



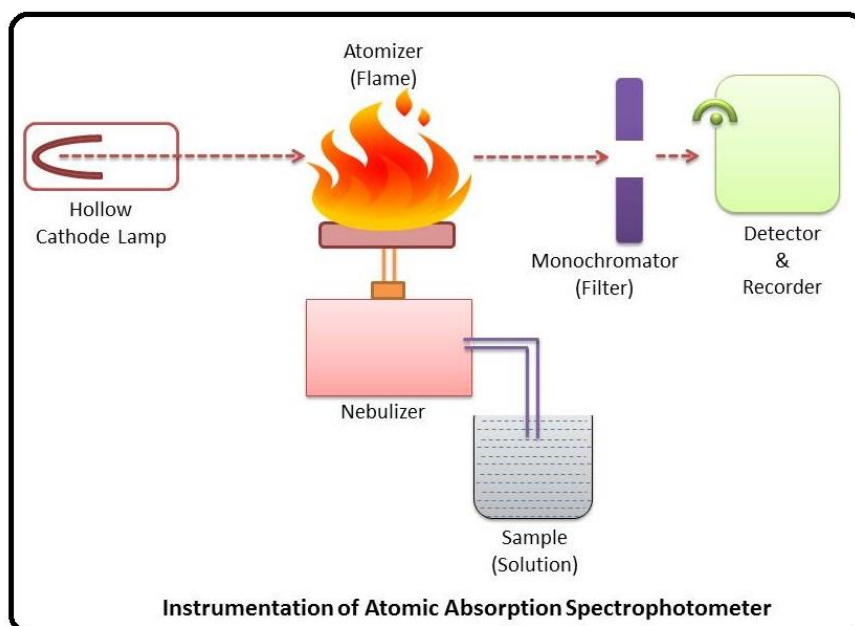
19CH103- ENGINEERING CHEMISTRY

Unit-5 INSTRUMENTAL METHODS OF ANALYSIS

AAS

The technique is based on the fact that:

- Ground state metals absorb light at specific wavelengths.
- Metal ions in a solution are converted to atomic state by means of a flame.
- Light of the appropriate wavelength is supplied and the amount of light absorbed can be measured against a standard curve.



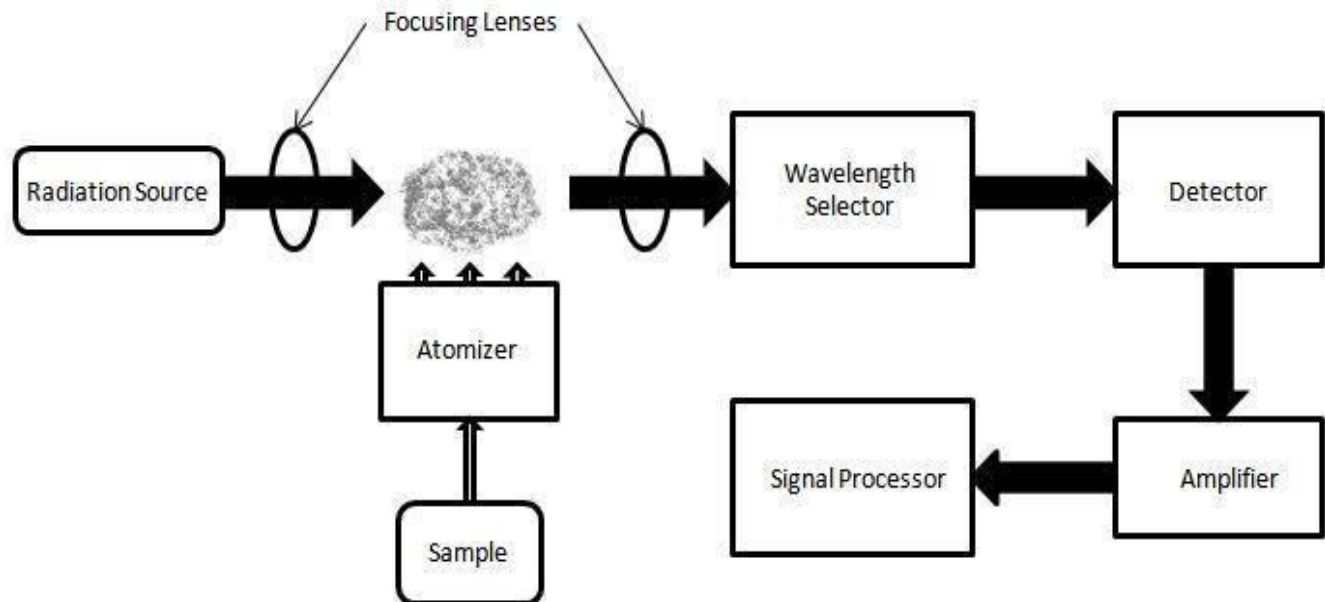
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BASIC PRINCIPLE

- The technique of (FAAS) requires a liquid sample to be aspirated, aerosolized, and mixed with combustible gases, such as acetylene and air or acetylene and nitrous oxide.
- The mixture is ignited in a flame whose temperature ranges from 2100 to 2800 °C.
- During combustion, atoms of the element of interest in the sample are reduced to free, unexcited ground state atoms, which absorb light at characteristic wavelengths.

- The characteristic wavelengths are element specific and accurate to 0.01-0.1nm.
- To provide element specific wavelengths, a light beam from a lamp whose cathode is made of the element being determined is passed through the flame.
- A device such as photomultiplier can detect the amount of reduction of the light intensity due to absorption by the analyte, and this can be directly related to the amount of the element in the sample.

Atomic absorption spectrometer “block diagram”



1. Radiation Source:

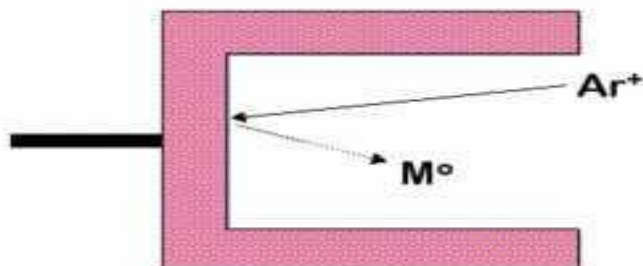
Hollow Cathode Lamp (HCL)

- A cathode lamp is a stable light source, which is necessary to emit the sharp characteristic spectrum of the element to be determined.
- A different cathode lamp is needed for each element, although there are some lamps that can be used to determine three or four different elements if the cathode contains all of them.
- Each time a lamp is changed, proper alignment is needed in order to get as much light as possible through the flame, where the analyte is being atomized, and into the monochromator



Hollow cathode lamp

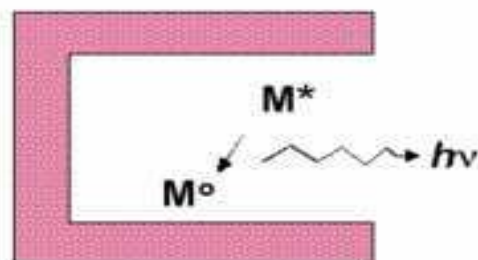
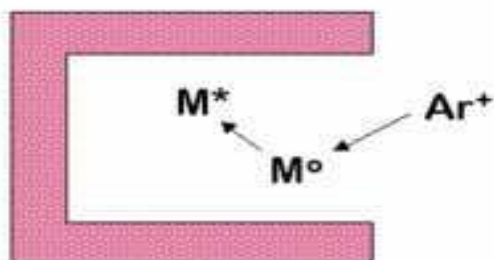
The lamp is filled with an inert gas like argon or neon. When a potential is applied, it causes the gas to become excited and it is driven towards the cathode.



Metal atoms are then sputtered off the surface of the cathode.

Hollow cathode lamp

Repeated bombardment of the metal atom by the gas causes it to be excited. It ultimately relaxes, producing specific atomic emission lines.





2. Chopper

- **Function:** is to fluctuate the source output
- It is a circular disc divided into four quarters two are mirrored and two are opened.
- The disc rotates at high constant speed, when the mirrored quarter in front of the lamp, it reflects the radiation.
- The second moment the open in front of the lamp and the radiation passes to the sample being absorbed by it and reaches the detector in pulses.
- The detector converts the radiation to alternating current signal and amplified it.
- The radiation coming from the flame itself and from atoms excited by the flame will reach the detector continuously and converted to direct current .
- signal which can be suppressed and eliminated.

3. Atomizer

a) Flame Atomizer:

Liquid samples introduced to atomizer through a nebulizer.

Uses a slot type burner to increase path length.

b) Non flame Atomizer “ Graphite Furnace)

heated electrically up to 6000°C and contains a ribbon or boat in which one can inject the sample.

Upon heating the furnace: the sample is ashen, then atomized by action of heat.

Advantages of non flame atomizer :

1. The sample volume is small
2. No need for fuel -oxidant mixture.
3. No flame noise.
4. Solid sample can be used directly.
5. Heat distribution is uniform and temperature is steady.
6. Unusual high sensitivity

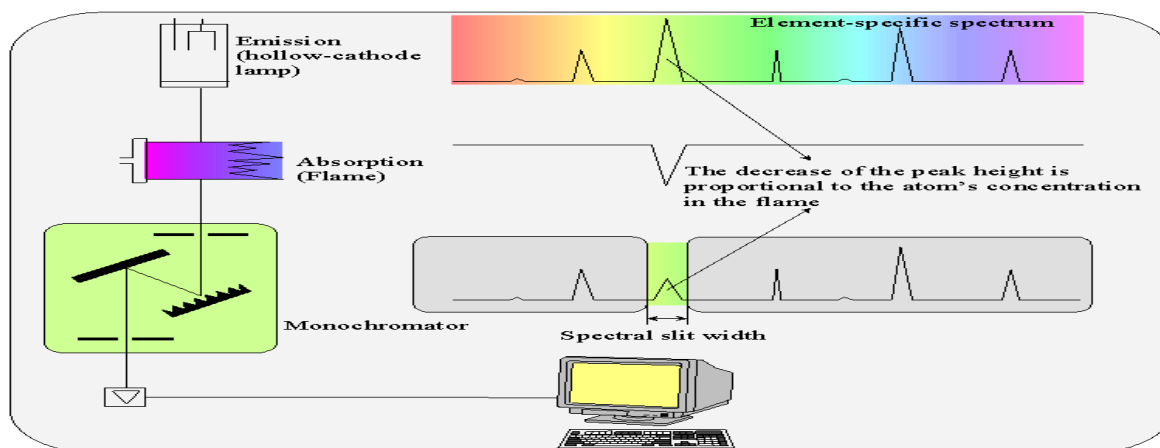
Types of flame:

- Different flames can be achieved using different mixtures of gases, depending on the desired temperature and burning velocity.
- Some elements can only be converted to atoms at high temperatures. Even at high temperatures, if excess oxygen is present, some metals form oxides that do not re-dissociate into atoms.
- To inhibit their formation, conditions of the flame may be modified to achieve a reducing, non-oxidizing flame.



4. Monochromator

- Is an optical device that transmits a mechanically selectable narrow band of wavelength of light or other radiation chosen from a wider range of wavelengths available at the input.



5. Detector

- The photomultiplier tube is almost universally used as the **detector** type in **AAS**
- The type of **detector** found in **AAS** is the photomultiplier tube - the principle of operation is the emission of electrons upon exposure to radiation. The **detector** contains a photoemissive cathode and a series of dynodes.

