CASE STUDY 44490

Improving analytical capabilities at Eurofins EAG Laboratories using an iCAP PRO Series ICP-OES

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Challenges of ICP-OES material testing

The key attributes that drive the selection of an ICP-OES instrument include the instrument footprint, ease of operation, user training, instrument stability, speed and cost of analysis, ease of maintenance, and instrument uptime. Other highly desirable options include the use of automated processes for operation, data processing and software workflows with data traceability.

The Thermo Scientific™ iCAP™ PRO Series ICP-OES and Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software provide unique features to improve the capabilities of a typical analytical laboratory. Some of these features are highlighted further in this document with the benefit they bring to the overall performance and productivity of the laboratory.

ICP analysis and Eurofins EAG Laboratories

Eurofins EAG Laboratories is a global leader in materials testing services with accredited laboratories in over 20 locations, serving more than 4,000 clients worldwide. Eurofins EAG utilizes various types of inductively coupled plasma (ICP) instruments in areas such regulatory compliance, supply chain testing, purity certification, additive manufacturing, ceramics, and failure analysis support.[1]

Eurofins EAG offers various ICP-based analytical techniques, including ICP-MS, ICP-OES, and LA-ICP-MS for accurate and precise determination of major, minor, and trace elements in environmental samples, food and beverages, and bulk chemical analyses of high-performance materials. The combination of complete dissolution, or digestion of a material, and its analysis by inductively coupled plasma-based techniques is therefore unique and extremely powerful. ICP techniques are considered among the most accurate and precise analytical techniques, not only within Eurofins EAG Laboratories' toolbox, but also throughout the world of analytical chemistry.

Eurofins EAG Laboratories analyzes various materials such as superalloys (high performance alloys used in aerospace, power, metal refining, and petrochemical applications). This analysis is carried out on a regular basis. Elements including tungsten, molybdenum, and chromium or rare earth elements are intentionally added with alloying components to improve specific properties of the material.

It is important to assess and control the level of these additive elements and possible elemental contaminants. The presence of elemental impurities can deleteriously affect the performance and lifetime of components and possibly lead to failures. Hence, there are strict specifications in place for superalloy chemical composition set by organizations such as the Society of Automotive Engineers (SAE), Military (MIL-SPEC), ASTM, and ISO.

Intuitive hardware and software for easy adoption by technicians

In high-throughput, fast turn laboratories, such as Eurofins EAG, one technician might operate various equipment from different manufacturers, so it is imperative to use instruments that do not require extensive training. The iCAP PRO Series ICP-OES instruments have a small footprint with no connections at the back, ensuring the instruments fit comfortably in the available laboratory space. The easily accessible sample introduction system, consisting of peristatic pump and tubing, spray chamber, nebulizer, injector tube and demountable torch, can be removed and re-fitted within a few minutes by users with basic training. This aspect leads to minimal change-over time for any laboratory. Since all components of the sample introduction system are pre-aligned, no special alignment is required when it is reassembled. The push-fit fittings simplify the setup of the sample introduction system so it can be easily performed by lab technicians without the need of special training. This feature is especially useful for testing laboratories that analyze high matrix containing samples with high sample throughput, where frequent cleaning of various parts of the sample introduction system is needed.

Automated and fast instrument readiness for day-to-day analysis

The Qtegra ISDS Software, which controls instrument operation, traceability in data acquisition, and data reporting, offers the 'Get Ready' feature, enabling users to operate the instrument without any manual interventions. It includes an automated sequence of processes, including plasma ignition, spectrophotometer optimization, auto-tune, performance check, and automatic shutdown at the end of sequence, permitting easy integration within the Eurofins EAG Standard Operation Procedures (SOP). By automating these tasks, the operator can focus on others, such as sample and standard preparation, setting up the autosampler, creating LabBook sample sequences, increasing overall sample throughput, and efficiency.

A direct effect of using automated startup is faster startup time and reduced purge gas requirements, with startup times of just 30 minutes from power off and 5 minutes from standby modes. This reduces the argon consumption for plasma stabilization and other non-productive steps and lowers the cost of analysis per sample, improving lab productivity.

The comprehensive instrument dashboard in Qtegra ISDS Software provides all necessary information related to the iCAP PRO Series ICP-OES and other third-party peripherals, such as the autosampler and fast sampling valves, instrument status indicators and safety interlocks. It also provides simple access to various functions like autotuning, auto-peak, measurement mode, and tune set editor, minimizing the need of switching from one application to another, which makes operation much easier and faster.

"The hardware and software design and capability ensure that there is minimal time required for training new technicians and operators at this Eurofins EAG site. The ease of assembling the sample introduction system coupled with the powerful Qtegra Software will improve overall productivity at Eurofins."

– Rajiv Soman, PhD (Eurofins EAG Laboratories)

Fast sample analysis for higher sample throughput

The new optical design and CID detector allows truly simultaneous measurement of emission signals in the entire spectral range of 167 nm to 852 nm in a single acquisition, improving the speed of analysis.

The use of available default optimized tune sets for aqueous and organic sample types reduces time-consuming method development efforts and provides accurate, precise, and reliable analytical data in the minimum possible time. Furthermore, it also offers advancement in terms of software controlled auto-tune depending upon the application. To achieve optimum performance for a specific application, software-controlled tuning of various instrument parameters including RF power, auxiliary gas flow, nebulizer gas flow, and radial viewing height can be performed.

The use of iFR mode allows the entire spectrum to be captured in one measurement, enabling Eurofins EAG Laboratories to improve analysis speed by >20%.

For Eurofins EAG Laboratories, where many different types of samples are expected to be analyzed every day, the speed of analysis is an important consideration. As the cost of analysis per sample depends directly upon the speed of analysis and the total number of samples analyzed per day, speedy sample analysis plays a vital role enhancing productivity and profitability of the analytical testing facility. It is also important to fulfill workload requirements, enabling the analytical testing laboratory to meet and exceed their valuable customer's expectations in terms of turnaround time and data quality.

Tools for easy method development

The analytical measurement with ICP-OES could be prone to spectral interferences; this is true for any ICP-OES instrument.

Therefore, to make reliable measurements of analytes, it is important to choose an interference-free analyte wavelength for the measurement. The comprehensive wavelength library available in Qtegra ISDS Software provides useful information while creating the analytical method regarding possible interferences and their relative intensities at the analyte wavelength. It enables the user to select the interference-free emission line of the analyte depending upon possible interferences. It helps develop methods quickly, avoiding trial and error, and helps avoid repeated analysis of samples, which otherwise need to be performed to assess method accuracy and precision.

The full-frame feature with the capability of acquiring a full emission spectrum of the sample covering the entire UV-visible wavelength region is a powerful tool to know the elemental composition of unknown samples (Figure 1). The data obtained through a full-frame scan are useful in the process of method development, as they give a fair idea about possible interferences and their concentrations. If required during method development, this allows technicians to choose additional appropriate and interference-free wavelengths for analysis and to pre-determine the linearity range and sample preparation steps, including sample weight and dilution factors.

"A true simultaneous capability of measurements in a single acquisition, coupled with ease of background correction algorithms, will enable a high degree of accuracy and precision of measurements, especially as we encounter complex sample matrices, and elemental compositions that can have a wide range of concentrations. This feature will improve overall productivity and help Eurofins EAG Laboratories with our ambitious goal of analysis done right, first time, and every time."

– Rajiv Soman, PhD (Eurofins EAG Laboratories)

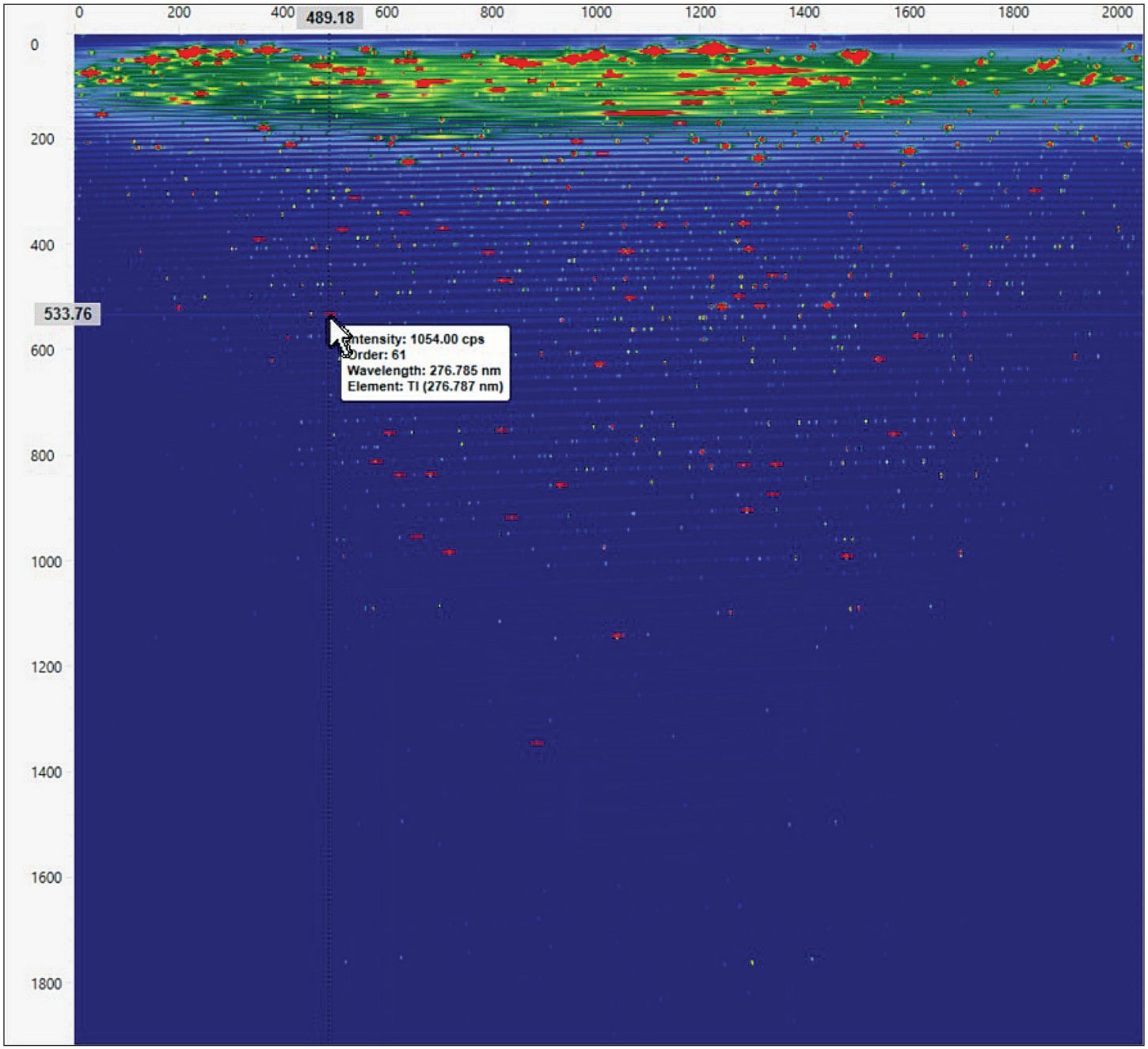

Figure 1. Full-frame image of the standard solution captured in iFR mode covering the entire UV-Visible region

"The full-frame feature coupled with tabulation of interfering elements in the proximity of the wavelength of analysis is certainly a key feature in efficient method development work on new types of samples."

– Rajiv Soman, PhD (Eurofins EAG Laboratories)

Addressing the needs for sensitivity and linear dynamic range

The capability of an analytical instrument to detect and quantify the lowest analyte concentration, which is often termed sensitivity, is a critical performance parameter for analytical testing laboratories including material testing. Sensitive instruments allow for the quantification of target analytes with the required degree of accuracy and precision in challenging samples, reducing the need for tedious method development.

The iCAP PRO Series ICP-OES has a unique optical design that has enhanced light transmission compared to previous generation ICP-OES. This offers improved detection limits for analytes over the wide range of wavelengths from 167 to 852 nm. The eUV measurement mode provides further improved sensitivity for the analytes at lower emission wavelengths (167–240 nm), such as aluminum, arsenic, selenium, phosphorus, and mercury, which are often required to be analyzed as contaminants and are considered challenging when quantified at lower concentrations (Table 1).

The iCAP PRO Series ICP-OES designed with a vertical plasma torch offers the flexibility to choose between the axial or radial plasma views depending upon the expected concentrations of target analytes and allows measurement of high matrix samples on a Duo instrument with a very high accuracy. In general, the axial view is useful for low concentrations, while the radial view provides reliable measurements of analytes with higher concentrations.

The iCAP PRO Series ICP-OES in combination with Qtegra ISDS Software offers another useful feature called "wavelength switching", where different wavelengths with different sensitivities can be used for a single analyte. This allows calibration of the analyte over a wider range of concentrations, using more than one analyte line. This offers more flexibility for analyzing samples with varying concentrations of analyte without the need of further dilution of samples with high analyte concentrations. This allows analytical testing laboratories to improve sample turnaround time, thus improving the productivity of the entire analytical process.

Table 1. Typical detection limit values achieved for elements measured using the axial iFR mode

Element	Wavelength (nm)	IDL (mg • L^{-1})	Element	Wavelength (nm)	IDL (mg • L^{-1})
Silver (Ag)	328.068	0.001	Potassium (K)	766.490	0.0007
Aluminum (Al)	167.079	0.0005	Lithium (Li)	610.362	0.001
Barium (Ba)	455.403	0.0001	Magnesium (Mg)	279.553	0.0001
Calcium (Ca)	393.366	0.0005	Manganese (Mn)	257.610	0.0001
Cadmium (Cd)	228.802	0.001	Sodium (Na)	589.592	0.001
Cobalt (Co)	238.992	0.001	Nickel (Ni)	231.604	0.002
Chromium (Cr)	283.563	0.0007	Lead (Pb)	220.353	0.005
Copper (Cu)	324.754	0.001	Vanadium (V)	309.311	0.001
Iron (Fe)	259.940	0.001	Zinc (Zn)	213.856	0.0006

> “Eurofins EAG Laboratories will greatly benefit by the “switching wavelength” software option, especially since we might encounter ranges of elemental compositions within a batch, or within multiple batches in a single run. This feature will not only ensure that the appropriate calibration range is used during analysis but also meet our stringent QC criteria, which is part of our accreditation requirements.”
>
> – Rajiv Soman, PhD (Eurofins EAG Laboratories)

In the example presented in Figure 2, sodium (Na) is measured in Radial-iFR mode using two different emission wavelengths with different sensitivities, i.e., primary wavelength of 589.592 nm and the relatively less sensitive 818.326 nm. A different range of linearity standards is used for each line using a single calibration block; linearity standards of 2.5, 25, and 100 ppm are used for 589.592 nm, whereas higher concentrations of 100 and 1,000 ppm are used for 818.326 nm.

In the method parameters, there is a provision to set the concentration switch point for each wavelength by virtue of which subsequent sample concentrations are reported using the appropriate wavelength.

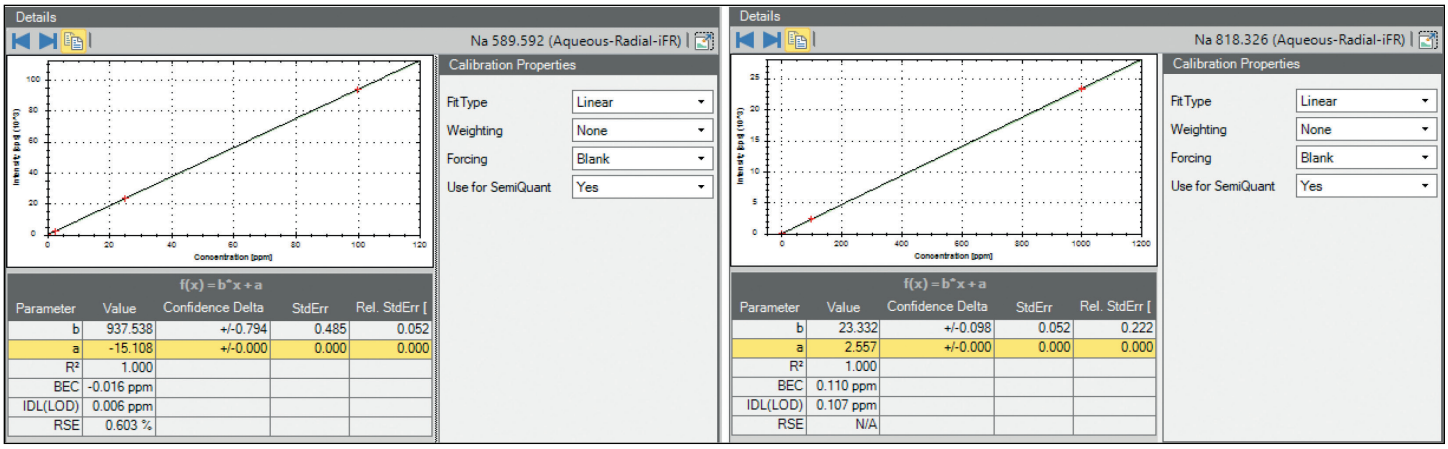

Figure 2A. Calibration curves and figures of merit for sodium at 589.592 nm and 818.326 nm

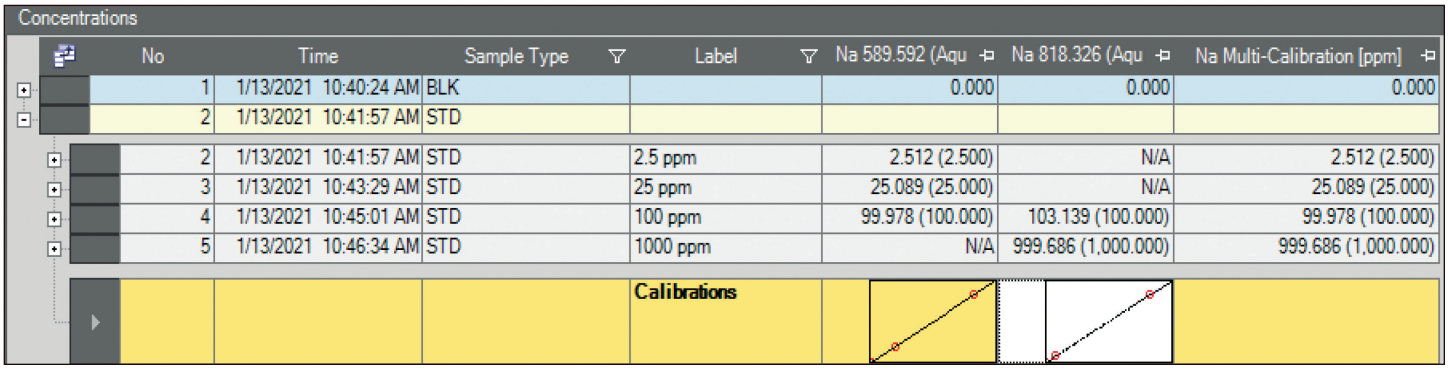

Figure 2B. Multi-trace quantification of sodium using the primary emission line of 589.592 nm and the less sensitive 818.326 nm

Minimal analytical signal drifts

The analytical signal measured by ICP-OES tends to vary frequently as it is almost impossible to regain the same plasma conditions every day and every time the plasma is ignited. The analytical signal also depends largely upon factors like the alignment of the sample introduction system components (nebulizer, injector tube, plasma torch) and steady and stable delivery of the liquid sample to the plasma through the peristatic pump tubing. Since the signal can vary, the general recommendation is to calibrate the instrument with known concentrations of analyte before the start of the analytical sequence once the plasma is ignited. Depending upon the internal SOP and the number of standards that need to be measured as part of the daily calibration, the entire process typically takes around 25–30 min. For a contract analytical testing laboratory, every minute is critical and has a huge impact on the sample turnover and overall productivity of the laboratory.

Despite this well-known limitation of such techniques and the associated challenges, it has been observed that over a period of almost three months the iCAP PRO Series ICP-OES delivers stable and consistent analytical signal for analytes with emission lines over the entire wavelength region (Figure 3). A maximum variation of 15% has been seen for the intensities of 1 mg/L multi-element standard solution of different analytes measured over a period of 90 days (60 discrete analysis measurements). By leveraging this kind of signal stability, one can avoid tedious daily instrument calibration and proceed with analysis by running and confirming the accuracy of the initial calibration verification (ICV) QC standard against the imported calibration block from the previous day(s).

Ensuring robust performance for every sample type

An ICP-OES instrument with a vertical torch is a proven configuration for improved matrix tolerance and robustness. The radial-only instrument with a vertical orientation of the plasma torch is a common and widely used instrument configuration for the analysis of high total dissolved solids (TDS) containing samples and organic solvents. The iCAP PRO Series ICP-OES instrument offers a vertical torch design for both duo and radial-only instruments, enhancing matrix robustness and achieving lower detection limits values at the same time.

The obvious major challenge with such a design is contamination and corrosion caused by deposition of salts on the plasma interface. Another challenge is exposure of the interface to the high plasma temperature and acid fumes, which damages the interface and optical components in the long run. These two challenges have been addressed smartly by introducing innovative geometry and airflow to avoid corrosion. The ceramic cones available for both axial and radial views are resistant to high temperature and to attack by acid fumes. Furthermore, these cones are easy to remove, clean, and refit when needed and can be done by a user whenever required. In addition, the inner torch box is removable and can be cleaned by the chemist whenever required as determined by the nature and frequency of the samples analyzed. From the perspective of an analytical testing laboratory, the instrument robustness and minimum downtime is a key factor in improving sample turnaround time and overall productivity and profitability of the laboratory's operation.

Figure 4 represents the matrix robustness of the iCAP PRO Series ICP-OES in terms of consistent recovery of 10 analytes spiked in 25% NaCl solution over extended period.[1]

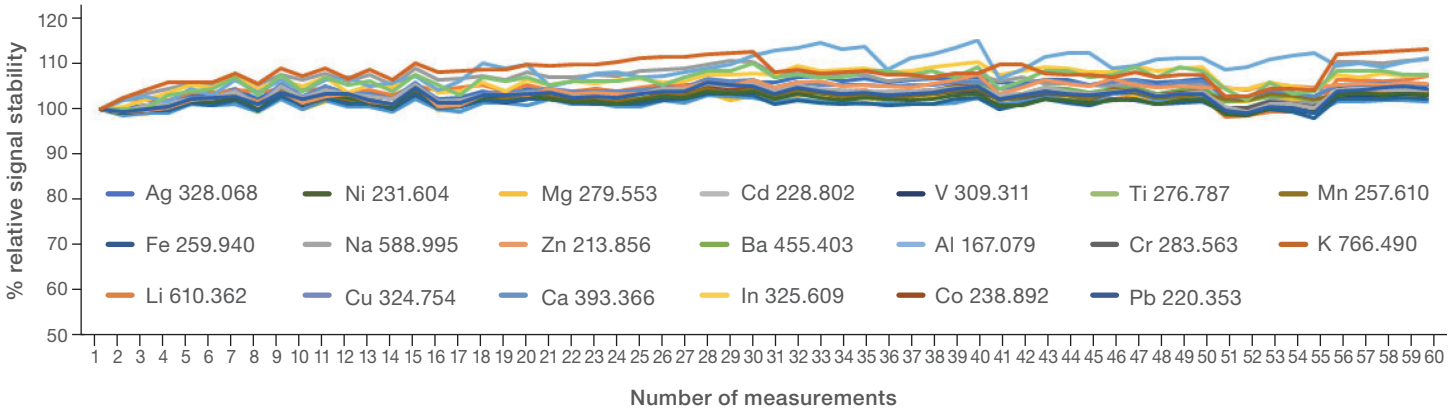

Figure 3. iCAP-PRO Series ICP-OES system stability as demonstrated by relative signal intensity (cps) obtained by measurements of a 1 mg/L standard solution of 20 analytes in 2% nitric acid over a period of 90 days (total 60 discrete measurements sets)

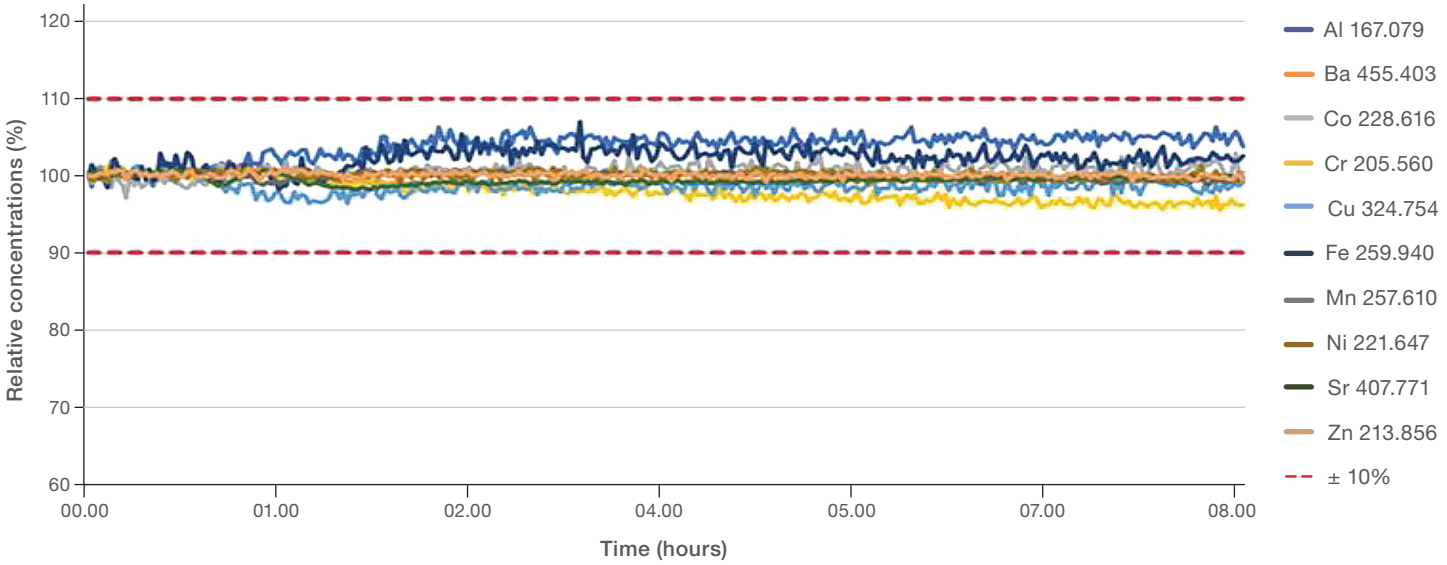

Figure 4. iCAP-PRO Series ICP-OES instrument robustness as demonstrated by % recovery of 10 analytes spiked in 25% NaCl solution over period of 8 hours

Simplification of data interpretation and data reporting with Qtegra ISDS Software

The most important part of day-to-day operation of an analytical laboratory is data processing, interpretation of the results including data reviewing, and reporting the results to customers. The simplified workflow of the Qtegra ISDS Software offers various features to ensure the quality of data, including linearity, QC standards behavior, out of range results, etc., with one glance at the generated data. The comprehensive sample list page allows the user to perform sample-specific final calculations by adding the sample weight, final volume made, and dilution factor at any stage of the analytical run, which helps to avoid tedious manual external calculations. The exclusive QC functions available in the Qtegra ISDS Software such as ICV *(Initial Calibration Verification)*, CCV *(Continuing Calibration Verification)*, LCS *(Laboratory Control Standard)*, and QCS *(Quality Control Standard)* in conjunction with the flagging feature help in keeping a check on the accuracy and repeatability of the analysis without the need to invest much time in data evaluation. The dedicated QC functions such as MXS *(Matrix Spike)*, PDS *(Post Digestion Spike)*, and LFB *(Laboratory Fortified Blank)* are useful tools for the automatic calculation of spike and recovery studies, which are some of the most important parameters of method validation.

The Qtegra ISDS Software offers different types of report templates to convert analytical data into a non-editable pdf format. Furthermore, default report templates can be edited to incorporate required information in the customized format including concentration data, raw intensities, linearity plots, QC recoveries, method parameters, and IS recoveries in a single analytical report.

All above mentioned calculation options and reporting features are extremely helpful to minimize manual efforts and ensure data quality and traceability, ultimately improving the productivity of the analytical laboratory. Some example reports useful for day-to-day data reporting available in Qtegra ISDS Software are presented in Figure 5.

LabBook: Example reporting.imexp

Index: 21
Sample: Spike sample
Sample type: QC
Analysis started at: 1/25/2021 5:54:34 PM

Standard DF: 1 Rack 0 Vial 4

Category	Ag 328.068 (Aqueous-Axial-iFR)	K 769.896 (Aqueous-Axial-iFR)	Co 228.616 (Aqueous-Axial-iFR)	Ni 221.647 (Aqueous-Axial-iFR)
Concentration average	9.97 ppm	10.23 ppm	9.98 ppm	10.00 ppm
Intensity average	64434.67 cps	176144.11 cps	14779.13 cps	33077.06 cps

Figure 5A. Simple sample report with concentrations and raw intensities

LabBook: Example reporting.imexp

Index: 21
Sample: Spike sample
Sample type: QC
Analysis started at: 1/25/2021 5:54:34 PM

Standard DF: 1 Rack 0 Vial 4

Analyte	Unspiked Amount (ppm)	Spiked Amount (ppm)	Expected Amount (ppm)	Total Amount Found (ppm)	Recovery (%)
Ag 328.068 (Aqueous-Axial-iFR)	0.22	10.00	10.22	9.97	97.5
K 769.896 (Aqueous-Axial-iFR)	0.25	10.00	10.25	10.23	99.7
Co 228.616 (Aqueous-Axial-iFR)	0.50	10.00	10.50	9.98	94.8
Ni 221.647 (Aqueous-Axial-iFR)	0.21	10.00	10.21	10.00	97.9

Figure 5B. Qtegra ISDS report with detailed information of matrix spike and recovery study

Summary

- The iCAP PRO Series ICP-OES with the Qtegra ISDS Software is a combination of intuitive hardware and software that facilitates easy adoption of the system by new technicians.
- Automation with the Get Ready feature offers unattended instrument readiness for day-to-day analysis, allowing technician's time for other activities. This automation also helps in reducing argon consumption, lowering cost of analysis per sample, increasing sample throughput, and improving laboratory productivity.
- Simultaneous measurement of the whole emission spectrum speeds up the analysis significantly. The iFR measurement mode provides reliable and consistent data in a very short time, which allows analysis of a greater number of samples within a given time.
- The comprehensive wavelength library and full-frame imaging provides useful information beforehand, which enables technicians to develop analytical method with minimum effort and trials.
- The optical design with enhanced light transmission improves instrument sensitivity, enabling accurate and precise determination of low-level impurities. The combination of axial and radial plasma view offers a wider linear dynamic range, which enables the determination of trace and major analytes in one sample run, minimizing the need of re-analysis of the sample after further dilution.
- The iCAP PRO Series ICP-OES with a vertical torch for both duo and radial configuration and its resulting system robustness offers great flexibility for analysis of a variety of challenging matrices, such as samples with high total dissolved solid and volatile and non-volatile organic solvents with great ease.
- The Qtegra ISDS Software with ease of use, comprehensive set of QC functions, flexible options for data exporting and reporting, limits, and flag functionality makes day-to-day analytical operation quick and easy, improving the productivity of the laboratory.

References

1. https://www.eag.com/wp-content/uploads/2020/06/M-050620-Purity-Brochure_sp.pdf
2. Application Note 44470 – High matrix tolerance of the Thermo Scientific iCAP PRO XP ICP-OES Radial and Duo equipped with a ceramic torch and sheath gas adapter. https://assets.thermofisher.com/TFS-Assets/CMD/Application-Notes/an-44470-icp-oes-metals-brine-ceramic-torch-sheath-an44470-en.pdf
3. www.thermofisher.com/ICP-OES

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