INTRODUCTION

Non- flame atomic emission techniques, which use electrothermal means to atomize and excite the analyte, include inductively coupled plasma and arc spark. It has been 30 years since Inductively Coupled Plasma – Atomic Emission Spectrometry ICP – AES (also known as ICP- optical emission spectrophotometry **ICP-OES**) began to be widely used, and is now one of the most versatile methods of inorganic analysis. Its features are often compared to atomic absorption spectrophotometry, in which the excitation temperature in the range 2000 to 3000 K, while the excitation temperature of argon ICP is 5000 to 10000 K, which efficiently excites many elements. Also, using inert gas (argon) makes oxides and nitrides harder to be generated.

PRINCIPLE OF ICP- AES

An ICP source consists of a flowing stream of argon gas ionized by an applied radio frequency field. This field is inductively coupled to the ionized gas by water-cooled coil surrounding a quartz torch that supports and confines the plasma. A sample aerosol is generated in an appropriate nebulizer and spray chamber and is carried into the plasma through a tube located within the torch. The sample aerosol is directed into the ICP, subjecting the constituent atoms to temperature of about 5000 to 10000°K. This high temperature results in almost complete dissociation of molecules, significant reduction in chemical interferences. The high temperature of the plasma excites atomic emission efficiently. The radiation emitted by the excited atoms is recorded by one or more optical spectrometers and when calibrated against standards the technique provides a quantitative analysis of the original sample.

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INSTRUMENTATION:

There are four basic components to an ICP – AES Instrument.

An ICP-AES instrument consists of a sample delivery system, an IC plasma to generate the signal, one or more optical spectrometers to measure the signal, and a computer for controlling the analysis.

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INSTRUMENTATION : ...cont'd

-1Sample delivery system

There are three basic parts to the sample introduction system. The Peristaltic pump draws up sample solution and delivers it to the Nebulize which converts the solution to an aerosol that is sent to the Spray chamber where The larger droplets fall to the bottom of the chamber and exit through the drain as mentioned in FAES ...Various types of spray chambers commonly used.



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INSTRUMENTATION : ... cont'd

2 - IC plasma :

The device which produces the IC plasma is commonly referred to as the ICP torch. It consists of two to four Argon flows depending on the manufacturer:

Nebulizer gas (inner Argon flow) carries the analyte aerosol

Sheath gas for producing a laminar flow to improve low excitation energy elements eg group I & II elements

ICP torch

•Torch is surrounded by high-energy induction coil connected to radio frequency generator which produce large electrical discharge in the troch.

Resistance in the movement of electrons and argon gas through the quartz tubes leads to generation of heat.
Sample is introduced in the argon stream in form

of liquid aerosol or vapor

- This step allow the evaporation of the solvent .
- The next steps are atomization and excitation of the analyte .

INSTRUMENTATION : ... cont'd

2 - IC plasma .. continued :



components of an ICP torch



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INSTRUMENTATION : ... cont'd

2 - IC plasma .. continued :The sample aerosol is directed into the center of the plasma.The energy of the plasma is transferred to the aerosol.

The main function of the energy source is to get atoms sufficiently excited such that they emit light. The temperature of the plasma is very high and varies from zone to zone .



INSTRUMENTATION : ... cont'd

2 - IC plasma :





the Radio Frequency generator



the Load coil



the Torch

INSTRUMENTATION : ... CONT'D

3- Spectrometer

Once the atoms in a sample have been excited by the plasma, they will emit radiation at specific wavelengths . <u>No two elements</u> will emit radiation at the same wavelengths. The function of the spectrometer is to diffract the emitted radiation from the plasma into wavelengths. Then each wavelength will be directed to a detector. These detectors are connected to a computer for data analysis .



APPLICATIONS

Plasma sources are rich in characteristic emission lines, which makes them useful for both qualitative and quantitative elemental analysis. The inductively coupled plasma yield significantly better quantitative analytical data than other emission sources do. The excellence of these results stems from their high stability, low noise, low background, and freedom from interferences.

ICP-AES is a major technique for elemental analysis. The sample to be analyzed, if solid, is normally first dissolved and then mixed with water before being fed into the plasma.

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Trace elements in a wide variety of aqueous matrices: drinking water, river, lake and ground water, waste water and effluent, and seawater.

Trace elements in solids after digestion: sediment, soil, sludge, road dust, air particulate matter, plant tissue and grain, rocks and minerals, etc.

Trace elements in samples of body fluids, including blood, plasma, and urine.

ICP - AES Advantage :

1. The plasma provides simultaneous excitation of many elements.

2.The analyst is not limited to analytical lines involving ground state transitions but can select from first or even second ionization state lines. For the elements Ba, Be, Mg, Sr, Ti, and V, the ion lines provide the best detection limits.

3. The high temperature of the plasma ensures the complete breakdown of chemical compounds (even refractory compounds) and impedes the formation of other interfering compounds e.g. oxides , thus virtually eliminating matrix effects. 4. The ICP torch provides a chemically inert atmosphere and an optically thin emission source, that is not subject to self absorption except at very high concentrations.

5. Excitation and emission zones are spatially separated : this results in a low background . Argon is Inert – non reactive with sample.

6.Low background, combined with a high S/N ratio of analyte emission, results in low detection limits, typically in the ppb range.

7. Wide linear region of analytical curve . Analysis of samples from ppb to ppm range in the same method.

8.High number of measurable elements - elements that are difficult to analyze in atomic absorption spectrometry such as Zr, Ta, rare earth, P and B can be easily analyzed.

Conclusion

ICP – AES is now highly rated as a multipurpose analysis technique. It is well regarded as an environmental measurement technique, along with atomic absorption spectrometry and its use is expected to expand even further in the future.

Plasma sources are rich in characteristic emission lines, which makes them useful for both qualitative and quantitative elemental analysis. ICP- AES yield significantly better quantitative analytical data than other emission sources do. The excellence of these results stems from their high stability, low noise, low background, and freedom from interferences.

Weaknesses of ICP-AES :

Differing viscosities can affect amount of sample uptake. Matrices can change nature of plasma Certain matrices can attack torch. Matrices can contain interfering spectral components. Instrumental Drift : Instrument reading can drift over a period of time due to physical changes in the optical system, or the configuration of the plasma. Standards need to be run at the beginning and end of each run in order to estimate and correct for this drift. Internal standards are used to compensate for differing matrices from sample to sample. Not effective for low levels of alkalis (less than 1-5 ppm). Only elemental data is provided - no direct structural information. Solids, can usually be dissolved using

various techniques.