SmartNotes

What are the benefits and considerations of upgrading to ICP-MS from GF-AA?

I have been a routine user of graphite furnace atomic absorption (GF-AA) to analyse my samples for many years. However my analysis requirements are changing and my instrument is old and needs to be replaced.

Should I purchase another GF-AA?

If you are already familiar with the technique having worked with it for a number of years, it might be an easy step to take. However, this means that you know the limitations of the technique on your work.

Although modern AA instruments have become more automated and easier to use you would still be constrained by the limitations of the basic technique. These include issues such as slow overall analysis times as it is a single element technique, the need for multiple hollow cathode lamps to cover all the elements you want to measure increasing cost, long analysis times due to the need for complex furnace temperature programs to remove the sample matrix before atomization, and also the need to add matrix modifiers to overcome chemical interference which complicates the analysis and increases the risk of contamination.

There would be a number of significant advantages for you to be gained by moving to a more powerful technique such as Inductively Coupled Plasma – Mass Spectrometer (ICP-MS).

Figure 1. Thermo Scientific ICAP RQ ICP-MS.

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Does ICP-MS need a special environment for installation?

It is true that if you are working with a semi-conductor application the instrument will ideally need to be located in a clean room. However, for routine applications such as environmental or pharmaceutical analyses the instrument can be installed in most normal laboratory situations provided they are of a reasonable standard and meet the installation requirements.

To reach the full capability of the instrument it will be necessary to use high quality reagents and make sure that any sample vessels are properly cleaned before use but these precautions are of a similar level to what you need to do now for your graphite furnace analyses.

The Thermo Scientific™ iCAP™ RQ ICP-MS has been designed to simplify installation and operation. It is a bench top instrument which results in a very compact and narrow design. It is likely occupy less bench space than your current graphite furnace instrument.

All service connections to the iCAP RQ ICP-MS are made on the left hand side of the instrument whilst all sample introduction connections are made on the right side. This means that there are no connections on the rear of the instrument and it can be positioned directly against a wall further simplifying installation.

Is ICP-MS very expensive to operate?

Your analysis cost using Graphite Furnace AA may appear quite small since the gas consumption is low, however when the cost of graphite cuvettes, contact cones and operator time is taken into account it is actually quite significant. This is especially true when calculated on a cost per sample basis since the analysis times are relatively long.

Although the analysis cost of ICP-MS will be higher this needs to be balanced against the benefits of a more powerful analysis technique.

It is a multi-element technique so overall analysis times will be very much reduced compared to Graphite Furnace AA. In addition, your entire suite of analysis elements can be measured with a single instrument rather than multiple techniques as you do currently. Elements such as Al, Si, Ti, V and B which are problematic with Graphite Furnace AA due to carbide formation have very good performance with ICP-MS.

ICP-MS also has significantly lower detection limits and the ability to carry out more detailed analysis such as isotope ratio or speciation. These benefits may be very useful for you in the future if legislation changes or your analysis requirements expand thereby effectively future proofing your laboratory.

Is ICP-MS difficult to use?

The iCAP RQ ICP-MS is the latest generation ICP-MS instrument and incorporates a number of both hardware and software innovations that greatly simplify setup and operation for the user.

Graphite Furnace AA is a deceptively complex technique to use and suffers from a number of complex vapour phase interference effects. Method development needs careful optimization of the temperature program, choice of matrix modifier and background correction to produce valid data.

By contrast the iCAP RQ ICP-MS has been designed with the user in mind. The sample introduction assembly is positioned at bench height for easy access and uses quick connect mechanisms without complicated connectors or screws. The high performance interface has been designed to operate at the optimum temperature minimizing sample deposition and reducing the need for user maintenance. The instrument also features a unique QCell technology with helium Kinetic Energy Discrimination (KED) measurement mode for effective and comprehensive interference removal.

Operation of the iCAP RQ ICP-MS is via the intuitive and easy to use Thermo Scientific Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software. A single button start up procedure is used to check instrument performance and if necessary optimize instrument parameters before the analysis proceeds.

Analyses are defined using a series of Thermo Scientific Qtegra ISDS Software LabBooks which contain details of samples to be measured along with calibration information, quality control checks and instrument parameters to be used for the measurements.

How easy is it to set up a method for ICP-MS?

The Qtegra ISDS Software makes method development extremely easy with the iCAP RQ ICP-MS.

It uses a simple and intuitive workbook based approach where all elements, parameters and samples to be measured are stored in a single LabBook for easy review later.

The LabBook can be quickly and easily built up from a series of supplied templates or alternatively a template can be created from a previous analysis or a new blank template can be created. The template contains elements to be analyzed, instrument measurement mode and parameters, calibration information and quality control information. The template approach minimizes the chance of mistakes and ensures that consistent analyses are carried out day by day.

The number of samples to be measured is then selected and the LabBook will be created automatically and the analysis is ready to run.

LabBooks can be run immediately or a series of LabBooks can be queued in the Scheduler which enables long fully automated analyses with differing requirements to take place.

When the analysis has been completed results can be reviewed easily on the screen, detailed raw data is able for each result and can be accessed by simply clicking the value.

Custom report templates can be created for routine analysis providing the necessary level of information required or alternatively results can exported to a LIMS system.

The Qtegra ISDS Software also provides a comprehensive series of features for laboratories that operate within a regulatory environment such as 21 CFR Part 11 such as permission controls, electronic signatures, and audit trails.

Can I measure all the elements I require for my analyses?

Element performance in Graphite Furnace AA is limited by the relatively low temperature of the atomizer, this is restricted to a maximum of around 2900 ˚C to avoid degradation of the graphite cuvette. In addition the graphite material is relatively reactive and will form stable carbides with a number of elements such as Al, Si, Ti and B which reduces performance.

These factors reduce the capability of Graphite Furnace AA to the determination of 50 elements. Dynamic range is also rather restrictive being at best around 10^2.

In contrast ICP-MS uses a high temperature plasma source to generate ions which are then measured by the mass spectrometer. The plasma source is generated by inductively heating a flow of ionized argon which results in a source temperature of around 10000˚C. This is more than sufficient to break all chemical bonds and efficiently ionize most of the elements in the periodic table.

Refractory elements such as Al, Si, or V will have very much lower detection limits in ICP-MS and in excess of 70 elements can be measured in. Latest generation ICP-MS instrument such as the iCAP RQ ICP-MS with its advanced engineering of the analyzer and SEM detector also offers >10^9 dynamic range.

Do I need a lot of sample to measure all my elements?

One of the key benefits of Graphite Furnace AA is the ability to analyze small sample volumes. Typically only 10 to 20 µL are injected into the furnace for analysis and with the use of small volume autosampler vials a sample volume of 100 µL is usually sufficient for most routine work. However, it must be remembered that Graphite Furnace AA is a relatively slow, single element technique with each analysis taking around 3 to 4 minutes. This tends to reduce the advantage somewhat.

The standard sample introduction system used in ICP-MS is a pneumatic nebulizer and cyclonic spray chamber. This will typically use a sample flow rate of around 1mL·min^{-1}. The advanced technologies used in the iCAP RQ ICP-MS mean that analysis times can be reduced significantly and 1 to 2 minutes is a typical time. This means that a sample volume of 2 to 3 mL is usually sufficient for most applications.

ICP-MS is a very sensitive technique with detection limits generally 1 to 2 orders of magnitude lower than Graphite Furnace AA. This means that sample solutions can generally be diluted by a factor of 10 to 20 and it will still be possible to measure accurate data which reduces the requirement for sample volume.

If sample volume is limited then it is also possible to use low uptake nebulizer designs of 20 to 50 µL·min^{-1} and reduced volume spray chambers to reduce the volume requirement without sacrificing performance.

There is also an optional discrete sampling valve available which uses a sample loop to inject the sample into a carrier stream. This has the benefit of reducing sample uptake and rinse delays as well as reducing sample volume requirements.

Does ICP-MS suffer with many interference effects?

It is true that early ICP-MS instruments suffered with isobaric interferences for elements such as As or Se and it was necessary to use complex correction equations to obtain reliable data.

With the development of collision/reaction cell technology these interference effects could be simply overcome by the use of a pressurized cell containing either inert He or reactive gases prior to the mass analyzer.

The advanced engineering of the iCAP RQ ICP-MS uses a proprietary flatapole design for the QCell. In addition to providing powerful interference reduction this also has excellent low mass performance. This means that when the cell is pressurized with He and the instrument operated in He-KED mode it provides adequate sensitivity at low mass along with enhanced sensitivity for higher mass elements that are often required for routine analysis. The result is that it is possible to operate the instrument in a single mode for routine analysis which simplifies operation and reduces analysis time by avoiding gas fill/flush delays.

In addition the iCAP RQ ICP-MS also has a unique optimized angular deflection lens to collect ions from the plasma and direct them into the QCell, neutrals from the plasma are not deflected and will pass out of the lens stack. This means that there is no impact on the QCell and long term stability is improved.

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Does ICP-MS require a lot of routine maintenance?

This is not the case and in many situations the maintenance requirement for ICP-MS will be less than for Graphite Furnace AA where much more maintenance is required. This includes items such as replacement of graphite cuvettes, cleaning or replacement of the contact cones, cleaning of the furnace windows and cleaning of the optical pyrometer used for temperature measurement. Due to restricted access some of these operations aren't easy to carry out.

The iCAP RQ ICP-MS has been designed to minimize the need for routine maintenance and make things as easy as possible when it may be required.

The sample introduction assembly is positioned at bench height and is of an open architecture design making access extremely easy. The spray chamber and nebulizer can be removed very easily for cleaning and quickly replaced. The plasma torch has a self-aligning injector and simply twists into place automatically connecting the plasma gases. Sample flow is provided by a close coupled peristaltic pump and tubing is simply clipped into place, this is usually replaced after around 40 to 50 hours of use.

The propriety plasma interface has been designed to operate at the optimum temperature to limit the build up of sample matrix on the sample cone and greatly extends the time between cleaning operations. The sample cone, skimmer cone, and extraction lens assembly are rigidly attached to a mount on the interface door. When the door is opened the entire assembly pivots outwards to maximize access and makes maintenance very easy.

The skimmer cone is a unique design featuring user exchangeable inserts that can be changed in less than a minute. A series of different inserts are available that allow the user to choose a balance between sensitivity and resistance to matrix, optimizing the system to the application.

Find out more at **thermofisher.com/SQ-ICP-MS**

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