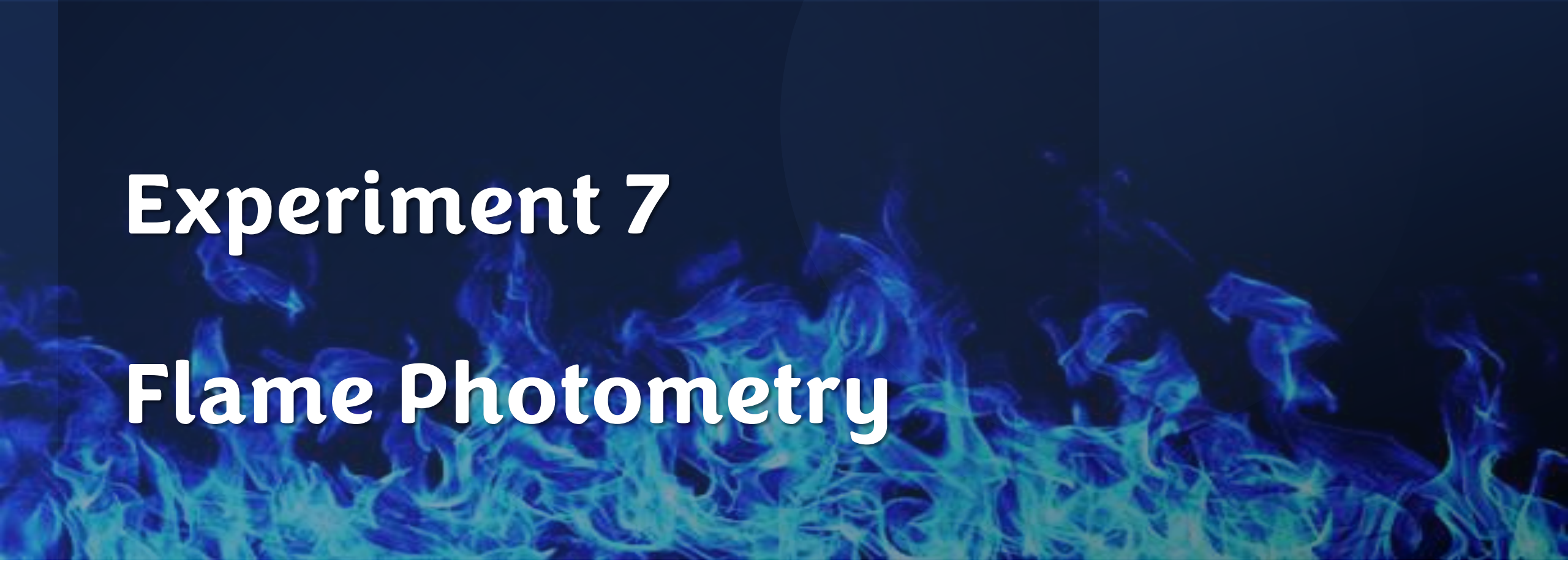


Experiment 7

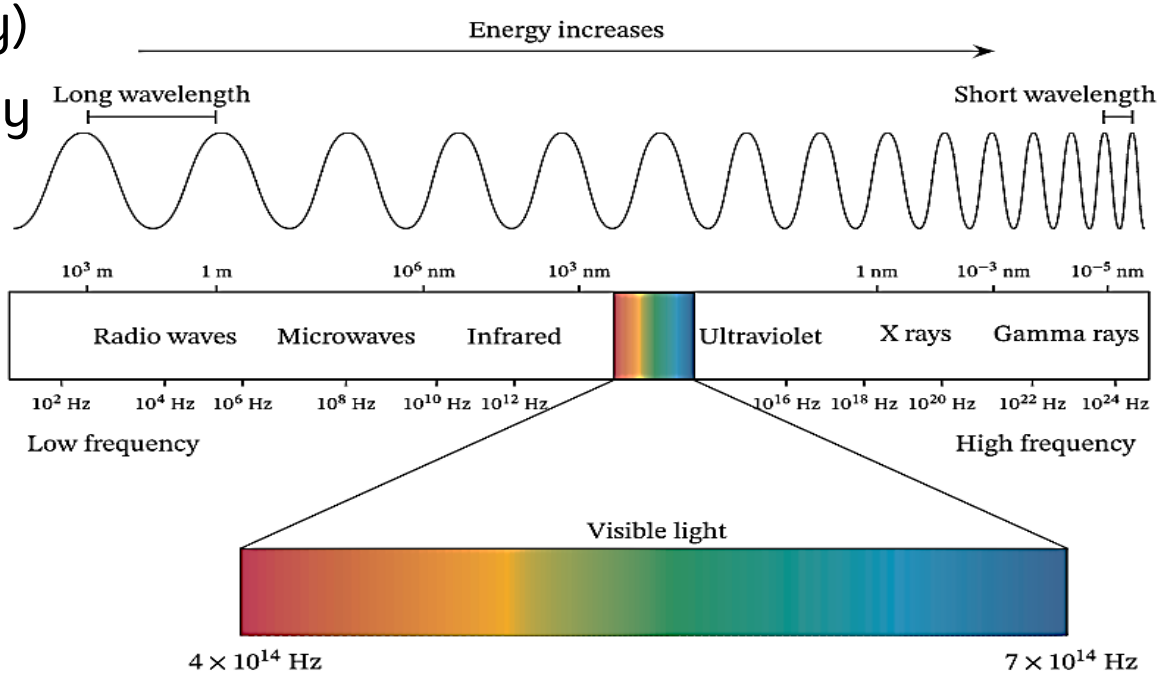
Flame Photometry

INTRODUCTION

Atomic spectroscopy is based on the absorption, emission, or fluorescence process of light by atoms or elementary ions. Information for atomic scale is obtained in two regions of the electromagnetic radiation (EMR) spectrum. These regions are UV/VIS and the X-ray.

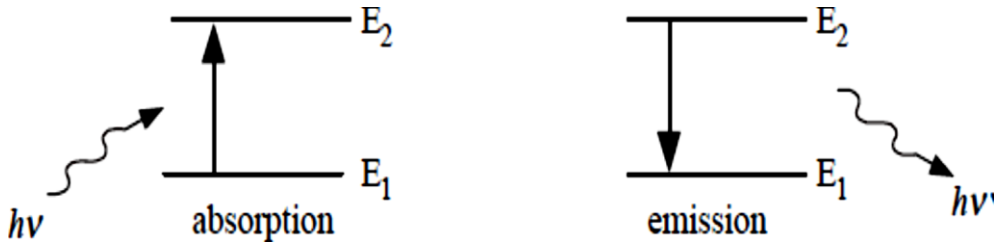
Atomic spectroscopy can be divided as:

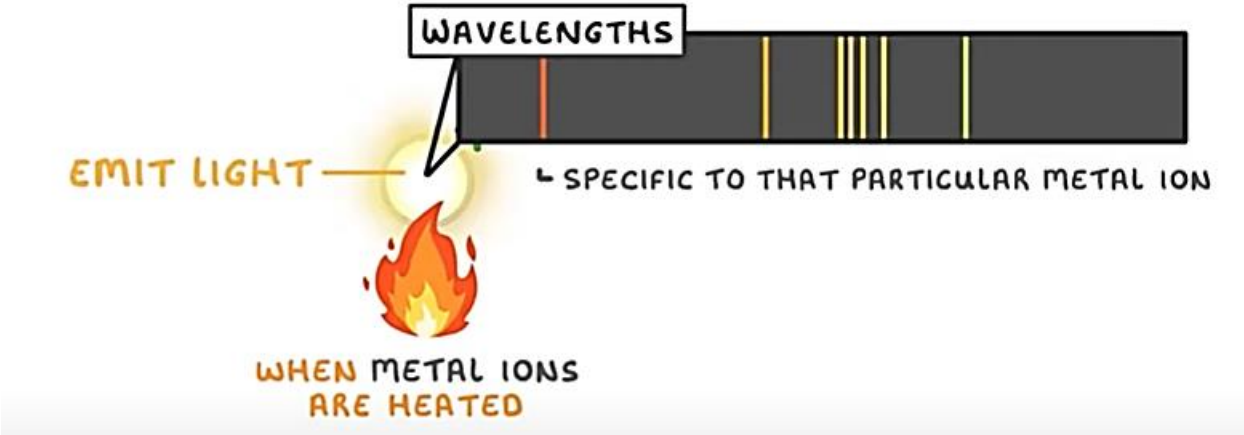
1. Absorption spectroscopy (UV-Vis)
2. Emission spectroscopy (Flame photometry)
3. Luminescence / fluorescence spectroscopy



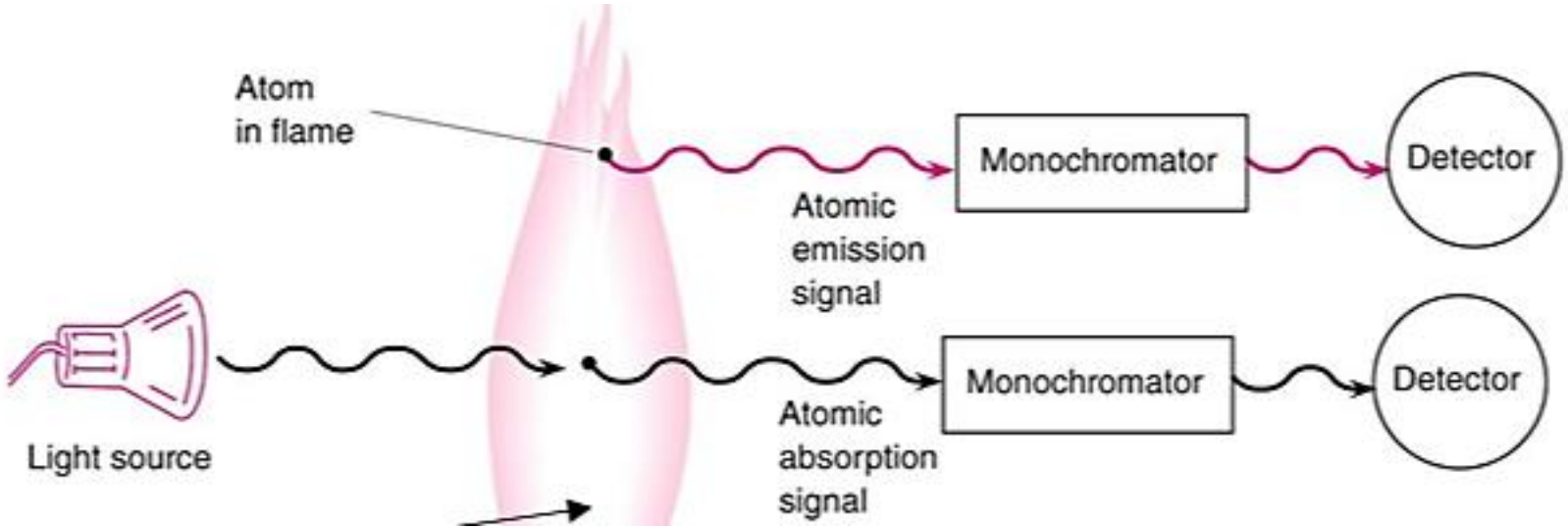
When sample interacts with light, absorption process occurs. Ground state electrons of the sample atom tend to move to the excited states with the energy of absorbed light. This process can also be called **excitation**.

- Apart from light, **heat** can cause excitation. Since excited state is unstable, electrons want to return back to the ground state. When an excited electron turns back to its ground state a radiation is emitted that is equal to the energy difference between excited and ground states. The emitted light is monochromatic, and it has the same wavelength as the light absorbed in the excitation process.



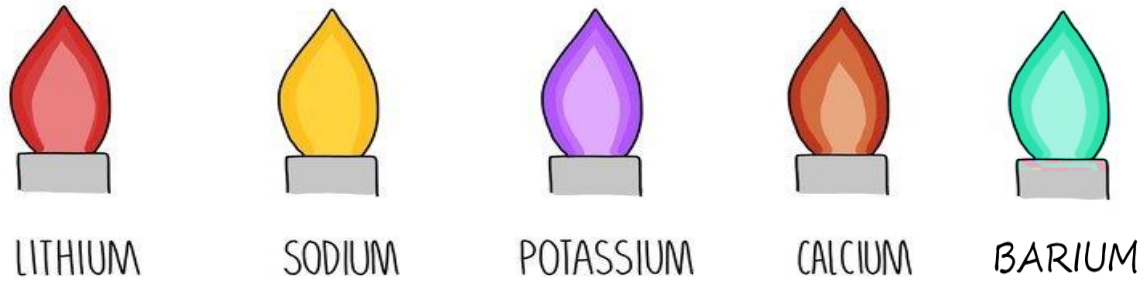


- Depending on the excitation technique, absorbed or emitted light is measured. If excitation source is flame, emitted radiation is measured. On the other hand, absorption is measured when lamp is used for excitation. Both are directly proportional with the number of atoms in the sample.



DEFINITION

Flame photometry or **flame emission spectroscopy** is an atomic emission technique used for the determination of elements which can be easily excited to higher energy levels at flame temperature such as **alkali (Cations) and alkaline earth metals**. These metals are **Na, K, Ca, Ba, Li.**



This method is based upon the measurement of **intensity of radiation emitted**, in the **visible region**, when a metal atom is introduced into a flame. The wavelength of the radiation (or the color), emitted tells us what the element is (**qualitative**), and the intensity of the radiation tells us how much of the element is present (**quantitative)**.

Flame

A flame can be described as a steady state gas phase reaction which takes place with emission of light. It is produced by burning a mixture of ***fuel*** *and* ***oxidant*** *in a burner. In flame photometry a variety of* ***fuels*** *can be used. O****xidant can g****enerally be air, oxygen or nitrous oxide (N_2O). The flame temperature depends on fuel to oxidant ratio.*

❖***The most commonly employed flames in flame photometry can be grouped into two types:***

1. Flames in which the fuel and oxidant as air or oxygen are well mixed before combustion, these are **called** pre-mix or laminar flames as they exhibit laminar flow.
2. Flames in which the fuel gas and the oxidant are first mixed in the flame itself. They are called unpremix or turbulent flames since they exhibit turbulence

Flame is formed by two components: fuel and oxidant.

Temperature of the flame changes depending on the fuel and oxidant types and their proportions.

In flame photometer generally **natural gas is used as a fuel** and **air is the oxidant**. Table 1 lists the different types of fuel, oxidant, and the temperature of the flame.

FUEL	OXIDANT	TEMPERATURE, °C
Natural Gas	Air	**1700**-1900
Natural Gas	Oxygen	2700-2800
Hydrogen	Air	2000-2100
Hydrogen	Oxygen	2550-2700
Acetylene	Air	2100-2400
Acetylene	Oxygen	3050-**3150**
Acetylene	Nitrous Oxide	2600-2800

Flame Structure

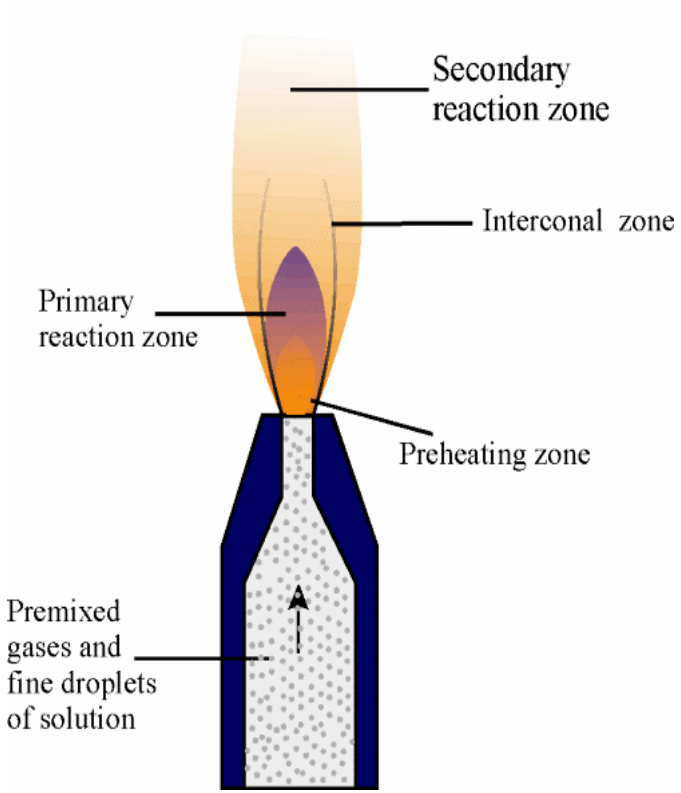
Flame consists of four important regions that their appearance and relative sizes changes with the fuel-oxidant ratio. These are:

i. **preheating zone** where the combustion mixture is heated to the ignition temperature by thermal conduction from the primary reaction zone.

ii. **The primary combustion** zone of the flame is **blue** in color. In this region, the concentration of ions and free radicals is very high and there is no thermal equilibrium. Therefore, it is not used in flame spectroscopy.

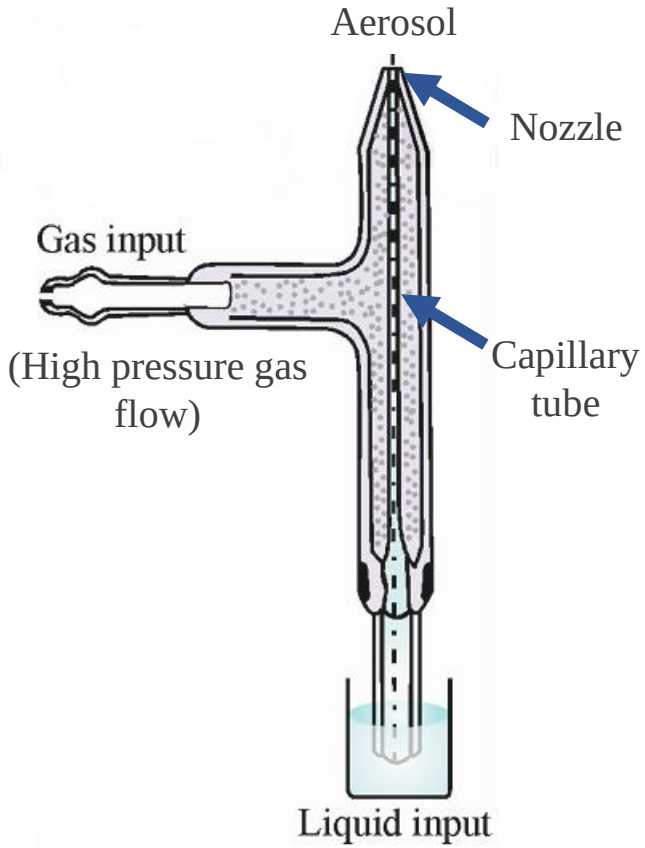
iii. **The interconal region** Where the maximum temperature is achieved just above the tip of the inner zone. It is rich in free atoms and is the most widely used region for flame spectroscopy.

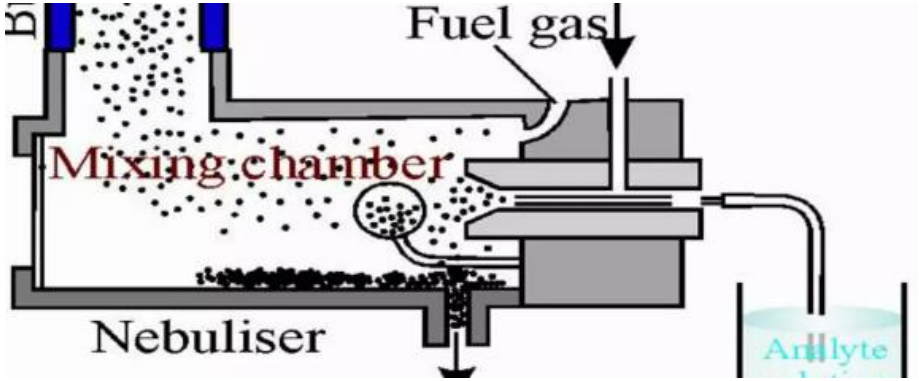
iv. **The outer cone (s**econdary reaction zone) – In this zone, the products of the combustion processes are burnt to stable molecular species by the surrounding air.

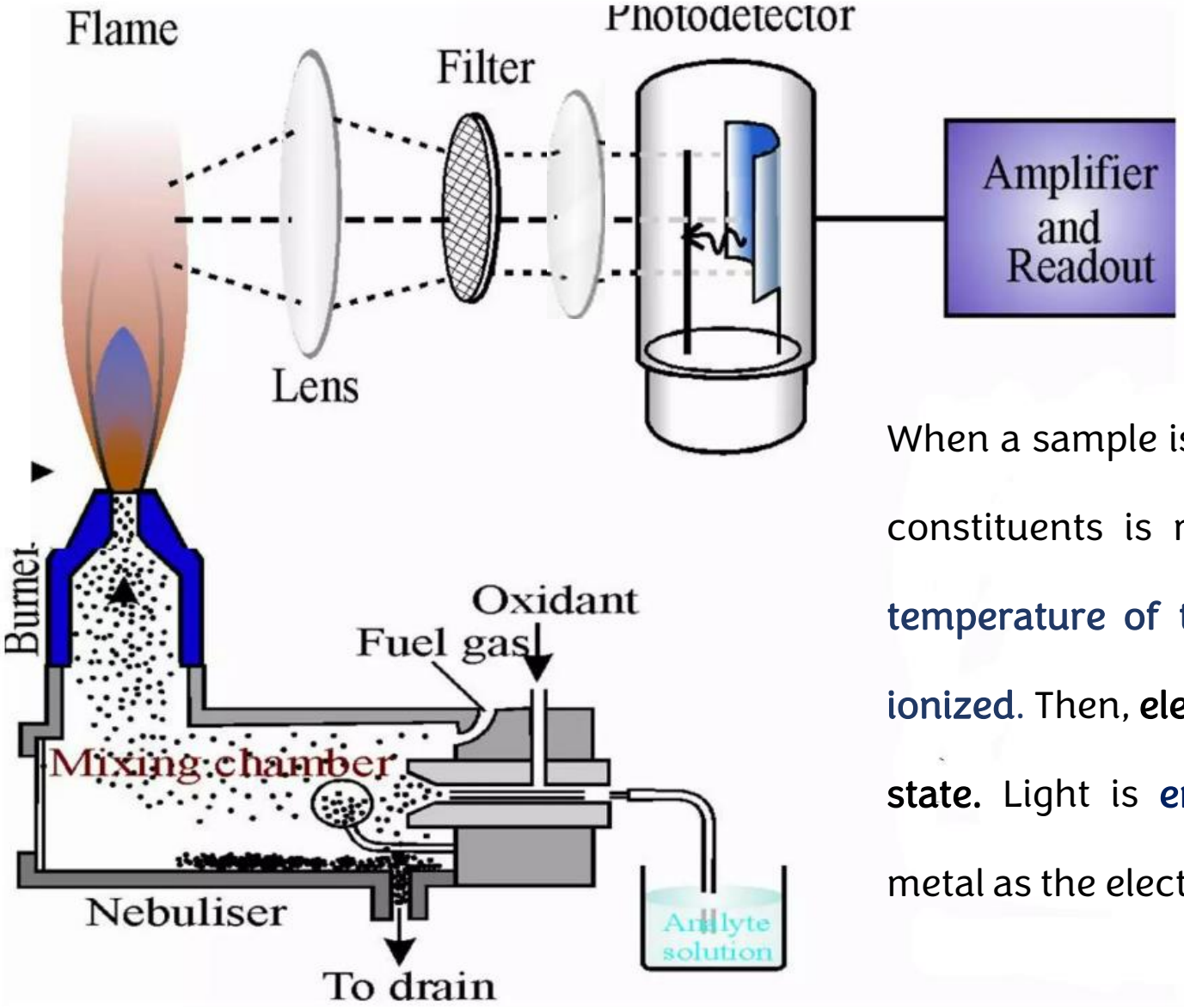


TECHNIQUE

In this technique, first aerosols are formed from sample solution by a jet of compressed gas. This process is called **nebulization.** Then the flow of the gas carries the aerosols into a flame where atomization takes place. **Atomization** is the conversion of sample aerosols into an atomic vapor by flame.



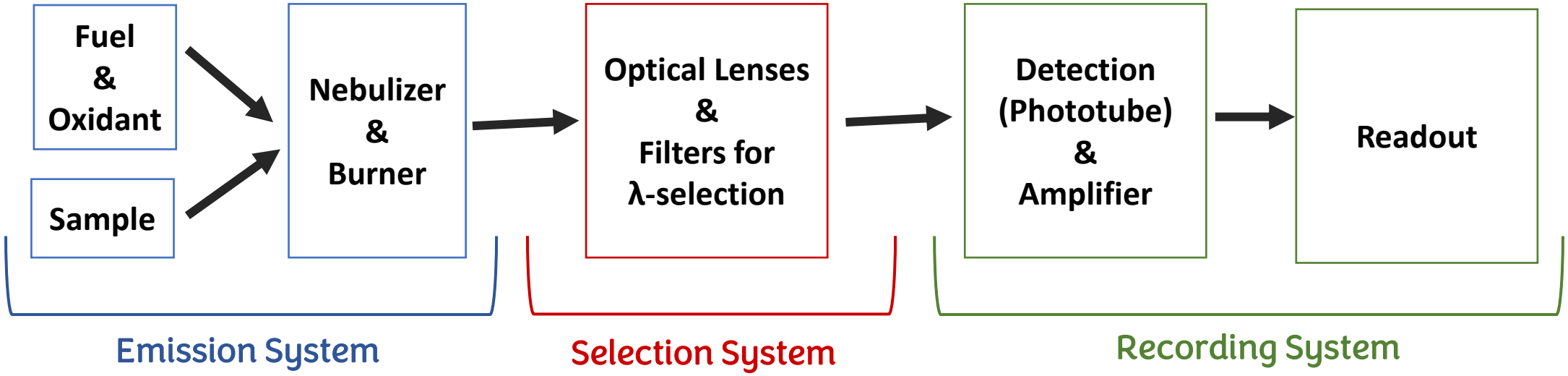




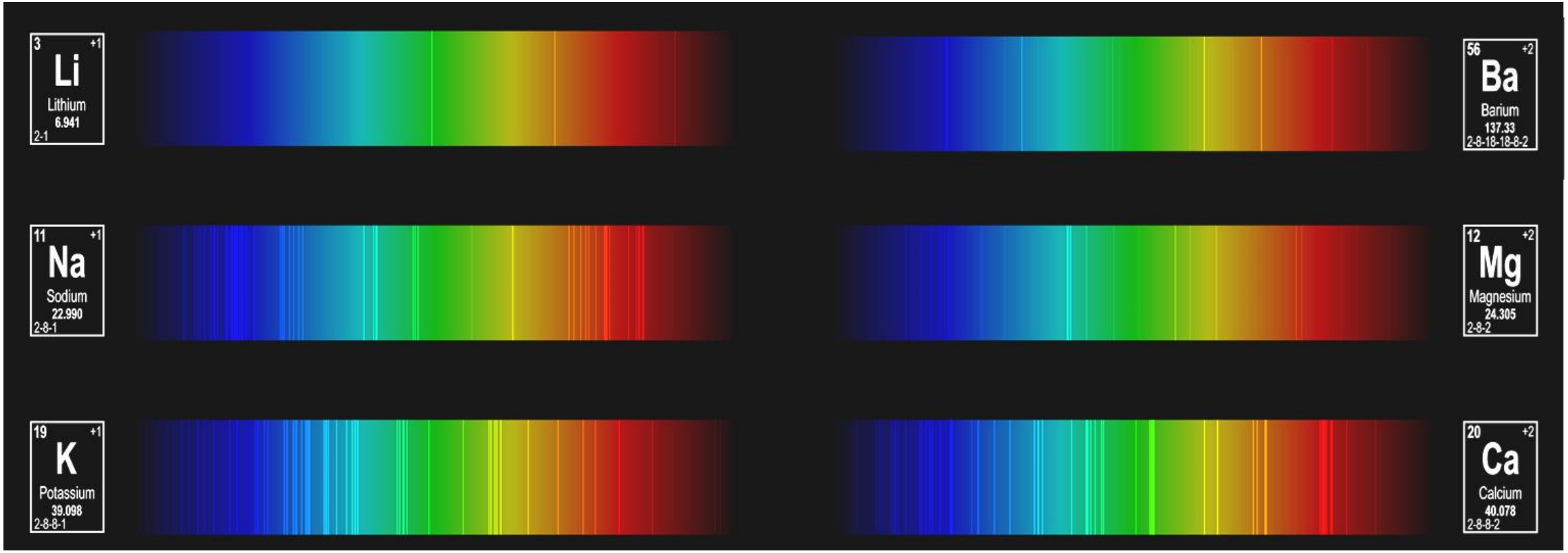
When a sample is atomized, a substantial fraction of the metallic constituents is reduced to gaseous atoms. Depending on the temperature of the flame a certain fraction of these atoms is ionized. Then, **electrons of the formed atoms are excited to upper state.** Light is **emitted at characteristic wavelengths** for each metal as the electron returns to the ground state.

Flame Photometer Systems

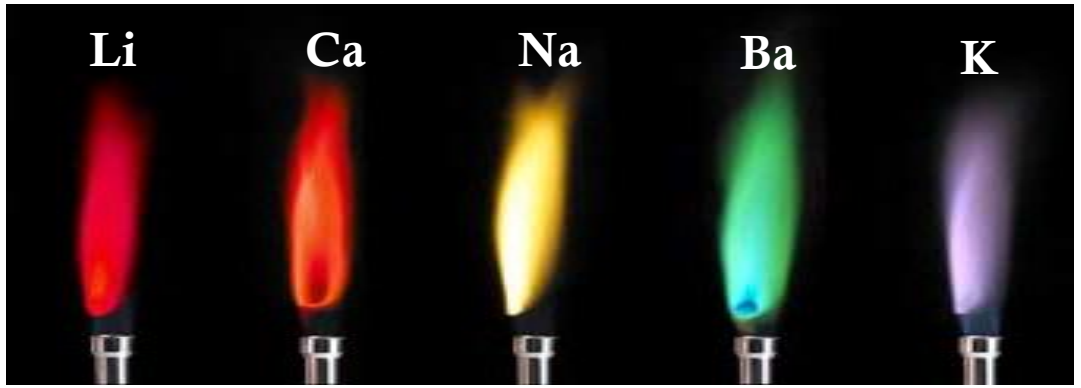
In flame photometer there are three fundamental systems which are **emission, λ-selection, and recording systems.**



1) Emission System: This consists of the flame, which is the source of emission.



Element	Emitted wavelength	Flame colour
Potassium (K)	766 nm	Violet
Lithium (Li)	670 nm	Red
Calcium (Ca)	622 nm	Orange
Sodium (Na)	589 nm	Yellow
Barium (Ba)	554 nm	Lime green



2) λ-Selection System: This includes the whole optical system of wavelength selection. In flame photometer the wavelength selector is a **filter**.

- The radiation emitted by the excited atoms is selected by using a filter which **transmits an emission line** of one of the elements **while absorbing the others.**

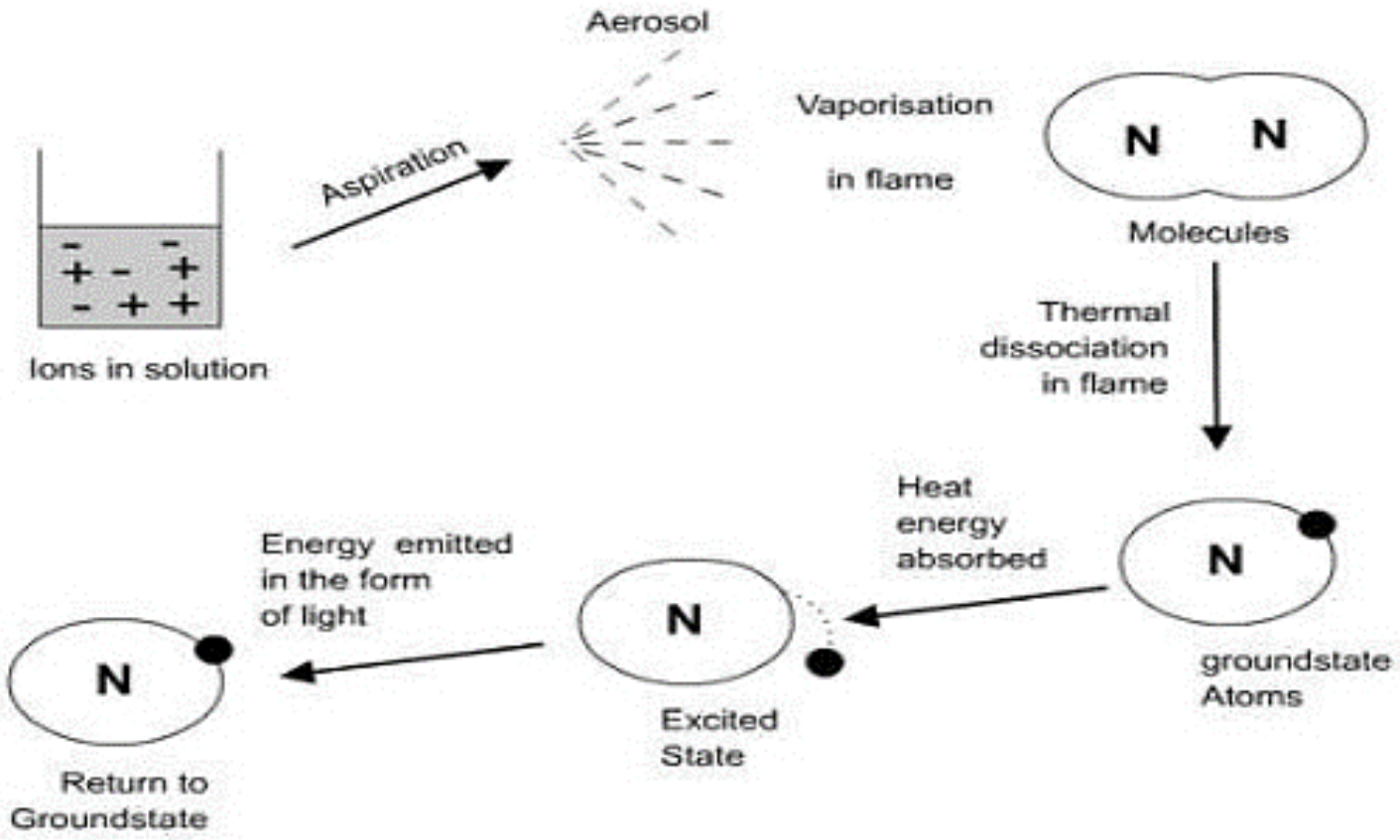
- **There are two types of filters**. These are **absorption** and **interference** filters. Absorption filters are restricted to visible region of the spectrum, but interference filters are used in UV, VIS and IR regions of the spectrum.

- **Absorption filters** are less expensive than the interference filters and they have been widely used for band selection in the visible region. These filters function by absorbing certain portions of the spectrum and transmitting the band of wavelengths belonging to the analyte element. The most common type consists of colored glasses.

- **Interference filters** rely on optical interference to provide relatively narrow bands of radiation. They consist of a transparent dielectric layer (CaF2 or MgF2) that occupies the space between two semi-transparent metallic films. This array is sandwiched between two plates of glass.

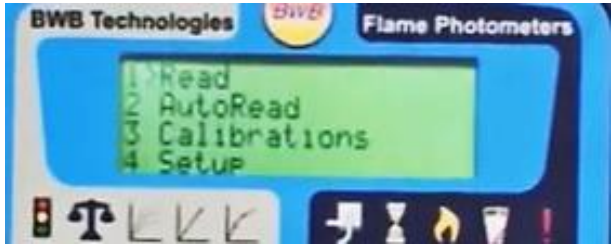
3) Recording System: This part consists of all the means of detection (*phototubes* or *photomultiplier tubes*), the *electronic devices of amplifying* and *electrical apparatus for measuring and direct recording*.

Flame Photometry process



The BWB XP Flame Photometer Instrument

The instrument that is used in this experiment is a **BWB XP model**, a five-channel digital flame photometer which is a low temperature, designed for the routine determinations of Na, K, Ca, Li, Ba. It is a direct reading digital instrument designed for use in clinical, industrial, and educational applications

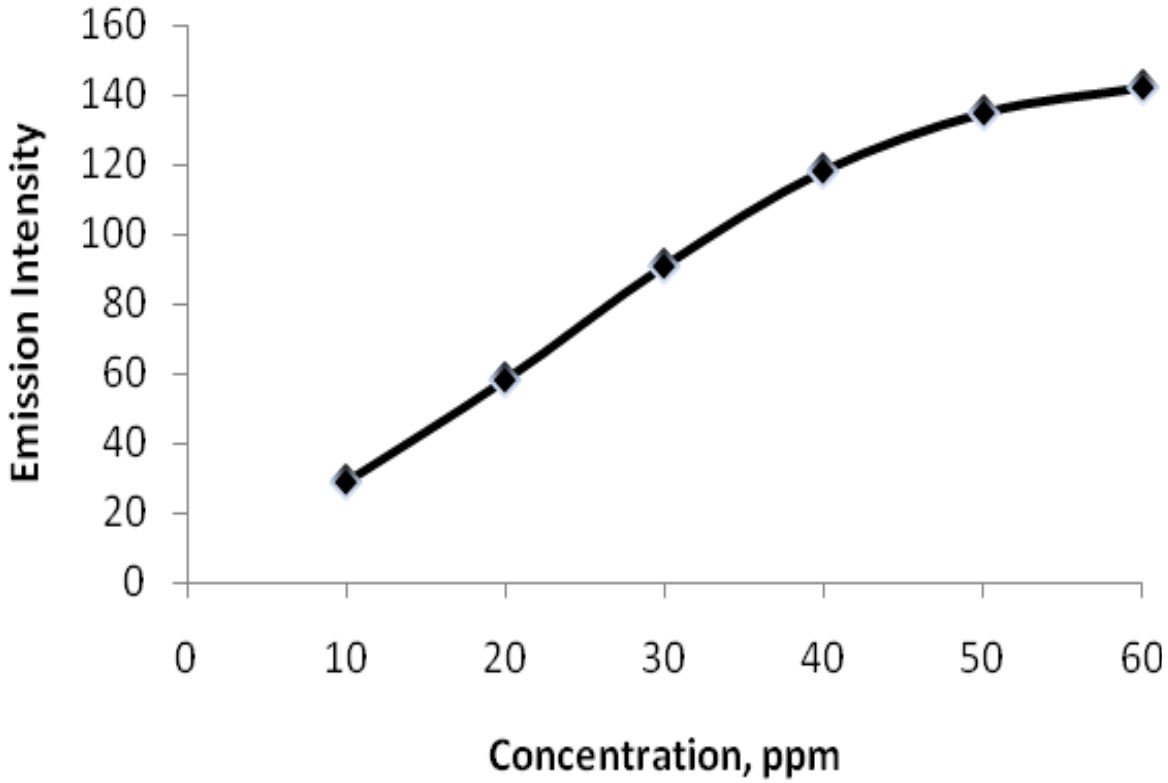

BWB Technologies
Flame Photometers
1 Read
2 AutoRead
3 Calibrations
4 Setup

BWB Technologies
BWB
Flame Photometers
Na~. 0.1 PPM
K~. 0.0 PPM
Li~. 0.0 PPM
Ca~. 0.7 PPM
read
calib
multi mode
single point
multi point
check u-tube
wait
flame out
over range
error
7
8
9
4
5
6
1
2
3
blank
0
.
accept
read
calibrate
multi mode
back STOP


read
calib
multi mode
single point
multi point
check u-tube
wait
flame out
over range
error

Calibration Curve

In flame photometry emitted light intensity from the flame is directly proportional to the concentration of the species being aspirated. The graph below shows that the direct relationship between **the emission** and **concentration** is true only at relatively low concentrations of mg/L level (up to 50 mg/L) → (ppm).



- If the samples being analyzed lie on the linear part of the curve, then user can take direct concentration readings from the digital display. However, if the concentration of sample is above the linear part, then user must dilute sample so that it lies on the linear part of the curve.
- A calibration curve is obtained by using standard solutions containing known concentrations of the elements to be determined.
- The concentration range covered by the calibration curve depends on the expected concentration so that the sample readings fall somewhere inside the calibration curve.
- Once the calibration curve has been plotted, the scale reading for the sample solution is compared with the curve to find the concentration.
- It is important to emphasize that each element has its own characteristic curve and separate curves must be constructed.

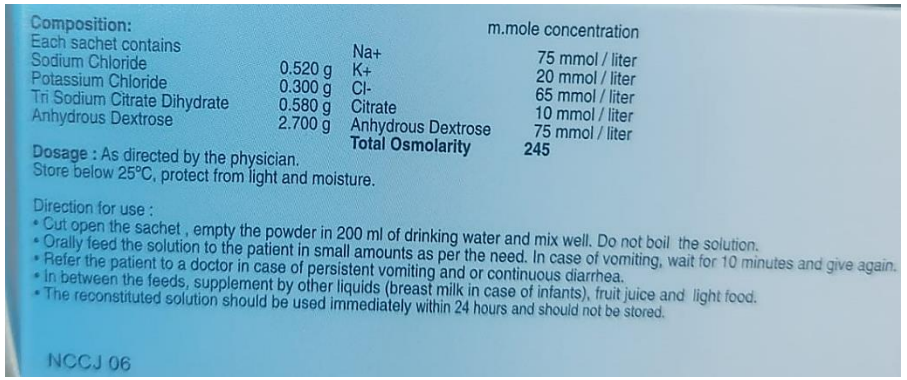

Practical Part

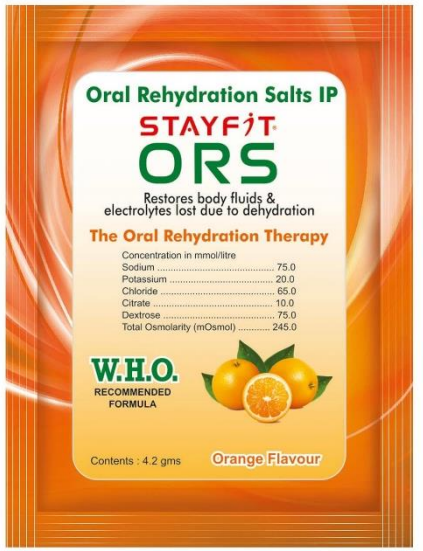
Objective

- Determination of **Na** and **K** assay in oral rehydration solution (ORS) as a pharmaceutical product using flame-photometer.
- Get familiar with the BWB Fame -photometer instrument and its usage.

Oral rehydration therapy is a type of fluid replacement used to prevent and treat dehydration, especially due to diarrhea. It involves drinking water with modest amounts of sugar and salts, **specifically <u>sodium</u> and <u>potassium</u>**.

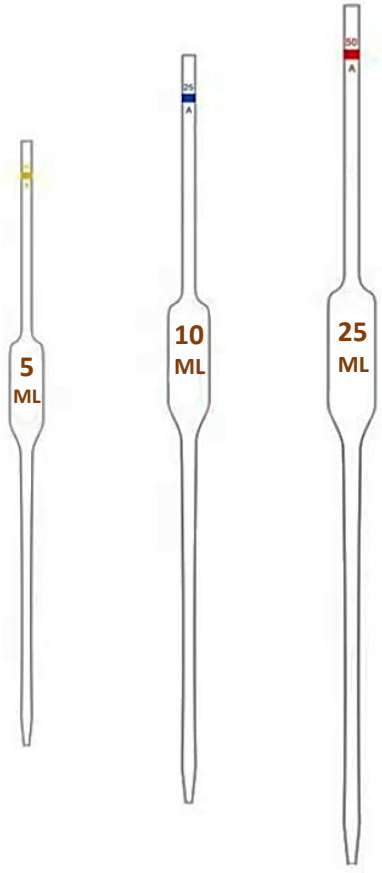
➢ Unlike other fluids, the ratio of the ingredients in an ORS matches what the body needs to recover from a diarrheal illness.





Glassware

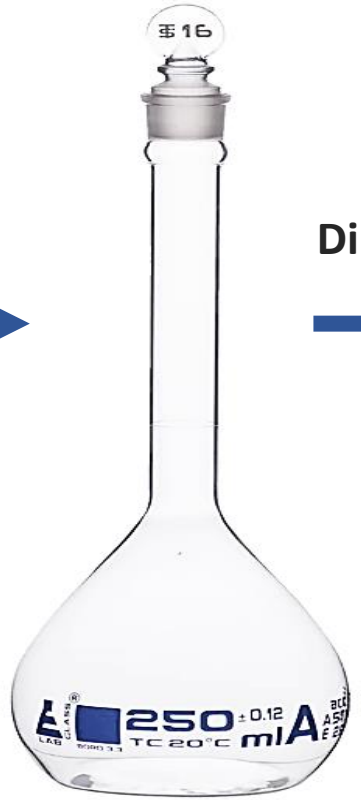
Each group will be supplied by the following glassware:



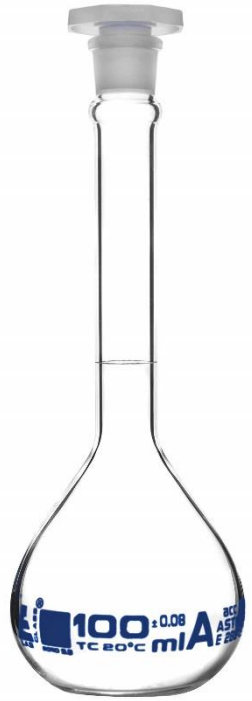
1 flask | 3 flasks | 1 flask | 1 flask

Procedure

Step 1: Preparing the ORS solution by dissolving the powder in one sachet in 250ml water. Then a dilution of 5ml to 100ml will be carried out.

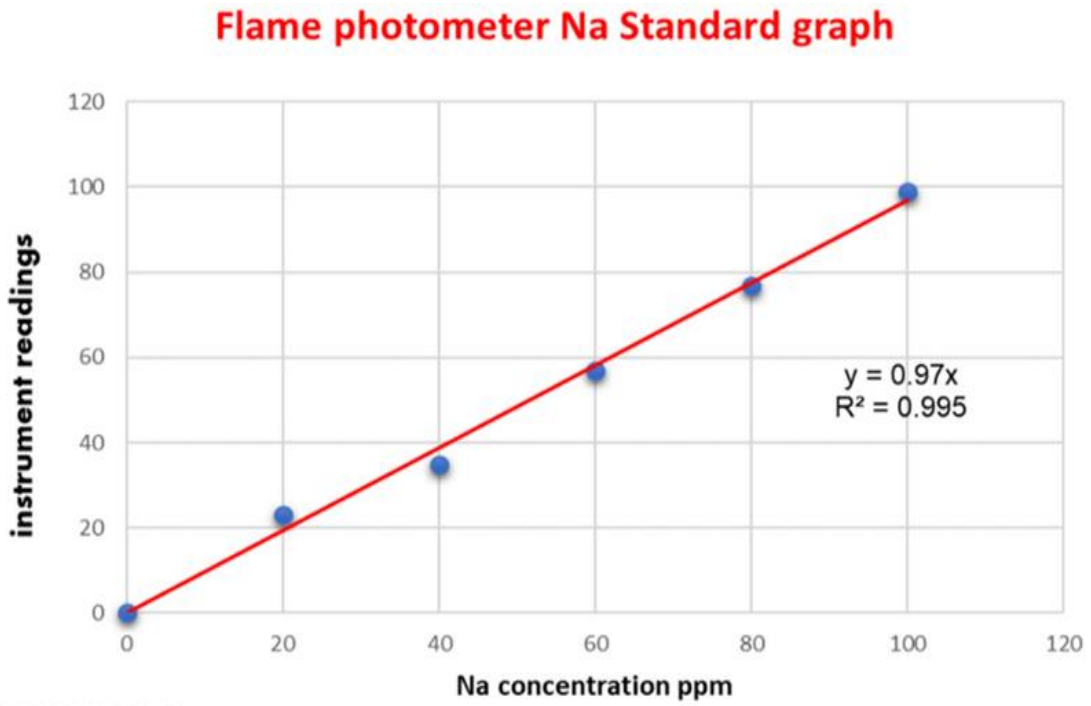


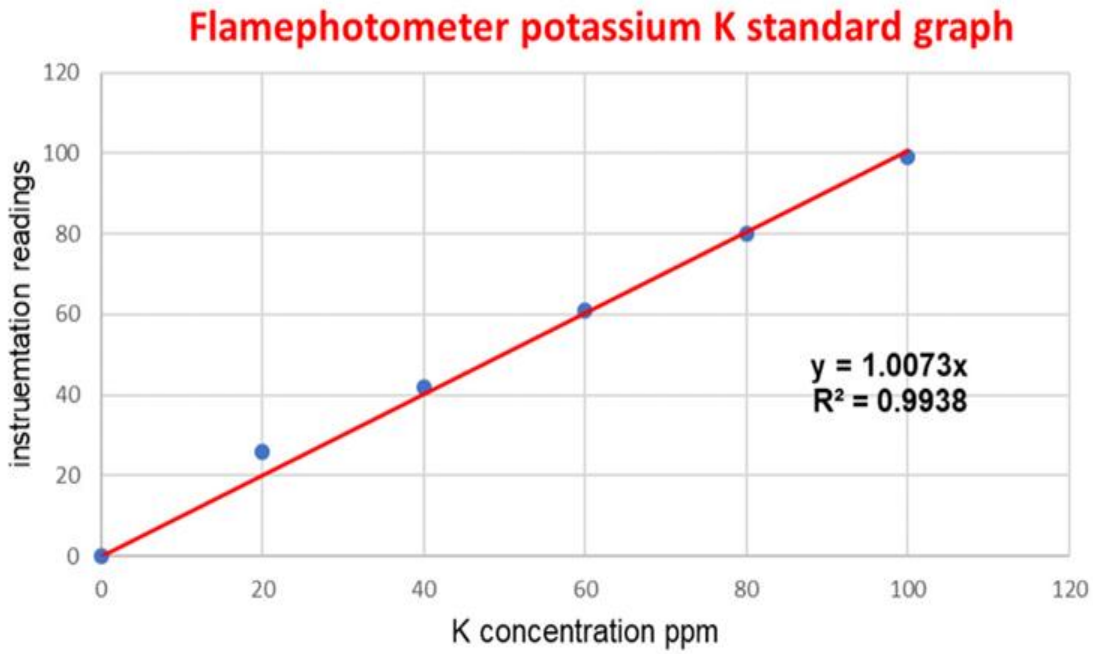
Dilute **5 ml** to **100 ml** D.W



Step 2: Preparing Standard Calibration curves

Preparing calibration curve for (**Na** and **K**) using NaCl and KCl salts. At first two stock solutions (250ml) with the concentration **200ppm** from both metals. Then carrying serial dilutions to obtain 4 standards with the concentrations (**100ppm, 80ppm, 40ppm**, and **20ppm**).





Weigh a certain amount of salt

200ppm

Up to 250 ml D.W

250ml

Stock solution

Dilute 25 ml from the stock solution to 50 ml D.W

50ml

100ppm

S1

Dilute 10 ml from the stock solution to 25 ml D.W

25ml

80ppm

S2

Dilute 10 ml from the stock solution to 50 ml D.W

50ml

40ppm

S3

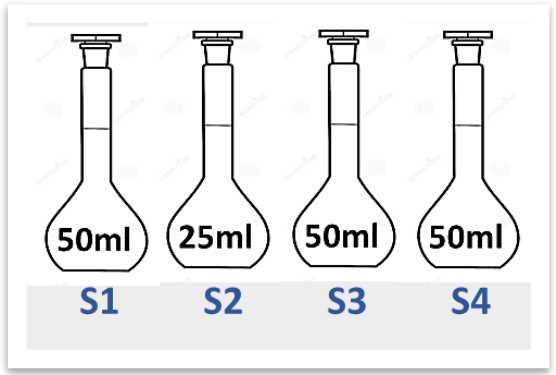
Serial Dilution
(2 steps dilution)

Then Dilute 25 ml from standard 3 (S3) to 50 ml D.W

50ml

20ppm

S4



Step 3: Take the standards to the flame-photometer to be measured

How to prepare a stock solution in ppm?

Example: Prepare **250ml** solution of Ca with a concentration of **100ppm**

$$ppm = mg/L$$

100ppm = 100mg/L → (250ml= 0.25L)

100mg → 1 L
??Ca → 0.25L
= 25mg (0.025g) of Ca per 250ml

How much should we weight of $CaCL_2$??

Equivalent weight

0.025g Ca in 250ml → 100ppm Ca

$CaCl_2 \rightarrow Ca^{+2} + 2\ Cl^{-1}$

1 mol of Ca = 1 mol of $CaCl_2$

Mol = weight / MWT

0.025g / 40.078 = ??g /110.98

0.00062 = ??g /110.98

Weight of $CaCl_2$= 0.06881 g = 68.81 mg

❖ Molecular weight for

(Ca= 40.078 g/mol)

(Cl= 35.453 g/mol)

($CaCl_2$= 110.98 g/mol)

Sodium from NaCl

250ml of Na^+ in a concentration of (200 ppm)

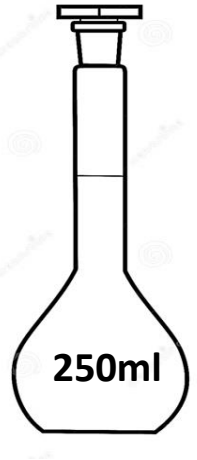
ppm = mg/L →

Na^+ ??

Equivalent Wt. →

NaCl ??

200 ppm of Na+



❖ Molecular weight for

(Na= 22.990 g/mol)

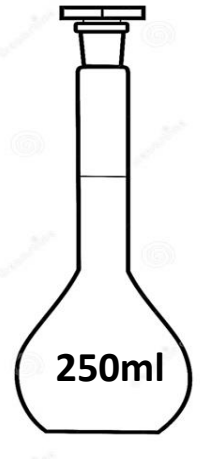
(Cl= 35.453 g/mol)

(NaCl= 58.44 g/mol)

Potassium from KaCl

250ml of K^+ in a concentration of (200 ppm) → ppm = mg/L → K^+ ?? → Equivalent Wt. → KCl ??

200 ppm of K+



❖ Molecular weight for

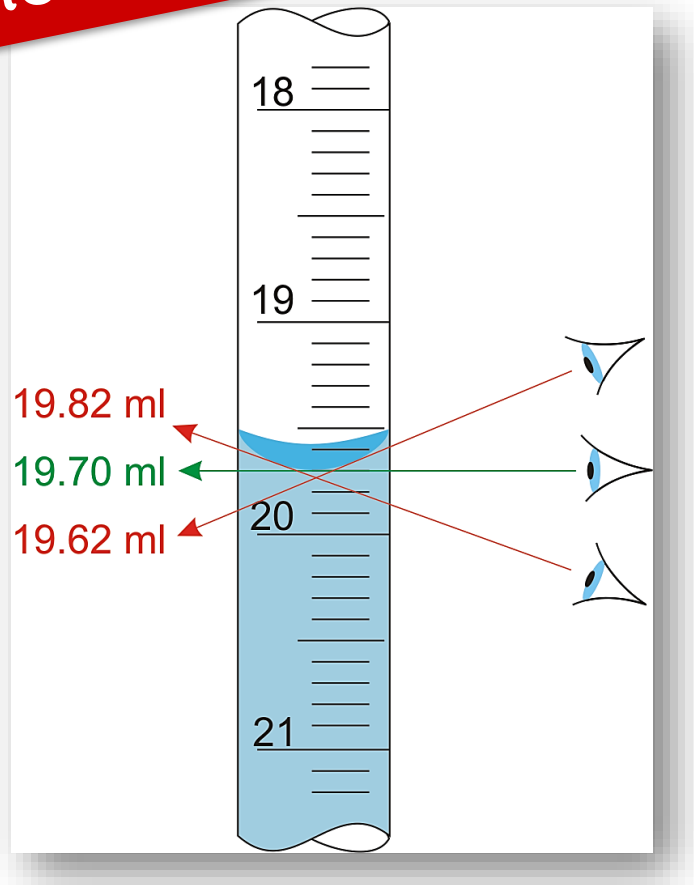
(K= 39.0983 g/mol)

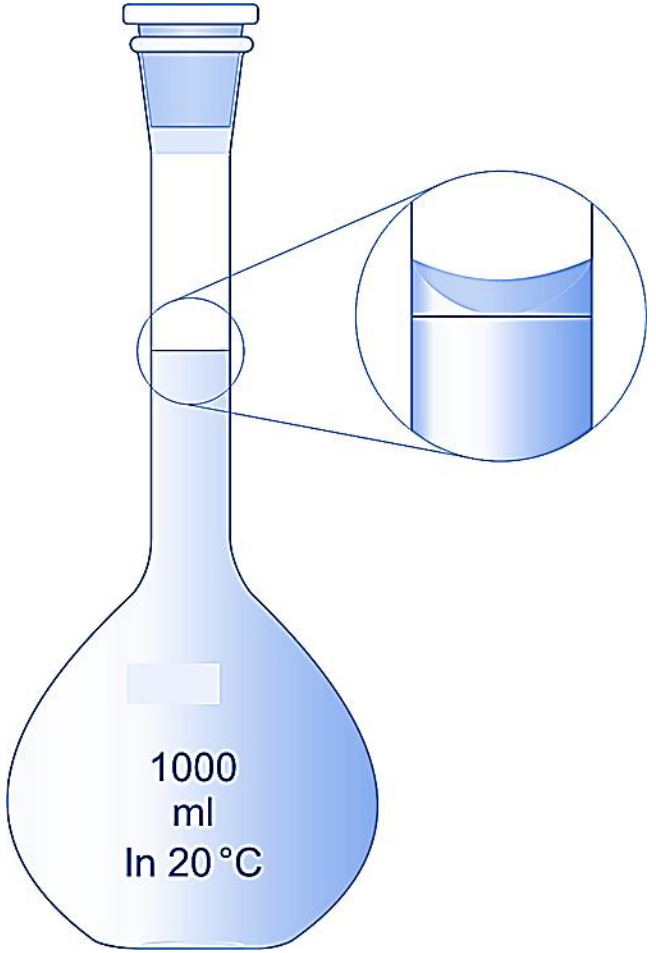
(Cl= 35.453 g/mol)

(KCl= 74.55 g/mol)

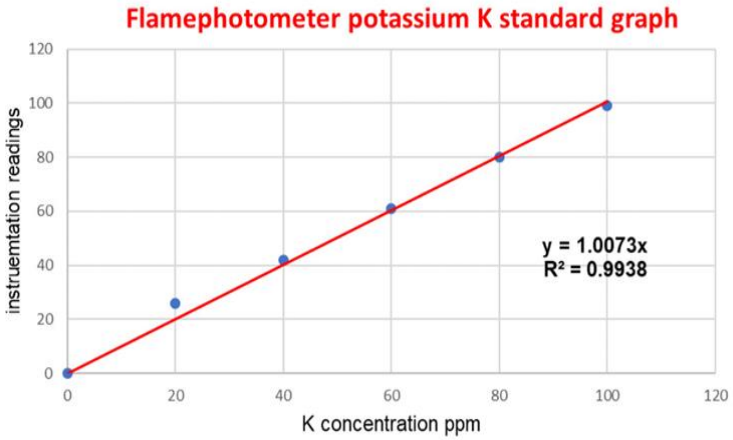
How to Obtain the accurate volume (liquid meniscus)

Remember





- ✓ *After finishing measurements, draw calibration curve then calculate the concentration for your Na and K metals in the sample(D.f).*
- ✓ *Determine the ORS sample Assay for both (Na and K) and discuss wither you are with or against its claim.*
  - ➢ *To determine the % assay, use the following formula:*



	m.mole concentration
Na+	75 mmol / liter
K+	20 mmol / liter
Cl-	65 mmol / liter
Citrate	10 mmol / liter
Anhydrous Dextrose	75 mmol / liter
Total Osmolarity	**245**

$$\% \text{ Assay} = \frac{\text{Actual Concentration}}{\text{Theoretical Concentration}} \times 100\%$$

MSc. Farah Hudaib