M.Sc. Chemistry
Practical Inorganic Chemistry (Paper- 4106)
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Atomic Absorption Spectroscopy

- Atomic absorption spectroscopy (AAS) is specifically designed for the analysis of the metals and metalloids substances.
- AAS is a quantitative analytical technique wherein the absorption of a specific wavelength of radiation by the neutral atoms in the ground state is measured.
- The more the number of the atoms in a given sample, the higher is the intensity of absorption and vice-versa.
- This is also known as ***metal analysis spectroscopy*** as it is mainly used for the analysis of metals.

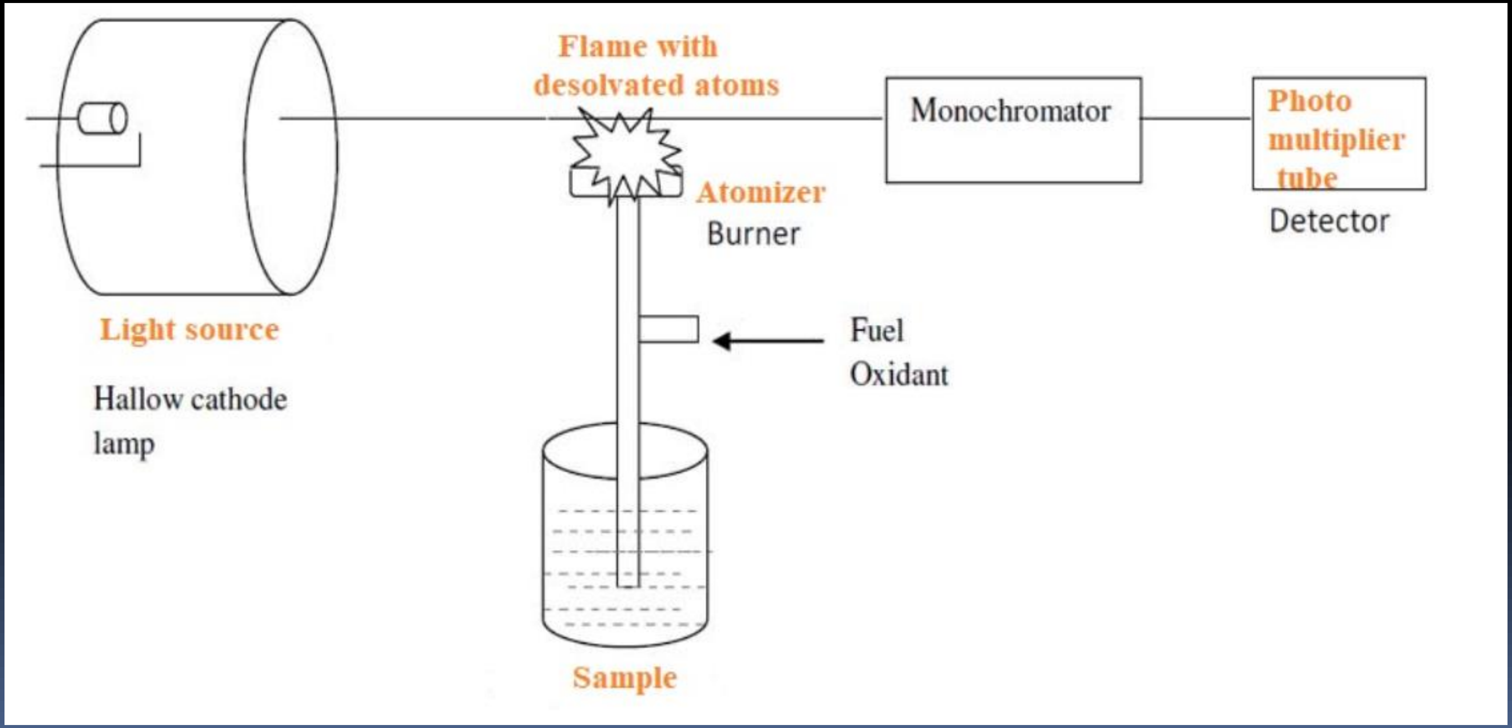
Working Principle:

"*The method relies on the principle of absorption spectroscopy*"

A liquid sample is allowed to convert into free atoms (desolvated and atomized). These free atoms absorb the light of a specific wavelength. The remaining unabsorbed light is detected and recorded. The intensity of absorption is directly proportional to the concentration of the sample.

Instrumentation

The Atomic absorption spectroscopy has simple instrumentation. But, unlike other spectroscopic methods, it has two additional requirements. These include a specially designed lamp to produce light of a desired wavelength and a burner to prepare the sample for absorption of light radiation. Additionally, the instrument also sprays the sample in the solution state over an atomizer (burner). This leads to evaporation of the solvent and leaves a fine dry residue. This residue has neutral atoms in the ground state. There are many representations available for the instrumentation but a simple graphical representation explained below:



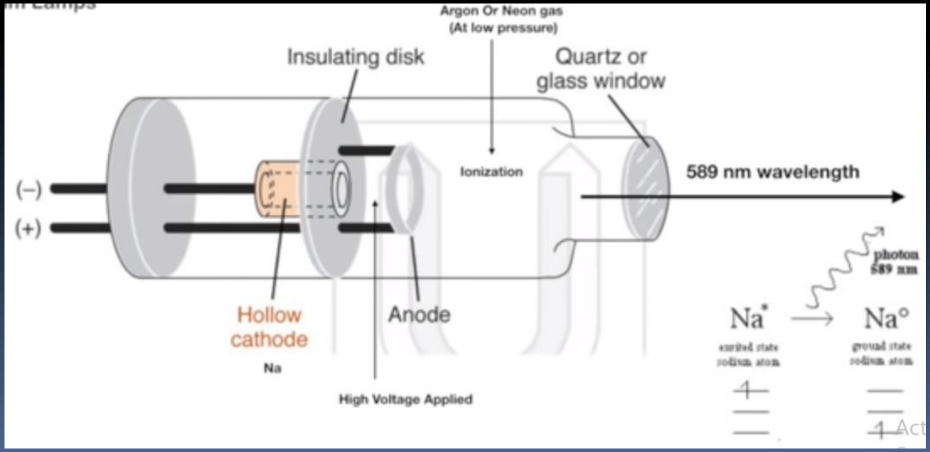
The Instrumentation includes following parts

- The atomizer (burner) to dry the sample ad produce atoms.
- Sample container.
- Fuel and oxidant to burn the sample by heat.
- Hollow cathode lamp to produce light of the desired wavelength.
- Detector to detect the absorption intensity.
- Amplifier and data recorder.

The instrument is available as single and double beam instruments.

Radiation Source: The radiation source should produce a narrow spectrum with little background noise. Besides the radiation should be stable and have sufficient intensity. Two types of light sources can be used based on the requirement.

1. **Hollow cathode lamp**: This is most widely used as a light source. Inside the lamp, the cathode is coated with a metal of analyte to be analyzed. For instance, if magnesium is to be analyzed from the sample, a cathode coated with magnesium is used.



Hollow cathode lamp

- Similarly, for all the other elements like Na, Ca, K, Zn, etc. analysis respective metal coated cathodes are used in the lamp. The lamp is filled with an inert gas like argon or neon which is ionized by an electric arc. The ions get attracted toward cathodes and strike it leading to excitation of metal ions. This leads to the emission of radiation with a characteristic wavelength of analyte metal.

- The advantages of Hollow cathode lamp is that it provides radiation with a bandwidth of 0.001 to 0.01nm. Use of other methods like monochromators gives radiation with a bandwidth of 1nm. So, these lamps give highly specific radiation.

- The disadvantage of this hollow cathode lamp is that for every metal different cathode lamp has to be employed.

2. **Electrode-less discharge lamps:** These lamps are less conventional in regular use but are essential of determination of Arsenic and selenium. A bulb containing an element of interest (with argon gas) is present in the lamp. This element is excited using microwave energy or radio frequency energy

Sample Insertion

Sample insertion id the second process of instrumentation which includes following steps

Sample container: This is a beaker-like a container of the sample which is placed below the burner preferably. A capillary tube drains the sample to the tip of the burner.

The burner (atomizer): Here the sample from the capillary rises to the tip of the burner. Here it is burned with the flame. This flame is produced by a fuel and oxidant combination. The sample after evaporation leaves a fine residue of neutral atoms.

Fuel and oxidant: This is a very important part of the entire process to be remembered. If the heat produced is not sufficient then the sample doesn't form neutral atoms. If the heat of the burner is more, the sample molecules may ionize instead of forming atoms. So both are undesirable for experimentation. Hence a proper combination of fuels and oxidant are to be used to produce recommended temperatures. Commonly used flues include propane, Hydrogen, and acetylene and oxidants are mostly air or oxygen.

Fuel combinations	Flame temperature	Metals Analyzed
Acetylene + Air	2550 degrees	For most samples
Acetylene + Nitrous oxide	2900 degrees	Aluminum (Al), Molybdenum (Mo), Silicon (Si), Titanium (Ti)
Hydrogen + Air	2200 degrees	Lead (Pb), Tin (Sn)

Monochromator: As discussed before, elements have specific absorption line. But some elements also have secondary absorption lines. Further, there is also emission from the lamp and the flame. Hence, we need to isolate the desired spectral line for the measurement of absorption. To achieve this a monochromator which can filter and provide a resolution of <1 nm is employed.

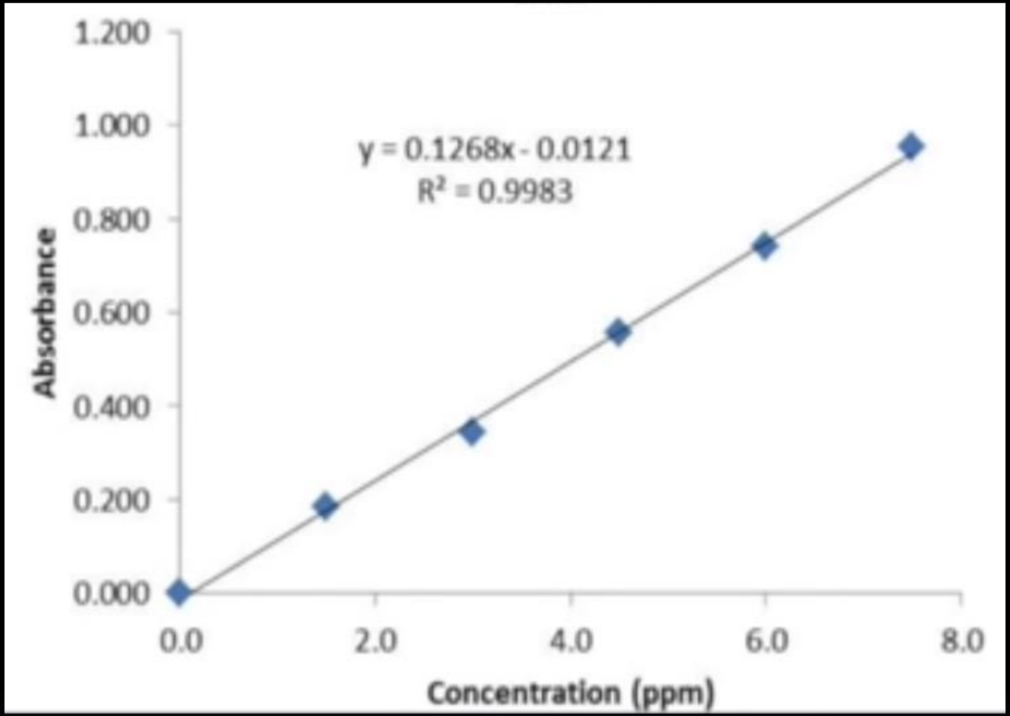
Detector: This part of the instrument detects the intensity of radiation absorbed by the elements. The detector consists of a photomultiplier tube or simple photocell. The current or potential recorded for the sample absorption is recorded in computer software and then analyzed.

Read device: This can be a display computer. It displays the absorbance at a specific wavelength.

Working Procedure

- Based on the metal of analysis a suitable cathode lamp is selected.
- The sample is dissolved in a polar solvent is placed in the container.
- With the help of fuel and oxidant in the presence of a mixer, the sample solution is sprayed on to the flame.
- The neutral atoms in the flame absorb light radiation from the cathode lamp. The unabsorbed radiation is recorded by the detector.
- Plot the graph of concentration against the absorbance reading to find out the concentration of the element in the sample.

Graph for Atomic Absorbance Spectroscopy



In atomic absorption spectroscopy data gathered in the form of transmittance which is converted using Beer–Lambert law to get the absorbance value.

Applications of Atomic Absorption Spectroscopy

- Atomic spectroscopy is used for quantitative analysis of metal elements in water, soil, plant material and ceramics.
- In health care, it is used to analyze ionic metal elements in blood, saliva, urine samples. The elements analyzed routinely include sodium, potassium, magnesium, calcium and zinc.
- To determine heavy metals like iron, manganese, copper, zinc, mercury, lead, nickel, and in urine and blood.
- This analysis is essential in case of heavy metal poisoning. Since heavy metal poisoning is mostly lethal a regular monitoring of poison levels in the patient blood are essential.
- To determine metal elements like copper, nickel and zinc in the food industry.
- To estimate Lead in petroleum products.
- To determine metal concentrations in groundwater and bore well samplings before using for drinking and irrigation.

Overview of Atomic Absorption Spectroscopy

- Atomic Absorption Spectroscopy (AAS) records the energy absorbed when electrons are excited.
- A solution is converted to a vapour in a flame. Light of a particular wavelength (energy level) is passed through the flame and atoms can absorb some of this energy.
- The light then passes through the monochromators (filter) to select the light of the chosen wavelength.
- Its intensity is measured by a detector. This can then be recorded as black bars on a coloured spectrum background or as a printed fact sheet.
- The amount of light absorbed indicates the quantity of the element present in the original sample.
- AAS is much more versatile than AES and can detect over 70 elements.
- It can accurately detect trace elements of up to parts per billion.
- AAS is one of the most widely used of modern instrumental techniques.
- Examples of uses: analysis of toxic metals in food and drink; urine and blood analysis; testing for air pollution. Readings from an AAS using known samples are used to draw up calibration curves- these can be used to determine the concentration of an unknown sample.

Recommended Books:

1. S. R. Koirtyohann, "A History of Atomic Absorption Spectrometry". Analytical Chemistry, 63 (1991)1024A–1031A.

2. Vogel's Quantitative inorganic analysis, 6th edition.

3. A. Walsh, H. Becker-Ross, S. Florek, U. Heitmann, High-Resolution Continuum Source AAS. Weinheim: Wiley-VCH Verlag GmbH & Co. KGaA, (2006)