# Lab Demo - 3&4

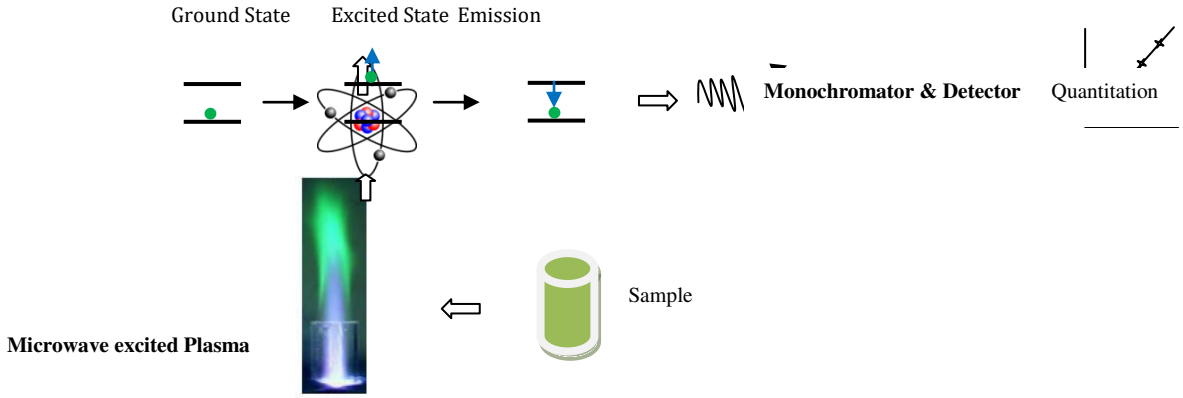
## 3. Microwave Plasma Atomic Emission Spectrometer (MP-AES)

### Introduction:

Spectroscopy is the study of the interaction between radiation and matter. Atomic emission spectroscopy (AES) is an analytical technique for determining the concentration of a particular metal element in a sample. AES is more sensitive than AAS-Flame and it can measure concentration of metals to sub parts per billion (ppb) level in a sample.

### Principle:

MP-AES uses emission spectrometry to assess the concentration of an analyte in a sample. Atomic emission occurs when a valence electron in a higher energy orbital returns to a lower energy orbital by emitting a set quantity of energy. This amount of energy (or wavelength) is specific to a particular electron transition in a particular element, and in general, each wavelength corresponds to only one element. This gives the technique its elemental selectivity. Atomic emission requires a means for converting a solid, liquid, or solution analyte into a free gaseous atom. The same source of thermal energy usually serves as the excitation source. The most common methods are flames and plasmas, both of which are useful for liquid or solution samples. Solid samples may be analyzed by dissolving in a solvent and using a flame or plasma atomizer.




### Instrumentation:

**Torch:** Plasma forms in this conventional torch using nitrogen as plasma gas.

**Microwave excitation assembly:** MP-AES instrument uses a microwave excitation assembly to create a concentrated axial magnetic field around a conventional torch and provides a robust, high temperature source in conventional torch (approx 5000 K) This focuses the microwave energy where it is needed to produce a toroidal plasma with a cooler central channel that is suitable for stable atomization.

### Monochromator & Detector:

When sample aerosol get introduced into nitrogen plasma the axial emission from the vertical oriented nitrogen plasma is directed into the fast scanning monochromater optics and wavelength specific emission are detected using a high efficiency Charged coupled device.

## 4. Atomic Absorption Spectrophotometer (AAS)

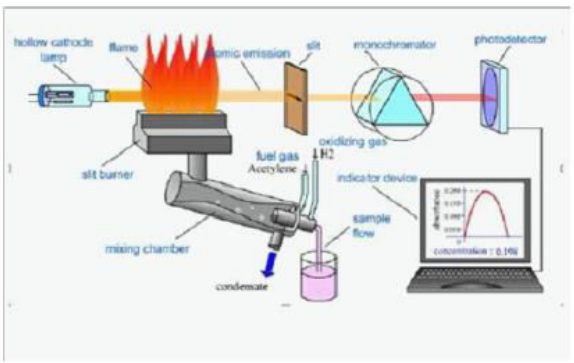
**Introduction:**

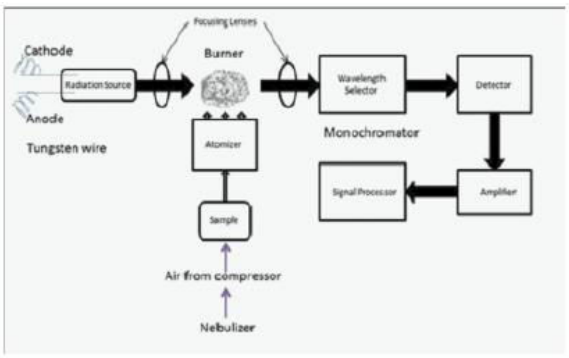
Spectroscopy is the study of the interaction between radiation and matter. Atomic absorption spectrometry (AAS) is an analytical technique that measures the concentrations of elements. Atomic absorption is so sensitive that it can measure down to parts per billion of a gram (μg dm−3) in a sample. The technique uses the wavelengths of light specifically absorbed by an element.

**Principle:**

Atomic absorption spectroscopy studies the absorption of radiation (both in UV and visible region) by neutral atoms in a flame. Thus, in atomic absorption spectroscopy, the sample is first converted to atomic vapours, and then absorption of atomic vapours is measured at a selected wavelength. This technique is also called absorption flame photometry because all the assays (or) analytical applications of atomic absorption involve spraying the sample into the flame. When an aerosol is delivered into a flame in atomic absorption spectroscopy, the following sequence of events occurs in rapid succession.

- Water/solvent is vaporized, leaving minute particles of dry salt.
- Dry salt is vaporized at the high temperature of the flame.
- Then part or all the gaseous molecules are dissociated into neutral atoms
- The neutral atoms in a gaseous state absorb radiation at a specific wavelength




**Radiation Source:**

To produce a beam of radiation with very narrow bandwidth, either

- A source of white light with a double monochromator

(Or)

- A hollow cathode discharge lamp is used.

The discharge lamps are specific to the element being analyzed. A sample of elements to be excited is held in a metal cup cathode and a tungsten anode. Argon carrier gases at low pressure and high voltage are used to produce a spectrum of the element.

**Nebuliser/Atomiser:**
Nebuliser is of scent spray type in which air is forced through the capillary tube dipping into the sample solution; thus, the sample is sprayed into the flame.

**Flame:**
To increase the optical path length of the sample, a series of burners may be used.

**Monochromator:**
A monochromator is used for more precise work, but the simple routine analysis, a simple filter may replace a monochromator.

**Detector:**
Photocell or photomultiplier tubes are generally used

**Advantage of AAS over Flame Emission Spectroscopy:**

- Atomic absorption spectroscopy is more sensitive because atoms of a specific element can absorb only radiation of their characteristic wavelength.
- Atomic absorption spectroscopy is independent of flame temperature.

**Disadvantages:**

- A separate cathode discharge lamp is required for each element to be determined.
- AAS cannot be used very successfully for the estimation of elements like Aluminium, Titanium, Tungsten, Molybdenum, Silicon, Vanadium, etc., because those elements give rise to oxides in the flame.