

**UNIVERSITY ABOU BAKR BELKAID OF TLEMCE**

**FACULTY OF SCIENCES**

**DEPARTEMET OF CHEMESTRY**



**Chemestry practical work**

**1<sup>st</sup> Year LMD SM**

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***1<sup>st</sup> semester***

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## 1<sup>st</sup> Practical worck

### Chemistry laboratory: Initiation, safety rules and materials

#### Introduction

**To the Student:** A significant amount of your training in chemistry will take place in the laboratory. The following instructions should be read carefully before you attend the first laboratory session. These instructions will help you make efficient use of your time while in the laboratory and also promote laboratory safety.

#### Objectives

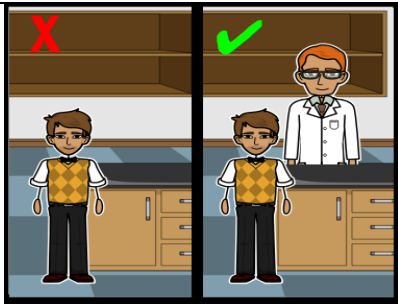

- Know the safety rules in the laboratory
- Identify the equipment used in the laboratory
- Know the different danger pictograms

#### Pre-lab preparation

Before coming to the laboratory, you must read carefully the practical work. The questions have been chosen to draw your attention to specific techniques and precautions that you should be aware of before you start the experiment. The experiments are designed to allow you to collect the data in **3 hours**, make sure you arrive on time, since your instructor will provide additional instructions for experiments as needed.

#### Laboratory safety

Prevention is the first basic safety step. Preventing accidents involves having a good knowledge of the work to be done, respecting safety signs, having good behavior in the laboratory, exercising effective personal protection, labeling, storing and disposing of chemicals correctly. The following table show the most of them:

Safety rules	Instructions to follow
 <p>Do not perform unauthorized experiments or work in a laboratory alone</p>	Teacher presence required
 <p>All personal belongings should be placed in the bookcases as you enter the laboratory</p>	workspaces must be free



\*wear lab coats at all times. Your legs must be completely covered below the knee by your lab coat.

\*Approved eye protection must be worn at all times in the laboratory.

\*Wearing gloves is required



\* Long hair and loose clothing must be confined while in a laboratory

\*The lab coat must be cotton

\*Eye protection must be splash proof chemical goggles.

\*If you do get a chemical in your eye rinse immediately with large quantities of water using the eye-wash stations.



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




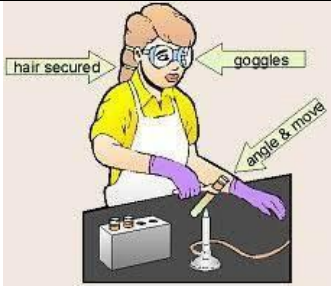


Smother the fire with a damp towel on a person: lay the person on the ground and cover them with the fire blanket





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

Thoroughly wash your hands after leaving the laboratory





 <p>Mouth suction is never used to fill a pipette.</p>	<p>rinse mouth Do not give drink Do not induce vomiting</p>
 <p>Never direct the open end of test tube toward yourself or anyone else</p>	
 <p><b>Never pour water into concentrated acid</b></p>	 <p><b>If an acid is to be diluted, pour acid slowly into the water with constant stirring.</b></p>
 <p>Liquid and solid waste containers must be properly used at all times ,and must be disposed of appropriately.</p>	<p>To avoid contamination of water and soil by the chemicals used</p> <p>Some used chemicals can be flushed down the sink drain with water.</p>
 <p>Never place chemicals directly on the balance pan.</p>	<ul style="list-style-type: none"><li>*Always use a proper weighing container when using a balance to weigh a chemical.</li><li>*Never return unused chemicals to their original container.</li><li>* Securely replace lids, caps, and stoppers after removing reagents from containers.</li><li>*Always wipe spatulas clean before and after inserting into reagent bottles.</li></ul>



 <p>Use the fume hoods when toxic or irritating vapors are involved</p>	Get out and breathe fresh air
 <p>HELP!</p>	<b>In any emergency, to get the lab supervisor attention is to SCREAM!</b>

### General laboratory procedures

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

General laboratory procedures	
 <p>Scrub inside and out with a brush, detergent, and tap water.</p>	
 <p>Rinse away all suds with tap water.</p> <p>Rinse the inside of the glassware two or three times with minimal amounts of distilled water. (Distilled water is expensive and should be used sparingly.)</p> <p>If dry glassware is needed immediately, rinse the equipment twice with small amounts of acetone. The residual acetone in the equipment will vaporize quickly and leave no residue.</p>	





Shake out as much rinse water as possible and dry the outside with a towel.





Make sure your work area is clean and dry.  
Clean up any spill immediately.  
Never place anything that is not directly required for the experiment on laboratory desks;  
other items may interfere with the experiment

- 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.
- 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.

### Chemical hazard information

The label affixed to the containers of marketed products has the role of informing the user about the dangerous properties. It must include :

- The name of the substance as well as its formula
- The dangers symbols (pictograms)
- One or more risk phrases
- One or more safety tips
- Some physicochemical properties and other indications.

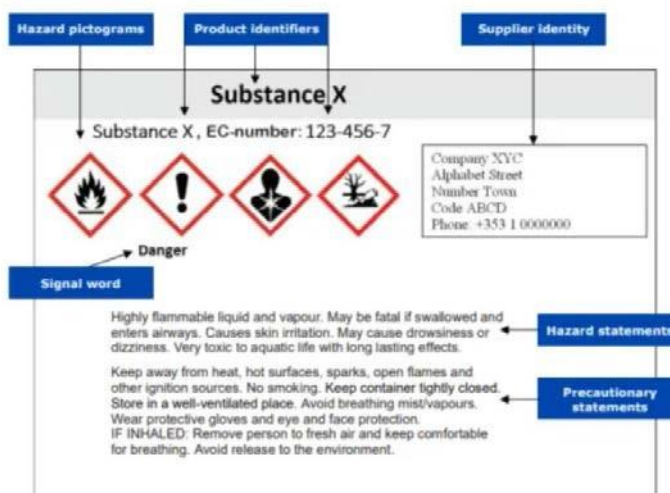
	Sulfate de Cuivre	1kg	CONTENANCE <b>1 kg</b>	UN 1469
	$\text{CuSO}_4$			
		95 %	Etiquetage CE 233-245-9	N° Lot K130/04048
Xn	Masse Molaire, Densité, Point de Fusion (m.p.) Point d'ébullition (b.p.)	R22 R36/38	Utiliser avant : 14/08/2006	
	S22		R 61-62-20/22-33 S 53-45	
<b>Plomb (II) nitrate</b>				
 RISQUE PENDANT LA GROSSESSE D'EFFETS NEFASTES POUR L'ENFANT. RISQUE POSSIBLE D'ALTÉRATION DE LA FERTILITÉ. ÉGALEMENT NOCIF PAR INHALATION ET INGESTION. DANGER D'EFFETS CUMULATIFS.				
T - Toxique Eviter l'exposition - se procurer des instructions spéciales avant utilisation. En cas d'accident ou de malaise, consulter immédiatement un médecin (si possible lui montrer l'étiquette).				

### Product Risk Phrases, Safety Phrases and Storage Phrases



There are three types of phrases on a product label:

- **Risk Phrases or H in English “Hazard”** : R1, R2, R68 etc...ex: R1 : Explosive when dry
- **Safety Phrases: S1, S2, ... S64,....**
- **Product storage and handling phrases** : S25 : Avoid contact with eyes; S49: Keep only in the original container.








### Symbols used on labels (pictograms)

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

<i>Symbol</i>	<i>Meaning</i>	<i>Risks</i>	<i>Precautionary advice</i>
	<b>Flame over Circle</b>	Oxidizers	These chemicals give off oxygen and can make a fire spread.
	<b>Flame</b>	Flammable • Pyrophoric • Self-heating • Emits Flammable Gas • Self-reactive • Organic peroxides	These chemicals burn or can release gases that burn.
	<b>Exclamation mark</b>	Irritant (skin and eye) • Skin Sensitizer • Acute Toxicity (harmful) • Narcotic Effects • Respiratory Tract Irritant • Hazardous to Ozone Layer	Cause health problems. Usually less toxic than chemicals labeled with the health hazard or skull and cross-bone pictograms. Also used for chemicals that can destroy the ozone layer.
	<b>Skull and Cross-bone</b>	Acute Toxicity (fatal or toxic)	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, bone.

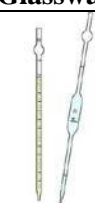
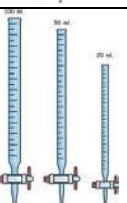





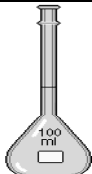


	<b>Exploding Bomb</b>	Explosive , self-reactive , organic peroxide	These chemicals can explode.
	<b>Corrosion</b>	Skin Corrosion/Burns • Eye Damage • Corrosive to Metals	These chemicals cause permanent damage to skin or eyes. These chemicals destroy metals.
	<b>Environment</b>	Aquatic toxicity	These chemicals are dangerous if they get into rivers, lakes or oceans.
	<b>Gas Cylinder</b>	Gases under pressure	Gases and liquids under pressure Can explode. This pictogram is used for both pressurized gases and liquefied gases such as liquid nitrogen.
	<b>Health Hazard</b>	Carcinogen . Mutagenicity .Reproductive Toxicity. Respiratory Sensitizer. Target Organ Toxicity. Aspiration Toxicity	Cause serious health problems. Some problems show up immediately, but some may show up much later.

### Usual glassware and utensils used in Practical Chemistry




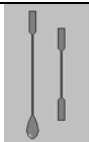


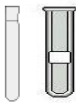
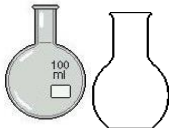




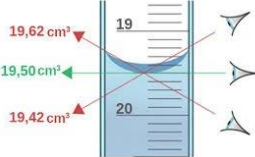
Laboratory equipment is generally used either to perform a manipulation or experiment or to perform measurements and collect data. The volume of the glassware used must be adapted to the handling:

Glassware	Utility and precision
	Pipette for very precise volume measurement Graduate pipette for various volume measurement
	Burette for precise volume measurement
	Graduated cylinder for precise volume measurement



	Volumetric flask very precise measurement
	Beaker, for inaccurate measurement , and container for transferring
	Erlenmeyer, for inaccurate measurement and chemical reaction (dosage, oxidation-reduction)

All chemistry laboratories mainly share common laboratory equipment, glassware and characterization devices. This includes:

Equipment and glassware	Utility
 Hot plate stirrer: for heating, stirring and mixing reagents	 The balance: to weigh reagents
 Wash bottle: may contain distilled water or other solvent	 Spatula: for solid powder samples
 Suction device (pear) to suck up the solutions	 Thermometer : taking reaction temperatures
 Test tube container for chemical tests	 Florence flask for synthesis reaction
   	 correct reading



## 2<sup>nd</sup> PRATICAL WORK

### PREPARATION OF SOLUTION

#### Objective of practical work:

The main objective of this manipulation is to prepare a solution from:

1. **Commercialised solution** of sulfuric acid  $\text{H}_2\text{SO}_4$ .
2. **Mother solution** prepared in question 1: the prepared solution in this case named **daughter solution**.
3. **A solid chemical** as well as potassium hydroxide KOH.

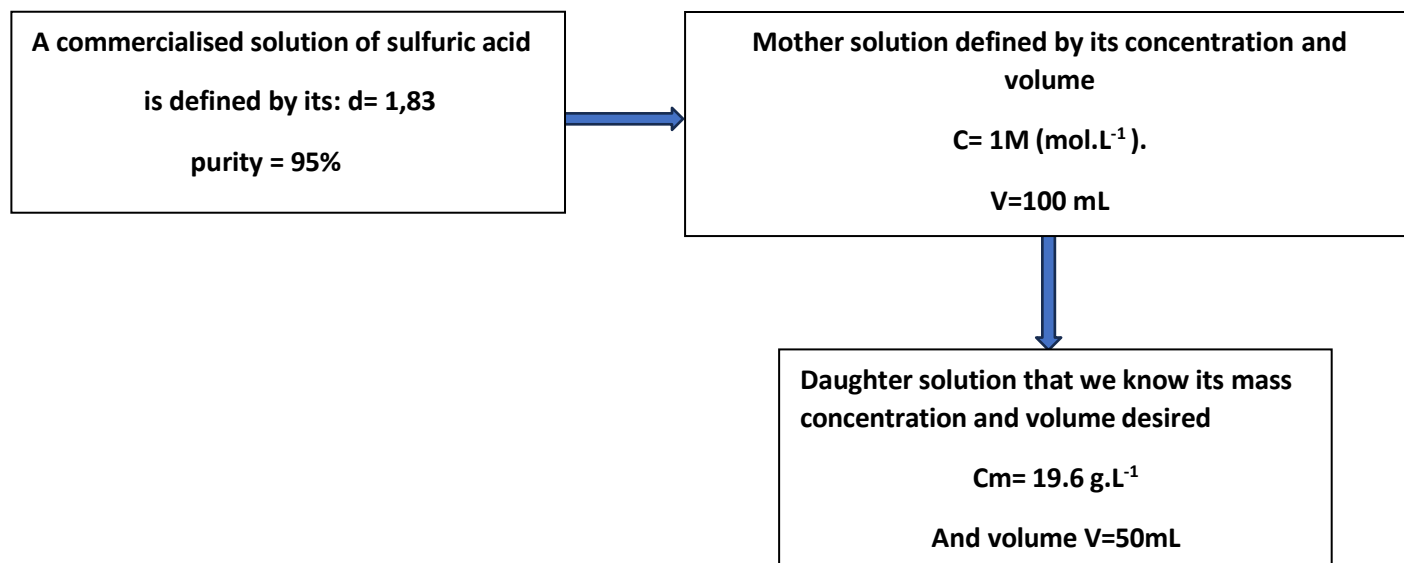
#### Principal of manipulation:

These solutions can be prepared by:

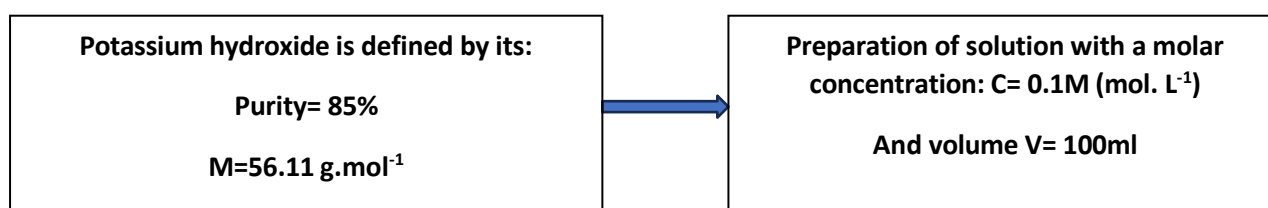
1. **Dissolution liquid-liquid**: in this case a precise volume taken from the commercialised solution is dissolved in volume of solvent (e.g. distilled water).
2. **Dilution**: consist in preparing, from a **mother solution**, that we know its concentration, a **daughter solution** whose concentration is lower.
3. **Dissolution solid-liquid**: here, a calculated amount of chemical solid is dissolved in solvent.

#### Operating mode:

1. Preparation of mother and daughter solutions of sulfuric acid: **Dissolution liquid-liquid**



2. Preparation of stock solution from chemical solid potassium hydroxide KOH: **dissolution solid-liquid**





**Reminder:**

- Normality (N): is the number of gram-equivalents of *solute* per liter of *solution*.
- Normality = number of gram equivalents x Molarity;  $N = z \cdot M$
- The gram-equivalent: is the quantity of solute comprising one mole of the particles considered ( $H^+$ ,  $OH^-$  etc.)
- The mass titer (mass concentration): is the weight concentration expressed in mass unit per liter of solution, generally expressed in  $g \cdot L^{-1}$ ;  $C_m = m/V$ ; MM (molar mass) =  $C_m/C_n$ .

**Note:** "Addition of solvent (eg. water) to a solution does not change the amount of solute, but it changes the concentration of solution, so in this case we can write:

$$n_1 = n_2 \Rightarrow N_1 V_1 = N_2 V_2 \text{ (when } z \neq 1)$$

$$\text{and } C_1 V_1 = C_2 V_2 \text{ (when } Z = 1)$$

**Equipment used**

Volumetric flask (V=50 and 100 mL);

Volumetric and graduated pipette (V=10 and 5 mL);

Beaker (V=50 and 100 mL); Pipette pear; Spatula;

Funnel; Electronic scale; Watch glass.

**Chemicals used**

Commercialised solution of  $H_2SO_4$  (95%,  $d=1.83$ );

Potassium hydroxide KOH;

Distilled water.

**Work to be done**

**Safety rule:** The dilution of a concentrated acid in water **releases heat**, can cause **spattering**. Therefore, we can receive droplets of acid solution on ourself. So, it is important to remember this **safety rule** when preparing acidic solution:

**when diluting a concentrated acid, put some water in the volumetric flask before to introduce the amount of concentrated solution. Mix and then top up to the gauge line.**

**1. Preparation of 100 mL of sulfuric acid (1 M): preparation of mother solution**

Calculate **the mass of solute (acid)** necessary for the preparation of the requested solution? Deduce **the volume of concentrated acid** needed?

In a **100 mL** volumetric flask, **put some distilled water**. Using a burette under **fume hood**, take the calculated volume of  $H_2SO_4$ . Fill with distilled water up to the mark, close then shake (**follow diagram 01**).

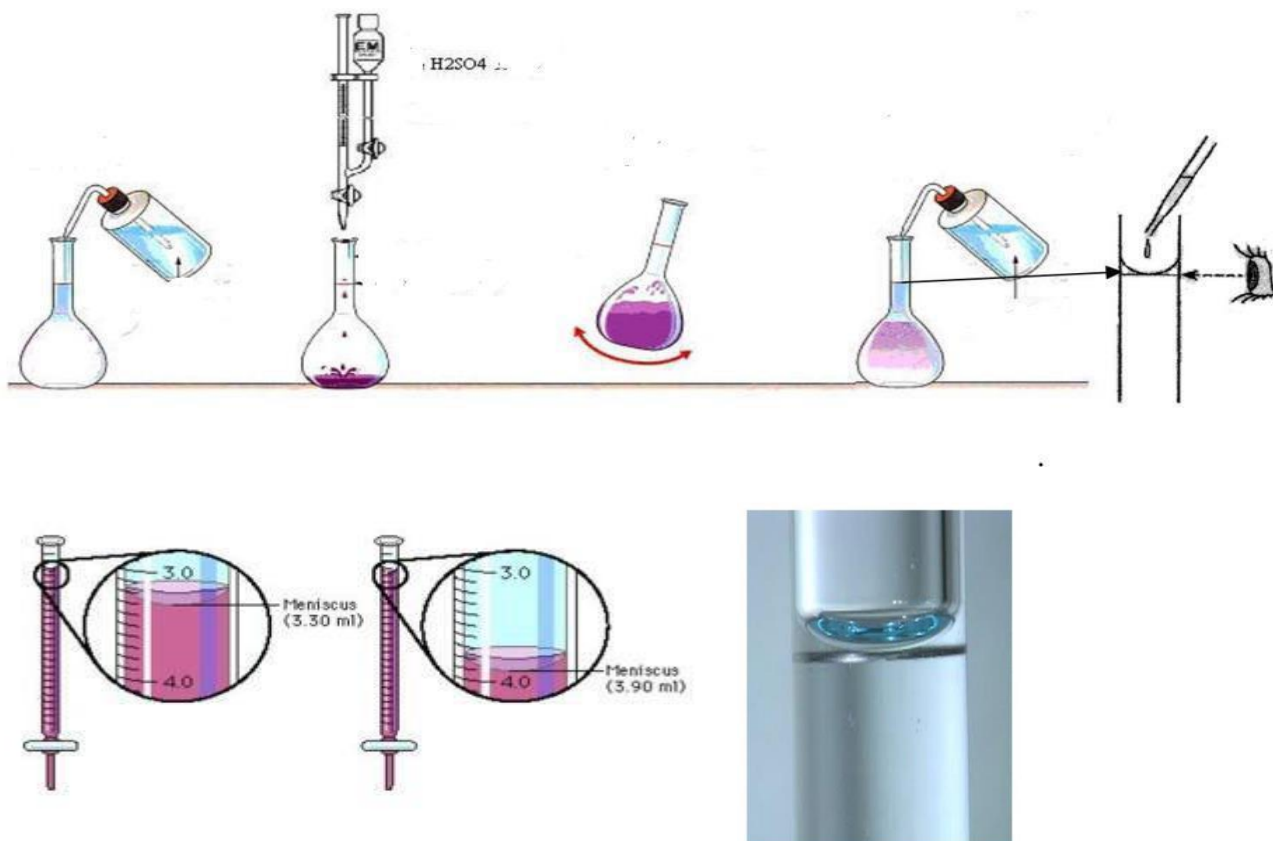


Diagram 01

## 2. Preparation of 50 mL of sulfuric acid (19.6 g/L): preparation of daughter solution

Calculate **the necessary volume** of to be taken from **the mother solution** for preparing **the daughter solution**?

In a **50 mL** volumetric flask, introduce the calculated volume using a graduated pipette. Fill the flask up to the mark with distilled water, close and shake. (**Follow the diagram 02**).

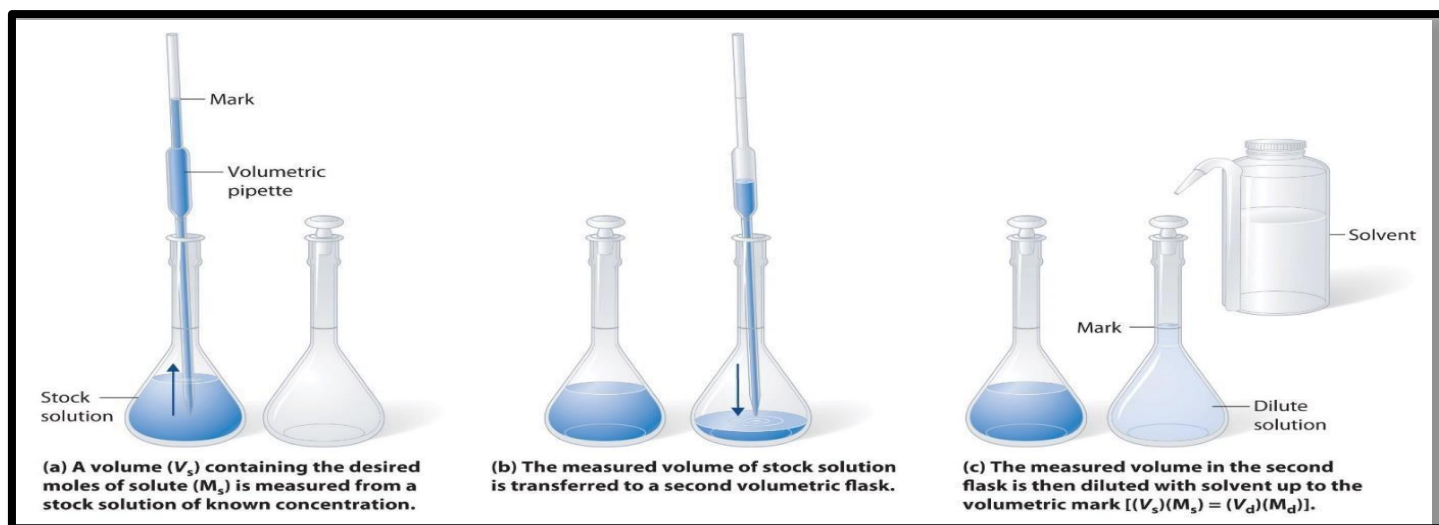


Diagram 2



3. Preparation of 100 mL of potassium hydroxide (0.1 M):

**Safety rule:** don't touch the pellets of hydroxide sodium with fingers. Use the watch glass to weigh and gloves to protect your hand, this chemical is very corrosive.

**Quickly close the bottle to prevent the sodium hydroxide from becoming hydrated and carbonated in the air**

1. Calculate the mass of sodium hydroxide (KOH) required for the requested solution?
2. Weigh the calculated mass.
3. In a 100 mL volumetric flask filled halfway with distilled water, add the calculated mass of KOH. Shake until complete dissolution then complete to the mark (Follow diagram 03).

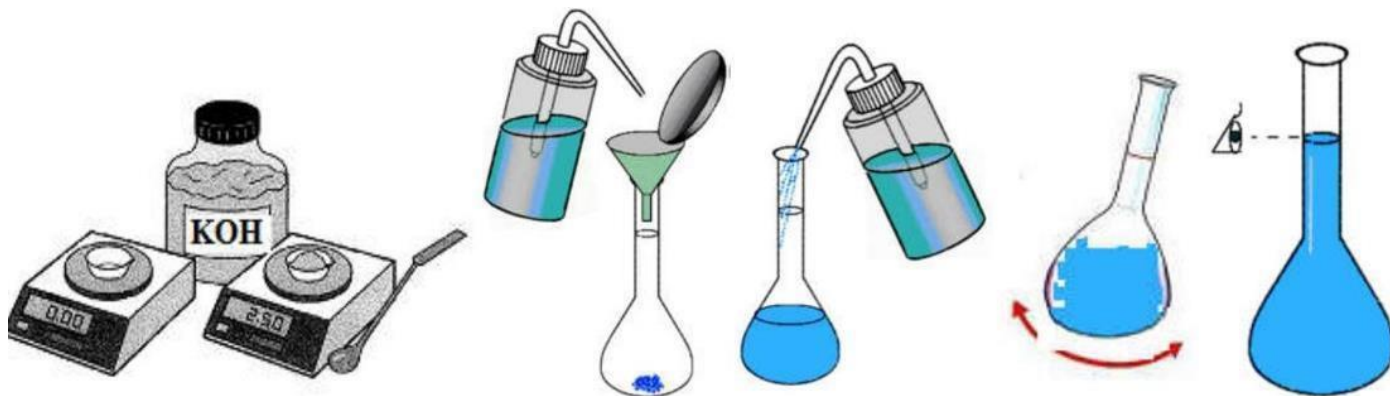


Diagram 3





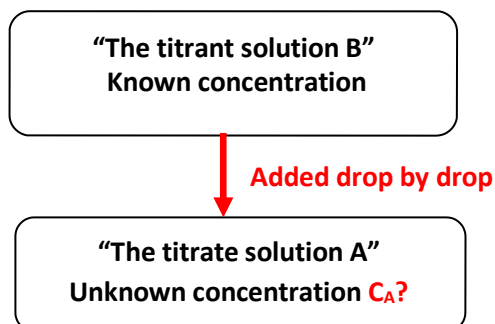
### 3<sup>rd</sup> Practical work: Volumetric acid-basic titration

#### 1/Titration of a solution:

A titration reaction is used to determine the concentration of a species in solution ( $C_A = \dots?$ )

To do this operation:

A first solution containing the species of **unknown concentration** “**the titrate solution A**” is brought into contact with a second solution containing a reagent of **known concentration** “**the titrant solution**”. The **titrant solution** is gradually poured into the titrate solution **drop by drop** until the equivalence point is reached.



At the equivalent point, the two reagents (**A** and **B**) are mixed in **stoichiometric proportions**, which allows the unknown concentration to be deduced.

$$\text{At the equivalence point} \\ n_{\text{eq.g}}(\text{Acid}) = n_{\text{eq.g}}(\text{Base})$$

A dosage (or titration) acid-base can be followed by:

**pH-metric:** we follow the evolution of the pH during the reaction.

**Colorimetry:** we use a colored indicator, which will be studied in this practical work.

**A colored indicator:** is a reagent whose color depends on the medium (or pH). It can be used to mark the end of a dosage if equivalence is reached in its turning area (table 1).

Table 1 : Exemples of colored indicators:

Indicator	Acid form	Turning area	Basic form
Helianthine	Red	3,1 - 4,4	Yellow
Methyl red	Red	4,08 - 6	Yellow
Bromothymole bleu	Yellow	6,0 - 7,6	Bleu
Phenol-phtaleine	Incolore	8,2 - 10,0	Pink
Alizarine Yellow	Yellow	10,1 – 12,2	Red



## 2/Objectives:

The main objective of this practical work is to:

Determine the molar concentration of an acid solution with acid-basic titration.

## 3/Principal of manipulation :

This titration is based on a colorimetric dosage.

## 4/Material and products:

- |   |  |
|---|--|
| -Burette (25-50 mL)                       | -Sodium hydroxide solution (0.1M NaOH)                 |
| -Graduated or volumetric pipettes (10 mL) | - HCl hydrochloric acid solution                       |
| -Pro-pipette                              | -H <sub>2</sub> SO <sub>4</sub> sulfuric acid solution |
| -Volumetric flasks (50 and 100 mL)        | -Colored indicator                                     |
| -Erlenmeyer flask (100 mL)                |  |
| -Beakers                                  |  |

## 5/Dosage of a monoacid using a strong base:

The dosage of the hydrochloric acid **HCl** solution **S2** will be carried out with a sodium hydroxide **NaOH** solution of molar concentration **C<sub>B</sub> = 0.1 mol/L**, with some drops of phenolphthalein as a colored indicator.

The global reaction equation is:



### 5.1/ Preparation of the solution to titrate (HCl hydrochloric acid solution S2):

1. Take a clean beaker, and pour a small quantity of HCl solution of unknown concentration S1.
2. Rinse the 10 ml pipette with the solution S1 (use the pro-pipette)
3. Take a volume of 10 ml of the solution S1 (use the rinsed pipette) .
4. Introduce this test portion into the 100 ml volumetric flask which contains a small quantity of distilled water
5. Complete with distilled water up to the jauge line
6. Close the volumetric flask with the stopper and shake .
7. You obtain a solution S2 (HCl of unknown concentration).

### 5.2/ Rapid dosage (equivalent volume supervision):

The rapid dosage is performed before the precis dosage to determine the equivalence point interval (the equivalence point will be included in a volume interval).

#### Work to be done :

1. Check that the burette tap is properly closed.
2. Rinse the burette with distilled water, then with the titrant solution (NaOH **C<sub>B</sub> = 0.1 mol/L**).
3. Degas the bottom of the burette by draining the titrant solution into a labeled beaker.
4. Fill the burette with NaOH **C<sub>B</sub> = 0.1 mol/L**.
5. Adjust the liquid level to the zero level of the burette by draining the NaOH excess into the labeled beaker 1.
6. Pour approximately 40 mL of solution (**S2**) into a labeled beaker 2
7. Take 10 mL of this solution using a clean volumetric pipette fitted with a pro-pipette.
8. Introduce this test portion into an erlenmeyer flask add an amount quantity of water



9. Add 1 to 3 drops of phenolphthalein indicator.
10. Place the erlenmeyer flask under the burette (*figure 1*)
11. Add the titrant solution NaOH mL per mL, and note the color of the solution by completing Table 2.
12. Shake manually.
13. The solution changes color when you added the equivalent volume of titrant solution (**V<sub>eq</sub>**). Indicate approximately this volume (by a frame): **V 1 mL < V<sub>eq</sub> < V 2 mL**

$$\text{..... } V_1 \text{ mL} < V_{eq} < \text{..... } V_2 \text{ mL}$$

Table 2 : Volumes equivalents

V <sub>NaOH</sub> titrant (mL)	1	2	3	4	5	6	7	8	9	10
Color solution										

### 5.3/ Precise dosage (drop dosage):

The precise dosage (by drop by drop), is carried out after the rapid dosage to determine equivalence points with precision. The titrant solution is poured up to 1.5 mL or 2 mL of the equivalence zone quickly, then by drop by drop until the equivalence. (first drop that changes the color of the solution)

#### Work to be done:

1. Adjust the liquid level to the zero level of the burette by adding the NaOH.
2. Pour approximately 40 mL of solution (**S<sub>2</sub>**) into a labeled beaker 2
3. Take 10 mL of this solution using a clean volumetric pipette fitted with a pre-pipette.
4. Introduce this test portion into an erlenmeyer flask add an amount quantity of water
5. Add 1 to 3 drops of phenolphthalein indicator.
6. Place the erlenmeyer flask under the burette (*figure 1*)
7. Quickly at the beginning add the titrant solution NaOH to the solution, until the value of **V<sub>1</sub>** (determined at the quick dosage)
8. Then add the titrant solution **drop by drop**, as the color change approaches (equivalence point). Close the tap of the burette as soon as the first drop changes color, shake.
9. Note the equivalent volume **V<sub>eq</sub>** and the color of the solution by completing Table 3.
10. Repeat the operation two to three times.

Table 3 : Equivalents volumes :

	1 <sup>st</sup> test	2 <sup>nd</sup> test	3 <sup>rd</sup> test
V <sub>eq</sub> (mL)			
Color solution			

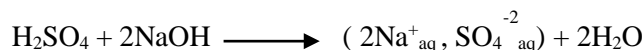
#### Questions:

1. Deduce the average equivalence volume (V<sub>eq-ave</sub>)?
2. Calculate the concentration of hydrochloric acid solution **S<sub>2</sub>**?
3. Deduce the concentration of solution **S<sub>1</sub>**



## 6/ Dosage of a diacid $\text{H}_2\text{SO}_4$ using a strong base $\text{NaOH}$ :

In this case , the dosage of sulfuric acid  $\text{H}_2\text{SO}_4$  will be carried out using a solution of sodium hydroxide  $\text{NaOH } C_B = 0.1 \text{ mol/L}$  , with some drops of phenolphthalein as a colored indicator  
The global reaction equation is:



### 6.1/ Rapid dosage (equivalent volume supervision):

1. Adjust the liquid level to the zero level of the burette by draining the  $\text{NaOH}$  excess into the labeled beaker 1.
2. Pour approximately 40 mL of  $\text{H}_2\text{SO}_4$  solution into a labeled beaker 3
3. Take 10 mL of this solution using a clean volumetric pipette fitted with a pro-pipette.
4. Introduce this test portion into an erlenmeyer flask add an amount quantity of water
5. Add 1 to 3 drops of phenolphthalein indicator.
6. Place the erlenmeyer flask under the burette (*figure 1*)
7. Add the titrant solution  $\text{NaOH}$  mL per mL, and note the color of the solution by completing Table 4.
8. Shake manually.
9. The solution change color when you added the equivalent volume of titrant solution ( $V_{\text{eq}}$ ) .
10. Indicate approximately this volume (by a frame):

$$\dots\dots V_1 \text{ mL} < V_{\text{eq}} < \dots\dots V_2 \text{ mL}$$

Table 4: Volumes equivalents

$V_{\text{NaOH titrant (mL)}}$	1	2	3	4	5	6	7	8	9	10
Color solution										

### 6.2/ Precise dosage (drop dosage):

11. Adjust the liquid level to the zero level of the burette by adding the  $\text{NaOH}$ .
12. Pour approximately 40 mL of solution  $\text{H}_2\text{SO}_4$  into a labeled beaker 3
13. Take 10 mL of this solution using a clean volumetric pipette fitted with a pro-pipette.
14. Introduce this test portion into an erlenmeyer flask add an amount quantity of water
15. Add 1 to 3 drops of phenolphthalein indicator.
16. Place the erlenmeyer flask under the burette (*figure 1*)
17. Quickly at the beginning add the titrant solution  $\text{NaOH}$  to the solution, until the value of  $V_1$  (determined at the quick dosage)
18. Then add the titrant solution **drop by drop**, as the color change approaches (equivalence point).
19. Close the tap of the burette as soon as the first drop changes color, shake.
20. Note the equivalent volume  $V_{\text{eq}}$  and the color of the solution by completing Table 5.
21. Repeat the operation two to three times.

Table 5 : Equivalents volumes

	1 <sup>st</sup> test	2 <sup>nd</sup> test	3 <sup>rd</sup> test
$V_{\text{eq (mL)}}$			
Color solution			

### Questions:

1. Deduce the average equivalence volume ( $V_{\text{eq-ave}}$ )?
2. Calculate the concentration of sulfuric acid  $\text{H}_2\text{SO}_4$  ?
3. Calculate the normality of the sulfuric acid solution?

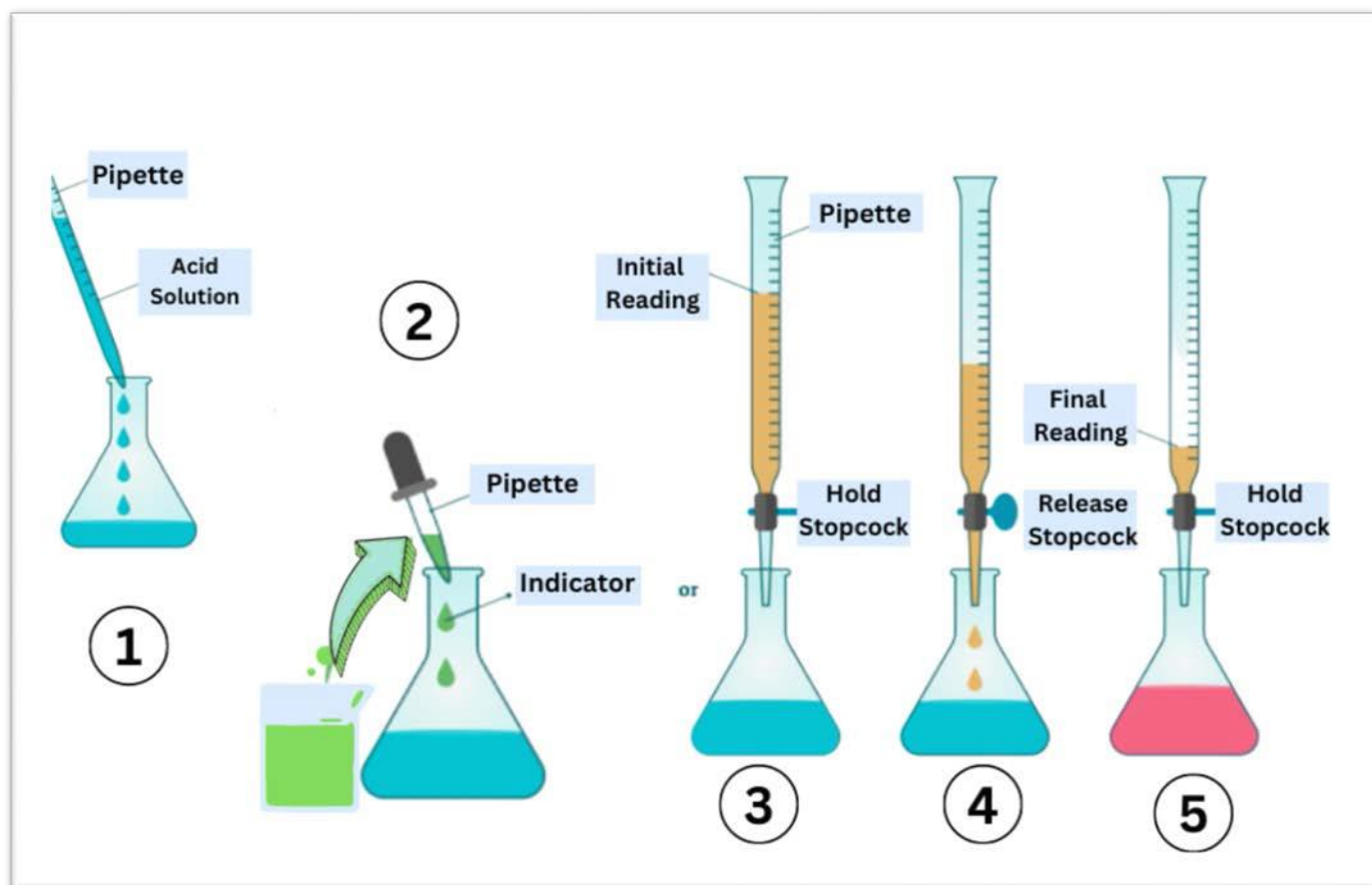


Figure 1 : Acid-base titration



## 4<sup>th</sup> Practical work: Titration by the Oxidation-Reduction Method

### Case: Manganimetry

### Terminology

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

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

i.e.:

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

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

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

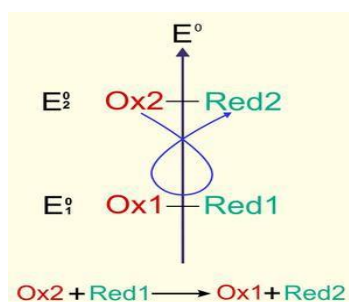
Or, **in an oxidation-reduction reaction, the reductant is oxidized and the oxidant is reduced**.



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

**The greater the  $E^\circ$ , the stronger the oxidant.**

for this, we can follow **the gamma rule**.



**the gamma rule**

### Examples:

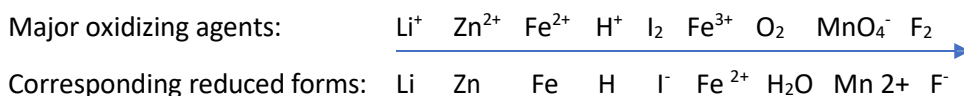
The transition of iron "Fe" into a solution corresponds to an oxidation:  $\text{Fe} \longrightarrow \text{Fe}^{2+} + 2 e^-$ .

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





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



## Objective of the manipulation

The main objective of this practical work is to determine the normality of a solution of  $\text{FeSO}_4$  by **manganimetry titration in an acidic medium**.

## Principal of practical work

**Manganimetry** is a dosing (titration) method based on **redox reactions** involving the **permanganate ion  $\text{MnO}_4^-$** , which is a chemical species capable of **capturing electrons**.

A **manganimetry titration** therefore involves the “colored” redox couple:  **$(\text{MnO}_4^-/\text{Mn}^{2+})$**  with a standard potential of (1.507 V).

Potassium permanganate  $\text{KMnO}_4$  is a strong oxidant for all chemical species, their corresponding reductant can be  $\text{Mn}^{2+}$  (**colorless**),  $\text{Mn}^{3+}$ , or  $\text{MnO}_2$  (**brown**), depending on **the reaction medium**, either **acidic** or **basic**. Thus, the **oxidizing form  $\text{MnO}_4^-$**  is **purple**, the **reducing form  $\text{Mn}^{2+}$**  is **colorless**, enabling the equivalent point to be determined **without the use of color indicators**.

**Note:** If the medium **is not acidic enough**, the couple involved is  **$\text{MnO}_4^-/\text{MnO}_2$** . As  $\text{MnO}_2$  is a brown solid with low solubility, the equivalence point can no longer be determined. It is therefore necessary to acidify the medium by adding concentrated sulfuric acid (hydrochloric acid and nitric acid are not used, as the former is oxidized by the permanganate ion and the latter is also an oxidizing agent).

## Operating mode

### Used materials:

### Used chemicals

-Burette (25-50 mL).	
-Graduated or volumetric pipettes (10 mL)	-Potassium permanganate solution $\text{KMnO}_4$
-Pro-pipette	
-Erlenmeyer flask (100 mL).	- $\text{H}_2\text{SO}_4$ sulfuric acid solution (20%).
-Hot plate	-Oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4$ , N1= 0.01N)
-Magnetic strippe	- $\text{FeSO}_4$ solution
-Beakers	

### 1. Determination of normality of $\text{KMnO}_4$ solution:

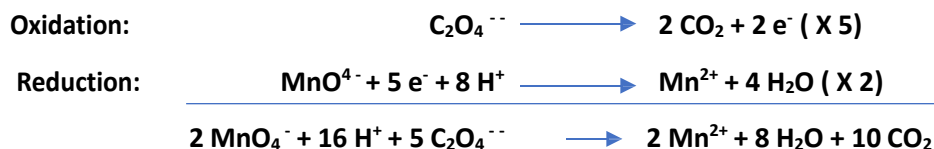
**Note:** follow the instructions given in PW N°03 (acid-basic titration) for preparing a dosing device.



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

This part is carried out using a reducing solution of **oxalic acid** ( $\text{H}_2\text{C}_2\text{O}_4, 2\text{H}_2\text{O}$ ), its concentration is **0.01 N**, as follow:

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



a) Fill burette with potassium permanganate solution ( $\text{KMnO}_4$ , oxidizing agent).

Place 10 ml of oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4$ ,  $N_1 = 0.01\text{N}$ ) and 10 ml of sulfuric acid  $\text{H}_2\text{SO}_4$  (20%) in the Erlenmeyer flask. Heat the mixture to around **60 °C** as the reaction is slow, and heat **speed up** (catalyse) the reaction, **the color disappears rapidly**.

b) Allow a few drops of  $\text{KMnO}_4$  to flow, shake, and wait until they **are decolorized**. Continue adding a few drops until an excess drop produces a persistent pale pink color (does not disappear).

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

Table 1:

	1 <sup>st</sup> test	2 <sup>nd</sup> test	3 <sup>rd</sup> test
$V_e$ (mL)			
Color			

- Deduce the average equivalence volume ( $V_{e,avg}$ )?
- Calculate the normality and deduce the concentration of the  $\text{KMnO}_4$ ?

## 2. Determination of normality of $\text{FeSO}_4$ :

Introduce into a 100 mL Erlenmeyer flask:

- 10 mL  $\text{FeSO}_4$  solution using a pipette.
- 10 mL 20%  $\text{H}_2\text{SO}_4$  using graduated cylinder.

Place the Erlenmeyer flask under the burette and start dosing **drop by drop** until a pale pink color is obtained due to the addition of a single excess drop of  $\text{KMnO}_4$ . Note the value of the volume  $V_e$  poured (Table 2). Repeat the operation three times.

	1 <sup>st</sup> test	2 <sup>nd</sup> test	3 <sup>rd</sup> test
$V_e$ (mL)			
Color			

- Deduce the average equivalence volume ( $V_{e,avr}$ )?
- Give the oxidation-reduction reaction involved?
- Calculate the normality of the titrate solution?