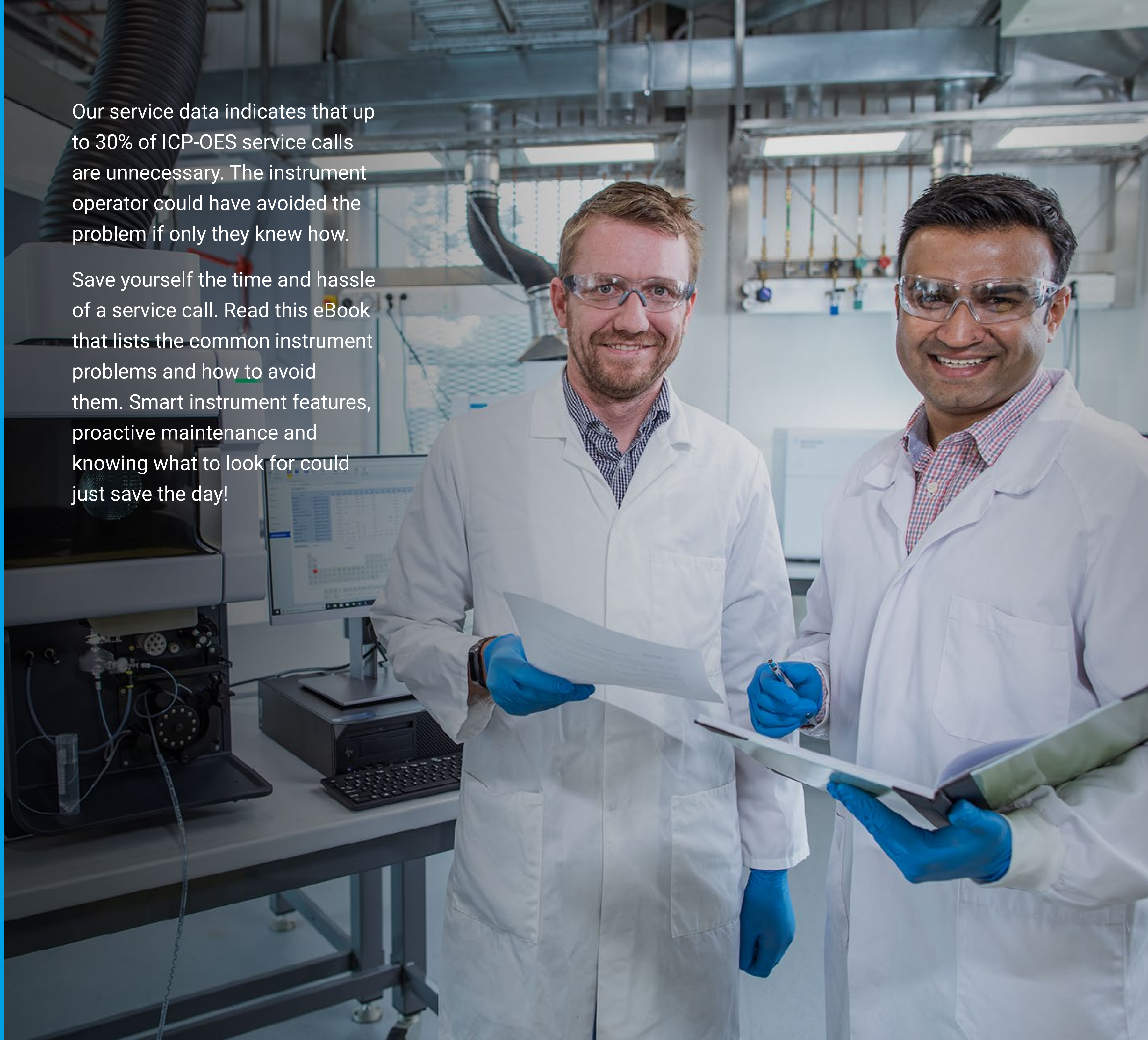
How to Prevent Common ICP-OES Instrument Problems

Tips and advice to prevent problems that cause time-wasting remeasurement of samples, from an ICP-OES expert

Agilent
Trusted Answers



Our service data indicates that up to 30% of ICP-OES service calls are unnecessary. The instrument operator could have avoided the problem if only they knew how.

Save yourself the time and hassle of a service call. Read this eBook that lists the common instrument problems and how to avoid them. Smart instrument features, proactive maintenance and knowing what to look for could just save the day!

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) is a well established technique for the measurement of elements in solution. ICP-OES is used in the mining, food, agriculture, energy, chemical, environmental monitoring, and pharmaceutical industries. The technique delivers sensitive, accurate, and precise measurements of the concentration of elements in a wide range of sample types. From analyzing sludges and sediments, through to drinking water and wine, ICP-OES is fast and reliable.

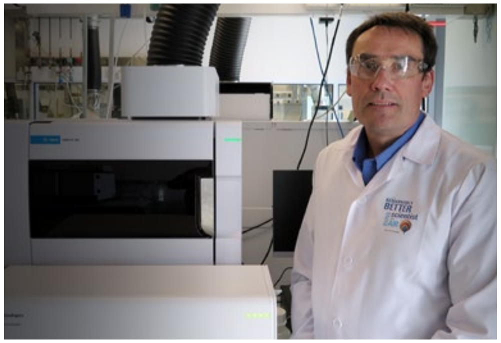
The technique is just one of a suite of atomic spectroscopy techniques offered by Agilent. The origins of atomic spectroscopy lie in flame atomic absorption spectroscopy (FAAS), invented in the 1950 s and still used in many labs today. At the other end of the family tree is inductively coupled plasma mass spectroscopy (ICP-MS). This technique is known for its high sensitivity, being able to measure elements at parts-per-trillion levels.

While the simple Beer-Lambert law is at the heart of all atomic spectroscopy techniques, most require a degree of knowledge and experience to achieve accurate and reproducible results. As instruments become more sophisticated, they can reduce the level of expertise required to do the analysis.

Modern cars have followed a similar development path. Features such as antilock braking, driver-assist technologies, and a multitude of monitoring systems have reduced the level of knowledge and skill required of the driver. Most people no longer carry the tool box that was needed for roadside repairs of cars in the 20th century. Similarly, current ICP-OES instruments offer a range of “smarts” that help the analyst identify and overcome problems. The analyst can then take action to avoid having to remeasure samples.

ICP-OES problems can be separated into two areas:

1. Problems caused by characteristics of the sample and mistakes made by operators during sample preparation and measurement, and
2. Problems caused by something going wrong with the instrument.



In this e-book, Agilent Technologies ICP-OES Marketing Manager Ross Ashdown, discusses ways laboratories can overcome instrument-related ICP-OES problems. Using common quality control methods and advances in ICP-OES instrumentation, analysts can avoid having to remeasure samples and can be confident in getting the right answer the first time.

Q:

Nebulizer blockages are a common problem for techniques that aspirate liquid samples. Are there ways to avoid or detect blockages?

A:

Aqueous solutions can contain fine particles that are virtually invisible to human eye. These particles can partially or fully block the small capillary tube at the tip of a glass concentric nebulizer. These blockages lead to many performance problems, which inevitably lead to having to remeasure samples.

A typical symptom of a partial nebulizer blockage is a low recovery for a continuing calibration verification (CCV) standard. The CCV solution is usually monitored periodically throughout an analysis. You only measure a CCV every 20–30 samples, so if it fails you have to remeasure all 20–30 samples that were measured prior to the failed QC.

A complete nebulizer blockage results in no signal at all, so is easy to diagnose as all results, including the internal standard, will show low or no emission signal.

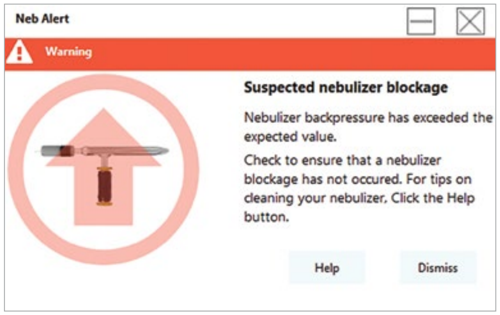
You can avoid having to remeasure samples due to a nebulizer blockage by monitoring both the CCV results and sample results over the course of an analytical run.

If you are frequently suffering from nebulizer blockages, consider taking the following actions:

- Filter or centrifuge the samples.
- Set the autosampler probe depth to sample from a significant distance above the base of the tube, to minimize the chance for particles on the bottom of the test tube to be sucked up into the probe.
- Change the type of nebulizer you are using to one with a larger internal diameter on the sample line that is more resistant to blockage.
- Add a switching valve to reduce the time the sample spends in the sample introduction system, and increase the rinse time without increasing sample analysis times, making blockages less likely.
- Use an argon humidifier to keep the tip of the nebulizer moist; solids are less likely to be deposited on the tip of the nebulizer, thereby reducing blockages from solutions with high % total dissolved solids (TDS).

The Agilent 5800 and 5900 ICP-OES instruments have a “Neb alert” function that alerts the analyst when the nebulizer back pressure changes. An increase in pressure can indicate a blockage is developing, whereas a decrease in pressure may be caused by a leaking connection. As soon as a predefined back pressure threshold is reached, an on-screen alert notifies the analyst of the problem. The run can be automatically stopped so the user can fix the problem before the quality of sample analysis is impacted.

Each type of nebulizer will run at a slightly different back pressure, so the alert threshold can be adjusted to suit the nebulizer type. To get an idea of what “normal’ is for the nebulizer being used, the analyst can review the plot of the nebuliser back pressure on the analysis page or use the separate Nebulizer Test function within the Agilent ICP Expert software.



The Neb Alert function of the Agilent 5800 and 5900 ICP-OES alerts the analyst when the nebulizer back pressure changes–indicating either a blockage or a leak.

Q:

What about other parts of the sample introduction system, like the plasma torch? How do they contribute to downtime, and how can this be avoided?

A:

The plasma torch is another component that is subject to harsh conditions. Aspiration of strong matrix samples like 100 g/L solutions can lead to crystalline deposition in the injector of the torch. These deposits can lead to a partially blocked torch injector and an erroneously reduced signal.

If various QC solutions are being monitored, downwards drift in the signal is a sign that torch blockage maybe happening. Different QCs can pick up this drifting signal. Frequent monitoring of a certified reference material (CRM) introduced as a QC solution will show declining recoveries if there's a torch blockage. Poor (low) recovery for quality control check solutions, such as continuous calibration verification solutions (CCV) is also an indication of a blockage.

If the instrument no longer produces the same reading for the calibration solutions when checked as a QC, then drift has occurred and a blockage somewhere in the sample introduction system is likely. Torches that have a horizontal orientation in the instrument experience this blockage with the highest frequency. Torches that are positioned vertically are more immune.

Another common type of torch problem is incorrect assembly or installation. This occurs when fitting a new torch or refitting one that has just been cleaned.

Running automated instrument performance tests at the start of each day that indicate a pass or fail based on values set by the manufacturer will highlight any sensitivity problems. The torch can then be checked to ensure it has been correctly assembled and installed.

Some torches have many components, which makes it easy to make a mistake when putting them back together. Incorrect torch assembly or installation can impact instrument sensitivity. Poor sensitivity will cause poor precision for low concentration analytes. This can be monitored and detected through inclusion of an appropriate QC solution and QC test.

When investigating the purchase of a new ICP-OES, it's a good idea to take a close look at the torch you'll most commonly use. Take it apart and put it back together again to see how foolproof the assembly is. The standard torch of an Agilent 5800 or 5900 ICP-OES instrument is a simple one piece assembly. It's impossible to assemble it incorrectly. Even our demountable torches have only 5 components. This compares to over 10 torch components for some other vendors' torch designs.

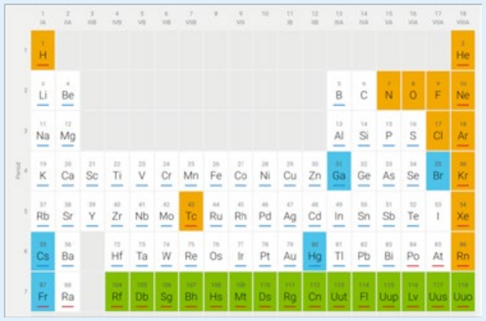
Torch alignment is another potential source of problems. You may have assembled the torch correctly, but is it in the correct position in the instrument to ensure optimum viewing of the emission signal? We have eliminated manual alignment of the torch in the Agilent ICP-OES instruments. The plug and play torch installation system ensures the torch is positioned perfectly with the instrument's pre-optics, without requiring analyst intervention or an optimization routine.

A plasma that fails to ignite is another cause of wasted time. Sometimes the causes of the failure are not obvious, forcing the analyst to spend time troubleshooting to fix the problem. The Agilent 5800 and 5900 ICP-OES instruments have sensors built in that monitor the plasma ignition sequence. These sensors allow the cause of the plasma failure to be pinpointed. It might be that the pump tubes aren't clamped or that the torch has not been dried and is still wet. With this information, the instrument can provide specific guidance on how to fix the problem.

High-Quality Single-Element Standards

Accurately detecting heavy metals and other elements in environmental, food, and elemental analysis samples depends on having high-quality reference materials. Agilent ULTRA standards are rigorously tested and manufactured using ISO 17025 and 17034 accreditations.

Use our interactive periodic table to find the element you need.

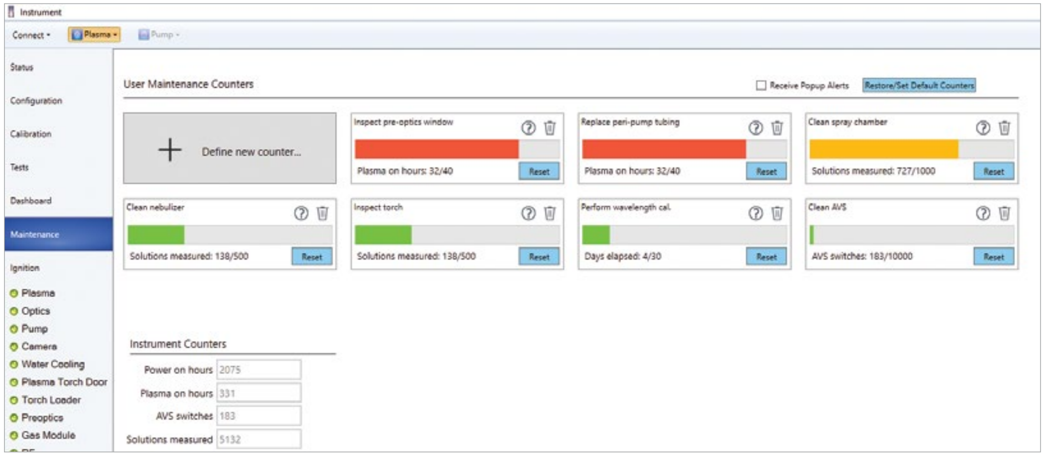


Q:

Labs can sometimes assume that analytical instruments just keep running within specification. How can they be sure that their instrument is operating as it should?

A:

It's a common misconception that instruments like an ICP-OES will keep on running, day in, day out, without any maintenance or attention. But with the potential for blockages in the sample introduction system from samples, high sample workloads and exposure to harsh environmental conditions in the laboratory, establishing a regular maintenance schedule can ensure optimum instrument performance and prevent small issues causing downtime during analysis.



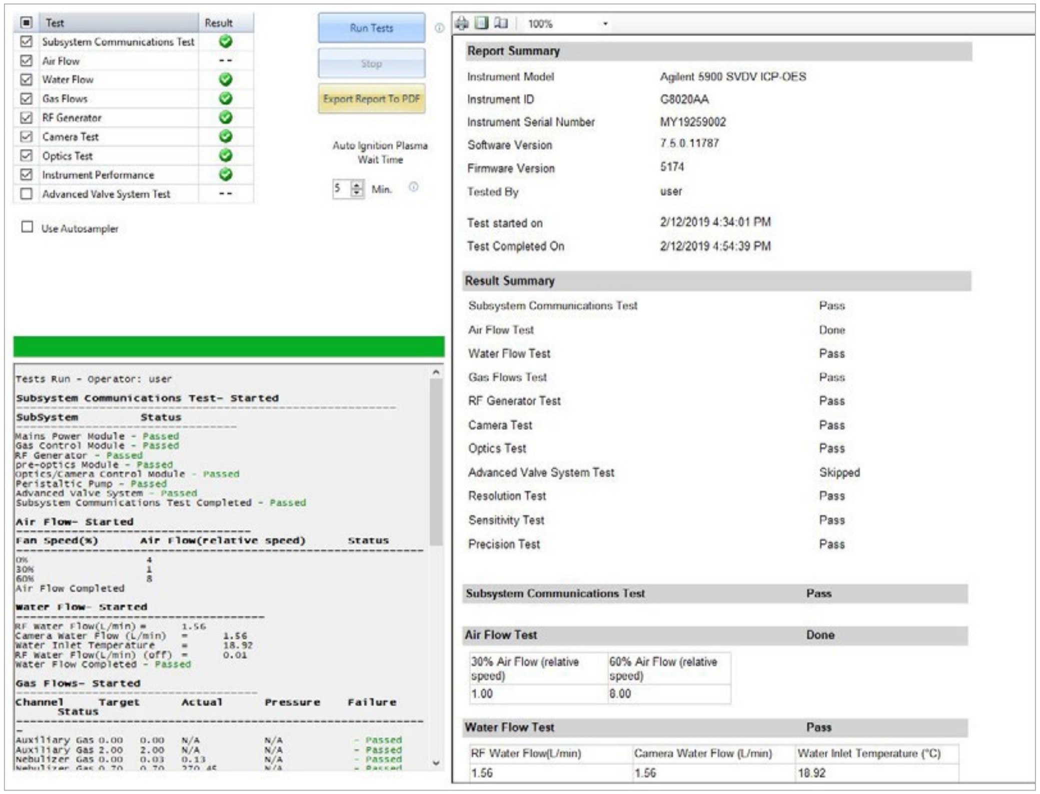
Both the Agilent 5800 and 5900 ICP-OES instruments include the early maintenance feedback function. This function counts the number of samples measured. It displays screen alerts, reminding the analyst to perform cleaning and maintenance functions, after the nominated number of samples have been measured.

Utilities required for instrument operation, such as argon, cooling water, or electrical power, can also malfunction. If utility supply is sub optimal, analytical performance can be compromised. This can impact result sensitivity, precision, and linear dynamic range as well as all other aspects of performance.

A good strategy is to always run an automated instrument performance test prior to starting analysis each day. This will immediately confirm correct operation, preventing remeasurement when you notice that your results aren't right. Most ICP-OES instruments have built in performance tests. Some also test utilities such as gas supplies.

In addition to the alerts, the 5800 and 5900 ICP-OES instruments have over 100 sensors that continuously monitor key parts of the instrument. Is your nebulizer getting blocked? You'll be notified. Did you forget to clamp your pump tubes? You'll be told about that too. Even the air intake filter in the instrument is monitored. If you think about the last time you cleaned your air conditioner filter at home, you'll realize how easy it is to forget these out-of-sight, out-of-mind components. If your lab is particularly dusty, the filter might be blocked and you won't know that the air supply to the instrument isn't high enough until something goes wrong. The 5800 and 5900 will let you know long before that situation arises.

We built-in some very useful instrument monitoring functions in the 5800 and 5900 ICP-OES instruments. The Early Maintenance Feedback (EMF) system allows alerts to be setup to prompt the analyst to run performance tests or do some preventative maintenance. These alerts are based on the number of samples that have been run. Just like the recommended service intervals for cars are based on how far they've been driven, performing maintenance based on the sample throughput, rather than just elapsed time, is a better way to maintain instrument performance. The alerts can be adjusted to suit the sample types a lab typically runs. For example, a lab analyzing predominantly clean water samples will need to do maintenance far less frequently than a lab measuring challenging samples such as soils that have been acid digested and have higher levels of dissolved solids.



Automated instrument performance test built into the 5800 and 5900 ICP-OES can be run quickly daily, to ensure everything is OK, prior to measuring samples.

Q:

Getting the method settings right is a critical activity in labs. What's the best way to ensure that correct settings have been selected?

A:

Instrument method settings can dramatically impact your results. For example, inadequate argon gas flows into the plasma and inadequate plasma power will lead to insufficient energy in the plasma. Not all the atoms and ions in your sample will be excited, with the result being reduced emission and reduced sensitivity. This will impact precision for analytes that are in trace levels. The precision will sometimes fall outside the lab threshold and when this occurs, it will mean the samples will need to be remeasured.

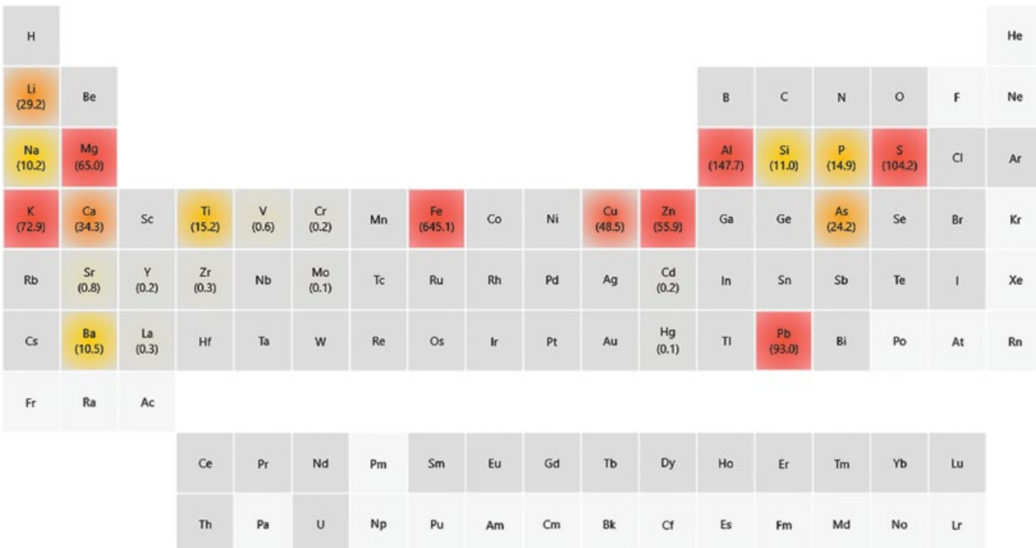
Other common mistakes include setting the wrong pump speed and an inadequate pre-read delay time. Rinse time is also critical. If you set the rinse time too short, you risk getting carry-over contamination from the previous sample. Set the rinse time too long and you are wasting time, power, and gas.

To determine the best method settings, it's a good idea to analyze a CRM with a similar matrix to that of your samples as part of your method development process. You should aim to get good recoveries at the trace levels when you measure the CRM (assuming the trace levels are within manufacturer's specification for your instrument). If you are unable to get good recoveries at the trace levels, further optimization of the method will be required.

The sample pump speed or delay time settings in your method can be assessed by monitoring the precision of a QC solution. It is also advisable to test both of these settings prior to starting an analysis. To test if the pump speed and uptake delay time are correct, manually activate the high pump speed and time how long it takes for the solution to go between the autosampler test tube and the spray chamber. This time should be entered as the uptake delay and the pump speed used should be manually inserted into the method.

Modern instruments often have a range of tools to help you define the optimum method settings. For example the Agilent 5800 and 5900 have a function called IntelliQuant Screening. This function does a quick scan of a sample to identify up to 70 elements in a sample and their approximate concentrations. If you use a sample that is representative of the samples you will typically analyze with this method, then you can use the IntelliQuant results to guide method settings. For example, if you see that there are low levels of your target elements in the sample, then you can increase the RF power and decrease the nebulizer flow to improve sensitivity for that element.

The IntelliQuant information can also be used to determine the concentration range of your calibration. By knowing the approximate concentrations of each element in a sample, you can select standard concentrations that will bracket the expected sample concentrations, to help you achieve an accurate calibration covering the full range.



The Agilent IntelliQuant function semi-quantifies up to 70 elements in a sample. This is useful when developing a new method or measuring unknown samples from a new source.

Tip: The IntelliQuant function also helps select the optimum analytical wavelengths, based on several factors, including spectral interferences. You can find out more about this in the ebook "How to Reduce ICP-OES Remeasurement Caused by Sample Problems and Errors"

Intelligent Rinse is another function that takes the guess work out of method development and avoids the need to use excessively long rinse times. When setting up a method, you are normally forced to specify a rinse time that will suit your highest concentration samples. This rinse time is then used for all samples, even those with much lower concentrations of analytes. The Agilent Intelligent Rinse function monitors nominated element wavelengths during the rinse period, automatically ending the rinse when these intensities reach a user-specified washout threshold. Intelligent Rinse removes the need for the analyst to specify a rinse time in the method, so it's also one less setting to define.

Q:

Are there some components of an ICP-OES that people often forget to check?

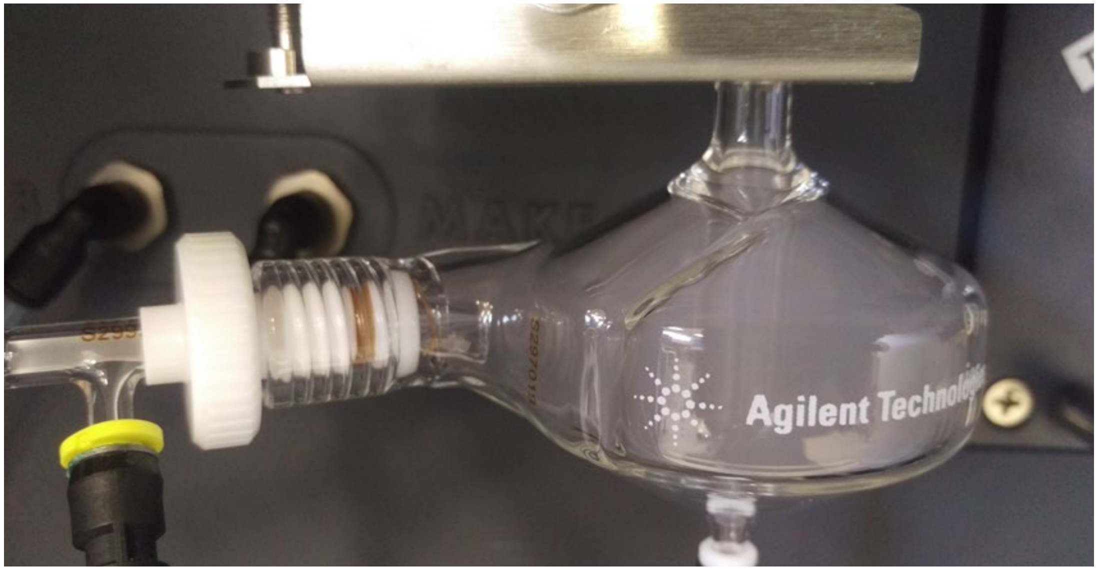
A:

Pre-optic windows are the round glass windows between the torch compartment and the chamber holding the optical components of the instrument. As contamination from samples being analyzed builds up on the windows, it reduces the amount of emitted light that passes into the optics to reach the detector. Dirty pre-optic windows will cause reduced sensitivity. This, in turn, leads to poor precision, which may result in samples having to be remeasured, particularly those with trace-level analytes that emit low levels of light. Monitoring the precision of the measurement of QC samples will identify this problem. However, there are many analytical performance issues that result in poor result precision. This makes it hard to pinpoint one cause.

Analysts should include the cleaning of pre-optic windows in the regular maintenance schedule for an instrument. Running the automated instrument performance tests each day will uncover any loss in instrument sensitivity.

The Agilent 5800 and 5900 have a handy function called Early Maintenance Feedback, or EMF. This function allows you set alerts based on the number of samples measured. Often, cleaning and maintenance schedules documented in lab procedures are forgotten or ignored, particularly when the lab is under time pressure. Not performing these tasks can have big impacts on results, which then wastes time because the analyst has to troubleshoot the problem and potentially remeasure samples.

The EMF function will display an alert reminding the analyst to do maintenance tasks, like cleaning the pre-optic windows–before results are impacted. The beauty of the EMF function is that it can be adjusted to suit your sample types. If a lab often runs high matrix samples, like soils and sludges, then they will need to do more frequent cleaning and maintenance than a lab that just runs water samples.



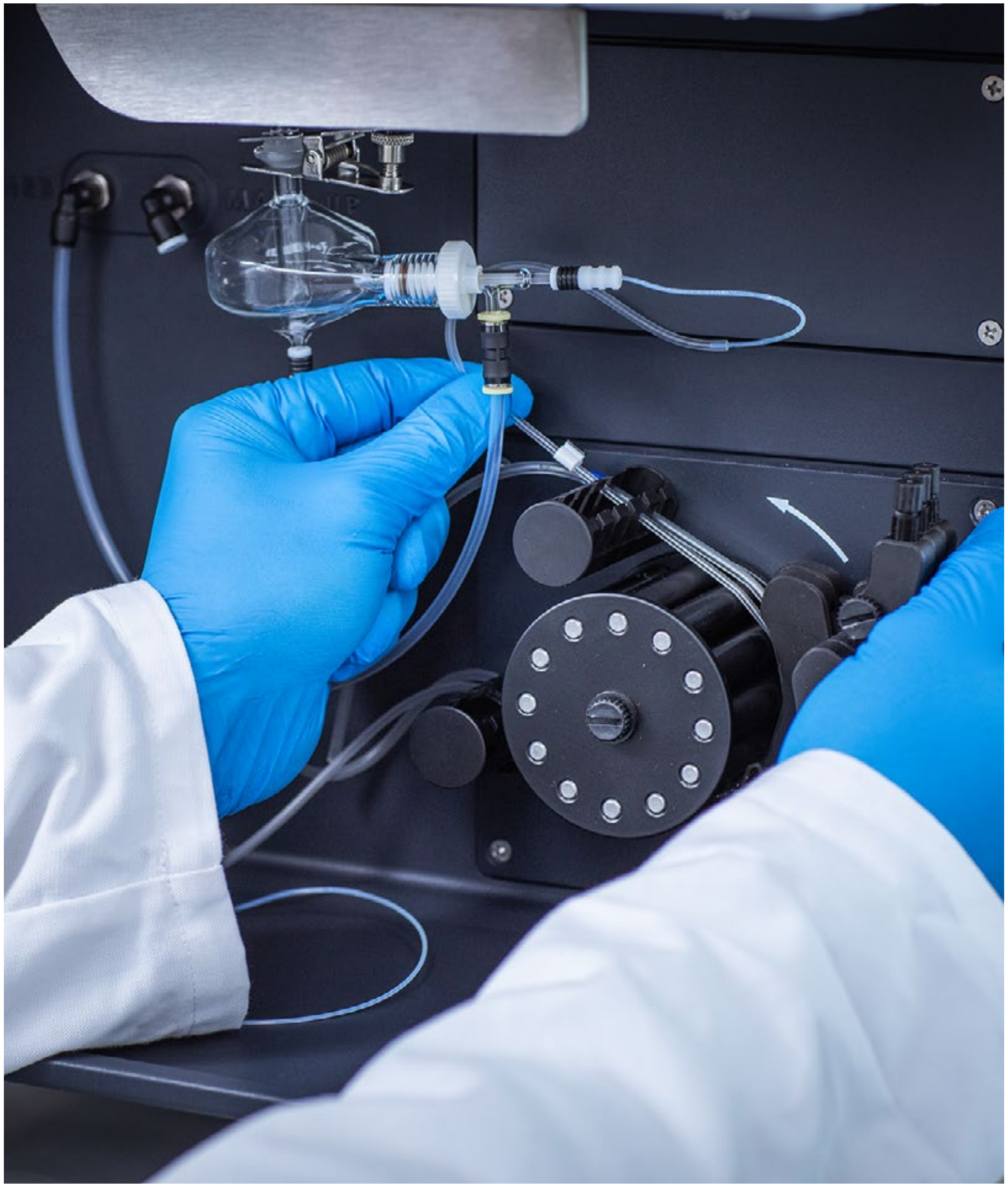
The other great benefit of the EMF function is the fact that it can be used as evidence during an audit. For example, if your lab has a three-week routine maintenance cycle, but during one of those weeks you only ran 50 samples, then the maintenance may have been delayed. If an auditor questions why the maintenance wasn't done, the data from the EMF function can be used to show that maintenance wasn't necessary because of the reduced sample load. In fact, you can move away completely from time-based maintenance schedules and get rid of hardcopy ICP maintenance records.
The EMF function retains all the data and will do the maintenance scheduling for you.

Q:

You mentioned tubing as being another common source of problems. What should labs do to prevent or identify tubing problems?

A:

Pump tubing wear is an underappreciated problem. Many labs will put up with gradually wearing tubing–not realizing the impact of worn pump tubing. Replacing worn tubing is a simple and relatively cheap maintenance task, but pump tubing that is not replaced regularly can have a big detrimental impact on results.



Leaking connections, incorrect tension, and air bubbles are other common tubing problems. Even forgetting to reclamp the peristaltic pump tubing before starting a sample run can trip analysts up.

Worn, leaking, or maladjusted peristaltic pump tubes will cause poor result precision, and this can also introduce drift during the analysis as the pumping efficiency of the worn tubing changes with use. Both precision and drift can be monitored through QC solutions, but they are often spaced 30–40 minutes apart, so waiting for a failed QC solution to address an issue wastes a lot of time, especially when you need to go back and remeasure samples that had been measured since the last valid QC.

Regular routine maintenance prevents the occurrence of peristaltic pump tube problems. Checking the tube's elasticity, roundness, connection, and tension at the start of each day, or after a certain number of samples, is important. These checks can reduce the risk of having to remeasure samples due to pump tubing problems.

The Early Maintenance Feedback function of the Agilent 5800 and 5900 ICP-OES instruments can be used to alert the analyst to perform tubing maintenance tasks. For example, an EMF alert can be set up to remind the analyst to check or change the pump tube on a time-based or sample-based frequency. The alert counter can be set to a value to suit the type of sample matrix. For example, if you are using 2% nitric acid solutions, you could set the counter to alert after 2000–3000 samples. If you are using a higher acid concentration, the alert counter will need to be set lower, to approximately every 1000 samples.

Another thing to consider is the type of pump tubing you are using. It needs to be chemically resistant to the sample matrix–therefore, organic and aqueous solutions need different types of pump tubing. PVC works well for most aqueous and acidic matrices, but it is not suitable for use with most organic solvents. It degrades quickly so that it doesn't pump properly, and with some solvents, it may even break down completely. Regularly checking the elasticity of the tubing is an easy monitoring task. As the tubing degrades, it goes hard, stretches, and loses elasticity.

Running a rinse solution through the instrument and unclamping the tubing and disconnecting the pump tubes (so it is no longer stretched over the pump rollers) at the end of the day is good practice. These actions will prolong the life of pump tubes. If you leave your sample matrix sitting in the tubes overnight, you may get sample leaching into the tubing, causing contamination of the first samples of the next run and faster degradation of the tubing.

The Outlier Conditional Formatting function of the Agilent 5800 and 5900 ICP-OES instruments is another useful tool that can identify worn pump tubing. It monitors sample results during a run and can flag when the %RSD exceeds a nominated threshold, internal standard failures, over ranges, or inconsistent results across wavelengths of the same element. Typically, worn pump tubing will cause an increase in the %RSD. If an alert is triggered, then the analyst has the opportunity to fix the problem before a lot of samples have been completed and have to be remeasured after changing the pump tubing.

Q:

The spray chamber is another critical component of the sample introduction system. Do you have any tips for maintaining the performance of the spray chamber?

A:

A dirty or contaminated spray chamber leads to poor drainage and uneven aspiration of the aerosol through to the plasma. This issue can be uncovered by watching how the solution runs down the inside of the spray chamber. The liquid should be running down the spray chamber as a uniform film. If there are droplets running down instead of a film, then the spray chamber is dirty.

Oils are particularly bad for spray chambers. Running samples containing oils causes variable spray chamber drainage. Flushing with clean solvent for a few minutes at the end of the analysis will flush the spray chamber. Some labs keep a separate set of sample introduction components for use with organic solvents or high matrix samples. These components can be installed as necessary. This preserves the life of the components used for everyday use with aqueous samples.

A dirty spray chamber leads to poor precision. Short-term precision can be monitored by reviewing the %RSD for the replicates per sample and medium-term precision can be monitored through the use of a QC solution. The problem is that QC solutions are often spaced 30 minutes apart. This means if the QC fails, all samples prior to the failure will need to be remeasured.

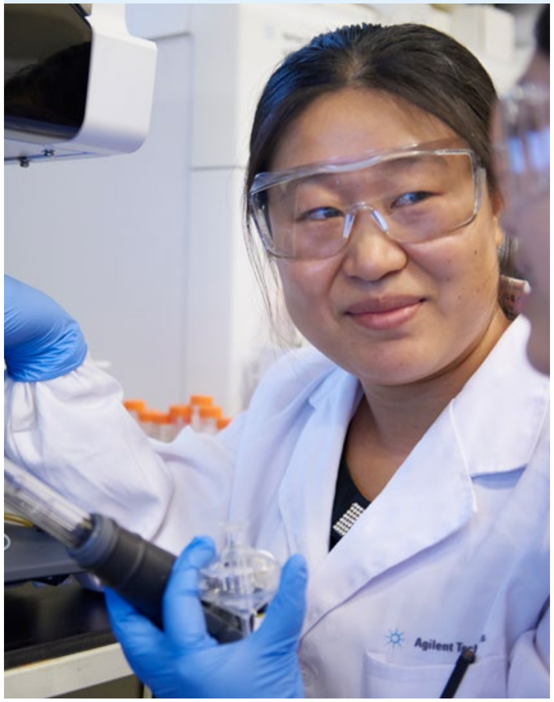
Include spray chamber cleaning as part of your routine maintenance. You should also run the automated instrument performance tests at the start of each day. This will determine if result precision is passing manufacturer's specification.

The Early Maintenance Feedback function of the Agilent 5800 and 5900 ICP-OES instruments can be used to set alerts to clean the spray chamber. If you are running oils, food samples, or other high matrix samples, you can set the alerts to appear after a few hundred samples. Running cleaner samples will allow you to set a sample count in the thousands to trigger an alert to clean the spray chamber.

Genuine Agilent Supplies

Agilent provides a range of high-quality ICP-OES supplies, including ICP-OES nebulizers and application kits, to support a range of atomic spectroscopy applications.

We supply application-specific parts and supplies such as peristaltic pump tubing and ICP-OES tubing kits for different solvents, as well as ICP-OES spray chambers, torches and gas filters.



Which ICP-OES best meets your needs?

Agilent 5800 ICP-OES

The 5800 has an ecosystem of embedded sensors and powerful processors, with smart algorithms and diagnostics, that identify problems that could impact results, preempt maintenance, and automate troubleshooting. Always working behind the scenes, the 5800 thinks like an expert, and can make recommendations and solve problems before they happen. This smart functionality reduces the number of samples you have to remeasure and gives you more confidence in your results.

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Agilent 5900 ICP-OES

The 5900 includes the smart features of the 5800, but offers the fastest sample measurement time of any ICP-OES, allowing you to increase revenue. The fast sample measurement time results in the lowest argon use, delivering more profits for your lab, and results you can confidently share with your customers.

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Agilent publication (5994-1278EN)

[Calibration Troubleshooting Checklist]
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