutralization reaction between aqueous sodium hydroxide and lactic acid?

7. An indicator solution is used to indicate when all the lactic acid has been consumed and that the reaction in complete. What is the name of the indicator solution? How exactly does the indicator let you know when the reaction is complete?

8. Determine the molar mass of lactic acid ($C_3H_6O_3$)?

9. Using the relation $C_{acid}V_{acid}=C_{base}V_{base}$, calculate the molar concentration of lactic acid in the milk?

10. Based on the result obtained above, determine the mass concentration of lactic acid in the milk?

11. Determine the Dornic degree?

12. Is the milk studied here fresh? Why?

Practical Work N°5: Titration of Vinegar

1. Introduction

Vinegar is a solution made from the fermentation of ethanol (CH_3CH_2OH), which in turn was previously fermented from sugar. The fermentation of ethanol results in the production of acetic acid or ethanoic acid (CH_3COOH). There are many different types of vinegar, each starting from a different original sugar source (e.g., rice, wine, malt, etc.). Acetic acid, the main component of vinegar, is widely used in food preservation, flavoring, and other industrial applications. The amount of acetic acid in vinegar can vary, typically between 4 to 6% for table vinegar, and up to three times higher (18%) for pickling vinegar. Therefore, it is essential to determine the concentration of acetic acid in vinegar accurately.

2. Outcomes

- **Describe** the chemistry of acid-base neutralization reactions and solve stoichiometry problems in the solution phase.
- **Prepare** standard acid solutions and use this to standardize a basic solution.
- **Determine** the concentration of a standard solution of vinegar and compare this with the stated concentration.
- **Use** the procedure to collect accurate and valid data and evaluate the outcome of the investigation in relation to the vinegar.

3. Backgrounds

Titration is a way to measure the unknown amount of a chemical in a solution; the titrant, by adding a measured amount of a chemical with a known concentration; the titrating solution. The titrating solution reacts with the titrant, and the endpoint of the reaction is monitored in some way. The titrant concentration can be calculated from the amount of titrating solution added, and the ratio of the two chemicals in the chemical equation for the reaction. In this practical work, we will determine the amount of acid using **acid-base neutralization** titration by adding enough hydroxyl ions from base to balance out the added hydrogen ions from acid. The OH^- ions will react with the H^+ ions to produce water and the identity of the salt can be determined by taking the cation from the base and taking the anion from the acid.

$$\boldsymbol{Acid + Base \rightarrow Water + Salt}$$

4. Titration of Vinegar

Vinegar is a dilute solution of acetic acid (CH_3COOH) in water, and it is often used in titrations to analyze its acid content. The acid-base reaction between acetic acid and sodium hydroxide can be written as:

$$\mathbf{CH_3COOH}\ (aq) + \mathbf{OH^-}\ (aq) \rightarrow \mathbf{CH_3COO^-}\ (aq) + \mathbf{H_2O}\ (l)$$

Acetic Acid + Sodium Hydroxide → Sodium Acetate + Water

Acetic acid is a weak monoprotic acid (contains one acidic hydrogen atom), meaning it does not fully dissociate in water. In order to obtain the concentration of acetic acid (CH_3COOH) present in the vinegar, sodium hydroxide (NaOH), a strong base, whose concentration is known is added until the reaction is complete. This means, exactly one mole of NaOH must be added to one mole of acetic acid. This point in the titration is called the Equivalence Point. The equivalence point is defined as that point in the titration when stoichiometrically equal amounts of acid and base are present. In the vinegar neutralization titration, that would be when one mole of NaOH has been added to one mole of CH_3COOH. Therefore the equivalence point depends on the reaction stoichiometry. At the beginning of the titration, the solution in the Erlenmeyer flask is acidic. As the base is added, it completely reacts with the acid and the solution in the Erlenmeyer flask continues to be acidic. But, at the equivalence point, the acid has completely reacted with the base. If even one tiny drop of base is added beyond that needed to arrive at the equivalence point, the solution in the Erlenmeyer flask is basic. By knowing the volume of the sodium hydroxide required to reach the equivalence and its concentration, the amount of acetic acid in the vinegar can be determined.

$$\mathbf{C_{acetic\ acid} . V_{acetic\ acid} = C_{sodium\ hydroxide} . V_{sodium\ hydroxide}}$$

An indicator is a chemical substance whose color depends on the acid/base property of the medium it is present in. Phenolphthalein is an indicator, which is colorless in an acidic medium and has a pink color in a basic medium. In this titration, a few drops of phenolphthalein should be added to the acid in the Erlenmeyer flask. The solution will remain colorless until the equivalence point. When the equivalence point has been crossed and the solution becomes basic, the phenolphthalein will take on a pink color. This is the reason to add the base drop by drop, so that even though the equivalence point will be crossed, the titration can be stopped at the appearance of the first permanent pale pink color.

5. Acidity of Vinegar

5.1. Definition: the degree of acidity (°) of vinegar measures its strength, defined as the mass in grams of pure acetic acid (**CH_3COOH**) contained in **100 g** of vinegar solution.

5.2. Examples:

- Distilled White Vinegar (5%): Often used for cleaning and cooking.

- Apple Cider Vinegar (4-5%): Used in salad dressings and for health purposes.

- Wine/Balsamic Vinegar (6-7%): Commonly used for gourmet culinary applications.

5.3. Acidity vs. pH: while the degree of acidity of vinegar measures the percentage of acetic acid, the pH (typically 2.4-3.4) indicates the concentration of the hydrogen ions (H^+). Higher degree of acidity (percentages) corresponds to lower, more acidic pH values.

In this PW, we will dose the acetic acid contained in a known volume of vinegar ($\mathbf{V_{acid}}$) using a base solution of known concentration ($\mathbf{C_{base}}$); sodium hydroxide solution (NaOH). At the equivalence, the number of moles of acetic acid initially present in the vinegar equals the number of moles of NaOH poured into the solution to be titrated, and the volume of NaOH solution required to reach the equivalence ($\mathbf{V_{base}}$) is determined. $\mathbf{V_{base}}$, $\mathbf{C_{base}}$, and $\mathbf{V_{acid}}$ are all known, so the concentration of the acid ($\mathbf{C_{acid}}$) can be determined as described above.

1. The mass of acetic acid in the sample can be determined from the number of moles present and the molar mass of acetic acid: $\boldsymbol{m_a = n_a \times M_a}$.

2. Moles of acid can be calculated from concentration of acid multiplied by the volume of acid used: $\boldsymbol{n_a = C_a \times V_a}$.

3. The volume of the acetic acid in vinegar can be calculated from mass of acetic acid divided by the density of acid: $\boldsymbol{V = m/\rho}$.

4. Finally, the degree of acidity of vinegar solution that was titrated can determined as follows: $\boldsymbol{D° = \frac{100}{\rho} \times C_a \times M_a}$

Where, $\mathbf{M_a}$ is the acetic acid Molar weight (M= 60 g/mol), and $\boldsymbol{\rho}$ is the density of acetic acid ($\boldsymbol{\rho}$ = 1,01).

6. Experimental Procedure

Laboratory tools:

- 25 mL graduated burette ;
- 10 mL graduated cylinder, funnel ;
- Wash bottle ;
- 10 mL volumetric pipette and a pipette bulb ;
- Beaker ;
- Magnetic stirrer and magnetic bar.

Laboratory Reagents:

- Sodium hydroxide solution (NaOH) with a concentration of 0.1 mol/L ;
- distilled water ;
- phenolphthalein solution (0.2% in ethanol) ;
- Marketing vinegar with degree of acidity to be determined.

7. Manipulation

A. Preparation of a 1/10th dilution of the studied vinegar

1- Take 10 mL of marketing white vinegar using a pipette.
2- Put it the 10 ml vinegar solution in a 100 mL volumetric flask.
3- Fill with distilled water up to meniscus line (Jauge line) to dilute the vinegar so that the volume of the solution becomes 100 mL.
4- Cap the flask and shake to homogenize the solution referred to as S1.

B. Colorimetric titration of the diluted vinegar solution

1- Take V = 10mL of solution S1 using a pipette and put it into a beaker or Erlenmeyer flask.
2- Add a few drops (3 drops) of phenolphthalein.
3- Fill the burette with sodium hydroxide solution of molar concentration C_B = 0,1mol/L.
4- Adjust the level of the solution to the zero graduation of the burette.
5- Place the beaker or Erlenmeyer flask on a white sheet under the burette to enhance the visibility of color changes (on the magnetic stirrer).
6- Introduce the magnetic stir bar.

7- Adjust the stirrer to homogenize the vinegar solution in the beaker.

8- Slowly drip the solution of sodium hydroxide into the vinegar solution, while stirring, until the color changes to a persistent pink color.

9- Note the value of the volume of the poured NaOH solution (V_B = …. mL).

10- Repeat the titration at least three times to obtain an average volume of NaOH used.

B. pH titration of the diluted vinegar solution

1. Adjust the liquid level to the zero level of the burette by adding the NaOH solution.

2. Pour 10mL of solution S1 into a clean beaker using a clean graduated cylinder.

3. Insert the pH probe into the S1 solution in the beaker.

4. By using a magnetic stirrer, place a magnetic stir bar in the beaker and start stirring.

5. Do the titration ml per ml, note the pH every 1ml and plot the curve representing the pH as a function of the volume of titrant solution added, to determine the value of V_{eq}.

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Test of the Practical Work N°5: Titration of Vinegar

1. How can you calculate the amount of a chemical in a solution using titration?

2. What range of pH values do you expect different types of vinegar to have?

3. What is the formula for calculating the degree of acidity (°D) of vinegar?

4. Write the balanced equation for the neutralization reaction between aqueous sodium hydroxide and acetic acid?

5. What was the purpose of the phenolphthalein indicator in this experiment? Be specific.

6. Deduce the molarity and normality of acetic acid in solution S1?

7. Calculate the Cm (mass concentration) of acetic acid in this vinegar?

8. Determine the dilution factor for the prepared vinegar solution?

9. Calculate the degree of acidity of the vinegar?

10. Suppose you added 20 mL of water to your vinegar sample instead of 10 mL. Would the titration have required more, less or the same amount of NaOH (aq) for a complete reaction? Explain.

11. Give the names of the glassware below:

















12. Give the names of these Pictograms:







Practical Work N°6: Redox Titrations (Permanganatometric Titration)

1. Introduction

The oxidation and reduction reactions in aqueous solutions involve the transfer of electrons between different species. During the oxidation process, electrons are transferred away from the species, whereas in reduction, electrons are gained by the species. Oxidation and reduction reactions occur simultaneously. A reaction that involves both oxidation and reduction is called a redox reaction. Titrations that involve redox reaction are called redox titrations. In acid-base titrations, indicators that are sensitive to pH changes are utilized to determine the endpoint. Similarly, in redox titrations there is a change in oxidation potential of the system. The indicators used in redox reactions are sensitive to change in oxidation potential. The ideal oxidation-reduction indicators have an oxidation potential intermediate between the values for the solution being titrated and the titrant, exhibiting a sharp readily detectable color change.

2. Outcomes

- **Understand** the concept of the oxidation-reduction titration.
- **Carry out** a redox titration of hydrogen peroxide (H_2O_2) solution with potassium permanganate ($KMnO_4$) solution.
- **Identify** the Equivalence point.
- **Determine** the unknown concentration/molarity of H_2O_2 solution.

3. Backgrounds

3.1. Oxidation state (O.S): it corresponds to the number of electrons, that an atom loses, gains, or appears to use when joining with other atoms in compounds.

An atom of a free element has an oxidation number of 0.

- Example: O.S of each Cl atom in Cl_2 = 0.

A monatomic ion has an oxidation number equal to its charge.

- Example: O.S (Cu^{2+}) = +2, O.S (Br^-) = -1.

Hydrogen generally has an oxidation state of +1 in most of compounds.

- Example: O.S of Hydrogen in the molecule of HCl = +1.

Oxygen generally has an oxidation state of -2 in compounds.

- Example: O.S of Oxygen in the molecule of H_2O = -2.

Neutral compounds have a total oxidation state of zero (Σ O.S = 0), whereas polyatomic ions have a total oxidation state equal to their charge (Σ O.S = charge).

- Example: O.S of $KMnO_4$ = O.S (K) + O.S (Mn) + [4xO.S (O)] = 0, O.S of SO_4^{2-} = O.S (S) + [4 x O.S (O)] = -2.

Note: To determine the oxidation state of the Mn atom in the neutral compound $KMNO_4$. O.S (Mn) = O.S of $KMnO_4$ – O.S (K) - [4xO.S (O)] = 0 - (+1) -[4x(-2)] = +7.

3.2. Oxidation: it refers to the process in which a molecule, atom, or ion molecule loses one or more electrons during a reaction. The oxidation number (O.S) of an element increases when it undergoes oxidation.

3.3. Reduction: it refers to the process in which a molecule, atom, or ion molecule gains one or more electrons during a reaction. The oxidation number (O.S) of an element decreases when it undergoes reduction.

3.4. Oxidizing Agent: it is defined as a substance that accepts electrons in reduction reaction.

3.5. Reducing Agent: it is defined as a substance that loses electrons in oxidation reaction.

3.6. Redox Reaction: it is a type of titration that investigates the oxidation and reduction of certain chemical species.

- When there is an atom that donates electrons, there is always an atom that accepts electrons.
- The transfer of electrons occurs from one atom to another.
- Redox titration is based on redox reaction between the analyte and the titrant.

3.7. Redox Potential: it is an empirical measurement expressed in volts and symbolized as E. This measurement is used for redox couples to predict the reactivity of chemical species towards each other. The standard potential E° is conventionally measured relative to the water/hydrogen couple (H^+/H_2), which has a potential of zero.

- In general, the half-equation with the more positive redox potential will be the reduction reaction and the other will be the oxidation.

  Example: $F_2(g) + 2e^- \rightarrow 2F^-$ E° = +2.87 V

  $Cl_2(g) + 2e^- \rightarrow 2Cl^-$ E° = +1.36 V

  $F_2(g)$ will be reduced (Red reaction) and Cl oxidized (Ox reaction), as +2.87 > +1.36.

4. Titration Reaction

To determine the concentration of hydrogen peroxide (H_2O_2) solution, we used a colorimetric titration. The potassium permanganate ($KMnO_4$) solution serves as the analyte, and the H_2O_2 solution is used as the titrant.

4.1. The two half-reactions

- Oxidation reaction: (Reducing agent) $H_2O_2 \rightarrow O_2 + 2H^+ + 2e^-$

- Reduction reaction: (Oxidizing agent) $MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$

The reduction reaction was obtained after the following procedures:

1- Balancing oxygens: $Mn\mathbf{O_4^-} \rightarrow Mn^{2+} + \mathbf{4}H_2\mathbf{O}$

2- Balancing hydrogens: $MnO_4^- + \mathbf{8H^+} \rightarrow Mn^{2+} + \mathbf{4}H_2O$

3- Balancing electrons: $MnO_4^- + 8H^+ + \mathbf{5e^-} \rightarrow Mn^{2+} + 4H_2O$

O.S of $MnO4^-$ = -1= O.S (Mn) + (4 x O.S (O)) → O.S of Mn in MnO_4^- = +7

- Balancing of half-reactions: $[H_2O_2 \rightarrow O_2 + 2H^+_{(aq)} + 2e^-]$ x 5

$[MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O]$ x 2

4.2. Total reaction: $5H_2O_2 + 2MnO_4^- + 16\ H^+ \rightarrow 5O_2 + 10H^+ + 2Mn^{2+} + 8H_2O$

5. Equivalence Point: the oxidized and reduced forms of permanganate have different colors. A solution of $MnO4^-$ is intensely purple. However, in an acidic solution ($8H^+$), the reduced form of permanganate, Mn^{2+}, is almost colorless. When using $MnO4^-$ as a titrant, the solution of the analyte remains colorless until the equivalence point s reached. The first drop of excess $MnO4^-$ produces a persistent pale pink color (Self-indicator).

Note:

- The choice and amount of acid are important in this redox titration because, in the half-reaction of reduction 8 moles of acid are needed for each mole of manganate (MnO_4^-).
- Insufficient volume of acid will mean that the solution will not be acidic enough and a brown solid, MnO_2, will be produced instead of Mn^{2+} ions. The brown solid will mask the color change and lead to larger volumes of manganate ions used than required.

6. Experimental Procedure

Laboratory Tools: Erlenmeyer flask, burette, graduated cylinder, pipettes, volumetric flasks, beakers, wash bottle, funnel, pipette bulb, stand, magnetic stirrer, and magnetic bar.

Laboratory Reagents: Distilled water, sulfuric acid (H_2SO_4), potassium permanganate ($KMnO_4$), and hydrogen peroxide (H_2O_2).

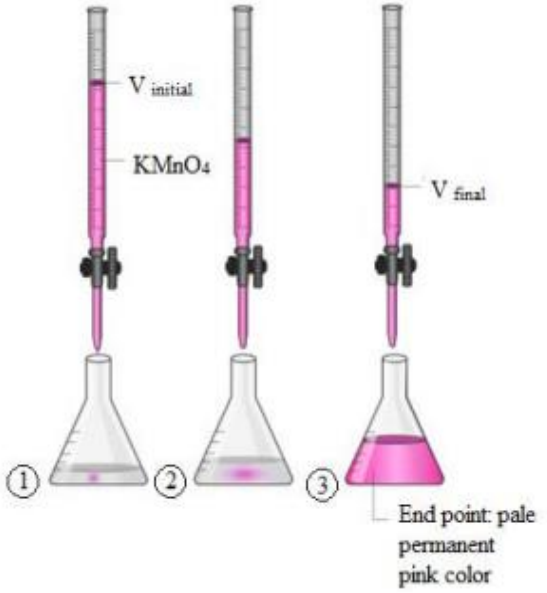
7. Manipulation

1- Rinse and fill a clean burette with potassium permanganate ($KMnO_4$) solution of 0,02M concentration. Remove the air bubble.

2- Take, using a pipette, V = 10 mL of H_2O_2 and pour it into an Erlenmeyer flask and add 3 drops of sulfuric acid (H_2SO_4).

3- Introduce the bar magnet, and place the Erlenmeyer flask on the magnetic stirrer. If there is no stirrer, use a beaker.

4- Titrate the H_2O_2 solution by adding the $KMnO_4$ solution drop by drop into the Erlenmeyer flask until the color change (the persistent violet color appears).



5- Notate the volume of the poured $KMnO_4$ solution at the equivalency point. Since the solution of $KMnO_4$ is of dark color, the upper meniscus should be considered for noting the burette readings.

6- Repeat the titration at least three times to obtain an average volume of $KMnO_4$ used.

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Test of the Practical Work N°6: Redox Titrations (Permanganatometric Titration)

1. What is the principle of redox titrations?

2. Give the definition of:

a) **Oxidation:**

b) **Reduction:**

3. Write the redox reactions.

a)

b)

4. Give the Reduction oxidation couple for both reactions?

5. How is the endpoint of a redox titration determined?

6. Give the names of the following glassware:






7. Give the names of these Pictograms:



8. What happens to $KMnO_4$ and H_2O_2 during the titration?

9. What specific name is given to the permanganate titrations? Which indicator is used in the permanganate titration?

10. At the end point of the titration, which chemical is present in the Erlenmeyer flask?

11. How many moles of potassium permanganate were titrated into the flask to reach the endpoint?

12. Write the formula of Potassium permanganate and its ionic form?

Practical Work N°7: Redox titration by Iodimetry

1. Introduction

Iodometric titration, also known as Iodimetry, is a method of analysis that involves the use of iodine/iodide in redox titration. The experiment is done to figure out how much analyte is utilized to make iodine. There are two types of Iodimetry; the direct method, and indirect method.

There are two main types of iodometric analysis**:** direct iodimetry and indirect iodometry**.** In the direct method, an iodine solution is used to titrate the reducing agents, which can be quantitatively oxidized at the equivalence point. As iodine is a weak oxidising agent, only a few substances can be oxidized by iodine. Thiosulphate, one of the common reducing agents, must be determined iodimetrically.

Iodine (I_2) is a mild oxidizing agent.

$I_2 + 2e^- \rightarrow 2I^-$ (reduction)

In the indirect method, also known as 'Iodometry' or 'Iodine titration', the oxidising agent that has to be estimated is treated with an excess of iodide ions and the liberated iodine is estimated by titrating with a standard thiosulphate solution.

Iodide (I^-) is a mild reducing agent.

$2I^- \rightarrow I_2 + 2e^-$ (oxidation)

In this practical work, we'll discuss about indirect Iodometric Titration in detail.

2. Outcomes

- **Gain** knowledge on the principle of the oxidation-reduction titration.
- **Carry out** an indirect titration using a complex experimental protocol and solve stoichiometry problems in the solution phase.
- **Identify** the equivalence point (according to the different appear colors).
- Carry out an
- **Determine** the unknown concentration/molarity of iodine in Iodimetry titration.
- **Use** the procedure to collect accurate and valid data and evaluate the outcome of the investigation in relation to the Iodimetry process.

3. Iodometric Titrations

Iodometric titration is used to determine the strength or the concentration of an oxidizing agent. In this method, $\mathbf{I^-}$ is oxidized into $\mathbf{I_2}$ in the presence of an oxidizing agent. The liberated iodine is estimated quantitatively by titrating with a standard thiosulphate solution. This method can be used to estimate almost all strong oxidizing agents. Here, starch is used as an indicator which gives a dark blue color in presence of $\mathbf{I_2}$.

The steps involved in an iodometric titration are as follows.

- Step 1: An oxidizing agent reacts with iodide ions (example: **KI**, potassium iodide).

$$KI + \textbf{Oxidizing agent} \rightarrow I_2$$

Equivalents of oxidizing agent = Equivalents of I^- oxidized = Equivalents of I_2 formed.

- Step 2: The iodine liberated is titrated with a standard hypo solution ($\mathbf{Na_2S_2O_3}$).

$$I_2 + \mathbf{Na_2S_2O_3} \rightarrow NaI + Na_2S_4O_6$$

From the equation, Equivalents of I_2 = Equivalents of $S_2O_3^{2-}$

Therefore, from the amount of iodine consumed by the thiosulphate ions, the amount of oxidizing agent can be calculated. The combined schematic representation can be given as:

$$\textbf{Oxidizing Agent + KI} \rightarrow \mathbf{I_2 + Na_2S_2O_3} \rightarrow \mathbf{NaI + Na_2S_4O_6}$$

The following are some of the widely used iodometric analyses in the laboratory.

1. $2MnO^-_4(aq) + 10I^-(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 5I_2(aq) + 8H_2O(aq)$

2. $BrO^-_3(aq) + 6I^-(aq) + 6H^+(aq) \rightarrow Br^-(aq) + 3I_2(aq) + 3H_2O(aq)$

3. $Cr_2O^{2-}_7(aq) + 6I^-(aq) + 14H^+(aq) \rightarrow 2Cr^{3+}(aq) + 3I_2(aq) + 7H_2O(aq)$

4. $IO^-_3(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3I_2(aq) + 3H_2O(aq)$

5. $2HNO_2(aq) + 2I^-(aq) + 2H^+(aq) \rightarrow 2NO(aq) + I_2(aq) + 2H_2O(aq)$

4. Experimental Procedure

Laboratory tools: Erlenmeyer flask, burette, graduated cylinder, pipettes, volumetric flasks, beakers, wash bottle, funnel, pipette bulb, stand, magnetic stirrer, magnetic bar and wand.

Laboratory Reagents: Distilled water, Sodium thiosulfate solution ($Na_2S_2O_3$) of 0.1 mol/L concentration, Potassium iodide (KI) or (I_2), and Starch (redox indicator).

5. Manipulation

1. Fill the burette with 0.1 M sodium thiosulfate solution ($Na_2S_2O_3$).
2. Using a pipette, take V = 10 mL of I_2 and pour it into an Erlenmeyer flask or beaker.
3. Introduce the magnetic stir bar, and place the Erlenmeyer flask under the burette on the magnetic stirrer.
4. Slowly drip the solution of the $Na_2S_2O_3$ (reducing agent) into the Erlenmeyer flask to titrate the solution of I_2.
5. When titration is almost complete (the solution takes on a straw-yellow color), add a few drops of starch (the color obtained is dark blue in the presence of iodine).
6. Continue the titration until the dark blue color disappears and becomes colorless (when the blue-black color disappears, the iodine has been completely reduced to the iodide ion).
7. Note the volume of sodium thiosulfate solution at the equivalence point.

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Test of the Practical Work N°7: Redox titration by Iodimetry

1. What's the objective of the practical Work?

2. What are the general steps of iodometric titration?

3. Write the reactions take place in iodometric titration?

4. Give the names of the glassware below:






5. Give the names of these Pictograms:



6. What is the purpose of adding starch solution to the iodometric titration?

7. Why can not the starch solution be added to an iodometric titration at the beginning and added only when the solution becomes pale yellow?

8. How can the end point of an iodometric titration be determined? Explain.

9. At the end point of the titration, give the oxidation state of iodine in the solution?

10. Write the formula of Hypo and sodium tetra-thionate?

11. Calculate the Concentration of sodium sodium tetra-thionate?

12. Calculate how many of iodine reacted with the mole of sodium sodium tetra-thionate?

Practical Work N°8: Titration of Bleach

1. Introduction

An aqueous solution of **sodium hypochlorite**, NaClO, appears as a slightly yellow liquid, and is commonly known as bleach. Aside from its uses as a bleaching agent, sodium hypochlorite solutions are also used as sterilizing agents and in water treatment. Its industrial applications extend to agriculture, food, paper production, and textiles. Furthermore, sodium hypochlorite is introduced into wastewater to mitigate odors.

2. Outcomes

- To determine the concentration of sodium hypochlorite (**NaClO**) in a sample of commercial bleach via a redox titration method and to compare the experimentally obtained concentration with the concentration stated by the manufacturer.

3. Background

Commercial bleaches are made by introducing chlorine gas into a sodium hydroxide solution. During this process, a portion of the chlorine is oxidized to form the hypochlorite ion, ClO^-, and some is reduced to the chloride ion, Cl^-. The solution remains strongly basic. The ionic chemical equation representing this reaction is:

$$\mathbf{Cl_2\,(g) + 2OH^-(aq) \rightarrow ClO\,(aq) + Cl^-\,(aq) + H_2O\,(l)}$$

The concentration of hypochlorite ions in bleach solutions can be determined using oxidation reduction titration. A desirable method would involve identifying a titrant that reacts with sodium hypochlorite to form a colored product. However, there are no simple colorimetric titrant-indicator systems that work effectively. Therefore, a multi-step method must be employed to titrate sodium hypochlorite.

4. Titration of Bleach

One of the best titration analysis of bleach solutions involves using iodide and thiosulfate ions. Iodide ion, I^-, is easily oxidized by almost any oxidizing agent. In acid solution, hypochlorite ions oxidize iodide ions to form iodine, I_2. The iodine that forms is then titrated with a standard solution of sodium thiosulfate.

The analysis takes place in a series of steps:

(1) Acidified iodide ion is added to hypochlorite ion solution, and the iodide is oxidized to iodine.

$2H^+$ (aq) + ClO^-(aq) + $2I^-$ (aq) → Cl (aq) + I_2 (aq) + H_2O (l)

(2) Iodine is only slightly soluble in water. It dissolves very well in an aqueous solution of iodide ion, in which it forms a complex ion called the triiodide ion. Triiodide is a combination of a neutral I_2 molecule with a I ion. The triiodide ion is yellow in dilute solution, and dark red brown when concentrated.

I_2 (aq) + I^- (aq) → I_3^- (aq)

(3) The triiodide is titrated with a standard solution of thiosulfate ions, which reduces the iodine back to iodide ions.

I_3^- (aq) + $2S_2O_3^{2-}$ (aq) → $3I^-$ (aq) + $S_4O_6^{2-}$ (aq)

During the last reaction the red-brown color due to triiodide ion fades to yellow and then to the clear color of the iodide ion. It is possible to use the disappearance of the color of the I_3^- ion as the method of determining the end point, but this is not a very sensitive procedure.

(4) Addition of starch to a solution that contains iodine or triiodide ion forms a reversible blue complex. The disappearance of this blue-colored complex offers a much more sensitive method of determining the end point. However, if the starch is added to a solution which contains a great deal of iodine, the complex which forms may not be reversible. Therefore, the starch is not added until shortly before the end point is reached.

I_3^- (aq) + starch → [I_3^-][starch] (aq)

triiodide + starch → triiodidestarch ion complex (**dark blue**)

The quantity of thiosulfate used in step (3) is directly related to the amount of hypochlorite initially present. The product obtained from oxidation of thiosulfate ion by iodine is the dithionate ion, $S_4O_6^{2-}$.

5. Experimental Procedure

Laboratory Tools:

- Balance, 0.01 g precision ;
- Glass pipettes, 5 ml and 25 ml ;
- Pipette bulb ;
- Weighing dish ;
- Burette and burette clamp ;
- Wash bottle with deionized water ;
- Ring stand Erlenmeyer flask, 125 ml or 250 ml.

Laboratory Reagents:

- Bleach, 5-6% NaClO, commercial, 15 ml ;
- Sodium thiosulfate solution, $Na_2S_2O_3$, 0.100 M, 70 ml ;
- Hydrochloric acid, HCl, 3 M, 6 ml ;
- Starch solution, 2%, 3 ml ;
- Potassium iodide, KI, 6 g.

6. Manipulation:

1. Record NaOCl concentration information from commercial bottle of bleach.
2. Use a pipette bulb and a 5 ml pipette to measure 5 ml of a commercial bleach solution into a 100 ml volumetric flask. To do this, first rinse the pipette with 5 ml "aliquot" (diluted fraction) of bleach and dispose of solution down the sink.
3. Dilute the 5 ml of bleach by adding deionized water to the 100 ml mark, stopper and mix well.
4. Weigh out approximately 2 g of KI. This is a large excess of that which is needed.
5. Pipette 25 ml of dilute bleach into an Erlenmeyer flask.
6. Add the solid KI and about 25 ml of deionized water. Swirl to dissolve the KI.
7. Working in a fume hood, add approximately 2 ml of 3 M HCl while stirring the solution. The solution should be a dark yellow to red-brown from the presence of the I_3^- complex ions.
8. Rinse the burette with deionize water, flush the burette with 5-10 ml of sodium thiosulfate solution to displace the water. This waste solution can go down the sink.

9. Fill the burette with 0.100 M sodium thiosulfate solution. Record the initial burette reading and the solution molarity.
10. Titrate with the standard 0.10 M sodium thiosulfate solution until the iodine color fades to light yellow.
11. Add one dropperful of starch solution. The blue color of the starch-iodine complex may appear.
12. Continue the titration until one drop of $Na_2S_2O_3$ solution causes the blue color to disappear. Record the burette reading.
13. Repeat the titration procedure, starting with step 3, two more times.
14. Dispose of solutions down the sink.
15. Rinse burette with copious amounts of deionized water.

Safety Precautions: Bleach is a corrosive liquid and can cause skin burns. The solution reacts with acid and heat, producing chlorine gas. The solution is moderately toxic by ingestion and inhalation. Keep away from skin and clothing. Hydrochloric acid solution is also toxic by ingestion or inhalation. It can cause corrosion to the skin and eyes.

It is corrosive to skin and eyes.

Caution: adding hydrochloric acid to bleach may release chlorine gas. Carry out procedure step 7 in a fume hood. Wash hands thoroughly before leaving the laboratory.

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Test of the Practical Work N°8: Titration of Bleach

1. Define oxidation and reduction?

- Oxidation:

- Reduction:

2. Write balanced oxidation and reduction half-reactions for the overall redox reaction in section 4 (Titration of Bleach section)?

$2H^+ (aq) + ClO^- (aq) + 2I^- (aq) \rightarrow Cl (aq) + I_2 (aq) + H_2O (l)$ Equation (1)

$I_3^- (aq) + 2S_2O_3^{2-} (aq) \rightarrow 3I^- (aq) + S_4O_6^{2-} (aq)$ Equation (2)

3. For each half-reaction, identify which substance is oxidized or reduced?

- Equation (1):

- Equation (2):

4. Give the names of the following glassware:







5. Give the names of these Pictograms:




6. What are steps for the reaction of titration of bleach? Briefly explain your answer.

a)

b)

c)

d)

7. Describe the procedure used for the titration of bleach?

8. Determine the number of moles of sodium thiosulfate that are equivalent to one mole of sodium hypochlorite. ? Show stoichiometry calculations clearly.

9. Calculate the average volume of $Na_2S_2O_3$ needed for the titration of 25 ml of diluted bleach?

10. Use the average volume and molarity of $Na_2S_2O_3$ to determine the molarity and normality of the diluted bleach?

11. Calculate the average molarity of the commercial bleach (before it was diluted)?

12. Assume the density of the commercial bleach is 1.08 g/ml. Calculate the average percentage by mass of NaClO in the commercial bleach?

Practical Work N°9: Determination of Water Hardness by Complexometric Titration

1. Introduction

Hard water is caused by the presence of naturally occurring calcium and magnesium salts in water. Water hardness is usually noticed because of difficulty in lathering soap and the formation of a scum when washing. Calcium ions (Ca^{2+}) and magnesium ions (Mg^{2+}) form insoluble salts with soaps causing precipitation of this soap scum. Also, hard water can react with dissolved carbonates in water to form a precipitate of insoluble calcium carbonate ($CaCO_3$). This "scale" (also called "lime scale") can build up on the inside of water pipes to such a degree that the pipes become almost completely blocked. Hard water is considered a problem to industries, agriculture and households that have to spend more money on extra soap and the replacement of blocked pipes. Different geographic areas have varying amounts of calcium and magnesium salts in the water, caused by soft rainwater filtering through different rocks dissolving these molecules on the way. Magnesium is found in rocks, such as dolomite and magnesite, whilst calcium is more abundant in limestone and some basalts.

The unit of measurement for hardness is milligrams per litre of calcium carbonate ($mg.l^{-1}$ $CaCO_3$). Water with less than $50\ mg.\ l^{-1}$ of calcium carbonate is regarded as soft, with quantities between 50 and 200 $mg.\ l^{-1}$ as intermediate, and with quantities greater than $200\ mg.l^{-1}$ as hard.

The effect of hard water on health is monitored by the World Health Organisation (WHO). It is found to potentially cause eczema in young children and there may also be a relationship between water hardness and cardiovascular health, so it is recommended to maintain a minimum total hardness of drinking water of $150\ mg.\ l^{-1}$ $CaCO_3$.

2. Outcomes:

- **Recognize** the general principles of a titration and Understand and the chemistry involved in a complexometric titration.
- **Know** about the drawbacks of high water hardness in our real life while performing water hardness test.
- **Appreciate** how to handle liquids in the laboratory.
- **Determine** the total hardness of given water samples through estimation of concentration of the total amount of calcium and magnesium salts in water.

3. Complexometric Titration

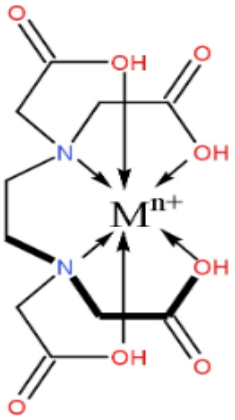
3.1. Definition:

Complexometric method reaction, also referred to as complexometry titration, is a method used to determine the concentration of metal ions that react with a ligand to form a colored metal-chelate complex involves the reaction of metal with.

3.2. Principle of Complexometric Titration

The core concept relies on a reversible reaction between a metal ion and a complexing agent, chelating agent, to generate a stable soluble complex. The endpoint is detected using an indicator that changes color when all the metal ions have been complexed.

- Metal ions (M^{n+}) combine with a chelating agent, ethylene diamine tetra acetic acid (EDTA), to form a stable metal-chelate complex.
- The overall reaction can be expressed as:



Metal ion (M^{n+}) + EDYA (HY^{4-})

↓

[Metal-EDTA complex (HY)]$^{(n-4)-}$ + H^{+}

- The endpoint is indicated by a color change by using a complexometric titration indicator.

3.3. Types of Complexometric Titrations

Depending on the application and nature of the metal ion, there are several types of complexometric titrations:

- **Direct Titration**: Metal ion is directly titrated with the chelating agent.
- **Back Titration**: Excess chelating agent is added to the sample, and the surplus is determined by titration with a standard metal solution.
- **Replacement Titration**: Metal ion replaces another metal ion from a complex, which is then titrated.
- **Indirect Titration**: Applied when direct titration is not possible; involves a reaction preceding the titration.

4. Determination of Water Hardness by Complexometric Titration

Water hardness can be measured using complexometric titration by adding a known concentration of a standard solution of EDTA, using as complexing agent, through a burette to a sample containing an unknown amount of dissolved salts of calcium (Ca^{2+}) and magnesium (Mg^{2+}). EDTA reacts and captures these metal ions creating a larger metal complex. So, whilst there are Ca^{2+} and Mg^{2+} ions in the solution the EDTA will react with them until all metal ions are used up. Since EDTA is insoluble in water, the disodium salt of EDTA is taken for this experiment. EDTA can form four or six coordination bonds with a metal ion. The end point of this titration is indicated by a change in the color of the solution, aided by the addition of an indicator. Mg^{2+} and Ca^{2+} ions are colorless at pH 10 but the solution will turn pink to red in color when adding the indicator Eriochrome Black T (ErioT). On titration with EDTA, the solution will eventually turn blue when sufficient EDTA has been added and reacted with all the Ca^{2+} and Mg^{2+} ions present in the sample.

An ammonia buffer solution is used to make sure that the sample stays at pH 10. This is important because at this pH, a marked color change is observed at the end point and the EDTA will not react with any other metal ions in the solution (such as Fe^{2+}) that may be present in the water sample.

5. Experimental Procedure

Laboratory tools: Burette (50ml), Erlenmeyer flask (250 ml), Pipette (25 ml), Magnetic stirrer, Beaker (100 ml).

Laboratory Reagents: Distilled water, 0.01 $mol.l^{-1}$ EDTA (ethylene diamine tetra-acetic acid), Eriochrome Black-T, Ammonia Buffer solution (pH 10), Water sample to be analysed.

6. Manipulation

a) Preparation of the sample:

- Take 50 mL of the water sample to be analysed and place in a 250 mL Erlenmeyer flask.

b) Add the indicator:

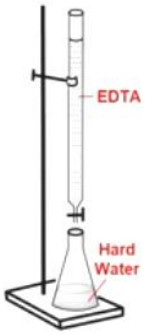
- Add about 2 to 3 drops of Eriochrome Black T indicator to the water sample.
- The solution should turn wine red in the presence of Ca^{2+} and Mg^{2+}.

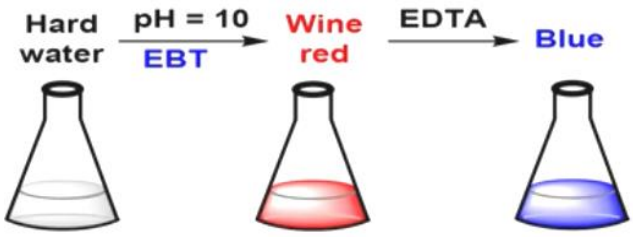
c) Add the buffer solution:

- Add 5 mL of ammonia buffer solution (pH 10) to the sample.
- This step is essential to maintain the optimum pH for the reaction between the metal ions and EDTA.

d) Titration:

- Fill the burette with the 0.01 M EDTA solution.
- Place the Erlenmeyer flask containing the sample on the magnetic stirrer.
- Titrate slowly with the EDTA solution while constantly shaking the sample.
- Observe the change in color of the solution.
- The end point of the titration is reached when the color of the solution changes from wine red to blue.
- Repeat the operation two to three times.





$$(M^{2+} = Ca^{2+} / Mg^{2+}) + EBT \rightarrow (M^{2+})\text{-EBT Complex}$$

(Blue) (Wine red)

$$(M^{2+})\text{-EBT Complex} + EDTA \rightarrow (M^{2+})\text{-EDTA Complex} + EBT$$

(Wine red) (Colorless) (Colorless) (Blue)

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Test of the Practical Work N°9: Determination of Water Hardness by Complexometric Titration

1. What is the principle of complexometric titration?

2. What are the types of complexometric titrations?

3. Differentiate between complexometric titration and redox titration?

4. How is the endpoint detected in complexometric titration?

5. What are the steps involved in the determination of water hardness using complexometric titration? Briefly explain your answer.

6. What is the unit of hardness of water?

7. Fill the Table and determine the volume of EDTA solution used to reach the end point?

	Trial 1	**Trial 2**	**Trial 3**
Initial burette reading (ml)			
Final burette reading (ml)			
Volume of EDTA solution used (ml)			

8. EDTA or other chelating agents can be found many products such as in shampoos, soaps, cleaning products, plant foods, salad dressings, canned foods, etc. Choose such a product and suggest the function of EDTA in that product?

9. Explain why we used a pH 10 buffer for our total water hardness titrations?

10. Using the following relationship: $\mathbf{C_{sample} = V_{EDTA} \times C_{EDTA} / V_{sample}}$. Determine C_{sample} in mol/l from the volume of EDTA added?

11. The total hardness (D) of the water is calculated using the following relationship:

D (°f) = C_{sample} / X

When; **C_{sample} = $[Ca^{+2}]+[Mg^{+2}]$ et X = 10^{-4} mol/l (limestone)**

Measure the mass concentration of calcium and magnesium in the selected water?

12. Compare the results obtained with the values indicated on the label of the water analyzed?

Notes: 1°f = 4 mg/l of Ca^{+2} = 2.4mg/l. of Mg^{+2}

1°f (French degree) of water hardness equals 10 mg/L (or 10 ppm) of calcium carbonate ($CaCO_3$).

General Conclusion

Colorimetric titration is a fundamental analytical technique with wide-ranging applications in domestic and industrial settings, including food quality control, detergent evaluation, and water treatment. Through this practical works, students have had the opportunity to examine several common household products and understand their chemical characteristics, such as acidity, bleaching potential and scale-inhibiting behavior. Recognizing these properties is crucial not only for scientific understanding but also for safe and responsible use of these substances in daily life.

The laboratory practical work focused on three titration techniques. Acid base titration was used to measure the acidity of vinegar and the Dornic degree of dairy products, linking chemical analysis to food quality. Oxidation reduction titration helped determine the chlorometric strength of bleach, demonstrating how redox reactions assess disinfectant effectiveness. Complexometric titration was applied to evaluate water hardness by measuring calcium (Ca^{2+}) and magnesium (Mg^{2+}) ions, providing insight into water quality.

It is essential to stress the importance of chemical safety. For example, mixing bleach with acidic substances like vinegar or certain detergents can generate chlorine gas (Cl_2), which is highly toxic and potentially fatal. The findings also underscore the importance of chemical safety in domestic environments. For example, mixing bleach with acidic substances such as vinegar or certain detergents can trigger hazardous chemical reactions, leading to the release of chlorine gas (Cl_2), which is highly toxic and potentially fatal. Students must understand not only how to perform titrations accurately but also the real-world implications of chemical reactions outside the laboratory.

In conclusion, these practical works reinforce the connection between theoretical chemistry and its applications. By mastering titration techniques, students gain critical skills for analyzing chemical properties, understanding product behavior, and promoting safety in both laboratory and everyday settings.

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