

Total Multi-line Concept

A revolutionary approach : from single-line to multi-line analysis .

The classical way of conducting the determination of an element in ICP-OES is to select a single line with adequate sensitivity and free from spectral interferences. If the nature and the concentration of the matrix elements are not constant, there is a risk of unexpected spectral interferences, which can become significant, and the concentration deduced from the use of a single line may then be wrong, with no means of detecting the bias.

When the analyte peak is interfered, a positive bias will be generated, while the bias may be negative when the matrix influences the background correction.

The ideal approach is to conduct multi-line analysis, a far more efficient way of taking benefit from all the available information of the plasma. With the use of multi-channel detection such as CCD detectors, the amount of spectral information has significantly increased and it is then possible to have acquisition of the full UV-visible spectrum for each element, and to use multiple lines per element.

Multi-line analysis makes the detection of outliers possible, thereby coping with unexpected spectral interferences and, consequently, improving the reliability of the results.

Because method development and its validation may be a complex task, performing multi-line analysis implies :







Bring ICP expertise to your lab with an easy-to-use instrument!

Be confident in the quality of your results!

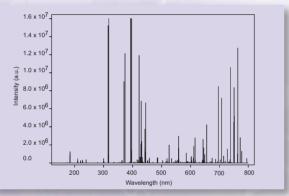
Innovative Interactive Assistance

Unique and proprietary S3-base

S³-base (Single-element Spectra, Spectroscopic data) is essential to take full benefit of multi-line analysis. Such a base responds to the need of reliability in terms of wavelength compilation and line intensity, because currently available wavelength tables are not based upon ICP experiments, but on alternative radiation sources such as arc or spark. As a consequence, ICP users must perform tedious experiments for line selection by overlapping spectra of the different elements expected in the sample, to find a sensitive line free from spectral interferences. When the multi-line concept is used, method development becomes even more complex.

Thanks to the specific instrumental configuration of ACTIVA M, single-element, entire spectrum acquisitions were performed under standard ICP operating conditions.

This allowed the assignment of ICP lines and the calculation of spectroscopic data, which led to the creation of a base with double access: a collection of single-element spectra and a data base containing wavelengths, ionisation state, excitation energy, sensitivity (S), background level, limit of detection (LOD), maximum concentration below detector saturation (C_{max}) and line width (FWHM).



Example of single-element spectrum (Ca 1 g/l)

More than 50 000 lines have been, then, assigned, with the corresponding spectroscopic data varying for example from 60 lines for Pb, 183 for Ca, ~3000 for Cr to ~5200 lines for Th.

S³-base provides appropriate spectroscopic data information for multi-line selection with MASTER.

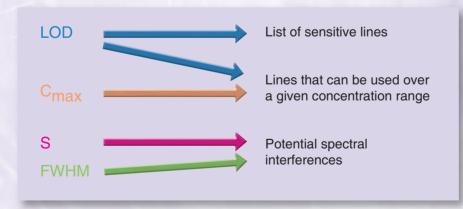
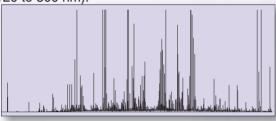




IMAGE.

Before developing the method, it might be necessary to identify the elements and estimate their concentration range. Semi-quantitative analysis is performed with IMAGE by using multiple lines, based on the acquisition of the complete spectrum

(120 to 800 nm).



Full spectrum acquisition (120-800 nm)

Spectra are stored for recalculation at any time for any element or line. All peaks can be visualised for confirmation. Real sample spectra can then be transported to MASTER, providing full interactivity between IMAGE and MASTER.

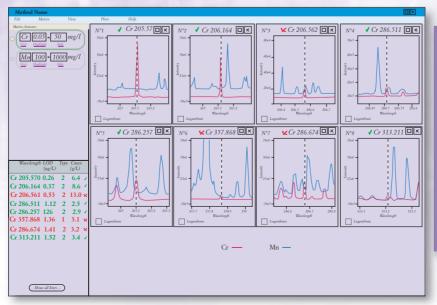


IMAGE provides a fingerprint of the samples and ensures traceability with possible re-processing

MASTER (Multi-line Analysis, Selection Tool for Enhanced Reliability) ensures the multi-line selection for each application. Thanks to S³-base, the line selection is facilitated through the automatic Filtering and the Display procedures.

Basically, the analysts just have to specify the list of elements and their concentration range. Based on the S3-base spectroscopic data, MASTER suggests lines for each analyte that are adequate for the defined concentration range and that are not interfered by the concomitant elements when present at their highest expected concentration, to be under the most pessimistic case. The number of lines to be suggested, as well as sensitivity and interference filtering criteria, are user-defined. As an example, for a concentration range 0.05 – 50 mg/L of Cr as the analyte, in the presence of Mn in the range 10 - 500 mg/L, the filtering procedure suggests at least 8 appropriate Cr lines.

Then, MASTER displays the analyte spectrum along with those of concomitant elements and the blank spectrum. This helps in the ultimate validation or rejection of a line, and to determine the correct background correction positions.



Lines and backgrounds of all elements are then exported, for an automatic set-up of the analytical method for analysis. Thanks to the proprietary S³-base, tedious line profiling optimisation is no longer needed.

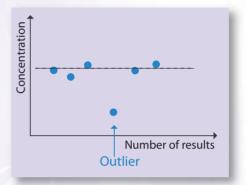
CALSTAT

The development of the method is completed afterwards with a statistical evaluation of the calibration procedure. Once the calibration is realised, automatic calculations in the CALSTAT tool helps the analyst to evaluate the following criteria: verification of linearity, estimation of uncertainty due to the

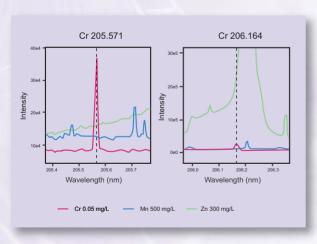
calibration procedure, determination of limits of quantitation, validation of background correction, and advice for weighting the regression.

SOS

SOS (Statistical Outlier Survey) is a statistical process of the multi-line results, for the rejection of possible outliers, to provide a single reliable element concentration. SOS copes with both positive and negative bias, and avoids recalculating the results.



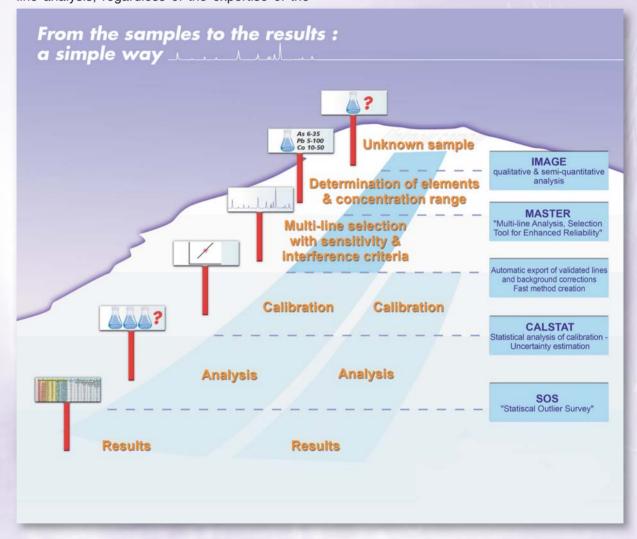
In our example, if Zn is present in one sample and was not expected, bias will occur: the right background of chromium 205.571 nm line is slightly increased and the 206.164 nm line is interfered. Both lines are rejected and three remain for the reliable calculation of Cr concentration.





SOS helps the analyst to optimise the method, as well as to perform routine analysis with security, by detecting outliers if unexpected interferences occur.

As a whole, the strong interaction between instrumental configuration and software tools is the essence of this revolutionary approach. ACTIVA M offers an incomparable and practical use of multiline analysis, regardless of the expertise of the user. The instrument is used at its best through time saving and user-friendly step-by-step operations. Everyone can easily become an expert in ICP analysis.





The Art of CCD-based ICP-OES

ACTIVAnalyst software, to operate the instrument at its best

Available in multi-language versions, the software provides a practical use for first-time operators, as well as optimisation capabilities for trained users.

Some software functions are keys to a secure multiuser operation of the instrument: on-line laboratory notebook; multi-user and multi-level security (biometric signature log-on available); computer programmable plasma conditions by method.

First-time users quickly become confident through assistance functions along the analytical methodology process: IMAGE for automatic semi-quantitative analysis; MASTER for lines selection; a Database of matrices, wavelengths and spectroscopic data, quality controls, sample tolerance tables and standards; optimization of integration time for each element based on sensitivity, speed and precision criteria from user's input; SOS for evaluation of results.

High sample throughput is achieved with the autosampler (benefiting from bar code identification of samples) and with automated analysis using a multimethod sequence for samples of various matrices requiring different methods. Programmable calibration re-sloping and QC checks, with user-defined actions, ensure the automatic sequence processing, and the SOS tool guaranties the quality of results. The ICP system offers increased productivity through automatic user programmable start up and shutdown. This also minimizes argon gas consumption.

Urgent samples can be easily inserted at any time in a sequence in-run, using either the current method or other methods.

Analytical criteria (accuracy, repeatability...) can be optimised if required using various correction methods: controlled Standard Addition Method; automatic calculation of additive and multiplicative

Inter-Element Correction factors; Internal Standard correction; auto-dilution process; drift correction & high precision data processing; results reprocessing after calibration or parameters changes.

The simultaneous measurement allows visualising the spectra of lines and their vicinity during analysis for confirmation or identification of unexpected spectral interferences.

Results are fully utilised through:

- Data transfer and import/export of sample information compatible with LIMS or third party software
- Results exporting and/or printing in real time, through batch reprocessing or on a sample-bysample bais
- Wide range of industry compliant reports, as well as additional reports such as stability report
- Full data archiving capabilities

Diagnostics of the instrument can be performed in different ways:

- TimeScan, transient signal acquisition (real-time output of instrument signal for troubleshooting or optimisation)
- Automatic export and calculation of parameters from Figures of Merit measurements
- Remote control and PlasmaCam remote plasma viewing for Service assistance
- SPC (Statistical Process Control) for X, R, SD and histograms charts of user selected solutions

The ACTIVAnalyst software is FDA 21 CFR Part 11 compliant, with electronic signature of a dedicated results report.

Expanding your capabilities !.

While the S³-base and assistant software tools make multi-line analysis a reality, without the advanced detector technology, optics and torch system of ACTIVA M, the assistance would be more limited in capability.

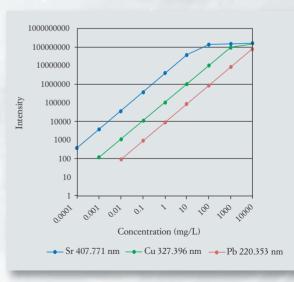
ACTIVA M maintains the strengths of HORIBA Scientific ICP spectrometers, while incorporating advanced CCD technology.



The Czerny-Turner optical system is equipped with a megapixel, unequalled low-noise CCD detector, leading to spectral windows up to 8 or 16 nm with simultaneous measurement of multiple lines and backgrounds.

Based on dual large back-to-back gratings, the resolution is, then, constant not only within a window but also over the spectral range. It is better than 10 pm with the 4343 grooves/mm holographic grating (in the range 120-430 nm), and better than 18 pm with the 2400 grooves/mm grating.

Considering the ACTIVA M low-noise, high-speed CCD detector, combined with possible exposure times in the range 1:1000, a practical dynamic range (signal before saturation-to-readout noise ratio) of 10⁷ can be achieved. Such a dynamic range is usually higher than the ratio of the maximum concentration to the limit of quantitation, with the maximum concentration corresponding to the beginning of graph curvature.



Calibration graphs: linear response range of 5 decades minimum (first point of each calibration graph corresponds to the limit of quantitation of the element)

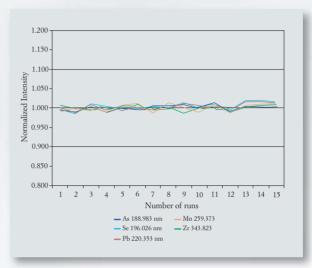
The addition of adjacent spectral windows allows the system to cover the entire 120-800 nm range: each entire spectrum is stored, allowing recalculation at any time on any element.

This configuration offers:

- Minimised spectral interferences, thanks to high and constant resolution
- Excellent limits of detection, regardless of the matrix complexity, thanks to high light throughput
- Ability to determine halogens in the far UV

The unique megapixel CCD based-camera allows observation of the Normal Analytical Zone of the plasma. This vertical torch-based Total Plasma View provides instrumental limits of detection comparable to axial or dual view systems, but is superior in terms of:

- •Warm-up time of typically 15 min (computer controlled automatic solid-state RF generator running at 40.68 MHz)
- Low argon consumption (12 L/min) and a quickmount demountable torch with less parts replacement
- Perfect reproducibility in torch positioning and stored operating parameters, facilitating multi-user operation
- Long-term stability even for difficult matrices, offered by the configuration of the torch associated.
- •Minimised matrix effects and few memory effects
- Allowing complex matrix, high dissolved solids or organic sample analysis (thanks to the large internal diameter, 3 mm, of the injector and to the unique sheath gas device)
- Providing better limits of detection
- Linear concentration range over a minimum of five decades
- High sample throughput with minimal re-sloping effort
- Incomparable ICP-OES performance for alkaline elements



Long-term stability for 1 mg/L of elements in 30% NaCl sample

(average RSD < 1 % over 3 hours without re-sloping or internal standard correction)



A Platform of support

HORIBA Scientific, the solution for inorganic analysis.

Besides our ACTIVA M, HORIBA Scientific proposes a full range of ICP-OES instruments, offering the best configuration suitable to your requirements.



HORIBA

ACTIVA family and ULTIMA 2 family provide a large choice of solutions for a laboratory.

ACTIVA S combines simplicity of operation together with robustness and stability. Based on CCD detection, it offers performance and flexibility.

ACTIVA M expands the application capabilities through assistant tools and enables the analyst to easily become an expert, regardless of the complexity of the application. High quality of results is achieved, ensuring optimum routine analysis conditions. Alternative wavelengths and halogens are measurable thanks to far UV capability.

ULTIMA 2 features the lowest detection limits available today, with single view radial plasma and the best optical resolution. Adding a direct viewed simultaneous optical system to this model (ULTIMA 2C and ULTIMA 2CHR) provides a complete solution for a laboratory with the speed and flexibility required for many applications where the need for a fast analysis time must not compromise instrument performance.



The simultaneous system also achieves excellent precision on major elemental determinations, and with ULTIMA 2CHR, optimum analytical performance is gained for the most complex materials.



HORIBA Scientific is completing the offer in ICP analysis with 2 separate ranges of inorganic standards, SPEX Certiprep (triple checked) standards and Précis standards. One can use different lots supplied by 2 sources for calibration and validation, as recommended by Good Laboratory Practices, ordered from a single vendor.

ICP spectroscopy is also used for WEEE/RoHS/ELV compliance testing when XRF screening indicates that a hazardous substance is present at or above the limit, or when the detection capability of XRF is not sufficient to detect the low level of these substances.

The XGT-WR series, EDXRF analytical Imaging Microscope, also offered by HORIBA Scientific, provides very good sensitivity for compliance screening and is complementary to the ICP performance.



HORIBA Scientific is also a major supplier for systems dedicated to Solid analysis. GDS (Glow Discharge Spectrometry, based on optical emission) is the ideal technique for both bulk analysis in solids (conductive or not) and depth profiling for layer characterisation of coatings or surface treatments.



For elemental analysis, HORIBA Scientific gives you the opportunity to choose the instrument that fits your needs for Carbon/Sulfur analysis (EMIA series) or Oxygen/Nitrogen/Hydrogen analysis (EMGA series). From ppm to 100%, C/S and O/N/H are analyzed in solids or powders, as a

complement to ICP-OES, for applications like steel, pure and precious metals, cement, alloys, soil... There is also the SLFA series to determine Sulfur from 0.03 ppm up to 10% in a wide variety of petroleum based products.

HORIBA Scientific, a broad range of support

Our commitment to support our users is expanded to the offer of both analytical and technical assistance directly or through our global network of representatives. A wide range of documents is available, including user and technical manuals as well as application and technical notes, for your support.

Training (on-site or at HORIBA Scientific application laboratories) and analytical assistance give the operators the help and knowledge to perform analysis under optimum conditions.

Radio frequency, solid-state 40.68 MHz, water cooled

Cooling system

GenCo water chiller for the generator

and the coil

Exhaust

Direct exhaust connection for

plasma compartment

Plasma

Fully demountable torch, 3 mm i.d., alumina injector, 12 L/min plasma

gas, 0.2 L/min sheath gas

Sample introduction

Concentric glass nebulizer, cyclonic glass spray chamber, 3 channels

peristaltic pump

Optical system

Thermo-regulated, 0.64 meter focal length, dual back-to-back gratings, 4343 g/mm and 2400 g/mm used in the 1st order with optical resolution <10 pm for 120-430 nm and <18 pm

for 430-800 nm 120-800 nm

Wavelength range Detection

Back thinned illuminated CCD,

2048x512 pixels, pixel size 13.5x13.5 μm,

Peltier cooled

AS-500 Autosampler with rinse station

Ar humidifier

Micro / high dissolved solids / inert material / organic

nebulizers

Scott / HF resistant / cooled spray chambers

Ultrasonic nebulizer

CMA for simultaneous measurement of hydride

forming elements and other elements

Oxygen kit for organic samples

Laser ablation system

PHYSICAL DATA

Refer to ACTIVA M Pre-Installation Guide for more details.

Depth

698 mm (27.5 in)

Width

1321 mm (52 in)

Height

604 mm (23.8 in)

Weight

195 kg (430 lb)

Power

Single-phase, 220-240 V, 50/60 Hz, 4 kVA

Cooling water

2 to 3 L/min, 2 bar

Argon

99.995% purity

....

160 to 190 nm, 99.999% purity

Nitrogen

120 to 160 nm, 99.9995% purity

Exhaust

250 m³/h (150 cfm)

Environmental

20 to 80% humidity non-condensing,

18-24°C at ± 2°C

www.horiba.com/scientific

JOBIN YVON Technology

Specifications subject to change without notice.



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