

Atomic Absorption Spectroscopy

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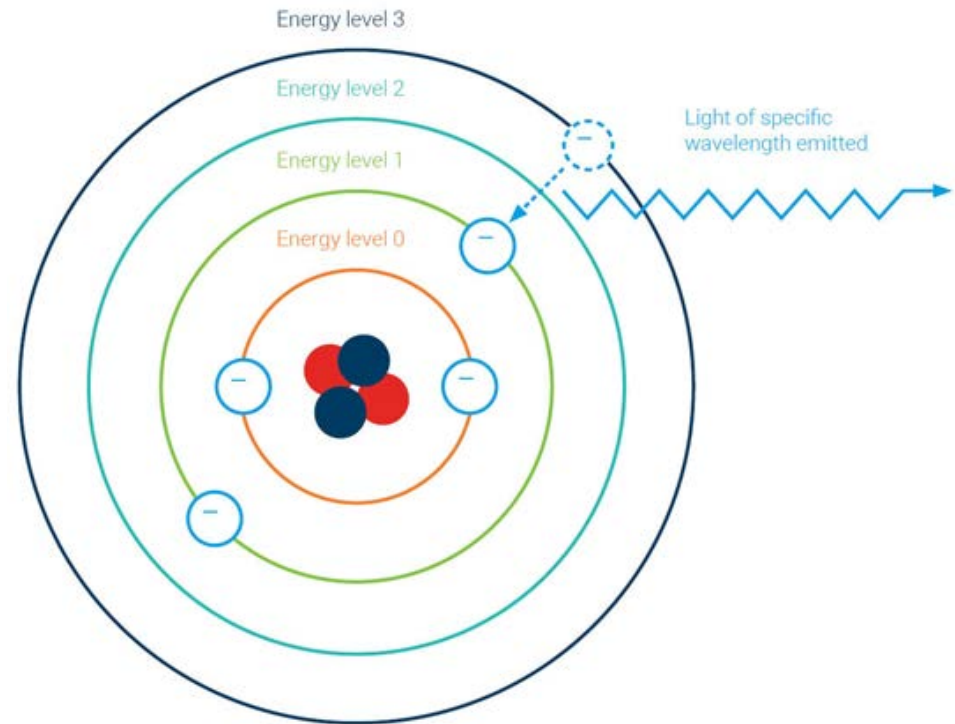
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What is Atomic absorption spectroscopy?

- Atomic absorption spectroscopy (AAS or AA spectroscopy) is one of the earliest elemental analysis techniques to be commercially developed.
- Flame atomic absorption spectroscopy (Flame AAS or FAAS) was developed in 1952 and first commercially released as an analytical technique in the 1960s.
- Since then, the technique has remained popular for its reliability and simplicity.
- AAS is an analytical technique used to determine how much of certain elements are in a sample.

Principle of atomic absorption spectroscopy

- 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.
- The radiant energy absorbed by the electrons is directly related to the transition that occurs during this process.
- Furthermore, since the electronic structure of every element is unique, the radiation absorbed represents a unique property of each individual element and it can be measured.



Working Principle

- A typical atomic absorption spectrometer consists of four main components: the light source, the atomization system, the monochromator and the detection system (Figure 1).
- In a typical experiment, the sample, either liquid or solid, is atomized in either a flame or a graphite furnace.
- The free atoms are then exposed to light, typically produced by a hollow-cathode lamp, and undergo electronic transitions from the ground state to excited electronic states.
- The Beer Lambert law describes the relationship between light absorption and concentration of the element. According to the law, the amount of light absorbed is proportional to the number of atoms excited from the ground state.
- The light produced by the lamp is emitted from excited atoms of the same element that is to be determined, therefore the radiation energy corresponds directly to the wavelength absorbed by the atomized sample.
- A monochromator is placed between the sample and the detector to reduce background interference.
- From here, the detector measures the intensity of the beam of light and converts it to absorption data.

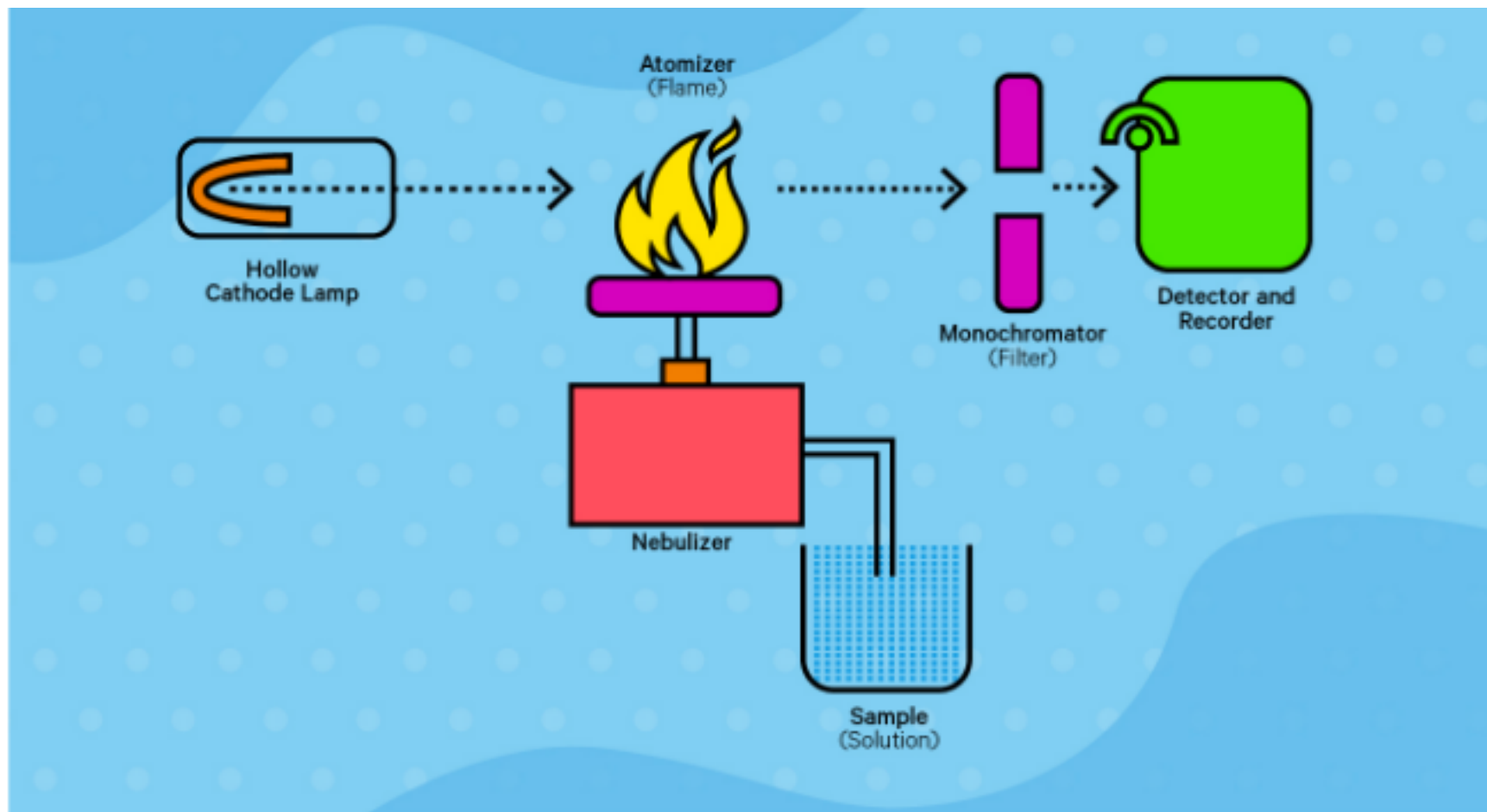


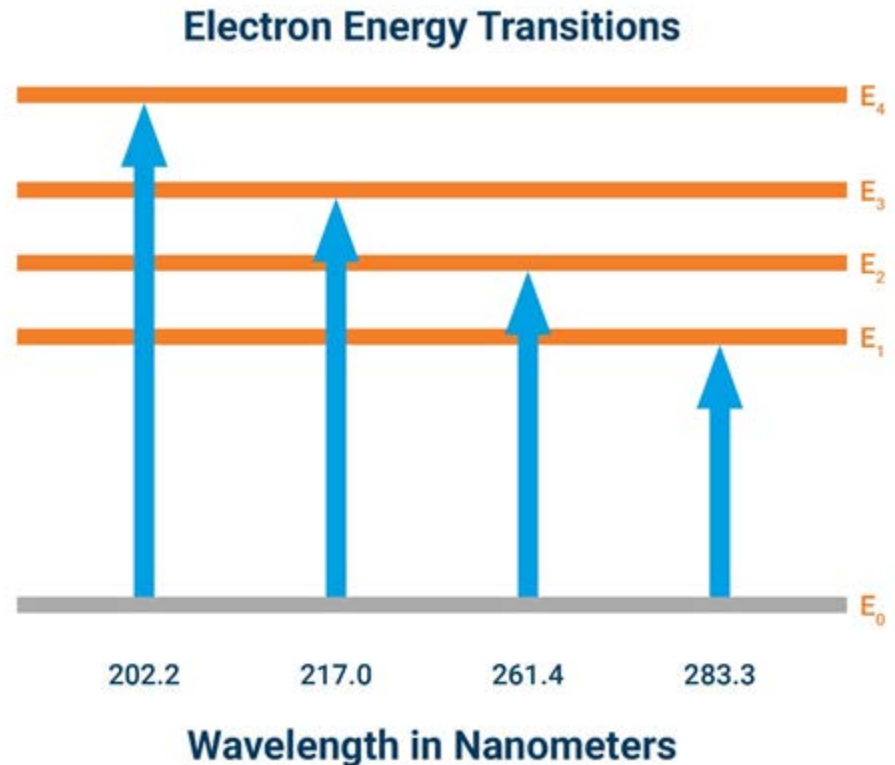
Figure 1: Schematic diagram of a typical atomic absorption spectrometer.

AAS in action, an example of Lead (Pb) Atom

- Energy from the heat of the flame causes atoms to freely dissociate.
- The amount of energy required for the electrons to move between energy levels corresponds to specific wavelengths of light.
- Eg. Moving an electron from the ground state of a Pb atom to the first energy level (E1) requires energy equivalent to light at 283.3 nm.
- It requires more energy to move an electron from the ground state to the second energy level (which is further away from the nucleus).
- For AAS analysis, the wavelength of the ground state to the E1 level is frequently of most interest, as it is the most intense.
- A strong absorbance band gives the best detection limits.
- In samples where the concentration of the element is higher, an alternate wavelength can be used.

...AAS in action, an example of Lead (Pb) Atom

- Only free, ground state Pb atoms in the flame will absorb at 283.3 nm.
- Electrons moving between other energy levels in the Pb atoms will absorb light at different wavelengths.



Instrumentation: Atomizing technique -Flame atomic absorption spectroscopy (FAAS)

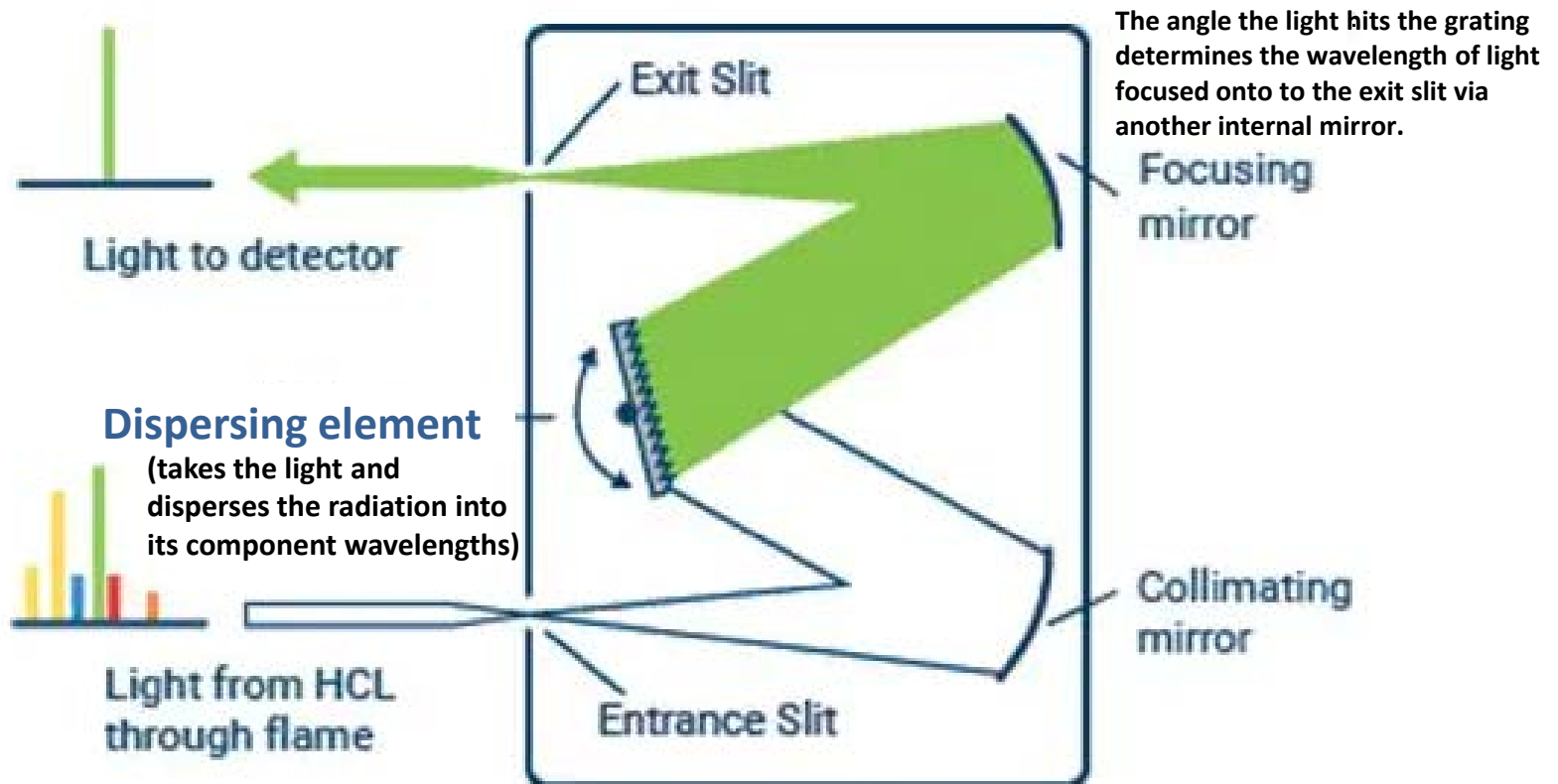
- *A sample introduction system:*
 - The pneumatic nebulizer makes use of the Venturi effect, the principle that fluid flows at a higher velocity through a narrower tube, to accelerate the solution stream.
 - The fluid then impacts a glass bead to create a fine spray of droplets, known as an aerosol.
 - Larger droplets drain to waste, while the fine aerosol is passed up into the spray chamber.
- *The burner (flame) and its associated gas supplies: air-acetylene or nitrous oxide-acetylene:*
 - In flame AAS, the burner converts the aerosol/gas mixture created by the spray chamber and nebulizer, into free, ground state atoms.
 - There are two common gas mixtures that are burnt to fuel the flame. They are air-acetylene (produces a flame ~ 2300 °C) and nitrous oxide-acetylene (~ 2700 °C).
 - The burner is specially designed with a thin slit, that is 5–10 cm long, depending on the type of burner used.
 - The slit defines the length of the flame in the spectrometer where the population of free ground state atoms exist, which determines the sensitivity, according to the Beer-Lambert Law.

...Instrumentation: Flame atomic absorption spectroscopy (FAAS)

- *A light source, the hollow cathode lamp (HCL):*
 - Hollow cathode lamps are filled with an inert ‘filler’ gas at low pressure, usually argon or neon.
 - A metal cathode, coated in the element of interest, is positioned opposite an anode.
 - A high voltage is applied, across the two electrodes, which ionizes the filler gas accelerating ions toward the cathode.
 - The cathode is bombarded by these ions with enough energy that metal atoms from the cathode material are ejected or “sputtered” creating an atom plume.
 - Inside the atom plume, further collisions between metal atoms occurs raising them to an excitation state.
 - When the atoms return to their preferred ground state, radiation is emitted, as light, at the characteristic wavelengths of that specific element.

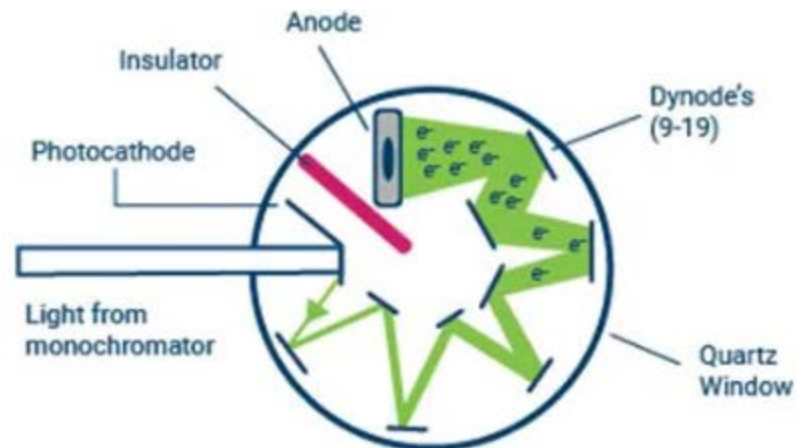
...Instrumentation: Flame atomic absorption spectroscopy (FAAS)

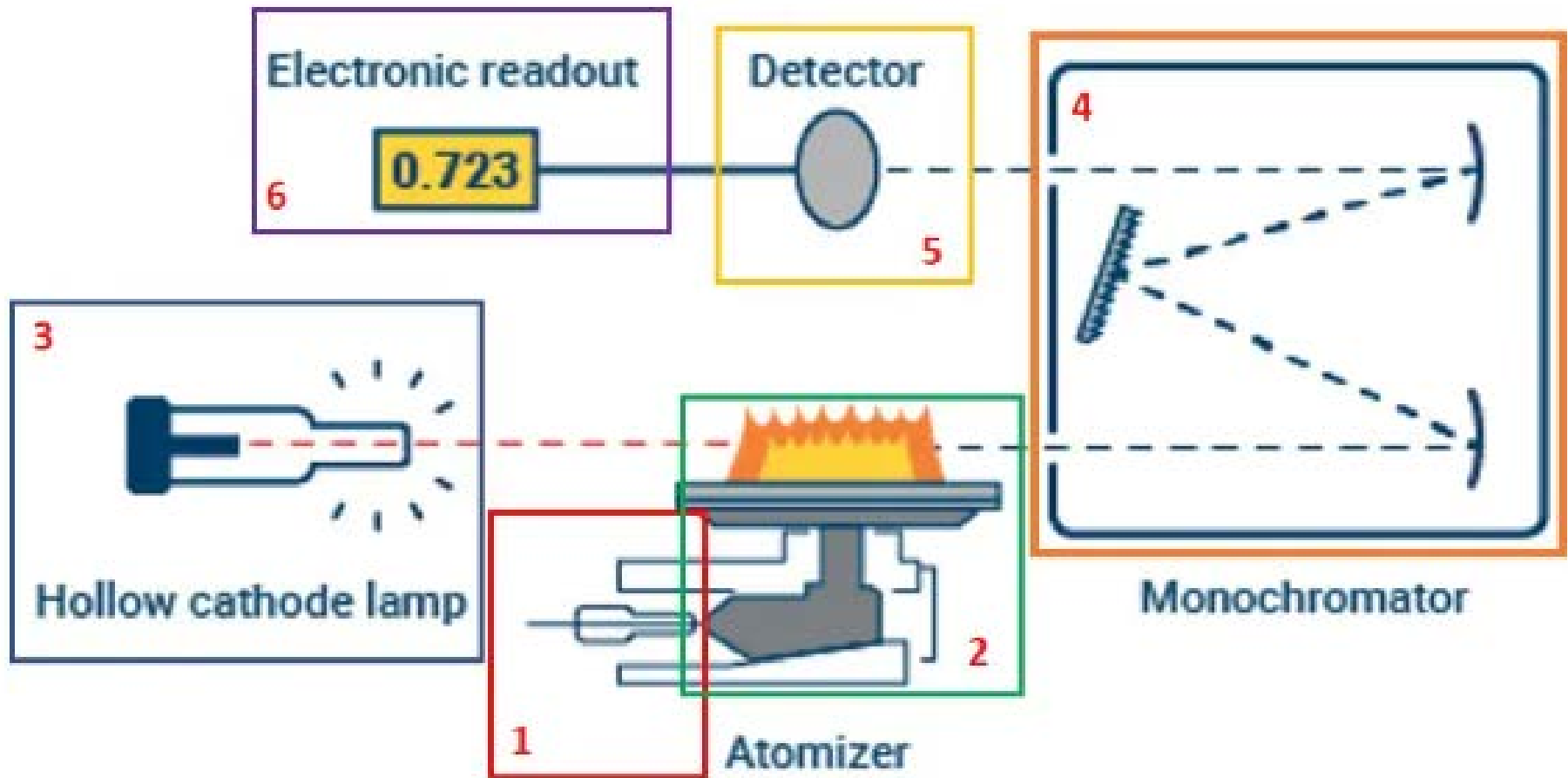
- *A monochromator:*
 - A [monochromator](#) is an optical device with the sole purpose of collecting light containing many wavelengths and isolating (selecting) a narrow wavelength band.



...Instrumentation: Flame atomic absorption spectroscopy (FAAS)

- *An optical detector (photomultiplier tube or PMT):*
 - The light from the exit slit of the monochromator enters the PMT and hits a photocathode, creating an electrical signal.
 - A series of dynodes amplify the signal and then it is collected (measured) on an anode and used to provide a quantitative measurement of absorption.
- *Computerized instrument control, data collection, and analysis:*
 - Specialized instrument control software calculates the concentration of each element in the sample, using the calibration that was performed before the sample analysis.
 - Statistical analysis of results, saving of instrument settings as analytical methods and the generation of reports on the analysis, are all done by the instrument software.





A simple flame atomic absorption spectrometer includes:

Atomizing techniques - graphite furnace atomic absorption spectroscopy (GFAAS)

- In GFAAS, a type of electrothermal atomization, a sample is placed in a hollow graphite tube which is heated until the sample is completely vaporized.
- GFAAS is much more sensitive than FAAS and can detect very low concentrations of metals (less than 1 ppb) in smaller samples.
- Using electricity to heat the narrow graphite tube ensures that all of the sample is atomized in a period of a few milliseconds to seconds.
- The absorption of the atomic vapor is then measured in the region immediately above the heated surface.
- Naturally, the detection unit does not have to contend with spectral noise, leading to improved sensitivity.

Hydride-generating atomizers

- Hydride-forming elements; As, Se, Sb, Bi, and Pb, are reacted to form a gaseous hydride.
- An acidified sample solution is added to a solution of sodium borohydride in a specially designed reaction cell.
- The resultant volatile hydride gas is passed into an optical cell (fused silica glass tube), where it can be electrothermally or flame heated.
- At high temperature, volatile hydrides decompose into neutral metal atoms that can absorb light from a hollow cathode lamp.

Cold-vapor atomization

- Mercury (Hg) is the only metal that does not atomize well in a flame or furnace.
- To analyze Hg, a special technique called, **cold-vapor atomization**, is employed.
- The Hg sample is acidified and reduced before it is swept through by an inert gas.
- The absorption of the gas is then determined.

AAS Applications

- **Mining and Geology** –
 - The elemental composition of minerals and rocks provide valuable information on the commercial feasibility of conducting mining activities in areas explored.
 - Trace metal analysis is of great value in prospecting for oil and water deposits.
- **Environmental Monitoring** – for trace metal contamination of industrial effluents, oceans, rivers and lakes.
- **Pharmaceuticals** – Trace metal analysis plays an important role in formulation development, catalyst efficiency and dosage limits.
- **Foods and Beverages** – In synthetic processed foods, metal pickup takes place due to contact with processing equipment and catalytic conversions.
 - manufacturers have to ensure that the trace metals do not exceed the permissible limits and this requires rigorous quality control through atomic absorption spectroscopy and other sophisticated instruments.

... AAS Applications

- **Oil and Petroleum** – Both edible oils and mineral oils require refining before consumption.
- Uptake of metals during such operations can lead to deterioration of performance or consumer hazards.
- Trace metal analysis of engine oil provides useful diagnostic information on the wear and tear of engine parts.
- **Agriculture** – Trace metal constitution of soils in addition to their acidic or basic nature is essential to establish their productivity and nutrient value.
- Trace metal composition of plants (leaves, stems and roots) gives a fair idea on how the uptake of minerals gets distributed under different growth conditions
- **Forensics** – Trace metal analysis provides valuable information on specimens such as stomach contents for food poisoning, paint chips, fibres and hair strands collected from the scene of a crime.

THANKS