

# Atomic absorption spectroscopy

Atomic absorption spectroscopy deals with the absorption of specific wavelength of radiation by neutral atoms in the ground state.

The phenomenon is similar to U.V spectroscopy, where absorption of radiation by molecules occurs.

Neutral atoms are obtained by spraying the sample solution of element using a burner.

Specific wavelength of radiation is produced by hollow cathode lamp.

Principle →

Liquid sample  
(sol<sup>n</sup> of metallic salt)

↓ sprayed in to flame

Formation of fine  
droplets

↓ Thermal energy  
(2000-6000)

Solvent in droplets  
get evaporate

↓ leaving

Fine residue

↓ which are  
Converted in to  
neutral atoms

↓  
Neutral atom absorbed  
specific wavelength of  
radiation from hollow  
cathode lamp

↓  
measurement of intensity  
of radiation absorbed  
by using photometric  
detector.

## Instrumentation of AAS -

Components of AAS - are hollow cathode  
lamp (light source)

burner

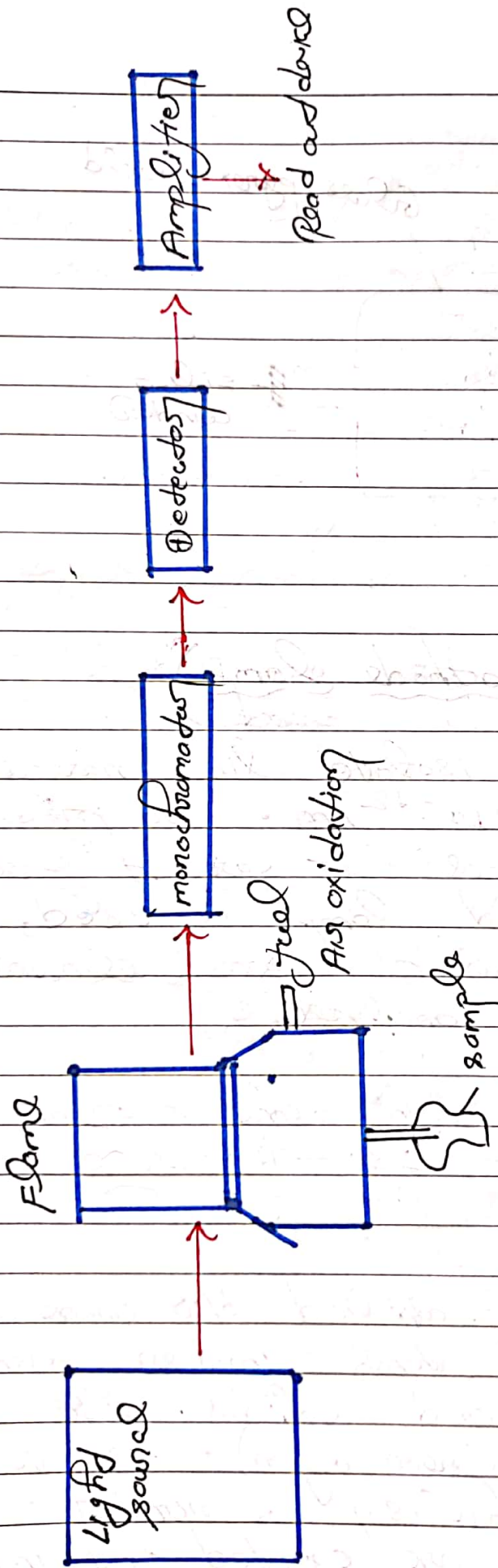
chopper

monochromator

Detector

Amplifier

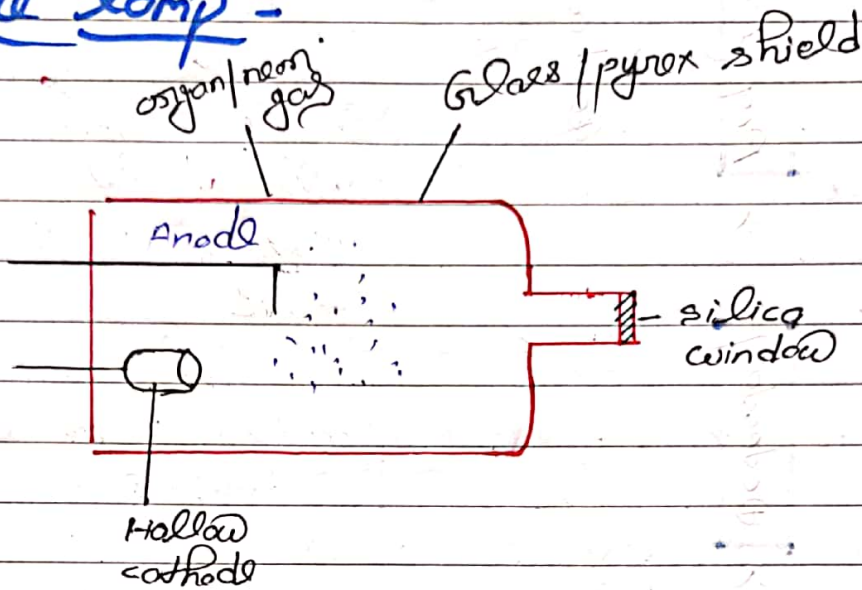
Read out device



Read and drive



# Hollow cathode lamp -



(Hollow cathode lamp).

monochromator can not isolate lines narrower than  $10^{-8}$  to  $10^{-12}$  nm. To produce the narrow lines of correct frequency, hollow cathode lamp is used, containing a vapour of same element as that being analysed.

H.C.L. is filled with neon or argon at pressure of  $130 - 700$  pascals (1-5 torr).

When current is applied b/w anode and cathode, metal atoms immerse from hollow cathode and collide with filler gas (neon, argon). Due to these collisions a number of metal atoms are excited and emit

SWOT

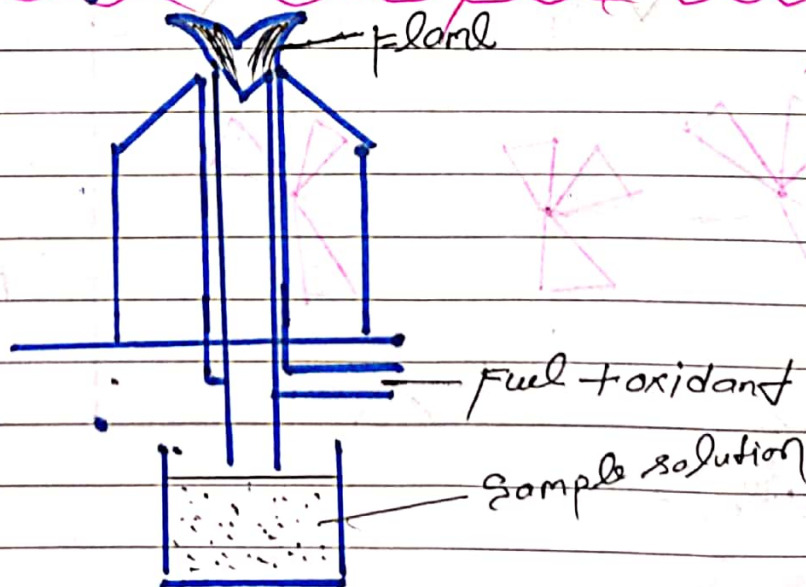
characteristic radiation. This radiation is absorbed by neutral atoms of the same elements in ground state, which occur in the flame when sample sol<sup>n</sup> is spray

Demerit in AAS is, for determination of every element separat H.C.D is required

Burner - in AAS. Automation (bulk liquid  $\rightarrow$  breaking up into small droplet) of sample solution is required.

There are different burners available  
 $\rightarrow$  Total consumption burner  
 $\rightarrow$  Laminar flow (premix) burner

- Total consumption burner -



SWOT



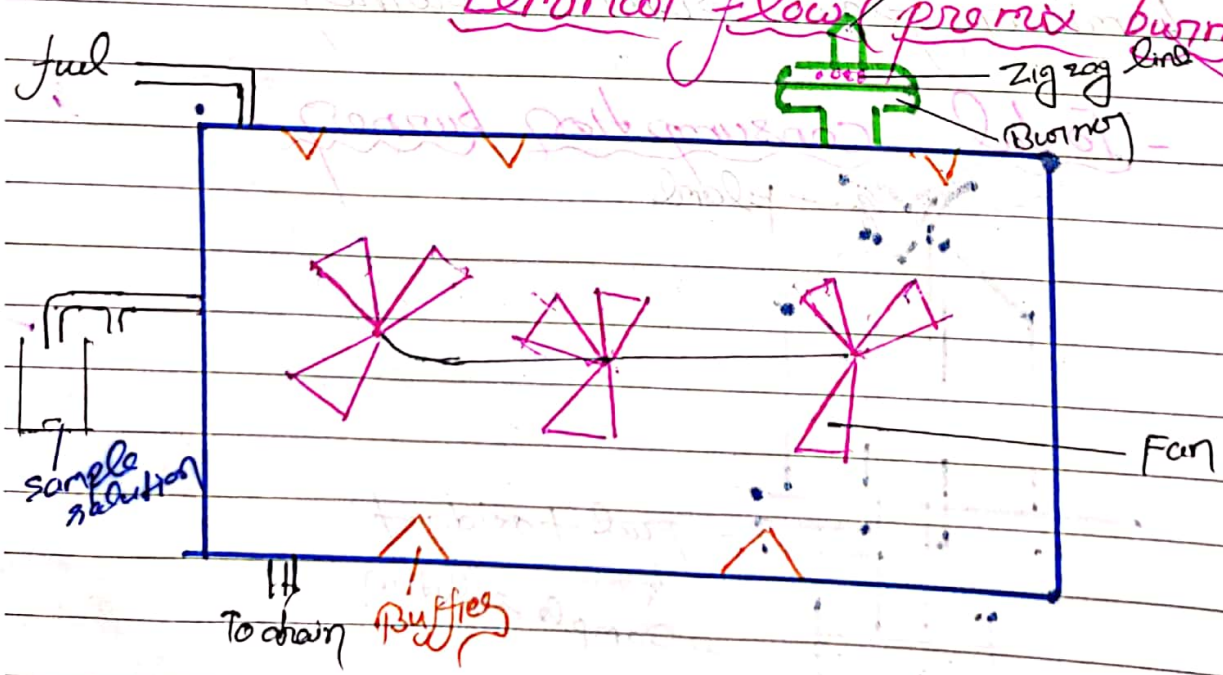
Sample solution  
 ↓ Pass through  
 capillary  
 ↓ by high pressure of  
 Fuel + oxidant  
 ↓ Burn at burner tip

Advantage:- Simple design  
 Consume indise complete sample

Disadvantage:- No uniform droplet size  
 Homogeneous flame is not obtain

Fluctuation in flame intensity

Laminar flow premix burner



Sample sol<sup>n</sup> + fuel + oxidant

↓  
mixed

↓  
Burn at burner tip

This type of burner system has 3 compartments -

i) Mixing chamber - made up of steel casting lined with plastic to avoid corrosion

ii) Burner head - made up of titanium which can stand very high temperature.

iii) nebulizer - nebulizer creates an "aerosol from liquid sample"

To maintain a sample uptake rate of 1 to 5 ml/minute

Advantages - uniformity in the flame intensity

Disadvantages - more complex structure



# Fuel and oxidant

Temp of flame

Low - No oxidation of residual atoms  
High - Ionization of atoms

Desired Temperature

Given

Total combination of oxidant fuel

Temp. of the flame is important activated by

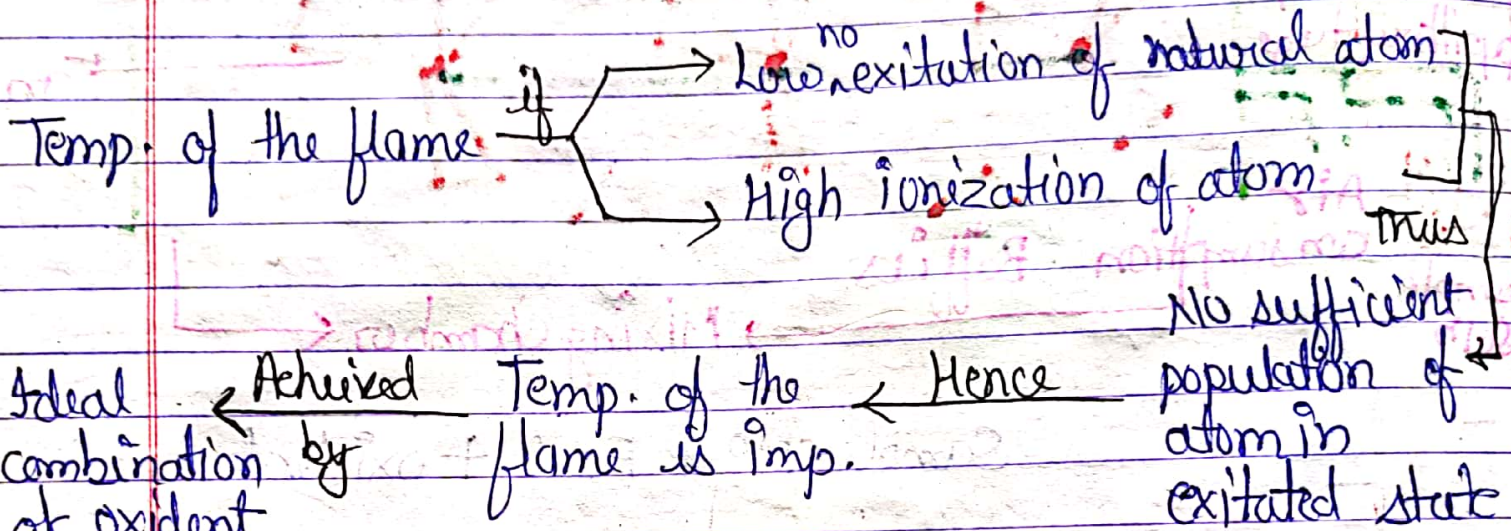
No sufficient population of atoms in excited state

Hence



Use of oxid.

Fuel & Oxidants :-



Gives → Desired Temp.

Fuel	Flame Temp.	
	Air	Oxygen
Propane	2100°C	2800°C
Hydrogen	1900°C	2800°C
Acetylene	2200°C	3000°C



### 3. Chopper :-

Present in earlier  
Instrument

Rotate like fan

Allow alternate  
radiation

From

Flame or HCl

Both

↓ Produces

Pulsating current  
(Signal)

Measured by Photo-  
metric detector

(Ex:- Photocell, Phototube  
Photomultiplier)

### NOTE

In modern instrument, flameless techniques  
(Electrothermal technique) are used



**NOTE** ①

Process occurs  
in Graphite  
furnace  
Furnace

Few microliters sample  
Injected in Graphite tube

~~Atomization through hole~~

↑se tube temp

Sample atomized

Radiation passes from one end  
of tube

Excites the analytes

Absorbed fraction  
↓ measured  
Detector





## NOTE

### ② Types of Atomization :-

1- Flame Atomizers :-

Ex:- Nebulizer

2. Electrothermal Atomizers :-

Ex:- Graphite furnace

3. Glow discharge atomizers :-

Ex:-

4) Hydride atomization

5) cold vapour atomization

4. Monochromator :-

- It select given absorbing line from spectrum
- line emitted from HCL.
-

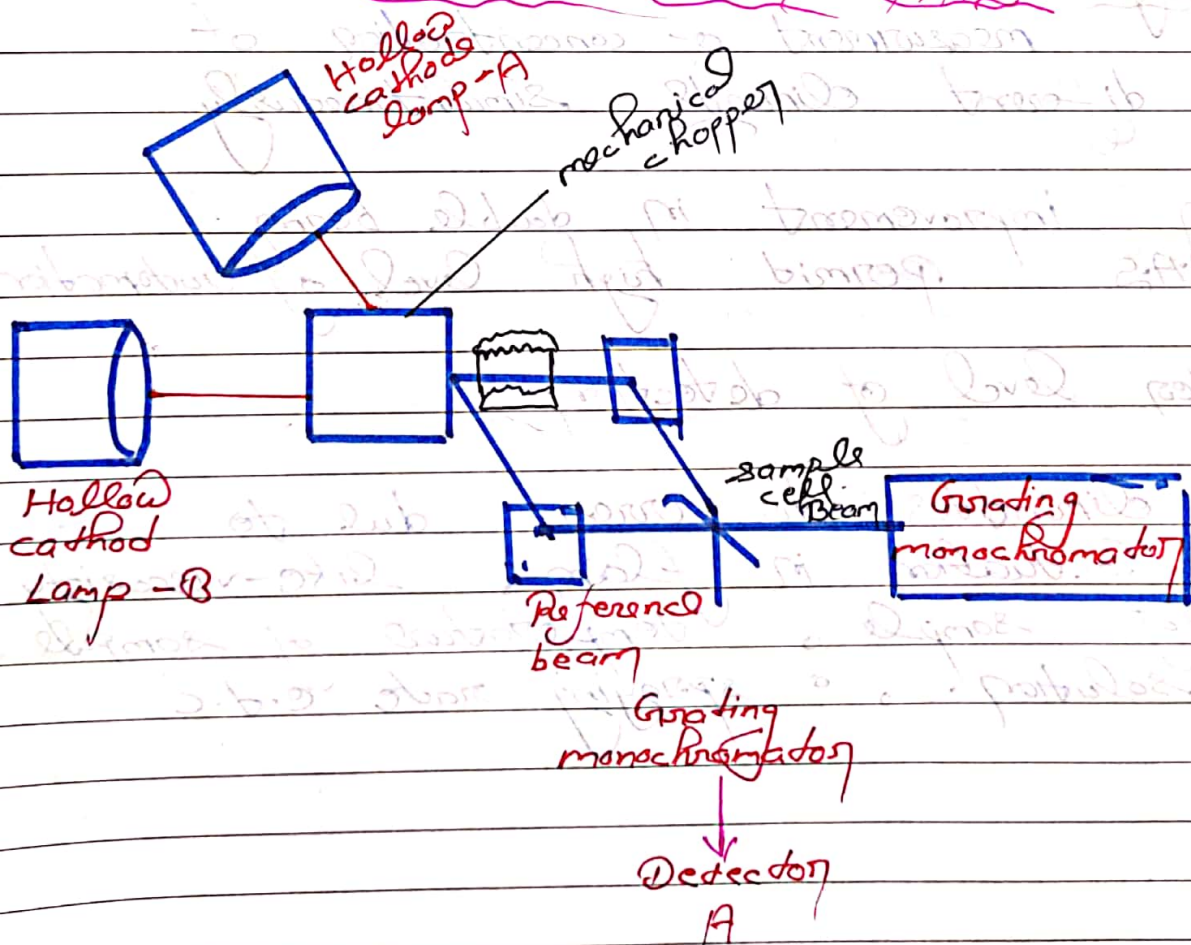


It displays the absorption spectrum as well as the absorbance at specific wavelength (similar to UV spectroscopy).

→ Types of A.A.S -

- > Single beam AAS
- > Double beam AAS

- Double beam A.A.S -



The two channel double beam A.A.S consist of two hollow cathode lamps for the determination of two elements.

The light beam from the source is split in to sample beam and reference beam by mechanical chopper

The reference beam monitors the lamp energy whereas the sample beam reflects sample absorption

→ Advantage -

measurement of concentration of two different elements simultaneously

modern improvement in double beam A.A.S permit high level of automation

Better level of detection

This eliminates the error due to fluctuation in flame, like-viscosity of sample & temperature of sample solution. • • spraying rate e.d.c



Iron  $324.7 \text{ nm}$  interfered with copper  $324.8 \text{ nm}$

## - Interference -

1) - Spectral interference  $\rightarrow$  unwanted signal overlapping.

analyte signal due to other elements or molecules in sample or with signal due to flame or furnace.

2) Chemical interference  $\rightarrow$  caused by any component of sample that decreases the atomisation of analyte to certain extent.

Ex  $\rightarrow$  Sulphate and phosphate ion decreases the atomisation of calcium ion by forming non volatile salt.

3) Ionisation interference  $\rightarrow$  Ionisation of analyte atoms decreases the concentration of neutral atoms.

4) Scattering effect  $\rightarrow$  this occurs due to the presence of high concentration of interfering element.

## n Application of AAS $\rightarrow$

AAS is mainly used for quantitative analysis. It is not used for qualitative analysis, since unless separate lamps are used, it is not possible to

to identifying various element present in the given sample

The various applications are

→ elements like - Iodine in carbencillin sodium can be determined quantitatively (2.48 nm)

→ Estimation of trace element in biological fluid (blood, urine e.t.c)

→ Estimation of elements like - copper, nickel, and zinc in food product.

→ Estimation of lead in calcium carbonate, petrol e.t.c

→ Estimation of zinc in zinc insulin injection

Estimation of magnesium, zinc etc in blood

→ Estimation of elements in soil sample, water supply, effluents, ceramic e.t.c

→ Estimation of mercury in thiomersal solution

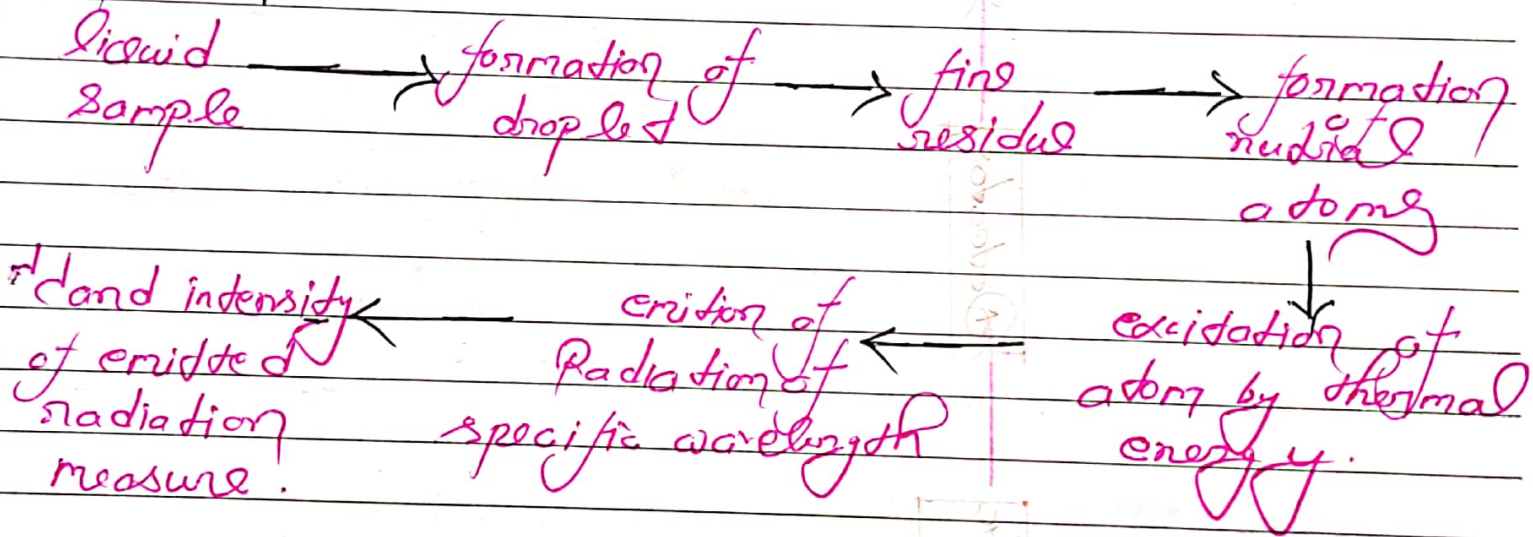


# Flame photometry

(Flame emission spectroscopy)

in flame photometry, the neutral atom are involved in the emission of radiation when introduced in to the flame.

Principle ->



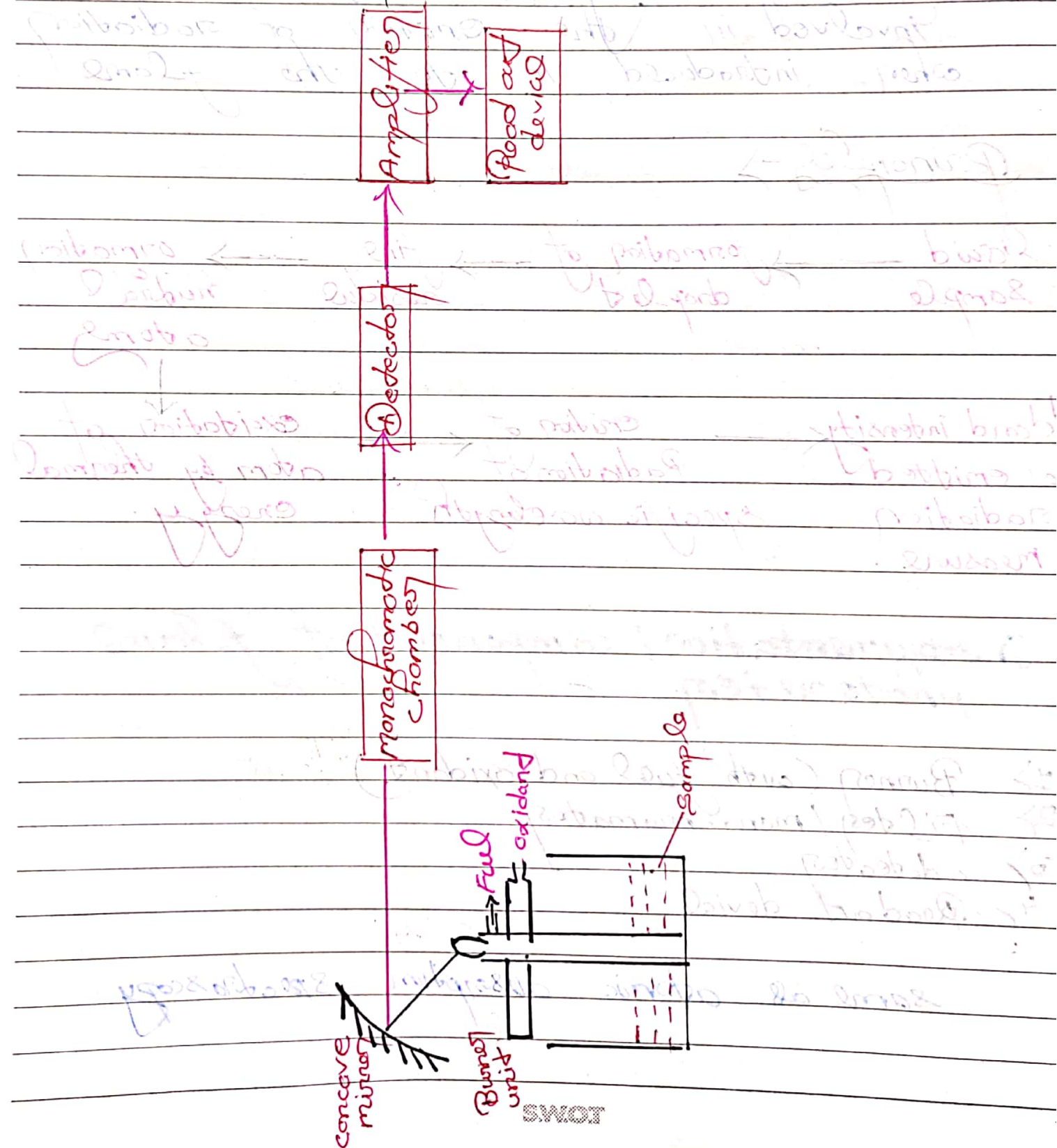
## Instrumentation / component of flame photometer

- 1> Burner (with fuel and oxidant)
- 2> Filter / monochromator
- 3> detector
- 4> Read out device

same as atomic absorption spectroscopy.

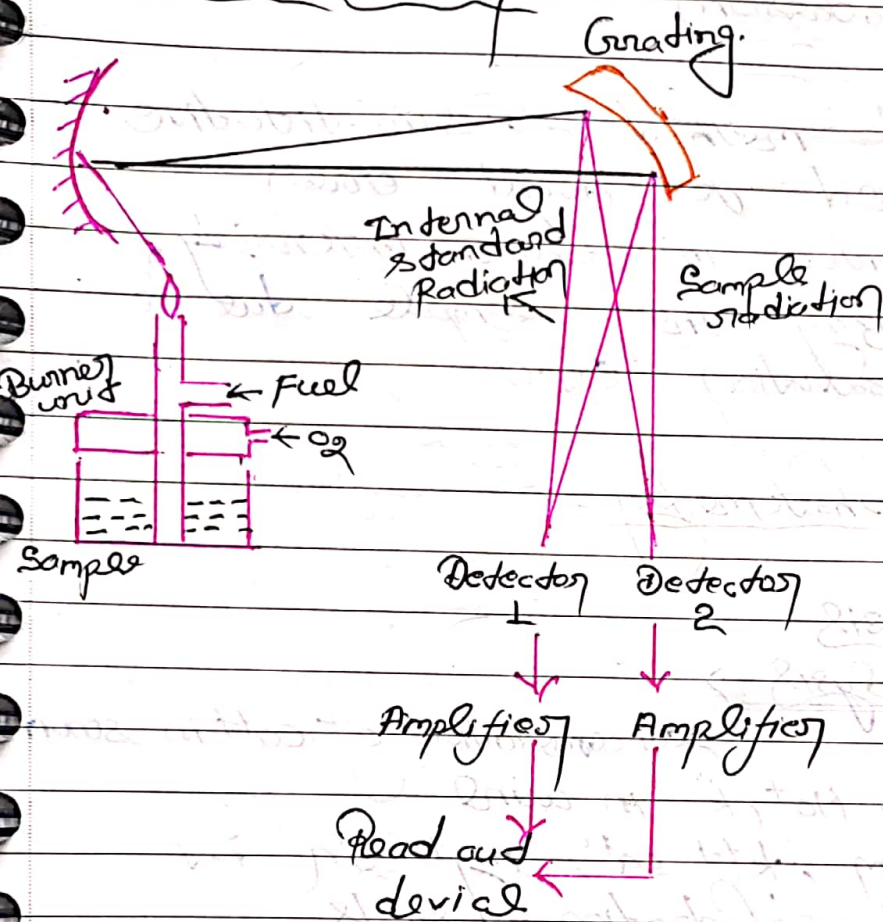
# Types of flame photometer ->

- 1) Single beam flame photometer
- 2) Double beam flame photometer.





# Double beam



In this, the radiation from the flame passes through a different filter or monochromator and the resulting beams fall on to two different detectors.

The double beam is used to monitor a sample element and internal standard element (a second substance used in quantitative analysis by internal standard method).  
For example, Lithium is used as internal standard in the estimation of

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calcium, sodium, potassium

internal standard method of quantitative analysis is used to avoid errors like - fluctuation in flame intensity, errors in atomizing the sample due to high viscosity of solution etc.

### Application of flame photometry -

For qualitative analysis

For Quantitative analysis →

- concentration of  $Ca^{2+}$ ,  $Na^{+}$ ,  $K^{+}$  in urine
- amount of sodium,  $K^{+}$   $Ca^{2+}$ ,  $Mg^{2+}$  in i.v fluids, oral rehydration salts

- Assay of Potassium chloride in syrup
- concentration of calcium in <sup>serum</sup> syrup for therapeutically drug monitoring