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SUBJECT NAME: INSTRUMENTAL METHOD OF ANALYSIS

SUBJECT CODE: BP701TP

CHAPTER 2 : FLAME PHOTOMETRY

FlamePhotometer:Principle,Components,Working Procedure,Applications,Advantagesand Disadvantages

Introduction

During 1980s Bowling Barnes, David Richardson, John Berry and Robert Hood developed an instrument to measure the low concentrations of sodium and potassium in a solution. They named this instrument as Flame photometer. The principle of flame photometer is based on the measurement of the emitted light intensity when a metal is introduced into the flame. The wavelength of the colour gives information about the element and the colour of the flame gives information about the amount of the element present in the sample.

Flame photometry is one of the branches of atomic absorption spectroscopy. It is also known as flame emission spectroscopy. Currently, it has become a necessary tool in the field of analytical chemistry. Flame photometer can be used to determine the concentration of certain metal ions like sodium, potassium, lithium, calcium and cesium etc. In flame photometer spectra the metal ions are used in the form of atoms. The International Union of Pure and Applied Chemistry (IUPAC) Committee on Spectroscopic Nomenclature has named this technique as flame atomic emission spectrometry (FAES).

Principle of Flame photometer

The compounds of the alkali and alkaline earth metals (Group II) dissociate into atoms when introduced into the flame. Some of these atoms further get excited to even higher levels. But these atoms are not stable at higher levels.Hence, these atoms emit radiations when returning back to the ground state. These radiations generally lie in the visible region of the spectrum. Each of the alkali and alkaline earth metals has a specific wavelength.

Element	Emitted wavelength	Flame color
Sodium	589 nm	Yellow
Potassium	766 nm	Violet
Barium	554 nm	Lime green
Calcium	622 nm	Orange
Lithium	670 nm	Red

For certain concentration ranges,

The intensity of the emission is directly proportional to the number of atoms returning to the ground state. And the light emitted is in turn proportional to the concentration of the sample.

Parts of flame photometer

A simple flame photometer consists of the following basic components:

Major Components:

1. Sample Delivery System

2. Source

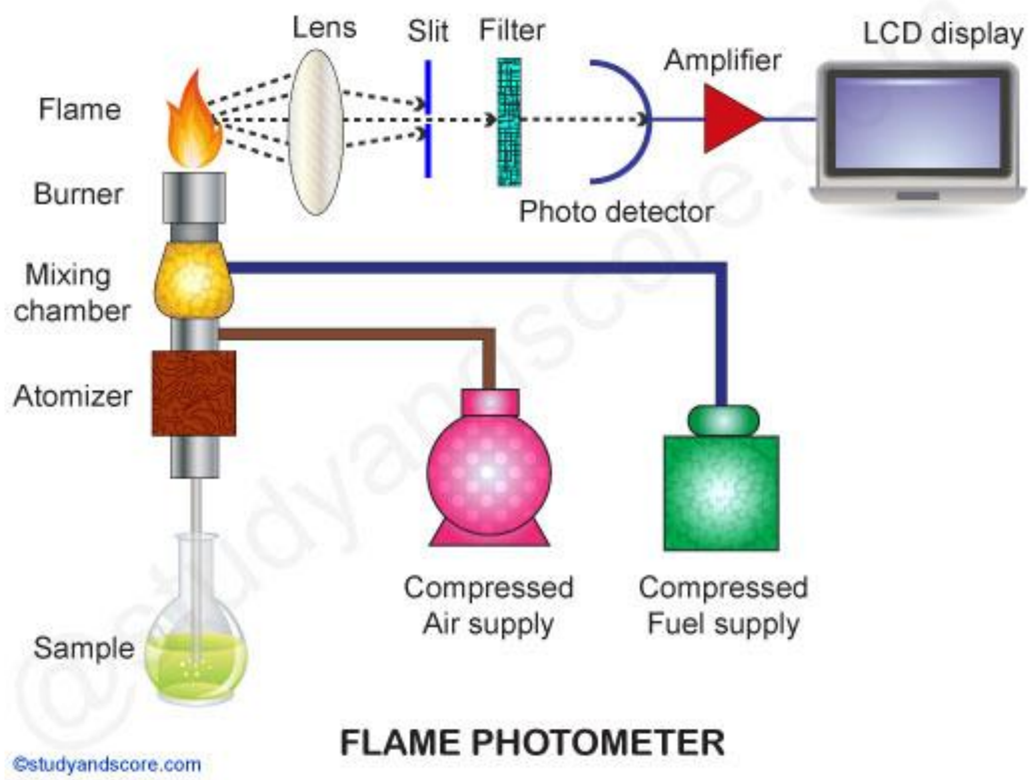
3. Monochromator

4. Detector

Sample Delivery System: There are three components for introducing liquid sample:

Nebulizer – it breaks up the liquid into small droplets. **Nebulization** the is conversion of a sample to a mist of finely divided droplets using a jet of compressed gas. The flow carries the sample into the atomization region. – Pneumatic Nebulizers: (most common) .

5.Read out device



Schematic Representation of the Flame Photometer

Aerosol modifier – it removes large droplets from the stream and allow only smaller droplets than a certain size to pass

Flame or Atomizer – it converts the analyte into free atoms

Source:

A Burner used to spray the sample solution into fine droplets. Several burners and fuel+oxidant combinations have been used to produce analytical flame including:

Premixed, Mecker,

Total consumption,

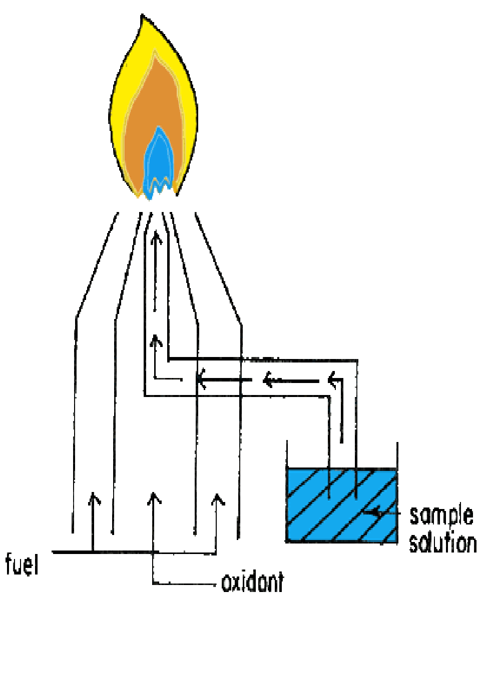
Lundergarh,

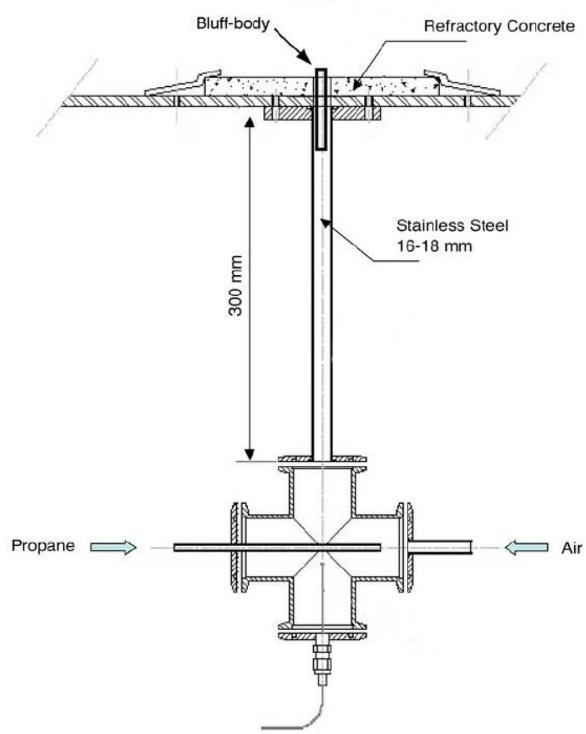
Shielded burner, and

Nitrous oxide-acetylene flames

- **Pre-mixed Burner:** – widely used because uniformity in flame intensity – In this energy type of burner , aspirated sample , fuel and oxidant are thoroughly mixed before reaching the burner opening. 13

Total Consumption Burner: – In this fuel and oxidant are hydrogen and oxygen gases – Sample solution is aspirated through a capillary by high pressure of fuel and Oxidant and burnt at the tip of burner – Entire sample is consumed.





Total Consumption Burner

Pre-mixed burner

Monochromator: –

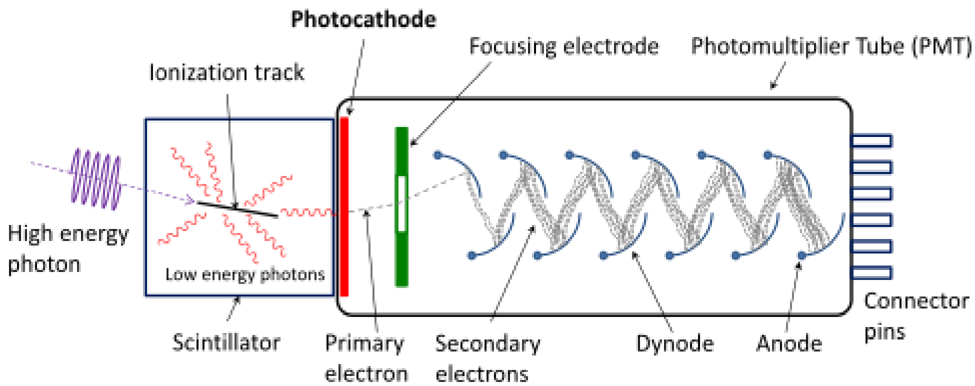
Prism: Quartz material is used for making prism, as quartz is transparent over entire region – Grating: it employs a grating which is essentially a series of parallel straight lines cut into a plane surface

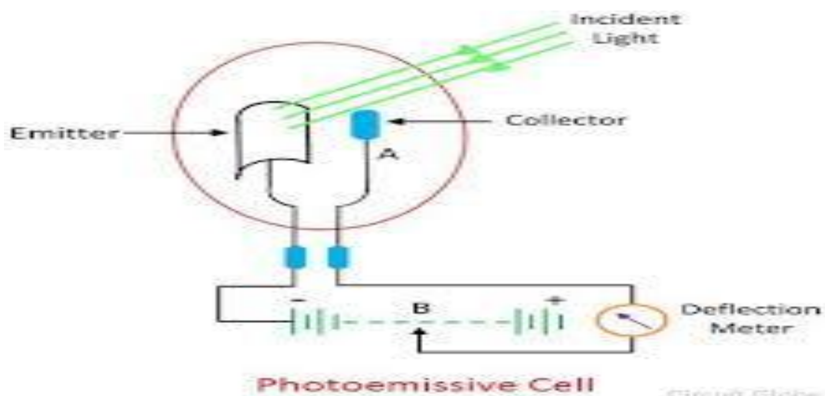
Detectors:

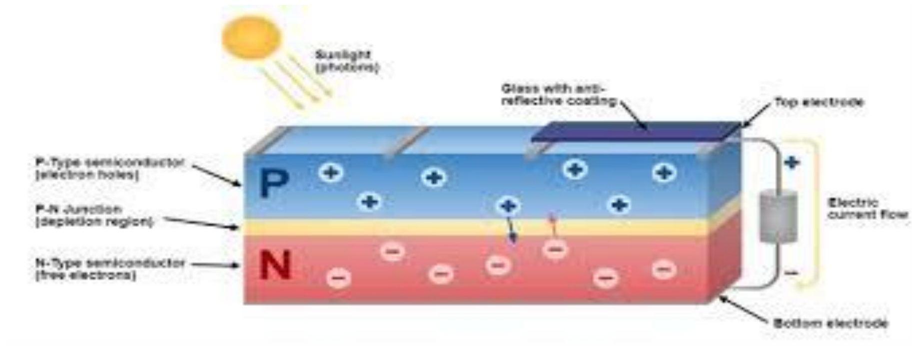
– Photomultiplier tubes

– Photo emissive cell

– Photo voltaic cell







Photovoltaic cell: • It has a thin metallic layer coated with silver or gold which act as electrode, also has metal base plate which act as another electrode • Two layers are separated by semiconductor layer of selenium, when light radiation falls on selenium layer. • This creates potential diff. between the two electrode and cause flow of current.

Photo-detector: The intensity of radiation emitted by the flame is measured by photo detector. Here the emitted radiation is converted to an electrical signal with the help of photo detector. These electrical signals are directly proportional to the intensity of light.

Read-out Device:

It is capable of displaying the absorption spectrum as well absorbance at specific wavelength.

Nowadays the instruments have microprocessor controlled electronics that provides outputs compatible with the printers and computers Thereby minimizing the possibility of operator error in transferring data.

Source of flame: A Burner in the flame photometer is the source of flame. It can be maintained in at a constant temperature. The temperature of the flame is one of the critical factors in flame photometry.

Fuel-Oxidant mixture	Temperature (°C)
Natural gas-Air	1700
Propane-Air	1800
Hydrogen-Air	2000
Hydrogen-Oxygen	2650
Acetylene-Air	2300
Acetylene-Oxyen	3200
Acetylene-Nitrous oxide	2700
Cyanogen-Oxygen	4800

Working procedure

Both the standard stock solution and sample solution are prepared in fresh distilled water.

The flame of the photometer is calibrated by adjusting the air and gas. Then the flame is allowed to stabilize for about 5 min.

Now the instrument is switched on and the lids of the filter chamber are opened to insert appropriate colour filters.

The readings of the galvanometer are adjusted to zero by spraying distilled water into the flame.

The sensitivity is adjusted by spraying the most concentrated standard working solution into the flame. Now the full scale deflection of the galvanometer is recorded.

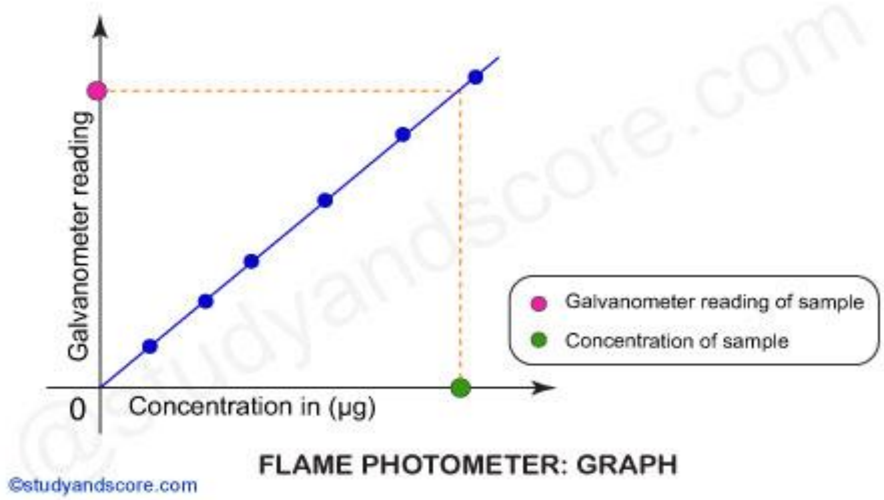
Again distilled water is sprayed into the flame to attain constant readings of galvanometer. Then the galvanometer is readjusted to zero.

Now each of the standard working solutions is sprayed into the flame for three times and the readings of galvanometer are recorded. After each spray, the apparatus must be thoroughly washed.

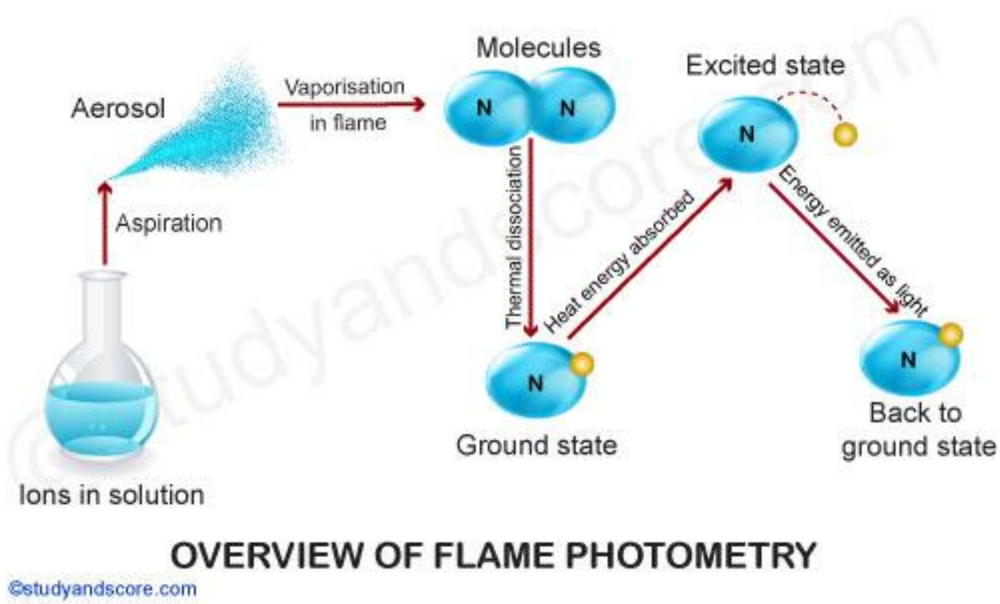
Finally sample solution is sprayed into the flame for three times and the readings of galvanometer are recorded. After each spray, the apparatus must be thoroughly washed.

Calculate the mean of the galvanometer reading.

Plot the graph of concentration against the galvanometer reading to find out the concentration of the element in the sample.



FLAME PHOTOMETER: GRAPH



overview of Flame Photometer

The solvent is first aspirated to obtain fine solid particles.

These molecules in the solid particles are moved towards the flame to produce gaseous atoms and ions.

These ions absorb the energy from the flame get excited to high energy levels from the ground state.

But as these ions are unstable, they return back to ground state. While returning they emit characteristic radiation.

The intensity of emitted light is proportional to the concentration of the element.

The oxidants in flame photometer are mainly air, oxygen or nitrous oxide. The temperature of the flame depends on the ratio of fuel and oxidant.

The processes occurring during flame photometer analysis are summarized below:

Desolvation: Desolvation involves drying a sample in a solution. The metal particles in the solvent are dehydrated by the flame and thus solvent is evaporated.

Vaporization: The metal particles in the sample are also dehydrated. This also led to the evaporation of the solvent.

Atomization: Atomization is the separation of all atoms in a chemical substance. The metal ions in the sample are reduced to metal atoms by the flame.

Excitation: The electrostatic force of attraction between the electrons and nucleus of the atom helps them to absorb a particular amount of energy. The atoms then jump to the higher energy state when excited.

Emission: Since the higher energy state is unstable the atoms jump back to the ground state or low energy state to gain stability. This jumping of atoms emits radiation with characteristic wavelength. The radiation is measured by the photo detector.

Scheibe-Lomakin equation

Scheibe-Lomakin equation describes intensity of light emitted with the help of following formula:

$I = k \times cn$

Where:

I = Intensity of emitted light

c = Concentration of the element

k = Proportionality constant

At the linear part of the calibration curve n~1, then I = k × c. In other words, the intensity of emitted light is directly related to the concentration of the sample.

emitted wavelength and flame colors of alkali and alkaline earth metals, flame color chart.

INTERFERENCES: In determining the amount of a particular element present, other elements can also affect the result. Such interference may be of 3 kinds:

Spectral interferences: occurs when the emission lines of two elements cannot be resolved or arises from the background of flame itself. – They are either too close, or overlap, or occur due to high concentration of salts in the sample

Ionic interferences: high temperature flame may cause ionisation of some of the metal atoms, e.g. sodium. – The Na+ ion possesses an emission spectrum of its own with frequencies, which are different from those of atomic spectrum of the Na atom.

Chemical interferences: The chemical interferences arise out of the reaction between different interferents and the analyte. Includes:

Cation-anaion interference: – The presence of certain anions, such as oxalate, phosphate, sulfate, in a solution may affect the intensity of radiation emitted by an element. E.g., – calcium + phosphate ion forms a stable substance, as Ca3(PO4)2 which does not decompose easily, resulting in the production of lesser atoms.

Cation-cation interference: – These interferences are neither spectral nor ionic in nature – Eg. aluminum interferes with calcium and magnesium.

Applications of flame photometer

Flame photometer can be applied both for quantitative and qualitative analysis of elements. The radiations emitted by the flame photometer are characteristic to particular metal. Hence with the help of Flame photometer we can detect the presence of any specific element in the given sample.

The presence of some group II elements is critical for soil health. We can determine the presence of various alkali and alkaline earth metals in soil sample by conducting flame test and then the soil can be supplied with specific fertiliser.

The concentrations of Na+ and K+ ions are very important in the human body for conducting various metabolic functions. Their concentrations can be determined by diluting and aspirating blood serum sample into the flame.

Soft drinks, fruit juices and alcoholic beverages can also be analysed by using flame photometry to determine the concentrations of various metals and elements.

Advantages of flame photometer

The method of analysis is very simple and economical.

It is quick, convenient, selective and sensitive analysis.

It is both and qualitative and quantitative in nature.

Even very low concentrations (parts per million/ppm to parts per billion/ppb range) of metals in the sample can be determined.

This method compensates for any unexpected interfering material present in the sample solution.

This method can be used to estimate elements which are rarely analysed.

Disadvantages of flame photometer

In spite of many advantages, this analysis technique has quite a few disadvantages:

The accurate concentration of the metal ion in the solution cannot be measured.

It cannot directly detect and determine the presence of inert gases.

Though this technique measures the total metal content present in the sample, it does not provide the information about the molecular structure of the metal present in the sample.

Only liquid samples may be used. Also sample preparation becomes lengthy in some cases.

Flame photometry cannot be used for the direct determination of each and every metal atom. A number of metal atoms cannot be analysed by this method. The elements such as carbon, hydrogen and halides cannot be detected due to their non-radiating nature.