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INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY: AN OVERVIEW

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ABSTRACT

Atomic emission spectroscopy (AES) is a method of chemical analysis that uses the intensity of light emitted from a flame, plasma, arc, or spark at a particular wavelength to determine the quantity of an element in a sample. The wavelength of the atomic spectral line gives the identity of the element while the intensity of the emitted light is proportional to the number of atoms of the element. Atomic emission spectroscopy that concerns the emission of radiation by the suitably excited atomic vapors of the analyte. Here, the emitted radiation and its intensity form the basis for the qualitative and quantitative applications of the technique. It is a multi-element analytical technique that can be used for the analysis of materials in gaseous, liquid, powder or solid form. Its high detection power and wide variety of excitation sources makes it the most extensively used method for analysis. Of the various different atomic emission spectroscopic, an inductively couple plasma (ICP) acts as an atomization- excitation source; the technique being called ICP-availability of sufficiently sensitive interference free spectral lines.

Keywords: Inductively coupled plasma (ICP), Atomic emission spectroscopy (AES), X-Ray fluorescence (XRF), Radio frequency (RF), Charge coupled devices (CCDs) or Charge injection devices (CIDs).

INTRODUCTION

Inductively coupled plasma atomic emission spectroscopy (ICP-AES), also referred to as inductively coupled plasma optical emission spectrometry (ICP-OES), is an analytical technique used for the detection of trace metals. It is a type of emission spectroscopy that uses the inductively coupled plasma to produce excited atoms and ions that emit electromagnetic radiation at wavelengths characteristic of a particular element. The intensity of this emission is indicative of the concentration of the element within the sample [1].

ICP-AES is an emission spectrophotometric technique, exploiting the fact that excited electrons emit energy at a given wavelength as they return to ground state. The fundamental characteristic of this process is that each element emits energy at specific wavelengths peculiar to its chemical character. Although each element emits energy at multiple wavelengths, in the ICP-AES technique it is most common to select a single wavelength (or a very

few) for a given element. The intensity of the energy emitted at the chosen wavelength is proportional to the amount (concentration) of that element in the analyzed sample. Thus, by determining which wavelengths are emitted by a sample and by determining their intensities, the analyst can quantify the elemental composition of the given sample relative to a reference standard [2].

ICP-AES analysis requires a sample to be in solution. Thus, interstitial waters can be analyzed simply, requiring only dilution in most cases. Igneous rocks, sedimentary rocks, and sediments, however, must be dissolved. This can be achieved either by a combined acid attack employing HF, HNO₃, and HCl acids, or by a LiBO₂ flux-fusion technique similar to that used for XRF preparation. In addition to being somewhat dangerous for routine shipboard work (because of the highly reactive nature of HF), the acid attack is not able to generate consistent and reliable data for Si because it volatilizes in

the presence of HF. The acid digestion procedure also often results in incomplete analysis of refractory elements such as Ti, Cr, and Zr because their host minerals are often difficult to dissolve. The flux-fusion approach is employed on board the *Resolution* for several reasons: (1) it is safer because HF is not involved; (2) it is a complete dissolution technique, allowing determination of all elements, including Si and the refractory elements; (3) the resultant solutions are similar in composition (or matrix) because they are dominated by the presence of the LiBO_2 flux; and (4) the solutions are stable in dilute HNO_3 acid and can be transported safely back to the scientist's laboratory for further shore-based study [3].

All ICP-AES systems consist of several components. We shall focus this discussion on three main aspects: the sample introduction system, the torch assembly, and the spectrometer. The sample introduction system on the ICP-AES consists of a peristaltic pump, Teflon tubing, a nebulizer, and a spray chamber. The fluid sample is pumped into the nebulizer via the peristaltic pump. The nebulizer generates an aerosol mist and injects humidified Ar gas into the chamber along with the sample. This mist accumulates in the spray chamber, where the largest mist particles settle out as waste and the finest particles are subsequently swept into the torch assembly. Approximately 1% of the total solution eventually enters the torch as a mist, whereas the remainder is pumped away as waste [4].

Humidification of the Ar gas injected into the nebulizer is important when analyzing samples with high dissolved solids, as is often the case with analysis of ODP rocks, sediments, and interstitial waters. Humidification takes place in the Ar humidifier, where Ar is bubbled through deionized water prior to its expulsion in the nebulizer.

The fine aerosol mist containing Ar gas and sample is injected vertically up the length of the torch assembly into the plasma. There are several recommended Ar flow rates used in the torch, as described in detail in the owner's manual and in the various publications provided. The radio frequency-generated and maintained Ar plasma, portions of which are as hot as 10,000 K, excites the electrons. When the electrons return to ground state at a certain spatial position in the plasma, they emit energy at the specific wavelengths peculiar to the sample's elemental composition.

The plasma is viewed horizontally by an optical channel. Light emitted from the plasma is focused through a lens and passed through an entrance slit into the spectrometer. There are two types of spectrometers used in ICP-AES analysis: sequential (monochromator) and simultaneous (polychromator). The JY2000 has a sequential spectrometer. This means that the diffraction grating in the spectrometer is analogous to a prism that refracts visible light into its component colors. The detector (photomultiplier tube) is fixed in space at the far

end of the spectrometer. Rotation of the diffraction grating sequentially moves each wavelength into the detector. The computer control ensures that the detector is synchronized with the grating so that the intensity at the detector at any given time is correlated with the wavelength being diffracted by the grating. The operator enters the wavelengths that he or she wishes to detect into the computer, the grating sequentially moves to the specified wavelengths, and the energy intensity at each wavelength is measured to provide a quantitative result that can be compared to a reference standard. Using standard spectroscopic techniques, sequential ICP-AES can provide extremely flexible and rapid analysis of a number of chemical elements. The spectrometer is flushed with N_2 gas to improve the detection limits of elements with emission wavelengths that are severely compromised by interference with air. This N_2 flush, which is constantly maintained in the instrument regardless of whether such elements are being analyzed, also protects the optics from the corrosive aspects of the atmosphere, which are particularly acute at sea [5].

MECHANISM

The ICP-AES is composed of two parts: the ICP and the optical spectrometer. The ICP torch consists of 3 concentric quartzglass tubes. The output or "work" coil of the radio frequency (RF) generator surrounds part of this quartz torch. Argon gas is typically used to create the plasma.

When the torch is turned on, an intense electromagnetic field is created within the coil by the high power radio frequency signal flowing in the coil. This RF signal is created by the RF generator which is, effectively, a high power radio transmitter driving the "work coil" the same way a typical radio transmitter drives a transmitting antenna. The argon gas flowing through the torch is ignited with a Tesla unit that creates a brief discharge arc through the argon flow to initiate the ionization process. Once the plasma is "ignited", the Tesla unit is turned off.

The argon gas is ionized in the intense electromagnetic field and flows in a particular rotationally symmetrical pattern towards the magnetic field of the RF coil. A stable, high temperature plasma of about 7000 K is then generated as the result of the inelastic collisions created between the neutral argon atoms and the charged particles [6].

A peristaltic pump delivers an aqueous or organic sample into an analytical nebulizer where it is changed into mist and introduced directly inside the plasma flame. The sample immediately collides with the electrons and charged ions in the plasma and is itself broken down into charged ions. The various molecules break up into their respective atoms which then lose electrons and recombine repeatedly in the plasma, giving off radiation at the characteristic wavelengths of the elements involved.

In some designs, a shear gas, typically nitrogen or dry compressed air is used to 'cut' the plasma at a specific spot. One or two transfer lenses are then used to focus the emitted light on a diffraction grating where it is separated into its component wavelengths in the optical spectrometer. In other designs, the plasma impinges directly upon an optical interface which consists of an orifice from which a constant flow of argon emerges, deflecting the plasma and providing cooling while allowing the emitted light from the plasma to enter the optical chamber. Still other designs use optical fibers to convey some of the light to separate optical chambers.

Within the optical chamber(s), after the light is separated into its different wavelengths (colors), the light intensity is measured with a photomultiplier tube or tubes physically positioned to "view" the specific wavelength(s) for each element line involved, or, in more modern units, the separated colors fall upon an array of semiconductor photodetectors such as charge coupled devices (CCDs). In units using these detector arrays, the intensities of all wavelengths (within the system's range) can be measured simultaneously, allowing the instrument to analyze for every element to which the unit is sensitive all at once. Thus, samples can be analyzed very quickly.

The intensity of each line is then compared to previously measured intensities of known concentrations of the elements, and their concentrations are then computed by interpolation along the calibration lines. In addition, special software generally corrects for interferences caused by the presence of different elements within a given sample matrix.

Atomic Emission Spectroscopy Based on Plasma Sources

Plasma is a high energy source which is an electrically neutral conducting gaseous mixture having a significant concentration of cations and electrons. As an electrical conductor it can be heated inductively by coupling with an oscillating magnetic field. The temperature of the plasma may be of the order of 5,000 to 8,000 K. The plasma based AES in principle, is similar to the flame photometry; the only difference being that flame is replaced by much more energetic atomization-excitation processes using plasma. In emission work, the argon plasma is frequently employed.

The analyte sample is introduced into the centre of the plasma as an aerosol with the help of a nebuliser using argon flow. As in a flame, in plasma also the sample undergoes various transformations like desolvation, vaporisation, atomisation, ionisation and excitation. The excitation and ionisation occurs due to collisions of the analytical atoms with high energy electrons. The high temperature assures that most samples are completely atomised, however, some molecular species e.g., N_2 , N_2^+ , OH, C_2 , etc. do exist in the plasma and are measurable. As the energy of the plasma source is quite high it ensures the

excitation of the atoms of all the elements present in the sample which then relax by emitting EM radiation of characteristic wavelengths of different elements. Thus, it is a multielement technique.

Depending on the composition of the analyte, the excited species consist of atoms, singly charged ions and sometimes the doubly charged ions. The emission lines observed are mainly from the excited atoms and singly charged ions; the emissions from the doubly charged ions however are relatively rare. The energies of the transitions are such that the emitted radiation falls in the ultraviolet and visible region of the spectrum i.e. between 160-900 nm. The emission lines from the atomic and ionic species are very narrow; the width being less than 5 pm [7].

PLASMA AND ITS CHARACTERISTICS

An ideal atomization-excitation source should have the following characteristics.

1. It should completely separate the analyte from its original matrix so as to minimise interferences.
2. It should have appropriate energy to ensure complete atomisation but keep ionisation to a minimum.
3. It should provide an inert environment, so as to keep the undesirable molecular species formation to a minimum.
4. It should have none or negligible background radiation.
5. It should provide for the analysis of samples in all possible forms like, solids, liquids, gases or slurries.
6. Of course, it should be inexpensive, need minimal maintenance and be easy to operate [8].

As always, it is difficult to attain ideality, however, plasma sources are quite promising for the purpose of atomisation-excitation job in atomic emission spectroscopy. In this, plasma source is an electrically neutral, highly ionised gas that consists of ions and electrons. It is sustained by absorbing energy from an electric or magnetic field. Three types of power sources have been employed in argon plasma spectroscopy. One is a powerful radio frequency generator that powers an induction coil, the magnetic field generated from it help in establishing the plasma. The second is a *dc* electrical source capable of maintaining a current of several amperes between electrodes immersed in the argon plasma and third one is a microwave frequency generator through which the argon flows. Accordingly, there are three types of plasma sources.

- Inductively coupled plasma (ICP)
- Direct current plasma (DCP)
- Microwave induced plasma (MIP)

Of the three, the radio-frequency, or inductively coupled plasma (ICP), source appears to offer the greatest advantage in terms of sensitivity and freedom from interference. On the other hand, the *dc* plasma source (DCP) has the virtues of simplicity and lower cost.

Inductively Coupled Plasma

The inductively coupled plasma (ICP) is plasma induced by radiofrequency. The energy transfer is mediated by an induction in that produces a magnetic field which helps in establishing and sustaining the plasma. The energy of a high frequency generator is transferred to a gas, generally argon, flowing at atmospheric pressure. A typical inductively coupled plasma source is called a torch. The most common ICP torch in use today has evolved over decades of development. It consists of a quartz tube whose upper part is surrounded by a radiofrequency work coil. The torch assembly is designed to deliver gases so that stable argon plasma is formed at the open end through which the sample aerosol can be injected [9].

Direct Current Plasma

In direct current plasma (DCP) the external source of energy is a *dc* electric source. The most recently developed DCP is the three electrodes DCP or inverted - Y shaped plasma. It has two anodes set at an included angle of about 60° and a cathode. The anodes are made up of graphite and are surrounded by ceramic sleeves. The cathode block consists of a small tungsten electrode also surrounded by a ceramic sleeve. The argon gas is introduced through the sleeves around the anodes and transfers the discharge to the cathode block. The sample in the form of an aerosol is introduced at a rate of about 2 cm³ /min below the intersection of the two anodic columns. The spectral observation region is just below this intersection. The background radiation is relatively low. However, the excitation region is relatively small, and good spatial stability is essential to prevent drifts in observed intensities. The DCP is to some matrix effects and fairly severe ionisation interferences, i.e. enhancement or depression in intensities due to the presence of an easily ionisable element. It has been reported that the detection limits for DCP-AES are inferior to those obtainable by ICP-AES

Microwave Induced Plasma

In a microwave induced plasma (MIP) source frequencies in the microwave region are used as external energy source. A microwave radiation of a frequency of 2450 MHz is commonly used. A microwave induced plasma is obtained when the ultrahigh frequency *ac* power is capacitatively coupled into a stream of noble gas (helium or argon) or nitrogen at about 3 dm³ /min. in a resonant cavity [10].

Low power MIP (50 to 100W) cannot accept solution aerosols without prior desolvation, high power MIP (800W) are more robust and can vaporise and atomise aerosols without desolvation. The MIP is also useful for the excitation of volatile hydride forming elements after stripping the hydrides from the excess of hydrogen. With different trapping techniques, detection

limits down to the sub-ng level can easily be obtained.

In one of the microwave plasma torches the microwave power generated by a 2 kW magnetron is guided to the tip of the torch using waveguides. A conical hollow nozzle acts as the tip of the torch through which the plasma gas argon flows. As there is only one flow channel, the sample is also injected through the same. The small size of microwave sources is an advantage over the ICP because it makes it more flexible and less expensive.

INSTRUMENTATION INVOLVED IN ICP FORMATION

Quartz Tube

The body of the torch consists of three concentric quartz tubes; the diameter of the largest tube being about 2.5 cm. The tube has three separate gas inlets. The argon gas enters the plasma through the outer channel with a tangential flow pattern at a rate of 8 – 20 L min⁻¹. The gas traveling up the central channel is called auxiliary gas and also has a tangential flow pattern. The innermost third inlet is connected to the nebuliser from which the gas enters the plasma along with the sample in a laminar flow.

Radio Frequency Power Generators

The majority of radiofrequency (rf) power generators are crystal controlled radiofrequency generators. These are designed to operate within the industrial frequency bands of 27.12 or 40.68 MHz, although plasma generators have been described operating at between 5 to 100 MHz. The radio frequency at which the torch operates is an important parameter in sustaining plasma operation. At the lower frequency range it is necessary to deliver large power up to 30 kW to sustain the plasma. At a frequency of 27.12 MHz, the generators are normally designed with maximum power ratings of between 2 and 5 kW, and are often used routinely to deliver about 1 kW to the plasma. The radiofrequency power supply is so controlled that it ensures a constant power transfer to the induction coil [11].

Work Coil

The work coil consists of a water-cooled induction coil that is powered by a radio frequency generator. The copper induction coil is wrapped two or three times around the ICP torch and has water flowing through it for cooling purposes. The lines of force, generated by the magnetic field, are directed along the axis of the solenoid inside the tube and take the form of an ellipse on the outside.

Argon Gas Supply

The conventional argon plasma torch uses between 13 and 20 liters of argon per minute, mainly to ensure that the outer quartz glass tubing adjacent to the plasma is adequately cooled. Such a high consumption of argon gas is met by installing a liquid argon supply

Dewar flask rather than relying on conventional compressed gas cylinders.

Mechanism of Plasma Formation

The ionisation of the argon gas flowing through the crystal tube inside the solenoid is initiated by a spark from a Tesla coil. The resulting ions and the electrons are subjected to the fluctuating magnetic field produced by the induction coil. This makes them flow in the closed annular paths within the coils. The induced or eddy currents are thus produced. These electrons rapidly acquire enough energy from the oscillatory field generated by the induction coil and sustain a high degree of ionisation. This leads to the formation of ring shaped toroidal plasma. The spacing between coil and torch, the distance between individual copper coils and the concentricity of both coil and torch are important parameters in the formation of stable plasma.

Appearance of ICP plasma

The typical plasma has a very intense, brilliant white, nontransparent core topped by a flame like tail. The analytical zone has a temperature of 5000-8000 K and is approximately 1 cm above the coils. It offers the best optical viewing area for maximum sensitivity. The plasma emits a continuum of background radiation that extends from the visible into the ultraviolet region. The radiation originates from electrons, Ar and Ar⁺ ions, as well as various atomic and molecular species in the matrix and their recombination products. In the region 10 to 30 mm above the core, the continuum fades, and the plasma is optically transparent. The spectral observations are generally made at a height of 15 to 20 mm above the induction coil. Here the background radiation is remarkably free of argon lines and is well suited for analysis [12].

INSTRUMENTATION FOR ICP-AES

The essential components of a plasma based atomic emission spectrometer are as given below.

- Plasma source: an atomisation-excitation device
- Nebuliser : a sample introduction device
- Monochromator: a dispersion device
- Detector: a radiation detection device
- Processing and readout device

The sample in the solution form is nebulised and injected into the plasma source with the help of a suitable nebuliser. The emitted radiation is passed through monochromator and after dispersion it is detected by a photodetector and sent to the processing unit for the processing and generating suitable output.

Sample Introduction

Inductively coupled plasma emission spectroscopy is used primarily for the qualitative and quantitative analysis of samples that are dissolved or

suspended in aqueous or organic liquids. The sample is brought as a fine aerosol into the plasma, which generally is achieved by pneumatic nebulisation. These are carried into the torch by argon gas flowing at a rate of 0.3 to 1.5 dm³/min through the central quartz tube. The aerosol enters the hot plasma zones with a low injection velocity so as to have a residence times of the order of ms that is required for an efficient atomization and excitation. Thus, there are two steps in the sample introduction. These are as follows.

- a) Sample preparation
- b) Nebulisation

a) Sample preparation

The solution preparation for the analysis using ICP-AES depends on the nature of the sample and the concentration of elements to be determined. There are two main types of sample preparation methods used for ICP analysis. These are explained below.

i) Acid digestion method

In the acid digestion method, the acids use their oxidising or reducing properties for the dissolution of the analyte. You would recall that acid digestion generally produces a clear solution of the analyte without loss of any of the elements to be determined. However, one must take care of the possible loss of volatile elements. For example, in the acid digestion using HCl, there is a possibility of the loss of As, Se, Sn as their volatile chlorides. Similarly, in case of sulphuric acid, the formation of precipitates of Ca, Ba, Pb is also a potential source of error [13].

Another aspect that needs attention of the analyst is that the quantity of acid or flux in the solution must be as low as possible so that there is minimum perturbation of the plasma. Some of the acids perturb the plasma more than the others. Accordingly, there is an order of preference of the acid to be used for sample preparation. The decreasing order of their preference is as follows. HNO₃ > HCl > HClO₄ > H₂SO₄ > H₃PO₄

ii) Dry attack method

In dry attack method, the sample preparation involves an alkaline fusion as well as high temperature calcination (450 - 600 °C) followed by acid recovery of ashes. However in this method the losses due to volatilization and insolubilisation are not negligible. More so there is a possibility of contamination of the solution due to the reagents.

b) Nebulisation

The analyte samples in all three states (solid, liquid, gas) have been successfully introduced into an ICP. For solutions, a nebuliser is used to convert the liquid stream into an aerosol consisting of particles that are 1–10 mm in diameter. Direct injection of liquids into the plasma would either extinguish the plasma or cause the atoms to be improperly desolvated, making excitation and emission

less efficient. You have learnt about nebulisation and different types of nebulisers in. Let us learn about different types of nebulisers employed in ICP-AES.

Nebulisers for ICP-AES

Nebuliser is a device that converts a solution of the analyte into finely divided droplets that are carried into the atomiser. You would recall that in concentric type pneumatic nebulisers the sample solution is aspirated through a capillary by the flow of a nebulising gas using Bernoulli's principle and is converted into a fine aerosol by the gas jet.

In a cross flow nebuliser, another type of pneumatic nebuliser, the aspirated sample is nebulised by a stream of argon, and the resulting finely divided droplets are carried into the plasma. A schematic diagram of the two types of pneumatic nebulisers used in ICP-AES [14].

- ❖ In a frit nebuliser, the sample solution is pumped to a glass frit membrane, consisting of a porous coral like synthetic material. The argon gas passes through the membrane and converts the sample into an aerosol spray. This is then directed into the plasma.

- ❖ Ultrasonic nebuliser, the sample is pumped on to a piezoelectric crystal vibrating at ultrasonic frequencies (50 kHz to 4 MHz). The vibrations of the crystal break the droplets into smaller particles which are transported to the plasma. Larger aerosol drops are drained out. A schematic diagram of the ultrasonic nebuliser used in ICP-AES

The electrothermalvaporisation and the hydride generation as the methods of introducing the sample in the atomiser. In electrothermalvaporisation method the sample is vapourised on a conductor like carbon rod or a heated metal and is carried by argon to the plasma. On the other hand in hydride generation method, some elements are converted into volatile hydrides which are then directed into the plasma. The schematic diagram of electrothermalnebuliser used in ICP-AES [15].

Electrothermalvaporisation coupled with a plasma torch offers the microsampling capabilities and low detection limits of electrothermal furnaces while maintaining the wide linear working range, the freedom from interference, and the multielement capabilities of ICP.

Monochromators

Emission from plasma is usually polychromatic in nature which implies that it consists of a large number of radiation of different wavelengths. More so, the bandwidths of optical emission lines from the plasma are typically of the order of 0.001 nm. These need to be suitably dispersed to analyse for the possible elements in the analyte. Resolution is important because of the complexity of ICP emission spectra, in terms of very large number of individual emission lines observed from

samples. If not resolved, it would cause extensive spectral interference. In ICP-AES spectrometers, the resolution of the complex spectra is achieved by using ruled grating monochromators having a large number of grooves on the grating. Some instruments on the other hand employ holographic gratings [16].

Detectors

There are three basic types of detector systems. These are photomultiplier tubes (PMTs), photodiode arrays (PDAs), and charge coupled devices (CCDs). Most of the instruments use photoelectric means of detection in the form of photomultiplier tubes (PMTs), or with photodiode arrays. In some cases photoplates are also used for detection purposes in AES. A number of modern instruments have now switched to solid state array detectors that use charge coupled devices (CCDs) or charge injection devices (CIDs).

Processing and Readout Device

Whether the detection of the analyte signal is done sequentially or simultaneously, a large amount of data needs to be handled in terms of the positions of the spectral lines and their intensities. Therefore the processing of the data needs good computers along with multichannel analog to digital converter to acquire and save the detected signals for further processing. The results of the determinations are printed out by a dedicated printer [17-18].

APPLICATIONS OF ICP-AES

The ICP-AES technique is versatile tool in the hands of analytical chemists. As many as 60 elements can be determined by it in a wide range of analyte samples such as rocks, minerals, soil, air, water, agriculture, forestry ecology, food analysis, etc. Therefore it has become an indispensable technique.

- ❖ **AGRICULTURAL SCIENCE:** Analysis of agricultural products and foods besides soil analysis.

- ❖ **HEALTH SCIENCES:** Determination of Aluminium in blood, Cu in brain, Se in liver, Na in breast milk

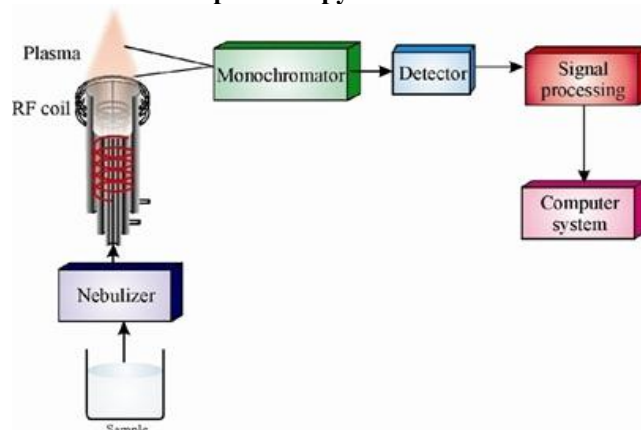
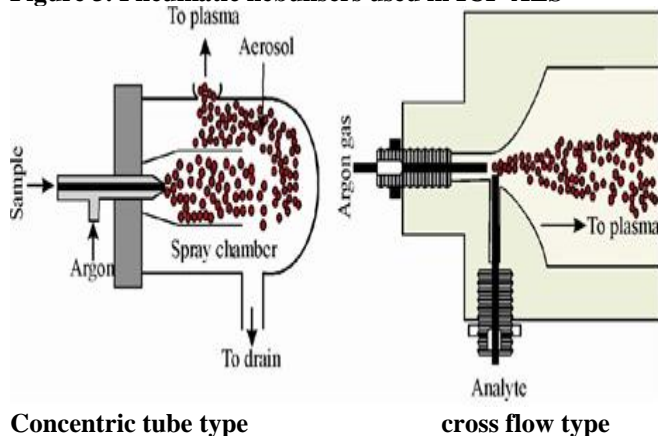
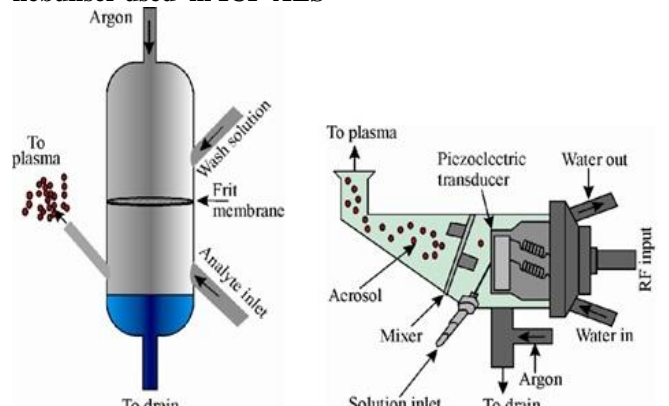
- ❖ **GEOLOGICAL SCIENCES:** Presence of lanthanides and other elements in rock samples

- ❖ **FORENSIC SCIENCES** Crime scene soil analysis

- ❖ **METALLURGY:** Analysis of trace elements in stainless steel.

- ❖ **ENVIRONMENTAL SCIENCE:** Waste water analysis, determination of pollutant metals in variety of matrices.

- ❖ **PHARMACEUTICAL INDUSTRY:** Presence of metals like Cu, Fe, Ni, and Si in lubricating oils or gasoline at tracer concentration. Traces of metals like Ca, Cu, Fe, Mn, Mg, P, K and Zn in beer or wine; determination of trace elements in polymers, evaluation of catalysts, and so on [19].

Figure 1. ICP-AES Spectrometer**Figure 2. A schematic layout of different components of an ICP-AES spectroscopy****Figure 3. Pneumatic nebulisers used in ICP-AES****Figure 4(a). Frit nebuliser and (b) ultrasonic nebuliser used in ICP-AES****REFERENCES**

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