New Radio Frequency Generator Technology for Thermo Scientific iCAP 6000 Series
ICP-OES; Powerful Technology Innovation for the most Rugged, Reliable Performance

Benefits in Brief

- Compact solid-state RF generator enables smallest instrument footprint
- Advanced design concept reduces the number of component parts and improves reliability and serviceability
- High efficiency energy transfer enables routine heavy matrix analysis at reduced plasma powers for optimum performance and cost efficiency
- Rapid response matching to changing plasma conditions enables easy transitions from aqueous, high salt and organic solutions

Why use plasma for Optical Emission Spectrometry?
An inductively coupled plasma (further referred to as ‘plasma’) is formed by seeding ions in a stream of argon which is flowing through 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 RF generator.

The plasma is contained within a quartz or ceramic tube known as a torch and can reach temperatures of up to 10000 °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 and forms a compact, stable ICP plasma (Figure 1).

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. 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

Why use plasma for Optical Emission Spectrometry?

The sample’s constituent atoms and ions will now be able to absorb the abundant energies 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 lines are possible 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 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:

- Size – RF generators are commonly large, heavy units, thereby forcing the ICP-OES instrument to be even larger.
- Robustness – old style 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 organics solutions.
• Efficiency – 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.

• Reliability – 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.

• Ease of service – old-style 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 - 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.

Rapid changes in plasma composition, such as changes in sample type or matrix, require an RF generator which 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.

**Solid State RF Generator Design and Effectiveness**

**Why Design a Solid State RF Generator?**

The Thermo Scientific iCAP 6000 Series ICP-OES predecessor, the Thermo Scientific Iris Intrepid II ICP-OES used a crystal controlled RF generator. Although robustness and stability compared well with other ICP-OES instruments on the market, the Iris Intrepid II lacked certain attributes which were needed to produce a robust, cost-effective, user and engineer-friendly generator. Computer aided design packages were used to ensure the iCAP 6000 Series ICP-OES RF generator design is both as robust and reliable as possible while maintaining a small footprint and producing efficient power output.

**Thermo Scientific iCAP 6000 Series ICP-OES Solid State RF Generator Design**

The Thermo Scientific iCAP 6000 Series ICP-OES solid-state RF generator is an advanced example of a free-running RF generator with solid-state chips and circuits replacing the vacuum power valves and other outdated components of the older style designs. Older designs tended to be slow to “match” the plasma to different matrices whereas the iCAP 6000 Series RF generator is solid-state and responds rapidly to changes within the plasma. In addition, the iCAP 6000 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 6000 Series RF generator is a free-running, solid-state 27.12 MHz RF generator capable of transferring over 2 kW of RF power into the plasma. The solid-state design provides stability, reliability and high-power in a small footprint.
General Principles

The iCAP 6000 Series ICP-OES RF generator utilizes solid-state chips, a 3kW solid state DC power supply, printed circuit boards and field-effect transistors (FET) in an integrated design. The design incorporates only 3 major parts plus the induction coil (Figure 6), all solid-state, providing a robust and reliable design.

Figure 6. iCAP 6000 Series ICP-OES RF generator simplified design – shows DC Power Supply Unit (PSU), feedback Power Supply Controller, main RF Generator Board and Induction Coil

The diagram in Figure 6 shows the simplicity of the iCAP 6000 ICP-OES Series RF generator design. In comparison to the crystal controlled RF generator shown in 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 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 4.

The Thermo Scientific iCAP 6000 Series ICP-OES RF generator capabilities and benefits

The highly advanced solid state RF generator has many advantages over the original power tube designs (which haven’t substantially changed in 20 years) and less efficient solid state designs.

Efficiency

The iCAP 6000 Series ICP-OES RF generator shows remarkable power efficiency of greater than 78 % 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 6000 Series ICP-OES to analyze challenging organic solvents and salt solutions containing high concentrations of metals, even when using low power settings. Analysis of impurities in metal plating solutions are a common, challenging matrix due to the high metal content - typically containing 50-100 g/l of metal or more. The analysis of 50 g/l of Nickel metal solutions is shown below with calibrations at various power levels.

The graph below shows a calibration graph of Ag 328.068 nm at a variety of power levels with five solutions of fifty grams per liter nickel (50 g/l Ni) spiked with silver to form a set of standards.

Figure 7. Calibration graphs of silver in 50 g/L Ni solution at various power levels

Note that all the calibrations are remarkably linear even when low RF power is used in the presence of high concentrations of Ni metal in solution. If a less efficient RF generator was used then there is the possibility that excessive curvature of the calibration may occur at lower RF powers or the plasma may even be extinguished.

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 RF to keep the plasma stable, even at ambient temperatures or lower RF powers. A 50 % methanol solution was analyzed on an iCAP 6000 Series ICP-OES using a standard organics sample introduction kit at ambient temperature. The solid state RF generator, with its efficient power transfer, produces excellent calibrations at high or low RF powers, enabling the user to determine the optimum power settings for the element suite to be analyzed.

Figures 8. Calibration graphs at various power levels with 50 % Methanol

The calibrations shown in Figure 8 demonstrate that the RF generator within the instrument is capable of enabling excellent linear calibrations at even low RF powers in the most difficult matrices. Although some RF generators cannot generate stable plasmas with high concentrations of metal and organic solvents, especially at low powers, the iCAP 6000 Series solid-state RF generator removes the restrictions on power for even the most difficult matrices.

The iCAP 6000 Series ICP-OES RF generator also demonstrates its efficiency by the minimal amount of heat energy it wastes. Even at the highest RF power settings, the amount of heat generated from the iCAP 6000 Series ICP-OES as an entire instrument is minimal (less than 700 W) with the main RF generator producing less than 300 W waste heat.
The efficiency of the iCAP 6000 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 RF generator is enhanced because of efficient power usage by the RF generator, more efficient 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 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.

The 8 hour stability graph (Figure 9) shows the excellent stability (with <2 % RSD over the 8 hour period) achievable by the iCAP 6000 Series ICP-OES RF generator. The solution used was a constantly aspirating 50 g/l Ni solution spiked with various concentrations of elements and the intensities were graphed as a percentage over the full 8 hour analysis.

The stability shown in Figure 9 enables fewer calibrations, fewer QC failures and thereby increased sample throughput and reduced costs compared to older style, legacy ICP-OES instruments RF generators.

**Robustness**

The iCAP 6000 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.

The benefit of the greater robustness compared to older style RF generators is improved stability when changing sample matrices in the same method and reduced stabilization time, reducing the analysis time as a whole.

Figure 10 displays the change in background corrected intensity when changing between de-ionized water and a spiked sample with a high matrix.

**Conclusion**

The development of a small, efficient, rapid response solid state RF generator has enabled Thermo Scientific to create the iCAP 6000 Series ICP-OES instrument with a very small footprint and robust sample handling capabilities.

Even at low RF powers, the iCAP 6000 Series ICP-OES RF generator’s robustness keeps the plasma stable and running analyses in challenging matrices that most other RF generators need much higher power settings to analyze.

Greater stability from the solid state RF generator compared to older style RF generators enables the analyst to utilize longer, more efficient analyses without the need for recalibration.

The iCAP 6000 Series ICP-OES Series RF generator solid state design has thus resulted in a more robust, efficient, smaller and more stable ICP-OES instrument.