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EXPERIMENTS IN GENERAL AND INORGANIC CHEMISTRY

LABORATORY EXERCISE INSTRUCTIONS



MOJCA SLEMNIK



University of Maribor

Faculty of Chemistry and
Chemical Engineering

Experiments in General and Inorganic Chemistry

Laboratory Exercise Instructions

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PREFACE

WORK INSTRUCTIONS

- **Students must come to each laboratory session well-prepared.**
- Before the session, students should carefully read the instructions for the experimental part of the exercise and study the relevant theoretical background, which can be found in the recommended textbooks. This theoretical knowledge is essential for solving the calculation tasks and successfully performing the experiment.
- Keep in mind that practical exercises are effective only when students are well-prepared and complete the tasks independently.
- During the laboratory sessions, the assistant may assess the student's knowledge - either orally or in writing. If the student is found to be insufficiently prepared, the assistant has the right to prohibit him/her from performing the experimental part of the exercise.
- Each exercise involves solving computational problems related to the theoretical content of the experiment.
- Prior to each laboratory session, the assistant will review and solve selected calculation examples with the students. Therefore, students must regularly attend seminar sessions, where they are introduced to chemical calculations.
- Successful problem-solving in chemical calculations requires a solid understanding of theoretical concepts and regular individual practice.

LABORATORY SAFETY PROTOCOLS

- Before starting practical exercises in the laboratory, **the student must be familiar with laboratory safety**: each student is given written instructions and must sign a declaration confirming their familiarity with laboratory procedures. These instructions must be strictly followed when working independently in the laboratory. In the event of any accident or injury, the laboratory assistant must be informed immediately.
- **Students must wear a lab coat when working in the laboratory, protective glasses and gloves when working with corrosive substances (acids, bases)!**
- Students must keep the laboratory in good order and may only carry out prescribed experiments.
- After the exercise, each student must **tidy up their workspace**. This means they must wash the equipment used during the practical part of the exercise, **rinse it with distilled water**, put it in a locker, and wipe down their workbench.
- **They must take care of their own inventory**, common inventory and laboratory furniture.

BRINGING AND CONSUMING FOOD AND DRINK, SMOKING AND USING MOBILE PHONES ARE STRICTLY PROHIBITED IN THE LABORATORY.

- All reactions that produce irritant or toxic gases must be carried out in fume cupboards.
- Only the designated disposal sinks located at the ends of the workbenches may be used for pouring out waste products. The sinks integrated into the workbenches themselves must not be used for this purpose. When disposing of waste materials into the drains, it is essential to flush thoroughly with running water.
- All substances that could have harmful effects on the environment when discharged into the sewage system (e.g., heavy metals, toxic substances, etc.) must be collected in special containers located next to the disposal sinks. These substances are later neutralized or transported to facilities that specialize in the collection and disposal of such waste.
- Before beginning the exercise, the assistant must inform the students about the proper collection procedures for hazardous waste.

- **Students are required to bring the following items to each laboratory session: a lab coat, safety goggles, gloves, a pipetting bulb, their own calculator, and a cloth for cleaning the workbench.**
- For any absence from a laboratory session, students must provide the assistant with a **valid medical certificate**. In the case of a planned absence, students must arrange an alternative date with the assistant to make up for the missed lab exercise.

THE LABORATORY NOTEBOOK GUIDELINES

Each student is required to keep a laboratory notebook. This should be an A5-sized notebook (60 to 100 sheets), used exclusively for recording laboratory work. **All observations, calculations, measurements, and results must be written clearly and legibly.**

Students must consistently follow the structure outlined below for each experiment:

Experiment number, title, and date

Data sheet number

1. Objective of the experiment
2. Measurements
3. Calculations
4. Experimental procedure
5. Results

All calculation tasks should be presented in a clear and logical format, showing the full process that leading to the final result. At the beginning of each calculation, students must list the data used—typically from the assigned data sheet.

For the experimental part, the student must clearly define the objective of the exercise. All measurements, calculations, and descriptions of the procedure must be recorded in an organized and concise manner.

In the procedure section, the student should briefly describe how the experiment was carried out and note any unusual occurrences that might have influenced the result. The procedure must be written in the **first-person singular** and in the **past tense**.

When writing the experimental section, students must use correct chemical terminology. While describing the experimental procedure, students may refer to the exercise instructions, but these should serve only as a guide. The procedure must be written in the student's own words. The description should be detailed and exact enough that another student could repeat the entire experiment based solely on what is written.

The results of each task must be clearly marked and presented in an organized manner.

In the second part of the course, most laboratory exercises focus on qualitative experiments. For these tasks, students should begin with a clearly defined objective, followed by a brief description of the experimental procedure and their personal observations. **All chemical reaction equations that occurred during the experiment must also be included.**

At the end of the description, students must provide explanations of their observations, conclusions, and interpretations - depending on what the exercise requires.

Any errors identified and specified by the assistant during notebook review must be corrected by the student. **Corrections should be written as a continuation in the same notebook, or the student must clearly indicate the page where the correction can be found.**

Incorrectly performed exercises must be repeated. If the student cannot repeat the experiment on the same day, they must arrange a new date for the repetition with the assistant in advance.

At the end of each laboratory session, the student must present their notebook to the assistant. The assistant will first **review the practical results** and then collect the notebook for correction. The student is considered to have completed the exercise only when the assistant has reviewed and signed all entries, along with the corresponding dates.

After the laboratory course is completed, the corrected and signed notebooks remain the property of the student.

LABORATORY EQUIPMENT

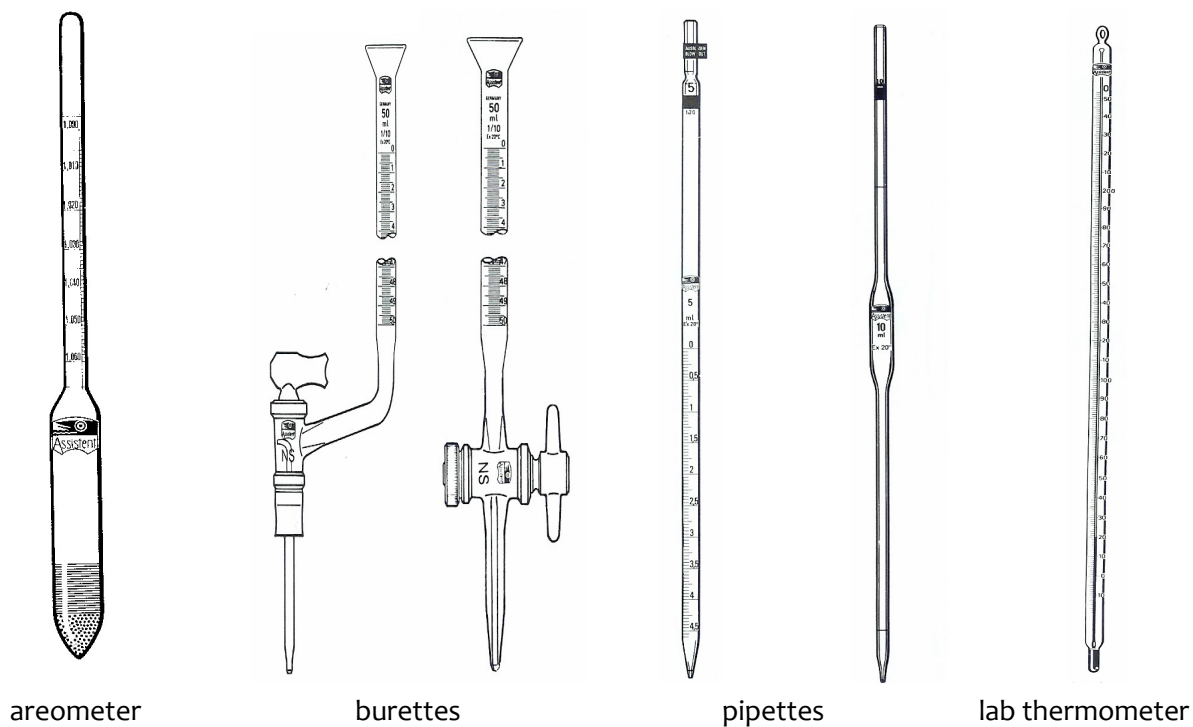


Figure 1. Laboratory equipment. [1]

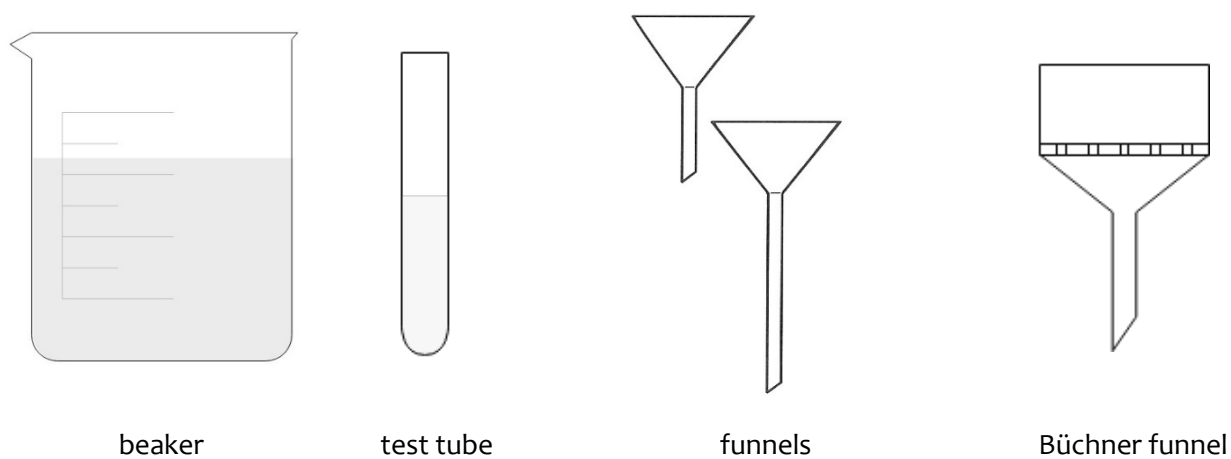
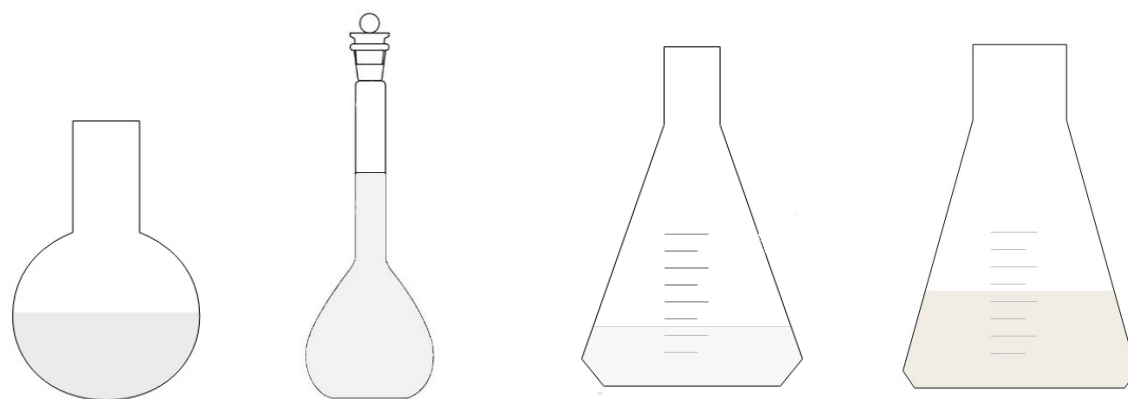


Figure 2. Laboratory equipment. [2]

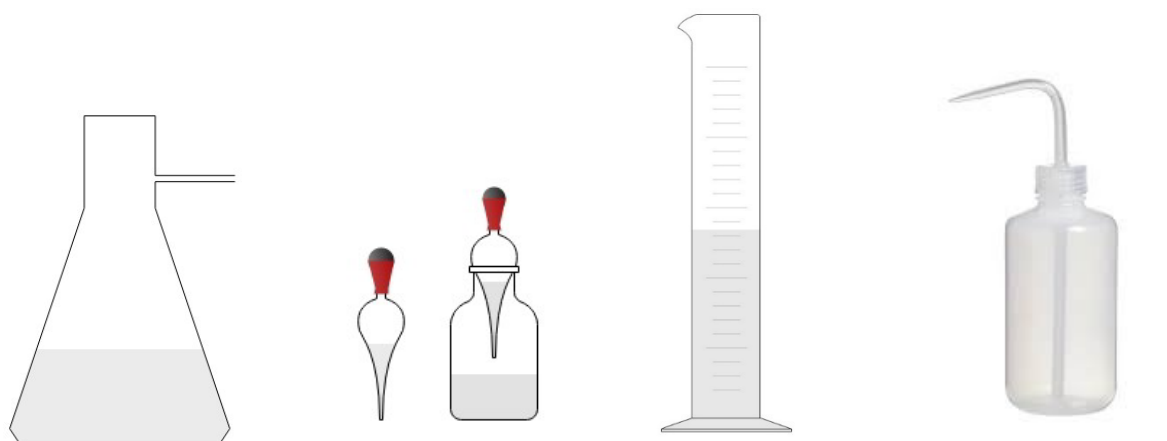


flask

volumetric flask

Erlenmeyer flasks

Figure 3. Laboratory equipment. [2]



suction flask

droppers

graduated cylinder

wash bottle

Figure 4. Laboratory equipment. [2]



weighing boat

evaporating dish

watch glass

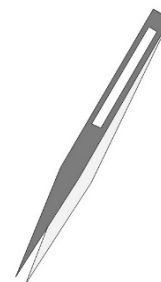
Figure 5. Laboratory equipment. [2]



magnets



crucible tongs

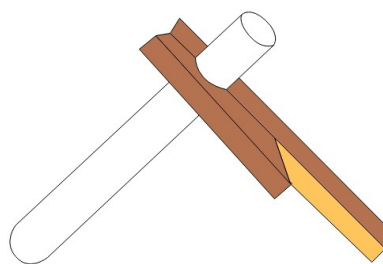


lab tweezer

Figure 6. Laboratory equipment. [2]



lab spoon with spatula

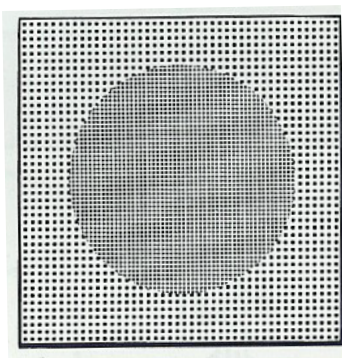


test tube clamp

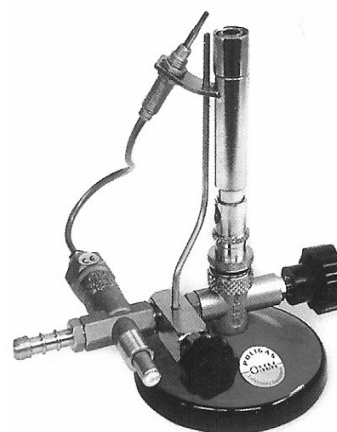
Figure 7. Laboratory equipment. [2]



rubber bulb



wire gauze

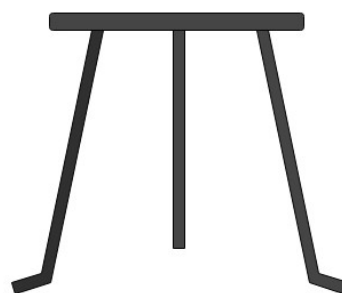


gas burner

Figure 8. Laboratory equipment. [1]



electronic balance



tripod

Figure 9. Laboratory equipment. [2]



test tube rack



pestle and mortar

Figure 10. Laboratory equipment. [2]



stand with clamps



double clamp

Figure 11. Laboratory equipment. [2]



Magnetic stirrer



protective glasses



rubber plug

Figure 12. Laboratory equipment. [2]

LABORATORY TECHNIQUES

WEIGHING

For weighing solid substances during laboratory exercises, we use analytical balances with **readings to two decimal places**. The following rules must be observed:

- **Chemicals must never be weighed directly on the balance pan.** Always use an appropriate container - such as an evaporating dish, beaker, watch glass, or weighing boat.
- **Never place hot objects on the balance!** Glassware must be cooled to room temperature before weighing. Weighing hot items gives inaccurate results and may damage the balance.
- **Avoid weighing in a draft.** Windows and doors near the balance should be closed during weighing.
- **Before weighing, check whether the balance is set to zero.** If it is not, reset it to zero using the appropriate button (TARE). If the balance cannot be zeroed, inform the assistant or technical staff!
- **First, weigh the empty container.** If its mass is needed for calculations, record it. Then, reset the balance to zero using the tare function and weigh the required reagent. Record the mass in your laboratory notebook!
- **If any chemical is spilled on the balance, clean it immediately** following the instructions of the assistant or technical staff.
- **After weighing, reset the balance to zero again.**

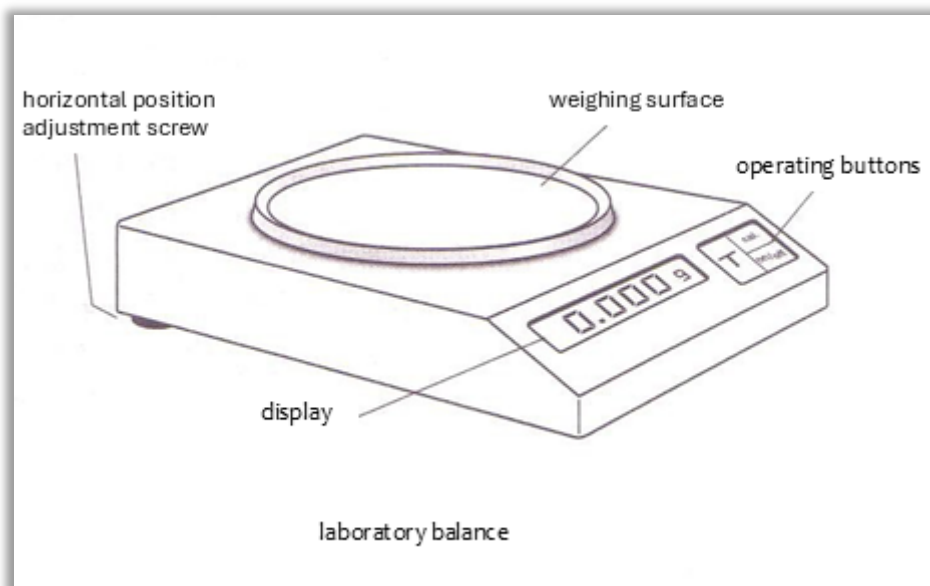


Figure 13. Laboratory balance. [1]

MEASUREMENT OF LIQUIDS, PIPETTES

For measuring liquid reagents and solvents, we use volumetric glassware that has calibration marks to determine volume. The most used are **graduated cylinders** of various volumes and **pipettes**, which are divided into graduated (measuring) and volumetric (filling) types.

Aqueous solutions in glass containers do not form a flat surface; instead, the surface curves into a concave shape called the **meniscus**. When reading the volume, be careful with the eye level—it must be aligned with the bottom edge of the meniscus!

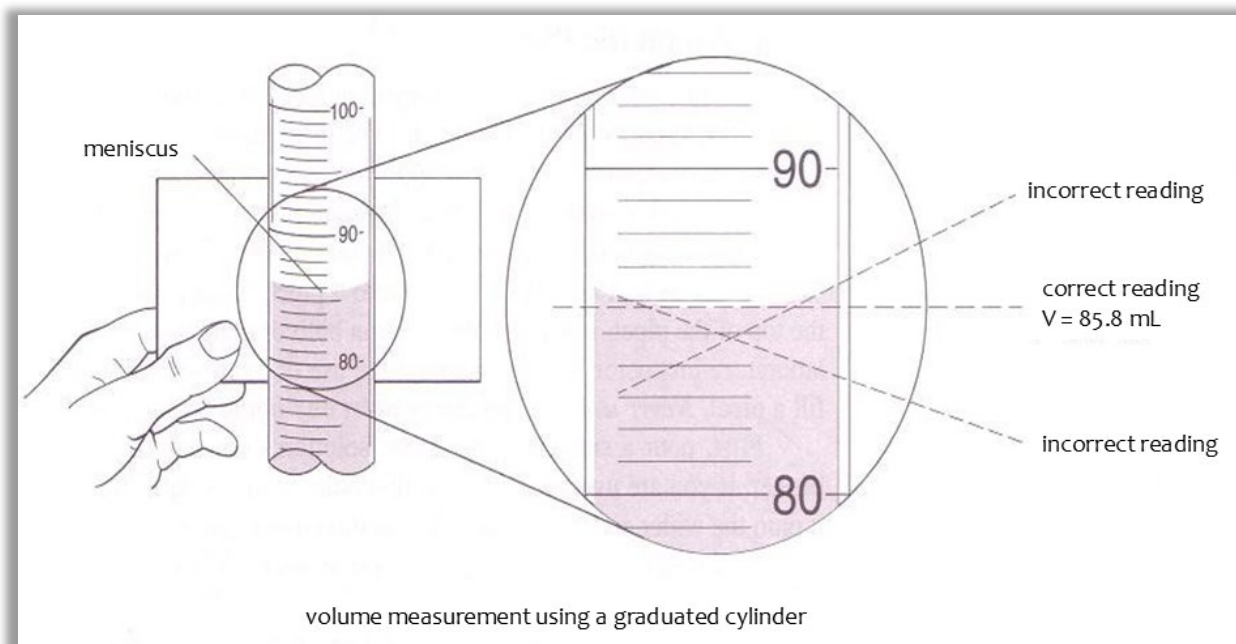


Figure 14. Volume measurement. [1]

When using pipettes, **under no circumstances should you pipette by mouth**, as this can be dangerous due to the risk of ingesting chemicals and inhaling vapours. Additionally, it is unhygienic. Instead, rubber bulbs (“pipette bulbs”) or plastic pipette aids are available.

When using pipette aids, follow the instructions of the lab assistant or technician. Be careful not to draw liquid into the pipette aid!

When emptying the pipette, **do not try to blow out the small volume of liquid** that remains in the tip - this volume has already been accounted for during calibration!

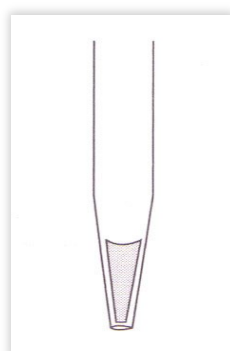


Figure 15. The residual liquid volume in the pipette tip is included in the pipette’s calibration. [1]

BURETTES

Burettes are long, narrow, calibrated glass tubes equipped with a stopcock (valve), which is used to control the flow rate. They are used for measuring liquids, most commonly in **quantitative analysis for titration**. The most frequently used burettes have a ground glass stopcock, but more recently, Teflon stopcocks have become increasingly common.

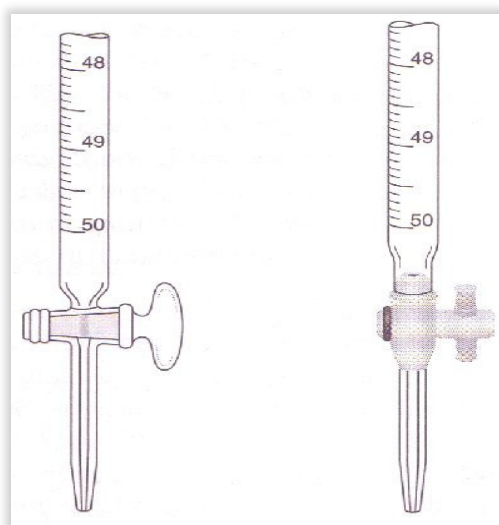


Figure 16. Burette with glass and Teflon stopcock. [1]

When working, ensure the **burette is securely clamped in a vertical position**, as this is necessary to obtain exact results. Check that the stopcock is closed, then fill the burette with the appropriate solution, using a glass funnel to assist. Always fill the burette slightly above the 0 mL mark, then **remove the funnel** and release the excess solution until the bottom edge of the meniscus aligns exactly with the 0 mL mark.

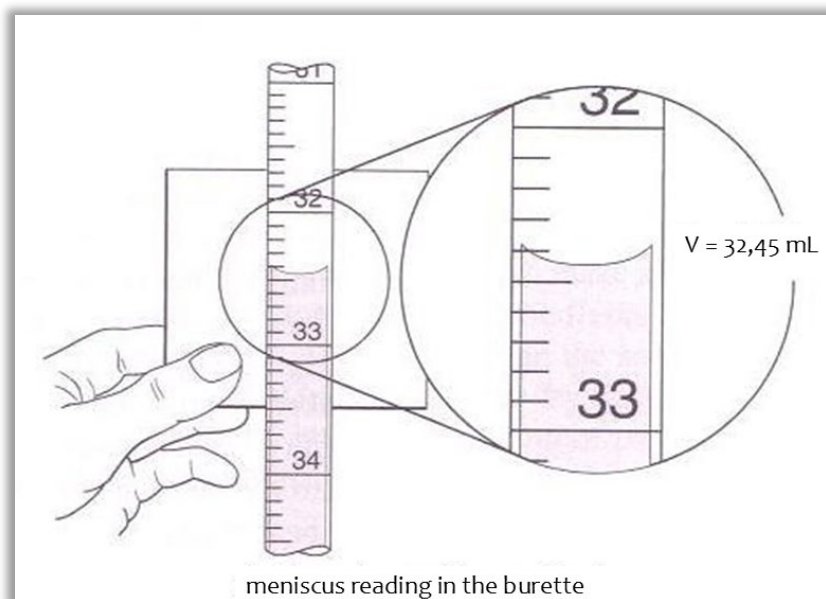


Figure 17. Correct meniscus reading. [1]

During titration, hold the burette with your left hand and the Erlenmeyer flask with your right hand. This prevents accidentally pulling the stopcock out of the clamp (this applies to right-handed users; see Figure 18.).

This is especially important with glass stopcocks and less so with Teflon ones. **Always observe the color of the solution against a white background.**

Stir the solution in the Erlenmeyer flask **continuously throughout the titration** using circular hand movements.

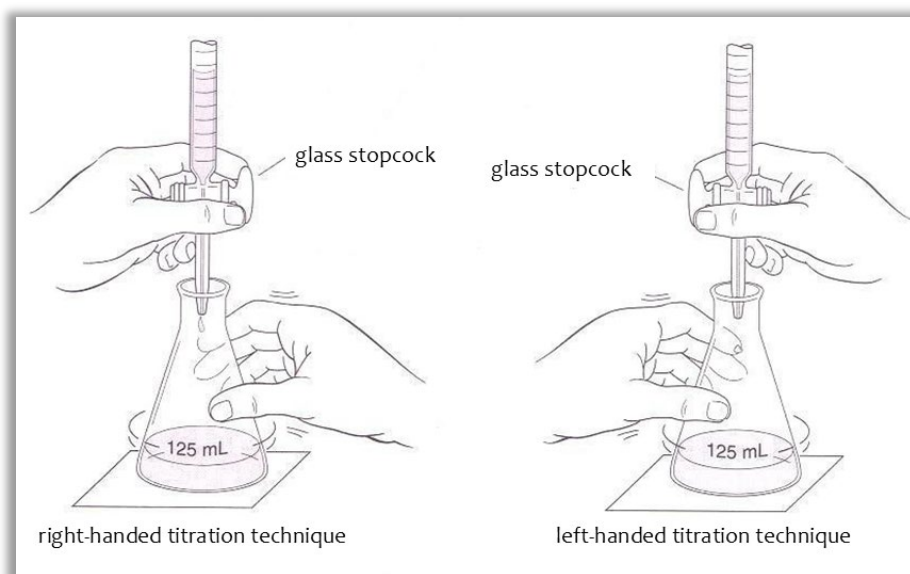


Figure 18. Titration techniques. [1]

VOLUMETRIC FLASKS

Volumetric flasks of various volumes are used to prepare exact defined volumes of solutions. For this purpose, each flask has a single mark on the neck indicating the volume to which it must be filled.

If preparing a solution from a solid solute, never weigh the solid directly in the flask. Instead, **dissolve the solute in a beaker with a volume of solvent less than that of the flask.** Transfer the solution quantitatively into the volumetric flask and rinse the beaker with solvent to ensure complete transfer.

If preparing a solution by diluting a concentrated stock solution, first pour some solvent into the flask (about one-third of its volume), then add the calculated volume of the concentrated solution (depending on required accuracy and precision, use a graduated cylinder or pipette).

In both cases, fill the flask up to the mark with solvent. Add the last drops slowly and carefully to avoid over-dilution. Use a dropper with a narrow tip to help. **When reading the volume, make sure to read at the bottom edge of the meniscus!**

MIXING LIQUIDS

When mixing a concentrated solution with water or two solutions of different concentrations, **always add the concentrated solution to the water** or the more concentrated solution to the less concentrated one! This rule is especially important when preparing acids from concentrated solutions and water. Add the concentrated solution gradually, in small portions.

PROPER USE OF DROPPERS

When adding reagents with droppers, take care not to contaminate the reagent in the dropper bottle. **Never place the dropper on the work surface and be careful not to touch the tip of the dropper to the test tube or any other container into which you are adding the reagent.**

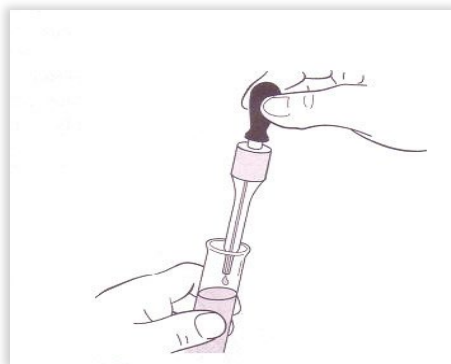


Figure 19. Use of dropper. [1]

FILTRATION

For proper filtration, you need a stand, a filtration ring, a glass funnel, a beaker, a glass stirring rod, and suitable filter paper. Fold or pleat the filter paper so that it fits snugly into the funnel. The funnel's stem should touch the wall of the beaker with its longer end. Pour the liquid from the beaker onto the filter paper, using the glass stirring rod to guide the flow.

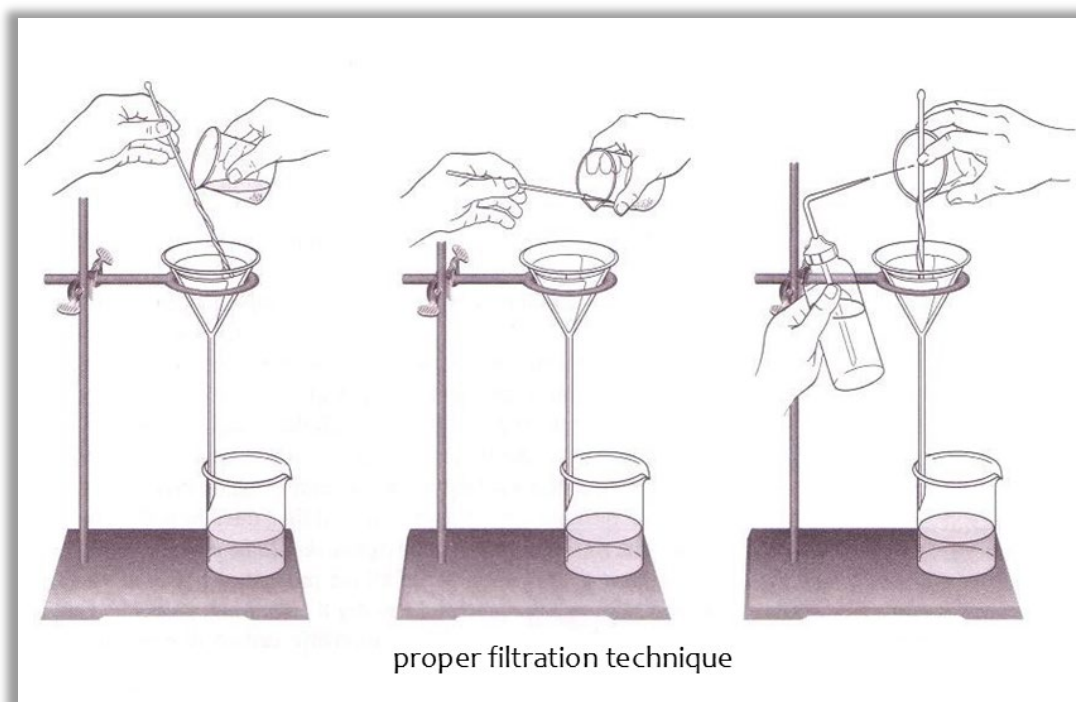


Figure 20. Filtration techniques. [1]

PROPER HEATING OF LIQUIDS IN A TEST TUBE

Non-flammable liquids, most often aqueous solutions, can be heated directly over a burner flame in a test tube, but certain safety precautions must be observed. Failure to do so can cause sudden and vigorous boiling of the liquid and/or breakage of the test tube due to rapid heating:

- Fill the test tube with liquid to a maximum of one-third of its volume.
- Hold the test tube with a test tube holder at an angle of approximately 45°.
- Do not place the test tube directly in the flame; position it just **above** the top edge of the flame.
- Gently and continuously swirl the test tube while heating.
- Keep the open end of the test tube pointed away from your face and others nearby.

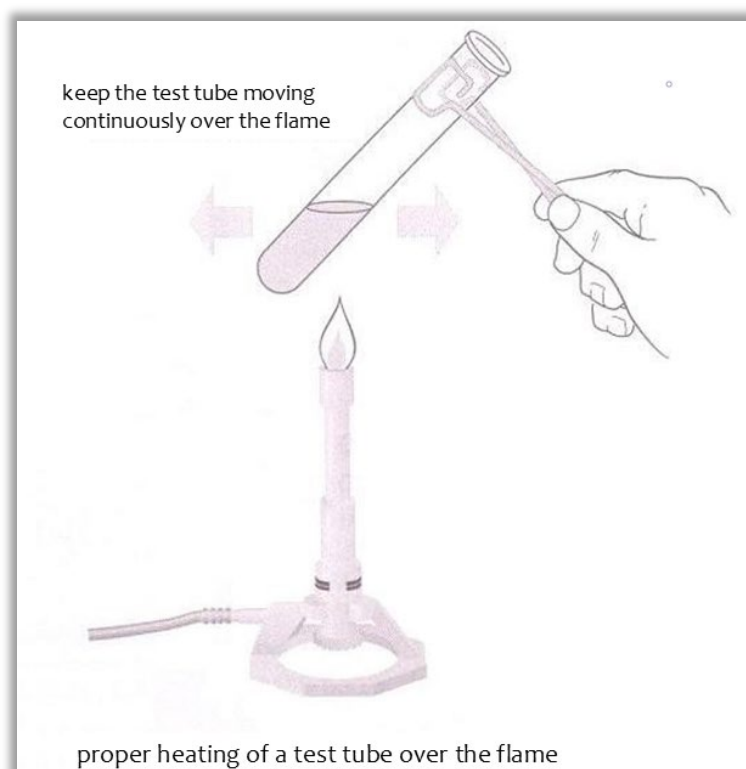


Figure 21. Proper heating of a test tube over the flame. [1]

SI BASE UNITS

Table 1. The International System of Units is based on 7 fundamental units.

QUANTITY		UNIT	
NAME	SYMBOL	NAME	SYMBOL
Length	<i>l</i>	meter	m
Mass	<i>m</i>	kilogram	kg
Time	<i>t</i>	second	s
Electric current	<i>I</i>	ampere	A
Temperature	<i>T</i>	kelvin	K
Luminous intensity	<i>I_v</i>	candela	cd
Amount	<i>n</i>	mole	mol

Table 2. Gas constant in different units.

VALUE of R	UNIT
8.314472	$\text{J} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$
0.0820574587	$\text{L} \cdot \text{atm} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$
$8.20574587 \cdot 10^{-5}$	$\text{m}^3 \cdot \text{atm} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$
8.314472	$\text{cm}^3 \cdot \text{MPa} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$
8.314472	$\text{L} \cdot \text{kPa} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$
8.314472	$\text{m}^3 \cdot \text{Pa} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$
62.3637	$\text{L} \cdot \text{mm Hg} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$
83.14472	$\text{L} \cdot \text{mbar} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$
1.987	$\text{cal} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$

Table 3. Decimal SI units derived from SI units and prefixes.

PREFIX		FACTOR	VALUE
NAME	SIGN		
exa	E	10^{18}	1 000 000 000 000 000 000
peta	P	10^{15}	1 000 000 000 000 000
tera	T	10^{12}	1 000 000 000 000
giga	G	10^9	1 000 000 000
mega	M	10^6	1 000 000
kilo	k	10^3	1 000
hecto	h	10^2	100
deca	da	10	10
deci	d	10^{-1}	0.1
centi	c	10^{-2}	0.01
milli	m	10^{-3}	0.001
micro	μ	10^{-6}	0.000 001
nano	n	10^{-9}	0.000 000 001
pico	p	10^{-12}	0.000 000 000 001
femto	f	10^{-15}	0.000 000 000 000 001
atto	a	10^{-18}	0.000 000 000 000 000 001

Derived SI units are derived from the base units with a factor of 1. For example:

$$1 \text{ J} = 1 \text{ N}\cdot\text{m}$$

$$1 \text{ Pa} = 1 \text{ N/m}^2$$

In addition to the base and derived SI units, some other units may be used in science and technology due to their practical significance.

Table 4. Some widely accepted non-SI units.

QUANTITY		UNITS		RELATION TO SI UNITS
NAME	SYMBOL	NAME	SYMBOL	
Volume	V	litre	L	1 L = 1 dm ³
Mass	m	tone	t	1 t = 10 ³ kg
Pressure	P	bar	bar	1 bar = 10 ⁵ Pa
Time	t	minute	min	
		hour	h	
		day	d	
Energy	W	electron volt	eV	1 eV = 1.602·10 ⁻¹⁹ J
Temperature	T	Celsius degree	°C	0°C = 273.15 K

In addition to the base and derived SI units and the widely accepted non-SI units, some obsolete units are still occasionally used. Generally, their use should be avoided; however, in certain fields of science and technology, they remain in everyday use. So, it is important to be familiar with them.

Table 5. Obsolete units.

QUANTITY		UNITS		RELATION TO SI UNITS
NAME	SYMBOL	NAME	SYMBOL	
Length	l	ångström	Å	1 Å = 10 ⁻¹⁰ m
		micron	my	1 my = 10 ⁻⁶ m
		inch	in	1 in = 25.4 mm
Pressure	p	atmosphere	atm	1 atm = 101325 Pa
		mm Hg	torr	1 torr = 133.3 Pa

Table 6. Derived quantities in chemistry.

QUANTITY	SYMBOL	DEFINITION	UNIT
amount of substance (substance X)	$N(X)$	$N(X) = n(X) \cdot N_A$	-
molar mass (substance X)	$M(X)$	$M(X) = \frac{m(X)}{n(X)}$	g/mol
density	ρ	$\rho = \frac{m}{V}$	g/mL
molar concentration (<i>molarity</i>)	$c(X)$	$c(X) = \frac{n(X)}{V}$	mol/L
mass concentration	$y(X)$	$Y(X) = \frac{m(X)}{V}$	g/L
molality of solute X in solvent Y	$b(X)$	$b(X) = \frac{n(X)}{m(Y)}$	mol/kg
mole fraction of substance X	$x(X)$	$x(X) = \frac{n(X)}{i} \sum n_i$	-
mass fraction of substance X	$w(X)$	$w(X) = \frac{m(X)}{\sum m_i}$	-
volume fraction of substance X	$\varphi(X)$	$\varphi(X) = \frac{V(X)}{i} \sum V_i$	-
solubility of substance X in solvent Y	t	$t = \frac{m(X)}{m(Y)} \cdot 100$	$\frac{g(X)}{100 g(Y)}$
degree of dissociation	α	$\alpha = \frac{N}{N_0}$	-

EXPERIMENTAL EXERCISES

EXPERIMENTAL EXERCISES



EXP. N°1: DETERMINATION OF A CRYSTAL HYDRATE FORMULA

REQUIRED KNOWLEDGE: Basic chemical laws. Atomic theory. Amount of substance. Mole. Avogadro's number. Relative atomic mass. Relative molecular mass. Chemical compound formulas. Chemical equations. Nomenclature of chemical compounds.

SAFETY: Follow the instructions when working with hot objects and devices!

LABORATORY TECHNIQUES

WEIGHING, page 10

KEY FORMULAS

amount of substance $n(x) = \frac{m(x)}{M(x)}$

no. of atoms $N = n \cdot N_A$; $N_A = 6.02 \cdot 10^{23} \text{ at. mol}^{-1}$

mass fraction $w(x) = \frac{m(x)}{\sum m_i}$

EXERCISES

1. CALCULATION EXERCISES (data will be provided on a separate sheet):

- Calculate how many atoms are in 1 mg of.....!
- Calculate the mass of 1 atom of.....!
- Determine the empirical formula of a compound from the given mass fractions!
- From the known chemical formula - calculate the mass fractions of the elements in the compound!

2. EXPERIMENTAL

Determine the empirical formula of the crystal hydrate provided by the lab assistant!

3. INVENTORY

Shared inventory

- Water bath
- Sand bath
- Laboratory spoon
- Balance (scale)

Personal inventory

- Porcelain evaporating dish
- Glass stirring rod

4. CHEMICALS

- Various crystal hydrates

5. PROCEDURE

- Weigh the empty evaporating dish and label it with your workstation number. If your sample requires a glass stirring rod (for mixing the salt), weigh the rod together with the dish. In this case the rod must remain in the dish for the entire experiment!
- Set the balance to 0.00 g (press the TARE button) and weigh the mass of the crystal hydrate.
- Carefully record both measurements!
- Place the evaporating dish with the crystal hydrate on the water bath. When opening the rings of the water bath, be careful not to splash samples that are already on the bath!
- Once all the water has evaporated, transfer the evaporating dish to the sand bath. Be sure to first place the dish with the salt on the surface of the sand. Only after a while should you bury it slightly deeper in the sand as the temperature inside the sand bath is higher than on the surface. Higher temperatures are needed to remove the crystal-bound water.
- When you believe that all the water has evaporated, cool the evaporating dish with the anhydrous salt and weigh it. Record the mass of the dish and salt in your lab notebook. If you included the glass rod in your initial weighing, it must remain in the dish for all subsequent weighings.

- **Ensure that the salt is dry** (i.e. drying to constant mass). To do this, return the evaporating dish with the dried salt to the sand bath for a while, then cool and weigh it again. **If there is no significant difference between the first and second weighings, you can conclude that all the water has evaporated from the crystal hydrate.**
- The mass of the crystal-bound water is the difference between the mass of the salt before and after evaporation. Calculate the mass of water into the amount of crystal water and express it in moles of water per 1 mole of the crystal hydrate! Dispose of the used salt in the designated container provided by the lab assistant.

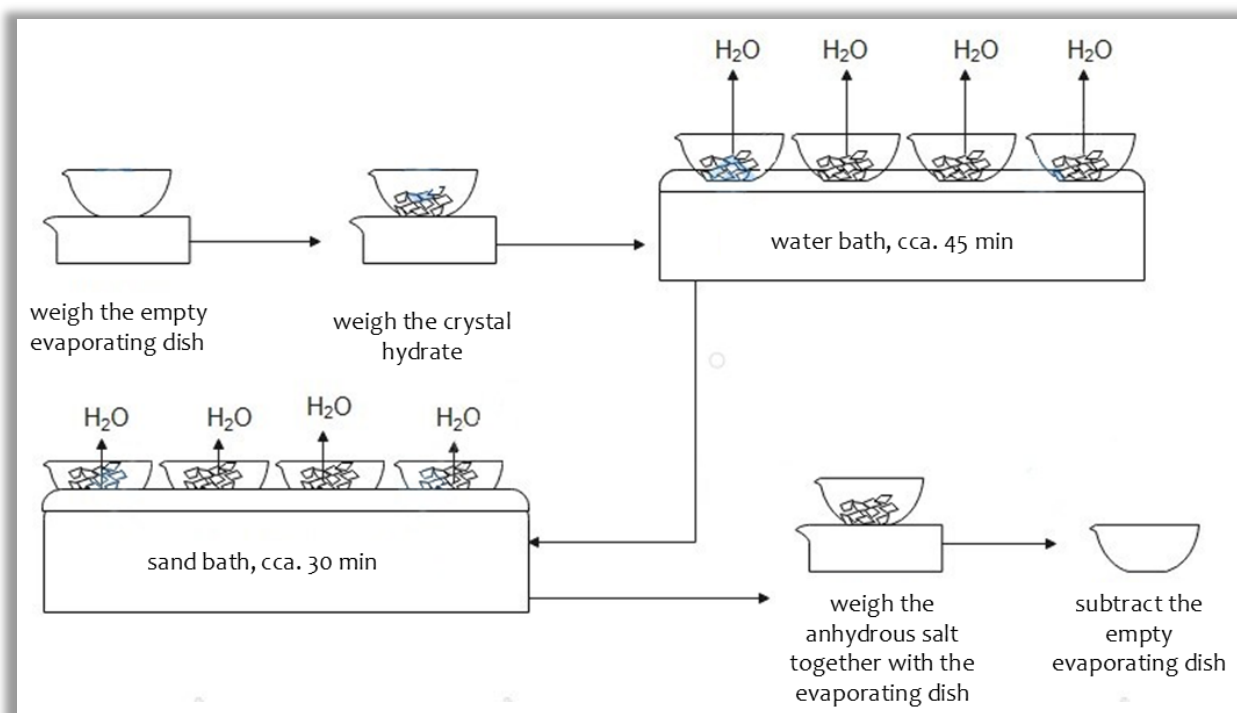


Figure 22. Procedure overview. Source: Author's own work.



LET'S CONSIDER

1. Why is it necessary to weigh the glass stirring rod together with the evaporating dish before weighing the crystal hydrate?
2. Why do we first dry the crystal hydrate on a water bath and only then on a sand bath? Why do we let the water evaporate completely on the water bath first?
3. Why do we initially place the evaporating dish on top of the sand in the sand bath, and only later embed it deeper into the sand?
4. What does "weighing to constant mass" mean?

EXP. N°2: THE GAS LAW

REQUIRED KNOWLEDGE: The Gas law. Amount of substance. Molar mass. Average molar mass. Gay-Lussac's law of combining gas volumes. Avogadro's law. Gas density. Partial pressure. Mole fraction.

SAFETY: Follow safety precautions when working with acids and during the generation of hydrogen gas! **SAFETY GOGGLES AND GLOVES ARE MANDATORY!**

LABORATORY TECHNIQUES

MEASUREMENTS OF LIQUIDS, PIPETTES,
page 11

KEY FORMULAS

$$P \cdot V = n \cdot R \cdot T$$

$$P \cdot V = \frac{m}{M} \cdot R \cdot T$$

$$P \cdot M = \rho \cdot R \cdot T$$

$$\bar{M} = \sum x_i \cdot M_i$$

EXERCISES

1. **CALCULATION EXERCISES** (data will be provided on a separate sheet):

- a) Calculate the molar mass of a gas. Where a mass of grams occupy a volume of litres at a temperature of °C and a pressure of kPa!
- b) A gas mixture of (gas A) and (gas B) has a density of g/L at a temperature of °C and a pressure of kPa.

Calculate the mass and volume fractions of both gases in the mixture!

Also calculate the partial pressures of the individual gas components in the mixture!

c) A mass of g of a hydrocarbon burns in air to form carbon dioxide and water vapor.

Calculate the **volumes** of both gaseous products formed during the combustion of the hydrocarbon at a temperature of °C and a pressure of kPa!

2. EXPERIMENTAL

Calculate the mass of magnesium by measuring the volume of hydrogen gas produced in the chemical reaction between magnesium and hydrochloric acid!

3. INVENTORY

Shared inventory

- Stand (support stand)
- Curved glass tube with a rubber stopper
- Copper wire
- 10 mL graduated cylinder
- Barometer (*if there is no barometer in the room, ask the assistant for the current atmospheric pressure*)
- Thermometer for measuring air temperature
- Thermometer for measuring water temperature

Personal inventory

- Double clamp holder
- Universal clamp
- Ring clamp with holder
- Test tube
- 1 L beaker
- 100 mL graduated cylinder

4. CHEMICALS

- Magnesium ribbon
- Hydrochloric acid (HCl). Concentration $c = 2 \text{ mol/l}$.

5. PROCEDURE

- Assemble the apparatus as shown in the Figure 23. Make sure the stopper fits tightly in the test tube!
- Fill the graduated cylinder completely with water and the beaker nearly to the top. To do this, fill the sink at the end of the lab bench with water. Turn the graduated cylinder (with the ring) upside down into the beaker, and then simultaneously submerge both into the sink. Check for any air bubbles that may remain in the graduated cylinder. Secure the cylinder to the stand. Be careful not to let any air enter the cylinder—if it does, repeat the filling process.
- Carefully pour 6 mL of hydrochloric acid (HCl) with a concentration of $c = 2 \text{ mol/L}$ down one side of the test tube. Make sure to wet only one side of the inner wall of the test tube!
- Hang the piece of magnesium (provided by the assistant) on the copper wire and gently place it against the *dry* wall of the test tube (the side *not* wetted with acid should face downward). Be careful not to let the magnesium touch the acid yet. Secure the test tube to the stand using a clamp and close it with the rubber stopper connected to the curved glass tube.
- Check that the end of the curved glass tube is placed beneath the lower edge of the graduated cylinder. Otherwise, some of the hydrogen gas produced during the reaction may escape.
- Once everything is correctly set up, pull the copper wire (with the magnesium) down between the test tube wall and the stopper, so that the magnesium falls into the acid.
- The reaction begins, producing hydrogen gas, the reaction is complete when gas is no longer evolving (i.e. no more bubbles are forming in the graduated cylinder).
- After the reaction is complete, read the volume of hydrogen gas produced. Try to equalize the water levels inside the graduated cylinder and in the beaker as much as possible.
- Record the atmospheric pressure using the barometer, the air temperature from the room thermometer, and the water temperature in the beaker.
- Subtract the partial pressure of water vapor (see [Table 7](#) – vapor pressure depends on the water temperature in the beaker) from the atmospheric pressure. Use the resulting data to calculate the **mass of the metal!**

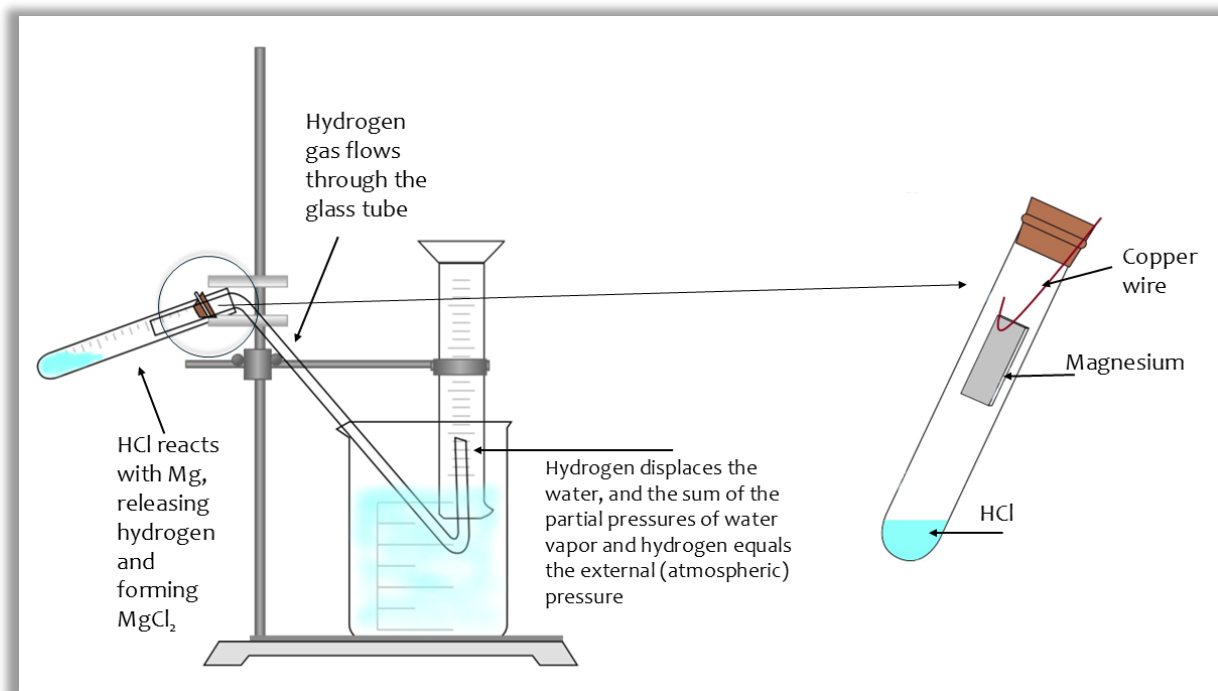


Figure 23. Apparatus Setup for the Experiment. Source: Author's own work.

Table 7. Dependence of Water Vapor Pressure on Water Temperature, (10 – 25°C).

$T(^{\circ}\text{C})$	p (kPa)	$T(^{\circ}\text{C})$	p (kPa)	$T(^{\circ}\text{C})$	p (kPa)	$T(^{\circ}\text{C})$	p (kPa)
10	1.22	14	1.60	18	2.07	22	2.64
11	1.32	15	1.71	19	2.20	23	2.81
12	1.40	16	1.81	20	2.33	24	2.98
13	1.49	17	1.93	21	2.48	25	3.17



LET'S CONSIDER

1. Why do we place the magnesium on the dry wall of the test tube?
2. Why do we equalize the water levels in the graduated cylinder and the beaker before reading the volume of the gas produced?
3. Why do we subtract the partial pressure of water vapor from the external (atmospheric) pressure?

EXP. N°3: SOLUTIONS

REQUIRED KNOWLEDGE: Solution concentration calculations. Solution density. Preparation of solutions.

SAFETY: When working with gas lines, ensure all valves are properly closed after use. Follow all safety protocols when handling concentrated acids and bases. When measuring the volume of concentrated acids or bases using a graduated cylinder, **IT IS MANDATORY TO WEAR SAFETY GOGGLES AND PROTECTIVE GLOVES.**

LABORATORY TECHNIQUES

WEIGHING, page 10
MEASURING OF LIQUIDS, PIPETTES, page 11
MIXING OF LIQUIDS, page 15
VOLUMETRIC FLASKS, page 15

KEY FORMULAS

mass fraction of substance: $w(x) = \frac{m(x)}{\sum m_i}$

molarity: $c(x) = \frac{n(x)}{V_{\text{solution}}}$

mass concentration: $\gamma(x) = \frac{m(x)}{V_{\text{solution}}}$

molality: $b(x) = \frac{n(x)}{m_{\text{solvent}}}$

EXERCISES

1. **CALCULATION EXERCISES** (data will be provided on a separate sheet)
 - a) **Preparation of a solution with a given mass fraction from a salt**
 Prepare grams of a% sodium chloride solution (based on the data provided on the sheet distributed by the assistant). Measure the density and temperature of the prepared solution. Then calculate the following concentrations: molar concentration, molal concentration, mass concentration.
 - b) **Preparation of a solution with a given mass fraction from a hydrate (crystalline compound)**
 Prepare grams of a% solution using (hydrate). Measure the temperature and density of the prepared solution. Then calculate its molar concentration.

c) Preparation of a solution with a given molar concentration

Prepare 250 mL of a M solution using a% stock solution with a density of g/mL. Calculate the mass percentage of the prepared solution. Measure its temperature and density.

2. EKSPERIMENTAL

Based on your calculations, prepare the required solutions. Verify your results with the teaching assistant before proceeding.

3. INVENTORY**Shared inventory**

- Balance
- Thermometer
- Areometer (hydrometer)
- Tripod stand
- Bunsen burner
- 10 mL graduated cylinder
- 5 mL graduated cylinder

Personal inventory

- Bakers
- Graduated cylinder
- Glass stirring rod
- Ceramic wire gauze
- 100 mL graduated cylinder
- 250 mL volumetric flask

4. CHEMICALS

- Salts provided by the teaching assistant.

5. PROCEDURE

- a) Calculate the mass of salt required to prepare the solution (*data provided on the information sheet*).
 - Weigh the calculated amount of salt into a clean beaker. Add the calculated volume of distilled water using a graduated cylinder. Stir the mixture thoroughly until all the salt has completely dissolved.

Table 8. Density of solution with different mass fractions in g/mL at 20 °C

H ₂ SO ₄		NaOH	
mass fraction	density	mass fraction	density
9.1 %	1.060	7.5 %	1.081
20 %	1.139	20 %	1.219
96 %	1.836	30 %	1.326

- Before measuring the density of the prepared solution, first rinse the graduated cylinder with a small portion of the solution. Then fill the cylinder with the prepared solution.
- Carefully immerse the hydrometer into the solution. During the density measurement, the **hydrometer must float freely** (you may use an empty test tube for support if necessary).
- Measure the **temperature** of the prepared solution.

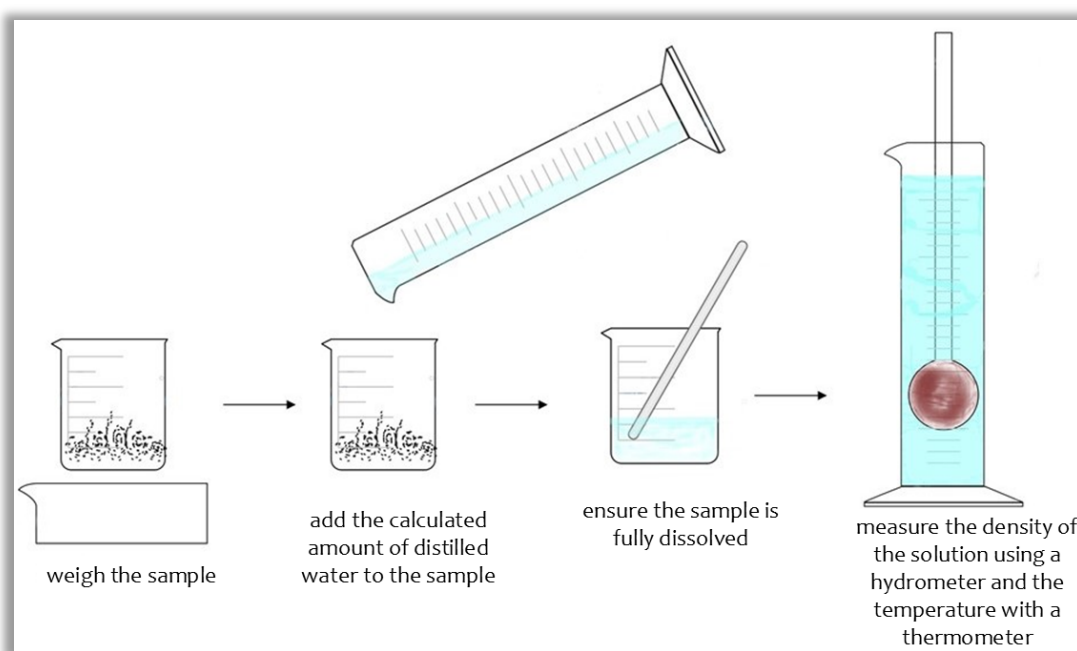


Figure 24. Procedure overview a). Source: Author's own work.

- b) **Prepare the solution** using the same procedure as in exercise a), but **in the calculation step, consider the mass of water of crystallization in the hydrate**. Subtract this mass of water from the calculated volume of distilled water.
- While dissolving the hydrate, stir the solution continuously. If the hydrate does not dissolve within a few minutes, place the beaker with the solution on a tripod stand with ceramic wire gauze and gently heat the solution.
 - Before measuring the density, cool the solution to room temperature by placing the beaker in a larger container filled with cold water.

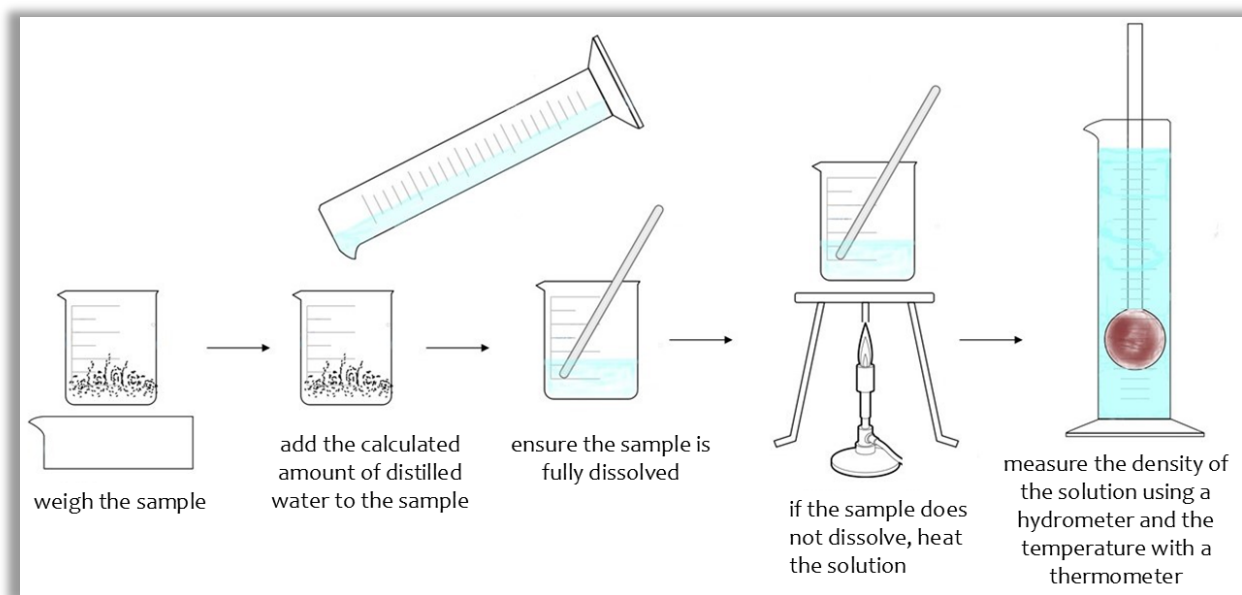


Figure 25. Procedure overview b). Source: Author's own work.

c) **Preparation of a solution with a specified molar concentration from a solution with a known mass fraction**

- Pour a small amount of distilled water (approximately one-third of the total volume) into a 250 mL volumetric flask.
- Measure the calculated volume of the solution with the known mass fraction and transfer it into the volumetric flask.
- **Be aware that the solution may heat up** during the transfer. If this occurs, cool the flask in cold water until the solution reaches room temperature.
- Then, carefully add distilled water **up to the calibration mark** on the neck of the volumetric flask (**ensure the bottom of the meniscus is aligned with the mark!**). Thoroughly mix the contents of the flask.
- Finally, measure and record the **temperature** and **density** of the prepared solution!

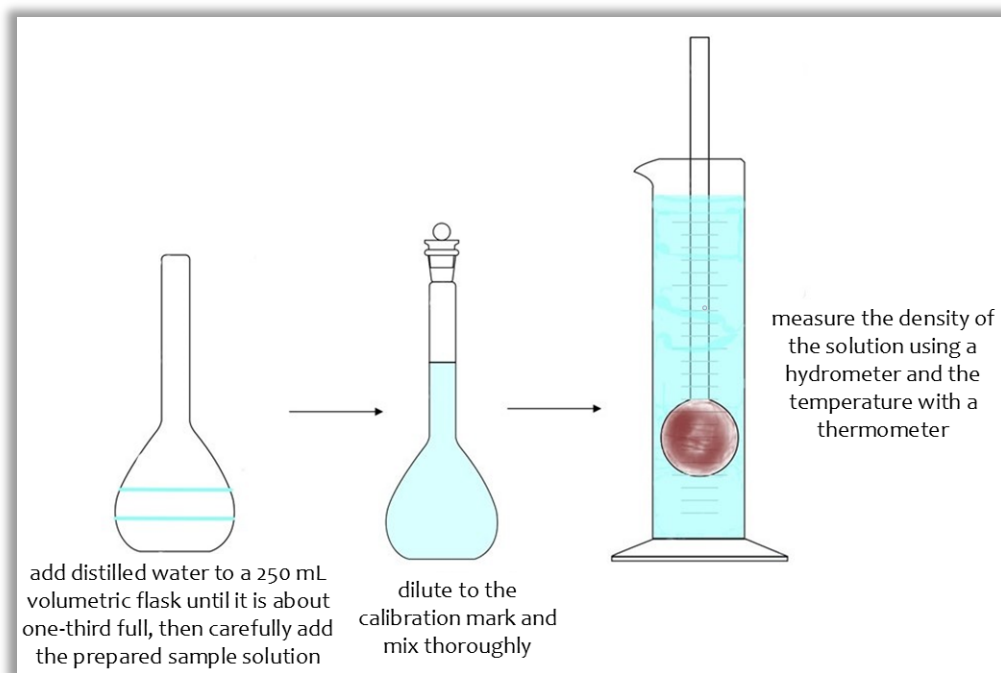


Figure 26. Procedure overview c). Source: Author's own work.



LET'S CONSIDER

1. How are mass concentration and molar concentration defined?
2. What is molal concentration?
3. How is the density of solutions defined?
4. What information is required to convert mass concentration to molar concentration?
5. Derive the formula to convert the mass fraction of a solute to molar concentration.
6. Which types of solution concentrations depend on temperature?

EXP. N°4: SOLUBILITY

REQUIRED KNOWLEDGE: Saturated solution. Concentration of a saturated solution. Dependence of solubility on the solvent. Dependence of solubility on external conditions (p , T). Solubility diagrams. Crystallization.

SAFETY: Exercise caution when handling hot objects and apparatus! Residues of metal salts should be disposed of in the designated containers.

LABORATORY TECHNIQUES

WEIGHING, page 101

MEASURING OF LIQUIDS, PIPETTES, page 11

FILTRATION, page 16

KEY FORMULAS

$$t = \frac{m(x)}{m(y)} \cdot 100 \text{ (g solute / 100g solvent)}$$

EXERCISES

1. CALCULATION EXERCISES (data will be provided on a separate sheet):

- Calculate how many grams of KNO_3 crystallize out if.....g of saturated KNO_3 at 100°C is cooled to 20°C ! The solubility of KNO_3 at 100°C is $246 \text{ g KNO}_3/100 \text{ g H}_2\text{O}$, and at 20°C it is $32 \text{ g KNO}_3/100 \text{ g H}_2\text{O}$.
- How many millilitres of water must be added to..... g of a solid mixture of Na_2CO_3 and $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ (the molar ratio between them is 1:2), to obtain a saturated solution at 60°C ? Solubility of Na_2CO_3 at 60°C is $46.4 \text{ g Na}_2\text{CO}_3/100 \text{ g H}_2\text{O}$.

2. EKSPERIMENTAL

Determine the solubility of the salt you obtained from the assistant!

3. INVENTORY

Shared inventory

- Stand
- Filter paper
- Water bath
- Sand bath
- Thermometer
- 10 mL graduated pipette
- Pipette bulb

Personal inventory

- 100 (or 150) mL and 250 mL beaker
- Glass rod
- Porcelain evaporating dish
- Joint with a ring clamp

4. CHEMICALS

- salt obtained from the assistant.

5. PROCEDURE

- In a 100 mL or 150 mL beaker, which you submitted, obtain salt from the assistant. Add 50 mL of distilled water to the obtained salt. Wait for 20 minutes and stir the solution several times.
- **The prepared solution is saturated**, meaning some insoluble salt remains at the bottom of the beaker in the solution.
- Filter the solution through crumpled filter paper into another beaker. Discard the first few millilitres of the filtrate.
- Measure the temperature of the filtrate and **pipette 10 mL of the saturated salt solution** into the **previously weighed evaporating dish**.
- **Weigh the evaporating dish** with the saturated solution and place it in a water bath.
- Evaporate the solution to dryness on the water bath, then dry the salt further **to constant mass** on a sand bath.
- Weigh the dried evaporating dish and calculate the mass of salt in 10 mL of the saturated solution.
- Calculate **the solubility of the obtained salt and the mass concentration of the saturated solution** at the measured temperature!

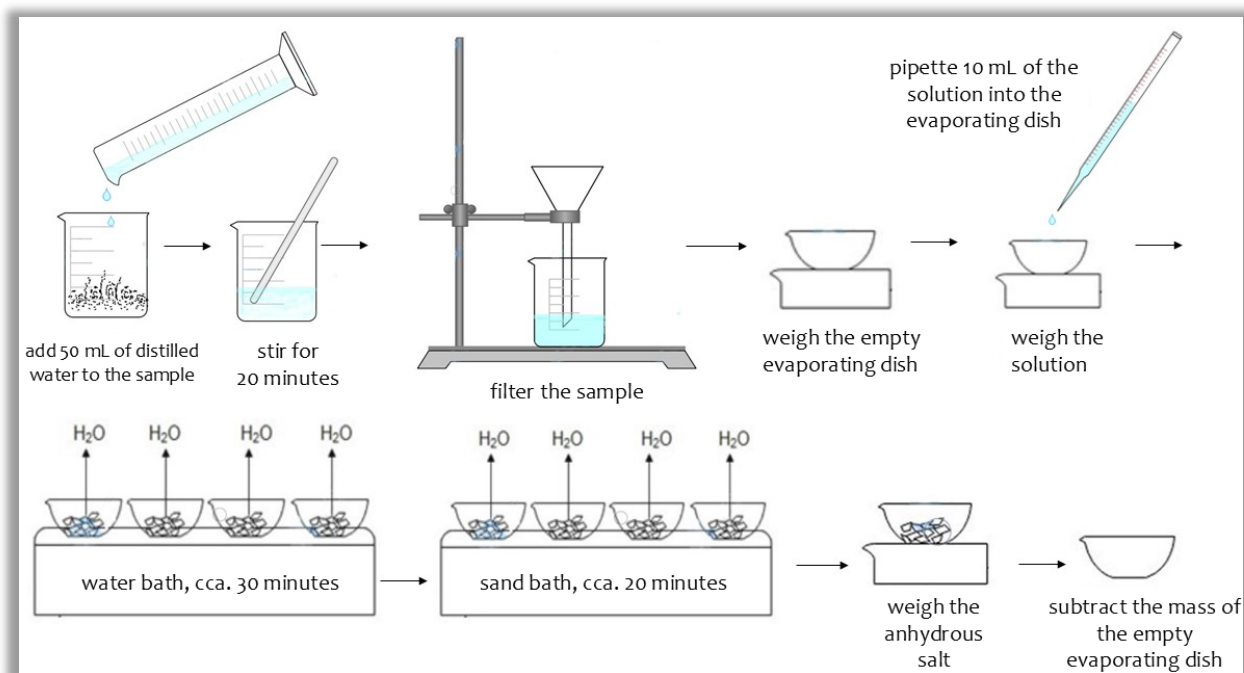


Figure 27. Procedure overview. Source: Author's own work.



LET'S CONSIDER

1. Why is it necessary to discard the first few mL of filtrate?
2. What is a saturated solution, and how do you prepare one?

EXP. N^o5: ACIDS, BASES AND SALTS, NEUTRALIZATION TITRATION

REQUIRED KNOWLEDGE: Acids, bases, and salts. Strong and weak electrolytes. Neutralization reactions. Ionic reactions. Indicators.

SAFETY: Follow the instructions for working with acids and bases. Ensure you **ALWAYS WEAR PROTECTIVE GOGGLES AND GLOVES!**

LABORATORY TECHNIQUES

PROPER USE OF DROPPERS, page 15
MEASUREMENT OF LIQUIDS, PIPETTES, page 11
VOLUMETRIC FLASKS, page 15
BURETTES, page 13

KEY FORMULAS

$$n = C \cdot V$$

$$\text{neutralization: } n_1 = n_2;$$

$$C_1 \cdot V_1 = C_2 \cdot V_2$$

EXERCISES

1. CALCULATION EXERCISES (data will be provided on a separate sheet):

- a) Calculate the molar concentration of the solution if 10 g of Na_2CO_3 , 10 g of $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$, 10 g of 5% Na_2CO_3 and a 10 mL of Na_2CO_3 solution of with a molarity of 1 mol/L is dissolved in mL volumetric flask, forming a solution, then diluted with distilled water to the mark!
- a) mL of HCl with molar mass of 1 mol/L neutralize 1 g of solid mixture of KOH and $\text{Ba}(\text{OH})_2$. Calculate the mass percentage of KOH in the original mixture!

2. EKSPERIMENTAL

a) Determine the color of the following indicators in acidic and basic media:

- Phenolphthalein
- Litmus
- Methyl red
- Methyl orange

b) Determine the difference in strength between hydrochloric acid and acetic acid!

- c) **Determine the difference in strength between sodium hydroxide and ammonia!**
- d) **Using titration, determine the concentration of the unknown sample of acid or base in the flask!**

3. INVENTORY

Shared inventory

- Burette
- Filter paper or paper towel
- Pipette bulb
- Test tube stoppers.

Personal inventory

- Test tubes,
- Test tubes stoppers
- Beakers
- 250 mL volumetric flask
- 25 mL graduated pipette or graduated cylinder
- 300 mL Erlenmeyer flask
- 10 mL or 5 mL graduated cylinders

4. CHEMICALS

- 0.1 M NaOH
- 2 M HCl
- 2 M CH₃COOH
- 2 M NaOH
- Copper plating solution
- Ethylacetate
- 0.1 M HCl
- 0.1 M NaOH
- Litmus paper (test strips)
- Phenolphthalein
- Litmus tincture
- Methyl red
- Methyl orange
- Zinc granules

5. PROCEDURE

a) Colour of indicators

- Place 8 test tubes in a test-tube rack
- To four of the tubes add 3.0 mL of 2.0 M HCl each, then add 2 drops of each indicator (phenolphthalein, litmus tincture, methyl red and methyl orange) to each tube.
- For each indicator, record the observed colour in the acidic medium.
- To the remaining four test tubes add 3.0 mL of 0.10 M NaOH each and repeat the procedure with the same indicators; record the observed colour of each indicator in the alkaline medium.

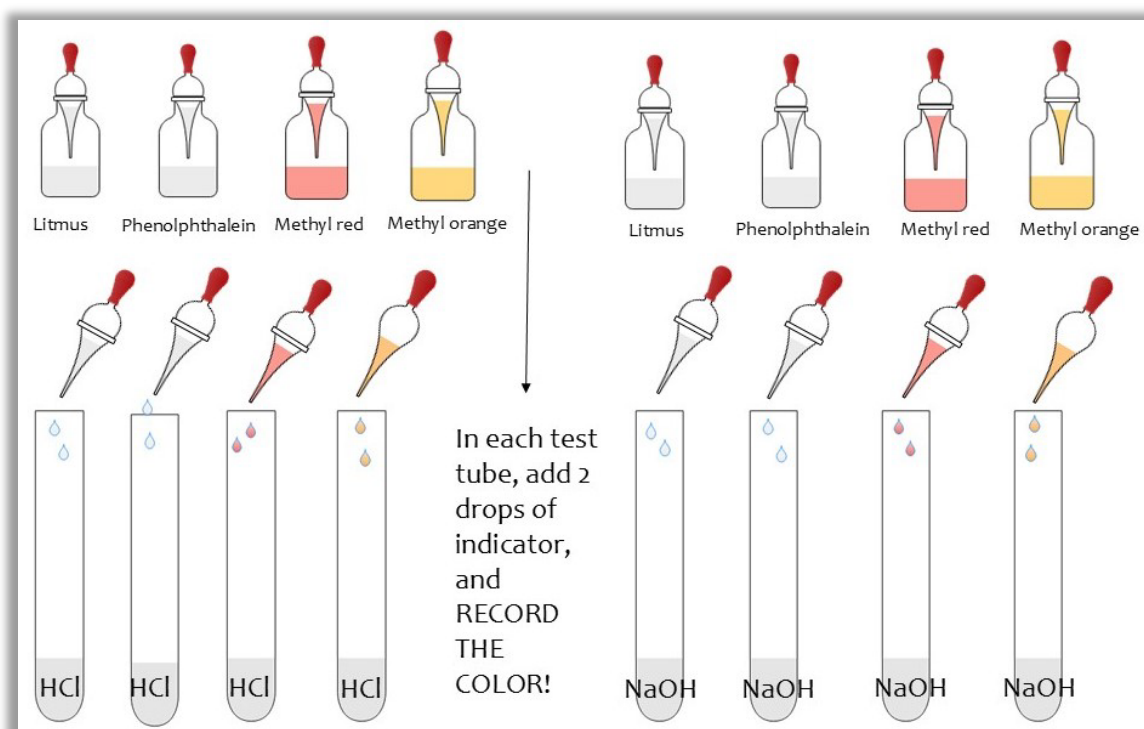


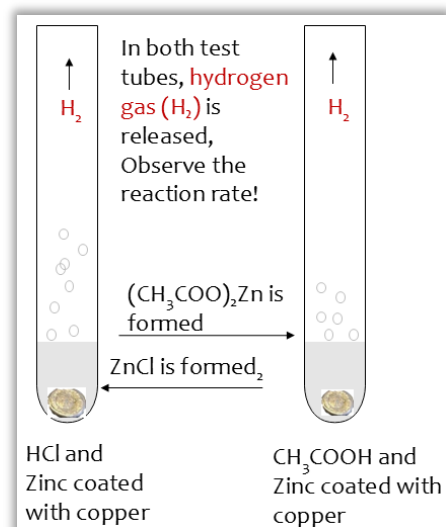
Figure 28. Procedure overview a). Source: Author's own work.

Table 9. Colours of indicators in acidic and basic media (fill in the table!)

Media	Litmus tincture	Phenolphthalein	Metyl red	Metyl orange
HCl (aq)				
NaOH (aq)				

b) Acid strength

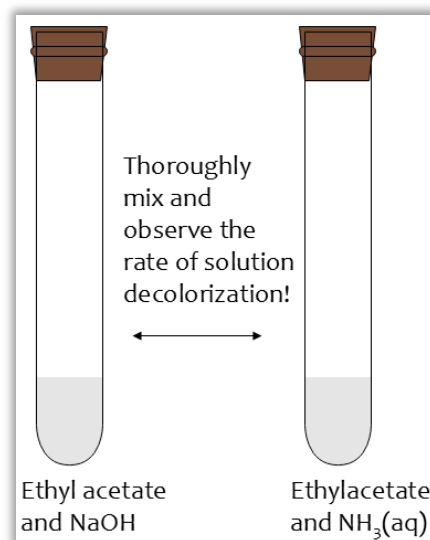
- In two test tubes, pour 5 mL of 2 M HCl and 2 M CH_3COOH , respectively. Add a piece of copper coated zinc to each test tube and observe the reaction process! Explain the reactions!
- Coat the zinc granules by pouring a copper sulphate (CuSO_4) solution over the zinc granules in a beaker and allowing the reaction to proceed for 10 seconds. Then, pour off the copper-plating solution and rinse the plated zinc with distilled water. Let it dry on a piece of filter paper or paper towel.

**Figure 29. Procedure overview b).**

Source: Author's own work.

c) Base strength

- In two test tubes, pour 1 mL of ethyl acetate each.
- To the first test tube, add 10 mL of 2 M NaOH; to the second test tube, add 10 mL of 2 M $\text{NH}_3(\text{aq})$
- Seal both test tubes with rubber stoppers and shake vigorously. Observe the rate of solution decolorization.
- Based on the rate of decolorization and the disappearance of the acetate phase, determine the difference in strength between NaOH and $\text{NH}_3(\text{aq})$.

**Figure 30. Procedure overview c)**

Source: Author's own work.

d) Concentration of the unknown sample

- Hand over a 250 mL volumetric flask to the assistant.
- Fill the prepared flask with distilled water up to the mark on the neck of the flask. Be very exact (the bottom of the meniscus must be exactly at the mark)!
- Thoroughly mix the solution in the flask!

- Use a litmus paper strip to determine whether the sample in the flask is acidic or basic.
- Using a graduated pipette, pour three times 25 mL of the solution from the flask into three Erlenmeyer flasks, adding 2 drops of the appropriate indicator to each.
- If the sample is **basic, use methyl red as the indicator**. When adding a few drops of **Methyl red** to the solution, it turns **yellow**.
- Titrate the sample with 0.1 M HCl until the solution turns to the color of *light brown onion*.
- Record the first volume of HCl at which the color change occurs. This first titration is a rough estimate; it gives an approximate volume of acid needed to titrate 25 mL of the sample.
- The volume of the acid used for titrating all three parallel samples should be approximately the same. If not, prepare another parallel sample for titration and repeat the process.
- For the average acid volume, consider only the titrations where the volumes of acid used are most similar.
- Calculate the average amount of acid used in the titrations.
- If the acid use varies significantly between titrations, the sample was unevenly mixed, and you should request a new sample from the assistant and repeat the titration.
- Using the measured volume of acid needed for titration and the concentration of the acid, calculate the concentration of the sample in the volumetric flask.
- If the sample in the flask **is acidic, add Phenolphthalein as the indicator**. The solution is **colourless** in an acidic medium.
- Titrate in the same manner as for basic titration but using 0.1 M NaOH.
- After the addition of the first drop of base, the solution turns light pink. Record the volume after the first drop that causes the color change.
- The color must remain stable— the solution should not decolorize. If it does, add another drop of base.
- Like the acid titration, calculate the average volume of base used in the three parallel titrations and finally determine the concentration of the sample in the volumetric flask.

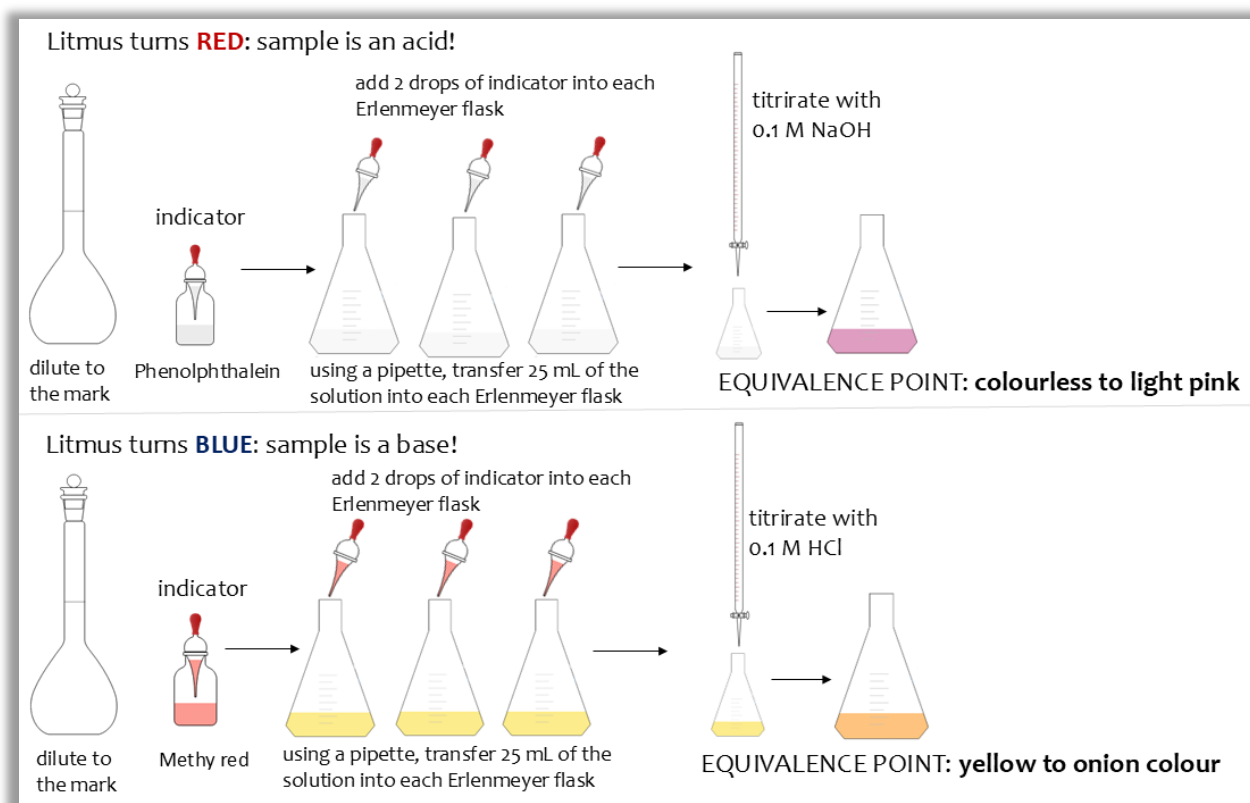


Figure 31. Procedure overview d). Source: Author's own work.



LET'S CONSIDER

1. Remember the colours of the indicators in acidic and basic media!
2. Explain why phenolphthalein is used as an indicator in acid-base titrations during the neutralization of acid with base, and why methyl red is used as an indicator during the titration of base with acid?
3. Write the reaction equation!
4. Explain what titration is!

EXP. N^o6: IONIC REACTIONS, DISTILLATION AND BACK-TITRATION

REQUIRED KNOWLEDGE: Concentrations of solutions, quantity of substances, ionic reactions, titration, distillation, weak and strong electrolytes.

SAFETY: Observe the rules for working with hot objects and apparatus, and follow the instructions for handling acids, bases, and gas equipment. Make sure that all gas valves in the laboratory are closed after finishing work. After the experiment, **do not pour the solution from the test tube down the drain;** instead, pour it into the container designated for collecting mercury-containing waste solutions.

LABORATORY TECHNIQUES

MEASUREMENTS OF LIQUIDS, PIPETTES, page 11
BURETTES, page 13

KEY FINDINGS

An ionic reaction occurs if one of the following is formed:

- sparingly soluble compound
- a poorly dissociated compound
- a readily volatile compound (gas)

EXERCISES

1. EXERCISE

Write and explain the ionic equations from the experimental exercise!

2. EXPERIMENTAL

Determine the mass of ammonium chloride in the obtained sample!

3. INVENTORY

Shared inventory

- 2 x stands
- Stopper with tubing for a funnel and a delivery tube
- Small test tube – test tube for adding agents
- Burette for 0.1 M HCl
- Burette for 0.1 M NaOH
- 2x tripod stands
- Boiling stones
- Gas burner

- 5 mL or 10 mL graduated cylinders
- Test tube stoppers

Personal inventory

- Test – tube rack
- Test tubes
- Bakers
- 1000 mL flat bottom flask
- Graduated cylinder
- Erlenmeyer flask
- 2x wire gauze
- Clamp holder
- Universal clamp
- Container for collecting Hg – waste solution

4. CHEMICALS

- CaCO_3 powder
- Stones
- 2 M HCl
- 0.1 M HCl
- 2 M NaOH
- 0.1 M NaOH
- 0.1 M AgCl
- 0.1 M AgNO_3
- 0.1 M $\text{Hg}(\text{NO}_3)_2$
- Methyl red

5. PROCEDURE

a) Formation of a gaseous product

- In a test-tube rack, place a test tube and add a knife-tip amount of powdered CaCO_3 .
- Using a graduated cylinder, measure 5.0 mL of 2.0 M HCl and pour the acid into the test tube containing the CaCO_3 powder.
- Observe the reaction and explain its course!
- In a second test tube in the rack, pour 5.0 mL of 2.0 M HCl and drop in a small stone (collected from the road).

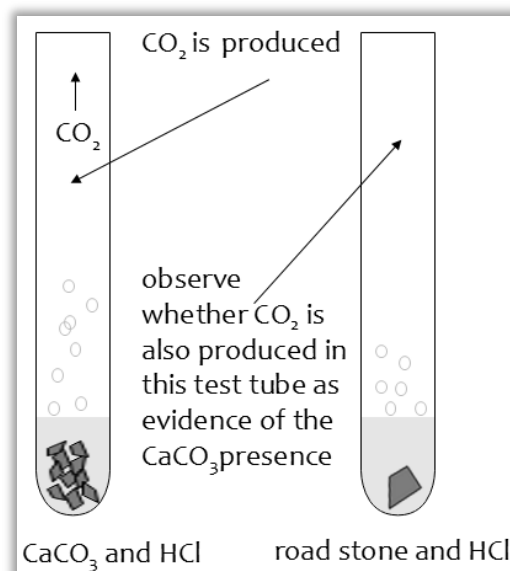


Figure 32. Procedure overview a).

Source: Author's own work.

- Observe whether a reaction occurs. If it does, explain its course!
- Why does the reaction not occur in some cases?

b) **Formation of a sparingly soluble and poorly dissociated substance**

- Pour approximately 1 mL of 0.10 M NaCl into the third test tube.
- Add two drops of 0.10 M AgNO₃.
- Observe the course of the reaction!
- To the white AgCl precipitate formed, add 0.10 M Hg(NO₃)₂ solution dropwise while gently shaking the test tube.
- What do you notice?
- Describe the course of the reaction, list the reactions that took place and write the corresponding reaction equations!

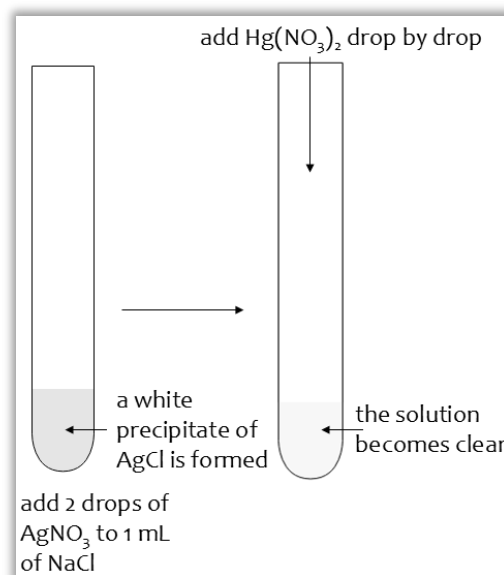


Figure 33. Procedure overview b).

Source: Author's own work.

c) **Determination of the mass of ammonium chloride in the sample**

- In pairs, hand the assistant a 1000 mL flat-bottom flask.
- Dilute the received sample in the flask with **50 mL of distilled water**.
- Add a few boiling stones (3–4) to the prepared solution.
- Find a stopper with an inlet tube that provides a good seal for the flask outlet.
- Using a burette, dispense **50 mL of 0.10 M HCl** into an Erlenmeyer flask.
- Assemble the apparatus according to the diagram.
- Using a 100 mL graduated cylinder, measure **50 mL of 2.0 M NaOH** and carefully pour it into the flask through the funnel at the top of the apparatus.
- Begin heating the solution in the flask and continue heating until the solution volume in the flask is reduced to one-third of the original volume. During the reaction ensure that the inlet tube always remains submerged in the HCl solution in the Erlenmeyer flask.
- **Heat the sample solution only to gentle boiling.**
- Replace the cooling water in the beaker that cools the contents of the Erlenmeyer as

needed. Take care not to lift the tubes out of the HCl solution in the Erlenmeyer.

- When the reaction is complete, first rinse the end of the inlet tube with a few mL of distilled.
- **Move the Erlenmeyer flask away from the apparatus, then extinguish the burner.**
- Cool the solution in the Erlenmeyer to room temperature, add a few drops of methyl red indicator, and titrate the excess HCl in the Erlenmeyer with 0.10 M NaOH to the first persistent color change. Titrate carefully and slowly, as you have only one replicate for calculating the mass of NH_4Cl !
- Write the reaction equations and calculate the mass of ammonium chloride in the sample!

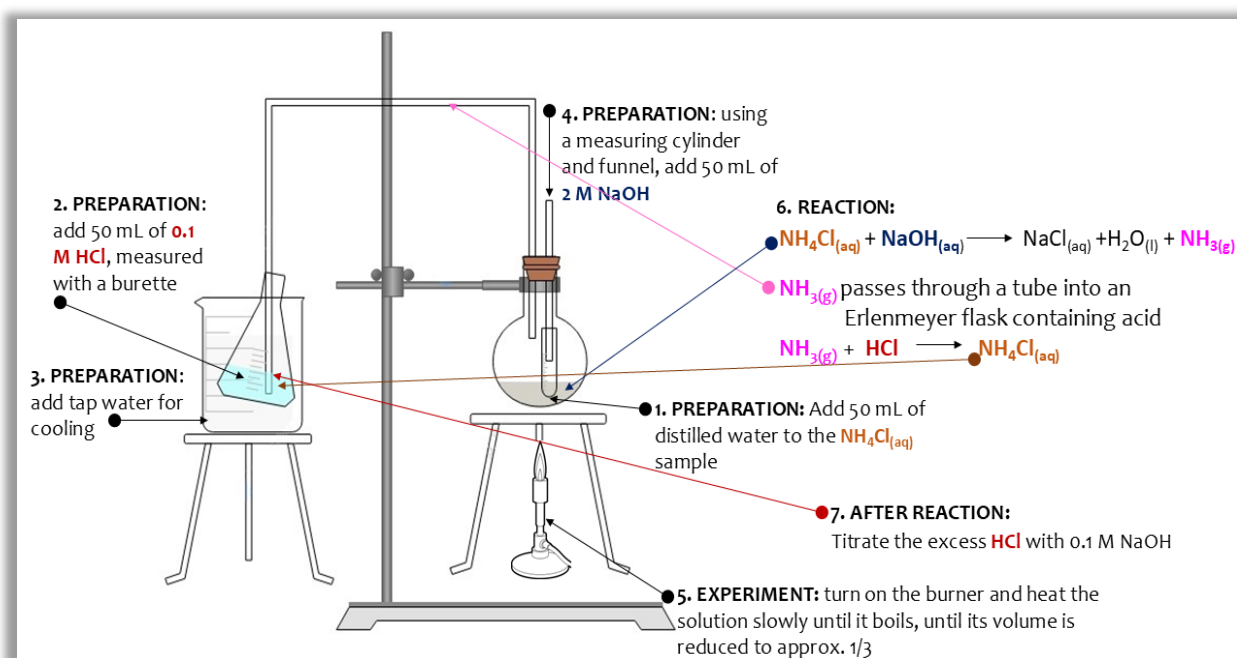


Figure 34. Procedure overview c). Source: Author's own work.



LET'S CONSIDER

1. Why are boiling stones added to the solution during heating?
2. Why is the sample NaOH added only after the apparatus is fully assembled and the acid is already in the Erlenmeyer flask?
3. What is the purpose of the liquid trap (liquid seal)?
4. Why must the acid solution in the Erlenmeyer be cooled during the reaction?
5. Why is methyl red used as the indicator for the titration?

EXP. N^o7: CHEMICAL EQUILIBRIUM, EQUILIBRIUM CONSTANT OF A CHEMICAL REACTION

REQUIRED KNOWLEDGE: Chemical equilibrium. Equilibrium constant K_c . Initial quantities.
Equilibrium quantities. Law of mass action. Le Chatelier's principle.
Ionization of water, pH.

SAFETY: Observe rules for working with hot objects and apparatus and follow the instructions for handling acids and bases. **WEAR SAFETY GOGGLES AND GLOVES ALL THE TIME!**

LABORATORY TECHNIQUES

MEASUREMENT OF LIQUIDS, PIPETTES, page 11
PROPRER USE OF DROPPERS, page 15

KEY FORMULAS

Dissociation of a weak acid:

$$K_a = \frac{[H^+][A^-]}{[HA]}$$

Dissociation of a weak base:

$$K_b = \frac{[BH^+][OH^-]}{[B]}$$

$$pH = -\log[H^+]$$

EXERCISES

1. CALCULATION EXERCISES (data will be provided on a separate sheet)

- a) Acid dissociation constant K_a (CH_3COOH) = $1.74 \cdot 10^{-5}$. Calculate the degree of dissociation (α) of acetic acid:
- using the approximation method
 - using the quadratic equation
- b) Calculate the pH of a 0.10 M base with a degree of dissociation of.....!
- c) Calculate the molar concentration (molarity) that has pH = and K_d =
The fraction of dissociated molecules is negligible.

2. EXPERIMENTAL

Perform experiments in which you will observe changes in a solution related to the law of mass action (Le Chatelier's principle regarding concentration changes).

3. INVENTORY

Shared inventory

- 5 mL or 10 mL graduated cylinders

Personal inventory

- 100 mL beaker
- 100 mL graduated cylinder
- Test tube rack
- Test tubes

4. CHEMICALS

- 0.5 M CH_3COOH
- 0.5 M CH_3COONa
- 0.015 M $\text{Fe}_2(\text{SO}_4)_3$
- 0.1 M NH_4SCN
- 3 M NH_4Cl

5. PROCEDURE

a) Color change

- Pour 10 mL of 0.50 M CH_3COOH into a 100 mL beaker (measured with a 10 mL graduated cylinder).
- Add a few drops of methyl orange indicator to the solution in the beaker and mix well.
- Then measure 10 mL of 0.50 M CH_3COONa with the graduated cylinder and add it to the acid solution; observe the colour change after adding CH_3COONa .
- Explain the change using the law of mass action (Le Chatelier's principle)!

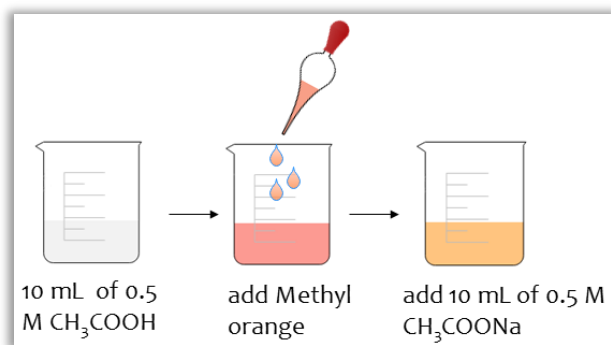


Figure 35. Procedure overview a). Source: Author's own work.

b) **Change in color intensity.** Before preparing solutions, wash glassware thoroughly, otherwise you will not see differences in color intensity!

- Pour 50 mL of distilled water into a 100 mL beaker (measured with a graduated cylinder).
- Using a 5 mL or 10 mL graduated cylinder, measure 1.0 mL of 0.10 M NH_4SCN and 1.0 mL of 0.015 M $\text{Fe}_2(\text{SO}_4)_3$; add both solutions to the distilled water in the beaker and mix thoroughly.
- A red-coloured solution of the poorly dissociated iron (III) thiocyanate complex is formed.
- Divide the solution into five test tubes to approximately equal heights.
- Add to the first test tube 2 mL distilled water; to the second 2 mL of 0.015 M $\text{Fe}_2(\text{SO}_4)_3$; to the third 2 mL of 0.10 M NH_4SCN ; to the fourth 2 mL of 3 M NH_4Cl .
- The fifth test tube is the control and receives no addition. Observe the change in color intensity by looking down the test tubes from above against a white background.
- The color intensity in each test tube depends on the concentration of the coloured iron (III) thiocyanate complex. Explain your observations using the law of mass action (Le Chatelier's principle)!

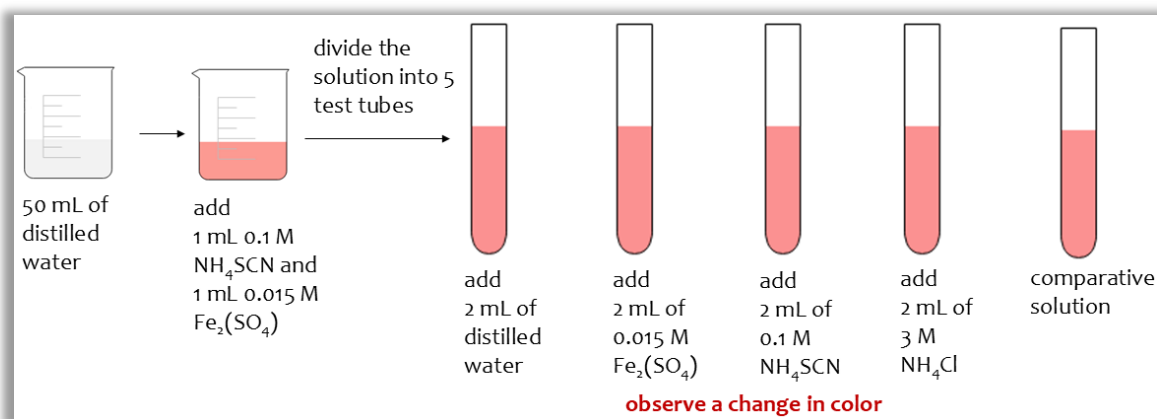


Figure 36. Procedure overview b). Source: Author's own work.



LET'S CONSIDER

1. What is the color of the acetic acid solution after adding methyl orange?
2. What is the color of the solution after adding sodium acetate, and why?
3. Write the equilibrium reaction for the formation of $[\text{Fe}(\text{SCN})]_3$! What happens after adding water, $\text{Fe}_2(\text{SO}_4)_3$ solution, NH_4SCN solution or NH_4Cl solution?

EXP. N°8: CHEMICAL EQUILIBRIUM, PROTOLYTIC EQUILIBRIA IN AQUEOUS SOLUTIONS, TITRATION CURVE

REQUIRED KNOWLEDGE: Ionic equilibria. Weak and strong electrolytes. Ionic reactions. Protolytic equilibria in aqueous solutions. Equivalence point. Neutral point.
Titration curve. Ionization of water, pH.

SAFETY: Follow instructions for working with hot objects: hold the test tube with a test-tube clamp while heating and heat the contents slowly to prevent splashing; after finishing work in the laboratory close all gas valves! **WEAR SAFETY GOGGLES!**

LABORATORY TECHNIQUES

MEASUREMENT OF LIQUIDS, PIPETTES, page 11

PROPER USE OF DROPPERS, page 15

PROPER HEATING OF LIQUIDS IN A TEST TUBE,
page 17

BURETTES, page 13

KEY FORMULAS



$$K_r = \frac{[M]^m \cdot [N]^n \cdot [O]^o}{[A]^a \cdot [B]^b \cdot [C]^c}$$

$$[X] = c(x)$$

EXERCISES

1. CALCULATION EXERCISES (data will be provided on a separate sheet):

Calculate the titration curve points (pH value at each mL of NaOH) and plot the titration curve for the following cases:

- 10 mL of ... M HCl is titrated by successive additions of 1 mL aliquots of ... M NaOH until a total of 20 mL NaOH solution has been added.
- 10 mL of ... M CH₃COOH is titrated by successive additions of 1 mL aliquots of ... M NaOH until a total of 20 mL NaOH solution has been added.

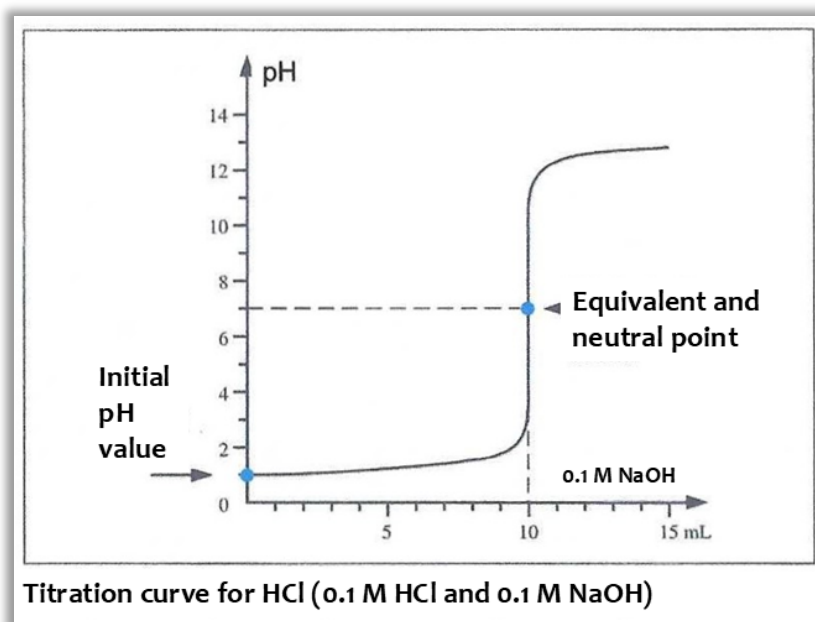


Figure 37. Example a) strong acid titrated with strong base: the equivalence point and the neutral point (pH = 7) coincide. [1]

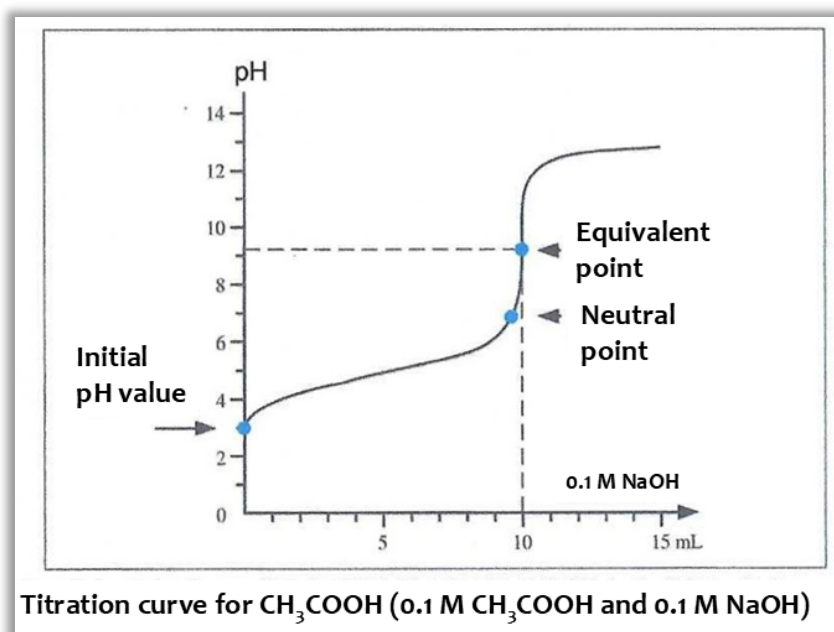


Figure 38. Example b) weak acid titrated with strong base. [1]

2. EXPERIMENTAL

Perform experiments to measure solution pH and determine carbonate hardness of water; describe and explain them!

3. INVENTORY

Shared inventory

- Stand
- Magnetic stirrer
- Stirrer magnets
- Stirring bar
- pH – meter
- Burettes for 0.1 M HCl.
- Bakers for rinsing waste (electrode rinse waste)
- 5 mL or 10 mL graduated cylinders
- Gas burner
- Spoons for salts

Personal inventory

- Bakers
- Test tubes rack
- Test tubes
- 100 mL graduated cylinder
- Test tube clamp.

4. CHEMICALS

- $\text{Na}_2\text{CO}_3 \cdot 10 \text{H}_2\text{O}$
- NH_4Cl
- 2 M NaCH_3COO ,
- Phenolphthalein
- Methyl orange

5. PROCEDURE

TECHNIQUE: Measuring the pH of solutions with a pH meter

- Before measuring pH, the pH meter must be calibrated.
- Between measurements the pH electrode should be kept immersed in distilled water.
- Before each measurement, rinse the electrode thoroughly with distilled water from a wash bottle and blot it dry with a soft paper towel. Do not rub the electrode with the towel!

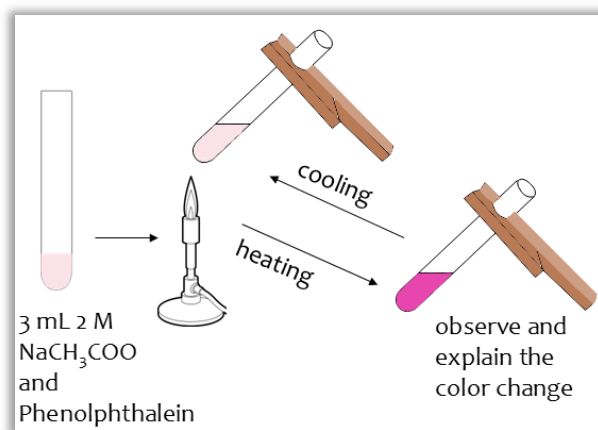
- Rinse and dry the stirring magnet with distilled water before use.
- Then immerse the electrode in the solution whose pH you intend to measure.
- Stir the solution continuously with a magnetic stirrer during measurement.
- Immerse the electrode at least 2 cm into the solution.
- Press the button marked "pH".
- Read the pH value only when the digits on the display stop changing.
- After the measurement, lift the electrode out of the solution, place a beaker for collecting rinse water underneath, and **rinse the electrode with distilled water from the wash bottle.**
- At the end of the session, leave the cleaned electrode immersed in distilled water to prevent drying and damage!

a) **pH of distilled water**

- Pour 50 mL of distilled water into a 100 mL beaker and measure its pH.
- Add 0.50 g $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ (about a teaspoon of the salt) to the water. Stir the solution until the salt dissolves, then measure the pH again.
- Explain why the pH changed after adding the salt!

b) **pH of tap water**

- Measure 50 mL of tap water into a 100 mL beaker and measure its pH.
- Add 0.50 g NH_4Cl (about a teaspoon of the salt) to the measured water, stir until all the salt dissolves, and then measure the pH of the solution again.
- Explain the change in the solution's pH!



c) **hydrolysis**

- Using a 5 mL or 10 mL graduated cylinder, measure 3.0 mL of 2.0 M sodium acetate into a test tube and add 3 drops of phenolphthalein. Heat the test-tube solution to boiling, then allow it to cool back to room temperature.

Figure 39. Hydrolysis. Source: Author's own work.

- Observe the colour change and explain why the color changed!

d) **Carbonate hardness of water**

- Give the assistant a 250 mL or 400 mL beaker!
- From the received sample, measure 100 mL into each of two Erlenmeyer flasks using a graduated cylinder and add 2–3 drops of methyl orange indicator to each flask.
- Titrate the water samples with 0.10 M HCl until the color changes from yellow to the “onion” colour (methyl orange endpoint).
- If the titration results differ significantly between the two parallels, request a new sample and perform another titration!
- Calculate the carbonate hardness of the sample from the volume of 0.10 M HCl used!

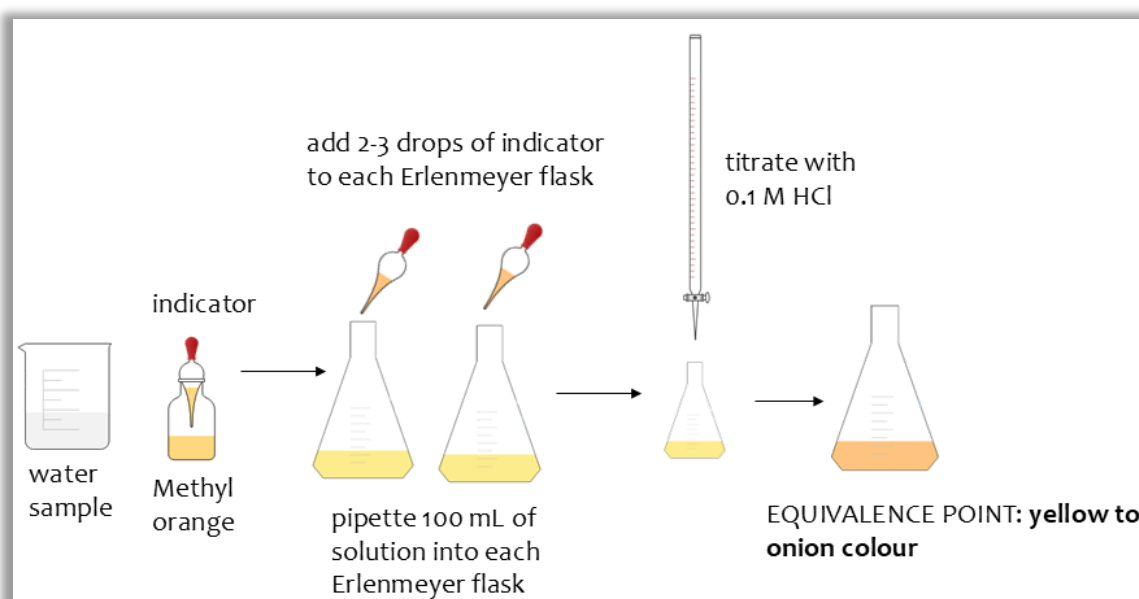


Figure 40. Procedure overview d). Source: Author's own work.

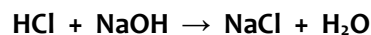


LET'S CONSIDER

1. What is the pH of distilled water and why? What causes the pH change after adding $\text{Na}_2\text{CO}_3 \cdot 10 \text{H}_2\text{O}$?
2. What is the pH of tap water and why does it change after adding NH_4Cl ?
3. What is carbonate hardness of water and how is it determined?

APPENDIX: TABLES FOR CALCULATING pH AS A FUNCTION OF VOLUME for the **TITRATION CURVE** (calculate and plot the curve using your own measured data from data sheet).
Calculate pH to two decimal places.

- a) **Titration of a strong acid with a strong base. Example:** 10.0 mL of 0.10 M HCl is titrated by successive additions of 1.0 mL aliquots of 0.10 M NaOH until a total of 20.0 mL of NaOH solution has been added.



First the equivalence point is calculated:

$$n_K = n_B \Rightarrow c_K \cdot V_K = c_B \cdot V_B$$

$$V_B = \frac{c_K \cdot V_K}{c_B} = \frac{0,1 \frac{\text{mol}}{\text{L}} \cdot 10 \text{ mL}}{0,1 \frac{\text{mol}}{\text{L}}} = 10 \text{ mL}$$

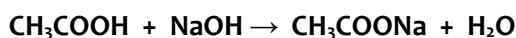
Table 10. Strong acid/Strong base.

V(NaOH) v mL	[H ⁺] or [OH ⁻]	pH
0 [H ⁺] = c _k		
1 [H ⁺] = (n _k - n _b) / (V _k + V _b)		
2		
3		
4		
5		
6	The equation remains the same up to the equivalence point!	
7		
8		
9		
10 mL: Equivalence point. Calculation is shown below!		
11 [OH ⁻] = (n _b - n _k) / (V _k + V _b)		
12		
13		
14		
15		
16	Equation remains the same until 20 mL!	
17		
18		
19		
20		

Equivalence point. Equation and calculation: n_k = n_b. K_w = [H⁺] · [OH⁻] =

$$10^{-14}. [\text{H}^+] = [\text{OH}^-] = x. x^2 = 10^{-14}. x = 10^{-7}. \text{pH} = \underline{7.00}$$

b) Titration of a weak acid with a strong base. Example: 10 mL of 0.50 M CH₃COOH is titrated by successive additions of 1.0 mL of 0.40 M NaOH until a total of 20.0 mL of NaOH solution has been added. $K_{\text{disoc.}} = 1.8 \cdot 10^{-5}$



First the equivalence point is calculated

$$n_K = n_B \Rightarrow c_K \cdot V_K = c_B \cdot V_B$$

$$V_B = \frac{c_K \cdot V_K}{c_B} = \frac{0,5 \frac{\text{mol}}{\text{L}} \cdot 10 \text{ mL}}{0,4 \frac{\text{mol}}{\text{L}}} = 12,5 \text{ mL}$$

Table 11. Weak acid/Strong base.

V(NaOH) v mL	[H ⁺] or [OH ⁻]	pH
0	$[H^+] = \sqrt{K_K \cdot c_K}$	
1	$[H^+] = K_K \cdot c_K / c_s = K_K \cdot (n_K - n_B) / n_B$	
2		
3		
4		
5		
6	The equation remains the same up to the equivalence point!	
7		
8		
9		
10		
11		
12		
12.5 mL: Equivalence point. Calculation is shown below!		
13	$[OH^-] = (n_B - n_K) / (V_K + V_B)$	
14		
15		
16		
17	Equation remains the same until 20 mL!	
18		
19		
20		

Equivalence point. Equation and calculation.

Given that we have a weak acid and a strong base, their salt will be slightly basic:

$$n_K = n_B \Rightarrow c_K \cdot V_K = c_B \cdot V_B$$

$$c_S = \frac{n_K}{V_K + V_B} = \frac{n_B}{V_K + V_B}$$

$$[OH^-] = \sqrt{\frac{K_W}{K_K} \cdot c_S}$$

EXP. N^o9: SOLUBILITY PRODUCT

REQUIRED KNOWLEDGE: Slightly soluble salts. Ionic equilibria. Solubility product. Solubility of a salt. Saturated solution concentration. Common-ion effect on salt solubility.

SAFETY: Follow the instructions for handling acids! **WEAR SAFETY GOGGLES AND GLOVES!**

LABORATORY TECHNIQUES

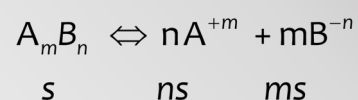
MEASUREMENT OF LIQUIDS, PIPETTES, page 11

PROPER USE OF DROPPERS, page 15

VOLUMETRIC FLASKS, page 15

FILTRATION, page 16

KEY FORMULAS



$$K = [A^{+m}]^n \cdot [B^{-n}]^m$$

$$= (ns)^n \cdot (ms)^m$$

$$= n^n m^m s^{n+m}$$

$$s = \left(K / n^n m^m \right)^{\frac{1}{n+m}}$$

EXERCISES

1. CALCULATION EXERCISES (data will be provided on a separate sheet):

- The solubility product of..... is How many mg of..... can dissolve in 1 L of water?
How many mg will dissolve in 1 L of M
- The solubility product of..... is Calculate the molar concentration (molarity) of the saturated solution! How many mg of..... can dissolve in 150 L of water?

2. EKSPERIMENTAL

- Perform the reaction between silver (Ag^+) ions and chloride (Cl^-) ions at different concentrations and determine the threshold concentration at which no precipitate forms!
- Decrease the solubility of lead chloride ($PbCl_2$)!
- Decrease the solubility of sodium chloride ($NaCl$)!

3. INVENTORY

Shared inventory

- Analytical balance
- Stand
- Filter paper
- 10 mL volumetric pipette
- Ring clamp
- Rubber bulb
- Laboratory spatula
- 10 mL graduated cylinder

Personal inventory

- Bakers
- Test tubes
- Glass funnel

4. CHEMICALS

- 0.1 M solution of AgNO_3
- PbCl_2
- Saturated solution of NaCl
- 2 M NaCl
- 1 M H_2SO_4 (in a dropper bottle)
- 37 % HCl

5. PROCEDURE

a) Different concentrations of AgNO_3

- Four students work together to prepare solutions; each contributes one 100 mL volumetric flask.
- Prepare the solutions as exactly as possible so that differences in turbidity after adding a few drops of 2 M NaCl are clearly visible.
- **Thoroughly wash glassware before preparing solutions;** otherwise, differences in precipitate intensity may not be visible!
- Using a volumetric pipette, transfer **10.0 mL of 0.10 M AgNO_3** into a 100 mL volumetric flask and dilute to the mark with distilled water. The prepared solution concentration is 0.010 M.
- From this solution, pipette 10.0 mL with the volumetric pipette into a second 100 mL volumetric flask and dilute to the mark with distilled water. This solution has concentration 0.0010 M.

- Repeat the dilution twice more to prepare solutions of 0.00010 M and 0.000010 M.
- **Pour the four solutions** into separate test tubes to one-third their height. Add a few drops of 2 M NaCl to each test tube, mix, and determine at which Ag^+ concentration no precipitate forms. Explain why no precipitate formed at that concentration!

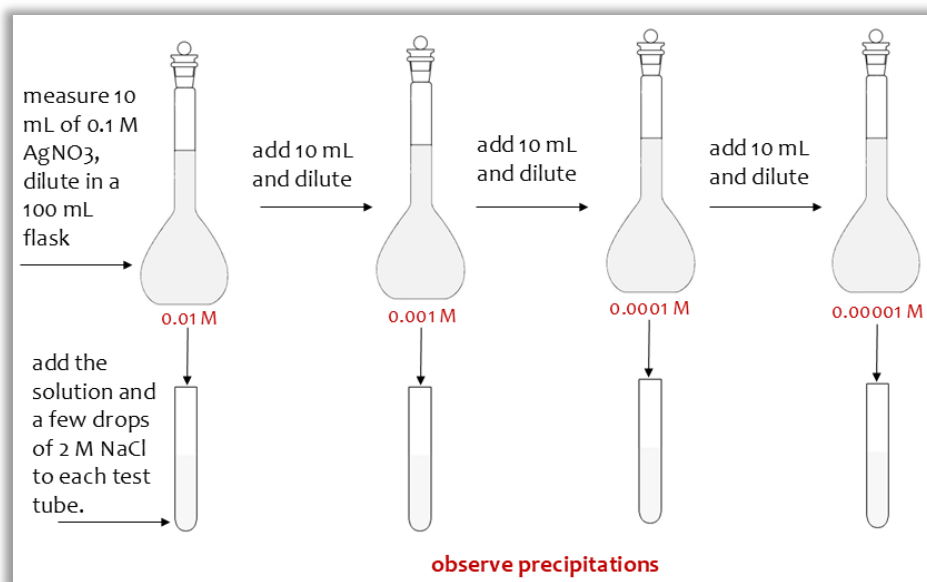


Figure 41. Procedure overview a). Source: Author's own work.

b) **Decreasing the solubility of PbCl_2**

- Prepare ~30 mL of a saturated PbCl_2 solution: dissolve 0.50 g PbCl_2 in 30 mL distilled water and stir for several minutes.
- Filter the saturated solution through filter paper into a 100 mL beaker and split the filtrate between two test tubes.
- To the first test tube add 1 mL of saturated NaCl solution. A white precipitate forms; filter it off again. Explain!
- To the filtrate remaining after the first filtration - and also to the filtrate from the second filtration - add a few drops of 1 M H_2SO_4 .

- Observe the reaction and write/explain the equations. In which test tube does more precipitate form, and why?

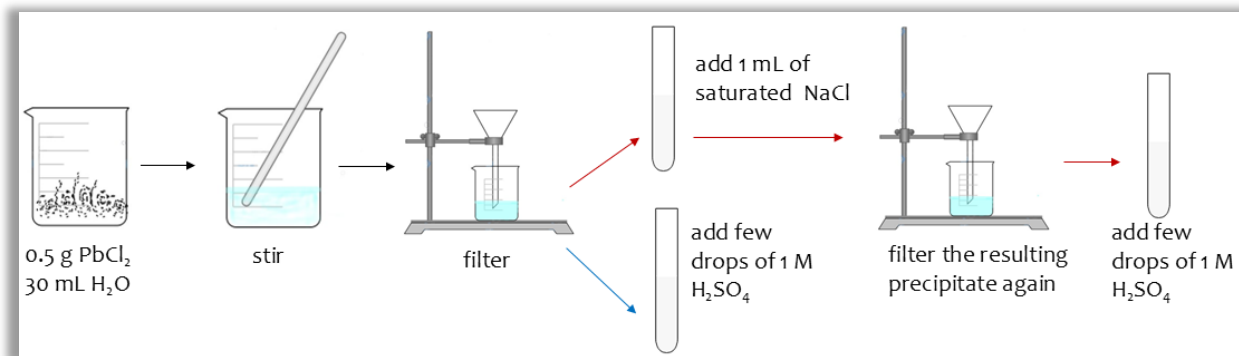


Figure 42. Procedure overview b). Source: Author's own work.

c) Decreasing the solubility of NaCl

- Set up a demonstration apparatus like the one used for determining ammonium salts.
- Because an irritating gas is produced, perform the experiment in a fume hood.
- Pipette 20 mL of 37 % HCl into a flask and add a few boiling stones. Place 20–30 mL of saturated NaCl solution in a beaker.
- Heat the HCl in the flask gently to produce a slow boil; gaseous HCl is generated and carried through a tube into the beaker containing the saturated NaCl solution.
- Describe and explain what happens in the beaker solution when gaseous HCl is bubbled through the saturated NaCl. After the experiment, pour water into the flask to prevent further release of HCl gas.

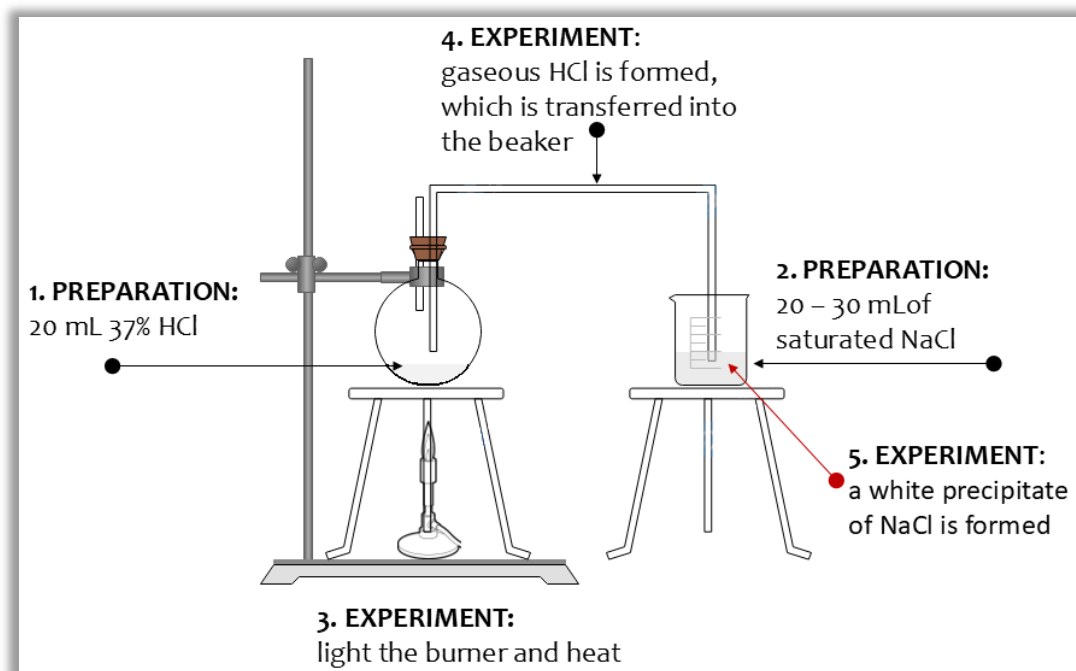


Figure 43. Procedure overview c). Source: Author's own work.



LET'S CONSIDER

1. Why did the solubility of lead (II) chloride decrease?
2. What caused the reduced solubility of NaCl in the solution after introducing gaseous HCl and describe how you observed this!

EXP. N^o10: OXIDATION-REDUCTION REACTIONS

REQUIRED KNOWLEDGE: Arrangement of redox equations. Oxidation numbers. Oxidizing agents and reducing agents in inorganic chemistry. Electrochemical series for metals and nonmetals.

SAFETY: Follow the instructions for handling acids and bases! Dispose of waste products containing CCl_4 into the designated container at the waste-drain area! WEAR SAFETY GOGGLES AND GLOVES!

LABORATORY TECHNIQUES

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VOLUMETRIC FLASKS, page 15

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KEY FINDINGS

Diluted HNO_3 (< 60 %) a gas NO is formed

Concentrated HNO_3 (\geq 60 %) a gas NO_2 is formed

EXERCISES

1. CALCULATION EXERCISES (data will be provided on a separate sheet)

Calculate how many millilitres of HNO_3 with a density g/mL are required to dissolve g of! Also calculate how many litres and which gas is produced in the reaction at a temperature of °C and a pressure of kPa.

2. EXPERIMENTAL

- In the electrochemical series, determine the positions of Zn and Cu relative to hydrogen!
- Determine which of the halogens is the stronger oxidizing agent!
- Establish the oxidizing strength of potassium permanganate (KMnO_4) in different media (acidic, neutral, alkaline)!
- Determine the mass of Fe^{2+} in the sample provided by the assistant by titrating the sample with 0.020 M KMnO_4 !

3. INVENTORY

Shared inventory

- Stand
- Burette
- 5 mL or 10 mL graduated cylinder

Personal inventory

- 250 mL volumetric flask
- Test tubes
- Test tubes rack
- 100 mL graduated cylinder
- 25 mL volumetric pipette
- Erlenmeyer flasks

4. CHEMICALS

- 2 M HCl
- Zn granules
- Pieces of Cu
- 0.25 M ZnCl_2
- 0.25 M CuCl_2
- 0.1 M NaBr
- 0.1 M KI
- Chlorine solution
- CCl_4
- 0.02 M KMnO_4
- 20 % NaOH
- 0.1 M Na_2SO_3
- 1 M H_2SO_4

5. PROCEDURE

a) Electrochemical series for metals

- Pour 3 mL of 2 M HCl into each of two test tubes. Place a zinc granule in the first tube and a piece of copper in the second.

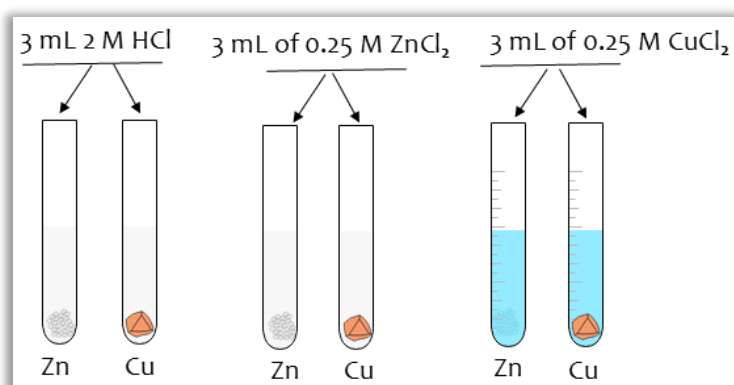


Figure 44. Procedure overview a). Source: Author's own work.

- Pour 3 mL of 0.25 M ZnCl_2 into each of two test tubes. Place a zinc granule in the first tube and a piece of copper in the second.
- Pour 3 mL of 0.25 M CuCl_2 into each of two test tubes. Place a zinc granule in the first tube and a piece of copper in the second.
- Observe the course of the reactions, explain them, and write all reaction equations that occurred!

b) **Electrochemical series for nonmetals**

- Into the first test tube pour 1 mL of 1.0 M NaBr; into the second test tube pour 1 mL of 0.10 M KI. To each test tube add 2 mL of chlorine solution and 1 mL of carbon tetrachloride (CCl_4).
- Cap the tubes, shake vigorously and observe the colours of the two separated layers.
- Explain the experiment and write the reaction equations!
- A brown color in the CCl_4 layer indicates elemental bromine; a violet color in the CCl_4 layer indicates elemental iodine.

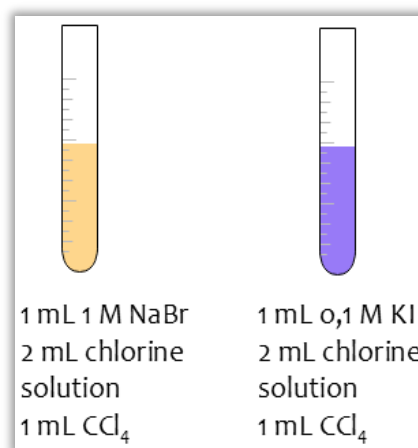


Figure 45. Procedure overview c).

Source: Author's own work.

c) **Oxidizing properties of KMnO_4**

- Into three test tubes, add 1 mL of 0.020 M KMnO_4 to each and dilute each with 3 mL of distilled H_2O .
- To the first test tube add 2 mL of 1 M H_2SO_4 ; to the second add 2 mL of distilled water; to the third add 2 mL of 20 % NaOH .
- To each of the prepared solutions, add 0.10 M Na_2SO_3 dropwise until the solution changes color. After adding Na_2SO_3 , mix the solution in the test tube.
- Write the redox reaction equations in ionic form!

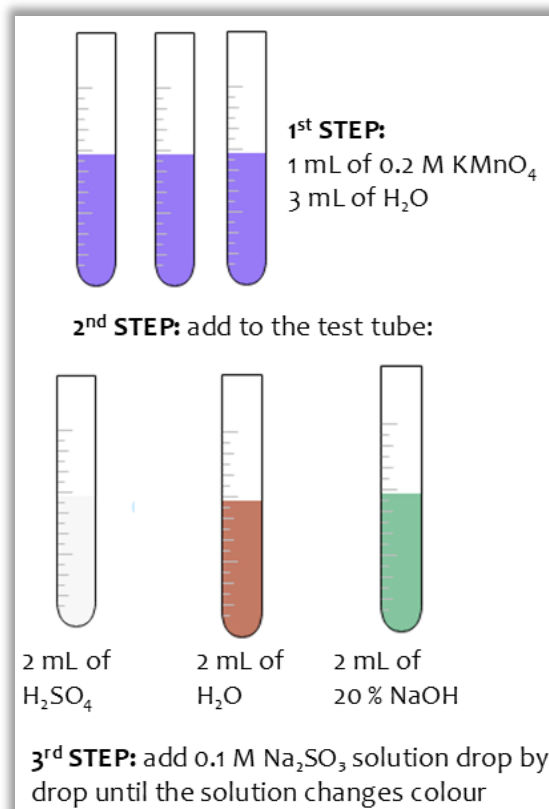


Figure 46. Procedure overview c).

Source: Author's own work.

d) **Determination of the mass of iron in the sample**

- Dilute the Fe^{2+} sample obtained from the assistant to the mark in a 250 mL volumetric flask and mix the solution thoroughly.
- Using a volumetric pipette, transfer 25 mL of the diluted sample into each of three Erlenmeyer flasks. Using a graduated cylinder, add 25 mL of 1 M H_2SO_4 to each flask.
- Titrate with 0.020 M KMnO_4 until the solution shows a persistent light pink color (end point).
- Calculate the average consumption of KMnO_4 from the titrations and determine the mass of iron in the entire sample.
- Also write the reaction equation!

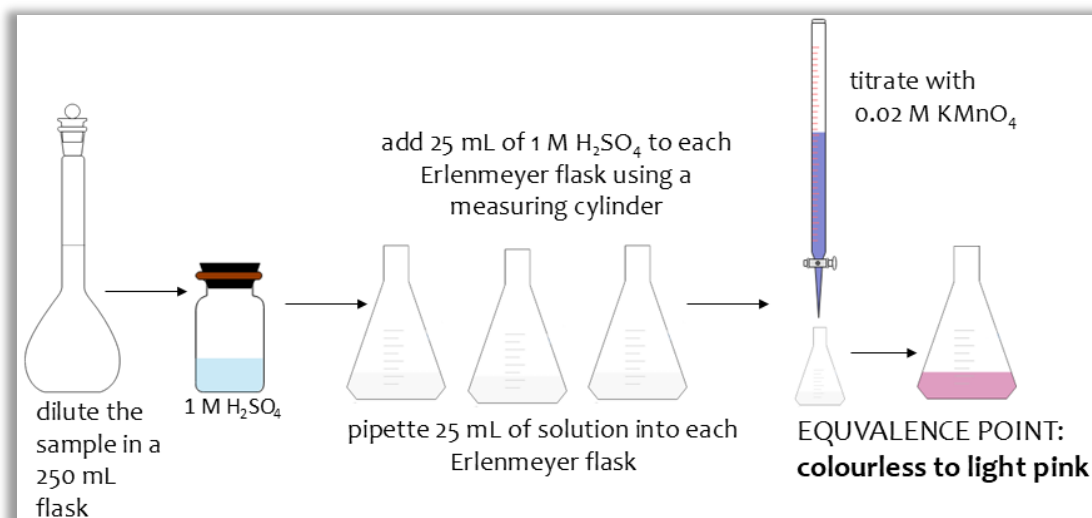


Figure 47. Procedure overview d). Source: Author's own work.



LET'S CONSIDER

1. Which reactions occur in the experiments under point a)?
2. How do you conclude which halogen is the stronger oxidant?
3. Describe the colours of the solutions in experiment c) (KMnO_4 with added Na_2SO_3) in acidic, neutral and alkaline media after adding Na_2SO_3 .

RECOMMENDED LITERATURE FOR FURTHER STUDY

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EXPERIMENTS IN GENERAL AND INORGANIC CHEMISTRY: LABORATORY EXERCISE INSTRUCTIONS

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This manual provides practical instructions for laboratory experiments in general and inorganic chemistry. Students learn the fundamentals of experimental work: laboratory equipment, basic techniques, and assembly of apparatus for chemical experiments. Calculations are linked to laboratory work, students practice writing reports, and they are introduced to laboratory safety.

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