

Lecture (4)

Flame emission spectroscopy

Submitted by:

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Introduction

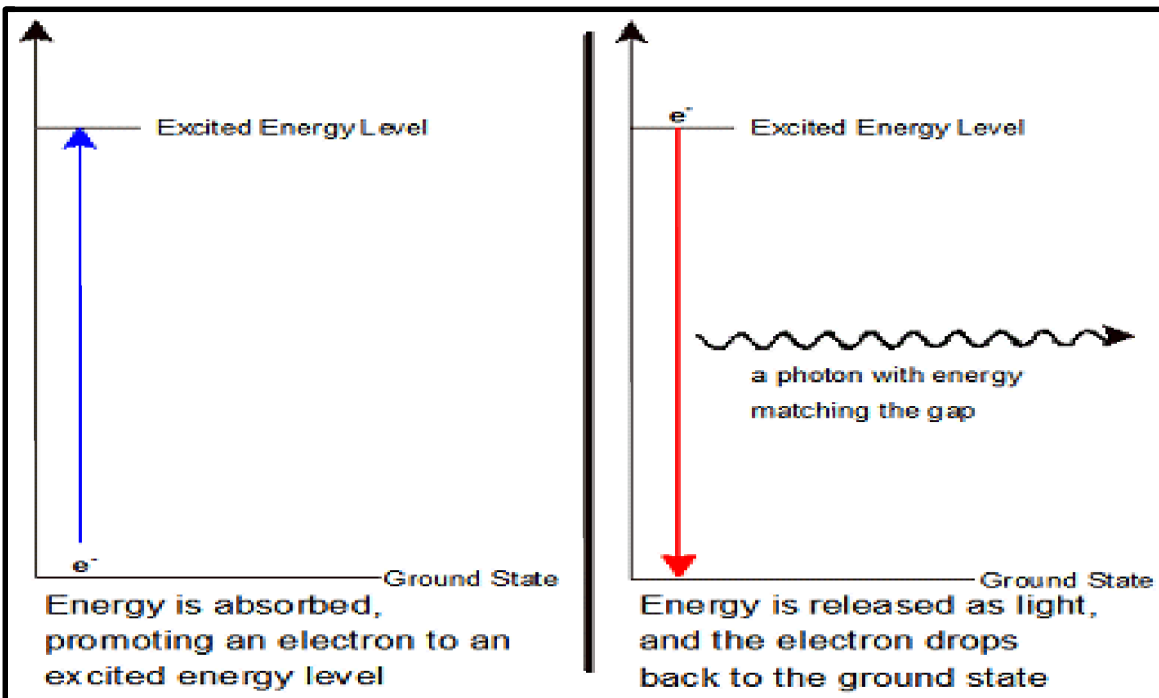
What is the Flame emission spectroscopy?

It is branch of spectrometry in which species examined in spectrometer as a form of atoms .

- *flame spectrometer is instrument used in inorganic chemical analysis to determine the concentration of **certain metal ions among them Na, K, Ca, Li** .*
- *Flame photometry is based on measurement of intensity if the light emitted when a metal is introduced into the flame*
- *The wavelength of colour tells what the element is (qualitative).*
- *The colour's intensity tells about how much of element present (quantitative)*

What 's the concept of flame emission spectroscopy ?

- *A sample of a material (analyte) is brought into the flame as either a gas, sprayed solution, or directly inserted into the flame by use of a small loop of wire, usually platinum. The heat from the flame evaporates the solvent and breaks chemical bonds to create free atoms.*
- *Gaseous molecule dissociated to give neutral atoms which can be unstable by thermal energy .*
- *The thermal energy also excites the atoms into excited electronic states that subsequently emit light when they return to the ground electronic state.*
- *Each element emits light at a characteristic wavelength .*



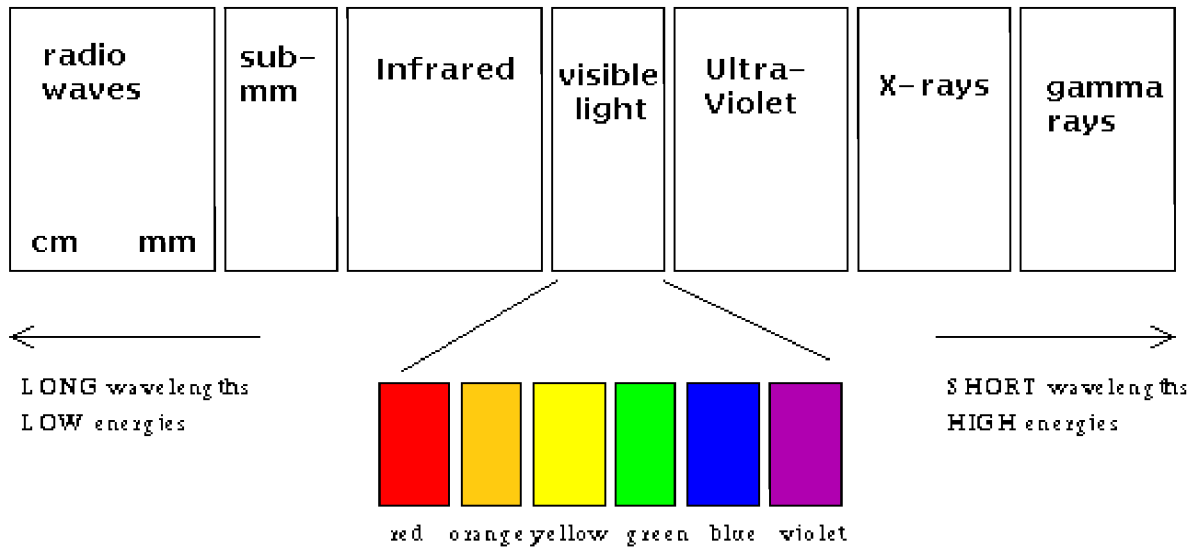
- Under constant and specific and controlled condition , the intensity of the light is proportional to the number of excited atoms which is proportional to the total number of atoms in the flame the sample concentration .

The number of atoms of an element excited by the flame depends on :

- 1- Flame temperature .
- 2- The energy difference between the excited and ground states.

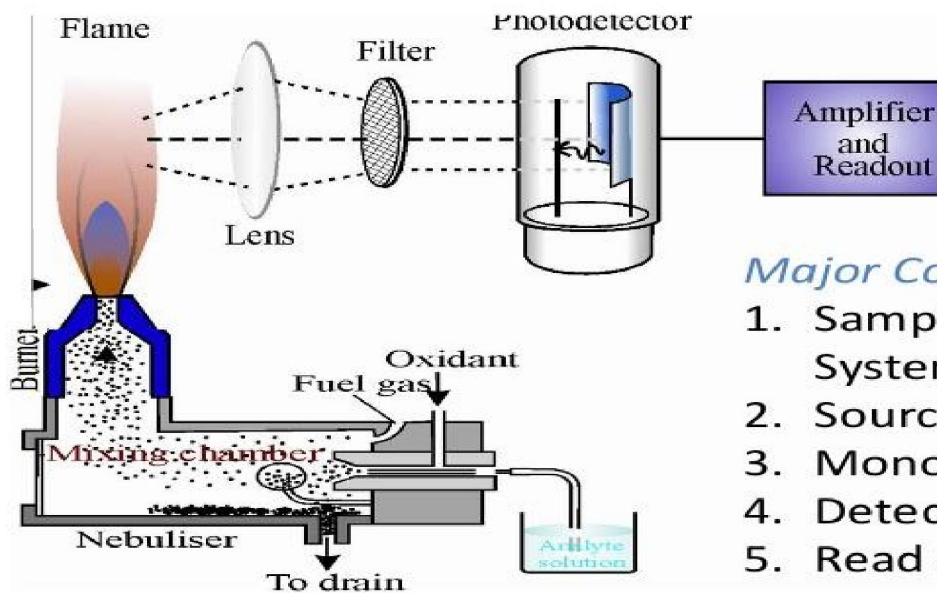
Various metals emit a specific colour of light when heated

ELEMENT	EMISSION WAVELENGTH(nm)	FLAME COLOUR
Sodium(Na)	589	yellow
Potassium(K)	766	violet
Barium(Ba)	554	Lime green
Calcium(Ca)	662	orange
Lithium(Li)	670	Red



INSTRUMENT FOR FLAME EMISSION

Schematic Representation of the Flame Photometer



Major Components:

1. Sample Delivery System
2. Source
3. Monochromator
4. Detector
5. Read out device

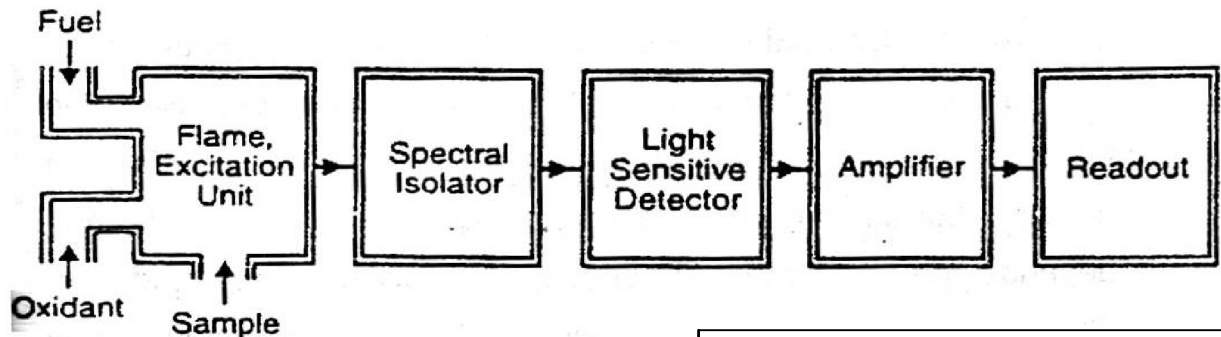
What are the INSTRUMENT FOR FLAME EMISSION Components ?

1-Flame atomizer.

3- Detector.

2- Monochromator

4-Readout meter



Flame Atomizer

a-Atomization of the sample

b- Source of thermal energy to excite the atoms.

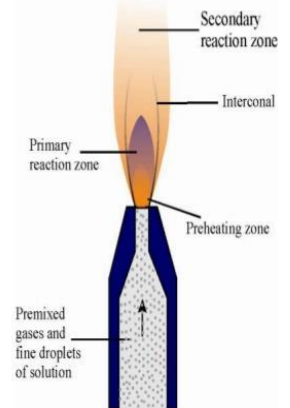
The atomizer is composed of :

- 1- Nebulizer
- 2- Burner

Structure of Flame:

As seen in the figure, the flame may be divided into the following regions or zones.

- Preheating zones
- Primary reaction zone or inner zone
- Internal zone
- Secondary reaction zone



Nebulizer: is a device by which sample solution is divided into very fine droplets which are aspirated into fine spray or aerosol.

As the oxidant flows it withdraws the sample from the capillary in very fine droplets

aerosol modifier : it removes the large droplets from the stream and allow smaller droplets than a certain size to pass .

- ❖ In case of potassium, sodium, lithium and calcium, they are atomized and excited below 20000 k above 25000 k ionization occurs.
- ❖ **The flame temperature must be**
 - ✓ an increase by 10^0 c is accompanied by increase of 4% in the excited atoms.
 - ✓ It must be sufficient to cause atomization only and not ionization.

Burner :

- ✓ Several burners and fuel oxidant combinations have been used to produce flame

Types of burners :

1- Premix burner .

2- Total consumption burner .

- This burner uses fuel hydrogen and oxygen gases .
- Sample solution is aspirated through a capillary by high pressure of fuel and oxidants .
- Entire sample is consumed.

Monochromator : it is a filter which allow the resonance wavelength to pass to the detector

➤ **Sodium**

- The major cat ion of the extracellular fluid is sodium. The typical daily diet contains 130-280 mmol (8-15 g) sodium chloride. The body requirement is for 1-2 mmol per day, so the excess is excreted by the kidneys in the urine

•

Reference range (intervals) for sodium	
Serum	136-145 mM
Cerebrospinal fluid	130-150 mM
Sweat	10-40 mM
Urine (varies with intake)	40-220 mmol/day

- **Hyponatraemia** : it is lower plasma of $[Na^+]$ level .
- **Hypertnatraemia** : raised plasma of $[Na^+]$ level .
- These are associated with a variety of diseases and illnesses and the accurate measurement of $[Na^+]$ in body fluids is an important diagnostic aid.

➤ **Potassium**

- **Potassium** : is the major cation found intracellularly .
- The average cell has **140 mM K⁺** inside but only about **10 mM Na⁺**
- K⁺ slowly diffuses out of cells so a membrane pump (the **Na⁺ /K⁺ - ATPase**) continually transports K⁺ into cells against a concentration gradient.
- **NOTE** : one K consumes 3 ATPs to be preserved inside the cell
- The human body requires about **50-150 mmol/day**.

Reference range (intervals) for potassium	
Serum	3.5-5.1 mM
Cerebrospinal fluid	about 70% of serum
Sweat	4.0-9.7 mM (men) 7.6-15.6 mM (women)
Urine (varies with intake)	25-125 mmol/day
Erythrocytes (intracellular)	105 mM

- **Hypokalaemia** :it is lower plasma level of [k⁺].
- **Hyperkalaemia** : it is rised plasma level of [k⁺].
- The high level of [k⁺] leads to hyperkaluria (increased urinary excretion of K⁺) , this is indicative of a variety of conditions .
- So the clinical measurement of [K⁺] is also of great importance.

The Flame Photometer

- A traditional and simple method for determining sodium and potassium in biological fluids involves the technique of emission flame photometry. This relies on the principle that an alkali metal salt drawn into a non-luminous flame will ionize, absorb energy from the flame and then emit light of a characteristic wavelength as the excited atoms decay to the unexcited ground state.
- You are probably familiar with the fact that if you sprinkle table salt (NaCl) into a gas flame then it glows bright orange (KCl gives a purple colour).
- A photocell detects the emitted light and converts it to a voltage, which can be recorded .
- Since Na⁺ and K⁺ emit light of different wavelengths (colours), by using appropriate coloured filters the emission due to Na⁺ and K⁺ (and hence their concentrations) can be specifically measured in the same sample
- A flame photometer can also be used to measure the element **lithium** in serum or plasma in order to determine the correct dosage of lithium carbonate, a drug used to treat certain mental disturbances, such as manic-depressive illness (bipolar disorder

- **The Experiment:**

- For preparing calibration curve :
- For example , there is conc. Solution of KCL , NaCL ... 2 mg/dl .
- Prepare 10 solutions with different concentrations of standard solution

Standards conc.(mg/dl)	standards volume (ml)	WATER (BLANK) ml
1- --	Blank (water)	1.0
2- 0.1	0.1	0.9
3- 0.2	0.2	0.8
4- 0.3	0.3	0.7
5- 0.4	0.4	0.6
6- 0.5	0.5	0.5
7- 0.6	0.6	0.4
8- 0.7	0.7	0.3
9- 0.8	0.8	0.2
10- 0.9	0.9	0.1
11- 1.0 (N1)	1.0	+ -- = 1 ml, V2
cons. Of diluted solutions		Diluted solution volume

- cons. Of diluted solutions which has been calculated by the following function :

$$N1.V1 = N2.V2 \dots \rightarrow N2 = N1.V1/V2$$

Conc. Diluted

ex: for calculation of concentration of the 5th solution

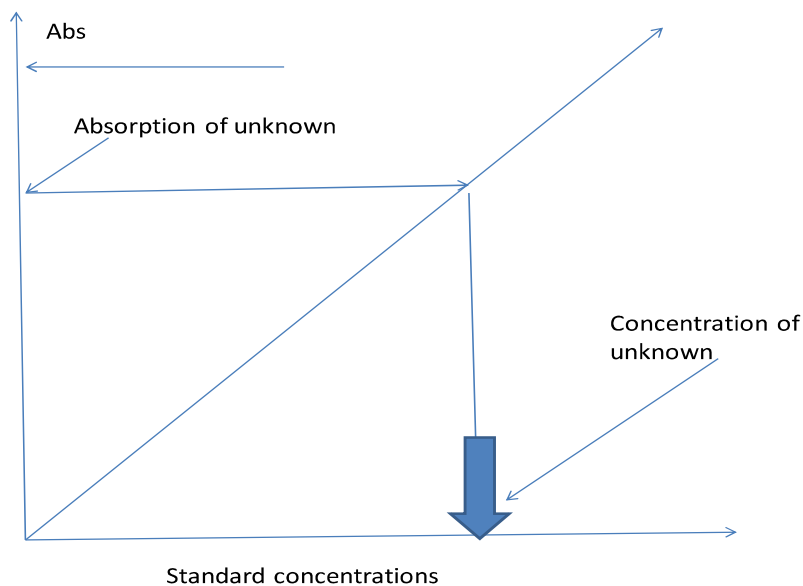
$$N2 = N1V1 / V2 \dots \dots \dots = 2.0 * 0.4 \text{ ml} / 1 \text{ ml} = 0.8 \text{ mg/dl}$$

N 2 = concentration of diluted standard solution .

V1 = volume of standard concentrated solution for a sample

V2 = volume of the final diluted solution

N1 = concentration of conc. Standard solution



- **NOTE:** In the flame emission experiment needs high volume of solution that analyzed, so we must dilute the solution for accurate determination .
- The water that's used in this experiment, should be **D-IONIZED**? WHY ?