

# Atomic Absorption Spectroscopy (AAS)

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PowerPoint

# What is (AAS)?

AAS is an analytical technique used to determine how much of certain elements are in a sample

Atomic absorption spectroscopy (AAS or AA spectroscopy) is one of the earliest elemental analysis techniques to be commercially developed.

It finds immense applications in the analysis for trace metals in soils, lakes, rivers, oceans, and drinking water, pharmaceuticals, foods and beverages, geological and mineralogical samples, petroleum products, biological fluids and specimens and forensic analysis.

# Pictures of AAS



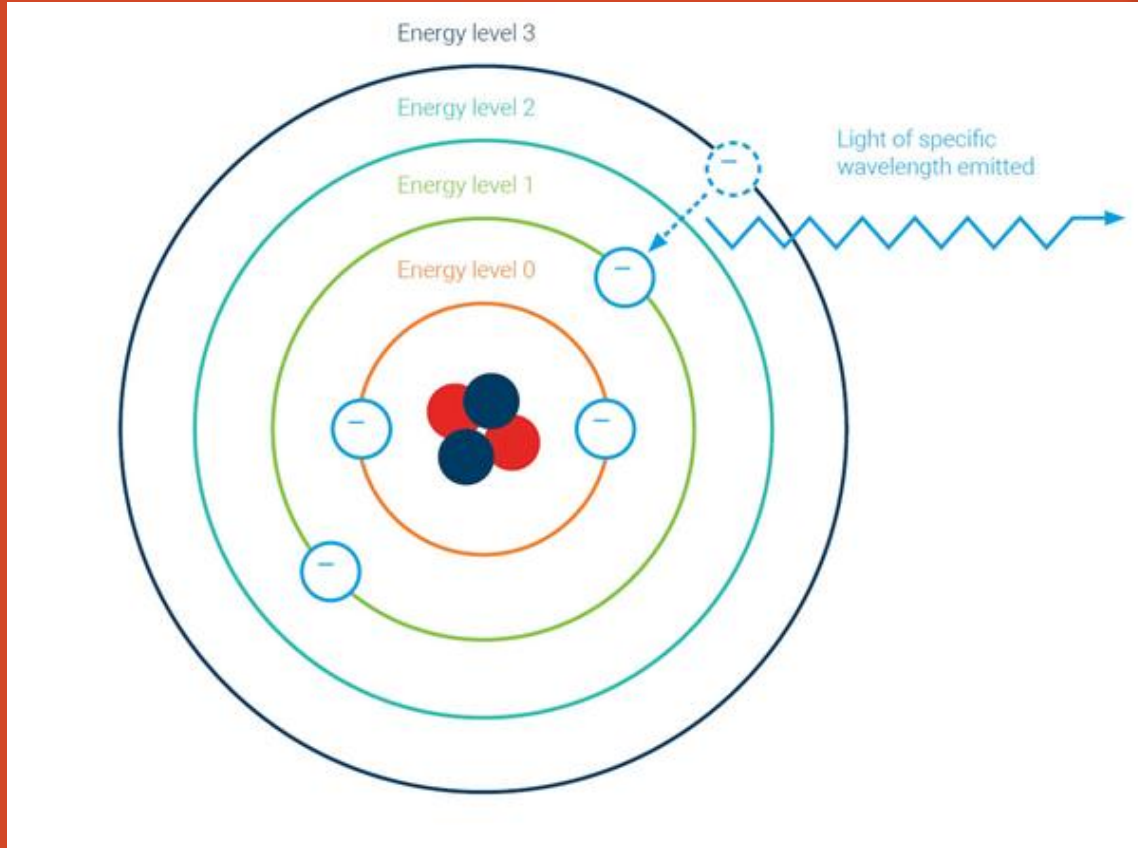


# History about AAS



Alan Walsh was the originator and developer of the atomic absorption method of chemical analysis, which revolutionized quantitative analysis in the 1950s and 1960s. Atomic absorption provided a quick, easy, accurate and highly sensitive method of determining the concentrations of more than sixty-five of the elements, rendering traditional wet-chemical methods obsolete. The method has found important application worldwide in areas as diverse as medicine, agriculture, mineral exploration, metallurgy, food analysis, biochemistry and environmental control, and has been described as 'the most significant advance in chemical analysis' in the twentieth century.

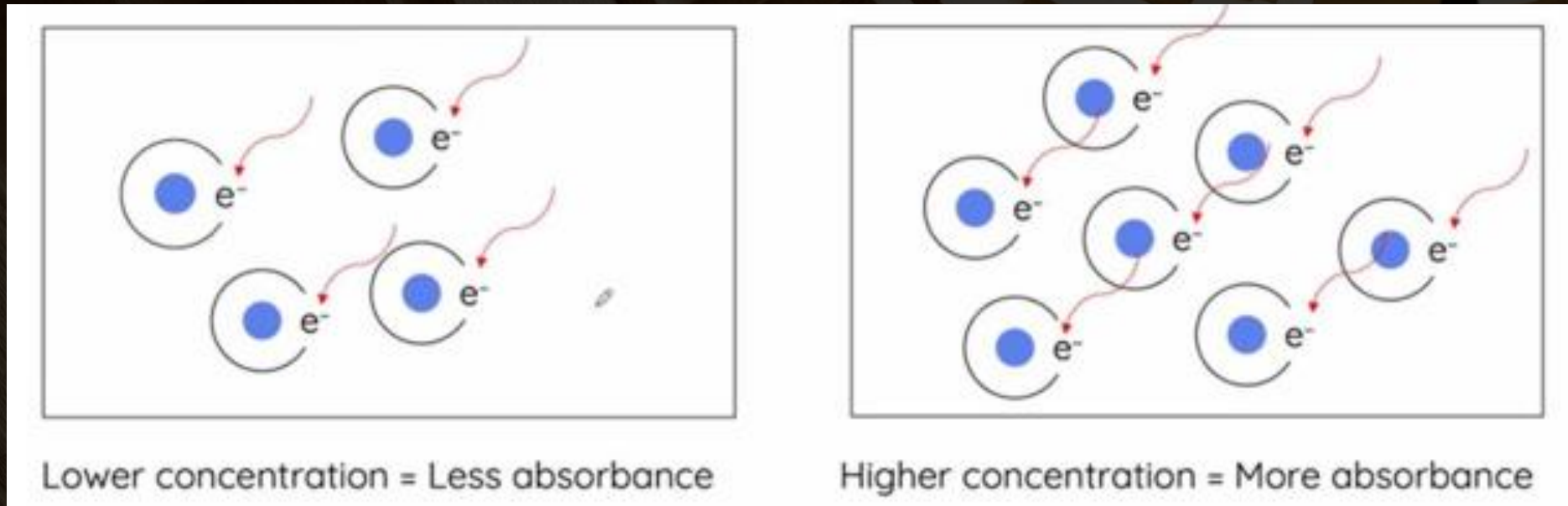
# Atomic Absorption



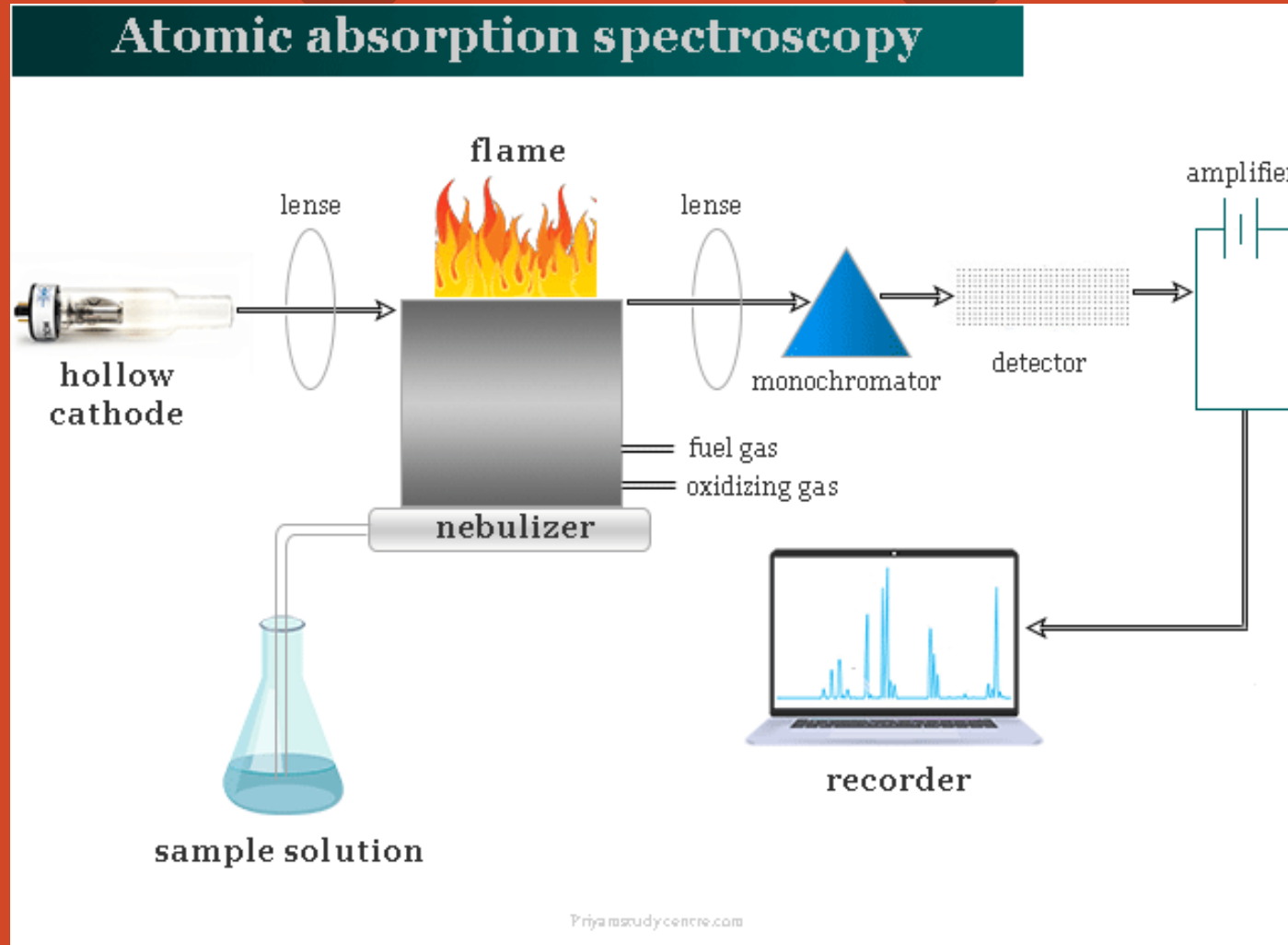
- 1 AAS is an analytical technique used to determine how much of certain elements are in a sample. It uses the principle that atoms (and ions) can absorb light at a specific, unique wavelength. When this specific wavelength of light is provided, the energy (light) is absorbed by the atom. Electrons in the atom move from the ground state to an excited state. The amount of light absorbed is measured and the concentration of the element in the sample can be calculated.
- 2 Energy absorbed by electrons = energy difference between atomic layers
- 3 Absorbed wavelength = emitted wavelength



# Relationship between concentration and absorption



# How the device works

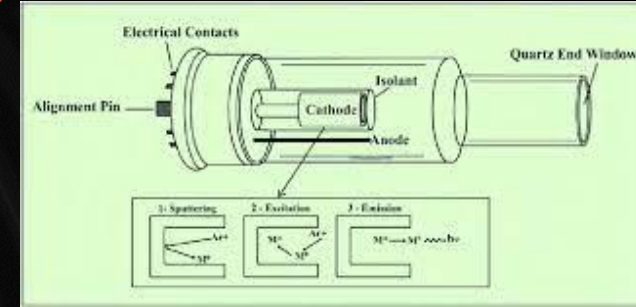


# Hollow cathode lamp

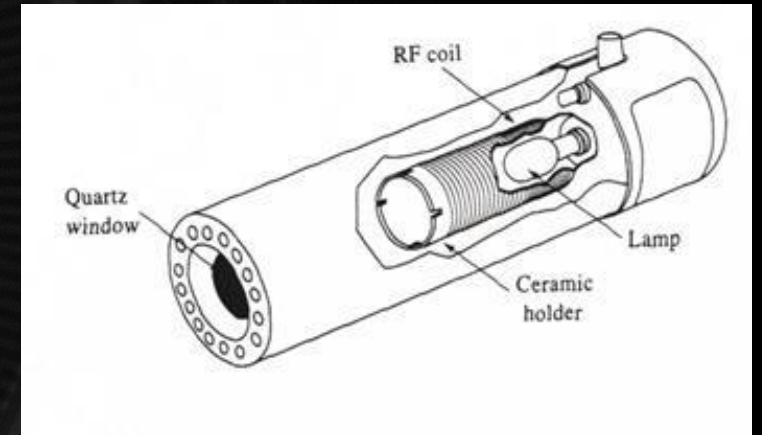
Electromagnetic wave generation

different types:

1 Hollow cathode lamp (HCL)



2 Electrode-less Discharge Lamps



3 deuterium lamp

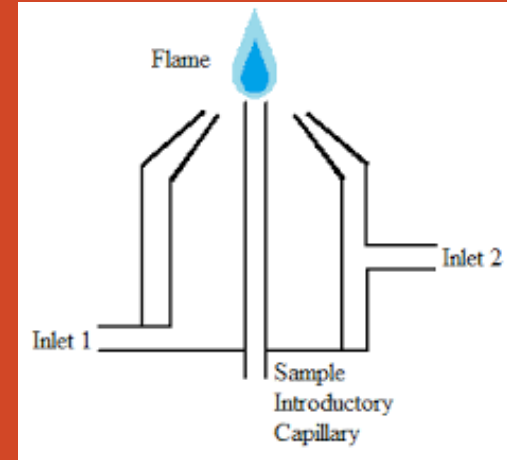




# Atomizer

- Atomic Absorption Spectroscopy requires the conversion of the sample to gaseous atoms, which absorb radiation. In AAS the sample is most commonly introduced as a solution. The solution is drawn in through a small tube and taken to the nebulizer where the solution is broken up into a fine mist (this is similar to an aerosol can). The fine mist is carried to the atomizer, such as a flame, by a carrier gas. When the mist reaches the flame, the intense heat breaks up the sample into its individual atoms. This final process is called atomization.

the heat of the flame produces the free atoms.



# Flame Atomizer

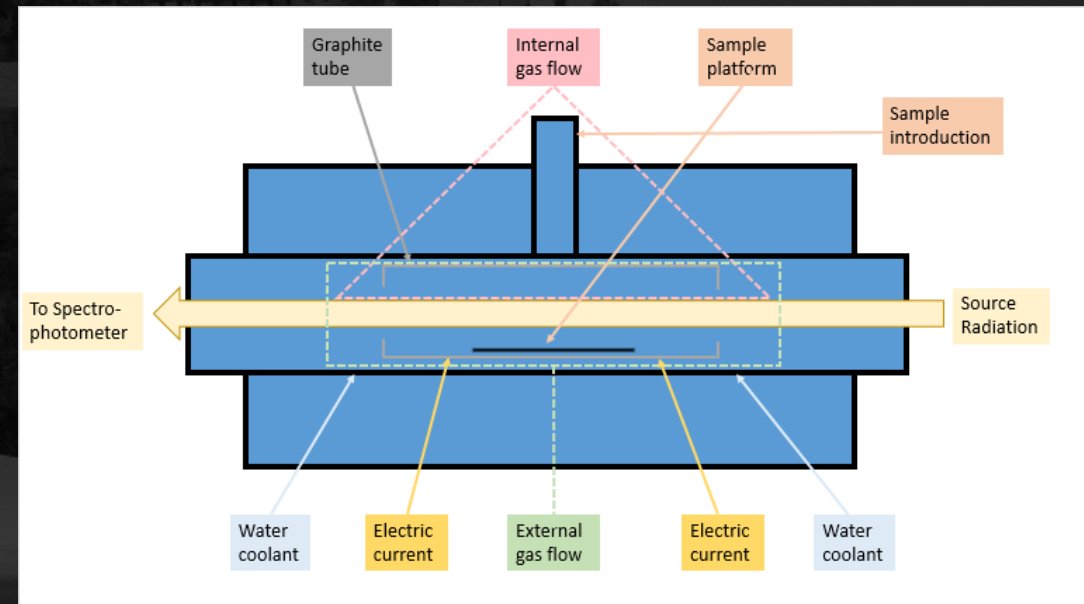
- In a flame atomizer, **a solution of the sample is nebulized by**
- **a flow of gaseous oxidant, mixed with a gaseous fuel**
- **and carried into a flame where atomization occurs.**
- As shown in Figure, a complex set of interconnected processes
- then occur in the flame



# Electrothermal atomizer

- 1 During electrothermal atomization, a sample goes through three phases to achieve atomization. First, the sample is dried at a low temperature. Then the sample is ashed in a graphite furnace (discussed below), followed by a rapid temperature increase within the furnace where the sample becomes a vapor containing atoms from the sample. Absorption is measured above the heated surface where the sample was atomized.
- 2 A graphite furnace is made up of a graphite tube open at both ends with a hole in the center for sample introduction. The tube is encased within graphite electrical contacts at both ends that serve to heat the sample. A supply of water is used to keep the graphite furnace cool. An external stream of inert gas flows around the tube to prevent outside air from entering the atomization environment. Outside air can consume and destroy the tube. An internal stream of inert gas flows through the tube, carrying away vapors from the sample matrix.

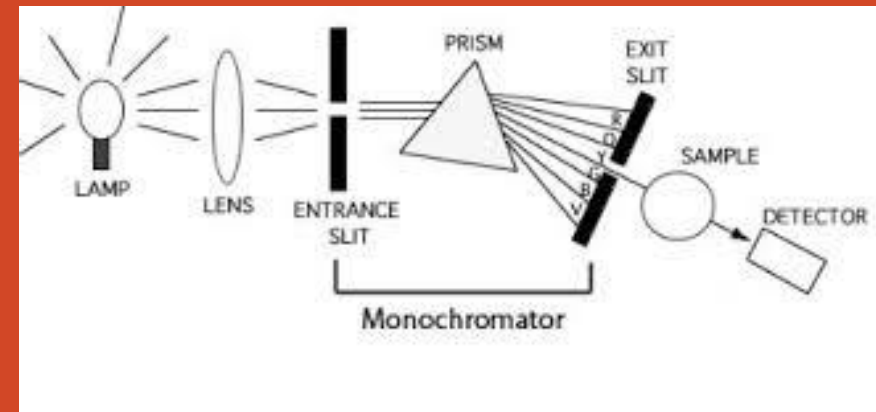
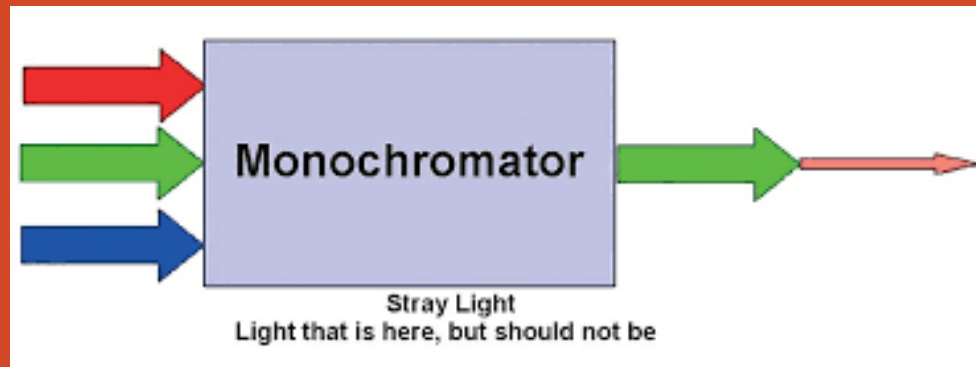
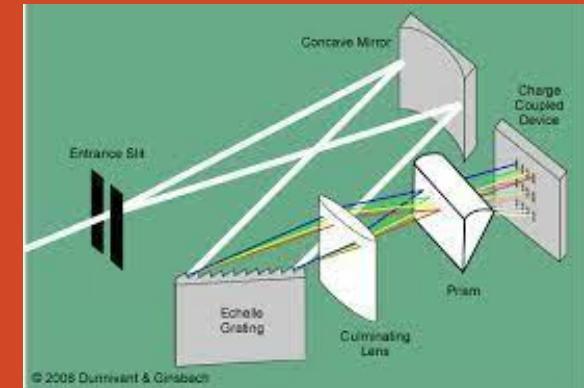
An electrothermal atomizer is here narrowly defined as **a device which is heated, to the temperature required for analyte atomization, by the passage of electrical current through its body.**





# Monochromator

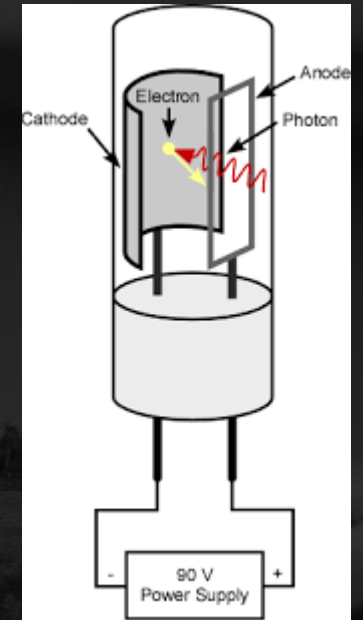
Monochromator is a vital optical component of the Atomic Absorption Spectrometer. It plays the function of isolating the wavelengths of interest from the broad spectrum of wavelengths emanating from the hollow cathode lamp



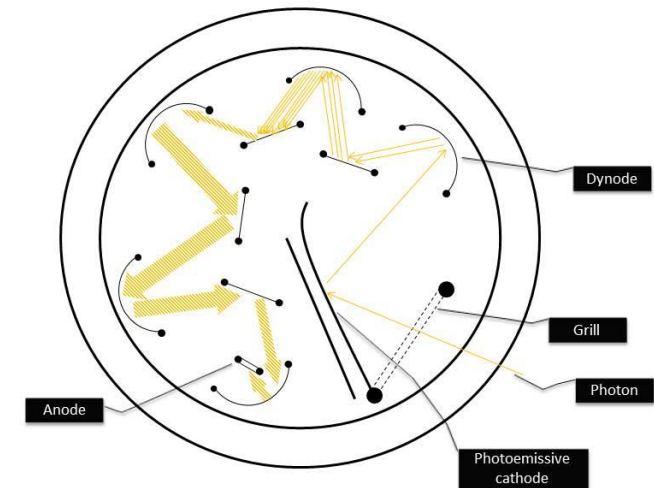
# Detectors

A detector can be a mechanical, chemical, or electrical device that measures the change of a variable in its environment. In Atomic Absorption Spectroscopy, the amount of radiation that passes through a sample is measured and quantitatively described by transmittance. As light passes through a sample, power is attenuated as it is absorbed by the analyte in the sample

A photomultiplier tube (PMT) is used for the measurement of low radiant power. The schematic of a PMT is similar to that of a traditional phototube. In a traditional phototube there are two electrodes, an anode and a cathode. When voltage is applied to the electrodes, the photons hit the cathode and generate electrons. The electrons flow to the anode. The anode generates a photocurrent that can be measured. A diagram of a traditional phototube is shown to the right.



Photomultiplier tube





# How we analyze?

- The basis of analysis is the Beer-Lambert law
- The Beer-Lambert Law is a relationship between the attenuation of light through a substance and the properties of that substance
- In AAS we have the incident intensity ( $I_0$ ) and the transmitted intensity ( $I$ ). So we can calculate the absorbance. We also have the  $\epsilon$  and  $l$ .
- Finally we can calculate the concentration of interested analyte.

$$A = \log_{10} \frac{I_0}{I}$$

$$A = \epsilon cl$$

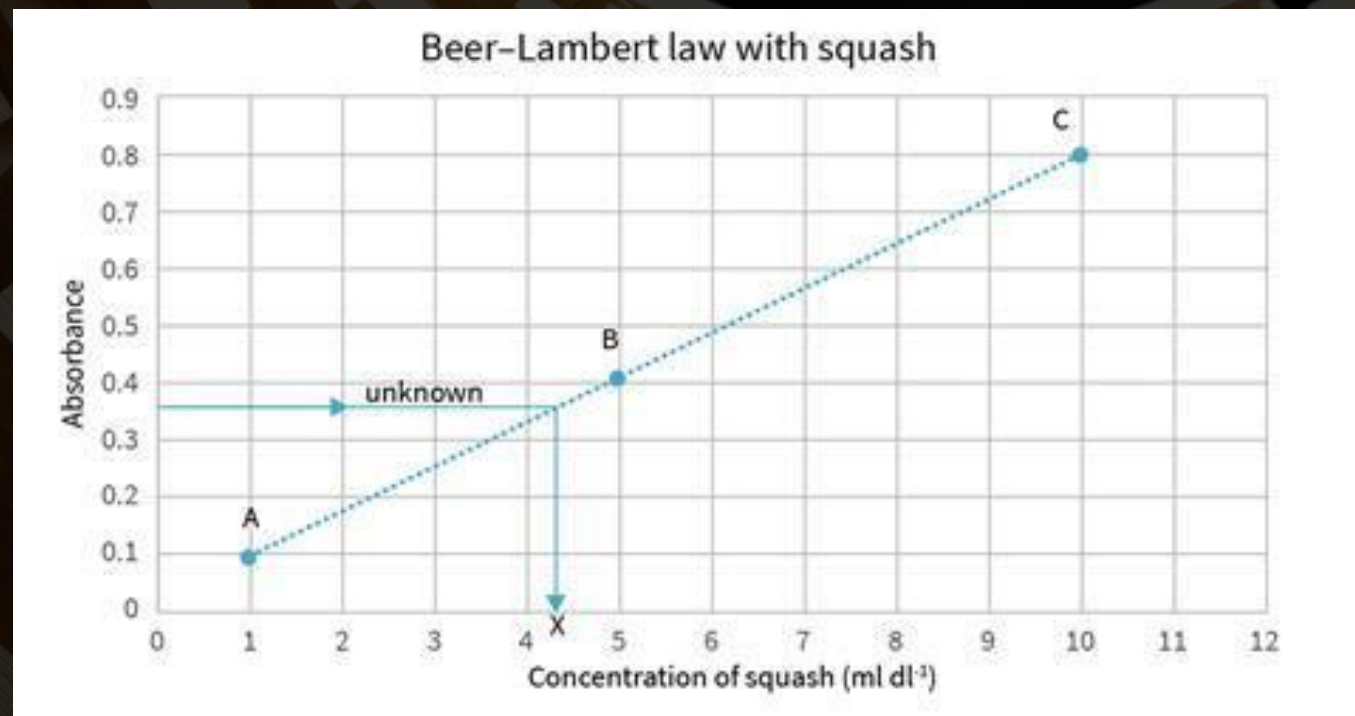
$A$	Absorbance	
$\epsilon$	Molar absorption coefficient	$M^{-1}cm^{-1}$
$c$	Molar concentration	$M$
$l$	optical path length	$cm$



# Calibration curve

- A, B and C are standard solutions of our interested analyte.
- The Beer-Lambert law states that there is a linear relationship between the concentration and the absorbance of the solution, which enables the concentration of a solution to be calculated by measuring its absorbance.

Note: we can't analyze high concentration elements with AAS. Because the overlap of the signals causes that the calibration curve miss its linear form. In these cases we should use ICP for its wide dynamic range.





# Standard solutions

The solutions and mixtures are made using materials of the highest purity, and therefore meet the requirements for instrumental analysis by AAS and ICP. All solutions are certified and can be traced to NIST standard reference materials. Solutions are produced according to ISO 17034 in an accredited environment. The solutions are tested in a laboratory accredited to ISO/IEC 17025 and supplied with a detailed, batch-specific certificate of analysis. AAS standard solutions are made from high-quality reagents, and therefore ensure maximum precision during calibration. Accuracy and precision for reliable measurement results.

## Properties:

- Concentration 1000 mg/l (1 g/l)
- Container sizes: 100 ml and 500 ml
- 24 months shelf life for unopened bottle & 12 months after the bottle has been opened



# Background correction

In real world situations, light from the lamps is absorbed by undissociated molecules. Light is also scattered by tiny particles in the solution. This loss of light is called the “background”. A high background reading may falsely indicate atomic absorption. This could result in an incorrect calculation of the analytical result. To combat this, there are several background correction techniques that can be incorporated into AA spectrometer instrument design.



## **Deuterium (D2) background correction technique:**

- D2 background correction is frequently used with flame AA spectrometer instrumentation. It uses a D2 lamp to measure the non-atomic absorption and is effective between 190 to 425 nm.
- To perform deuterium background correction, the continuum signal from the D2 and the HCL resonance light sources are rapidly alternated.
- The signal from the D2 lamp is subtracted from the HCL signal. This gives a background-corrected signal.

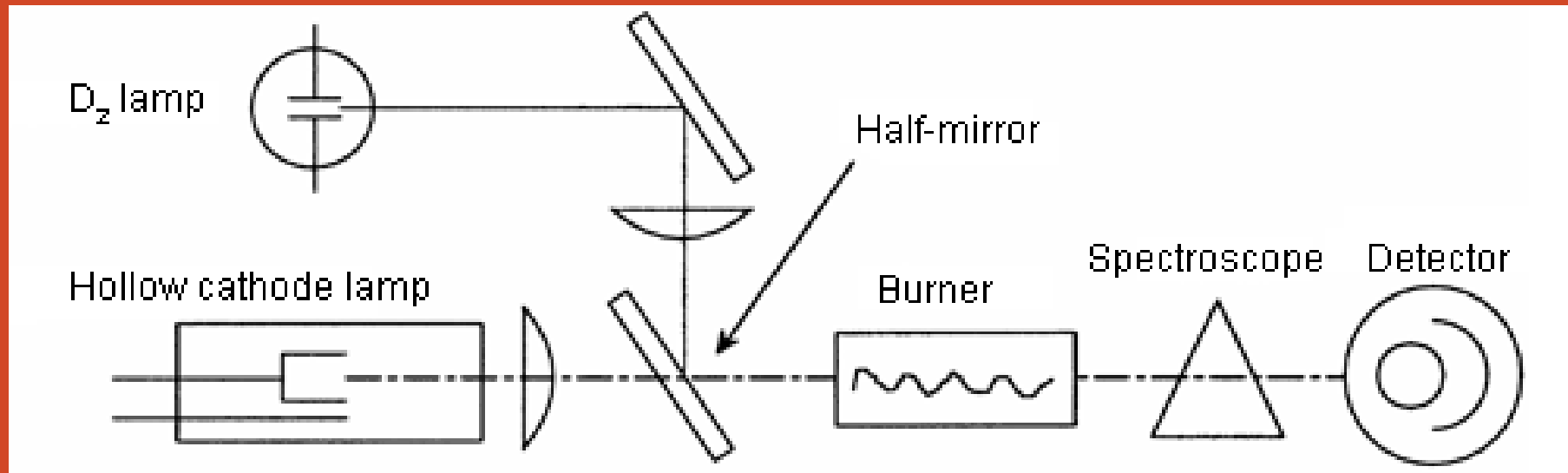
Hollow cathode lamp signal = atomic absorption + background

Deuterium lamp signal = background only

Electronically processed signal = atomic absorption only

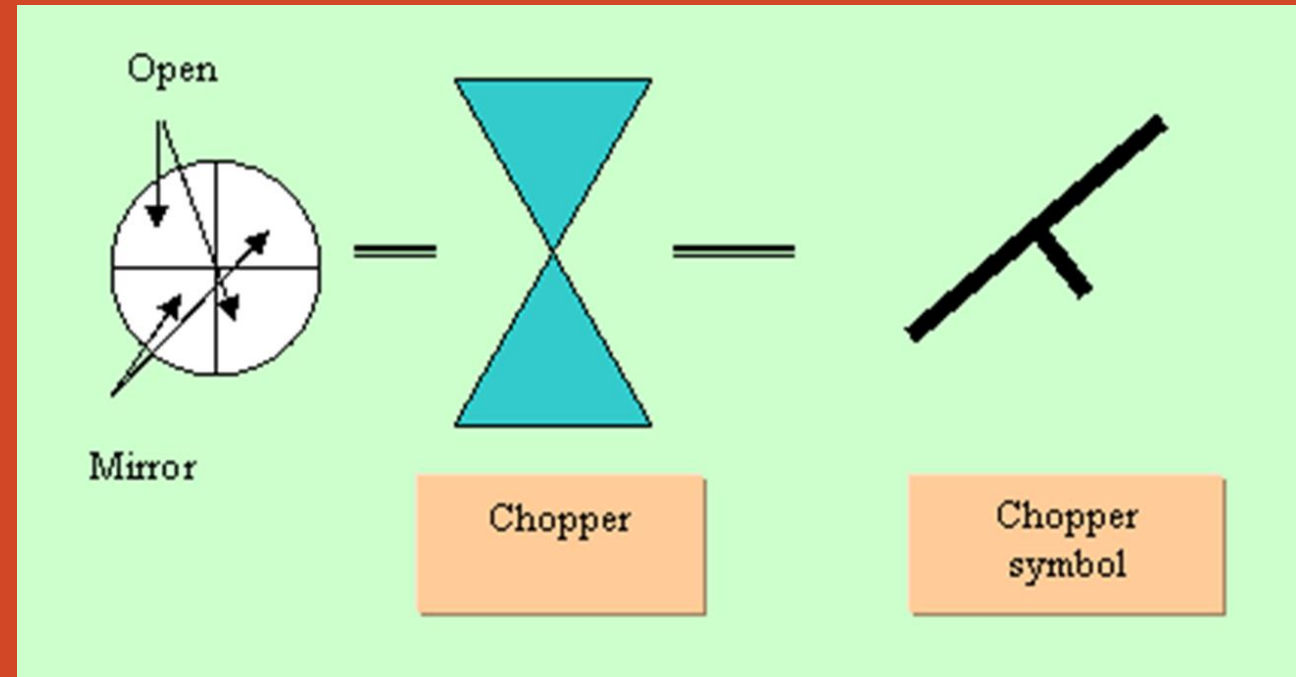


# Deuterium (D<sub>2</sub>) background correction technique



# Chopper

The function of the chopper is to chop the light leaving the source so that when the incident beam hits the chopper at the solid surface, the beam will be blocked and detector will only read the emitted signal from the D2 lamp. As the chopper rotates and the beam emerges to the detector, the detector signal will be the sum of the transmitted signal plus that emitted from the D2 lamp. The signal processor will be able to subtract the first signal from the second one.



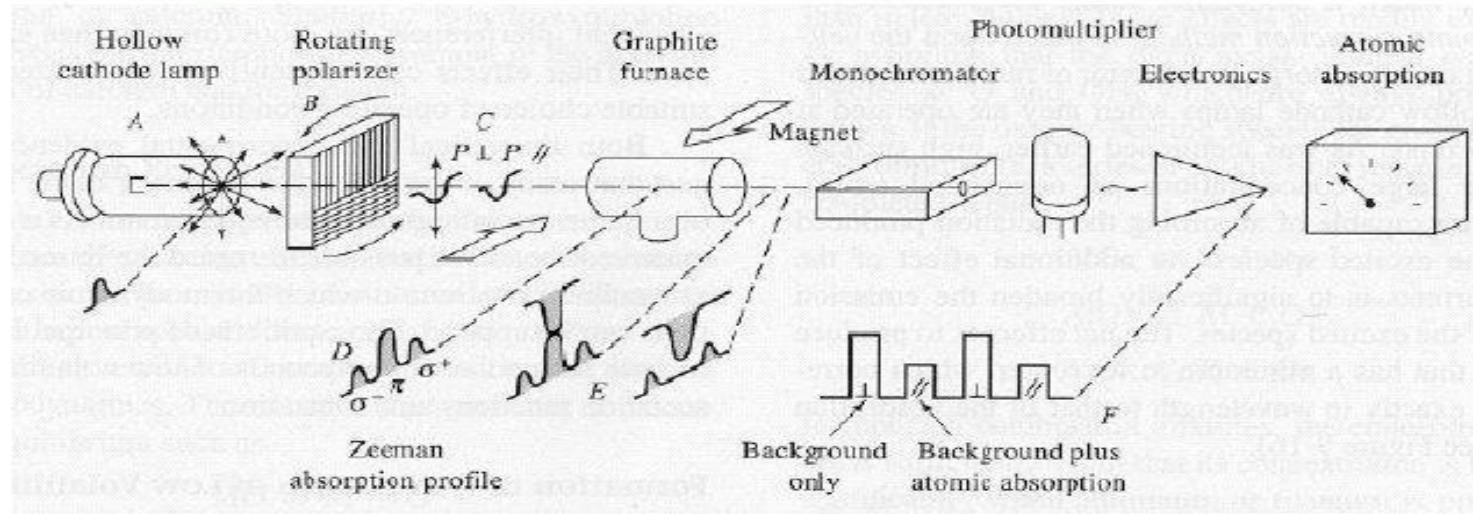
# Zeeman effect correction

- In a Zeeman Effect instrument, a magnetic field is applied to atoms, splitting the electronic energy levels. This causes multiple absorption lines to be present, and the sum of these absorption lines is equal to the original line that formed them. In it, the absorption line is split into two components: the pi component, which is present at the original wavelength, and sigma component which is both negatively and positively shifted so that two absorption lines are present.
- Pi lines correspond to radiation that polarized parallel to the magnetic field, while  $\sigma$  lines correspond to radiation polarized perpendicular to the field.
- In a DC Zeeman correction system, radiation from a hollow cathode lamp passes through a rotating polarizer to a furnace surrounded by a permanent magnet. When the radiation from the HCL passes through the rotating polarizer two forms of polarized light are produced: light that is polarized parallel to the field, and light that is polarized perpendicular to the field. When light is polarized parallel to the field, both atomic and background absorption occurs, and when light is polarized perpendicular to the field, only background absorption occurs.
- In an AC Zeeman correction system, a stationary polarizer is present in front of the source, and an electromagnet is present around the furnace. When the magnetic field is off, both the atomic and the background absorption occurs. When the magnetic field is on, only background absorption occurs.



# Zeeman effect correction

## Zeeman effect correction



- B - Rotating polarizer
- E – absorption at // polarization
- Very sensitive correction technique

The Zeeman Effect is preferred over most other forms of background correction, as it tends to be more accurate. AC Zeeman systems tend to be more expensive than DC Zeeman systems, but they are more sensitive and have larger linear working ranges.

## Accuracy

It means correctness and indicates the degree of conformity of the test results with the real value. It is expressed as a percentage and for AAS it is in the range of 0.5 to 5%.

## Precision

It expresses the accuracy and shows the reproducibility of the test. May be the result that every time

We get from the test that it is not correct, but it is equal to the previous value or close to it. In this case, we say that although the test is not accurate, it is accurate. Accuracy is expressed as a percentage and for AAS it is between 1 and 3%

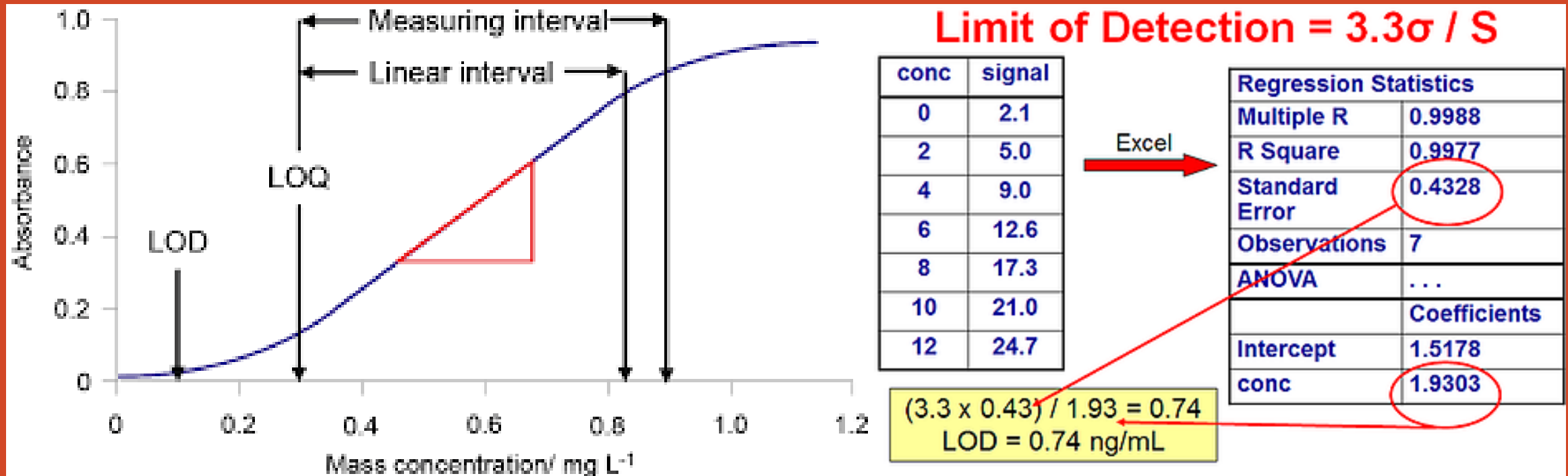
## LOD, LOQ

are terms used to describe the smallest concentration of a measurand that can be reliably measured by an analytical procedure.



# LOD, LOQ

- LoD is the lowest analyte concentration likely to be reliably distinguished which detection is feasible.
- LoQ is the lowest concentration at which the analyte can not only be reliably detected but at which some predefined goals for bias and imprecision are met. ( $LOQ = 10 \sigma / s$ )





# Advantages and Limitations

Advantages	Limitations
Low cost per analysis	Cannot detect non-metals
Easy to operate	New equipment is quite expensive
High sensitivity (up to ppb detection)	More geared towards analysis of liquids
High accuracy	Sample is destroyed
Mostly free from inter-element interference	When we have many elements in our solution, we need more lamps and this can be inappropriate.
Wide applications across many industries	



Mining and  
Geology

agriculture

Environmen  
tal  
monitoring

**Applications**

Oil and  
petroleum

Materials  
developme  
nt

Food and  
beverages

pharmaceu  
ticals

# References

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# Questions

1. What are the LOD and LOQ? and how they determined?
2. What is the duty of the chopper and how it works? describe the structure of chopper.
3. Explain zeeman background correction in AAS. how many types does it have?
4. Why we can't determine the high concentrations with AAS?
5. Why we can just analyze metals with AAS?
6. what is the basis of AAS and how does it work?
7. What is the difference between alan walsh method and the old method for measuring concentration?
8. What are the advantages of flame atomizer compared to the other atomizers?
9. Name the type of AAS lamps and mention their differences
10. what is the function of the detector in AAS?





**Thank you for your attention**