SPECTROSCOPY
TUTORIAL

A Beginner's Guide to ICP-MS

Part II: The Sample-Introduction System

ROBERT THOMAS

Part II of Robert Thomas' series on inductively coupled plasma mass spectrometry looks at one of the most critical areas of the instrument — the sample introduction system. He discusses the fundamental principles of converting a liquid into a fine-droplet aerosol suitable for ionization in the plasma, and provides an overview of the different types of commercially available nebulizers and spray chambers.

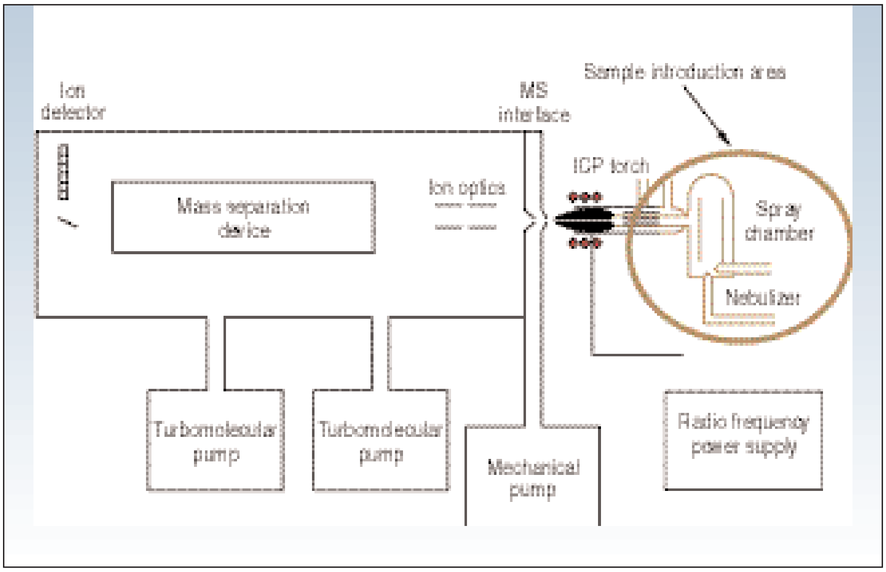

Figure 1. ICP-MS system diagram showing the location of the sample introduction area.

The majority of inductively coupled plasma mass spectrometry (ICP-MS) applications involve the analysis of liquid samples. Even though spectroscopists adapted the technique over the years to handle solids, it was developed in the early 1980s primarily to analyze solutions. There are many ways of introducing a liquid into an ICP mass spectrometer, but they all basically achieve the same result — they generate a fine aerosol of the sample so it can be efficiently ionized in the plasma discharge. The sample-introduction area has been called the Achilles heel of ICP-MS because it is considered the weakest component of the instrument, with only 1–2% of the sample finding its way into the plasma (1). Although there has recently been much improvement in this area, the fundamental design of an ICP-MS sample introduction system has not dramatically changed since the technique was first introduced in 1983.

Before discussing the mechanics of aerosol generation in greater detail, let us look at the basic components of a sample introduction system. Figure 1 shows the proximity of the sample introduction area relative to the rest of the ICP mass spectrometer, while Figure 2 represents the individual components.

The mechanism of introducing a liquid sample into analytical plasma can be considered as two separate events — aerosol generation using a nebulizer and droplet selection by way of a spray chamber. Sharp carried out a thorough investigation of both processes (2).

AEROSOL GENERATION

As mentioned previously, the main function of the sample introduction system is to generate a fine aerosol of the sample. It achieves this purpose with a nebulizer and a spray chamber. The sample is normally pumped at ~1 mL/min via a peristaltic pump into the nebulizer. A peristaltic pump is a small pump with lots of minirollers that rotate at the same speed. The constant motion and pressure of the rollers on the pump tubing feed the sample to the nebulizer. The benefit of a peristaltic pump is that it ensures a constant flow of liquid, irrespective of differences in viscosity between samples, standards, and blanks. After the sample enters the nebulizer, the liquid is broken up into a fine aerosol by the pneumatic action of gas flow (~1 L/min) smashing the liquid into tiny droplets, which is very similar to the spray mechanism of a can of deodorant. Although pumping the sample is the most common approach to introducing it, some pneumatic nebulizers, such as the concentric design, don't need a pump because they rely on the natural venturi effect of the positive pressure of the nebulizer gas to suck the sample through the tubing. Solution nebulization is conceptually represented in Figure 3, which shows aerosol generation using a nebulizer with a crossflow design.

DROPLET SELECTION

Because the plasma discharge is inefficient at dissociating large droplets, the spray chamber's function is primarily to allow only the small droplets to enter the plasma. Its secondary purpose is to smooth out pulses that occur during the nebulization process, due mainly to the peristaltic pump. Several ways exist to ensure only the small droplets get through, but the most common way is to use a double-pass spray chamber where the aerosol emerges from the nebulizer and is directed into a central tube running the whole length of the chamber. The droplets travel the length of this tube, where the large droplets (greater than ~10 µm in diameter) fall out by gravity and exit through the drain tube at the end of the spray chamber. The fine droplets (~5–10 µm in diameter) then pass between the outer wall and the central tube, where they eventually emerge from the spray chamber and are transported into the sample injector of the plasma torch (3). Although many different designs are available, the spray chamber's main function is to allow only the smallest droplets into the plasma for dissociation, atomization, and finally ionization of the sample's elemental components. Figure 4 presents a simplified schematic of this process.

Let us now look at the different nebulizer and spray chamber designs that are most commonly used in ICP-MS. This article cannot cover every type available because a huge market has developed over the past few years for application-specific customized sample introduction components. This market created an industry of small OEM (original equipment manufacturers) companies that manufacture parts for instrument companies as well as selling directly to ICP-MS users.

NEBULIZERS

By far the most common design used for ICP-MS is the pneumatic nebulizer, which uses mechanical forces of a gas flow (normally argon at a pressure of 20–30 psi) to generate the sample aerosol. The most popular designs of pneumatic nebulizers include concentric, microconcentric, microflow, and crossflow. They are usually made from glass, but other nebulizer materials, such as various kinds of polymers, are becoming more popular, particularly for highly corrosive samples and specialized applications. I want to emphasize at this point that nebulizers designed for use with ICP-optical emission spectroscopy (OES) are not recommended for ICP-MS. This fact results from a limitation in total dissolved solids (TDS) that can be put into the ICP-MS interface area. Because the orifice sizes of the sampler and skimmer cones used in ICP-MS are so small (~0.6–1.2 mm), the concentration of matrix components must generally be kept below 0.2% (4). Therefore, general-purpose ICP-OES nebulizers that are designed to aspirate 1–2% dissolved solids, or high-solids nebulizers such as the Babbington, V-groove, or cone-spray nebulizers, which are designed to handle as much as 20% dissolved solids, are not ideal for use with ICP-MS. The most common of the pneumatic nebulizers used in commercial ICP mass spectrometers are the concentric and crossflow designs. The concentric design is more suitable for clean samples, while the crossflow is generally more tolerant to samples containing higher levels of solids or particulate matter.

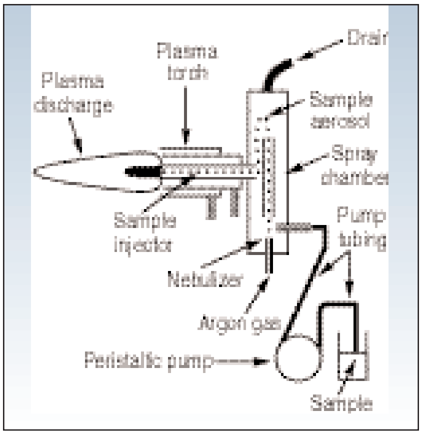

Figure 2. Diagram of the ICP-MS sample introduction area.

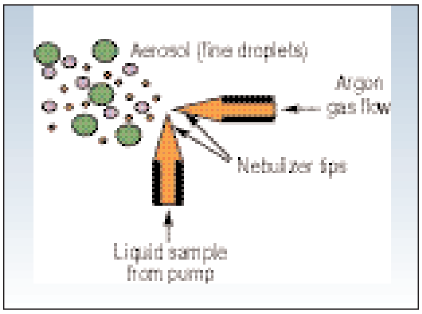

Figure 3. Conceptual representation of aerosol generation with an ICP-MS nebulizer.

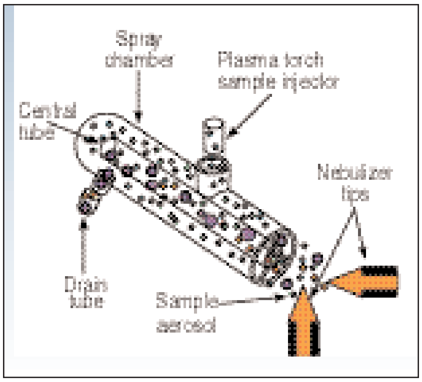

Figure 4. Simplified representation of the separation of large and fine droplets in the spray chamber.

Concentric design. In the concentric nebulizer, the solution is introduced through a capillary tube to a low-pressure region created by a gas flowing rapidly past the end of the capillary. The low pressure and high-speed gas combine to break up the solution into an aerosol, which forms at the open end of the nebulizer tip. Figure 5 illustrates the concentric design.

Concentric pneumatic nebulizers can provide excellent sensitivity and stability, particularly with clean solutions. However, the small orifices can be plagued by blockage problems, especially if large numbers of heavy matrix samples are aspirated.

Crossflow design. For samples that contain a heavier matrix or small amounts of undissolved matter, the crossflow design is probably the best option. With this design the argon gas is directed at right angles to the tip of a capillary tube, in contrast to the concentric design, where the gas flow is parallel to the capillary. The solution is either drawn up through the capillary tube via the pressure created by the high-speed gas flow or, as is most common with crossflow nebulizers, forced through the tube with a peristaltic pump. In either case, contact between the high-speed gas and the liquid stream causes the liquid to break up into an aerosol. Crossflow nebulizers are generally not as efficient as concentric nebulizers at creating the very small droplets needed for ICP-MS analyses. However, the larger diameter liquid capillary and longer distance between liquid and gas injectors reduce clogging problems. Many analysts feel that the small penalty paid in analytical sensitivity and precision when compared with concentric nebulizers is compensated by the fact that the crossflow design is far more rugged for routine use. Figure 6 shows a cross section of a crossflow nebulizer.

Microflow design. A new breed of nebulizers is being developed for ICP-MS called microflow nebulizers, which are designed to operate at much lower sample flows. While conventional nebulizers have a sample uptake rate of about 1 mL/min, microflow nebulizers typically run at less than 0.1 mL/min. They are based on the concentric principle, but

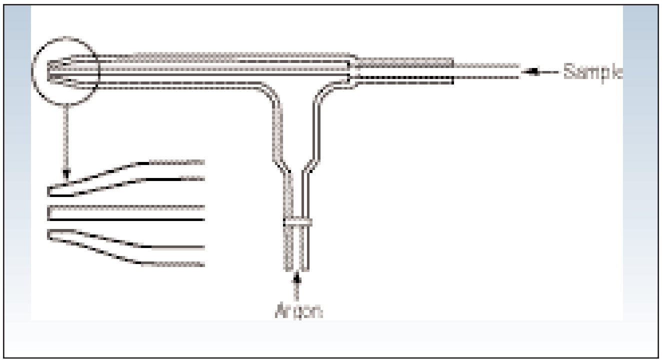

Figure 5. Diagram of a typical concentric nebulizer.

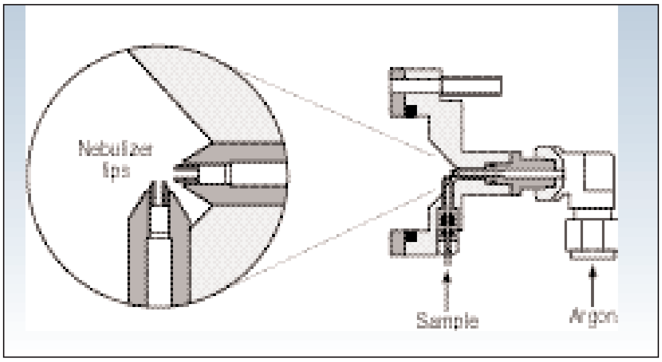

Figure 6. Schematic of a crossflow nebulizer.

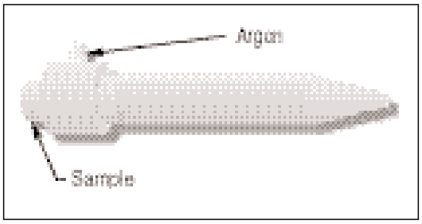

Figure 7. A typical concentric microflow nebulizer. Printed with permission from Elemental Scientific (Omaha, NE).

they usually operate at higher gas pressure to accommodate the lower sample flow rates. The extremely low uptake rate makes them ideal for applications with limited sample volume or where the sample or analyte is prone to sample introduction memory effects. These nebulizers and their components are typically constructed from polymer materials such as polytetrafluoroethylene (PTFE), perfluoroalkoxy (PFA), or polyvinylidene fluoride (PVDF). In fact, their excellent corrosion resistance means that they have naturally low blank levels. This characteristic, together with their ability to handle small sample volumes such as vapor-phase decomposition (VPD) applications, makes them an ideal choice for semiconductor labs that are carrying out ultratrace element analysis (5). A typical microflow nebulizer made from PFA is shown in Figure 7.

SPRAY CHAMBERS

Let us now turn our attention to spray chambers. Basically two designs are used in commercial ICP-MS instrumentation — double pass and cyclonic spray chambers. The double pass is by far the most common, with the cyclonic type gaining in popularity. Another type of spray chamber based on the impact bead design (first developed for flame AA and then adapted for ICP-OES) was tried on the early ICP-MS systems with limited success, but is not generally used today. As mentioned earlier, the function of the spray chamber is to reject the larger aerosol droplets and also to smooth out pulses produced by the peristaltic pump. In addition, some ICP-MS spray chambers are externally cooled (typically to 2–5 °C) for thermal stability of the sample and to minimize the amount of solvent going into the plasma. This can have a number of beneficial effects, depending on the application, but the main benefits are reduction of oxide species and the ability to aspirate volatile organic solvents.

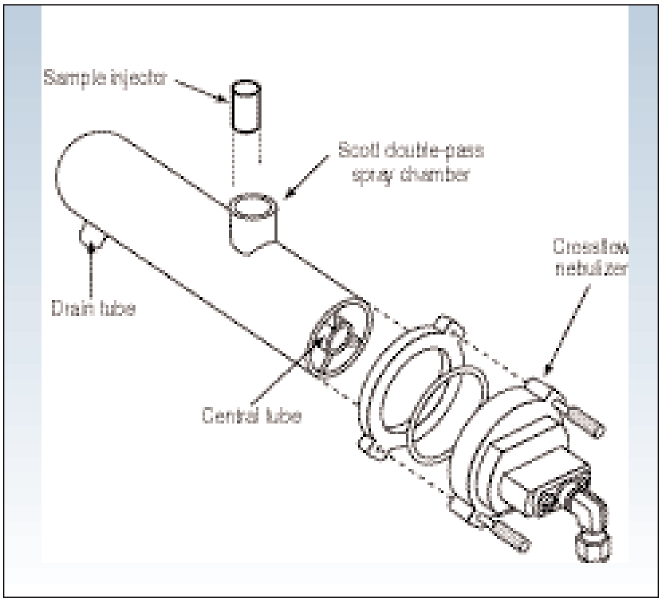

Figure 8. Schematic of a Scott double-pass spray chamber (shown with crossflow nebulizer). Printed with permission of PerkinElmer Instruments (Norwalk, CT).

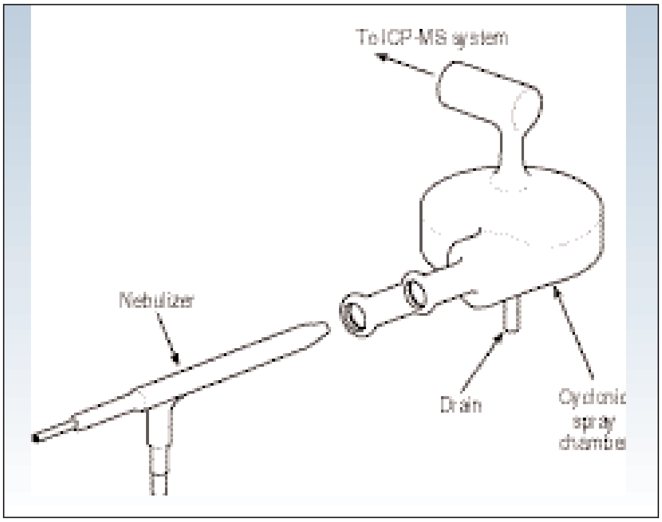

Figure 9. Schematic of a cyclonic spray chamber (shown with concentric nebulizer).

Double pass. By far the most common design of double-pass spray chamber is the Scott design, which selects the small droplets by directing the aerosol into a central tube. The larger droplets emerge from the tube and, by gravity, exit the spray chamber via a drain tube. The liquid in the drain tube is kept at positive pressure (usually by way of a loop), which forces the small droplets back between the outer wall and the central tube, where they emerge from the spray chamber into the sample injector of the plasma torch. Scott double-pass spray chambers come in a variety of shapes, sizes, and materials, but are generally considered the most rugged design for routine use. Figure 8 shows a Scott spray chamber made of a polysulfide-type material, coupled to a crossflow nebulizer.

Cyclonic spray chamber. The cyclonic spray chamber operates by centrifugal force. Droplets are discriminated according to their size by means of a vortex produced by the tangential flow of the sample aerosol and argon gas inside the chamber. Smaller droplets are carried with the gas stream into the ICP-MS, while the larger droplets impinge on the walls and fall out through the drain. It is generally accepted that a cyclonic spray chamber has a higher sampling efficiency, which, for clean samples, translates into higher sensitivity and lower detection limits. However, the droplet size distribution appears to be different from a double-pass design, and for certain types of samples, can give slightly inferior precision. An excellent evaluation of the capabilities of a cyclonic spray chamber was made by Beres and co-workers (6). Figure 9 shows a cyclonic spray chamber connected to a concentric nebulizer.

Many other nonstandard sample introduction devices are available that are not described in this particular tutorial, such as ultrasonic nebulization, membrane desolvation, flow injection, direct injection, electrothermal vaporization, and laser ablation. However, they are becoming more and more important, particularly as ICP-MS users are demanding higher performance and more flexibility. For that reason, they will be addressed in a separate tutorial at the end of this series.

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"News Spectrum" continued from page 13

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A Beginner's Guide to ICP-MS

Part III: The Plasma Source

ROBERT THOMAS

Part III of Robert Thomas' series on inductively coupled plasma–mass spectroscopy (ICP-MS) looks at the area where the ions are generated — the plasma discharge. He gives a brief historical perspective of some of the common analytical plasmas used over the years and discusses the components that are used to create the ICP. He finishes by explaining the fundamental principles of formation of a plasma discharge and how it is used to convert the sample aerosol into a stream of positively charged ions.

Inductively coupled plasmas are by far the most common type of plasma sources used in today's commercial ICP–optical emission spectrometry (OES) and ICP-MS instrumentation. However, it wasn't always that way. In the early days, when researchers were attempting to find the ideal plasma source to use for spectrometric studies, it was unclear which approach would prove to be the most successful. In addition to ICPs, some of the other novel plasma sources developed were direct current plasmas (DCP) and microwave-induced plasmas (MIP). A DCP is formed when a gas (usually argon) is introduced into a high current flowing between two or three electrodes. Ionization of the gas produces an inverted Y-shaped plasma. Unfortunately, early DCP instrumentation was prone to interference effects and also had some usability and reliability problems. For these reasons, the technique never became widely accepted by the analytical community (1). However, its one major benefit was that it could aspirate high levels of dissolved or suspended solids, because there was no restrictive sample injector for the solid material to block. This feature alone made it attractive for some laboratories, and once the initial limitations of DCPs were better understood, the technique became more accepted. In fact, for those who want a DCP excitation source coupled with an optical emission instrument today, an Echelle-based grating using a solid-state detector is commercially available (2).

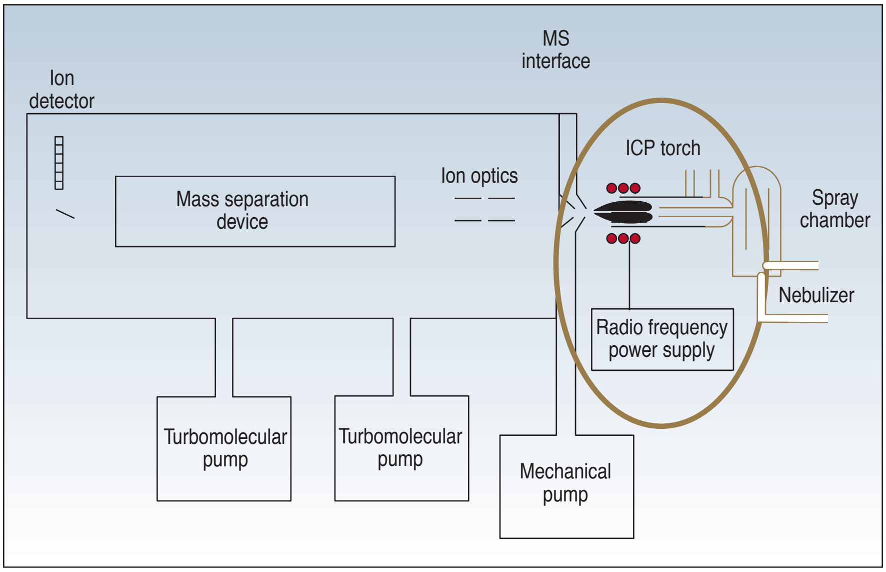

Figure 1. Schematic of an ICP-MS system showing the location of the plasma torch and radio frequency (RF) power supply.

Limitations in the DCP approach led to the development of electrodeless plasma, of which the MIP was the simplest form. In this system, microwave energy (typically 100–200 W) is supplied to the plasma gas from an excitation cavity around a glass or quartz tube. The plasma discharge in the form of a ring is generated inside the tube. Unfortunately, even though the discharge achieves a very high power density, the high excitation temperatures exist only along a central filament. The bulk of the MIP never gets hotter than 2000–3000 K, which means it is prone to very severe matrix effects. In addition, they are easily extinguished during aspiration of liquid samples. For these reasons, they have had limited success as an emission source, because they are not considered robust enough for the analysis of real-world, solution-based samples. However, they have gained acceptance as an ion source for mass spectrometry (3) and also as emission-based detectors for gas chromatography.

Because of the limitations of the DCP and MIP approaches, ICPs became the dominant focus of research for both optical emission and mass spectrometric studies. As early as 1964, Greenfield and co-workers reported that an atmospheric-pressure ICP coupled with OES could be used for elemental analysis (4). Although crude by today's standards, the system showed the enormous possibilities of the ICP as an excitation source and most definitely opened the door in the early 1980s to the even more exciting potential of using the ICP to generate ions (5).

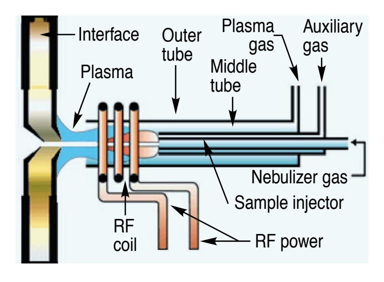

Figure 2. Detailed view of a plasma torch and RF coil relative to the ICP-MS interface.

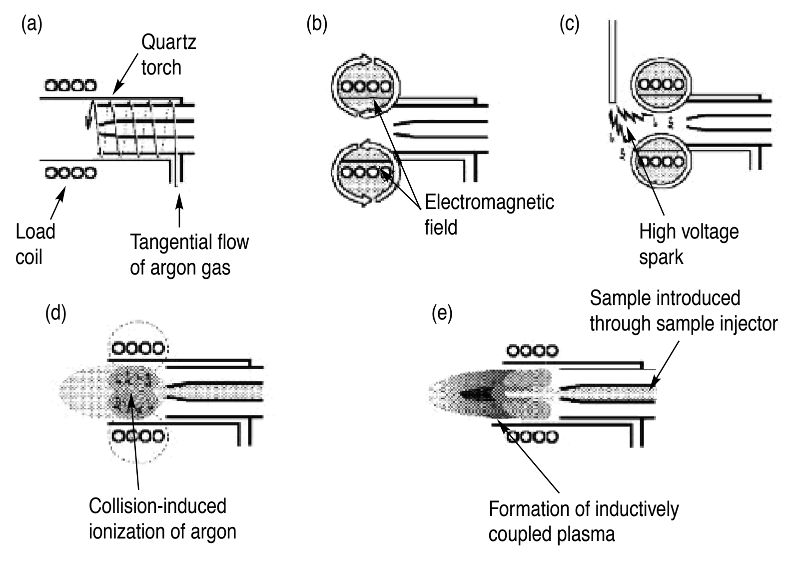

Figure 3. (right) Schematic of an ICP torch and load coil showing how the inductively coupled plasma is formed. (a) A tangential flow of argon gas is passed between the outer and middle tube of the quartz torch. (b) RF power is applied to the load coil, producing an intense electromagnetic field. (c) A high-voltage spark produces free electrons. (d) Free electrons are accelerated by the RF field, causing collisions and ionization of the argon gas. (e) The ICP is formed at the open end of the quartz torch. The sample is introduced into the plasma via the sample injector.

THE PLASMA TORCH

Before we take a look at the fundamental principles behind the creation of an inductively coupled plasma used in ICP-MS, let us take a look at the basic components that are used to generate the source: a plasma torch, a radio frequency (RF) coil, and RF power supply. Figure 1 shows their proximity to the rest of the instrument; Figure 2 is a more detailed view of the plasma torch and RF coil relative to the MS interface.

The plasma torch consists of three concentric tubes, which are usually made from quartz. In Figure 2, these are shown as the outer tube, middle tube, and sample injector. The torch can either be one-piece with all three tubes connected, or it can be a demountable design in which the tubes and the sample injector are separate. The gas (usually argon) used to form the plasma (plasma gas) is passed between the outer and middle tubes at a flow rate of ~12–17 L/min. A second gas flow, the auxiliary gas, passes between the middle tube and the sample injector at ~1 L/min and is used to change the position of the base of the plasma relative to the tube and the injector. A third gas flow, the nebulizer gas, also flowing at ~1 L/min carries the sample, in the form of a fine-droplet aerosol, from the sample introduction system (for details, see Part II of this series: *Spectroscopy* **16**[5], 56–60 [2001]) and physically punches a channel through the center of the plasma. The sample injector is often made from materials other than quartz, such as alumina, platinum, and sapphire, if highly corrosive materials need to be analyzed. It is worth mentioning that although argon is the most suitable gas to use for all three flows, there are analytical benefits in using other gas mixtures, especially in the nebulizer flow (6). The plasma torch

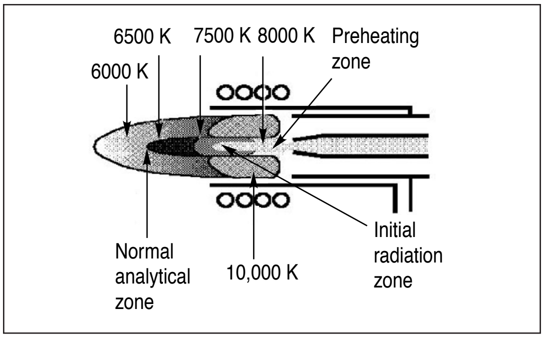

Figure 4. Different temperature zones in the plasma.

Droplet (Desolvation) Solid (Vaporization) Gas (Atomization) Atom (Ionization) Ion

$$M(H_2O)^+X^- \longrightarrow (MX)_n \longrightarrow MX \longrightarrow M \longrightarrow M^+$$

From sample injector ⟶ To mass spectrometer

Figure 5. Mechanism of conversion of a droplet to a positive ion in the ICP.

is mounted horizontally and positioned centrally in the RF coil, approximately 10–20 mm from the interface. It must be emphasized that the coil used in an ICP-MS plasma is slightly different from the one used in ICP-OES. In all plasmas, there is a potential difference of a few hundred volts produced by capacitive coupling between the RF coil and the plasma. In an ICP mass spectrometer, this would result in a secondary discharge between the plasma and the interface cone, which could negatively affect the performance of the instrument. To compensate for this, the coil must be grounded to keep the interface region as close to zero potential as possible. I will discuss the full implications of this in greater detail in Part IV of this series.

FORMATION OF AN ICP DISCHARGE

Let us now discuss the mechanism of formation of the plasma discharge. First, a tangential (spiral) flow of argon gas is directed between the outer and middle tube of a quartz torch. A load coil, usually copper, surrounds the top end of the torch and is connected to a radio frequency generator. When RF power (typically 750–1500 W, depending on the sample) is applied to the load coil, an alternating current oscillates within the coil at a rate corresponding to the frequency of the generator. In most ICP generators this frequency is either 27 or 40 MHz. This RF oscillation of the current in the coil causes an intense electromagnetic field to be created in the area at the top of the torch. With argon gas flowing through the torch, a high-voltage spark is applied to the gas, which causes some electrons to be stripped from their argon atoms. These electrons, which are caught up and accelerated in the magnetic field, then collide with other argon atoms, stripping off still more electrons. This collision-induced ionization of the argon continues in a chain reaction, breaking down the gas into argon atoms, argon ions, and electrons, forming what is known as an inductively coupled plasma discharge. The ICP discharge is then sustained within the torch and load coil as RF energy is continually transferred to it through the inductive coupling process. The sample aerosol is then introduced into the plasma through a third tube called the sample injector. This whole process is conceptionally shown in Figure 3.

THE FUNCTION OF THE RF GENERATOR

Although the principles of an RF power supply have not changed since the work of Greenfield (4), the components have become significantly smaller. Some of the early generators that used nitrogen or air required 5–10 kW of power to sustain the plasma discharge — and literally took up half the room. Most of today's generators use solid-state electronic components, which means that vacuum power amplifier tubes are no longer required. This makes modern instruments significantly smaller and, because vacuum tubes were notoriously unreliable and unstable, far more suitable for routine operation.

As mentioned previously, two frequencies have typically been used for ICP RF generators: 27 and 40 MHz. These frequencies have been set aside specifically for RF applications of this kind, so they will not interfere with other communication-based frequencies. The early RF generators used 27 MHz, while the more recent designs favor 40 MHz. There appears to be no significant analytical advantage of one type over the other. However, it is worth mentioning that the 40-MHz design typically runs at lower power levels, which produces lower signal intensity and reduced background levels. Because it uses slightly lower power, this might be considered advantageous when it comes to long-term use of the generator.

The more important consideration is the coupling efficiency of the RF generator to the coil. The majority of modern solid-state RF generators are on the order of 70–75% efficient, meaning that 70–75% of the delivered power actually makes it into the plasma. This wasn't always the case, and some of the older vacuum tube–designed generators were notoriously inefficient; some of them experienced more than a 50% power loss. Another important criterion to consider is the way the matching network compensates for changes in impedance (a material's resistance to the flow of an electric current) produced by the sample's matrix components or differences in solvent volatility. In older crystal-controlled generators, this was usually done with servo-driven capacitors. They worked very well with most sample types, but because they were mechanical devices, they struggled to compensate for very rapid impedance changes produced by some samples. As a result, the plasma was easily extinguished, particularly during aspiration of volatile organic solvents.

These problems were partially overcome by the use of free-running RF generators, in which the matching network was based on electronic tuning of small changes in frequency brought about by the sample solvent or matrix components. The major benefit of this approach was that compensation for impedance changes was virtually instantaneous because there were no moving parts. This allowed for the successful analysis of many sample types that would probably have extinguished the plasma of a crystal-controlled generator.

IONIZATION OF THE SAMPLE

To better understand what happens to the sample on its journey through the plasma source, it is important to understand the different heating zones within the discharge. Figure 4 shows a cross-sectional representation of the discharge along with the approximate temperatures for different regions of the plasma.

As mentioned previously, the sample aerosol enters the injector via the spray chamber. When it exits the sample injector, it is moving at such a velocity that it physically punches a hole through the center of the plasma discharge. It then goes through a number of physical changes, starting at the preheating zone and continuing through the radiation zone before it eventually becomes a positively charged ion in the analytical zone. To explain this in a very simplistic way, let's assume that the element exists as a trace metal salt in solution. The first step that takes place is desolvation of the droplet. With the water molecules stripped away, it then becomes a very small solid particle. As the sample moves further into the plasma, the solid particle changes first into a gaseous form and then into a ground-state atom. The final process of conversion of an atom to an ion is achieved mainly by collisions of energetic argon electrons (and to a lesser extent by argon ions) with the ground-state atom (7). The ion then emerges from the plasma and is directed into the interface of the mass spectrometer (for details on the mechanisms of ion generation, please refer to Part I of this series: *Spectroscopy* **16**[4], 38–42 [2001]). This process of conversion of droplets into ions is represented in Figure 5.

The next installment of this series will focus on probably the most crucial area of an ICP mass spectrometer — the interface region — where the ions generated in the atmospheric plasma have to be sampled with consistency and electrical integrity by the mass spectrometer, which is under extremely high vacuum.

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