thermoscientific

TECHNICAL NOTE 43334

High performance radio frequency generator technology for the Thermo Scientific iCAP 7000 Plus Series ICP-OES

Keywords
Free-running, Plasma, RF generator, Solid-state

Using inductively coupled plasmas for optical emission spectrometry

An inductively coupled plasma (henceforth referred to as a ‘plasma’) is formed by seeding ions in a stream of argon which is flowing through Radio Frequency (RF) and magnetic fields in the region of the induction coil (Figure 1). The ions are formed by subjecting the argon gas to a high voltage spark and this causes small amounts of ionization of the argon. These electrons and ions are then accelerated by the magnetic field and collide with other argon atoms, causing further argon ionization and the subsequent formation of a plasma. The plasma is sustained by continual ionization of the argon via collisions induced by the magnetic field. This transfer of energy is known as inductive coupling. The energy that is transferred to the electrons and ions originates from the RF generator.

The plasma is contained within a quartz or ceramic tube known as a torch and can reach temperatures of up to 10,000 °C. To prevent melting or damage to the torch the argon is forced to flow tangentially at a high velocity, this cools the inner surface of the torch, forming a compact and stable plasma (Figure 1).

ThermoFisher SCIENTIFIC

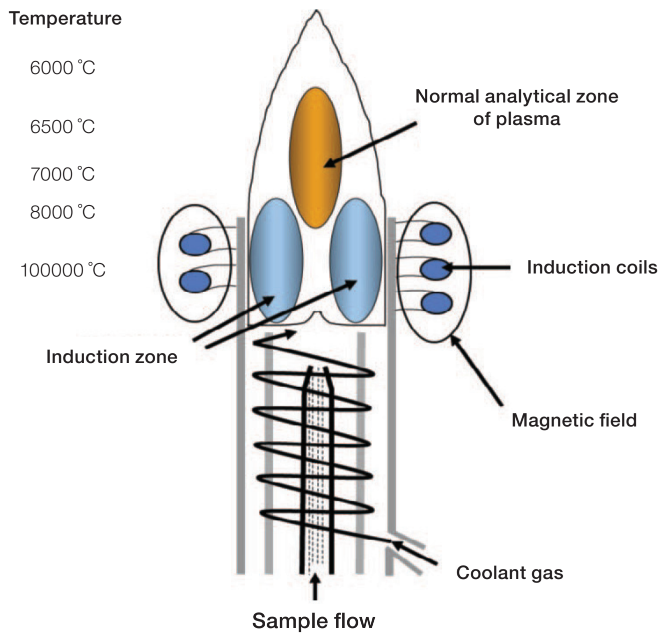

Figure 1. Plasma diagram, heating and viewing zones.

Aerosols generated from liquid or solid samples are fed into the plasma from various types of sample introduction systems or external accessories through a centrally located tube inside the torch. The plasma is at its most energetic and hottest where the sample is injected and the liquid evaporates and completely dissociates into its constituent atoms and ions within milliseconds (Figure 2). An efficient and powerful RF generator will develop and continually sustain a plasma that is capable of completely dissociating almost any sample matrix, thereby reducing oxide formation and other chemical interferences to a minimum.

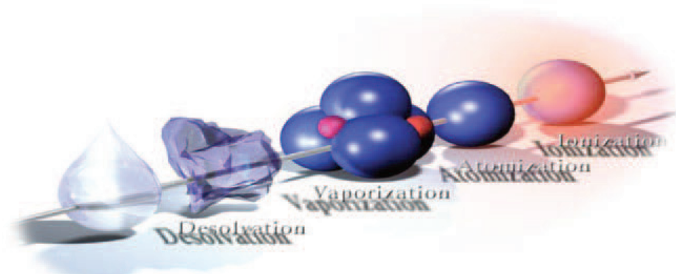

Figure 2. Sample processes in the plasma.

The constituent atoms and ions of the sample will now be able to absorb the energy from the surrounding plasma and will emit light at their own characteristic wavelengths. The emissions result from electrons absorbing energy in specific amounts, entering an excited state and subsequently releasing that energy as a photon of characteristic wavelength after returning to their original state. Due to the large energies present in the plasma, intense emission wavelengths are obtained making the plasma a very good tool to enable sensitive measurements of trace-level impurities with simultaneous measurement of high concentration sample constituents.

Common challenges for plasma generation

The design challenges for plasma generation in new Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) instruments are most noticeable in the older style generators. If the challenges are not solved then the effectiveness of the ICP-OES instrumentation is reduced. These challenges may include considerations such as size, robustness, efficiency, reliability and ease of service:

- RF generators are commonly large, heavy units, thereby forcing the ICP-OES instrument to be even larger.
- Traditional generators use power tubes and may not be able to cope with rapid transitions between heavy matrices, such as high metal or salt content and organic solutions.
- ICP-OES instrumentation with power tube generators have always been inefficient. Much of the power that is used with these instruments must be extracted as heat, this is wasteful and measures must be taken by laboratory to counteract the heat generated.
- Generators utilizing power tubes will need to have the power tubes replaced approximately every two years, depending on usage, and breakdowns due to old power tubes can occur at any time. Other generators may use many different parts, increasing the likelihood of failure.
- Traditional generators are complex items which require time-consuming procedures to repair if a failure occurs. Simplified generators can be serviced in a very short time with minimum downtime.

The stability of an RF generator greatly depends on its ability to adjust for changing conditions in the plasma due to differing samples or sample matrices by changing the power conditions to suit the variations – this is known as 'matching'. Historically, there have always been two main approaches to the control and matching of RF generators, crystal controlled or free-running. Crystal-controlled generators match and lock the frequency of the RF generator to the oscillation of a reference crystal (Figure 3). Free-running generators match the power generated to the power required by the plasma and allow the frequency to vary slightly (Figure 4).

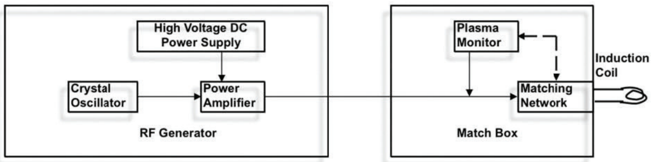

Figure 3. Simplified crystal-controlled RF generator design.

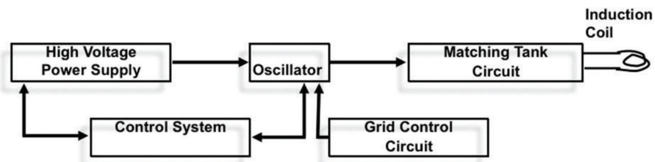

Figure 4. Simplified free-running RF generator design.

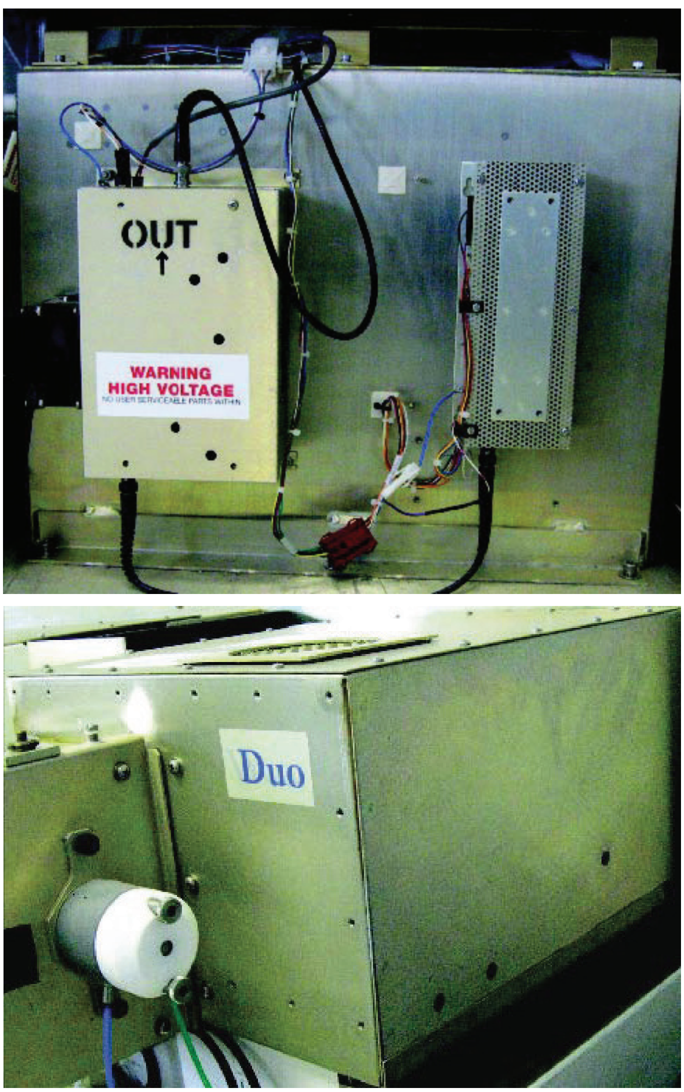

Figure 5. Pictures of a traditionally designed crystal-controlled RF generator. The power supply (top) and match box (bottom) give a total mass of both is approximately 90 – 100 kg. The total volume and weight of this generator is greater than that of a complete iCAP 7000 Plus Series ICP-OES instrument.

Rapid changes in plasma composition, such as changes in sample type or matrix, require an RF generator that will quickly respond to changes and achieve stable plasma as rapidly as possible. For instance, the plasma may be extinguished on older, slower reacting RF generators by introducing an organic solvent into the plasma directly after an aqueous sample.

Thermo Scientific iCAP 7000 Plus Series ICP-OES solid-state RF generator design

The Thermo Scientific™ iCAP™ 7000 Plus Series ICP-OES solid-state RF generator is an advanced, free-running RF generator, with solid-state chips and circuits replacing the vacuum power valves and other outdated components of traditional designs. Historic designs tended to be slow to 'match' the plasma to different matrices whereas the iCAP 7000 Plus Series ICP-OES RF generator is solid-state and responds rapidly to changes within the plasma. In addition, the iCAP 7000 Plus Series ICP-OES RF generator is a simple design which is compact, robust, power efficient and reliable. The efficient design with fewer components enables easier, rapid servicing and helps to reduce instrument downtime since the components can be easily replaced if required.

The iCAP 7000 Plus Series ICP-OES RF generator is a free-running, solid-state, 27.12 MHz RF generator capable of transferring 1600 W of RF power into the plasma with power regulation better than 0.1%. The solid-state design provides stability, reliability and high-power in a small footprint.

This innovative RF generator utilizes solid-state transistors, a 3kW solid state DC power supply, printed circuit boards and field-effect transistors (FET) in an integrated design. The design incorporates only three major parts plus the induction coil (Figure 6), all solid-state, providing a robust and reliable design.

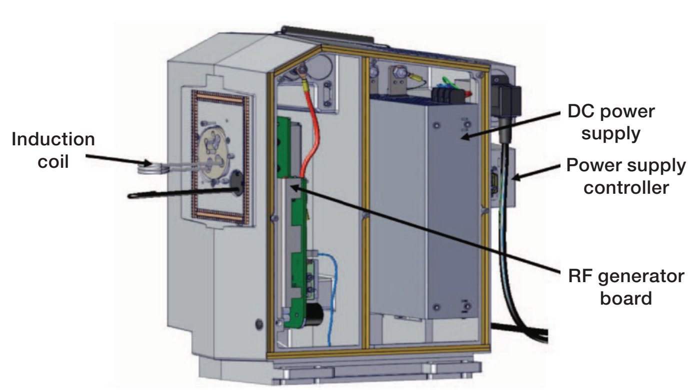

Figure 6. The schematic shows the DC Power Supply Unit (PSU), feedback Power Supply Controller, main RF Generator Board and Induction Coil.

The diagram in Figure 4 shows the simplicity of the iCAP 7000 Plus Series ICP-OES RF generator design. In comparison to the crystal-controlled RF generator (Figure 3), the new RF generator board performs the same function as the match box and the new DC power supply equates to the large, heavy power cube. The entire mass of the iCAP 7000 Plus Series ICP-OES RF generator is approximately 20 kg with the casing forming the majority of the mass and less than one quarter of the volume of the crystal-controlled generator in Figure 5.

Thermo Scientific iCAP 7000 Plus Series ICP-OES RF generator capabilities and benefits

The iCAP 7000 Plus Series ICP-OES highly advanced solid-state RF generator has many advantages over the original power tube designs and less efficient solid-state designs.

Efficiency

The iCAP 7000 Plus Series ICP-OES RF generator provides high efficiency of greater than 78% DC to RF power even under the most difficult conditions. This means that the RF generator supplies a minimum of 78% of the incoming power to the plasma, enabling the iCAP 7000 Plus Series ICP-OES instrument to analyze challenging organic solvents and salt solutions containing high concentrations of metals, even when using low power settings.

Impurities in metal plating solutions are a common, challenging analysis due to the high metal content of the matrix (typically containing 50-100 g·L^{-1} of metal or more). Figure 7 shows a calibration graph of silver standards (using the Ag 328.068 nm wavelength) in 50 g·L^{-1} nickel solutions at various power levels. All calibrations obtained are remarkably linear even when low RF power is used and this in the presence of high concentrations of nickel in solution. If a less efficient RF generator was used, there is the possibility that excessive curvature of the calibration may occur at lower RF powers or the plasma may even be extinguished.

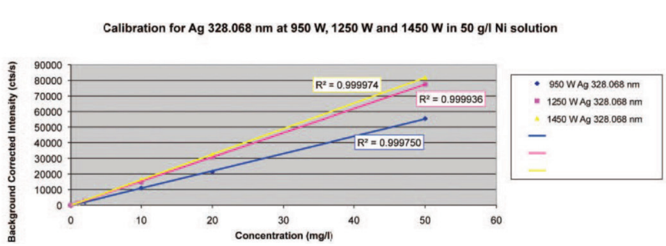

Figure 7. Calibration graphs of silver in 50 g·L^{-1} nickel solutions at various power levels.

Methanol is a volatile water-miscible organic solvent. When analyzed by ICP-OES, the solvent may generate too much organic aerosol and extinguish the plasma. Commonly, these types of volatile solvents are analyzed at high power settings and the spray chamber is cooled to reduce the amount of organic vapor produced, sometimes as low as -15°C. Efficient transfer of energy into the plasma enables the iCAP 7000 Plus Series ICP-OES RF generator to keep the plasma stable, even at ambient temperatures or lower RF powers. A 50% methanol solution was analyzed for copper (Cu 327.396 nm) using a standard organic sample introduction kit at ambient temperature. The calibrations shown in Figure 8 demonstrate that the RF generator within the instrument is capable of enabling excellent linear calibrations in the most difficult matrices.

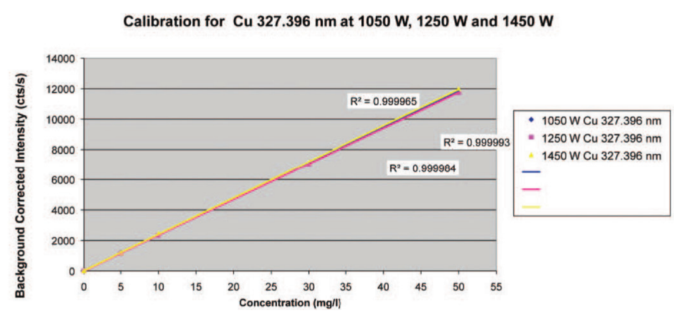

Figure 8. Calibration graphs of copper in 50% methanol at various power levels.

Although some RF generators cannot generate stable plasmas with high concentrations of metal and organic solvents, especially at low powers, the iCAP 7000 Plus Series ICP-OES solid-state RF generator removes the restrictions on power for even the most difficult matrices. The instrument solid-state RF generator, with its efficient power transfer, producing excellent calibrations at high or low RF powers, enables the user to determine the optimum power settings for the element suite to be analyzed.

The iCAP 7000 Plus Series ICP-OES RF generator also demonstrates efficiency by the minimal amount of heat energy it wastes. Even at the highest RF power settings, the amount of heat generated from the entire instrument is minimal (less than 700 W) with the main RF generator contributing less than 300 W.

The efficiency of the iCAP 7000 Plus Series ICP-OES RF generator enables reduced power consumption, lower waste heat generation coming from the generator and a greater capacity to be able to reduce power while still achieving the same performance, with or without challenging matrices.

Stability

Stability of the iCAP 7000 Plus Series ICP-OES RF generator is enhanced because of its efficient power usage better cooling and less susceptibility to intensity drift due to environmental change. In addition, matrix composition does not affect the RF generator performance as much as in older designs due to the rapid response and power efficient design. The iCAP 7000 Plus Series ICP-OES solid-state RF design allows the plasma to reach a stable state after changing matrices much more rapidly than older designs. This produces more consistent, stable intensities when changing samples, organic solvents or even from water to high salts solutions.

Figure 9 shows the excellent stability (with <2% RSD over the 8 hour period) achievable by the iCAP 7000 Plus Series ICP-OES RF generator. The solution used was a 50 $g \cdot L^{-1}$ nickel solution spiked with various concentrations of elements. The solution was continuously aspirated and the intensities plotted as a percentage over the full 8 hour analysis. Such stability enables fewer calibrations, fewer QC failures and, hence, increased sample throughput and reduced costs compared to older style, legacy ICP-OES instruments RF generators.

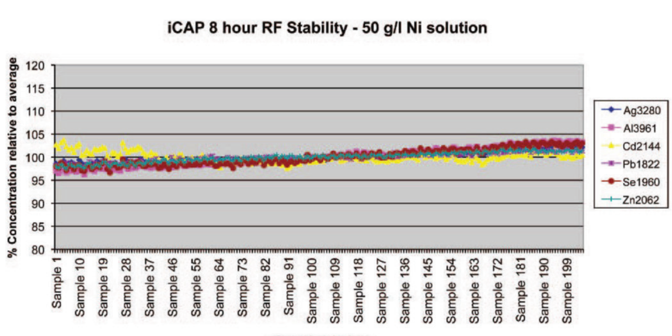

Figure 9. Stability analysis (8 hours) in a 50 $g \cdot L^{-1}$ nickel solution.

Robustness

The iCAP 7000 Plus Series ICP-OES RF generator is especially robust with a rapid response time to changes in the plasma loading. In contrast to older designs, solid-state RF generators are known for their rapid reaction times and robustness when dealing with changing matrices. This rapid response enables the intensity to stabilize rapidly, giving a flat plateau immediately after the wash-in period. This enables the analyst to shorten the stabilization time since the RF generator requires less time to reach its optimum and stable power level. The benefit of the greater robustness of the iCAP 7000 Plus Series ICP-OES is improved stability when changing sample matrices in the same method and reduced stabilization time, reducing the analysis time as a whole.

Conclusion

The development of a small, efficient, rapid response solid-state RF generator enables the Thermo Scientific iCAP 7000 Plus Series ICP-OES instrument to have a very small footprint and robust sample handling capabilities. Even at low RF powers, the RF generator robustness keeps the plasma stable and enables analyses in challenging matrices that most other RF generators need much higher power settings to perform. Greater stability from the solid-state RF generator compared to traditional RF generators also enables the analyst to utilize longer, more efficient analyses without the need for recalibration. The Thermo Scientific iCAP 7000 Plus Series ICP-OES RF generator's innovative solid-state design has resulted in an efficient, ICP-OES solution which provides the lowest cost of analysis per sample.

Find out more at **thermofisher.com/ICP-OES**

ThermoFisher SCIENTIFIC