

A vertical photograph on the left side of the page showing a bright blue flame in a laboratory setting, likely used for atomic absorption spectroscopy (AAS).

Why Flame AAS Users Are Moving Up to ICP-OES

Introduction

Atomic absorption spectrometry (AAS or flame AAS) and inductively coupled plasma optical emission spectrometry (ICP-OES) represent two long-established technologies used in a wide variety of analytical applications. ICP-OES has traditionally come with a considerably higher price tag than AAS. Thus, many users have felt constrained to consider only AAS instruments for a variety of tasks where lower costs were essential.

Recently, however, price points have shifted. Manufacturing efficiencies and other changes

now offer the possibility of obtaining an ICP-OES product at not much more than the cost of an AAS device. (Case in point: the SPECTRO GENESIS ICP-OES analyzer.) So a number of users previously locked into evaluating AAS technology alone are now considering ICP-OES for their next analytical instrument purchase.

This paper briefly outlines conventional flame AAS and ICP-OES technologies, and offers comparisons to help users choose the analyzer that's right for them.

Basic principles

AAS technology operates on the principle of atomic absorption, using a flame to atomize a sample. Most AAS instruments utilize flames fueled by an air/acetylene mixture, which generates temperatures at about 2300° C, or a nitrous oxide/acetylene mixture, which enables temperatures up to 2900° C. A liquid or dissolved sample is nebulized into an aerosol and introduced into the flame. The flame-generated ground state atoms can absorb radiation. Light of a specific wavelength, generated by an analyte element-specific hollow cathode lamp and passing through the flame, is partially absorbed by the atoms of the analyte element. Radiation absorbance is measured by a detector, and through an empirical calibration, the concentration of an element present in the sample is determined.



Flame AAS, which uses a hollow cathode lamp for each element to be analyzed, has long been the hot choice for many analyses. But the increasing affordability of advanced, high-productivity ICP-OES casts a brighter light on AAS disadvantages.

ICP-OES technology relies on atomic/ionic emission, breaking down a sample within a high-temperature (typically up to 10000° C) argon plasma to generate atoms and ions. In the basic ICP-OES method, sample elements emit a characteristic number of specific spectral lines with different wavelengths when excited within the plasma. Emitted light is resolved into these separate lines by optical components such as diffraction gratings; the light is finally directed onto a detector array that quantifies light intensities at these different wavelengths. Elemental components of a sample may be identified and their concentrations determined using a suitable calibration.

AAS analyzers are always *sequential*: they must analyze each element in a sample one at a time. Some ICP-OES analyzers are also sequential. Others are *simultaneous*: they analyze the entire relevant emission spectrum of elements in a sample at the same time. When faced with samples containing more than a few elements, sequential examination demands relatively long analysis times. This difference can have critical implications for throughput, as discussed later in this paper.

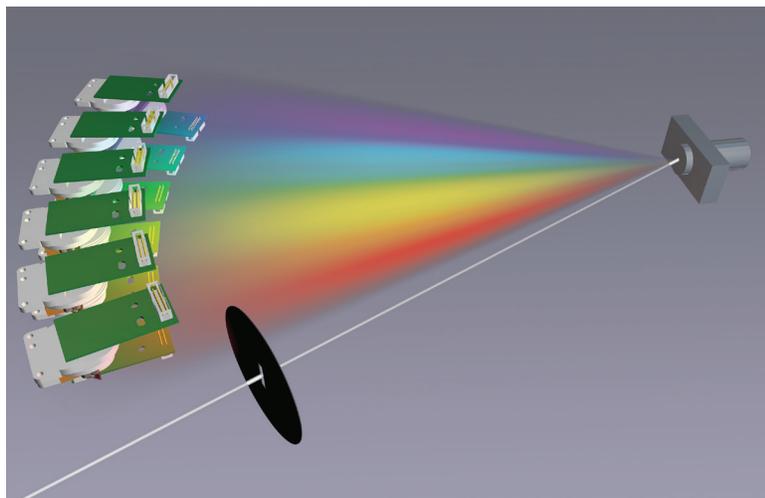
Ease of use

AAS instruments are widely used for routine analyses in a variety of industries for several reasons — high among them, ease of operation.

In seeming contrast, ICP-OES is the technology of choice for more complex laboratory analysis. However, basic operation can be greatly simplified in models intended for routine analysis. An instrument such as the SPECTRO GENESIS is optimized to eliminate many of the difficulties associated with using a new technique or instrument. Sample preparation is much easier. Unlike AAS, which suffers from a limited linear range, with ICP-OES a single sample dilution is normally sufficient to measure all elemental concentrations. Furthermore, with an ICP-OES instrument's relative freedom from matrix effects, buffers or matrix modifiers common in AAS are typically not required.

Overall, SPECTRO GENESIS is designed for minimal installation and training requirements, plus easy operation and maintenance. It's available with a complete set of factory methods plus step-by-step standard operating procedures (SOPs) for a number of common analytical applications. It can also be equipped with an automated

Advanced ICP-OES optics: the ORCA polychromator system in a SPECTRO GENESIS analyzer separates light emitted in the plasma, and enables full simultaneous measurement of the relevant spectrum and elements.



front-end sample preparation system. So users can move straight into “plug & analyze” performance without time-consuming method development.

Detection limits

For some measurement targets — notably Group I elements such as lithium, sodium, and potassium — AAS provides limits of detection (LODs) that are superior to those of most instruments employing ICP-OES technology. However, AAS cannot analyze a number of nonmetals at all. And its LODs for phosphorus are unimpressive.

By contrast, an ICP-OES instrument such as SPECTRO GENESIS provides substantially better LODs on refractory elements such as aluminum, titanium, boron, and vanadium. It provides good results on nonmetals such as sulfur and phosphorous — elements where AAS fails. In fact, for phosphorus, ICP-OES analysis offers LODs three orders of magnitude lower than those obtained with an AAS analyzer.

In a related limitation, AAS technology typically requires that an instrument be equipped with hollow cathode lamps specific to any of the elements to be analyzed. In all, depending on lamp availability, an AAS instrument can analyze (in sequence) up to 67 elements. Conversely, an ICP-OES instrument such as SPECTRO GENESIS uses no lamps at all, and captures the entire relevant spectrum — from 175 to 777 nanometers (nm) — in a single analysis. All told, it can simultaneously analyze up to 78 elements.

Throughput

Throughput differences may play a key role in many users' purchasing decisions.

It all comes down to the instrument's likely duty cycle. How many elements must typically be analyzed for each sample? With three replicates, an AAS instrument requires about 10 seconds for each element sequentially, for 30 seconds of total analysis time. By contrast, SPECTRO GENESIS takes about 60 seconds for three replicates. But in that time, it can analyze any number of elements simultaneously.

Reliable estimates put the light duty/heavy duty breakpoint at around 50 samples and 10 elements per day. If an instrument's workload typically remains at or under this limit, AAS devices may have an edge. However, if an instrument must routinely analyze more than 50 samples and 10 elements per day, a heavy-duty, simultaneous ICP-OES such as SPECTRO GENESIS can definitely deliver higher throughput rates, and will usually be the better choice.

Safe walkaway automation

Because their operation depends on flames and pressurized flammable gases, flame AAS instruments cannot safely run unattended.

By contrast, an ICP-OES like SPECTRO GENESIS does not require flammable gases. Properly set up, it can run safely without supervision for an entire shift or more — including overnight stints.

This level of true walkaway automation frees up laboratory personnel for other critical tasks. It can also increase throughput substantially.

Dynamic range

AAS instruments typically exhibit a linear dynamic range of 10^3 — fairly low compared to other major spectroscopic techniques. It makes these instruments ideal for measuring trace element concentrations, but less suitable for dealing with samples exhibiting wide concentration ranges — particularly those containing high concentrations of a given element or elements.

Compare that with the performance of an ICP-OES instrument such as SPECTRO GENESIS. It possesses a typical linear dynamic range of 10^5 — among the widest such ranges available. This enables analyses from parts per billion to hundreds of parts per million. Example: SPECTRO GENESIS can measure copper using the emission line at 324.74 nm from its approximately 0.002 ppm LOD to more than 200 ppm. Automatic switching to alternative lines, where available, can often extend the dynamic range even further — with no throughput penalty, since all lines are measured simultaneously.

Chemical interferences

Interferences caused by chemical interactions within a sample are relatively common with AAS technology.

Temperatures reached within the AAS flame don't exceed 3000° C, so a number of chemical bonds, most notably in refractory oxides, may persist.

With the ICP-OES plasma, it's a different story. Virtually all chemical bonds are totally destroyed by about 6000° C. Since temperatures in the plasma can reach up to 10000° C, this technology completely eliminates chemical interferences.

Ionization interferences

AAS technology experiences ionization interference in trying to analyze Group II elements such as magnesium, calcium, and barium, using a nitrous oxide flame. Operators must take the extra step of adding an appropriate buffer to the sample solution to counteract this.

With ICP-OES, minimal ionization interferences are encountered. One exception: some ICP-OES analyzers do

experience easily ionizable element (EIE) interferences when encountering matrices containing Group I elements such as lithium, sodium, and potassium (and to a lesser extent, also Group II elements). However, ICP-OES models such as SPECTRO GENESIS eliminate effects of this interference via radial plasma observation. No buffering is required.

Spectral interferences

Since AAS analyzers use only a single wavelength, they exhibit practically no spectral interferences.

ICP-OES analyzers "see" the entire relevant spectrum, for a much more information-rich environment. This means they may encounter spectral interferences when dealing with closely spaced, line-rich matrices, such as those associated with metals. However, with a larger choice of emission lines for a single element, spectral interferences can typically be prevented by appropriate line selection. Even where spectral interferences cannot be avoided fully, ICP-OES analyzers apply mathematical correction to compensate, securing accurate results.

Stability and TDS

AAS analyzers suffer from a lack of long-term *stability*. An AAS instrument often requires several recalibrations over any 8-hour shift.

SPECTRO GENESIS ICP-OES provides accurate, high-productivity analyses — along with low operating and consumables costs plus an optional automated sample introduction system.



Long-term stability for an ICP-OES such as SPECTRO GENESIS is excellent. At less than 2 percent long-term instability over an 8-hour period, the instrument avoids the need for frequent recalibration.

Also, ICP-OES instruments can — and AAS devices cannot — tolerate high levels of *total dissolved solids* (TDS).

Lamp costs

As mentioned, AAS technology generates light of a specific wavelength to be partially absorbed by the target element's atoms within the flame. So an AAS instrument must be equipped with a hollow cathode lamp (HCL) to generate that light. In fact, it requires a separate lamp (sometimes ganged in a multi-element array) for each element to be analyzed.

Unfortunately, such lamps are relatively costly consumables with fairly short lifetimes. The effort and expense needed to purchase, inventory, and replace

them can be a significant factor in AAS cost comparisons.

Even from lower-cost, aftermarket suppliers, HCL list prices in a recent survey ranged from \$200 to \$600 per lamp, depending mainly on the element-specific cathode material.

As for lamp longevity: heavy-duty usage — for instance, running an AAS instrument more or less continuously, for 8000 hours annually — may require up to five HCLs per element per year! And even in low-use cases, these products can exhibit quite short shelf lives. Since lamp lifetimes are often rated at 1 year, even users who utilize their instruments only occasionally might have to purchase an HCL for every analyte of interest every year.

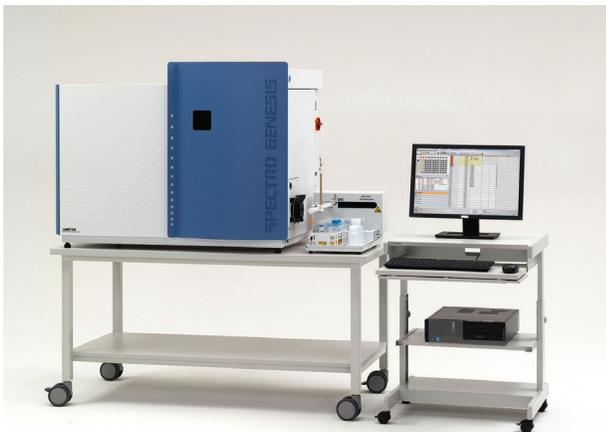
In sharp contrast, since their technology makes no use of HCLs, ICP-OES instruments incur no lamp purchase or replacement costs.

Summary costs and times

AAS	ICP-OES (SPECTRO GENESIS)
Costs to purchase/replace lamp(s): variable but often significant (see above)	Costs to purchase/replace lamp(s): none
Costs to analyze 1 sample with 16 elements: <ul style="list-style-type: none"> • Bottle of acetylene (8500 liters) \$120.00 • Acetylene consumption 4 liters/minute; \$0.15 per analysis • Electricity 0.03 kilowatt hour at \$0.01 	Costs to analyze 1 sample with any number of elements: <ul style="list-style-type: none"> • Bottle of argon (15000 liters) \$80.00 • Argon consumption 18 liters/minute; \$0.15 per analysis • Electricity 0.03 kilowatt hour at \$0.01
Total analysis time: 160 seconds	Total analysis time: 90 seconds
Throughput: At 16 elements, can analyze up to 180 samples in an 8-hour shift	Throughput: At any number of elements, can analyze up to 320 samples in an 8-hour shift

Conclusion

Now that pricing for AAS analyzers and some ICP-OES analyzers is beginning to converge, direct comparisons of technical advantages and disadvantages become useful. Users should carefully consider the issues raised in this report to select the best technology for their unique elemental analysis applications.



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