

FLAME PHOTOMETRY

BASIC CONCEPTS, INSTRUMENTATION, AND APPLICATION

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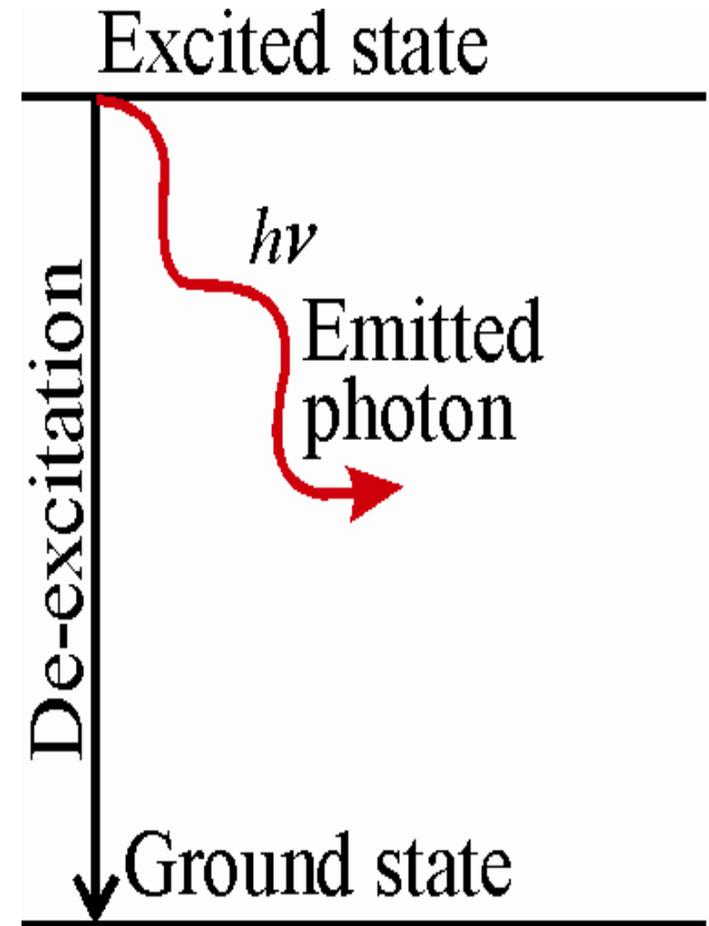
INTRODUCTION:

- Flame photometry (more accurately called *Flame Atomic Emission Spectrometry*) is a branch of spectroscopy in which the species examined in the spectrometer are in the form of atoms
- A photoelectric flame photometer is an instrument used in inorganic chemical analysis to determine the concentration of certain metal ions among them sodium, potassium, calcium and lithium.
- Flame Photometry is based on measurement of intensity of the light emitted when a metal is introduced into flame.
 - The wavelength of colour tells what the element is (qualitative)
 - The colour's intensity tells us how much of the element present (quantitative)

- The basic principle upon which Atomic Spectroscopy works is based on the fact that *"Matter absorbs light at the same wavelength at which it emits light"*.
- Atoms of elements → subjected to hot flame → specific quantum of thermal energy absorbed by orbital electrons → become unstable at high energy level → release energy as photons of particular wavelength → change back to ground state.
- When a metal salt solution is burned, the metal provides a colored flame and each metal ion gives a different colored flame.
- Flame tests, therefore, can be used to test for the absence or presence of a metal ion

BASIC CONCEPT:

- Liquid sample containing metal salt solution is introduced into a flame,
- Solvent is first vaporized, leaving particles of solid salt which is then vaporised into gaseous state
- Gaseous molecule dissociate to give neutral atoms which can be excited (made unstable) by thermal energy of flame
- The unstable excited atoms emit photons while returning to lower energy state
- The measurement of emitted photons forms the basis of flame photometry.



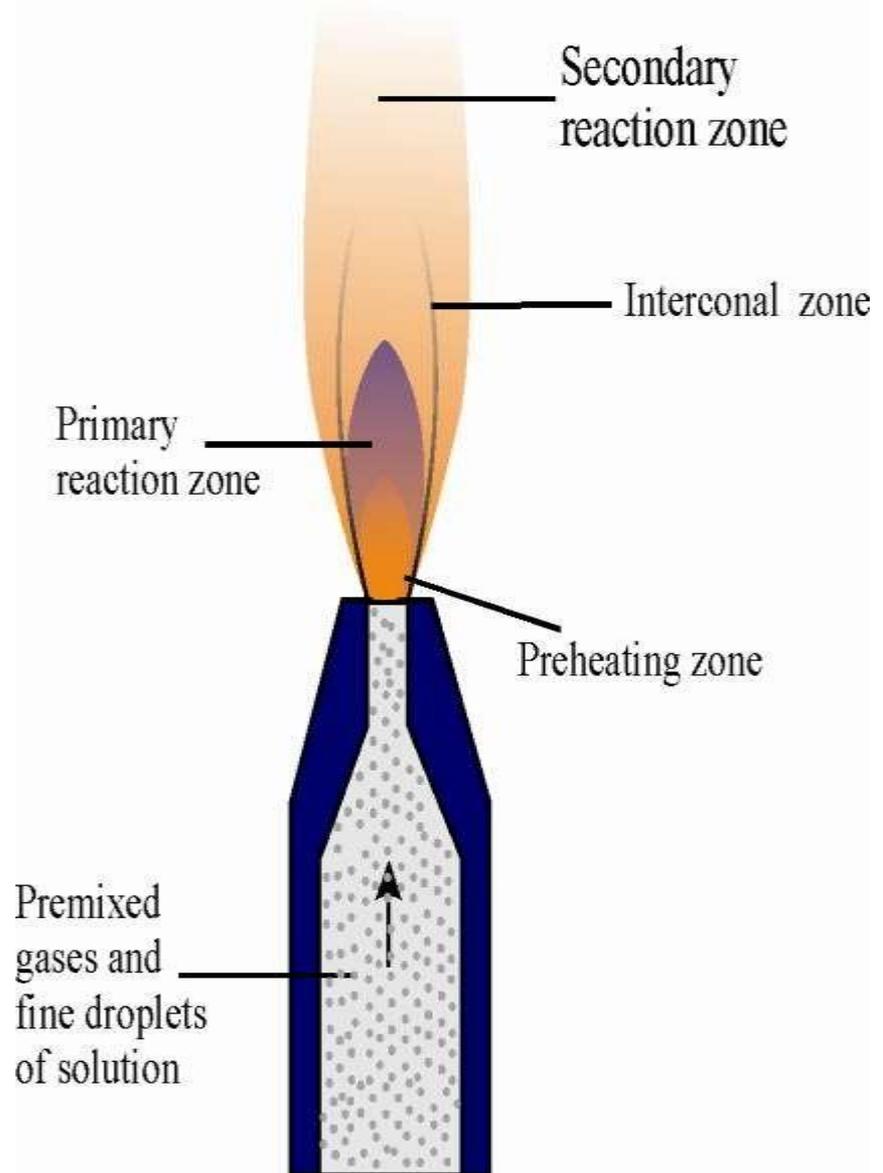
- Under constant and controlled conditions, the light intensity of the characteristic wavelength produced by each of the atoms is directly proportional to the number of atoms that are emitting energy, which in turn is directly proportional to the concentration of the substance of interest in the sample.
- Various metals emit a characteristic 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

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



- **preheating zone**- In this, combustion mixture is heated to the ignition temperature by thermal conduction from the primary reaction zone.
- **primary reaction zone**- This zone is about 0.1 mm thick at atmospheric pressure
 - There is no thermodynamic equilibrium in this zone and the concentration of ions and free radicals is very high.
 - This region is not used for flame photometry.
- **interconal zone** – It can extend up to considerable height. The maximum temperature is achieved just above the tip of the inner zone.
 - This zone is used for flame photometry.
- **secondary reaction zone** - In this zone, the products of the combustion processes are burnt to stable molecular species by the surrounding air.

INTERFERENCES:

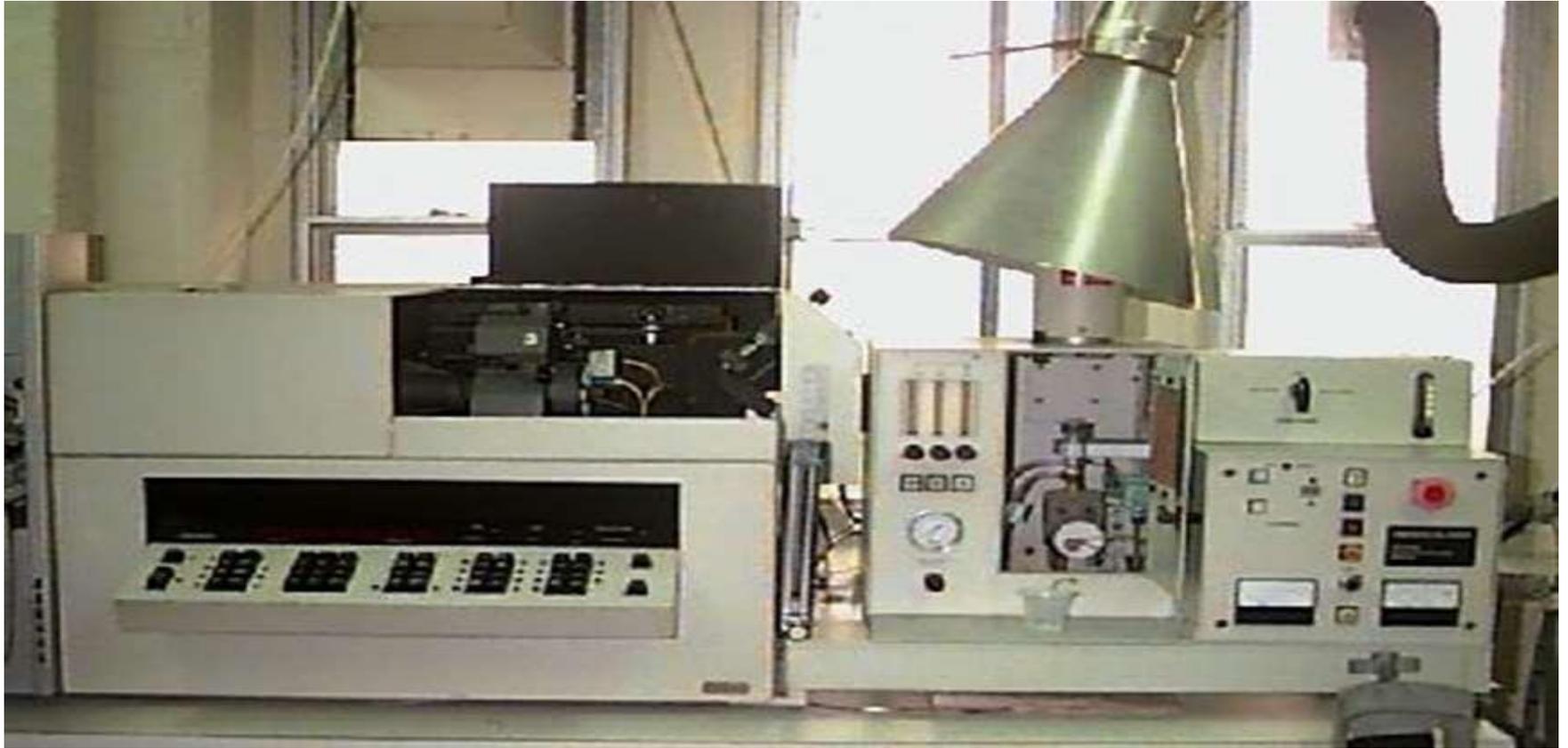
- In determining the amount of a particular element present, other elements can also affect the result.

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.

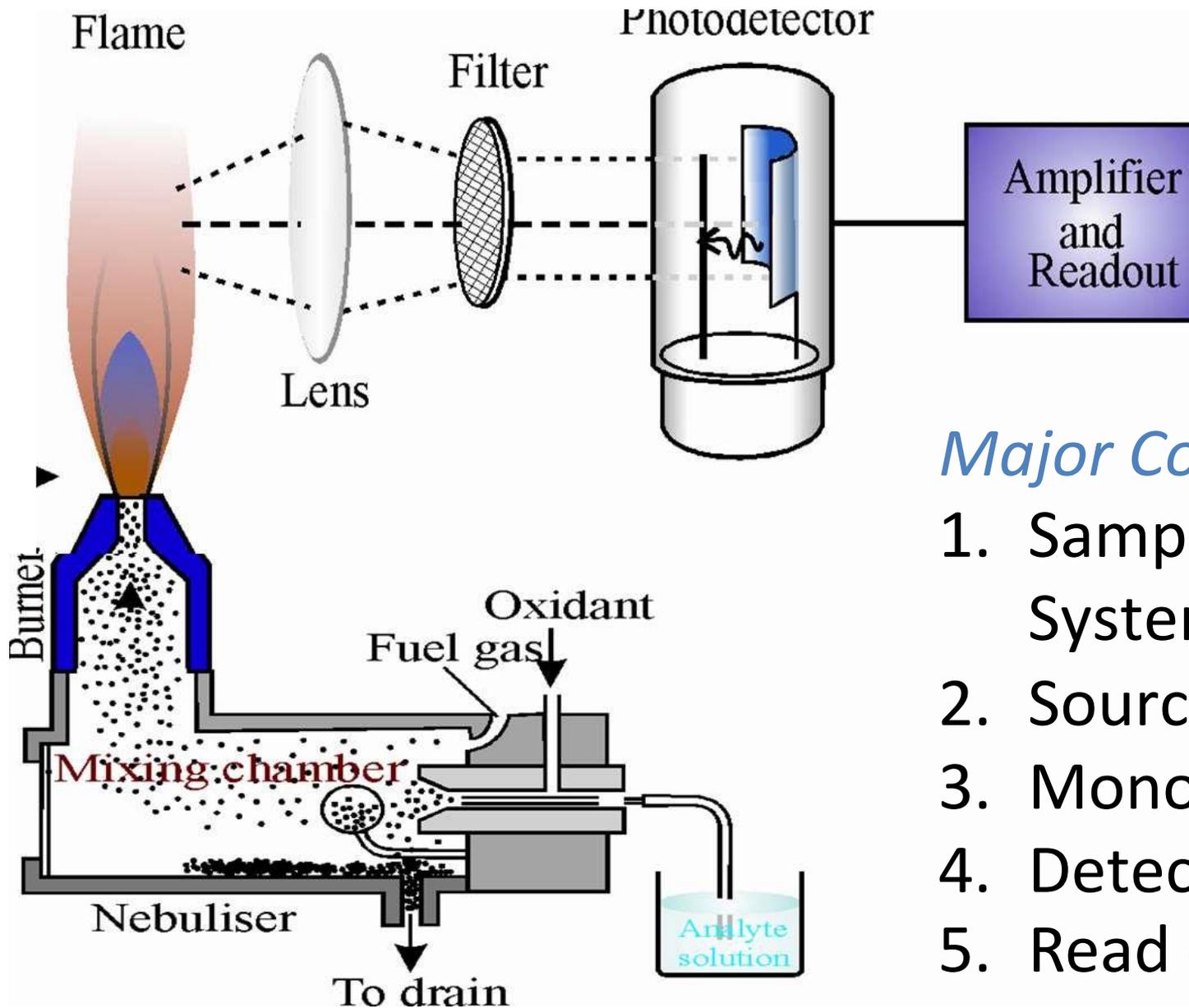
- *Chemical interferences*: The chemical interferences arise out of the reaction between different interferences and the analyte. Includes:
 - Cation-anion interference:
 - The presence of certain anions, such as oxalate, phosphate, sulfate, in a solution may affect the intensity of radiation emitted by an element. E.g.,
 - calcium + phosphate ion forms a stable substance, as $\text{Ca}_3(\text{PO}_4)_2$ 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.

INSTRUMENTATION:



THE FLAME PHOTOMETER

Schematic Representation of the Flame Photometer



Major Components:

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

Sample Delivery System:

There are three components for introducing liquid sample:

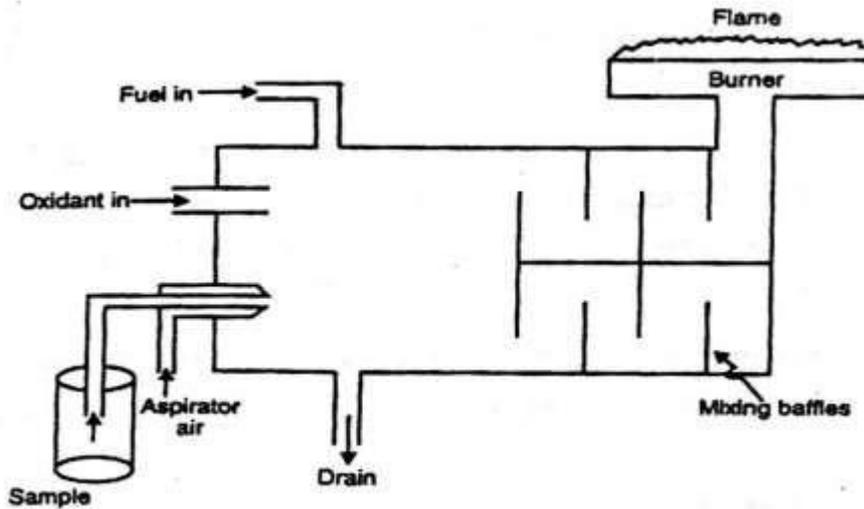
- **Nebulizer** – it breaks up the liquid into small droplets.
 - Nebulization is the conversion of a sample to a mist of finely divided droplets using a jet of compressed gas.
 - The flow carries the sample into the atomization region.
 - *Pneumatic Nebulizers:* (most common)
- **Aerosol modifier**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

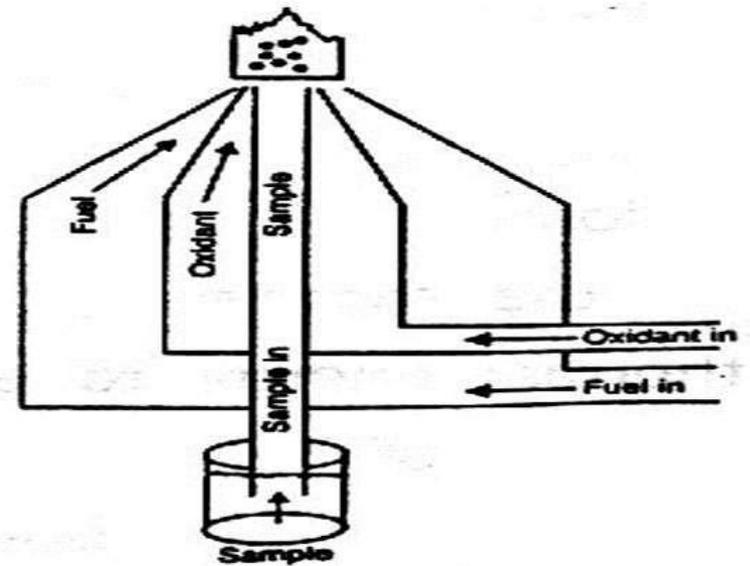
- *Total Consumption Burner:*

- 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.



A pre-mixed type.

Pre-mixed burner



Total Consumption 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.

APPLICATIONS:

- To estimate sodium, potassium, calcium, lithium etc. level in sample of serum, urine, CSF and other body fluids.
- Flame photometry is useful for the determination of alkali and alkaline earth metals.
- Used in determination of lead in petrol.
- Used in the study of equilibrium constants involving ion exchange resins.
- Used in determination of calcium and magnesium incement.