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# ANALYTICAL TECH NIQUES FOR ELEMENTAL ANALYSIS: A REVIEW

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## ABSTRACT
Analytical chemistry is the study of analytical techniques and their instrumentation which play a very essential role in determination and evaluation of the quality of pharmaceutical preparation and various elements present in samples.[12] The pharmaceuticals development shows their efficacy if the presence of impurities is not detected in the sample.[5] Therefore, several analytical methods are developed time to time in order to estimate the pharmaceutical matter. For determination of inorganic elements several techniques used is atomic absorption spectrophotometer and inductively coupled plasma atomic emission spectrometry. For the trace element analysis inductively coupled plasma atomic emission spectrometry (ICP-AES) is used and atomic absorption spectrometry is used for the determination of environmental samples as well as industrial toxicology. X-ray fluorescence spectroscopy (XRFS) is a type of analysis technique for qualitative and quantitative determination used mainly for determination of heavy metals. The relevant use of analytical methods of which is remarked to employ more efficient and fast techniques which is certified by pharmaceutical quality control. Further, Graphite furnace atomic absorption spectroscopy, Cold vapour atomic absorption spectrometer (CVAAS), Atomic emission and fluorescence spectroscopy (AES and AFS), Inductively Coupled Plasma Mass spectrometry (ICP-MS) are also the various analytical techniques.[1,2]

**KEYWORDS**: -Analytical techniques, atomic absorption spectrophotometer, X-ray fluorescence spectroscopy, inductively coupled plasma atomic emission spectrometry, pharmaceutical quality control.

## INTRODUCTION
Analytical Chemistry is that branch of chemistry that deals with the analytical study of accurate qualitative and quantitative measurements of the elements present in the samples.[12] The heavy metals are present in environment, if these elements are present in more than trace amount then they may act as toxic or dangerous and these metal toxicities can cause adverse effects to the living beings. Several analytical methods were developed to estimate the elements. Therefore, pharmaceutical quality control tests are also performed for determining the use of analytical method having more efficiency with reasonable cost.[7] Spectroscopy is the study about the electromagnetic radiation its process of absorption and emission depending on the wavelength of the respective radiation, which occurs due to the interaction between matter and energy/radiation.[8,3] It has analytical as well as physical applications. The major goal of atomic analytical spectroscopy is to recognition of elements and determining the concentration of element in various sample media.[12] like different samples of nano materials, foods, drugs, biomaterials and waste of industries.[3]

### Atomic absorption spectroscopy
Atomic Absorption Spectrometry (AAS) is a technique that deals with absorption of specific wavelength of radiation in a ground state by neutral atoms for measuring quantities of chemical elements present in environmental samples. These neutral atoms were obtained by using a burner by spraying the sample solution of element.[3] As it estimates the concentration of the element due to the passage of light with specific wave length emitted by a radiation source of a specific element through the sample having cluster of atoms. This specific wavelength of radiation is generated by using a hollow cathode lamp.[4] This is the process in which unexcited atoms diminish the radiant power of the source by absorbing the characteristic radiation from a source in a flame or furnace.[9,5]

### Principle
Atomic-absorption spectroscopy quantifies the absorption of ground state atoms in the gaseous state. When the metallic salt solution is sprinkled on to the flame, due to the thermal energy of the flame, fine droplets are formed and evaporation of the solvent in the droplets takes place, and a fine residue is left which are further transformed into neutral atoms. The atoms absorb ultraviolet or visible light and make transitions to higher

electronic energy levels. The analyte concentration is determined from the amount of absorption.[9,5]

**Instrumentation**

Various components of atomic absorption spectrophotometer (AAS):

1. A light source - A hollow cathode lamp
2. An atom cell (atomizer) – Burner
3. A Chopper
4. A monochromator
5. A detector – Photomultiplier tube or phototube
6. Read out device

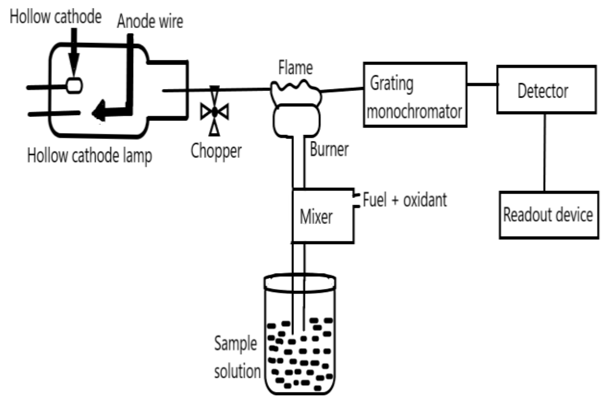



**Fig.1.1 Block diagram of instrumentation of Atomic absorption spectroscopy.**

1. **Light source:** The radiation source can be HCL or electrodeless discharge lamp (EDL)[4] Generally, a hollow cathode lamp of the element is being used as light source that is measured. It consists of two cylinders located co-axially.[8] Hollow cathode lamp is made up of specific element or alloys of element or coating of element and anode made up of tungsten. An inert gas either neon or argon are filled and sealed in a glass tube usually made up of glass/ Pyrex shield. For the analysis of element, every element has their own unique lamp which is to be used during the analytical process.[3,5] When current or potential difference is applied between the anode and cathode, metal atoms appear from hollow cups and interact with the gas leading to the ionization of atoms present in gas. Then the process of sputtering occurs in which collision with the cathode takes place which leads to ejection of metal atoms. [5] The atoms which were in excited state emit some of their characteristics radiation, the neutral atoms in the ground state absorb the radiation and the sputtered atoms after emission, return to ground state.[9,5]

2. **Atomizer:** Burner with fuel and oxidant: Atomisation of the sample solution is required therefore; different types of atomizers that can be used in atomic absorption spectrometry are a graphite furnace, or a quartz tube, flame and burner. [4]The process of atomisation takes place by shattering the molecules into atoms after the dissociation of particles into single molecules all this is done by disclosing the sample to high temperature that can be done in a burner, flame or furnace. Then the process of vaporization takes place in which the solid particles transform into gaseous form after the process of desolation in which atoms are mixed with liquid sample (9, 3), atoms can absorb radiation from the source in this form.[5]

3. **Chopper:** Chopper are the type of component which looks like fan, as it rotates, these were used earlier, for constructing a pulsating signal used for the measurement of the intensity of light absorbed by the elements.[9]

4. **Monochromator:** Monochromators is mainly choosing the specific wavelength of light determines the particular element by eliminating the scattered light[4] of other wavelengths.[5] As some of the elements have single absorption line that can be called as principle line and some of the elements have several lines called as secondary line. Therefore, monochromator is used. Usually, monochromator provides the resolution of 1nm or less than 1 nm.

5. **Detector:** Commonly, the photomultiplier tube also called phototube is used as a detector in which the process of conversion of light signal into electrical signal takes place. (4)Which are corresponding with the light intensity.

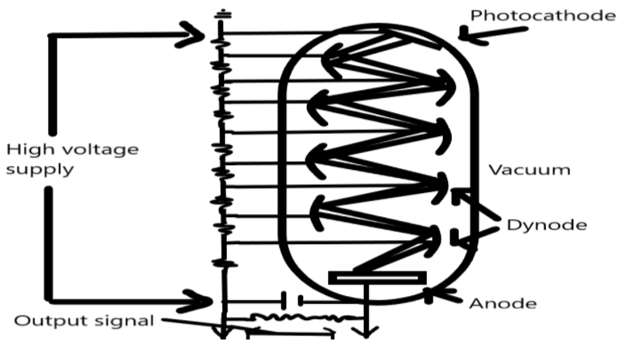



**Fig 1.2 Photomultiplier tube or phototube detector.**

6. **Read out device/ data read out:** The signal amplifier processes the electrical signal that is produced in the read-out device. This can be stored as a data.

**Working**

1. Sample preparation- If solid is mixed with solvent with help of tube sample solution is sent to nebulizer.
2. Sample solution gets aspirated and converts into fine droplets. With the help of inert gas, fine droplets send to the surface of flame.
3. To maintain the temperature of flame the supply of fuel and oxidant should be maintained.
4. Different types of fuels, oxidants are used and in that basis temperature of flame is maintained.

5. To maintain the temperature of flame is mandatory as various steps occur on the base of the flame such as:

**Desolvation –** Due to high temperature evaporation of the liquid solvent takes place.
**Vaporization –** The remaining sample molecules converts into gas.
**Atomization –** These molecules dissociate and produce atoms.

Excitation and emission- Atoms absorb thermal energy and reaches to excited state and emits radiation as the state is not stable.[5]

6. Photomultiplier tube detector detects the radiation and the signal is transferred to the recorder.[4,5]

**Applications**
1. Used for the detection of trace elements.
2. For environmental analysis.
3. Used for determination of industrial toxicology.[9]

**Table 1.1 various analytical techniques and process of detection.**

| Analytical Techniques | Types of elements detected | Units used to determine analyte concentration | Method of detection of elements |
|---|---|---|---|
| Flame atomic absorption spectrometer (FAAS) | Heavy metals were detected For example-lead (Pb), copper (Cu)cadmium (Cd), and zinc (Zn) | Parts per million (ppm) | Sample solution is prepared and then sample as an aerosol or gaseous form is aspirated in the atomizer by the nebulizer. As a flame atomizer supplies nitrous oxide or air-acetylene.[4,9] |
| Graphite furnace atomic absorption spectroscopy (GFAAS) | Lead (Pb),Cadmium (Cd) and Nickle (Ni) | Parts per billion (ppb) | Before atomization process sample is prepared by stirring with modifiers and gets distributes into atomizer called graphite tube. To dissociate the atoms in the left-over sample and to separate the solvent and matrix components, Heating takes place in the sequence of steps that comprises of drying, ashing, and atomizing. The sample containing dissociated atoms within the tube is kept for long time.[3,4] |
| Cold vapour atomic absorption spectrometer (CVAAS) | Mercury (Hg) | Nanometre(nm) | By applying the strong reducing agents like stannous chloride or sodium borohydride, In the elemental state reduction of mercury takes place. By the carrier gas- Argon the vapour of mercury moves to the absorption cell which is located in the light path present in the spectrophotometer.[4,5] |
| Hydride generation atomic absorption spectroscopy (HGAAS) | Metalloids are detected for example- antimony, selenium,arsenic, and tellurium | Parts per million (ppm) | Sodium borohydrideas a reducing agent interacts with the aqueous samples that are acidified this is comprised in a continuous flow system, which ultimately produces volatile hydride. Then this is conveyed to the atomizer that is heated quartz cell, then along with the argon gas of the conventional AAS with the optical axis.[4,9] |
| Fluorescence spectroscopy | Calcium (Ca), magnesium (Mg) potassium (K), and sodium (Na) are detected. | Parts per million (ppm) | In the flame the atoms in the ground state by concentrating on a beam of light gets excited in the form of vapours. And as the atoms releases energy they return back to the ground state, that energy is measured.[4] |

**Table 1.2 Advantages and Disadvantages of the techniques used for the detection of elements.**

| Techniques | Advantages of the techniques | Disadvantages of the techniques |
|---|---|---|
| Flame atomic absorption spectrometer (FAAS) | 1. Fast analysis of the sample On an average 10-15 s for the analysis per sample.<br>2. Precision is good.<br>3. Relatively low cost.[15]<br>4. Interferences can be easily corrected.[5] | 1. For the induction of overall atomisation utmost atomisation temperature is not adequate.[5] |
| Graphite furnace atomic absorption spectroscopy (GFAAS) | 1. For majority of elements detection limits are good, because 20 µl is essential for analysis which is a very small sample size.[4] | 1. Working range is restricted.2. Process of analysis is slow.3. Cost is high.[4] |
| Cold vapour atomic absorption spectrometer (CVAAS) | 1. Detection limits are low.<br>2. Measurement is fast (30-50 second per sample).<br>3. Interferences are minimum.[4] | 1. Analytical procedures are monotonous.<br>2. Chemical reagents are highly consumed.[4] |
| Hydride generation atomic absorption spectroscopy (HGAAS) | 1. Detection limits are low.<br>2. Interferences are minimum.<br>3. Measurement is fast.[4] | 1. Chemical reagents are highly consumed.<br>2. Analytical procedures are monotonous.[4] |

**Inductively coupled plasma-atomic emission spectrometry (icp-aes)**

In analytical atomic spectroscopy, Inductively Coupled Plasma-Atomic Emission Spectroscopy is one the technique which is preferably and widely used for various elemental analysis. As the source of atomization and excitation plasma is used. A highly ionized and neutralized gas which consists atoms, electrons and ions.[8,5]

**Principle**

When the element emits light in the sample which is instigated in the argon based ICP source, at the temperature of about 6000 TO 8000° K at this high temperature elements present in the sample gets dissociated into atoms and ionization takes place, emitting their characteristics radiation, these emitted radiations are measured.[5]

**Instrumentation**

1. Source- Inductively coupled plasma
2. Nebulizer
3. Pumps
4. Spray chambers
5. Torch
6. Radio frequency generators
7. Wavelength dispersive devices
8. Detector -Photomultiplier tube
9. Data collection

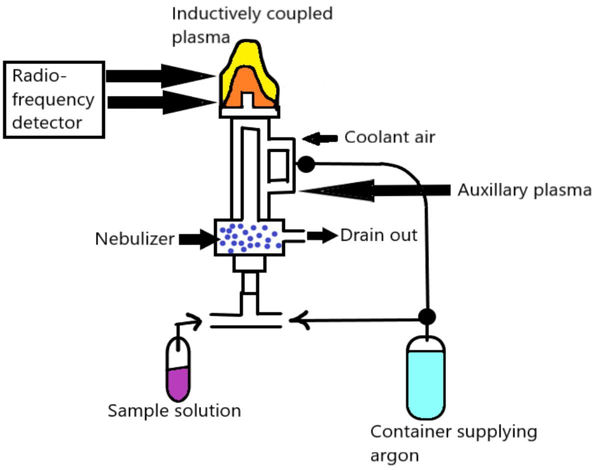
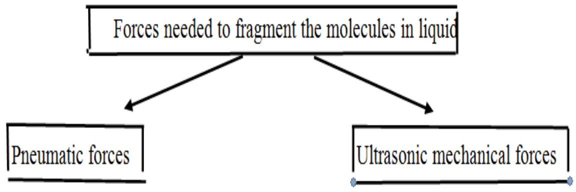



**Fig. 1.3: Inductively coupled plasma-atomic emission spectrometry.**

**Nebulizer**

To prevent adsorption of metals, with 2-3%in $HNO_3$ the sample solution is acidified, before establishment into plasma, the adsorption takes place in bottle sample or glassware or into tubing instrument. Liquid sample is converted into aerosol & is conveyed into plasma this causes the excitation of the atomic species in the form of aerosol.[12,5]



**Pumps**

Peristaltic pumps are the type of pumps that are used in the analysis. The samples are passed through tubes that are used in the pumps; this is for preventing the immediate contact with the solution avoiding contamination of the sample.[5]

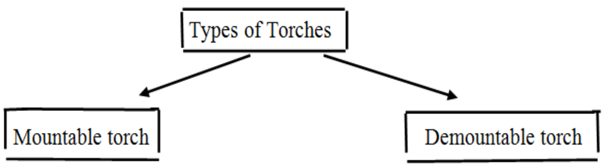
**Spray chambers**

When nebulizer produces sample aerosol, then it is transferred to the torch before injecting the plasma. Between the torch and nebulizer, the spray chamber is set, its mainoperation is to eliminate the large particles present in the aerosol. During nebulization when smaller particles move through the flow of air and moves into the torch, its secondary function is to erase the pulses that arise during the process.[16,5]

**Torch**

The solvent which is usually water from the analyte salts evaporates, and dissociate the atoms present in the salts; therefore excitation of the atoms occurs is the motive of the torch. For the flow of argon& aerosol injection a total of three tubes are used. At high velocity, the appearance of gas with the narrow space between the two outer tubes. The flow of gas from the central tubecarrying the aerosol sample is inserted in the plasma.[5,12]

Almost 700 to 1500 watts power is provided by this device for the generation of the plasma discharge, power is transferred by a load coil to plasma gas covering the top of the torch.[5]




**Wave length dispersive devices**

The most frequently used techniques for physical dispersion are - diffraction and Echelle grating, Prisms, filters and interferometers. A specific element or a molecule that emits light constituting wavelengths is monochromatic radiation. Various elemental analyses are attainable with polychromators, as in polychromators slits are lined up and permits only a particular wavelength of radiation to precede to a detector.[5]

**Detectors**

Detectors are used for measuring the intensity of emitted radiations that are remoted by the spectrometer. The vacuum tube consisting of photo cathode and a dynode, when the light hits the cathode it started ejecting electrons. The electrons are then move towards the dynode, every electron hitting the surface the dynode ejects 2 to 5 secondary electrons. Further, these secondary electrons hit another dynode and ejection of electrons takes place till the last dynode, 8 -10 dynodes is present in the detector. At last, these secondary electrons are gathered from the last dynode. For example- Photo multiplier tube is the frequently used detector.[16,5]

**Data collection**

From the detector the signals are transferred to the system where the signals are collected and recorded for the interpretation of graph.[12]

**Working**

1. **Sample Preparation:** Acids, microwave digestion and heating methods are used for the preparation of samples.
2. **Nebulization:** Sample in Liquid is transformed into aerosol through nebulization.
3. **Desolvation:** By mixing of sample and solvent, evaporation of solvent takes place.
4. **Vaporization:** After solvent is evaporated only sample molecules are left which converts into gas due to high temperature.
5. **Atomization:** After converting into gas the molecules dissociate and atoms are formed.
6. **Excitation:** The temperature of the plasma should be more at this phase and due to this the atoms absorbs the thermal energy or due to interactions of the atoms, attains energy and reaches to high energy state.
7. **Emission:** As high energy state is unstable therefore, and emits light of a characteristic wavelength.
8. **Separation and Detection:** The light is separated by grating and is measured quantitatively.[12]

**Applications**

1. Determination of presence of heavy metals like-lead, cadmium, copper etc in honey and sugar.[15]
2. Determination of elements from the environmental samples[13]
3. Determination of various trace elements in sample matrices.[12]

**X RAY FLUORESCENCE**

The X-ray fluorescence spectroscopy (XRFS) is a type of analysis technique for qualitative and quantitative determination[10] for evaluation of various elements in which the phenomenon of the x rays and matter interacts used mainly for the detection of elements from sodium to uranium this requires very little amount of sample for preparation. [11,4]

**Principle**- Sample is irradiated through x ray radiation by the source. If the energy is sufficient it will interact with the inner shell atom and those atoms are thrown out, immediately within $10^{-8}$ sec, relaxation process takes place in which the outer shell electrons falls into the inner shell. As a result, a specific amount of energy is released in the form of electromagnetic radiation.[10]

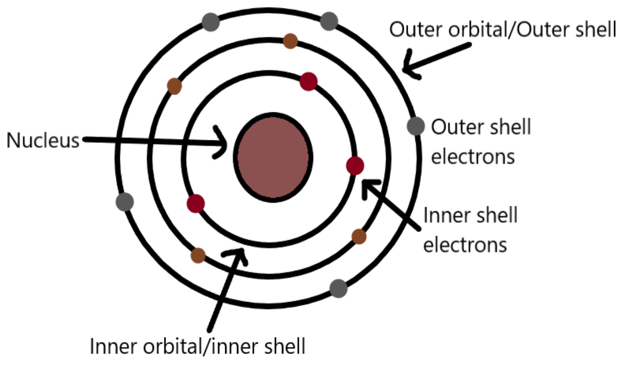



**Fig. 1.4: Shells of atom.**

## Instrumentation
The X-ray fluorescence spectroscopy generally includes:
1. Source - X-ray radiation.
2. Sample chamber
3. Detector
4. Data Processor.

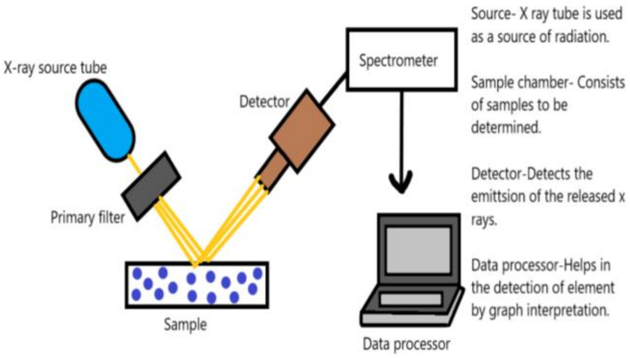



**Fig 1.4 Instrumentation of X ray fluorescence.**

## Working
1. When an atom is strike by X-ray radiation of high intensity, the one or more atom present in the inner orbital are kicked out of the inner orbital.
2. Due to the process atoms becomes unstable and the empty space in the inner orbital is filled by the atoms in the outer orbital.
3. The electrons from the inner shells have a more energy than the outer orbital electron. This causes release of x ray radiation.
4. As each element have different electronic energy levels, therefore the peak formed can be tally up to a particular element of X-ray fluorescence.[16]

## Applications
1. To check and evaluate the level of heavy metals in soil.
2. Used in various metal processing plants.
3. Used in the laboratories where geochemical analysis is performed.[16]

## CONCLUSION
Analytical techniques like atomic absorption spectroscopy are mainly used for the determination of various elements in the environmental samples and industrial toxicology. And another method, Inductively Coupled Plasma-Atomic Emission Spectroscopy detects heavy metals in various sample matrices. Whereas, x ray fluorescence evaluates heavy metals in soil and used in metal processing plants etc. These methods are accurate and reliable for use to prevent the toxicity of the elements by detecting the concentration.

## REFERENCES

1. Masoom Raza Siddiqui, Zeid A. AlOthman, Nafisur Rahman Arabian Journal of Chemistry, 2017; 1: 1409-1421.
2. A. J (Tom) Van Loon, Analytical Atomic Absorption Spectroscopy: Selected Methods, 2012.
3. Muhammad Akhyar Farrukhs, Atomic Absorption Spectroscopy, 2012.
4. ABM Helaluddin, Reem Saadi Khalid, Mohamed Alaama and Syed Atif Abbas, Tropical Journal of Pharmaceutical Research, 2016; 15(2): 427-434.
5. Swapna G; Analytical techniques for analysis Of Inorganic Elements – A Review; PharmaTutor, 2017; 5(2); 39-45.
6. Nagashwar R., Kumar talluri M.V.R, Journal of pharmaceutical and biomedical analysis, 2007; 43: 1-13.
7. Rudy Bonfilio, Magali Benjamim De Araujo, Herida Regina Nunes Salgado, WSEAS transactions on biology and biomedicine, 2010; 4: 7.
8. G.L. Moore, Introduction to Inductively Coupled Plasma Atomic Emission Spectrometry, 2012.
9. Bernhard Welz, Michael Sperling - Atomic Absorption Spectrometry, 2008.
10. Rudolf Muller, Spectrochemical Analysis by X-Ray Fluorescence, 2014.
11. Burkhard Beckhoff, Birgit Kanngießer, Norbert Langhoff, Reiner Wedell, Helmut Wolff, Handbook of Practical X-Ray Fluorescence Analysis.
12. Thomas j. Manning and william r. Grow 1 / 2, no. 1 issn 1430-417 The chemical educator
13. Petya Kovacheva, Rumyana Djingova, Analytica Chimica Acta, 2002; 464: 7–13.
14. Ravali R, Phaneendra M, Bhanu Jyothi K, Ramya Santhoshi L and Sushma K, J Bioanal Biomed, 2011; 11.
15. M.D. Ioannidou, G.A. Zachariadis, A.N. Anthemidis, J.A. Stratis Talanta, 2005; 65: 92–97.
16. Gerhard Schlemmer, Lieve Balcaen, José Luis Todolí, Michael W. Hinds, Elemental Analysis: An Introduction to Modern Spectrometric Techniques.