

Experiment 7

Flame Photometry

Ayaayyash

INTRODUCTION

زي ماينعرف ال atomic spectroscopy
بتدرسلي العلاقة بين ال radiation
matter الي بتظهر على اساس

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:

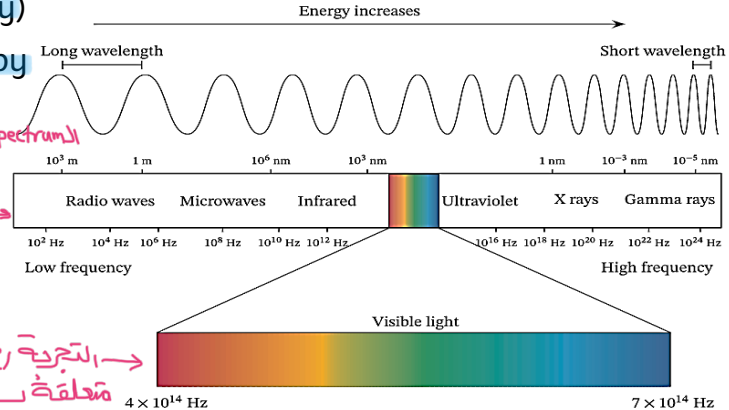
1. Absorption spectroscopy (UV-Vis)
2. Emission spectroscopy (Flame photometry)
3. Luminescence / fluorescence spectroscopy

يعني بصريتا
excitation or
atomization
عن طريق ال flame

① UV/VIS - 15 Region
② X-ray

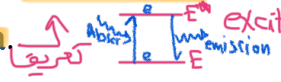
photon s concentration intensity علاقة بين spectrum
بعضني زي علاقة بين

different
radiation →



→ التجربة رح تكون
متعلقة بـ
light

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**.



تفاعل المادة مع الضوء يؤدي إلى انتقال إلكترون من الحالة الأرضية إلى حالة مثارة

➤ 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.

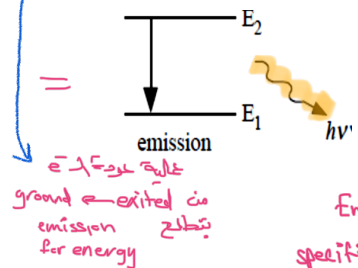
وجود إلكترون في الحالة المثارة غير مستقر ويحاول الرجوع إلى الحالة الأرضية المنخفضة الطاقة
excited state
in stable form
ground state

انتقال إلكترون من State إلى State آخر يعتمد أنه يكتسب الطاقة
انتقال إلكترون من State إلى State آخر
بمقدار الطاقة



SPECIFIC TO THAT PARTICULAR METAL ION

لأنه يعني ربح أو فقد إلكترون من المدار الخارجي للمعدن
لأنه سوف الضوء الذي ينتج عن احتراقه
لهذا specific



عند تسخين المعادن وتكون specific
Emission
ويعطي light different ويكون specific

وهو key mark حتى انزل
ال metal عن بعضها

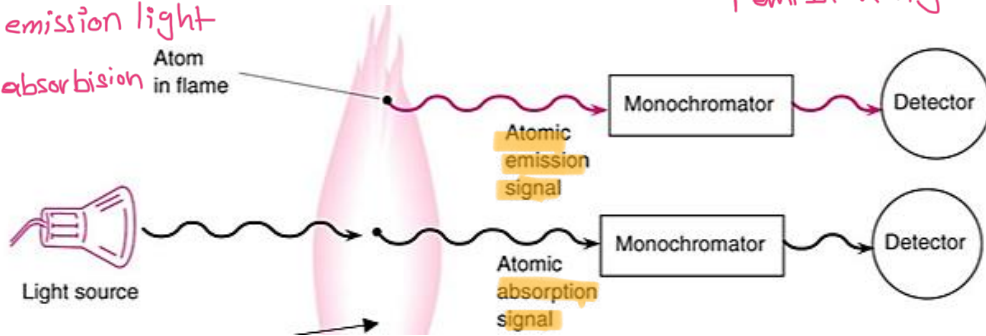
احتمال تقييس

➤ 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.

↑ concentration
↑ emission light

Flame → emission light

lamp → absorption

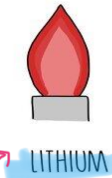


DEFINITION

معناه انه تحديد العنصر الي سهل انه اهيجها
وانقلها لل exited level عن طريق ال flame

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**.

mette | في طريقة حرق اذ
الي عندي بخط اذ ions على
Bunsen Burner
وسوف اضعو الي
حرق تطلع وبيرف المادة بس طريقة
في effect ل mixture of ions
بيجي دور
اي يقسي
وجد اذ
Mixture
of ions
لا تفرق
فلتر يرجع لاني
ion



LITHIUM



SODIUM



POTASSIUM



CALCIUM



BARIUM

Na
K
Li } مجموعة
الاولى

Ca
Ba } مجموعة
الثانية

advantage of flame

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**).

qualitative → emitted
quantitative → intensity

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. Oxidant can generally be air, oxygen or ^{pure} nitrous oxide (N_2O). The flame temperature depends on fuel to oxidant ratio.

كلما زاد تركيزه كلما كانت pure أكثر كل ما كان حرارة flame أكثر
وكلما اختلفت ال ratio بين ال fuel وال oxidant رحت تختلف الحرارة



❖ 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.

mixed قبل عملية الاحتراق

يشكل منتظم وما
منه عشوائية

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.

mixed أثناء عملية الاحتراق

على شكل موج أو أمواج

Flame is formed by two components: **fuel** and **oxidant**. ①

Temperature of the flame changes depending on the **fuel** and **oxidant** types and their proportions. ②

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.

مع اختلاف ratio يتغير درجة الحرارة

FUEL	OXIDANT	TEMPERATURE, °C
Natural Gas	Air	1700-1900
② Natural Gas	Oxygen	2700-2800
Hydrogen	Air	2000-2100
③ Hydrogen	Oxygen	2550-2700
Acetylene	Air	2100-2400
① Acetylene	Oxygen	3050-3150
Acetylene	Nitrous Oxide	2600-2800

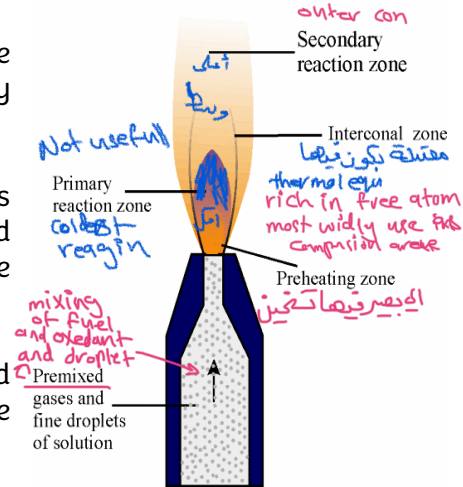
فأنا لندرم اعرفا إنه ال-oxidant هو ال flame

oxygen > Nitrous oxide > Air

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 (secondary reaction zone)** - In this zone, the products of the combustion processes are burnt to stable molecular species by the surrounding air.



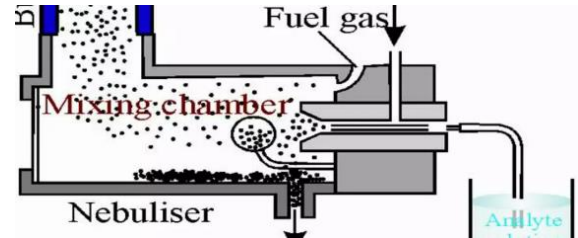
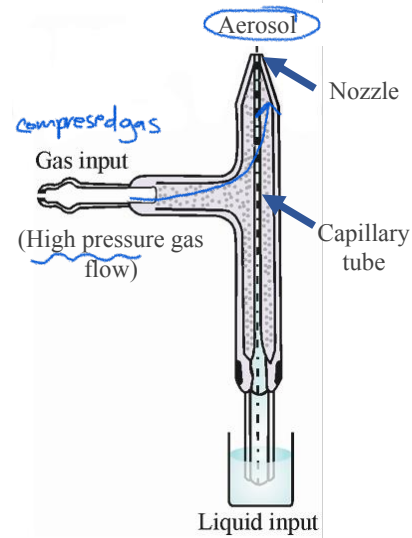
TECHNIQUE

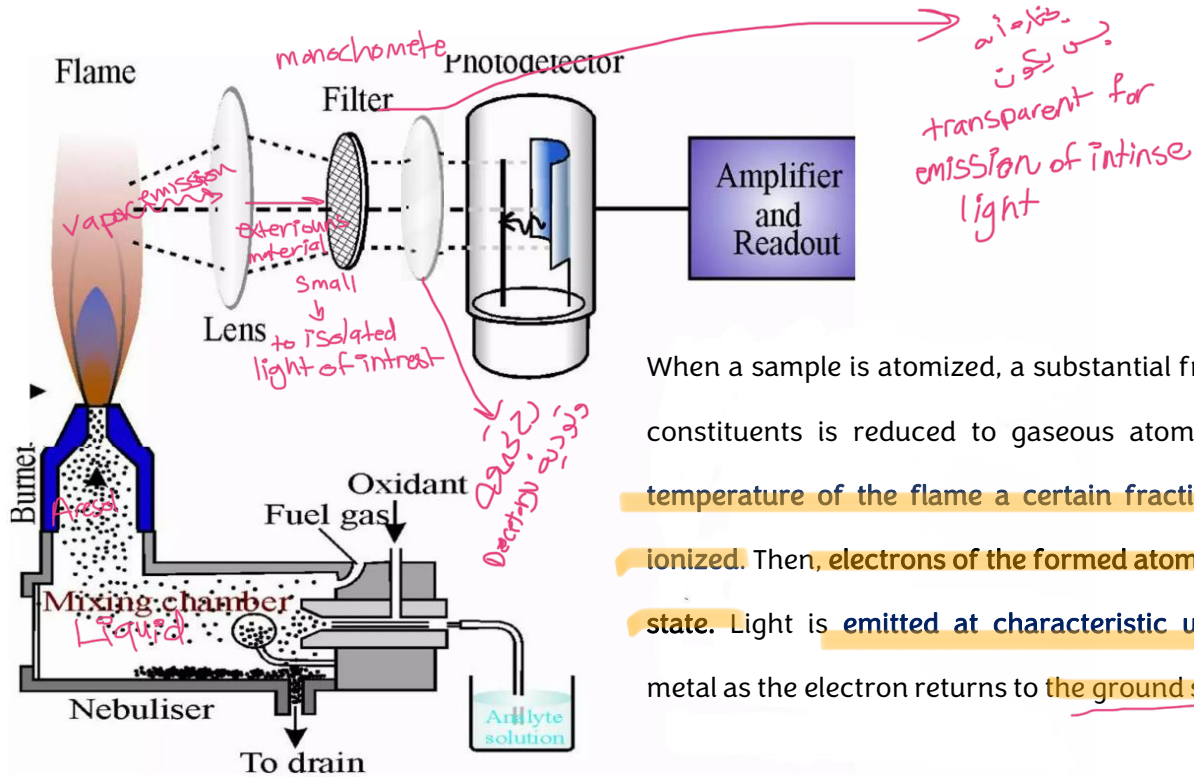
In this technique, first aerosols are formed from sample solution by a jet of compressed gas. This process is called **nebulization**. Then the flow of the gas carries the aerosols into a flame where atomization takes place. **Atomization** is the conversion of sample aerosols into an atomic vapor by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

الى تبخير فيه تحول ل sample
Aerosol ← liquid -
نابض

في Atomization في flame
نابض

Vapor ← Aerosol تحول
نابض

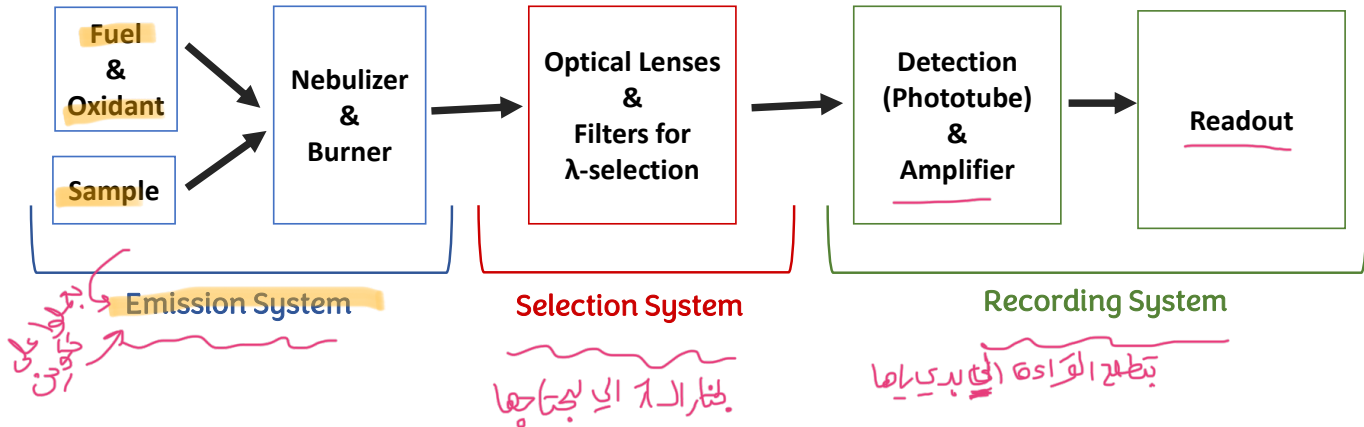




When a sample is atomized, a substantial fraction of the metallic constituents is reduced to gaseous atoms. Depending on the temperature of the flame a certain fraction of these atoms is ionized. Then, electrons of the formed atoms are excited to upper state. Light is emitted at characteristic wavelengths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

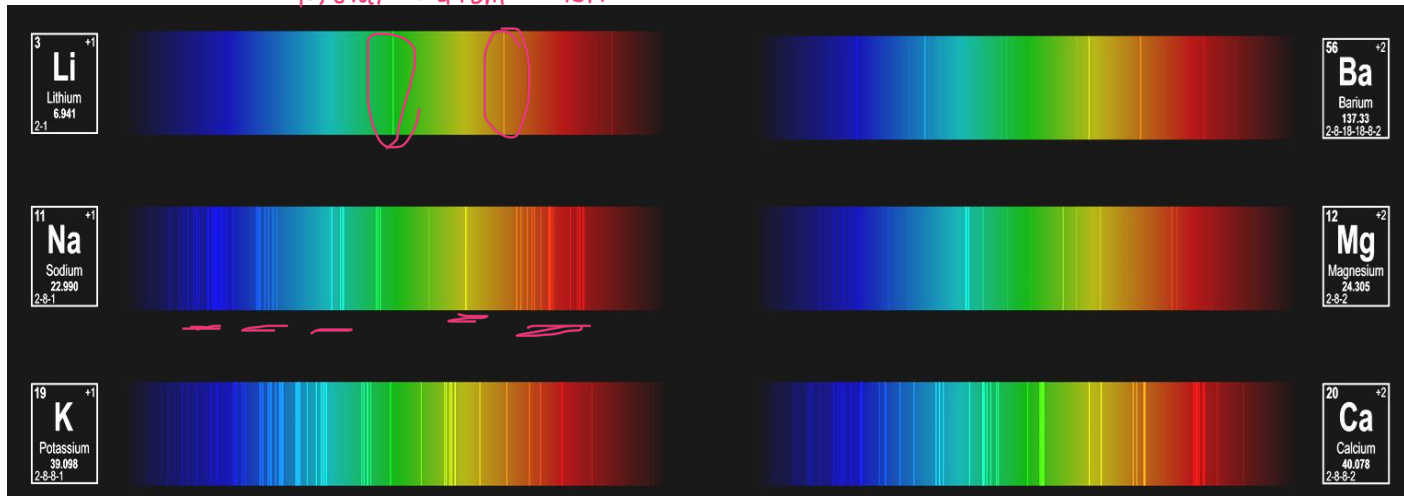
Flame Photometer Systems

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.

metal $\rightarrow$ atom $\rightarrow$ ion



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 



يفلتر كل الأطوال الموجية
التي تخرج من Flame ويترك
العينة التي مطلوبة ويبقى الباقي

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. more sensitive, more accurate, more expensive.

- **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 (CaF_2 or MgF_2) that occupies the space between two semi-transparent metallic films. This array is sandwiched between two plates of glass.

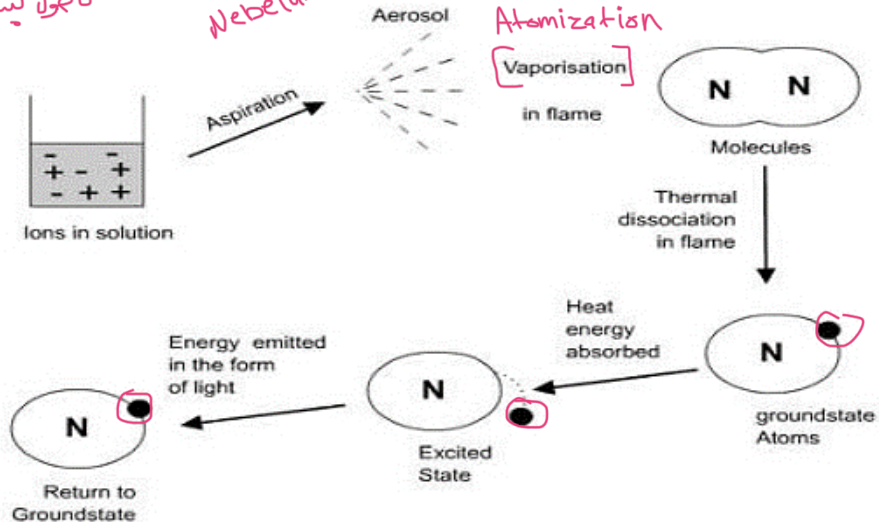
3) **Recording System**: This part consists of all the means of detection (**phototubes or photomultiplier tubes**), the *electronic devices of amplifying and electrical apparatus for measuring and direct recording.*

عملیاتی پیمانہ

Nebulization

Atomization

Vaporisation
in flame

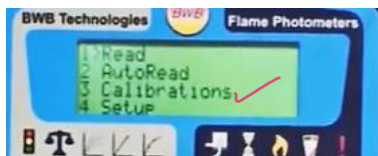


Flame Photometry process

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

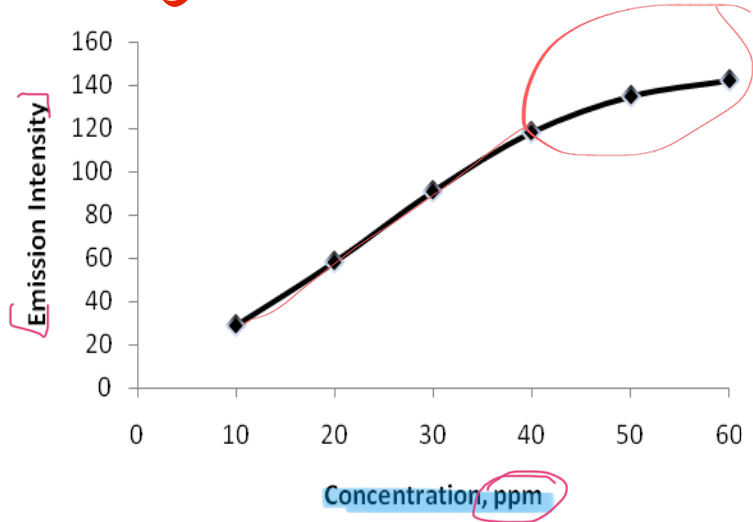




Calibration Curve

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).

عالي لما
Conc deviation



انا دائما بال calibration range دائما باخذ العينات ونحضر يغطي تركيز الانون يعني انا عارفه انه الانون بتركيز معين اذا كان الانون تركيزه او الانتينستي طلعت تاعته ضمن الخط تبع قبل شوية
خذ direct reading for unknown conc from calibration equation

❖ 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.

لازم التراكيز ماتزيد عن ١٠٠٠ ppm mg/l

❖ 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.

A laboratory setting featuring a blue flame photometer on the left with a digital display showing '25.00ppm', '1.00ppm', '3.00ppm', and '12.00ppm'. To its right is a tall grey instrument with a metal top and warning labels. In the foreground, several glass vessels containing dark liquids are arranged: two Erlenmeyer flasks, a round-bottom flask, a large Erlenmeyer flask, and a beaker. A white rack filled with test tubes is visible on the right. The background shows a blurred laboratory environment with a microscope and a computer monitor.

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 Flame -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 sodium and potassium**.

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

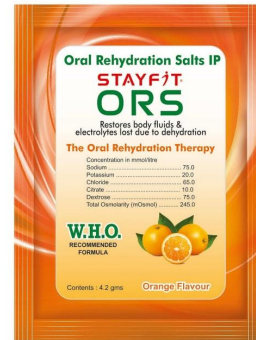
Composition:			m.mole concentration
Each sachet contains			
Sodium Chloride	0.520 g	Na ⁺	75 mmol / liter
Potassium Chloride	0.300 g	K ⁺	20 mmol / liter
Tn Sodium Citrate Dihydrate	0.580 g	Cl ⁻	65 mmol / liter
Anhydrous Dextrose	2.700 g	Citrate	10 mmol / liter
		Anhydrous Dextrose	75 mmol / liter
		Total Osmolality	245

Dosage : As directed by the physician.
Store below 25°C, protect from light and moisture.

Direction for use :
• Cut open the sachet , empty the powder in 200 ml of drinking water and mix well. Do not boil the solution.
• Orally feed the solution to the patient in small amounts as per the need. In case of vomiting, wait for 10 minutes and give again.
• Refer the patient to a doctor in case of persistent vomiting and/or continuous diarrhea.
• In between the feeds, supplement by other liquids (breast milk in case of infants), fruit juice and light food.
• The reconstituted solution should be used immediately within 24 hours and should not be stored.

NCCJ 06

Reference



Glassware

Each group will be supplied by the following glassware:



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.

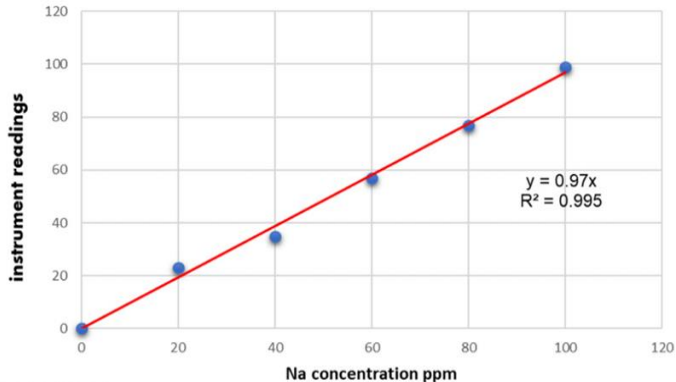


Step 2: Preparing Standard Calibration curves

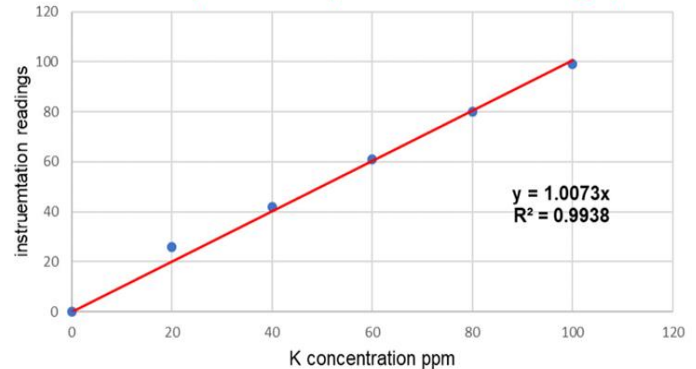
Preparing calibration curve for (Na) and (K) using NaCl and KCl salts. At first two stock solutions (250ml) with the concentration **200ppm** from both metals. Then carrying serial dilutions to obtain 4 standards with the concentrations (**100ppm**, **80ppm**, **40ppm**, and **20ppm**).



Flame photometer Na Standard graph



Flamephotometer potassium K standard graph



Weigh a certain amount of salt



200ppm

Up to 250 ml D.W



Stock solution

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



S1

100ppm

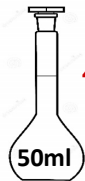
Dilute 10 ml from the stock solution to 25 ml D.W



S2

80ppm

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



S3

40ppm



S1

S2

S3

S4

Serial Dilution
(2 steps dilution)

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



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 ^{mg/L}

$$\text{ppm} = \text{mg/L}$$

$$100\text{ppm} = 100\text{mg/L} \rightarrow (250\text{ml} = 0.25\text{L})$$

$$100\text{mg} \rightarrow 1\text{L}$$

$$??\text{Ca} \rightarrow 0.25\text{L}$$

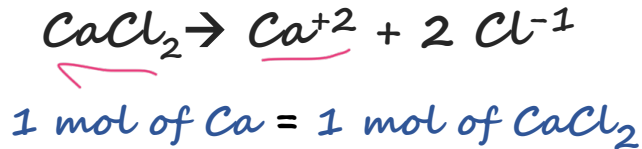
$$= 25\text{mg} (0.025\text{g}) \text{ of Ca per } 250\text{ml}$$

How much should we weight of CaCl₂??

$$\begin{array}{l} 100\text{mg} \rightarrow 1\text{L} \\ ?? \rightarrow 0.25\text{L} \\ = 25\text{mg} \rightarrow 0.025\text{g of Ca} \\ \text{per } 250\text{ml} \end{array}$$



****Equivalent weight****



$$\text{Mol} = \text{weight} / \text{MWT}$$

$$0.025\text{g} / 40.078 = ??\text{g} / 110.98$$

$$0.00062 = ??\text{g} / 110.98$$

$$\text{Weight of } \text{CaCl}_2 = 0.06881\text{ g} = \underline{68.81\text{ mg}}$$

0.025g Ca in 250ml → 100ppm Ca

❖ Molecular weight for
(Ca= 40.078 g/mol)
(Cl= 35.453 g/mol)
(CaCl₂= 110.98 g/mol)

$$\text{Mol}_{\text{Ca}^{+2}} = \frac{0.025}{40.078} = 0.00062$$

$$\frac{\text{g}}{110.98} = 0.00062$$

Sodium from NaCl

ppm $\rightarrow$ — mg $\rightarrow$ 1L
 $\rightarrow$ — بالوزن
 ① كم كمية Na⁺

② Equivalent wt
 1 mole of Na⁺ = 1 mole of NaCl

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

ppm = mg/L

Na⁺ ??

Equivalent Wt.

NaCl ??



200 mg $\rightarrow$ 1L
 ? $\rightarrow$.25 L

50 mg = .050g ♦ Molecular weight for

(Na= 22.990 g/mol)

(Cl= 35.453 g/mol)

(NaCl= 58.44 g/mol)

$$n = \frac{g}{M \cdot n} = \frac{0.050g}{22.99}$$

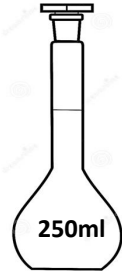
$$= 2.18 \times 10^{-3} \text{ mol}$$

$$2.18 \times 10^{-3} \times 58.44 =$$

0.127 g of NaCl

127 mg of NaCl

200 ppm of Na⁺



Potassium from KCl

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

ppm = mg/L

$K^+ ??$

Equivalent Wt.

$KCl ??$



❖ Molecular weight for
(K= 39.0983 g/mol)
(Cl= 35.453 g/mol)
(KCl= 74.55 g/mol)

$$200 \text{ mg} \rightarrow 1 \text{ L}$$

$$\frac{?}{?} \rightarrow 0.25 \text{ L}$$

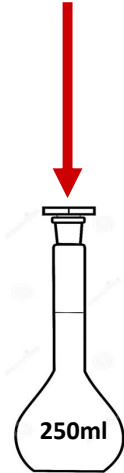
$$50 \text{ mg } K^+ = 0.05 \text{ g } K^+$$

$$\frac{0.05}{39.0983} = 1.278 \times 10^{-3} \text{ mole } K^+$$

$$* 1 \text{ mole } K^+ = 1 \text{ mole } KCl$$

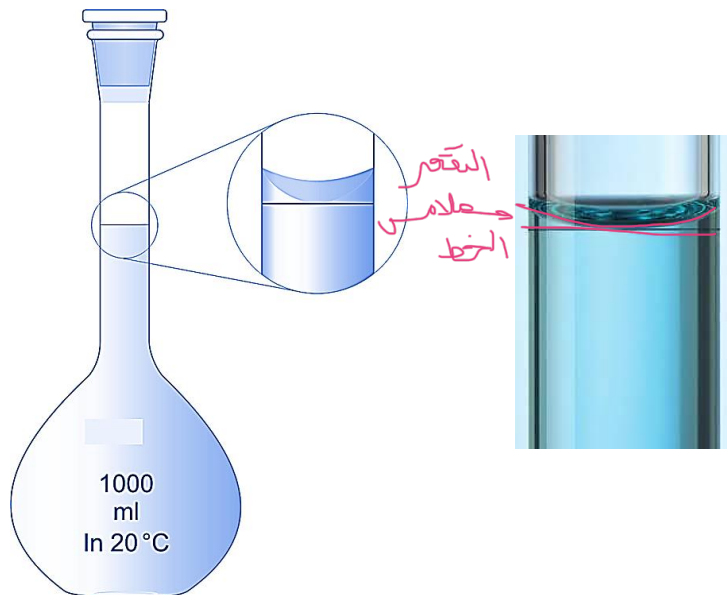
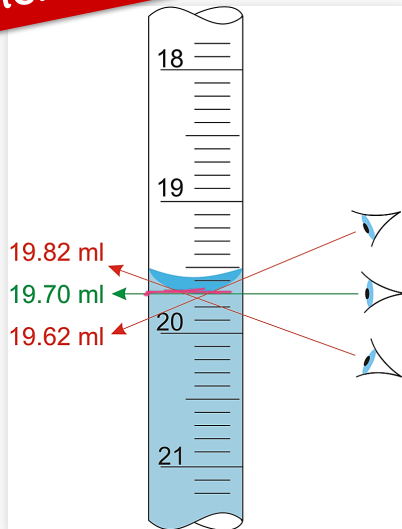
$$10^{-3} * 1.278 * 74.55 = 200 \text{ ppm of } K^+$$

$$0.09533 \text{ g} = 95.33 \text{ mg of } KCl$$



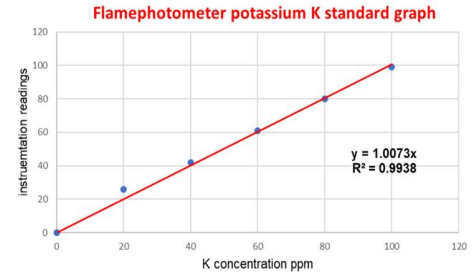
How to Obtain the accurate volume (liquid meniscus)

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:

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



	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



GOOD
LUCK!

MSc. Farah Hudaib