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UPGRADING TO ICP-OES FROM AAS

iCAP 7000 Plus Series ICP-OES

# SmartNotes

QA

## What are the benefits and considerations of upgrading to ICP-OES from AAS?

I have been a routine user of atomic absorption (AA) to analyze my samples for many years. However, the lab is now very busy and my instrument is old and needs to be replaced.

### Should I just purchase another AA?

You are already familiar with the technique having worked with it for a number of years so it might be an easy step to take. However, this also means that you know the limitations of the technique and restrictions it places 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 relatively 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, the need for different flame types to determine your suite of elements which complicates the analysis and increases your operating costs, and also the use of the flame limits the possibility of unattended operation reducing the effectiveness of your laboratory.

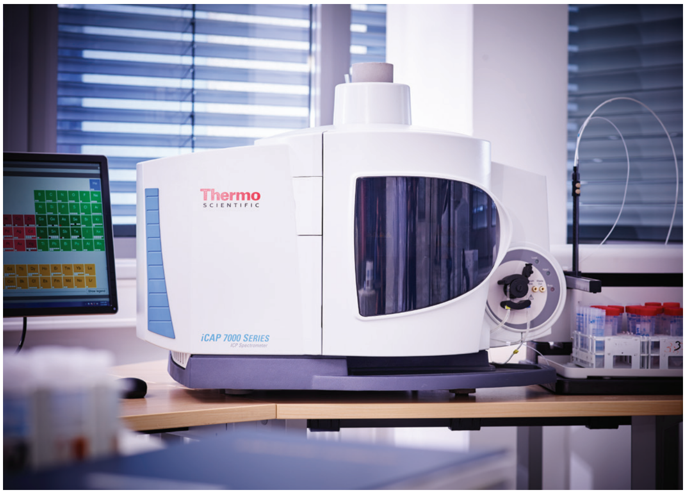

**Figure 1. Thermo Scientific iCAP 7000 Plus Series.**

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There would be a number of significant advantages for you to be gained by moving to another technique such as Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES).

## Does ICP-OES need a special environment for installation?

A modern ICP-OES such as the Thermo Scientific™ iCAP™ 7000 Plus Series ICP-OES (Figure 1) is a very rugged and reliable instrument and can be installed in most normal laboratory situations.

It is a very compact benchtop instrument and will only require a similar amount of bench space to your current AA.

The services required to operate the instrument are simpler than your AA, a single 220/240v 30amp supply is required for power and only a single gas, argon, is required for the plasma source.

The optical spectrometer is contained in a sealed housing which is purged with argon during operation to avoid problems of contamination and dust. The gas flow is reduced during standby and can be switched off if the instrument is not being used to save cost.

The detector is also contained in a sealed and purged housing to avoid issues with contamination. It is cooled to -45 ˚C by an integral peltier cooler to offer improved performance and the lowest detection limits.

Finally, the instrument is fully protected by a comprehensive series of interlocks that are constantly monitoring the status of the instrument. Should any issue be detected the instrument will be shut down to avoid potential damage.

## Is ICP-OES very expensive to operate?

Although the overall cost will depend on your local requirements, it is very likely that the relative cost for your complete analysis will be comparable to AA.

Currently you must use compressed air, acetylene and nitrous oxide for your flame analyses and argon for your graphite furnace and hydride generation analyses. There is also a significant additional cost for the graphite furnace cuvettes which typically last around 150 samples each (depending upon the number of repeaters per sample).

By comparison the ICP-OES will only require argon gas for the plasma source and to purge the detector. The iCAP 7000 Plus Series ICP-OES has also been designed to use a minimum gas flow for analysis and a reduced flow during standby further improving efficiency.

The real differentiator will become apparent when you consider the overall analysis cost. Since AA is a single element technique there is a relatively high amount of operator involvement switching between flame, furnace and vapour measurements to analyze all your elements.

By contrast, ICP-OES is a multi-element technique and all of your elements will be analyzed for each sample in a single measurement greatly increasing throughput and reducing the cost per sample.

A further benefit is that the ICP-OES is an inherently safe technique and only uses argon gas. This means that unattended operation is not a problem and you will be able to continue measuring samples during the night to improve the efficiency of your lab and reduce operating costs further.

## Is ICP-OES very difficult to use?

No not at all. Unlike flame AA where you need to select the correct burner and flame type for the element that you want to measure, ICP-OES uses a common torch for all analyses.

Only when analyzing organic solvents or very high levels of dissolved solids is a change required. In this case it is simply a matter of replacing the centre tube of the torch but since the iCAP 7000 Plus Series ICP-OES torch uses a self aligning design this is a very simple operation.

The single type of gas used for the plasma source also makes optimisation a much simpler operation than for AA. It is not necessary to optimize fuel and oxidant flows and the position of the burner and impact bead for different elements.

For routine analysis almost all aqueous samples can be analyzed with a common set of plasma parameters while organic solvents would typically use a second set of parameters.

If the very best detection limits are required then optimization of the plasma parameters and plasma viewing position can be carried out. The Thermo Scientific Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software used with the iCAP 7000 Plus Series ICP-OES has several automatic routines and that can be used to quickly and easily optimize the instrument.

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

The Qtegra ISDS Software makes method development extremely easy with the iCAP 7000 Plus Series ICP-OES. 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.

Elements are easily selected from a periodic table with the optimum wavelength for analysis being automatically displayed by Element Finder plug-in for the Qtegra ISDS Software. This selection is the result of an extensive element wavelength library containing information of common interferences.

The wavelength selection is also intelligently monitored as the LabBook is built up with subsequent elements using the optimum wavelength based on those already selected.

Optimum plasma parameters will also be selected by the Qtegra ISDS Software based on the type of sample to be measured. In most cases the default parameters will yield perfectly good results but they can be automatically optimized if required.

Any changes made to a LabBook will be stored for later review so an audit trail of the method development process is provided for information.

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

Element performance in AA is limited by the relatively low temperature of the atomizer. This is around 3000°C for both a nitrous oxide flame and a graphite furnace. Some elements have very high atomization energies and so have either poor performance in AA due to inefficient atomization or cannot be measured at all. This results in a total of 64 elements than can be analyzed by AA.

Conversely, the plasma source used in ICP-OES 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 atomize 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-OES.

In addition, the spectrometer and optical path in the iCAP 7000 Plus Series ICP-OES are sealed and purged of air or water vapour which dramatically improves optical transmission in the deep UV part of the spectrum. This allows the iCAP 7000 Plus Series ICP-OES to operate over a wavelength range of 167-847 nm which gives access to wavelengths for elements such as S, P or B which cannot be measured by AA.

In total it is possible to measure 73 elements routinely with ICP-OES.

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

In AA the oxidant flow used to sustain the flame is also typically used to provide the nebulizer gas which is then mixed with the fuel and sample aerosol. This is usually a relatively high gas flow of around 10 L·min which results in a liquid uptake rate of 5-6 ml·min.

Although graphite furnace can be used with sample volumes of 10 to 20 µl, it suffers with chemical interference and cannot be used for all elements.

Conversely, in ICP-OES the gas flows used for the plasma source are entirely separate from the flow used for the nebulizer. This results in finer control and a much lower gas flow of around 0.7 L·m is used. The nebulizer design can be optimized to provide best efficiency at low gas flow which results in sample flows of around 1 ml·min.

For applications where sample volume is limited micro nebulizer designs are also available that can operate with sample flows of 50 to 100 µl·min.

In addition, as already mentioned ICP-OES is a multi-element technique with analysis times generally around 1.5 to 2 minutes. This means that all elements can easily be measured with a total sample volume of 3 to 4 ml greatly reducing the amount of acids and reagents required for analysis and the amount of waste produced.

By comparison, a typical analysis of 10 elements using AA could take around 3 minutes of total measurement time and would require 20 to 25 ml of solution.

## Does ICP-OES suffer with many interference effects?

In many respects interferences in ICP-OES are much less severe than in AA and easier to deal with.

The high temperature of the plasma source is the main reason for the relative freedom from interference in ICP-OES. There is sufficient energy available to break all chemical bonds which means chemical interferences are extremely rare.

This is not the case with AA where there are many examples of chemical interference such as phosphorus suppression of Ca and Mg in flame AA or vapour phase interference of chloride in graphite furnace. Often it is necessary to use ionization buffers or mixtures of matrix modifiers to overcome the interference which complicates the analysis and increases the risk of contamination.

Background interference occurs in both AA and ICP-OES and results in falsely high results. It is very easy to see in ICP-OES since the spectra for all measurements are stored by the Qtegra ISDS Software and can be displayed. It is usually corrected automatically by subtracting background measurements from the total of peak and background.

It is more complicated to correct for in AA since it typically involves the use of a second light source such as a deuterium lamp. This is not ideal as there is insufficient energy from the deuterium lamp to allow correction at wavelengths greater than 425 nm.

The Zeeman effect can be used with a graphite furnace to magnetically shift the analyte peak and allow a background measurement to be taken.

The most common issue in ICP-OES is spectral interference due to the high energy of the plasma source which results in a high number of emission wavelengths being produced. The element wavelengths are very well documented and the Qtegra ISDS Software uses an extensive library for wavelength selection.

The software also provides a routine for automatic correction of spectral interference based on measurement of the interfering element and assessing the contribution on the analyte at the analysis wavelength.

Another option is that since the iCAP 7000 Plus Series ICP-OES is a simultaneous instrument adding additional element wavelengths to an analysis does not compromise the analysis time. There are often many wavelengths available for analysis for each element and generally there will be several of sufficient sensitivity to be used.

The Qtegra ISDS Software is able to automatically make a suggestion for additional wavelengths which can be added if spectral interference is likely to be a problem for some elements. The results can then be viewed in detail post run and data from the best wavelength used for reporting.

## Can I process data retrospectively?

Data can be processed retrospectively as this is a very powerful tool, which can be used for method development.

The CID86 detector used in the iCAP 7000 Plus Series ICP-OES provides continuous coverage of the spectrum being measured. This means that not only is data collected for the analyte peak but it is also collected for a region of the spectrum either side of the analyte peak. If there was to be a problem with background interference or other nearby spectral lines then it is very easy to move the points used to correct the baseline and recalculate the data.

In some applications the use of internal standardization is common to improve precision or correct for physical effects such as viscosity differences. Sometimes it can be difficult to choose the optimum element to be used as the internal standard due to compatibility with the sample type or problems of availability. In this case several elements can be assigned as possible internal standards and the data processed using in each one in turn. Once a best match has been found it can then be used for future analyses.

Typically only areas around each analyte peak are saved for routine analysis. However, it is possible to save an image of the entire spectrum which can then be used at a future date. If a result for an element is required that was not calibrated at the analysis time it is possible to measure it now.

## Does ICP-OES require a lot of routine maintenance?

In many situations the maintenance requirement for ICP-OES is less than that for AA. This is certainly the case for graphite furnace where more maintenance is required.

In AA it is very important for the burner to be kept clean, by definition the flame requires a fuel to be burnt which means that carbon can be deposited along the slot of the burner, especially when using a nitrous oxide flame. The carbon build up will disturb the flame causing the signal to drift and poor stability.

It is also necessary to clean the spray chamber on a regular basis to ensure that deposits do not collect and interrupt the draining of waste sample. The nebulizer should also be cleaned on a regular basis to ensure a stable flame and best sensitivity.

Reassembly of the spray chamber and nebulizer is a safety critical operation since the acetylene gas passes through it and it is necessary to check for leaks on completion.

Maintenance of the graphite furnace is more complex and involves 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 are not easy to carry out.

By comparison ICP-OES is more intuitive as far as maintenance is concerned. There is nothing burning in the plasma torch since the plasma is formed by ionized argon so carbon build up is not usually a problem. Only when analyzing organic solvents there is a possibility of carbon forming and usually this is because incorrect plasma gas flows have been set. Once set correctly it is common to measure for many hours without carbon build up.

It will be periodically necessary to clean the centre tube of the torch especially if samples with high levels of dissolved solids are analyzed. This is usually done by soaking in water and reassembly is easy due to the self aligning design.

The spray chamber and nebulizer also need to be cleaned periodically as in AA although in this case there are no safety concerns. If a leak were to be created then the plasma would just extinguish due to the air entrainment.

One additional operation for ICP-OES comes from the fact that the sample flow is pumped to the nebulizer rather than using a venturi effect. This is typically a peristaltic pump and the tubing would need to be replaced after around 40 to 50 hours of use. It is an easy operation since the tubing simply clips in place and the cost of the parts is very low.

Find out more at **thermofisher.com/ICP-OES**

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