Scope For Elemental Analysis And New Academia Performance

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Abstract - **Spectrometers that identify and measure the elemental composition of samples have long been key tools in university and other academic laboratories. However, as academic needs change and requirements increase, some users have felt constrained by the performance of older spectrometers. These demanding users need truly future-proof instruments that can satisfy their analytical needs today and tomorrow.**

I. INTRODUCTION

This paper will discuss recent advances in both ICP-OES and ED-XRF technology that have resulted in a unique class of instrumentation — one that brings academic researchers a new level of uncompromising performance, scope, and flexibility. Included will be hardware advances that have increased the capabilities of these technologies across a wider range of elements, concentration ranges, and applications. So academic users can now truly future-proof their research requirements. The trouble with specialization Elemental analysis instruments are often optimized with certain discrete applications in mind.

For example, many analysers employ inductively coupled plasma optical emission spectrometry (ICP-OES) technology. However, as these popular instruments have developed over the years, manufacturers have tended to equip most models with one selected plasma viewing technique (radial and axial are the two main options). Each option trades off certain performance characteristics in favour of others. Unfortunately, this results in an inherently compromised system [1-5] one best suited for certain applications and not others, and thus unduly specialized for a specific set of tasks.

Another widely used technology class, energy-dispersive X-ray fluorescence (ED-XRF) spectrometry, often suffers similar issues. A given analysers excitation parameters may be selected and optimized for the analysis of specific elements in specific matrices. Results a compromised system design with limited excitation parameter flexibility, so that the system determines the concentrations of certain elements in certain matrices preferentially over others. Of course, this kind of analytical specialization may be suitable for many types of industrial applications. It can also be a good fit for certain specialized academic laboratories. For example, a university lab that concentrates on routine environmental analyses — testing samples of soil, water, and wastewater for given sets of nutrients or toxic metals — will often find its present and anticipated needs met with a good midrange ICP-OES spectrometer (such as the SPECTROGREEN from SPECTRO Analytical Instruments).

However, high-end academic laboratories increasingly require even more flexible, high-performance analytical capabilities.

II. EVOLVING RESEARCH REQUIREMENTS

For universities and institutes at the forefront of research, several factors drive the need for maximum flexibility in the performance and scope of their elemental analysis instrumentation. A number of leading academic institutions have centralized their analytical equipment within analytical chemistry centers. These central laboratories offer services for the diverse requirements of a variety of departments across the campus. Each day may bring the need to analyze samples of anything from soils and waters to ceramics or metals.

Another factor: changing avenues of investigation. In any academic term, professors may receive grants for new studies that require analyzing different elements, perhaps at new (usually lower) levels of concentration.

Buying cycles plus grant and budgeting processes bring added pressures. Older instruments may not be able to handle current or anticipated tasks, necessitating upgrading or replacement. And with equipment funding usually determined on a yearly basis, if not even longer-term, academic laboratories must carefully plan ahead to make sure they can meet both current and future analytical needs.

The nature of certain scientific specialties also maximizes analytical requirements. For example, in the earth sciences or subspecialties such as biogeochemistry, samples are often highly varied. Complex matrices a challenging puzzle for many spectrometers can be commonplace and analysers may encounter high concentration levels in one sample, while facing the need for trace element determination in the next.

Finally, student education forms part of many laboratories' missions. The obligation to train young researchers on up-to-date technology is taken seriously even at many research-intensive institutions.
Innovative solutions for academia's unique needs fortunately, it's easy to see that this is a new golden age of elemental analytical power for academic research.

Instead of the compromised, overspecialized ICP-OES and ED-XRF instruments that many university labs have applied in the past, a new class of instruments has improved on these technologies to eliminate most of their constraints. Probably the best examples: the SPECTRO ARCOS MV ICP-OES and SPECTRO XEPOS HE ED-XRF analysers.

Both offer unique new performance and flexibility that's already been successfully applied by a wide range of university and institutional users. These spectrometers are becoming the ICP and XRF instruments of choice for academic research worldwide.

ICP-OES — SPECTRO ARCOS MV

Background: the importance of plasma viewing choices the optical system in a radial-view spectrometer observes a cross-section of light (emitted by samples excited in the plasma) from the side. This is opposed to an axial-view system, where the plasma is observed from end to end. In recent years, a demanding group of users in industry and academia users who wished to perform elemental analysis of metals and other materials with high precision, high stability, and high matrix compatibility have purchased radial- view ICP-OES analysers. These users have included a key group of high-end academic research institutions.

They have favoured radial versus axial plasma observation due to radial benefits such as the following:

- Larger linear range
- Higher matrix compatibility (handles up to 20% total dissolved solids (TDS))
- Fewer matrix effects
- Easier handling
- Better stability

However, this has meant forgoing axial plasma observation characteristics such as these:

- Lowest detection limits
- Highest sensitivity for trace analysis

A main consideration: the vulnerability of axial-based systems to drawbacks such as recombination effects, influences from the plasma load, self-absorption, and the easily ionizable element (EIE) effect. Some spectrometers have tried to combine the benefits of both viewing techniques, offering systems with some variation of dual- view or twin-interface technology. However, each of these must compromise one view or the other. Their designs inevitably favour either radial or axial observation with a direct view, while adding an indirect view for the other mode via a small periscope optic.

Seeking a truly versatile capability, SPECTRO developed the Multi View option for its high- end SPECTRO ARCOS ICP-OES the latest in the SPECTRO ARCOS line that has led the industry for more than 13 years. The design eliminates added mirrors or periscopes that would degrade sensitivity via light loss. A simple mechanical adjustment lets the researcher literally "turn" the instrument from one view to another, in 90 seconds or less. So users can choose true radial view or true axial view, without compromise and without buying two separate instruments to suit the sample being analyzed. This unique solution has already proven to produce successful results for researchers in high-end academic labs across the globe.

ICP-OES: unique technology

The SPECTRO ARCOS MV analyser delivers peak performance furnishing the industry's highest optical resolution over its wide spectral range (130-770 nm), plus stellar sensitivity. And it provides the highest dynamic range on the market, with a readout system capable of acquiring signals up to 1.2 billion counts per second. All this performance is paired with outstanding throughput and productivity. Much of this superior performance arises from exclusive technologies [6-7]. In addition to the no-compromises Multi View plasma observation system mentioned above, SPECTRO ARCOS MV provides no-purge UV-PLUS sealed gas purification and a no-external-cooling OPI-AIR interface. And its ultra-stable, 2000-watt (W) LDMOS generator provides extreme power reserves to handle even fast-changing/extreme plasma loads. Taken together, all these innovations help make SPECTRO ARCOS MV probably the most versatile ICP-OES analyser available.

ICP-OES: exclusive flexibility

The Multi view component of SPECTRO ARCOS MV demonstrates that future- proof flexibility is at the heart of its design. Offering both true radial and true axial plasma observation on a high-end ICP- OES instrument, it provides the utmost in wavelength range; optical resolution, precision, and sensitivity; ultra-low limits of detection (LODs); ensured stability; and outstanding matrix compatibility. Yet the system can complete a single analysis in as little as 20 seconds — and is able to capture a complete spectrum in less than 3 seconds.

When researchers' duties extend into instruction, SPECTRO ARCOS MV has the flexibility to function as an advanced teaching tool. Its ORCA Paschen-Runge optical design, offering simultaneous capture of the complete spectrum with every measurement, provides the opportunity to easily educate students about emission spectroscopic analytical concepts, and even allows the use of hyphenated techniques such as laser ablation, IC/LC coupling, ETV, or DC Arc that require the acquisition of transient signals. Its Multi View technology also presents the clearest possible illustration of the characteristics of radial- based plasma observation versus axial- based, and, combined with the high-power, free-running LDMOS generator, allows the study of plasma effects.

ICP-OES: myriad applications

It's no surprise that what may be the most versatile ICP-OES analyser on the market lends itself to a wide variety of academic applications. It should furnish excellent results for researchers' needs, today and tomorrow.

Providing the highest continuous optical resolution over a wide spectral range allows SPECTRO ARCOS MV to easily separate neighbouring lines, minimize spectral interferences, simplify method development, and improve accuracy with line-rich matrices. Its direct light- paths and optimized transfer optics, as well as its hermetically sealed and fully environmentally controlled UV-Plus optic, provide high transparency and offer the best sensitivity in its class, especially in the VUV/ UV range enabling parts-per-trillion (ppt) to sub-parts-per-billion (sub-ppb) detection for many applications. So it performs very well in any kind of material evaluation, from trace elements in line-rich material to high-precision analysis of major components.

The SPECTRO ARCOS MV's direct light- path axial plasma observation mode, with its unique OPI interface, is ideal when the highest sensitivity plus a large dynamic range are needed. Examples: elemental analyses for any kind of environmental work, and/or the measurement of elements from ppb to major-component percentage levels in chemicals, ceramics, or metals.

In turn, the unit's radial observation mode, with its freedom from plasma and matrix effects, is the choice for any higher-matrix or organic application. That includes elemental analysis of any petrochemical material including volatile organics, as well as trace element analysis in high-salt matrices. The optimized light-path still provides high sensitivity with even higher precision and stability. When stability is an especially key factor (for example, with high-volume / high-throughput applications such as soils analysis), applying specialized techniques like bracketing can achieve relative standard deviations (RSDs) well below 0.1%. This makes SPECTRO ARCOS MV the perfect tool for applications where major compounds require highly accurate analysis (e.g., precious metals), or where exact concentrations ratios are important (e.g., battery materials).

High plasma loads are particularly enabled by the SPECTRO ARCOS MV's 27 MHz free- running LDMOS generator. With up to 2000 W, it's one of the highest-powered systems available. It provides extreme agility, requires no mechanical match work, and offers the same characteristics as a free- running tube system. The magnesium (Mg) ratio test measurements demonstrate this benefit impressively. Whether plain water or even 50% isopropyl alcohol solution is introduced into the plasma, the Mg ratio remains almost constant suggesting that the same amount of energy is transferred into the plasma regardless of load.

Finally, unlike many other spectrometers, the instrument can achieve excellent results for the difficult-to-analyze halogen elements especially chlorine (Cl), iodine (I), and bromine (Br), which are important for environmental, petrochemical, and agronomy analyses.

On the SPECTRO ARCOS MV, its UV-PLUS sealed gas purification system avoids the need to constantly purge the optical system with costly argon (Ar) gas. (Savings: up to $3800 per year in gas consumables.) Also, the fully air-cooled design eliminates expensive, breakdown-prone external water chillers. (Savings: up to $5,000 versus separate system, leaks, and maintenance.) ICP-OES: welcome ease of use Advanced software provides academic users with short learning curves, streamlined workflows, simplified method development, accelerated data handling (up to 1500x faster than previous models), and high productivity. Meeting researchers absolute need for reliable record-keeping of data and results, SPECTRO ARCOS MV with ICP Analyser Pro software supplies full forensic capabilities. That includes powerful version management and audit trail functions, covering every event and change for solid security and total traceability. Full spectrum storage with every measurement plus advanced editing and recalculation capabilities enable post-analytical evaluation of data, even long after a given analysis occurs. This allows the evaluation of data elements even outside the initial scope of the analysis.

Academic users also appreciate ease of use that extends to backup by a responsive, reliable service organization. For example, SPECTRO ARCOS MV is supported by SPECTRO's AMECARE performance service. So researchers get efficient installation and training; the ability to enhance user skills by providing access to expert advice; and timely support in the rare case that a service issue arises.

ED-XRF SPECTRO XEPOS HE[8-11].

In the past, researchers with elemental analysis applications suited to XRF would often choose wavelength-dispersive X-ray fluorescence (WD-XRF) analysers. On the downside, these instruments were typically twice as costly as their ED-XRF counterparts. However, they frankly outmatched ED-XRF models for important performance metrics: including lower LODs, greater resolution, and better precision, as well as higher sample throughput rates and shorter measurement times. For many applications, that's no longer the case. ED-XRF technology has been continuously refined and greatly improved in recent years. So an advanced ED-XRF spectrometer such as SPECTRO XEPOS HE though still significantly less costly for both purchase price and long-term expenses than any WD-XRF model can now provide comparable performance in many academic applications. In fact, for some trace-level analyses, SPECTRO XEPOS HE achieves even lower limits of detection than typical WD-XRF instruments.

ED-XRF: unique technology

SPECTRO XEPOS HE is perhaps the industry's most powerful ED-XRF spectrometer. Its recent technological improvements include innovative high- count detector, readout, and X-ray tube designs. These deliver unique new adaptive excitation, as well as an optimized combination of a thick binary palladium/ cobalt (Pd/Co) alloy anode X-ray tube with direct excitation, excitation via a bandpass filter, and polarized excitation all to provide optimized performance for different groups of elements. (It's like having three tubes in one instrument)

Results up to 10x greater sensitivity and up to 3x better precision than previous models. And the analysers long-term stability now matches that of WD-XRF models. These qualities are critical for multi-element analyses of major, minor, and trace element concentrations. For elements commonly found in environmental, geological, food, pharmaceutical, or certain other samples, researchers can choose one of SPECTRO's precalibrated application packages. On the other hand, users may encounter completely unknown samples in liquid, solid, or powder form, and need to perform a quick elemental screening. For this and many other applications, the instrument's default TURBOQUANT II screening method can be utilized to obtain excellent analysis results.

ED-XRF: unprecedented flexibility

The great flexibility of SPECTRO XEPOS HE enables coverage of a wide array of elements, storing full spectra with every measurement. So researchers get fast, accurate analysis of many more elements than previously possible. The analyser covers the range from carbon (C) to uranium (U) for rapid qualitative screening analysis, and can deliver quantified results from fluorine (F) to uranium(U) at lower levels and with better precision than previous generations of instruments. Students and researchers can experiment: operating the instrument with different types of excitation to get the best sensitivity for the elements of interest. They can also discover the unique benefits of full-spectrum measurement.

SPECTRO XEPOS HE flexibility extends beyond just the widest possible spectrum. Example: researchers can choose to optimize either precision or speed. For challenging measurement, they may select the analysers highest sensitivity. This lets them determine both minor and major elemental concentrations with great precision, taking advantage of exceptionally low detection limits (often less than 1 part per million (ppm)) for critical trace amounts including analysis of heavy elements such as cadmium (Cd). Or, if a busy lab schedule stacks up multiple routine analyses to run, they can prioritize speed — accepting slightly less precision to achieve dramatically reduced measurement times, such as cutting analysis for many samples from 6 minutes to 60 seconds.

Finally, the unit offers excellent flexibility of sample handling for the diverse requirements of many academic labs. A spacious measurement compartment and multiposition auto sampler allow analysis of larger and/or irregularly shaped samples. An optional helium (He) purge permits analysis of light elements in liquids and powders. Users may also use the vacuum mode for analysis of elements with low atomic numbers in pressed powder pellets, fused beads, or solid samples.

ED-XRF: a range of applications Versatility and flexibility are hallmarks of this latest class of instruments. So an advanced ED-XRF analyser [12] will be designed to give excellent results in a wide array of typical academic applications.

One example: the bulk analysis of peat samples for hydrogeology research. Taken in particular from peat lands that receive liquid inputs only from precipitation, these can provide valuable information on retention of nutrient and trace elements, as well as deposition history. The SPECTRO XEPOS HE analyser demonstrates very good correlations for minor and trace element concentrations in these samples even at very low sample masses (down to approximately 500 milligrams). And measurements are non-destructive, so samples are available for additional testing using other techniques.

Another illustrative case: the determination of nutrients, trace elements, and heavy metals in plant tissue - an important nutritional source for humans and animals alike. SPECTRO XEPOS HE shows very good correlations for minor and trace element concentrations in plant materials, with tight agreement between certified and analyzed concentrations. Minimal sample preparation is required. Once again, the testing is non-destructive.

ED-XRF: lower cost of ownership

This unique class of spectrometers is designed with numerous cost-saving features — a welcome advantage given most academic labs' budget constraints. For example, the affordable SPECTRO XEPOS HE analyser exhibits significantly lower investment and ownership costs than a typical high-powered WD-XRF model. It offers lower power consumption (overall only 200 W). Its components are selected to achieve long life cycles. And when components finally must be replaced, they're often less expensive: a new tube for SPECTRO XEPOS HE is around one-third the cost of a high-powered WD-XRF tube. Bottom line: performance similar to a WD- XRF in many academic applications — for the purchase price of a bench top ED-XRF.

ED-XRF: excellent ease of use

An instrument like SPECTRO XEPOS HE is the product of years spent refining every aspect of the user experience. It suits the needs of academic labs at every phase of operation. Its workflow provides proven, effective procedures for achieving accurate results in minimum time, in a high-productivity research environment. For accurate evaluation of a new series of samples, users first calibrate the instrument, test the precision of the analysis and the unit's limits of detection, and validate its measurement against known reference samples. The analyser operating software interface has been redesigned and optimized using user input and testing to be powerful, intuitive, and exceptionally easy to learn and use.

When high-accuracy, quantified analysis is necessary, XRF often requires sample preparation. (For questions about selecting a suitable prep method, users can consult the experienced SPECTRO application team.) But in many cases, for a quick screening analysis, sample preparation is minimal. Users just place the sample into the measurement compartment and run the screening test.

Simply put: a student or researcher doesn't have to be a spectrometry expert to get good analysis results in a reasonable time with SPECTRO XEPOS HE. Academic users also appreciate ease of use that extends to backup by a responsive, reliable service organization. As with SPECTRO ARCOS MV, SPECTRO XEPOS HE is supported by SPECTRO's AMECARE performance services. So users get efficient training and installation; the ability to enhance user skills by providing access to expert advice plus advanced product and application tools; intuitive software; and timely support in the rare case that a service issue arises.

III. CONCLUSION

Top-tier academic research laboratories must conduct elemental analysis of a wide array of often-difficult materials. When selecting the correct spectrometer, lab planners must carefully weigh key characteristics such as technology, flexibility, applications range, costs, and ease of use. To add a final challenge, their choices must reflect both their current and future needs for analytical performance and scope. Fortunately, recent developments have given rise to a unique new generation of ICP-OES and ED-XRF analysers that can help academic researchers future-proof their analysis capabilities.

IV. REFERENCES

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