




# Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES): a Powerful Analytical Technique for Elemental Analysis

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## Abstract

ICP-OES is a powerful, versatile, and advanced analytical technique with excellent detection properties. Due to its extraordinary features, it has been widely employed for the analysis of a wide variety of chemical elements in the past few years with great success. It offers the least detection time, lower detection limits, broader linear dynamic range, and greater matrix tolerability as well as negligible chemical interferences. Beyond this, it can handle multiple varieties of samples including aqueous, inorganic, organic liquids, and solids as well. It comprises complex instrumental makeup which enables it to detect up to 2 to 70 elements simultaneously with great accuracy. Recent reports evidenced that this hyphenated technique has been employed in several analytical determinations including food analysis, agricultural investigations, geological studies, drug/metabolite analysis, and environmental and forensic sciences. In the present compilation, a detailed description of the fundamental principles of ICP-OES has been provided along with its various sophisticated functional components. Also, the reported applications of this technique in different fields have been discussed highlighting the basic experimental setups and outcomes by presenting the data as tables. This summarization may be helpful to analysts to get insights into the working as well as performance characteristics of this technique.

**Keywords** ICP-OES · Detection limits · Food analysis · Geological · Nebulizers · ICP torch

## Introduction

In the last few years, the inductively coupled plasma optical emission spectrometry (ICP-OES) technique emerges out as the most prominent and exploited technique within the pharmaceutical industry (Giussani et al. 2009). It is a modern and multi-functional analytical technique that is primarily used for the analysis of an element in different samples like environmental, industrial, plant, tissue, and pharmaceutical samples with a wide variety of concentrations (Li et al. 2020). Inductively coupled plasma with optical emission spectrometry (ICP-OES) is one such hyphenated technique having the capability of rapid and multiple elemental analysis with much greater sensitivity like parts per billion (ppb) to parts per million (ppm) with very little interference (Pollard et al. 2007). It is based upon the spontaneous photons

emission from ions or atoms whose excitation takes place in a radio frequency discharge also these photons possess some characteristic energies which are mainly determined by the help of a quantized level of energy structure for the ions and atoms (Hou et al. 2006). It is a versatile sensitive technique with low detection limits, fewer chemical interferences, and a shorter time-consuming process in comparison to other spectrometric techniques which results in specific and accurate outcomes (Charles and Fredeen 1997). Nowadays, ICP-OES has proved to be an important instrument in elemental impurities determination in various sample matrices and is being used in the various pharmaceutical industries to measure trace metal contents of active pharmaceutical ingredients (API) or intermediates against specification limits specified in various pharmacopeias resulting in efficient findings up to parts per billion scale and gives high purity (Balaram 2016).

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## Fundamental Principles and Modes of Operation

The basic principle of inductively coupled plasma optical emission spectrometry (ICP-OES) depends upon the spontaneous emission of photons generated from ions or atoms and these ions and atoms excited by a radio frequency discharge (Majumdar and Dubey 2017). In ICP-OES, firstly the samples were introduced into inductively coupled argon plasma through a sample injection system in which molecules are atomized and ionized in the plasma and eventually excited (Soltanpour et al. 1996). When electrons return from an excited energy level to a lower energy level, ions and atoms release photons which are called ionic or atomic emission, and the emission is then collected to a detector using Echelle or Rowland circle optics, which separate different emission wavelengths from each other (Kulander et al. 1993; Ghosh et al. 2013). The detector then measures the emission signal intensities of respective wavelengths and this emission wavelength corresponds to the energy difference between the excited state and ground state of the electron and the emission signal strength is proportional to the element's abundance in the plasma (Bibinov et al. 2007; Thiagarajan et al. 2004; Aguilera et al. 2009).

In ICP-OES, a sample usually introduced as a liquid sample into the instrument that is injected towards the central channel of radiofrequency (RF) induced argon plasma, where a liquid sample is transformed in the form of an aerosol called as the aerosol nebulization process, whereas solid samples previously required the extraction of the sample or acid digestion and the gas samples could be introduced directly into the instrument (Donati et al. 2017; Bendicho and de Loos-Vollebregt 1991).

At its core, ICP-OES maintains very high temperatures (8000–10000K) to rapidly dry and vaporized the aerosol, releasing the elements as an analyte in the gaseous state as free atoms (Warren 1993). Additionally, the aerosol sample conveyed to the plasma where by the help of plasma sample desolved, vaporized, atomized, and excited as well as ionized to convert them into ions and promote them to an excited state by emitting photons and the ground state is then achieved by both excited atoms and ions (Walkup et al. 1986). Then, the photons of ICP-OES are collected with the help of a concave mirror or lens, and this focusing optic lens forms an image of the ICP on the entrance aperture with the help of a wavelength selection device named a monochromator and the selected specific wavelength then converted by a detector mainly of a photodetector which converts them into an electric signal which is then processed and amplified by integrators and recorded by a computer (Radehaus et al. 1998; Wilson et al. 1996). The monochromator is like a simple monochromator or

photomultiplier tube (PMT), which can be used for single element detection while the combination of polychromator and advanced array detector is used to perform simultaneous multielement determination (up to 70 elements) (Thompson 2012).

## ICP-OES Edge over ICP-MS

ICP-OES is a recently developed technique for trace elements analysis compared to the ICP-MS technique and carries certain differences (Olesik 1991). The ICP-OES and ICP-MS both are using the same type of source but the main difference is that the ions of the analyte formed in the ICP-OES are sent with the help of radiofrequency discharge (RF) while in the ICP-MS technique analyte ions are transported through a mass spectrometer where the ions of analyte are separated through the magnetic field according to their mass to charge ratio ( $m/z$ ) (Thomas 2001; Tyler and Jobin 1995; Schenk and Almirall 2012). The ICP-OES technique is not ideally suited as a part of the sample introduction system with any other chromatographic technique like gas chromatography (GC) or liquid chromatography (LC), whereas the ICP-MS is ideally suited with chromatographic techniques (Duyck et al. 2007). The mass detector coupled with ICP-MS makes this hyphenation technique highly sensitive and highly specific as compared to ICP-OES and this hyphenation technique leads to the identification of the compound via mass to charge ratio (Kannamkumarath et al. 2002).

## Basics Instrumentation of ICP-OES

The fundamental components and layout of a typical ICP-OES instrument have been outlined in Fig. 1. The ICP-OES instrument consists of nebulizers, spray chambers, sample injector, ICP torch, RF generator, optics or the spectrometer, emission detectors, signal processing, and software.

### Nebulizers

In the case of the ICP-OES instrument, firstly samples are converted into liquid form, and with the help of a peristaltic pump, samples are pumped into the instrument by a device named a nebulizer which converts the liquid into an aerosol or mist (Bruins et al. 1987). In ICP-OES, one of the critical steps is the nebulization process in which all the liquid samples should be converted into an aerosol by a perfect nebulizer in such a way that the discharge of plasma could desolvate, vaporize, atomize, and ionize as well as be excited in a consistent manner (Lienemann et al. 2007). In ICP-OES instruments, two types of nebulizers are mainly used, the first one is the Pneumatic type nebulizer (Fig. 2) which makes use of high-velocity gas flows to produce an aerosol as well as available commercially. The second one



droplets as compared to cross-flow nebulizers due to the large capillary tube used inside them which makes them comparatively more clogging resistant well as it is made up of other than glass or quartz which makes them more robust and corrosion-resistant (Wang 2004). The first two pneumatic nebulizers are not so popular, the third class of pneumatic nebulizer is the Babington nebulizer in which the liquid samples are allowed to flow through a small orifice which is over a smooth surface, and the argon gas of high-speed scissors the liquid sample sheet into an aerosol sheet (Wang 2004). As this type of nebulizer is having a large hole or sample-path which makes them less likely to clog, due to this the sample having high viscosity as well as high total dissolved solids (TDS) can be nebulized by this kind of nebulizer (Soltanpour et al. 1996). The Babington nebulizer having one of the variants named as V-groove nebulizer which is also explained here. In the V-groove nebulizer, a small hole in the center through which argon gas of the high-speed is flowing and the samples flow down to a groove rather than flowing towards the nebulizer smooth surface. It is used for the nebulization of sample solutions like sea water, urine samples because it contains high salt content (Charles and Fredeen 1997). The last category in the nebulizer is the ultrasonic nebulizer (USN) in which a sample is run towards an oscillating piezoelectric transducer which breaks the liquid sample into extremely fine aerosols due to the transducer's rapid oscillations. In terms of efficiency, the USN type nebulizer is 10–20% more efficient as compared to 1–2% of other nebulizers like concentric or cross-flow nebulizers because a large number of samples are reaching the ICP-discharge as well as the instrument equipped with USN type nebulizer has much more sensitivities which are of about 10–50 times more as compared to concentric or cross-flow nebulizer (Charles and Fredeen 1997).

### Spray Chamber

The spray chamber is used to carry out the nebulized sample from the nebulizer to the ICP torch, where it is fed to the plasma discharge (Jin et al. 1991). It is placed between the torch and the nebulizer for this purpose (Jin et al. 1991). The secondary function of the spray chamber is too diminished smooth out pulses that are produced by the peristaltic pump that occur during nebulization. In an ICP-OES instrument, a spray chamber is designed in such a way that allows only the droplets having a diameter of about 10 microns or smaller to pass into the plasma and the sample droplet of 1–5% range that may reach the plasma and the remaining 90–95% sample is drained out into a waste container. The spray chamber is made up of quartz or other corrosive resistant materials that allow the introduction of the samples containing hydrofluoric acid as well as nebulized into the plasma (Fassel and Kniseley 1974; Yurkov 2017).

### Sample Injector

The sample injector is used for introducing the sample and in general, the sample required in the form of liquid for the analysis purpose. In ICP-OES, the ability to introduce samples in the form of solid or gas would extend the technique's applicability and the sample that occurs naturally in the form of solid which required converting them into a liquid form for ICP-OES analysis (Carey and Caruso 1992). For ICP-OES analysis, various types of sample introduction systems are established, but the hydride generation method is the most popular and well-liked. In the hydride generation method, samples required for the analysis are converted into the liquid form, after that dissolving the samples in dilute acid, the samples are mixed with the reducing agent which is a solution of sodium borohydride in dilute sodium hydroxide. After that, the compounds are separated and carried to the plasma by a high stream of argon gas, with excellent sensitivity and detection limit (Shen et al. 2015; Torres et al. 2007; Makishima and Nakamura 2009). The second technique for the sample introduction system is the electrochemical vaporization (ETV) or graphite furnace technique. In this technique, electrochemical vaporization is mostly utilized to vaporize a liquid or solid sample small portion in research facilities, and the remaining vapor is carried out by high stream argon gas to the plasma discharge as well as the introduction of samples are non-continuous, but in the case of an ICP-OES spectrometer, the transient signal capability and processing is required (Wang 2004; Carey and Caruso 1992). In this technique, the greatest sensitivity is possible to achieve but other aspects like optimized operations conditions like analysis of more than one element as well as some severe matrix effects limit the applicability of this technique to the ICP-OES sample introduction system. This technique did not gain much commercial attention and success in ICP-OES, but it acts as a substitute for the sample introduction system in ICP-MS (Wang 2004).

The third technique for sample introduction system is the laser ablation technique in which optimum energy is used to vaporize the sample using a laser beam that is shown over the sample, and a strong stream of argon gas transports the vaporized sample to the plasma discharge. This method applies to a wide variety of samples with different materials like non-conducting, conducting, inorganic, organic, and solid materials (Wang 2004). One of the major drawbacks of this technique is that it is not possible to quantify large number of samples due to lack of matrix-matched standards (Wang 2004). However, this technique is not commercialized (Wang 2004). In a nutshell, sample introduction techniques studies in an ICP-OES instrument have been a broad area and will continue to be an active area of research soon (Günther et al. 1999).

## ICP Torch

In the early days, ICP-OES torches were firstly reported by Fassel and the torches used in today's ICP-OES are quite similar to those reported by Fassel in terms of design and functions. It contains three concentric tubes used for aerosol injection and argon flow and between the two outer tubes the spacing is kept narrow mainly for the introduction of argon gases at high speed between them and the outside chamber are tangentially designed which makes a spiral of the gas around the chamber which further proceeds it upwards (Wolnik 1988). The major function of argon gas is to maintain the torch's walls cool which is made up of quartz and this argon gas flow gradually is called "coolant flow or plasma flow," now it is called as "outer" gas flow which is normally about 7–15 L per minute for argon ICPs. Under the plasma toroid, the chamber directly sends argon gases between the outer flow and inner flow which maintains the plasma discharge apart from the injector tubes and the intermediate which ultimately eases the introduction of an aerosol sample into the plasma. In the normal mode of operation, previously this flow was called auxiliary flow but nowadays the flow of an intermediate gas is about 1.0 L/min and this gas flow is utilized generally when organic samples are analyzed which reduces the carbon formation over the tip of the injector tube (Klostermeier et al. 2005; Wiederin et al. 1991; Johnson et al. 2004). Currently, the most popular ICP-OES torches are of the demountable type which can be removed easily so that only tubes can be replaced or modified concerning the entire replacement of the torch. One of the main advantages of the demountable type torch is the ability to use a variety of injector tubes such as corrosion-resistant ceramic injectors, narrow-bore injectors, and wide-bore injectors as well as lower-cost replacement of ICP-OES torch. The narrow-bore injectors were used for the analysis of organic solvent while wide-bore injectors were used for the introduction of highly dissolved solid content samples (Johnson et al. 2004). A typical outline of the ICP-OES torch is presented in Fig. 4.

## Radio Frequency Generators

A device that supplies the power for the plasma discharge is the radio frequency (RF) generator which is to be generated and sustained and the power of this generator ranges around 700 to 1500 W, which is transferred on every side of the top of the torch to the plasma gas by the help of a coil i.e. load coil which acts as an antenna to convey the power of the radio frequency to the plasma gas and the antenna is made up of copper tubing that is cooled by water or gas during the operation (Sheeran et al. 2016). In ICP-OES, the majority of the radio frequency generators operates at frequencies from 27 to 56 MHz and the specific frequency that

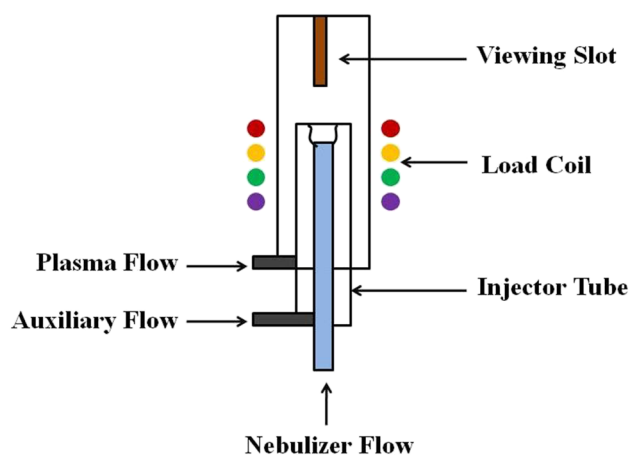


Fig. 4 Outline of an ICP-OES torch

is intended to be used in the instrument of an ICP-OES is set in part by the US Federal Communications Commission (FCC) or related agencies that are nominated as industrial and scientific agencies worldwide (Ghosh et al. 2013). In previous days, most of the ICP-OES generators were operated at 27.12 MHz although, some improvements in reductions of background emission intensity as well as coupling efficiency at this frequency lead to operating at 40.68 MHz in the number of instruments at an increasing rate; however, greater than 40-MHz frequencies were also used they were unsuccessful at commercial scale (Ghosh et al. 2013; Taggart Jr et al. 1987). In ICP-OES instruments, generally, two types of radio frequency (RF) generators were used such as piezoelectric quartz crystal, which produces an oscillating RF signal with the help of crystal-controlled generators then amplified by the radio frequency generator. The second type of radio frequency (RF) generators is the free-running generators which operate based on oscillation frequency which depends on two conditions such as electronic components of the generator circuitry as well as plasma discharge conditions (Ghosh et al. 2013; Sheeran et al. 2016). Some recent advancements in generator design have been taken the lead of this dependency by a free-running ICP-OES generator design that automatically adjusts the output power of the generator and compensate the plasma certain changes and the free-running ICP-OES generators generally made up of simpler parts which are frequently smaller as well as less costly as compared to crystal-controller generators (Charles and Fredeen 1997; Thompson 2012).

## Optics and the Spectrometer

As previously described with the help of the ICP-OES instrument, the emission is collected through a focusing optic named as a concave mirror or a convex lens which generally collects the emission radiation and produces the image



of a discharge on the wavelength-dispersing device's input slit (Ghosh et al. 2013). One of the critical steps in an ICP-OES instrument is the ability to separate the radiation components or to discriminate the spectral radiation from the plasma discharge (Wang 2004). In earlier years of the 1990s, most of the commercially based ICP-OES instruments were created by using the dispersive devices of grating-based and the diffraction grating is merely a mirror which composed of a steel or glass and onto its surface at regular intervals it contained parallel spaced lines or grooves, and dispersion gratings based ICP-OES instrument contained groove density of about 600–4200 lines/mm (Wang 2004). The grating is mostly consolidated in the spectrometer of an ICP-OES instrument, and the spectrometer's function is to convert plasma radiation into a clear beam, disperse it according to wavelength, and focus the component of dispersed light on an exit plane or circle (Ghosh et al. 2013; Thompson 2012). To pass only the selected wavelengths and blocking the wavelengths of undesirable types to the detector, exit slits are used. By using conventional dispersive optics, the multiple elemental analysis is performed in two ways. The first way is the monochromatic way which contains only one exit slit and one detector, and to perform the multi-elemental analysis, monochromators are used which performs quick scanning of wavelength from one wavelength to another wavelength and it is mainly achieved due to diffraction grating angle change or the detector moved to the exit plane when the grating is in the fixed position. The fast sequential multi-elemental analysis is only possible when the monochromator is capable to scan lines with sufficient speed and the most popular configuration in monochromator type is the Czerny-Turner type (Floyd et al. 1980; Palmer and Loewen 2002; Harrison 1973). The second way to perform the multiple elemental analysis, polychromators are used which uses the several exit slits and (all of them are on the periphery location called Rowland circle), and the same spectrometer detectors are used to perform the simultaneous multielement analysis. Each of the exit slit polychromators is aligned to a specific element and the most popular configuration in polychromator type is the Paschen-Runge mount type (Hou et al. 2006; Wang 2004). During recent years, ICP-OES instruments based on the Echelle grating have become very popular due to the combining features of two dispersing systems. In this configuration, two optic components are arranged at right angles, of which is echelle grating whose rulings range from 8 to 300 grooves/mm (Charles and Fredeen 1997; Pennebaker et al. 1999). The 2D array wavelength is having two directions, one for the wavelength and another for the spectral orders, and it has a distinct advantage over conventional grating-based spectrometers in that it produces a very high quantum yield in each spectral order. It also has admirable resolution because it is primarily used in higher spectral orders and the higher spectral resolution obtained

by using higher orders allows for a more compact instrument (Ghosh et al. 2013; Welz 2006).

In the ICP-OES instrument, spectrometers based on the monochromator and polychromator type possess their advantages and disadvantages. In polychromator-based spectrometers, each emission spectrum can be analyzed primarily during the period of whole sample introduction; hence, it has a higher output rate of the sample (Welz 2006). For each spectrum, a direct reader can be used, but the less requirement of space in the system limited the available lines or channels near to about 64 and only spectral lines like 20–30 are fitted with most of the commercial based polychromators (Welz 2006). In monochromator-based spectrometers, flexibility is one the most significant advantages which mean to state that at any time, it is having the capability to access any particular wavelength which is available within the range of the monochromator and enabled us to measure and determined any element emission line through this technique. Additionally, it is having the scanning capability, and scanning is performed only of that particular region which is adjacent to the interest line or measuring the interest line simultaneously of immediate vicinity is significantly helpful in the analytical result validation (Ghosh et al. 2013; Cortez and Pasquini 2013). The instruments based on monochromators are very much suitable for those applications which require complex background corrections which is often impossible in the ICP-OES analysis. In monochromator-based ICP-OES instruments, elements are consecutively measured that require a large amount of sample and it is having a very much low sample through output rate as compared to polychromatic-based ICP-OES instruments (Bilhorn et al. 1987a).

### Emission Detectors

The intensity of dispersed ICP radiations is further measured with the help of a detector (Charles and Fredeen 1997). In earlier, ICP-OES instruments, the photomultiplier tube (PMT) was used which consists of sensitive photo materials such as an anode collection and the photocathode. The dynodes which consist of several electrodes separate the anode and the cathode which provides the multiplication of electron or gain of an electron at each biased of dynode with favorable potential (Ghosh et al. 2013). In photomultiplier tubes, 9–16 dynodes are there which multiply the electrons which are then recorded as response (Rogalski 2012). In the 1960s, solid-state devices based on silicon properties such as transistors and diodes were introduced into the electronic sector in the form of an integrated circuit (IC), which were expanded to the digital sector electronics and the cost of these devices and systems such as digital computers, was gradually reduced (Yu et al. 2015).

Solid-state detectors are light-responsive silicon-based sensors that can be quickly integrated into linear and 2D arrays. PDA, CID, and CCD are the advanced types of commercially available solid-state detectors (Wang 2004; Bilhorn et al. 1987b). Silicon-based detectors are mainly classified into two categories i.e. photoconductive and photovoltaic detectors. The photoconductive detectors are based upon the conductivity of the photodiode and junction detectors which produce a potential difference between the junctions of n-type and p-type semiconductors and work in both photoconductive and photovoltaic modes (Rogalski 2004). In photovoltaic mode, the interaction of light with a photodiode of semiconductors produces an electron-hole pair and both holes and electrons are migrated towards the junction opposite site. In photoconductive mode, across the junction of p-n type semiconductors, the reverse-biased is applied and the conductance across the junction is increased due to electron-hole pairs. The photoconductive mode's conductance and voltage are directly proportional to the intensity of light (Sweedler et al. 1989).

The photo-diode array (PDA) detectors are made up of many individual silicon diodes, which are integrated into the form of integrated circuits (ICs). In photoconductive mode, the PDA detectors operate during the charging of each capacitor which is over the chip and this array detectors is a register of shifts which consists of a series of capacitors closely spaced over the chip which store as well as transfer the signals which are produced either electronically or optically. The PDA detector's stored charge transfers are similar to the brigade bucket in which signals go to side capacitors and sequentially read at the bottom array. PDA detectors for low-level light analysis primarily come along with intensifiers as well as chillers (Mermet and Ivaldi 1993).

The charge-coupled device (CCD) detector consists of a two-dimensional (2D) array of silicon photodiode with the integrated circuits (IC) over the individual detector elements in the order of hundreds or thousands. The working of the CCD detector is very similar to the linear PDA detectors, except for signal digitalization, a multichannel converter converts the signal from analog to digital (A/D). The charge on the detector must be sequentially read as well as it must be destroyed in the meantime (Smith 1997).

The charge-injection device (CID) detector is very much similar to the charge-coupled device (CCD) detector, except for the individual detector elements accessed and controlled. The charge-injection device allows the detector elements to access individually as well as controls each element's exposure time. To put it another way, each element of the detector in the array could be randomly integrated to determine the amount of charge that has accumulated during the time the device has been light exposed (Mermet and Ivaldi 1993). Efficient cooling is highly required in CID detectors because it produces an

inherently higher dark current as compared to PDA and CCD type detectors (Mermet and Ivaldi 1993). Later, for echelle grating-based ICP-OES instruments, a new type of CCD detector known as a segmented array charge-coupled device (SCCD) detector was introduced. It consists of individual smaller sub-arrays collection of 20–80 detector elements despite using a giant charge-coupled device (CCD) of contiguous detector elements in hundreds or thousands of numbers (Smith 1997). In an ICP-OES spectrometry, 70 elements are observed but these subarrays correspond to 200 or more than 200 important elements of ICP spectral lines.

Most of the CID and CCD detectors on commercial based have a lack of sensitivity below the wavelength of 350nm due to the absorption of photons by the electrodes which are embedded on the devices' surfaces (Smith 1997). As we know that there are no embedded electrodes in the individual subarrays of the detector elements in the segmented array charge-coupled device (SCCD) detector, the SCCD detector possesses an excellent light response in the wavelength ranging from 160 to 782nm. Solid-state detectors such as CCDs or CIDs are always fitted to the most popular commercially based ICP-OES instruments with Echelle gratings spectrometers and only the array PDA type detector taking the benefits from the Echelle grating spectrometers' 2D capability. As compared to traditional photomultiplier tube (PMT) detectors, the noise issue of these detectors can be greatly reduced with proper cooling (Smith 1997).

## Signal Processing

In ICP-OES instruments, electronics are used for signal processing through photomultiplier tubes (PMT), and the information they provide is straightforward. The electrical current measured at the anode of a photomultiplier tube (PMT) is converted to computer data and used by the computer for signal processing, and by the use of digital processing signal it converts the voltage signal into a digital signal using an analog to digital (A/D) converter, then sends the digital signal to a computer for signal processing, and finally passes the final result information to the computer system (Ghosh et al. 2013). In automatic ICP-OES instruments, the majority of the function is controlled through an on-board computer and the computer is mainly required for the simpler multi-element analysis as well as it handles the huge amount of data that are produced by the instrument, although there are various types of virtually commercial ICP-OES tools that are available today that use a certain computer type which controls the spectrometer to collect, manipulate, and report as well as the computer control amount over the other function of the instrument and this function greatly varies from model to model (Charles and Fredeen 1997; Ghosh et al. 2013).

## Software

The ICP-OES instrument software is having a feature it can prepare standards and samples with a single keystroke and develop an analytical method as well as apart from analyzing the sample it can also prepare the results. The primary objective of a good software package in the ICP-OES instrument is not limited to the instrument's automatic features during analytical data collection; however, the main objective of the software package is to simplify the instrument overall operation and this simplified operation is mainly important in developing the analytical method and results reporting and not only to running an analysis (Ghosh et al. 2013). The most appropriate parameters for proper analysis during the method development task are wavelengths, a voltage of PMT, background correction points, and the selection of standard concentration, and these parameters are selected only to view the graphically displayed spectral data with the least number of efforts (Ghosh et al. 2013).

## Applications of ICP-OES in Quantitative Analysis

### In Food Analysis

An ICP-OES technique has substantiated itself an ultrafast approach in quantitative analysis of foods. For quantitative analysis of a variety of food materials, a technique called ICP-OES is used to find out the level of essential nutrients in food and level of toxic elements in foods as well as in analysis of food materials. A bit of the noteworthy applications in food analysis has been discussed in the following section.

### In Quantitative Determination of Non-hydride and Hydride Elements in Fish

One of the most important parameters in the testing of food products is quality control which includes the analysis of analytes like nutrients and micronutrients as well as toxic elements in various types of food products at a wide range to ensure that their products are both safe and effective (Drvodelic *n.d.*). The non-hydride and hydride elements like Se, As, Hg, and Sn produce a considerable impact on the health of an individual. The ICP-OES technique with the introduction system of multimode sample is more sensitive as compared to the conventional nebulization technique by delivering accurate results and accurate speed as well as reducing the operating costs (Drvodelic *n.d.*). By utilizing the ultrasensitive and advanced properties of this technique, Neli Drvodelic et al. have carried out a successful estimation of the concentration in fish samples of certain non-hydride and hydride elements (Drvodelic *n.d.*). Using

the dual-mode MSIS accessory, this method was described and quantified in a single analytical run for non-hydride and hydride element analysis in fish tissue (Drvodelic *n.d.*). In this technique, first of all in fish tissue, the non-hydride and hydride-forming elements were recovered and the recovery result was found between the range of 92 and 106 % as well as the method detection limits (MDL) of these elements were found between 0.01 and 0.98 µg/L respectively. Furthermore, calibration curves of all elements were linear with the value greater than 0.999 shows excellent linearity for both non-hydride and hydride elements (Table 1). To determine the non-hydride and hydride elements in fish tissue samples, this developed and validated method has been successfully applied. This method showed high sensitivity, excellent linearity, and high analytical performance in a very short time as well as quite significant in further analytical research for estimation of non-hydride and hydride elements in fish (Drvodelic *n.d.*).

### In Quantitative Determination of Elemental Impurities in Whiskey

Whiskey is one of the most popular spirit-based drinks and it is almost consumed worldwide. It is a spirit drink that is mainly prepared from the malted grains or saccharified by the diastase enzyme of the malt by the distillation process (Nelson et al. *n.d.*). The impurity profiling in whiskey is important to ensure the product's safety and purity. The elemental impurities like Mg, Cu, and Zn produce a significant impact on an individual's health (Nelson et al. *n.d.*). Depending on the quantitative determination of elemental impurities in whiskey samples, different atomic spectroscopy analytical techniques like ICP AES, MP AES, and ICP-MS can be used but among them ICP-OES is an attractive technique mainly for analysis of elemental impurities due to its high sensitivity, faster analysis, better results, and wide multi-elemental coverage (Nelson et al. *n.d.*). By utilizing the above properties of the ICP-OES technique, Jenny Nelson et al. developed a newer analytical technique mainly for the elemental impurity profiling in whiskey based on the ICP-OES technique with Mass Profiler Professional (MPP) software (Nelson et al. *n.d.*). They identified and quantified 69 whiskey samples for elemental impurity profiling which have different characteristics. This method is rapid and accurate for the quantitative elemental impurity profiling in whiskey by ICP-OES method with MPP software (Nelson et al. *n.d.*). In this technique, they found that the elements like Mg, Cu, and Zn demonstrate correlation coefficients of 0.999, 0.999, and 1.000, and linearity over the entire calibrated range is excellent. The method detection limit (MDL) of all elements was found between 0.02 and 8.86 µg/L with read time 20–40 s and QC spike mean recoveries of 3



**Table 1.** Applications of ICP-OES in food analysis

Sr. no.	Analyte	Sample	Technique	Conditions	Run time	MDL	R <sup>2</sup> value	RSD	LOD	LOQ	Recovery	Ref.
1.	Hydride and non-hydride Elements	Fish tissue	ICP-OES MSIS	Pump speed 25 RPM, Rf power 1400W, plasma flow 12 (L/min), aux. flow 1.0 (L/min), nebulizer flow 0.65 (L/min)	20 s	0.01–0.98 µg/L	0.9999	<10%	N.D.	N.D.	92–106%	(Drvodelic <a href="#">n.d.</a> )
2.	Elemental profiling	Whiskey	ICP-OES MPP	Pump speed 12 RPM, Rf power 1200 W, plasma flow 12 (L/min), aux. flow 1.0 (L/min), nebulizer flow 0.70 (L/min)	20–40 s	0.02–8.86 µg/L	0.9999–1.0000	<10%	N.D.	N.D.	90–112%	(Nelson et al. <a href="#">n.d.</a> )
3.	Elements	Edible oil	EIEB ICP-OES	Pump speed 15 RPM, Rf power 1000 W, plasma flow 15 (L/min), aux. flow 1.5 (L/min), nebulizer flow 0.75 (L/min)	30 s	N.D.	0.9998	N.D.	N.D.	N.D.	96–109%	(Bakircioglu et al. <a href="#">2013</a> )
4.	Essential metal intake	Yogurts	ICP-OES	Pump speed 50 RPM, Rf power 1200 W, plasma flow 0.5 (L/min), aux. flow 0.5 (L/min), nebulizer flow 0.5 (L/min)	N.D.	N.D.	N.D.	N.D.	0.0003–1.097 (mg/L)	0.001–3.655 (mg/L)	>96.5%	(Luis et al. <a href="#">2015</a> )

Table 1. (continued)

Sr. no.	Analyte	Sample	Technique	Conditions	Run time	MDL	$R^2$ value	RSD	LOD	LOQ	Recovery	Ref.
5.	Elemental analysis	Nuts and seeds	ICP-OES	Analytical wavelength 120–825 nm, Rf power 1400W, plasma flow 12 (L/min), aux. flow 1.0 (L/min), nebulizer flow 1.0 (L/min)	N.D.	N.D.	0.9911–0.9995	1.1–6.7%	N.D.	0.00072–0.0532 mg/L	95%	(Naozuka et al. 2011)
6.	14 elements	<i>Aesculus hippocastanum</i> seeds and extracts	ICP-OES	Ultrasonic nebulizer, charge-coupled detector, power 1400W, plasma flow 15 (L/min), aux. flow 1.3 (L/min), nebulizer flow 1.2 (L/min)	40–60 s	N.D.	0.9979–0.9999	3.5–11.3%	N.D.	0.001–10 mg/L	95.8–105.7%	(Durante et al. 2021)
7.	15 elements	Vegetable oils and fats	ICP-OES	Pneumatic concentric nebulizer, charge-coupled detector, power 1.4KW, plasma flow 15 (L/min), aux. flow 1.0 (L/min)	0.6 s	N.D.	N.D.	N.D.	0.20–4.5 µg/mL	0.70–59.0 µg/mL	App. 100%	(Martinez et al. 2020)

Table 1. (continued)

Sr. no.	Analyte	Sample	Technique	Conditions	Run time	MDL	R <sup>2</sup> value	RSD	LOD	LOQ	Recovery	Ref.
8.	27 elements	Vegan milk and oils	ICP-OES	Pneumatic nebulizer, power 1600W, plasma flow 12 (L/min), aux. flow 1.0 (L/min)	120 s	N.D.	N.D.	N.D.	0.17–17 ppb	5–25 ppb	90–110%	(Karasakal 2020)

MDL, method detection limit; RSD, relative standard deviation; LOD, limit of detection; ICP-OES MS/MS, inductively coupled plasma optical emission spectroscopy multimode sample introduction system; ICP-OES MPP, inductively coupled plasma optical emission spectroscopy Mass Profiler Professional; EIEB ICP-OES, extraction induced by emulsion breaking inductively coupled plasma optical emission spectroscopy; ICP-OES, inductively coupled plasma optical emission spectroscopy; RPM, revolutions per minute; Rf, radiofrequency; N.D., not determined

samples that lies within the expected CCV value  $\pm 10\%$  (Table 1) (Nelson et al. n.d.). The data analysis of all the 69 whiskey samples was analyzed using the software Mass Profiler Professional (MPP) and the study indicates that the elemental profiles in whiskey were mainly utilized to differentiate the samples based on age and type as well as the origin of the sample (Nelson et al. n.d.). By using MPP software, principal component analysis (PCA) of all different elements was found with ( $P$ -value  $< 0.05$ ) which were able to confirm whether the different types of whiskey are separated based on their elemental profiles. This method was an accurate, sensitive, and specific as well as viable tool for elemental profiling of elements in whiskey through the ICP-OES technique with MPP software.

### In Determination of Trace Elements Concentration in Edible Oils

The concentration of trace elements in edible oils corresponds to the quality of edible oils. An edible oil contains various trace elements that are responsible for oxidative degradation as well as toxic effects that can occur in humans beings depending upon their concentration in the oil. Trace elements like Mg, Mn, and Co responsible for oil rancidity while ill-effects in humans are caused by elements such as Cd, Hg, and Pb (Bakircioglu et al. 2013). Depending on the trace elements concentration estimation in oil samples, different techniques like GF AAS, and F AAS can be utilized but for the analysis of edible oil, ICP-OES is an attractive as well as a popular technique because it can handle the wide concentration ranges found in oils with high organic content. The ICP-OES techniques are more sensitive as compared to other techniques in terms of results, speed, and cost of operation. By utilizing ultrasensitive and advanced properties of the ICP-OES technique, Dilek Bakircioglu et al. have carried out successful estimation of trace element concentration by applying ultrasonic technique of extraction and wet digestion as well as induced emulsion breaking extraction procedures in oil samples. By this technique, they found trace elements concentration in 5 edible oil samples by using the EIEB extraction procedure and they were between the ranges 0.022–0.058, 0.126–7.106, 0.570–4.504, 8.004–12.588, 0.035–0.054, 0.908–2.182, 0.099–0.134, and 2.206–8.982 mg/kg for trace elements like cadmium, chromium, copper, manganese, nickel, iron, lead, and zinc and elements like iron and manganese have highest and lowest metal concentrations in all samples respectively (Table 2). Also, analyte recoveries in oil samples for all metals under study situated between 96 and 109% (Table 1) (Bakircioglu et al. 2013). This validated and developed technique applied successfully in trace element concentration determination in edible oil sample (Bakircioglu et al. 2013).

**Table 2.** Estimated concentrations of trace elements in edible oils

Sr. no.	Elements	Metal concentrations after EIEB		Ref.
		Min. (mg/kg)	Max. (mg/kg)	
1.	Cd	0.022	0.058	(Bakircioglu et al. 2013)
2.	Cr	0.126	7.106	
3.	Cu	0.570	4.504	
4.	Fe	8.004	12.588	
5.	Mn	0.035	0.054	
6.	Ni	0.908	2.182	
7.	Pb	0.099	0.134	
8.	Zn	2.206	8.982	

### In Quantitative Determination of Metal Intake from Yoghurts in the Diet

Yogurt is one of the most popular nutritional-based food products and it is accepted worldwide by consumers. It is a coagulated milk product produced by lactic acid bacteria (LAB), primarily *Lactobacillus bulgaricus* and *Streptococcus thermophilus*, who ferment lactic acid (Luis et al. 2015). These milk derivatives contain numerous dietary supplements like proteins, vitamins mainly of B<sub>12</sub> and B<sub>2</sub>, and minerals likely Ca, Mg, and Zn which produce a beneficial effect on human health predominantly to balance the intestinal flora and diminish the blood cholesterol level as well as a preventive effect against colon malignancy (Luis et al. 2015). Depending on the determination of trace elements and mineral contained in yogurts, different techniques such as F AAS, GF AAS, ICP-MS, INNA, and XRF are available but among them ICP-OES is an attractive technique mainly in multiple elemental yogurt samples analysis because of low detection limits, reduced analysis time, and accurate and precise determinations over the broad range of concentrations. By utilizing the ultrasensitive and advanced properties of the ICP-OES technique, G. Luis et al. have carried out successful estimation and quantification of trace elements in yogurt samples (Luis et al. 2015). In this technique, they found the 20 trace elements contents in which 4 macro elements such as Na, K, Ca, and Mg as well as 16 trace elements like Al, B, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Li, Pb, Zn, Ni, Sr, and V were analyzed in the sample of yogurt. Firstly, for each of the 20 metals, standard deviations as well as mean concentration were examined in yogurt (plain vs. flavored) and the value was found to be ( $p < 0.05$ ). Additionally, in yogurt (plain and flavored), trace element Zn was the most abundant with the value 3.10 and 2.47 mg/kg in plain and flavored yogurt after the other microelements respectively and the recovery result was found to be greater than 96.5 % (Table 1). Thenafter, 20 elements (macro and trace) contents were quantified through the ICP-OES technique in 72 yogurt samples, and the mean

**Table 3.** Estimated amounts of daily consumption of essential and trace elements

Sr. no.	Category	Daily consumption of essential and trace metal		Ref.
		Ca	Zn	
1.	Men	6.43%	1.7%	(Luis et al. 2015)
2.	Women	4.8%	1.2%	
3.	Children	6%	2%	

weight concentrations (mg/kg) of all elements were 455, 1101, 1018, 115.1, 0.59, 0.07, 0.40, N.D., 0.002, 0.02, 0.27, 0.33, 0.52, 0.02, 0.04, 0.01, 0.002, 0.02, N.D., and 2.79 for Na, K, Ca, Mg, Al, B, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Li, Pb, Zn, Ni, Sr, and V respectively. In addition, yogurt daily consumption has been identified and mentioned in Table 3 which states that the detected metal levels revealed no toxicological risk to the consumer (Luis et al. 2015). This method would be of quite significance in further analytical research for the analysis of trace elements by ICP-OES techniques.

### In Elemental Analysis of Nuts and Seeds

Nuts and seeds are complex nutrient-dense foods free from cholesterol as well as a rich source of important nutrients such as unsaturated fatty acids and proteins. Other than proteins and fats, major nutrients like vitamin (B<sub>6</sub> and E), folic acid, niacin, and minerals are present which are likely to produce beneficially impact health outcomes (Naozuka et al. 2011). The analysis of elements in nuts and seeds is of paramount importance for the purpose of safety and purity. Depending on the quantitative elemental analysis in nuts and seeds, different atomic spectroscopy methods such as ICP-MS and GF AAS can be used due to the multi-elemental analytical technique. Based on the nuts and seeds importance as a source of mineral as well as complex compositions of mineral, Juliana Naozuka et al. developed accurate, specific, and rapid methods for the simultaneous estimation of metals, metalloids, and non-metals in sapuca nut, babassu nut, cupuassu (seed and pulp), coconut, and cashew nut by the ICP-OES technique (Naozuka et al. 2011). The results of analytical parameters such as the average RSD for calibration solution repeatability measurements ranged from 1.1 to 6.7%, and quantification limits (LOQ) were (0.00072–0.0532 mg/L) for micro and (0.0522 mg/L) for macro elements (Table 1) (Naozuka et al. 2011). Furthermore, all analytes were compared between experimental and certified values using peach leaves as a certified reference material [66] and results obtained by comparison which demonstrates technique was selective, accurate, and precise with 95% limit of confidence as Student's *t*-test. The mean



RSD for repeatability measurement ( $n=5$ ) differ from 1.4% for (P) to 8.5% for (K) and 19% for Zn, the worst result was observed (Naozuka et al. 2011). In addition, range of macro and micro nutrient in various types of nuts and seeds were identified in terms of dietary reference intake (DRI) and found that the nutritional value was not exceeded in comparison with the regulations of the Food and Drug Administration (FDA) except for Mn in the babassu nut (Naozuka et al. 2011).

### In Elemental Analysis of Seeds and Extracts of *Aesculus hippocastanum*

Nowadays, formulations from plant origin have been widely developed and marked by many industries for medicinal as well as nutraceutical purpose (Sirtori 2001; Jarzebski et al. 2019). *Aesculus hippocastanum* (horse chestnuts) is among such medicinal plants that have revealed diverse pharmacological as well as nutraceutical properties. Its seeds and extracts are commercialized as food supplements and also available as herbal medicines. Its seeds contain starch, several proteins, fatty acids, purines, saponins, flavonoids, etc. (Bombardelli et al. 1996). Laura Maletti et al. have applied the ICP-OES technique for determination of some metal contents in seeds and extracts of *Aesculus hippocastanum*. The samples were monitored for a time period of 4 years by selecting the seeds from a pure species (AHP) and another hybrid species (AHH). The horse chestnut flour from seeds of both varieties was evaluated for the contents of 14 elements (Al, Ca, Cu, Fe, K, Mg, Mn, Na, Ni, P, Se, Si, Sr, Zn) for a time period of 2016–2019. The concentrations were determined in mg per 100 g of the flour for both the varieties. Similarly the crude extracts (50% alcoholic tincture) were also evaluated for the same elemental contents for 4 years. It was worth notable that iron content was more throughout the 4 years in AHP (79.0–80.0 mg/kg) variety as compared to AHH flour samples (1.42–1.82 mg/kg) (Table 1). Along with this, levels of K, Ca, Mn, Ni, and Cu were also higher in AHP variety (Durante et al. 2021). Principle component analysis was used for statistical distribution of data on the basis of variables.

### In Multi-elemental Analysis of Vegetable Oils and Fats

Presence of certain metals or metalloids in edible oils even at minute concentrations may lead to problems and health issues. Therefore, detection of such elements with a great accuracy in food stuffs is of utmost importance. ICP-OES is the technique of choice for this purpose. José-Luis Todolí et al. applied the ICP-OES technique for determination of 15 elements in vegetable oils and fats. They have reported a newer dilution and shot analysis method for the very first time. Along with this, they have used a high-temperature

torch integrated sample induction system (hTISIS). The approach was used to quantify Al, Ba, Ca, Cd, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Si, Ti, and V in solid fats and liquid oils with high accuracy and precision. The optimized dilution factor was set as 1: 20 using xylene as solvent. Accuracy was performed using hTISIS where external calibration using xylene based standards was used. This reduced the matrix effects which were usually seen in case of traditional cyclonic spray chamber. The limits of detections were also improved up to 4 to 10 folds. Iron content was comparatively higher in case of cooking oil as well as vegetable oils. This element may come from stainless steel components. Another abundant element was silicon with concentration ranging between 2.6 and around 100 mg kg<sup>-1</sup> (Table 1). Barium was also found in frying oil as well as used cooking oil (Martinez et al. 2020).

### In Quantitative Trace and Major Elements Determination in Vegan Milks and Oils

In respect to human nutrition, trace and major elements are needed in the diet for biological function. Apart from the toxicity of some trace and major elements, it possesses a potential therapeutic action if plants contain these elements. In vegan milks and oils, the source of trace elements is soil and from which it absorbed and finally plant growth and development take place as well as these trace elements would be harmful depending upon their concentration and exposure. A well-known substituted cow milk is vegan milk which is free from lactose which notably a plant based milk and possess significant advantages over cow milk due to lactose tolerance. It possesses crucial advantages over other milk due to the presence of significant amount of minerals, vitamins, and mono- and polyunsaturated fats as well as free from cholesterol. Ayca Karasakal et al. have successfully carried out trace and major elements determination in vegan milks and oils through the ICP-OES technique after microwave digestion. Within the optimized conditions of this technique, the concentrations of Na, Mg, K, Ca, P, Fe, Cu, B, Mn, Zn, Al, S, As, Bi, Cd, Co, Cr, Mo, Ni, Pb, Pt, Sb, Se, Sn, Ti, W, and Hg in 81 vegan milk samples (soybean milk, coconut milk, and almond milk) and 125 vegan oil samples (soybean oil, coconut oil, bitter almond oil, sweet almond oil, and walnut oil) in Turkey was determined. The concentration of trace and major elements in vegan milk ranged between 307.4–501.2 for Na, 1.8–15.6 for Mg, 478.8–1300.4 for K, 276.3–1189 for Ca, 197–797.8 for P, 18.7–241.4 for S, 0.09–0.42 for Mn, 0–0.58 for Zn, 0.02–1.06 for Cu, 0.34–1.56 for B, 0.26–0.67 for Sb, and 3.4–30.4 for Sn respectively. The concentration of trace and major elements in vegan oils ranged between 6.8–31.2 for Na, 403.5–425.2 for K, 142.8–160.4 for Ca,

71.65–149.8 for P, 0.35–0.85 for Mg, 14.2–35.2 for S, 0.02–0.27 for Mn, 0.07–0.36 for Zn, 1.90–4.64 for Se, 0.92–5.36 for Fe, 0.01–0.05 for Cu, 1.02–1.66 for Sb, and 21.2, 35.0 for Sn respectively. Furthermore, the methods were validated in terms of linearity, limit of detection and limit of quantification, precision, and accuracy. The limits of detection were found in the range of 0.17 to 10.60 ppb respectively and the values of limit of quantification were found in the range of 5–25 ppb respectively. The accuracy of the method was found in the range of 90–110% which represents the accuracy of the method compared to certified reference material [72] (Table 1). This validated and developed technique applied successfully in trace and major element concentration determination in vegan milks and oils.

### In Agriculture Analysis

The ICP-OES method has also been used to examine a wide range of agricultural materials. In agriculture analysis, the ICP-OES technique analyzes different samples including fertilizers, soils, and soil toxicity of metal contamination. Some of the important applications in agriculture analysis have been discussed in the following sections.

#### By Using Advanced Valve System, Ultra-high-speed Analysis of Soil Extracts

Soils are porous media that are highly complex and structurally heterogeneous across a wide range of spatiotemporal scales and micro-organisms mediate or modulate a wide variety of soil reactions (Cauduro 2016). It contains numerous amounts of macronutrients, micronutrients, and heavy metals in their composition which produces numerous effects on the health of soil texture and alters the growth of the crops. The ICP-OES technique is easy, faster, and cost-effective and provides simpler quantitative analysis mainly of micronutrients and heavy elements in soil sample. By utilizing the ICP-OES technique, John Cauduro et al. developed an ultra-high-speed new analytical technique for determination of micronutrients and heavy metals in soil samples which is based upon the ICP-OES technique with an advanced valve system. In this technique, DTPA soil extract solution was analyzed 120 times which shows excellent stability as well as precision obtained through this technique was excellent with value ( $< 3.4\%$ ) throughout the run (Table 4) (Cauduro 2016). This method was accurate, faster analysis time, and without any compromise in analytical performance and implies them a best analytical technique for quantitative analysis of heavy metals and micronutrients in the soil sample.

### Elemental Nutrient Determination in DTPA-Extracted Soil

Soils contain heterogeneous elements in their composition and the elemental content in soil impacts the development of plants and yields better crops (Kulikov 2016). The presence of elements in the soils determines plant-based products' safety consequently to assess the fertility of soil, analysis of micronutrients is required while analysis of heavy metals in soil to identify any potential toxicity which produces an undesirable effect on the health of an individual (Kulikov 2016). Different extraction methods and analytical techniques are used depending on the elements of interest in soils, for example, the ICP-OES technique provides low-cost analysis and a wide concentration range for soil testing. The ICP-OES technique with Synchronous Vertical Dual View (SVDV) is more sensitive as compared to Radial View (RV) ICP-OES by providing excellent reliability, flexibility, stability, simplicity as well as ease of operation and performance. By utilizing the ICP-OES technique with SVDV, Elizabeth Kulikov et al. have successfully performed out elemental nutrients determination in the extracted soil sample from DTPA by extracting soil as a solution that contains chelating agents like diethylenetriaminepentaacetic acid (DTPA) used mainly for the micronutrients extraction from soil. In this technique, they obtained excellent linearity as well as with excellent method detection limits over a broad concentration range. Method detection limits (MDL) of each element like Cu, Fe, Mn, Zn, Cd, Co, Ni, and Pb were observed to be less than 0.025 mg/kg as well as spike recoveries of all element were found to be within  $\pm 10\%$  of the expected values (Kulikov 2016). In addition, spike recoveries within  $\pm 10\%$  of target values were used to validate the method, which demonstrate the method of accuracy (Table 4). This method was robust, sensitive, good, linear, excellent method detection limits without any compromise in analytical performance by the ICP-OES technique with Synchronous Vertical Dual View (SVDV) system and implies them a best analytical technique for the quantitative determination of elemental nutrients in extracted soil sample (Kulikov 2016).

### Determination of Vital Nutrients in Plant Samples

Plants contain a variety of essential nutrients that are necessary for the development, growth, and maintenance of plant tissues. The investigation of plant materials is mainly for macronutrients, micronutrients, and contaminants determinations which is important for the sources of nutrients and minerals and also aid in the restitution of contaminated soils to sort out the mineral deficiency in plants that solemnly affects the human population. The main elements like B, Al, Ca, Ba, Cu, K, and Fe are vital nutrients for the maintenance of plants which is responsible for plant nutrition and development as well as for the metabolism of the plant

**Table 4.** ICP-OES in agriculture analysis

Sr. no.	Analyte	Sample	Technique	Conditions	Run time	MDL	R <sup>2</sup> value	RSD	LOD	LOQ	Recovery	Ref.
1.	Micronutrients (Cu, Fe, Mn, Zn, Co, Ni) Heavy metals (Cd, Pb)	Soil	ICP-OES	Pump speed 25 RPM, Rf power 1200W, plasma flow 12 (L/min), aux. flow 1.0 (L/min), nebulizer flow 0.70 (L/ min)	11.7 s	N.D.	0.999	<3.4%	N.D.	N.D.	N.D.	(Sirtori 2001)
2.	Elemental nutrients	Soil	ICP-OES	Pump speed 12 RPM, Rf power 1200W, plasma flow 12 (L/min), aux. flow 1.0 (L/min), nebulizer flow 0.70 (L/ min)	30 s	<0.025 mg/kg	0.999	N.D.	N.D.	N.D.	93–96%	(Jarzebski et al. 2019)
3.	Elemental nutrients	Alfalfa, corn, sugarcane sample, and apple leaves	ICP-OES	Pump speed 12 RPM, Rf power 1500W, plasma flow 12 (L/min), aux. flow 1.0 (L/min), nebulizer flow 0.60 (L/ min)	20 s	<0.01–5.85 mg/kg	0.999	N.D.	N.D.	N.D.	94.2–108.6%	(Bombardelli et al. 1996)
4.	Phosphorus	Soil	ISAM ICP-OES	Wavelength 197.248– 214.914 nm, Rf power 1400W, plasma flow 18 (L/min), aux. flow 0.5 (L/min), nebulizer flow 0.5 (L/ min)	15 s	N.D.	0.999	N.D.	0.092	0.279	90–110%	(Durante et al. 2021)

**Table 4.** (continued)

Sr. no.	Analyte	Sample	Technique	Conditions	Run time	MDL	$R^2$ value	RSD	LOD	LOQ	Recovery	Ref.
5.	Geographical origin for seven elements (Ba, Ca, Fe, Mg, Mn, Na, and Sr)	Red garlic	MSA ICP-OES	EURACHEM guidelines	N.D.	N.D.	0.993–0.998	<15%	0.02–0.90 ( $\mu\text{g/g}$ )	0.06–3.03 ( $\mu\text{g/g}$ )	86–95%	(Martinez et al. 2020)

MDL, method detection limit; RSD, relative standard deviation; LOD, limit of detection; LOQ, limit of quantitation; ICP-OES, inductively coupled plasma optical emission spectroscopy; ISAM ICP-OES, internal standard addition method inductively coupled plasma optical emission spectroscopy; MSA ICP-OES, multivariate statistical analysis inductively coupled plasma optical emission spectroscopy; RPM, revolutions per minute; N.D., not determined

(Barros et al. 2016). The ICP-OES techniques are more sensitive by providing faster analysis, per sample less argon consumption, high throughput analysis, excellent sensitivity with a broad dynamic range as well as ease of operation and performance. By utilizing the ICP-OES technique with the technology Dichroic Spectral Combiner (DSC), Juan A.V.A. Barros et al. have successfully carried out the determination of vital nutrients like B, Al, Ca, Ba, Cu, K, Fe, Mg, Mn, S, Sr, P, and Zn in alfalfa, corn, sugarcane, and an apple leaves plant samples by microwave acid digestion method. In this technique, for primary nutrients like P and K; secondary nutrients like Ca, Mg, and S; and micronutrients like B, Cu, Fe, Mn, and Zn; method detection limits (MDL) and background equivalent concentrations (BEC) were discovered with low MDL value as well as the recovery of analyte in apple leaves by using ICP-OES in SVDV mode of all elements were ranged from 94.2 to 108.6% which is within the limit of  $\pm 10\%$  targeted value (Table 4). Additionally, by this technique calibration curves with value greater than 0.999 were prepared for elements like Al, B, P, and S which is less than 10% calibration error (Barros et al. 2016). This method shows excellent performance and flexibility as well as accurate to find out vital nutrients in plant leave without any compromise in analytical performance by the ICP-OES technique with Synchronous Vertical Dual View (SVDV) system.

#### Determination of Phosphorus in Soil

The presence of phosphorous is very important in the soil mainly for agriculture purposes as phosphorus is related to crop production. The presence of phosphorous in the soil is mainly due to the phosphorous cycle in the ecosystem as well as due to the ecology and geology of the soil (Yang et al. 2018). By utilizing the ICP-OES technique with an improved standard addition method (ISAM), Jing Yang et al. have successfully carried out phosphorus determination in the soil which is based upon conventional standard addition method (CSAM) and calibration curve method (CCM). In this technique, firstly the standard soil whose phosphorous determination was performed by ISAM method and the obtained values by ISAM method were matched with the certified values and ISAM method results compared with CSAM and CCM results. Additionally, correlation coefficient, quantification limit, and limit of detection of all these three methods were calculated. The results obtained through the method of ISAM of the above parameters were 0.9996, 0.092, and 0.279, respectively, by CCM method the results obtained were 0.9999, 0.059, and 0.180 respectively and by CSAM the results obtained were 0.9998, 0.032, and 0.097 respectively (Table 4) (Yang et al. 2018). Thenafter, method recovery was calculated by soil and blank samples rate of recovery which is within the range of 90–110%. The errors between the results of CCM and ISAM were less than 10%, with *t*-test value ( $P > 0.05$ ) which demonstrates no significant



difference between the two methods (Yang et al. 2018). This method shows excellent performance and reliability to some extent and matrix effect overcame in this propose method. It was an acceptable method for rapid and accurate batch analysis, particularly for phosphorus content in soil samples with an obvious matrix effect.

### Red Garlic Produced in Italy Classified According to Its Geographical Origin

Since ancient times, *Allium sativum* L. (garlic) has been used as a food condiment as well as a curative and preventive agent in traditional medicine. It contains complex and unique volatile organosulfur compounds like alliin which is an active precursor for raphem constituents as well as it contains thiosulfonates and thiosulfonates responsible mainly for characteristic taste and flavor (D'Archivio et al. 2019). The analysis of element in garlic on the basis of geographical discrimination is useful for tracing plant-based foods which ensure the soil from where these are grown. By utilizing the ICP-OES technique and multivariate statistical analysis, Angelo Antonio D'Archivio et al. have successfully carried out the trace elements concentration determination in garlic. The observed linearity was excellent and the  $R^2$  values found between 0.993 and 0.998; sensitivity was quantified in terms of LOD and LOQ limits (Table 4) (D'Archivio et al. 2019). The method precision of ICP-OES and accuracy was carried out by taking test concentration ( $n=3$ ) of garlic samples fortified before microwave digestion. The values of precision were less than 10% for Ba, Cu, Fe, Na, and Sr and higher values (11–14%) for Mn, Zn, Ca, and Mg have been determined. Therefore, %RSD was still below 15 % of the analyzed components which is within the acceptable limits for ICP-OES analysis and for most of the garlic samples, the recovery rate of each garlic sample was determined and acceptable mean recoveries (86–95%) were observed (D'Archivio et al. 2019). This method shows excellent performance and flexibility as well as a suitable technique for concentration determination of major mineral which is used for garlic geographical classification by utilizing multivariate statistical analysis through the ICP-OES technique.

### In Geological Analysis

A wide range of geological materials have been studied using the ICP-OES method. The ICP-OES technique application was mainly involved in the geological analysis of major, minor, trace element, determination of composition in different type of soils, sediments, rocks and its related materials which mainly were involved in determining the origin of rock formations. Some of the important applications in geological analysis have been discussed in the following sections.

### Determination of Pb in Geological Samples

The low concentration of lead in various matrices, such as food, industrial, environmental, clinical, or biological samples, is directly linked to its high toxicity, which has an impact on human health and safety. The determination of lead concentrations in various matrices needs pre-concentration methods before measurements. In calcium-rich materials like dolomites, Pb estimation was performed by atomic spectrometric techniques such as ICP-OES, AAS, and AFS and can be utilized mainly for the plumbane generation which is a very effective technique for separating Pb from complex matrices but among them, hydride generation (HG) coupled with ICP-OES is an interesting technique for quantitative determination of lead in trace amounts in the geological samples (Welna et al. 2015). The HG ICP-OES techniques are more sensitive as compared to other techniques by providing high selectivity, high sensitivity, and reduced sample preparation time. By utilizing the high sensitivity and high selectivity of this technique, Maja Welna et al. have successfully carried out determination of Pb in geological sample using HG ICP-OES with dilute acids after ultrasonic extraction techniques. In this technique, LODs under the optimized conditions were found in the range of  $2.3\text{--}5.6\text{ }\mu\text{g L}^{-1}$  and the precision (%RSD) observed between 2.2 and 3.9 which is below 5% were achieved, linearity ( $R^2$ ) relationship observed was 0.999 in the concentration up to  $2.5\text{ }\mu\text{g mL}^{-1}$  (Welna et al. 2015) (Table 5). Additionally, certified reference materials (CRMs) analysis as well as by recovery method, accuracy method of Pb was determined which is found between 94–108% and 92–114% for CRMs analysis and standard addition method respectively (Welna et al. 2015). This developed and optimized technique was applied successfully mainly for Pb determination in dolomites utilized by various fertilizer factories and an excellent technique with high sensitivity and selectivity in a very short period of time.

### Rare Earth Element Determination in Geological Samples

A better understanding of mineral reserves is required for chemical analysis of geological materials as well as geological and geochemical studies. The valuable information about the origin and geochemical formation is provided by rare earth elements (REEs) determination in geological samples. Mineral ores are rich source of REEs and their determination is critically needed in geological samples for its military applications. The ICP-OES technique is a good alternative technique for REEs determination with excellent sensitivity as compared to other techniques by providing high selectivity, high sensitivity, linearity, and simplicity with less sample preparation time (Amaral et al. 2016). By utilizing the high sensitivity and high selectivity

of this technique, Clarice D. B. Amaral et al. successfully carried out rare earth elements (REEs) determination in mineral samples of geological origin. In this technique, the method detection limits (MDLs) under the optimized conditions were less than 0.08 mg/kg, excellent linearity was excellent with value 0.9990, and recovery studies of samples were ranging from 90.1 to 107% with satisfactory results (Table 5) (Amaral et al. 2016). This method was considered as an excellent method with high accuracy, precision, sensitivity, and provide high throughput of sample in a very short time.

### Extraction of Rare Earth Elements and Their Identification

Solvent extraction is used to extract rare earth elements, which is a simple alternative method that has been widely used over the years due to its ease of use. To extract the lanthanides from various matrices, various extraction techniques involving many organic reagents such as organophosphorous reagents are mainly used (Pradhan and Ambade 2020). The ICP-OES technique is more sensitive as compared to other techniques by providing high selectivity, high sensitivity and reduced sample preparation time. By utilizing the high sensitivity and high selectivity of this technique, Susanta Kumar Pradhan et al. have successfully carried out lanthanides determination in geomaterials like soil and rock. The method validation was performed by rock and soil samples certified reference materials (CRMs). The precision (%RSD) varied over the 1 to 10% range but less within 10% which is acceptable and depends upon lanthanides concentration, linearity ( $R^2$ ) relationship observed near to one i.e. (0.999) for each elemental component (Table 5). The accuracy of the method developed for all lanthanides was checked by analysis of recovery test and the recovery studies were analyzed and the recovery of all lanthanides was found to be in the range of 90–100% (Pradhan and Ambade 2020). This developed and optimized method was applied successfully in the individual lanthanides determination through the ICP-OES technique.

### In Drug/Pharmaceutical Analysis

The hyphenated technique ICP-OES has proven to be a reliable method for elemental analysis, beginning with individual ingredient testing and continuing through production to final quality control. The ICP-OES method was applied successfully in the raw materials components analysis to finished product testing to ensure the safety of products, and researchers are developing ICP-OES techniques for elemental analysis in various pharmaceutical sample matrices. A bit of the noteworthy applications in drug/pharmaceutical analysis has been discussed in the following section.

**Table 5.** ICP-OES in geological analysis

Sr. no.	Analyte	Sample	Technique	Conditions	Run time	$R^2$ value	RSD	LOD	LOQ	Recovery	Ref.
1.	Pb	Geological	HG ICP-OES	Detection generator 40.68 MHz, Rf power 1000 W, Plasma flow 13 (L/min)	0.10 s	0.999	2.2–3.9%	2.3–5.6 ( $\mu\text{g/L}$ )	N.D.	94–108%	(Karasakal 2020)
2.	Rare earth elements	Geological	ICP-OES	Fast pump 80RPM, Rf power 1500W, Plasma flow 12 (L/min)	20 s	0.990	N.D.	N.D.	N.D.	90.1–107%	(Cauduro 2016)
3.	Lanthanum	Geological	ICP-OES	Detection generator 40.68 MHz, Rf power <5 W, plasma flow 11 (L/min)	N.D.	0.999	1–10%	N.D.	N.D.	90–100%	(Kulikov 2016)

*RSD*, relative standard deviation; *LOD*, limit of detection; *LOQ*, limit of quantitation; *HG ICP-OES*, hydride generation inductively coupled plasma optical emission spectroscopy; *ICP-OES*, inductively coupled plasma optical emission spectroscopy; *RPM*, revolutions per minute; *Rf*, radiofrequency; *N.D.*, not determined

### In Vitro Phosphate Binding Study of Eliphos Tablets in Estimation of Free Calcium and Phosphorus

The active pharmaceutical ingredient present in the Eliphos tablets is calcium acetate which is used for hemodialysis and peritoneal dialysis. ICP-OES is a striking technique for the inorganic metal impurities due to well detection limits, faster analysis time, low quantification limits, high sensitivity, and selectivity with accurate and precise determinations over wide ranges. Venkata Vivekanand Vallapragada et al. have successfully carried out in vitro phosphate binding study in Eliphos tablet samples for estimation of free calcium and phosphorous (Vallapragada et al. 2011). Within the optimized conditions of this technique, the development of the method was carried out in the presence of placebo and other matrices to quantitate calcium and phosphorous. The method precision of this technique by analyzing calcium acetate and solutions of sodium phosphate in five replicates ( $n=5$ ) and the %RSD of both solutions was 2.10% and 0.26% which is to be within the specification limit. The linearity of the method was correlated with the correlation coefficient, and  $R^2$  was found to be 0.9999 which indicates good linearity of calcium and phosphorous (Table 6). The recovery of calcium in placebo presence ranges from 98.3 to 101.1% (Vallapragada et al. 2011).

### Quantitative Content Determination of Boron in Tamsulosin Hydrochloride

It is an alpha-blocker drug used primarily to treat urinary problems due to enlargement of the prostate and their intake relaxes the muscles of bladder to enable free urine flow. The steps involved in the synthesis of tamsulosin hydrochloride include sulfate of sodium borohydride as reducing agent, and ultimately trace amounts of boron can have a significant impact on the health of an individual as residual impurities (Rajput et al. 2010). Mithlesh Rajput et al. have carried out successfully quantitative content determination of boron in tamsulosin hydrochloride samples by utilizing the ICP-OES technique. Under the optimized conditions, boron was extracted adequately and brought into the solution form, from tamsulosin hydrochloride in the presence of calcium hydroxide to prevent boron volatilization. The linearity of the method with correlation coefficient of boron standard solutions was found to be 0.99978 which indicates good linearity. The precision (%RSD) of the method of intra-day ranged from 2.5 to 4.9%, for inter-day ranging from 2.4 to 5.0% which is within the acceptable limits (Table 6) (Rajput et al. 2010). The accuracy of boron from the spiked tamsulosin hydrochloride samples at three distinct spiking levels was found in the acceptable range of 90–98% which is also within the acceptable limits. The validated methods

LOD and LOQ were 15 µg/L and 25 µg/L respectively. This developed method is quite useful and routinely utilized in the quantitative determination of boron with high sensitivity and selectivity with the procedure of fast sample preparation to ensure the quality of tamsulosin hydrochloride (Rajput et al. 2010).

### Estimation of Copper, Magnesium, and Zinc in Escitalopram Oxalate Drug

During the manufacturing process, escitalopram oxalate drug contained curtailed amount of inorganic impurities which leads to unwanted effects on the health of an individual. Veeramachaneni et al. have successfully utilized the ICP-OES method to quantitate the trace amount of copper, magnesium, and zinc in the sample of escitalopram oxalate. The linearity obtained by standard solutions and the  $R^2$  value mainly was found to be 1.000, 0.9999, and 0.9999 for copper, magnesium, and zinc respectively that shows excellent linearity (Veeramachaneni and Jayavarapu 2013). The accuracy of the method was achieved through spiked sample solutions of the respective elements and recovery ranged between 86.8 and 117.3% which is within the acceptable limits (Table 6). The precision of the method was checked and (%RSD) value of copper, magnesium, and zinc was found to be 3.1%, 1.3%, and 2.0% which is within the acceptable limits. Additionally, validated method (LOD) and (LOQ) were 33 µg/L and 10 µg/L respectively which is within the acceptable limits (Veeramachaneni and Jayavarapu 2013). This method is an alternative economic method for estimating the contents of elements in escitalopram oxalate.

### ICP-OES Method for Simultaneous Determination of Chromium, Molybdenum, and Selenium in Infant Formulas

Khan et al. have carried out successfully the simultaneous estimation of Cr, Mo, and Se in infant formula samples using the ICP-OES technique which is effective and sensitive. Under the optimized conditions in these techniques, different methods of digestion such as dry-ashing, wet-digestion, and microwave digestion methods evaluated mainly for sample preparation in certified reference material and non-fat milk powder. Firstly, by using a dry ashing furnace method certified reference method (CRM) using  $\text{HNO}_3$ , the recovery values of Cr and Se were 146.1% and 27.2%, respectively, after that the second dry ashing method was by using a hot plate followed by  $\text{HNO}_3$  and  $\text{HCl}$  and the values of recovery for Cr and Se were 138.4% and 209.0% respectively and finally the third dry ashing method was by

**Table 6.** ICP-OES in drug/pharmaceutical analysis

Sr. no.	Analyte	Sample	Technique	Conditions	R <sup>2</sup> value	%RSD	LOD	LOQ	Recovery	Ref.
1.	Ca, P	Eliphos tablets	ICP-OES	Rf power 1500W, plasma flow 15 (L/min), aux. flow 0.2 (L/min), nebulizer flow 0.8 (L/min)	0.9999	2.10% (calcium acetate), 0.26% (sodium phosphate)	N.D.	Lowest	98.3–101.1% (calcium acetate), N.D. (phosphate)	(Barros et al. 2016)
2.	B	Tamsulosin hydrochloride	ICP-OES	Rf power 1200W, plasma flow 15 (L/min), aux. flow 1.50 (L/min), nebulizer flow 0.56 (L/min)	0.9997	2.5–4.9% (intra-day), 2.4–5.0% (inter-day)	15 µg/L	25 µg/L	90–98%	(Yang et al. 2018)
3.	Cu, Mg and Zn	Escitalopram oxalate	ICP-OES	Pump rate 500 RPM, Rf power 1150W, plasma flow 15 (L/min), aux. flow 0.5 (L/min), nebulizer flow 0.6 (L/min)	1.0000 (Cu), 0.9999 (Mg), 0.9999 (Zn)	3.1% (Cu), 1.3% (Mg), 2.0% (Zn)	33 µg/L	10 µg/L	86.8–117.3%	(D'Archivio et al. 2019)
4.	Cr, Mo and Se	Infant formulas	ICP-OES and ICP-MS	Rf generator 27.12 MHz, Rf power 1300W, plasma flow 16 (L/min), aux. flow 1.5 (L/min), nebulizer flow 0.94 (L/min)	N.D.	1.18% (Cr), 1.02% (Mo), and 1.27% (Se)	4.443 (Cr), 0.909 (Mo), 5.890 (Se) µg/kg by ICP-OES, 0.063 (Cr), 0.062 (Mo), 0.133 (Se) µg/kg by ICP-MS	14.8 (Cr), 3.03 (Mo), 19.06 (Se) µg/kg by ICP-OES, 0.21 (Cr), 0.21 (Mo), 0.44 (Se) µg/kg by ICP-MS	100.7% and 1.04–4% (Cr), 94.5% and 103.6% (Mo), 98.6% and 94.9% (Se)	(Wadna et al. 2015)
5.	Macro and Trace Element	Multivitamin preparation	ICP-OES with slurry sample introduction	Rf generator 27.12 MHz, Rf power 1100W, plasma flow 0.6 (L/min), aux. flow 10 (L/min), nebulizer flow 0.65 (L/min)	N.D.	5–10%	1000–100000 µg/L for Ca, Mg, P, K, Fe, Mn, Zn, Cu and 5–50 µg/L for Cr, Ni, V	N.D.	N.D.	(Amaral et al. 2016)

RSD, relative standard deviation; LOD, limit of detection; LOQ, limit of quantitation; ICP-OES, inductively coupled plasma optical emission spectroscopy; ICP-MS, inductively coupled plasma mass spectrometry; RPM, revolutions per minute; Rf, radiofrequency; N.D., not determined



using  $\text{HNO}_3$ ,  $\text{HCl}$ , and  $\text{H}_2\text{SO}_4$  and the recovery values of Cr, Mo, and Se were 123.1%, 92.1%, and 173.2% respectively (Khan et al. 2013). In the wet digestion method, by using  $\text{HNO}_3$  and  $\text{H}_2\text{O}_2$ , the recovery values of Cr, Mo, and Se were 119.2%, 95.7%, and 101.6%, respectively, and also by microwave digestion method the recovery values of Cr, Mo, and Se were 111.5%, 102.2%, and 98.9% respectively (Table 6). The results revealed that the sample preparation techniques of wet digestion and microwave provided satisfactory results (Khan et al. 2013).

### In Multivitamins Preparations Quantitative Determination of Macro and Trace Element

The preparation of multivitamin represents as one of the largest categories of vitamin supplements, accounting for 48 % of the supplement market. Concerning multivitamins preparations, safety and toxicity is of paramount importance. So, analysis of multi-element is applicable and used easily for accurately verifying the content of elements mentioned on the labels of multivitamin to screen toxic elements contamination. To determine macro and trace elements, Krejčová et al. analyzed multivitamin preparation samples using the ICP-OES technique. For the determination of macro and trace elements such as Fe, Mn, Zn, Cu, Cr, Ni, Ca, Mg, P, K, and V in multivitamin preparations, an effective and sensitive method has been developed. First of all, slurry compositions were evaluated and a slurry concentration of 0.1–0.2% was found optimum for determination of macro and trace element in multivitamin preparations (Krejčová et al. 2006). The samples were mineralized for result comparison and 0.1–0.2% m/v in 6% v/v  $\text{HNO}_3$  as slurry concentration were used as analysis purpose. Simultaneously, calibration was tested using water standard solutions, slurry standards, and standard additions for the determination of the above elements in slurries. The %RSD for macro elements ranged from 5 to 10%, and the measured concentrations in the in-home control samples were in good agreement with independent laboratories suggesting that good precision is provided by the method as well as the concentration of the detected element is compared to the quantity declared for the analyzed multivitamin preparations by the producer (Table 6). This method was found to be appropriate for the monitoring of quality control in multivitamin preparations and could be useful as a routine procedure in the analysis of macro and trace elements in multivitamin preparations as well as in the ICP-OES slurry analysis (Krejčová et al. 2006). This method was found significant and appropriate for multivitamin preparations, and slurry analysis provides satisfactory results mainly for multivitamin preparations monitoring for the purpose of quality control (Krejčová et al. 2006).

## Conclusion

Determination of the quality and safety of any consumable product is the ultimate goal of every analytical process. It is unfortunate that even after prominent care various elemental impurities get incorporated into the finished products. ICP-OES is an advanced technique for detection as well as quantification of such incorporated trace amounts of elements in a broad range of samples. Its sophisticated, advanced, and ultrapowerful instrumentation has led it to be the technique of choice for the above-said purpose. The presented review summarized the basic principles, components, and modes of operation of ICP-OES along with its merits over the other techniques. The various presented reports have made it evident that this technique has been adopted by several research groups and industries to ensure the quality and purity of various products including foodstuffs, agricultural products, soil, pharmaceutical products, and geological samples as well. Although this approach offers several advanced features, still it has been less explored. This compilation may be helpful for budding analysts to understand its basics and strategies to employ it in their analytical work.

## Declarations

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**Informed Consent** Informed consent not applicable.

**Conflict of Interest** Declared None

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