Review Article



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A review on inductively coupled plasma optical emission spectroscopy and its advancement in analysis of metals

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ABSTRACT

Spectroscopy is a standout amongst the most generally utilized procedure for estimation of atoms at nuclear and molecular level by deciding their energy states by concentrate the light retained or transmitted Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) has developed as an amazing diagnostic system in recent days. This instrument is utilized for fluid examples which are atomized and further de-solvated, vaporized, atomized, and energized and additionally ionized by the plasma. This energized particles and particles discharge their characteristic radiation which is a wavelength received for translation of focus data for the analyst. The ICP-OES can detect even different components with higher level of affectability and exactness even up to PPB range. 'The regulatory administrative bodies are anticipating a significant position in the advanced investigative research facilities for routine examination. The present review features the different parts of ICP-OES, the benefits of ICP over other expository systems for metal analysis and methods employed in subjecting samples.

Keywords: Spectroscopy, ICP-OES, Metals, Analysis.

INTRODUCTION

Inductively coupled plasma optical emission spectroscopy is a sensitive and multi-elemental technique. Inductively coupled plasma is done with optical emission spectroscopy (OES), mass spectrometer (MS) and atomic emission spectrometer (AES). The discussion concentrates on ICP-OES. It is one of the most versatile method in analysis. It

quantifies trace level metal analysis or elemental impurities. It detects and quantifies only cations and in very low concentrations. The detection level of metals are up to ppb. The plasma maintains the generation of temperature i.e., plasma in ICP-OES is 8000-10000°c, where the sample gets de-solvated into gas and then dissociates individual molecules into atoms. Because of the higher temperature, the atoms and ions in sample gets excited to produce at

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particular wavelength characteristic to element present in the sample solution. The intensity of emitted light relates to concentration of element.

Table 1. Comparing with Other Instruments in Metal Analysis [9]

Flame Photometer	Atomic Absorption	ICP-OES	ICP-MS
	Spectrometer		
a)Only group II	a)Source- cathode	a)Sensitive andmulti-	a)Advanced technique
metals are	Hollow		
detected i.e., Na, K, Li	lamp principle involved -	elemental technique	b)Very expensive
,Ca,			
Ba, Ce	Absorption	principle-	c)Detection limit: PPTto
		emission	
Principle-Emission	b)It is for individual analysis.	b)Metals/Elemental	PPQ
b)Limit of detection :PPM	for every element ,lamp	impurities can be quantified	d)High sensitivity and wide
c)Temperature:1000- 4800 °C	source of that particular	in trace level concentrations.	dynamic range
based on fuel-oxidant	element should be placed for	c)Detection limit:PPM to	e)Determines quality and
mixture combination.	detection	PPB	quantity quickly.
	c)Temperature of	d)Temperature:8000-10000	f)Temperature :10000 °C
	plasma:2000-3000°C.	°C	
	d)Detection limit: 0.1PPM	e)Easy and rapidly	
	to	producing	
	100 PPB	results.	
	e)Time taking process		

Principle

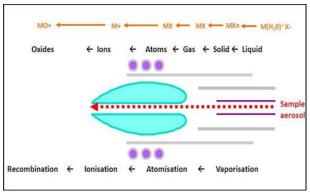


Fig 1. Principle

The name itself indicates the principle of "emission". The atoms gets excited at high temperature on absorbing energy. At a particular state the atoms gets back to ground state by

releasing light of particular wavelength characteristic to the element present in it. Sample gets vaporised and gets atomized at plasma and

ionized on reaching from higher level to ground level.

Plasma: Gas in which a significant number of atoms are ionized will interact with magnetic field

inductive coupling between varying field and plasma. [2]

Components



Fig 2.ICP-OES instrument

Chiller: It circulates liquid nitrogen around plasma to reduce high temperatures and allows it to OES through inlet and outlet as high temperatures can cause damage in OES.

Exhauster: It is maintained to suck out harmful fumes by mixing them with air and releases out.

Gas supply

Nitrogen: Yellow colour indicated inlet pipe for supply

-Used for purging in OES and also to remove gases other than oxygen and moisture so as to get accurate result and mirrors get cleaned.

Argon: Green colour indicated feed pipe for supply

- It is an inert gas and is stable; hence do not react with sample.
- It is required for higher temperatures i.e., plasma generation.
- RF power involved in electron removal which become unstable

Shear gas: It is compressed air which is helpful in supply of filtered or purified air into instrument.

-It tare's /cuts the plasma into tail and head as the temperature in same level can't be maintained in OES done by magnetic plates called Flag plate technique.

Nebulizer: Sample fed through the inlet pipe which is connected to peristaltic pump to nebulizer where it gets aspirated in plasma.

Detectors: mono chromator, charge coupled device

Air conditioner: To control and maintain the temperature in the location /lab. Temperature should be maintained at $25\pm2^{\circ}$ C.

Sample preparation methods for ICP-OES [5, 6]

Heavy metals are soluble in acids and solution should be clear for aspiration. Most commonly used acids for digestion are HNO3 (Nitric acid), HCl (hydrochloric acid), HF (hydrofluoric acid), H2SO4 (sulphuric acid), H2O2(hydrogen peroxide), HClO4(perchloric acid) etc.,. If there is any presence of precipitate on addition of water for dilution indicates the reaction between cations and anions.

Microwave Digestion System

Most popular and widely used method in sample preparation. It is safe, effecient and rapid. Because of its less risk external contamination results in improved detection and accuracy in result. In this method two steps are involved in which aliquot of samples are to be digested were placed in clean Teflon digestion vessel with concentration of acid mixture HNO3:HF = 3:1 v/v ratio for first microwave digestion procedure. In second step of procedure ,sample is treated with acid mixture HNO3:HF:HCl =3:1:0.7 v/v/v .After the vessel gets cooled ,place the tubes in rotor ,where microwaves

are passed and then raise the temperature over a period of 6 min to 200°C and holding the same temperature for 20 min. After cooling, get it opened carefully .All the digested samples transferred to VF quantitatively with ultra pure water .These set to be analyzed after dilutions using external calibrated standard.

Open Hot-Plate digestion

In this beakers with aliquot of samples are placed on hot plate digested with acid mixture HNO3: HF 3:2 v/v and subjected to heating at 150°C.Organic matter evaporates at low temperatures. This method develops a risk of contamination with atmosphere as performed in open systems.



Fig 3. Hot Plate

Ash Plate technique

It involves 1g sample is taken in beaker and placed on muffle furnace (Pt/Silica)and subjected to temperature gradually increasing to 1000°C till the organic fumes get stops and ash remains set to

room temperature .Then 1g of ash powder (inorganic matter) is taken and acid is added and heated followed by making up with water. Filter it and subject to aspirate.

Instrumentation

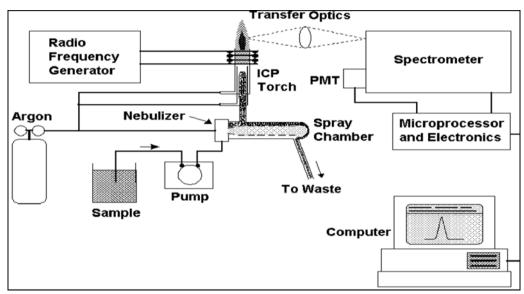


Fig 4. Schematic representation of instrumentation in ICP-OES

The sample is introduced into system by peristaltic pump into a nebulizer. Where the sample is changed over into an aerosol. A spray chamber is among nebulizer and light which drains the abundance drops in aerosol through outlet and enables sample to go into light.

In the torch when plasma energy is given to an examination test from outside, the components

(molecules) is energized. To produce plasma, first argon gas is provided to torch coil, and high recurrence electric flow is connected to the work loop at the tip of the torch cylinder. Utilizing the electromagnetic field made in the torch tube by the high recurrence current, argon gas is ionized and plasma is produced.



Fig 5. ICP Torch

This plasma has high electron thickness and temperature (10000K) and this energy is utilized in the excitation outflow of the sample. At the point when the energized particles come back to low energy position, outflow spectrum rays are discharged and the emission rays that relate to the photon wavelength is gathered by utilizing different optics and wavelength gadgets are estimated by

utilizing different detectors i.e., Photo multiplier tube(PMT) / Charge coupled device(CCD) . The component type is resolved dependent on the situation of the photon beams, and the substance of every component is resolved dependent on light ray intensity. Signal processing units and output devices are available to peruse the outcome by the analyst. [3]

METHODOLOGY

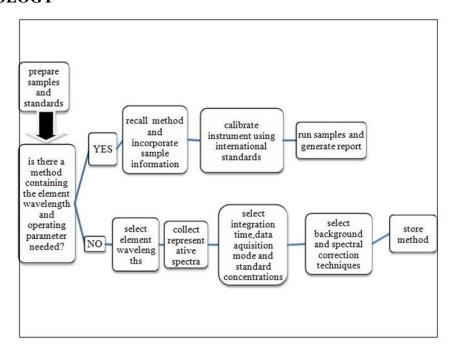


Fig 6. Methodology of ICP-OES

Advantages [8]

- 1. Very fast and sensitive
- 2. Flexible to element
- 3. Greater dynamic range
- 4. Analysis speed:1-3 min
- 5. Dozens of elements can be recorded simultaneously i.e., multi-elemental technique
- 6. Appreciable level of ionization for few segments.

Disadvantages

- 1. Consumption of sample is high (1-5 ml/min)
- 2. Interferences in spectrum
- 3. Expensive and very complicated
- 4. Relatively poor detection limits

Applications [1]

- In environmental analysis: Water ,soil ,air and sludges
- Metallurgy: Metals like steel, bronze ,brasses ,alloys and traces of contaminants
- Geological analysis: Sediments, ceramics, cements, rock samples and also employed in survey and screening about quality and purity.

- Food analysis: Micro nutrients, water we drink and food we eat and also food fed to animals. Analysis of crops and fields.
- Petrochemical analysis: Oils, greases, paints, inks, additives and in examination of industrial by-products.

CONCLUSION

ICP-OES is one of the attractive and sophisticated technique and is at its best in getting opted by analysts. Compared to other techniques like AAS, ICP-MS and flame photometry, ICP-OES pose many benefits involving rapid and fast analysis, simple to operate, detection is good at resulting, it enables quantification of selected metals .Hence it as an ideal choice for laboratory research with high productivity.

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