

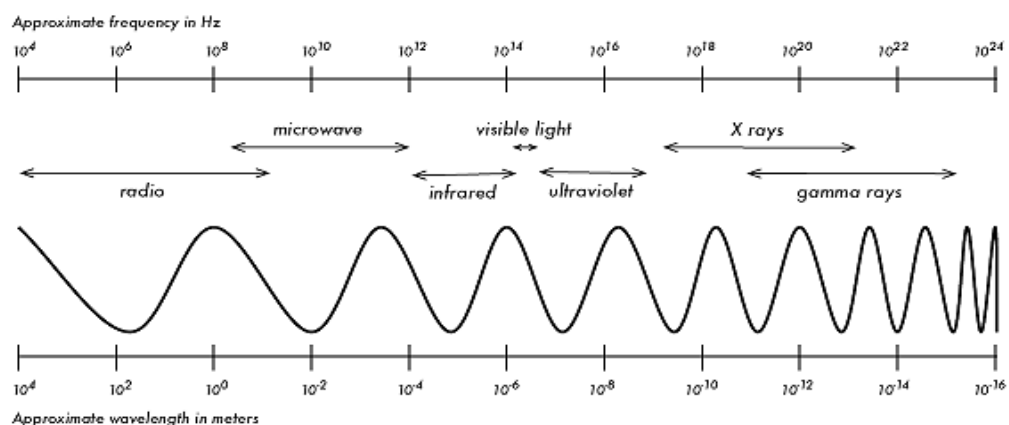
FLAME PHOTOMETRY

AIM

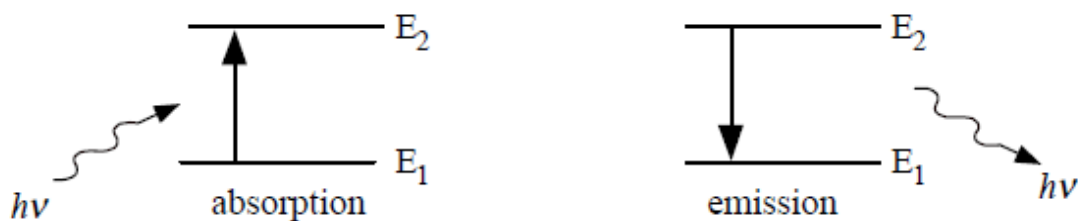
INTRODUCTION

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.

Electromagnetic Spectrum:



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.



Flame photometry or flame emission spectroscopy is an atomic emission technique. There is no need for light source. Flame serves both as an atomizer and excitation source. It is suitable for qualitative and quantitative determination of several cations, especially for metals that are easily excited to higher energy levels at flame temperature. These metals are Na, K, Ca, Ba, Li.

In this technique, first aerosols are formed from sample solution by a jet of compressed gas. This process is called nebulization. The design of the nebulizer is shown in Figure 1. 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 are reduced to gaseous atoms and also depending on the temperature of the flame a certain fraction of these atoms are 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.

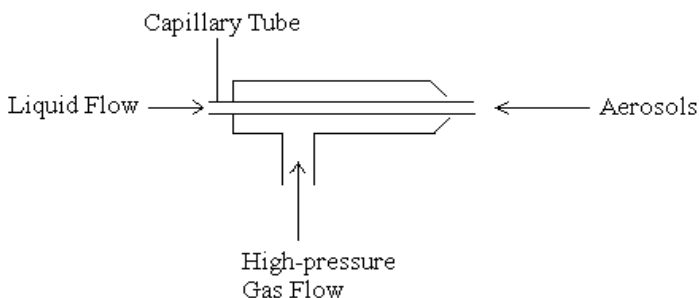


Figure 1. Design of a nebulizer

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.

Table 1. Flame components and temperatures

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 consists of three important regions. These are the primary combustion zone, interconal region and outer cone (secondary combustion zone). The appearance and the relative sizes of these regions can be changed with the fuel-oxidant ratio. Regions of the flame are shown in Figure 2.

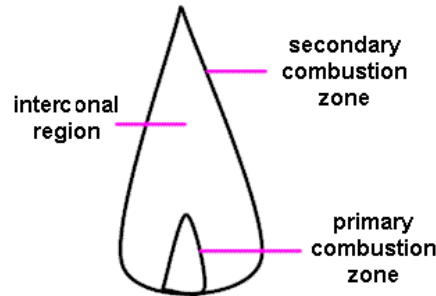


Figure 2. Schematic appearance of flame

The primary combustion zone of the flame is blue in color. In this region, there is no thermal equilibrium. Therefore, it is not used in flame spectroscopy. The interconal region is rich in free atoms and is the most widely used region for the spectroscopy. In The outer cone the products of the inner core are converted to stable molecular oxides.

In flame photometer there are three fundamental systems which are emission, λ -selection and recording. The general flow diagram is shown in Figure 3.

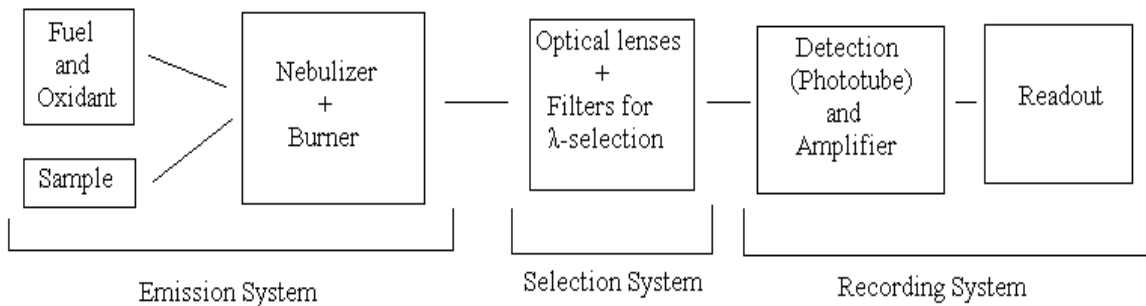


Figure 3. A general scheme of a Flame Photometer

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

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

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.

The instrument that is used in this experiment is a JENWAY PFP7 model which is a low temperature, single channel emission Flame Photometer designed for the routine determinations of Na, K, Ca, Li and Ba. It is a direct reading digital instrument designed for use in clinical, industrial and educational applications.

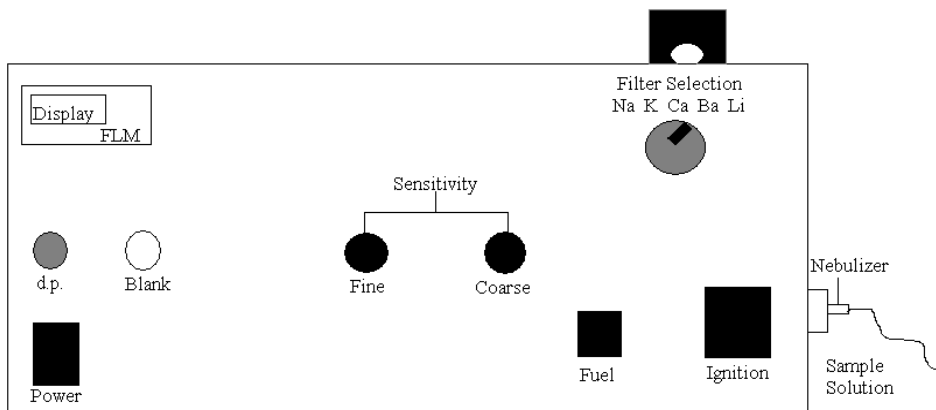


Figure 4. Front Panel Controls for the instrument

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).

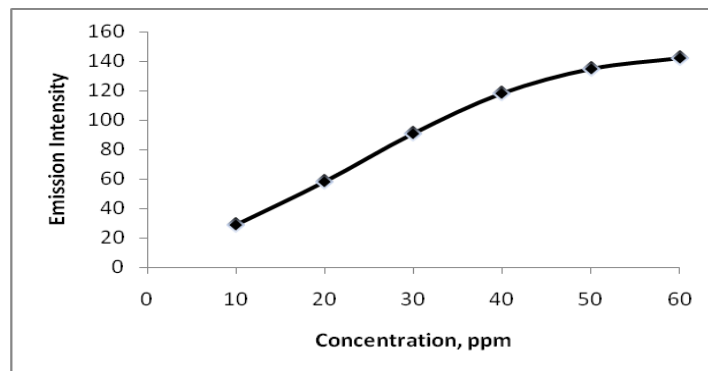


Figure 5. Calibration curve

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 has to 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.

REAGENTS AND APPARATUS

- Calcium Carbonate, $\text{Ca}(\text{NO}_3)_2$ (in the balance room)
- Sodium Chloride, NaCl (in the balance room)
- Do not forget to bring **sour cherry juice**.
- 100 mL volumetric flask
- 5 mL pipette

PROCEDURE

A. Flame Photometric Determination of Calcium in tap water and sour cherry juice

- 1) Prepare 100.0 mL of 1000 mg/L Ca stock solution from $\text{Ca}(\text{NO}_3)_2$.
- 2) Prepare 100.0 mL of 10.0, 20.0, 30.0, 40.0 and 50.0 mg/L standard Ca solutions from 1000 mg/L Ca stock solution.
- 3) Select Ca as analyte from the instrument.
- 4) Adjust the fuel/oxidant ratio and burner position by aspirating 30.0 mg/L standard Ca solution.
- 5) Measure the emission intensities of all standard Ca solutions (including the blank solution) and sample solution after necessary dilutions.
- 6) Make 10 replicate measurements of 10.0 mg/L Ca standard solution to calculate Limit of detection (LOD).
- 7) Draw the calibration plot and find the concentration (in mg/L) of Ca in beverages and tap water.
 - *After finishing measurements, draw calibration curve and discuss the applicability of it to the unknown samples by looking at correlation coefficient and dynamic range and if necessary, prepare more solutions.*

B. Flame Photometric Determination of Sodium in tap water and sour cherry juice

- 1) Prepare 100.0 mL of 1000 mg/L Na stock solution from NaCl .

- 2) Prepare 100.0 mL of 10.0, 20.0, 30.0, 40.0 and 50.0 mg/L standard Na solutions from 1000 mg/L Na stock solution.
- 3) Select Na as analyte from the instrument.
- 4) Adjust the fuel/oxidant ratio and burner position by aspirating 30.0 mg/L standard Na solution.
- 5) Measure the emission intensities of all standard Na solutions (including the blank solution) and sample solution after necessary dilutions.
- 6) Make 10 replicate measurements of 10.0 mg/L Na standard solution to calculate Limit of detection (LOD).
- 7) Draw the calibration plot and find the concentration (in mg/L) of Na in beverages and tap water.
 - *After finishing measurements, draw calibration curve and discuss the applicability of it to the unknown samples by looking at correlation coefficient and dynamic range and if necessary, prepare more solutions.*

PRE-LAB STUDIES

Read the introduction from the manual.

- 1) Describe the basic differences between atomic emission and atomic absorption spectroscopy?
- 2) What kinds of experimental setups can be used for the detection of atomic emission ? Give more than one example considering the filters, monochromators, and different types of detectors which you can use in this manner.
- 3) What are the differences between flame emission and absorption spectrometry in terms of instrumentation and process?
- 4) Can we use flame photometer for the determination of all of the elements in periodic table?
- 5) Can we analyze solid, gas and organic samples using flame photometry? If yes explain how.
- 6) What is the function of nebulizer used in flame photometer?

POST-LAB STUDIES

- 1) What could be the weak sides of determining the concentration of an unknown by Flame Photometry?
- 2) Discuss the possible matrix interference effect for the determination of Na in beverages. What are your suggestions to eliminate matrix interference if there is?

Name surname:

Section:

Date:

REPORT SHEET
FLAME PHOTOMETRY

<u>Part A: Determination of Calcium</u>		<u>Part B: Determination of Sodium</u>	
Sample	Emission	Sample	Emission
blank		blank	
10 ppm		10 ppm	
20 ppm		20 ppm	
30 ppm		30 ppm	
40 ppm		40 ppm	
50 ppm		50 ppm	
Tap water		Tap water	
Sour cherry juice		Sour cherry juice	

Part A: Determination of Calcium	
Line Equation ($y=mx+n$)	
R ²	
Limit of Detection	
C _{Ca} in tap water (ppm)	
C _{Ca} in sour cherry juice	

Part B: Determination of Sodium	
Line Equation ($y=mx+n$)	
R ²	
Limit of Detection	
C _{Na} in tap water (ppm)	
C _{Na} in sour cherry juice	

TA's Name and Signature: