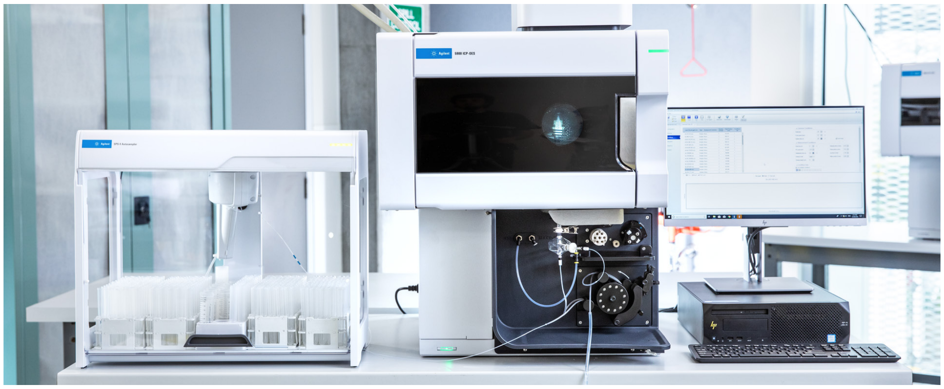
Executive Overview

Agilent
Trusted Answers

Maximize Your ICP-OES Instrument Performance and Uptime

A guide to maintenance of ICP-OES instruments to help you achieve the best analytical performance



Introduction

Agilent Technologies commissioned an independent global survey of laboratory managers with the primary objective being to understand laboratory and instrumental "pain points" and develop strategies for addressing any concerns. A secondary goal was to uncover key differences faced by these laboratory managers with respect to instrument operation. The survey, performed by Frost & Sullivan, involved 700 people across four countries: Germany, the UK, the USA and China; and the individuals surveyed varied with respect to experience, company size, role and primary function. The key findings of this research can be found at: www.agilent.com/about/newsroom/presrel/2017/07jun-ca17019.html

What became evident from the survey is that most users want to reduce maintenance and downtime, and improve overall workflow in the laboratory. Consequently, this article will outline some tips that can help maximize ICP-OES performance and address common challenges faced in the laboratory.

Prevent nebulizer blockage

How can you reduce or prevent nebulizer blockage? Remember, when dealing with sample nebulization, flows are usually relatively low. The fine capillary that carries the sample into the spray chamber has limited tolerance to undissolved solids and large particles. So, when running challenging samples there is a high risk of both annulus and nebulizer capillary blockage, leading to sensitivity problems. What can be done about this? First, and most crucially, make sure that the sample introduction system is rinsed with a suitable reagent blank before extinguishing the plasma. This will prevent any deposition taking place in the nebulizer itself. Second, consider your sample preparation strategies: filtering or centrifuging samples to remove particulates can help prevent nebulizer blockage. For challenging samples, the use of autosampler enclosures can also help prevent dust or dirt being transferred into samples whilst they are in storage and waiting for analysis. In addition, adjusting the autosampler probe height so that sampling occurs above any dissolved solid or precipitate can help reduce the chance of nebulizer blockage. The key word to remember is "prevention".

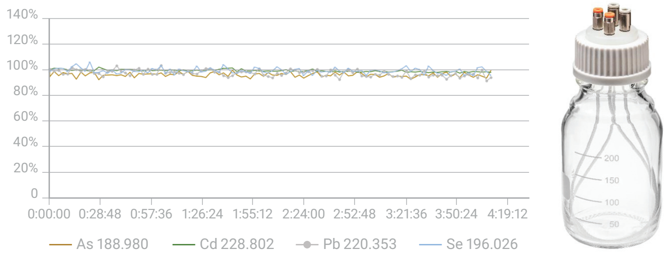
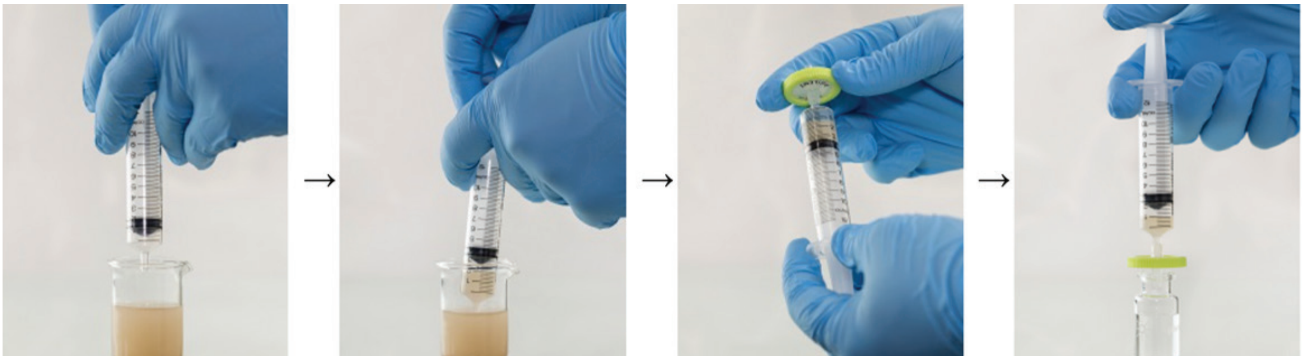


Figure 1. Long-term stability for 250 ppb multi-element standard in 25% NaCl with ICP-OES VDV configuration (axial mode) using the humidifier.

Another approach to reduce the chance of nebulizer blockage, particularly with challenging samples, is to use an argon humidifier accessory (Figure 1, right). The fine tubes inside the bottle are actually a permeable membrane. By filling the bottle with deionized water, the permeable membrane allows water to humidify the nebulizer gas. A moist nebulizer gas flowing through the nebulizer can help to reduce the chance of blockage due to salt build up and consequently reduce the amount of drift. Figure 1 (left) also shows an example of a challenging sample: 25% sodium chloride for over four hours with continuous aspiration – using a sample introduction system that is suitable for high-dissolved solids, including the argon humidifier accessory. Long-term stability with < 2.5% precision over that full period of the test is achieved.

A third approach is to review the nebulizer being used. The glass concentric nebulizer is the most common and while this offers good sensitivity, it is not ideal for samples with high TDS levels or large particles. For a challenging matrix, you can reduce blockages by selecting an alternate nebulizer. Agilent provides a selection tool that can recommend the best nebulizer for your application.

A fourth approach to reduce or prevent nebulizer blockage is to filter samples prior to analysis. Of course, most users should adopt this approach, but many prefer not to do so because it impacts on productivity. However, this approach is highly recommended. By way of example, Figure 2 shows the Agilent Captiva syringe filters need only four steps to realize the full benefits of filtration.



Before filling the sample, draw approximately 1 mL of air into the syringe. This will minimze fluid retention.

Draw your sample into the syringe, then draw in about 1 mL of air. Invert the syringe and wipe residue off tip.

Connect the syringe to the syringe filter using a luer connection. Twist gently to ensure a secure seal.

Filter syringe contents into a vial. Afterwards, remove the syringe filter, draw air into the syringe, reattach the syringe filter, and press the plunger to filter the residual sample. This will maximize sample recovery.

Figure 2. Recommended steps to filter samples using Agilent Captiva syringe filters.

In addition to filtering of samples, there are also some things to keep in mind from a sample preparation point of view that can first help to improve the accuracy of the results, but also reduce the chance of nebulizer blockage. Consider if you are working with the most appropriate digestion procedure. Are the analytes being quantitatively extracted and dissolved? In many cases, the digestions you work with may only be a partial extract and you may also face the potential that some volatile analytes may be lost during digestion. This is where it helps to take a certified reference material, preferably a solid certified reference material, through the sample preparation and analysis procedures. In this way, it can be checked whether any loss of analyte occurs during the digestion steps. In addition, you need to check whether the digestion remains stable or whether some precipitates or suspension occur after digestion, or any contamination occurs. To check this, you should work with a reagent blank. Ideally, it should be included with every sample batch; that is, take a pure water sample through the sample preparation process. With a pure water sample the expected results should be very close to zero. If higher values are observed, for any analyte in that particular sample, potential contamination is highlighted and further investigation to identify where the particular contamination was coming from would be required.

So far, strategies have been discussed to prevent nebulizer blockage, but blockages do still occur. The next question to address is how can blockages be removed? It is important to remember that nebulizers are fragile, so they should never be sonicated in an ultrasonic bath or cleaned with a cleaning wire (in the case of conventional glass concentric nebulizers or OneNeb nebulizers). To remove a nebulizer blockage, you should backflush the nebulizer using a nebulizer cleaning tool – this approach is very efficient and allows you to force some cleaning solution, typically methanol, through the tip of the nebulizer. Even without a cleaning tool the same effect can be achieved by applying suction on the back of the nebulizer; for example, using peristaltic pump tubing, or by applying a vacuum connection on the back of the nebulizer. With a very stubborn deposit in the nebulizer, soaking the nebulizer overnight in concentrated nitric acid is recommended.

Clean your sample introduction system

How are those other critical components in the sample introduction system of the ICP-OES cleaned and maintained? The first component to consider is the spray chamber. The glass cyclonic spray chamber is probably the most common type of spray chamber in use today on an ICP-OES system; in most cases, it will work efficiently, but over time droplets can build up on the walls of the spray chamber (Figure 3). In such an instance, the spray chamber needs to be cleaned immediately, as the droplet formation will affect precision. The best approach for cleaning the spray chamber is to soak it overnight (preferably for 24 hours) in a 25% detergent solution (Triton X-100, Decon, Fluka RBS 25 will all clean effectively). After cleaning, the spray chamber should be rinsed and returned to the instrument ready for the next analysis.

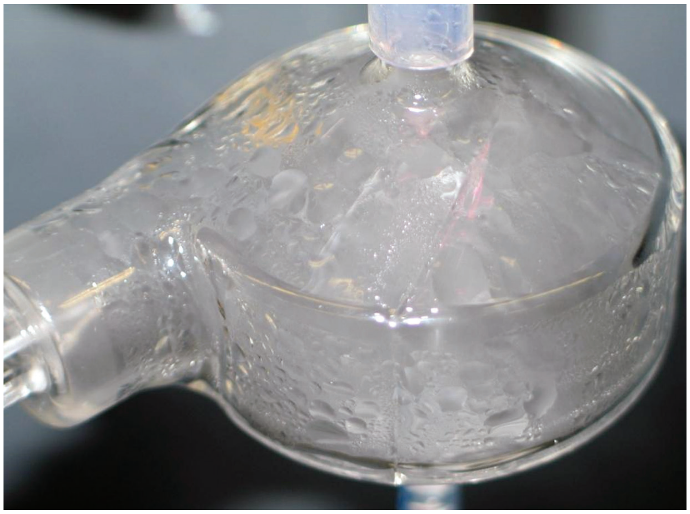
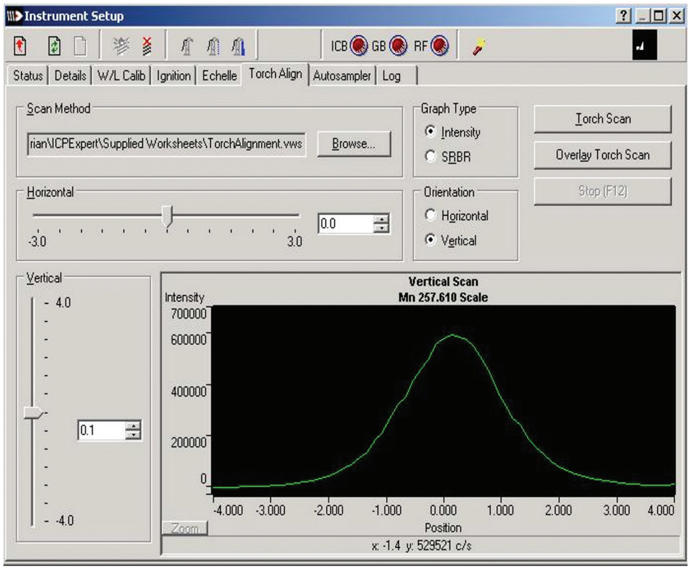


Figure 3. Clean the spray chamber when a build-up of droplets on the inside surface is visible, or if performance is degraded.

The next component to consider is the torch for the ICP-OES instrument. To clean the torch of the Agilent 5000 Series ICP-OES instrument, the outer tube should be soaked in aqua regia (mixture of hydrochloric acid and nitric acid) for one hour – Agilent offers a very convenient cleaning stand for this purpose. After cleaning, both the inside and outside of the torch should be rinsed with de-ionized water and compressed gas (air, nitrogen or argon) pumped through the three gas supply ports to remove any remaining liquid.

For older systems, such as the Agilent 700 Series ICP-OES instrument, the process is virtually the same, except that the torch should be soaked overnight to remove any deposition that has taken place. Again, after cleaning it should be rinsed thoroughly to remove any remaining liquid and then, importantly, dried carefully before being returned to the instrument.

On the 700 Series ICP-OES instruments the torch needs to be positioned manually, so after placing the torch into the torch holder the setting should be checked – the distance between the RF coil and the intermediate tube should be between 2 to 3 mm. This will ensure the correct location for efficient plasma formation and efficient sample excitation. The torch alignment routine also provides a means to verify that the torch is in the correct location – this routine allows both the vertical and horizontal positioning of the torch to be set, ensuring that the instrument is looking at the highest intensity region from the torch (Figure 4). This can also be a very useful way to do a quick performance check on the instrument because the maximum intensity should be consistent from day-to-day. Any changes in the intensity readings for the sample provides an indication that there is a potential blockage somewhere else in the instrument. Similarly, the ideal settings for the horizontal and vertical position of the torch should also remain fairly consistent and, again, sudden changes in these could indicate another issue with the torch.



Scan Method: rian\ICPExpert\Supplied Worksheets\TorchAlignment.vws

Graph Type: ● Intensity ○ SRBR

Horizontal: 0.0

Orientation: ○ Horizontal ● Vertical

Vertical: 0.1

x: -1.4 y: 529521 c/s

Figure 4. The torch alignment scan allows you to optimize the viewing positions of the torch to ensure the optimum part of the plasma is being viewed.

Prepare accurate standards

ICP-OES is widely used for fast, multi-element analysis in environmental, industrial, agricultural, and materials science applications. Its broad dynamic range and ability to handle complex matrices make it a powerful tool, but its accuracy hinges on well-prepared, matrix-appropriate calibration standards. Because the instrument measures emission intensity for the unknown samples relative to your prepared calibration standards, any inconsistencies in standard concentration or matrix compatibility can directly affect calibration linearity, detection limits, and spectral resolution. In high-throughput or regulated labs, this can lead to poor recoveries, invalid data, or costly sample reruns.

What are the different quality standards (RM versus CRM)?

In atomic spectroscopy workflows, both Reference Materials (RMs) and Certified Reference Materials (CRMs) are valuable, depending on the precision required. When choosing a standard, it is important to understand the distinction between RMs and CRMs.

Both RMs and CRMs are tested using high-performance instrumentation and come with a Certificate of Analysis. One major difference is that CRMs are subjected to additional quality control steps, including testing for homogeneity and long-term stability (typically over 2 to 3 years). CRMs are then certified for homogeneity and long-term stability, making them essential for high-precision applications and or for regulated methods where full traceability and defined measurement uncertainty are required.

RMs are manufactured in the same ISO 17025 and 17034 accredited facilities as CRMs, ensuring they are traceable, tested, and a cost-effective alternative for routine analysis, method development, or internal quality control checks where enhanced homogeneity or stability testing past their shelf life is not critical. Choosing the right level of certification depends on whether your lab needs ISO/IEC 17025 traceability and regulatory compliance or simply reliable, consistent performance.

Agilent CrossLab

From Insight to Outcome

Agilent Inorganic Certified Reference Materials and Standards

Your essential resource for single, multi-element and metallo-organic standards for spectroscopy and IC instrumentation

Agilent

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Figure 5. Agilent offers a offers a comprehensive portfolio of analytical standards, spectroscopy standards, reference materials, and certified reference materials for environmental, food safety, elemental analysis, instrument calibration, tuning mixes, and more.

Agilent standards manufacturing process

Agilent CRM and RM standards are formulated and manufactured using rigorous quality controls to ensure exceptional purity, accuracy, and full traceability. All standards are gravimetrically prepared using ultra-pure raw materials and typically validated for trace-level impurities using Agilent ICP-MS systems. Production occurs in ISO-9001 certified facilities, with testing performed in ISO/IEC 17025-accredited laboratories. Certification complies with ISO 17034 guidelines, ensuring traceability to the NIST SRM 3100 series elemental standards. The final products are packaged in acid-leached, pre-cleaned HDPE bottles and sealed in protective bags to guard against contamination during shipping and storage. All RMs and CRMs are backed by stability studies demonstrating a shelf life of one year or more. RMs and CRMs are both supplied with a comprehensive Certificate of Analysis (CoA) with certified concentrations. The CoA for the CRMs provides expanded data, including measurement uncertainty and trace impurity levels for up to 68 elements.

Tips for preparing standards for your workflow

Proper preparation of calibration standards is critical to ensuring accurate, reproducible, and traceable results with your analyses. Even small mistakes with dilution, storage, or matrix matching can introduce significant errors, especially when working at trace concentrations or with complex sample matrices.

Follow these essential lab practices when using or preparing standards to help improve the accuracy and reliability of your calibration standards:

- **Use in-date standards:** Always check and use standards within their expiration date (especially important for low-concentration or organic-based solutions). Expired standards may have low concentrations due to degradation or precipitation.
- **Calibrate pipettes and glassware:** Regularly calibrate and verify your pipettes and glassware. Regular verification ensures accurate volume delivery and minimizes dilution errors.
- **Perform serial dilutions:** Avoid large single-step dilutions to reduce errors. Use intermediate dilution steps to improve accuracy and reproducibility
- **Stabilize working standards:** Acidify your prepared standards using the appropriate acid, based on the analyte, to stabilize metal ions and extend shelf life. Follow supplier guidance on acid type (e.g. HNO_3, HCl, HF, or mixed acids), concentration and compatibility.
- **Matrix match:** Prepared calibration standards should closely match the acid matrix in which your samples are prepared to avoid ionization or other enhancement/suppression effects in the atomizer.
- **Storage:** Store your prepared standards in pre-cleaned, high-purity HDPE or PFA containers. Avoid glass containers to prevent contamination and adsorption losses.
- **Reduce contamination risks:** Use only natural (uncolored) pipette tips to avoid trace metal contamination (especially for elements such as zinc, cadmium, and iron).
- **Prepare fresh standards as needed:** Prepare fresh standards frequently (daily for ultra-trace work). This is particularly important for volatile or unstable elements, or when working at trace concentrations (< 1 ppm).
- **QC Checks:** Include reagent blanks with each batch to check for potential background contamination. Use spike recoveries and matrix spikes to confirm method performance and identify matrix effects.

Reducing potential contamination

Contamination can distort sensitivity and skew calibration curves. It can occur at any point in the workflow: storage, handling, digestion, dilution, or measurement. Common sources include working with poor quality (impure) reagents, improperly maintained water systems, colored pipette tips, or leachable elements from solutions stored in glassware. Cross-contamination between high and low concentration standards can also occur.

Follow these essential lab practices when using or preparing standards to help minimize contamination:

- **Review the CoA:** Always check the CoA for every reagent and consumable used during sample preparation for potential contaminants. Even between different batches of the same product, impurity levels can vary significantly.
- **Use ultra-pure 18.2 MΩ·cm deionized water**.
- **Use natural (uncolored) pipette tips:** While colored tips may add interest in the lab, use only natural (uncolored) pipette tips to avoid trace metal contamination (especially for elements like zinc, cadmium, and iron).
- **Validate digestion workflows:** Choose your digestion method (microwave, hot-block, acid) based on sample matrix and target analytes. Verify accuracy by running a CRM through the full digestion and analysis workflow to verify recovery and accuracy.
- **Use QC checks in each batch:** Include reagent blanks with each batch to check for potential background contamination. Use spike recoveries and matrix spikes to confirm method performance and identify matrix effects.

Why select Agilent standards?

Agilent standards are trusted worldwide for their exceptional purity, traceability, and performance. Offering both single- and multi-element RMs and CRMs, Agilent supports a wide range of applications, including heavy metals in wastewater, nutritional elements in food, and wear metals in petroleum products. Agilent also offers specialty metallo-organic, biodiesel, and solvent-based standards, along with tuning and wavelength calibration solutions to verify instrument performance.

In addition, Agilent can offer custom inorganic standards allowing you to order the exact elements required in the concentrations needed, together with the matrix that you want those standards in, with assured fast delivery. Custom standards are manufactured to your specific requirements, then are qualified and certified by our team of expert chemists in our ISO 9001, 17025, and Guide 34 accredited facility.

Sourcing your standards from a trusted provider like Agilent is essential. Backed by detailed impurity profiles, NIST traceability, and rigorous quality control, Agilent delivers consistent, reliable standards to ensure confidence in your analytical results.

Don't neglect the pump tubing

Another area for potential concern is peristaltic pump tubing – critical to achieve the best performance from our instrumentation. Ideally the waste tubing should have a larger internal diameter (id) than the sample tubing to make sure that you are getting efficient removal of waste liquid from the sample introduction system (Figure 6). The tubing also needs to be resistant to the solvent that you are using. PVC tubing is good for most types of acid digests, but when working with an organic solvent you need to pay more attention to chemical compatibility, and you may need to work with Viton or Marprene pump tubing to ensure solvent resistance. It is always good practice to clean new tubing to remove potential contamination and the tubing should be replaced regularly. Old pump tubing can cause lots of issues, particularly with precision, stability and drift. As a general guideline, if you are running your instrument five days a week, then you should expect to replace the tubing at least once a week. The most critical thing is that when you are finished with your analysis, take the pump tubing out of the pump holders, and release the pressure bar and remove it from its location around the pump. That will enable the tubing to relax and recover a little. Before you refit it on the instrument roll the tubing between your fingers to determine if there are any flat spots on the tube. If it looks obviously worn, or is stretched (Figure 7), then make sure you replace it immediately.

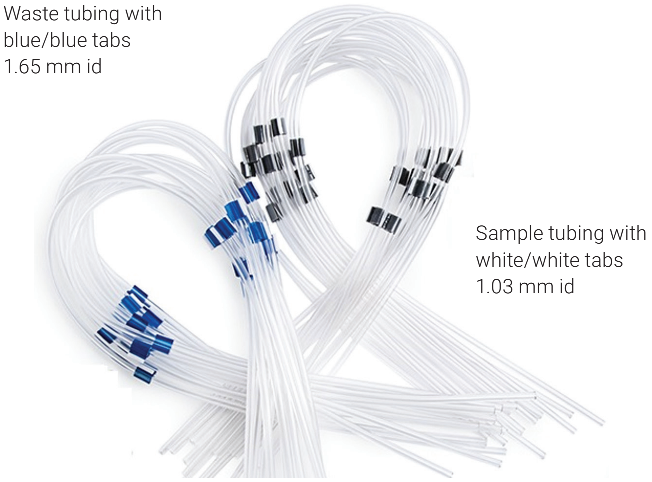


Figure 6. Default tubing recommended for most applications is PVC peristaltic pump tubing.

Figure 7. Regularly check the condition of the peristaltic pump tubing, looking for stretching, flat spots, discoloration, or general wear – and replace if you are uncertain.

In fact, if you have any doubts at all about the tubing, then replace it immediately. Erratic flow of liquid into the sample introduction system can occur if you haven not applied the correct pressure on the pump tubing. Remember not to overtighten the tubing – as long as it is pumping smoothly and efficiently that is all that is required. If any bubbles are seen in the liquid stream, then you should check for any loose connectors or a leak somewhere in the system. If the flow of liquid coming through the nebulizer is not consistent, then that is an indication that you have plugging in your sample introduction somewhere. At that point, check whether those components need cleaning.

Importantly, at the end of the analysis there are some simple steps that should be taken to get better lifetime out of your pump tubing and also reduce the chance of nebulizer or injector blockage. Make sure you aspirate a suitable rinse solution for a few moments before shutting off the plasma. This will help to prevent any sample deposition in the tip of the nebulizer. You can then shut off the plasma after making sure that you pump out any remaining liquid from the pump tubing and from the sample introduction system. Next, you can take the tubing out of the pressure bars on the instrument and remove the bridges from the securing slots such that the tubing is no longer stretched over the rollers in the pump and giving it a chance to recover. You should then empty the waste vessel and leave the instrument in standby mode, enabling the fastest possible start-up time.

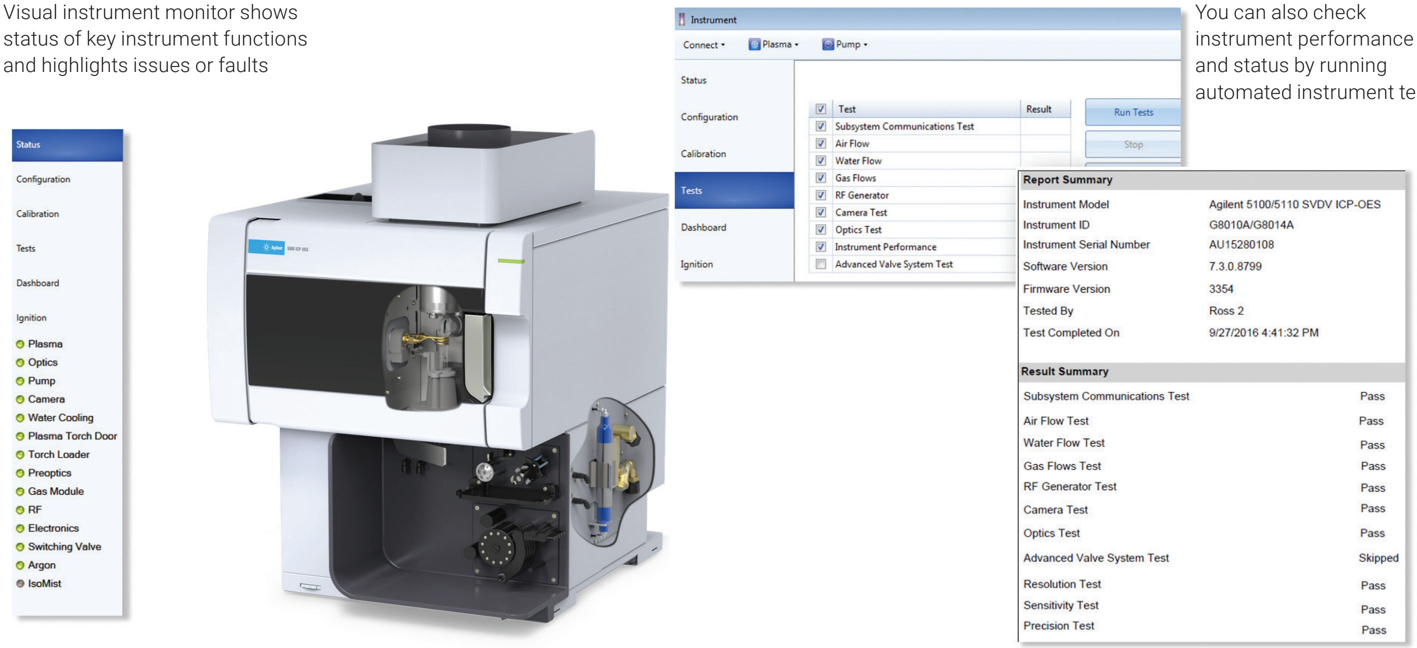
Check analytical sensitivity

Now look at wavelength calibration. This is something that you should be doing periodically – typically about once a month. It is a process that allows the instrument to relate the positioning of actual emission signals to the actual pixels on the detector chip. To do this, Agilent recommends working with the Agilent pre-prepared wavelength calibration solution, as this means there is no chance of any missing components giving improved reproducibility with the convenience of a pre-mixed solution. If you see a wavelength calibration value that is typically < 100%, the first thing to consider is whether the sample has reached the plasma. The next is to verify that the optics boost purge is enabled and stable. In many cases that is the first thing to be forgotten – the boost purge has not been on long enough and so you miss some of the wavelengths in the UV region.

In addition to the wavelength calibration routine, there are several other approaches you can use to check the health of your particular instrument. With the current software available for Agilent ICP-OES instruments, there is a visual monitor that indicates the status of the current instrument functions and highlights immediately whether there are any particular issues (Figure 8). In addition, the software gives you the capability to run performance tests at any time and this can be a convenient way to verify that your instrument is performing correctly.

You may not choose to run a full sequence of tests – you can simply choose the ones you want. For example, by focusing on the instrument performance tests you can see immediately whether there are any potential issues with sensitivity or precision, which could highlight a potential issue with your sample introduction system. This is a quick and convenient way to check the condition of your instrument before you start a long-term analysis.

There are also several other sensors throughout the instrument that can be invaluable for performing diagnostics or troubleshooting. The gauges will indicate immediately if there are any issues; for example, looking at the nebulizer backpressure, you can see immediately whether you are starting to see any build-up occurring in the nebulizer. As the build-up or blockage starts to form, you will start to see the backpressure in the nebulizer go up. Now, the instrument will flag that for you and stop the analysis if it gets too high, but even before then, you can still periodically check through the dashboard to see the health of your particular instrument. You will see there is a nebulizer test that will give you the capability to test the performance of your nebulizer before you start your analysis.



Visual instrument monitor shows status of key instrument functions and highlights issues or faults

You can also check instrument performance and status by running automated instrument tests

☑ Test	Result
☑ Subsystem Communications Test	
☑ Air Flow	
☑ Water Flow	
☑ Gas Flows	
☑ RF Generator	
☑ Camera Test	
☑ Optics Test	
☑ Instrument Performance	
☐ Advanced Valve System Test	

Report Summary

Instrument Model	Agilent 5100/5110 SVDV ICP-OES
Instrument ID	G8010A/G8014A
Instrument Serial Number	AU15280108
Software Version	7.3.0.8799
Firmware Version	3354
Tested By	Ross 2
Test Completed On	9/27/2016 4:41:32 PM

Result Summary

Subsystem Communications Test	Pass
Air Flow Test	Pass
Water Flow Test	Pass
Gas Flows Test	Pass
RF Generator Test	Pass
Camera Test	Pass
Optics Test	Pass
Advanced Valve System Test	Skipped
Resolution Test	Pass
Sensitivity Test	Pass
Precision Test	Pass

Figure 8. View the interactive Instrument Status page to check the current status of the instrument and check instrument performance.

Another challenge, particularly for a novice user, is knowing which wavelengths to use for method development. Again, there are software tools available in the instrumentation that can help you to simplify this process. One of the most important of those is the Intelliquant software tool for the Agilent 5000 Series ICP-OES. This allows you to run a sample and create a heat map that highlights the relative concentrations of all the elements that have been found (Figure 9). From a method development perspective this is invaluable. The Intelliquant tool will also give an indication of the concentrations of those particular analytes, which you can see visually through the spectrum displayed to the user.

Figure 9. IntelliQuant is a fast semiquantitative data acquisition routine for ICP-OES that can collect data across the entire spectral range to provide quick sample insight.

To help you with quantification when you are using this tool, Agilent has a range of calibration standards that are used with the Intelliquant procedure to provide improved accuracy. These standards are available in a kit, but can also be purchased individually. Running these standards will improve the quantification of the default calibration to give better accuracy when using the software. Although this software capability is not supported with earlier Agilent 700 Series ICP-OES instruments, the Intelliquant standards can be used with the semi-quantitative worksheets to provide similar capabilities.

Another common challenge that many users face is measuring samples of low concentration. How can you get better sensitivity for these low concentration analytes? One simple thing you can do is use a longer read time when you are doing your replicate readings. Going from a 1 to a 5-second read can give you more accurate determination of the signal and, of course, going to an even longer read time will enable you to get more accurate determination for your sample. This means that you will be able to get a significant improvement in your detection limit capability.

Another approach suitable for samples with a clean matrix is to change the type of spray chamber that is fitted on your instrument. Going to a single-pass design, that has no internal baffle will improve sample transport efficiency resulting in more sample into the plasma and a significant improvement in both signal and detection limits. As shown for arsenic, selenium and lead, changing to a single-pass spray chamber can give up to a 2x improvement in detection limit capability (Figure 10).

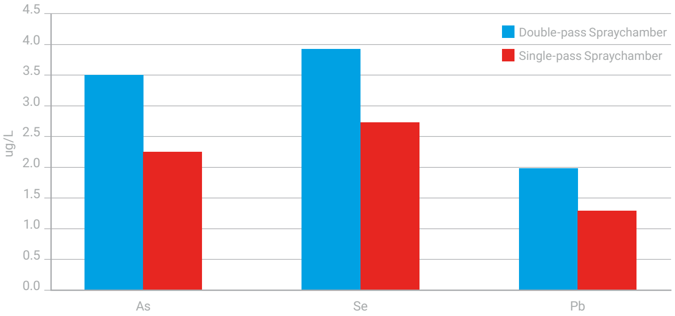


Figure 10. The single-pass glass cyclonic spray chamber provides higher sensitivity and faster washout with clean matrices, improving detection limits (30 second axially viewed).

For elements that form hydrides, particularly elements such as arsenic, selenium and mercury, you can switch to a multimode sample introduction system (MSIS). This is a spray chamber used with ICP-OES and MP-AES instruments that enables the simultaneous determination of routine elements at the same time as measuring hydride-forming elements; that is, preparing one sample digest and potentially measuring all the elements from that single digest (Figure 11). Using a four-channel pump enables one to work with this device. Alternatively, you can use a standalone peristaltic pump if you do not have a four-channel pump on the instrument. Sample is pumped through the nebulizer in the conventional way and that is used to do determinations of routine elements. For hydride-forming elements, pump some of that sample in through the bottom of the spray chamber and pump a suitable reductant in through the top of the spray chamber. The sample and reductant combine enabling us to form the hydride or chemically separate the analyte from the matrix. The hydride vapor is then swept into the plasma along with the sample aerosol allowing simultaneous determination.

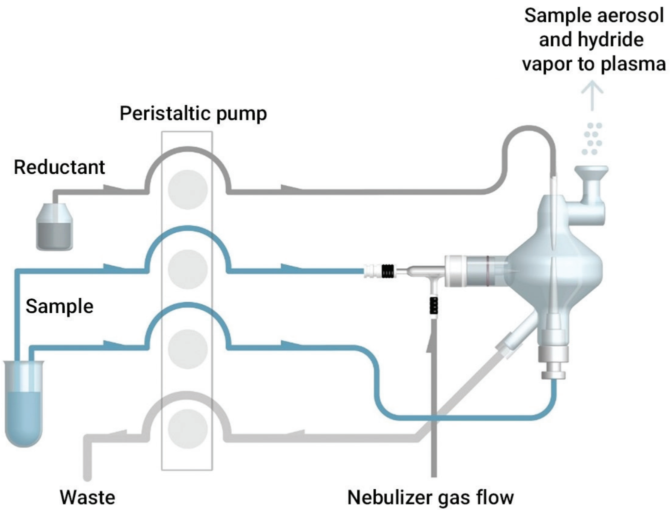


Figure 11. Schematic of MSIS configured for simultaneous analysis of hydride elements and conventional elements.

So, why do that? Well, the benefit is improved detection limit capability particularly for those challenging elements that have generally low sensitivity – elements such as arsenic, selenium, antimony, and mercury. Working with an MSIS system can offer an order of magnitude improvement in detection limit, that is, down in the sub-ppb range, enabling more accurate determination of those analytes. Other element detection limit performance is relatively unaffected allowing good performance for all the routine elements and significant improvements for more challenging elements.

Routine maintenance

Here are some recommended maintenance schedules to ensure you are getting the best performance out of your ICP-OES instrument:

Daily

Check your exhaust system and your argon gas pressures, check the sample introduction system for any potential blockage whether it is in the injector, torch, or in the nebulizer. Check the peristaltic pump tubing for excess stretching or any flat spots and do a visual inspection of the spray chamber for any liquid droplets building up on the inside to make sure that it's draining smoothly and efficiently.

Weekly

Clean the sample introduction components, such as the bonnet on the torch or the pre-optics cone on the axial ICP, and check the water level in the chiller that is being used with your ICP-OES instrument.

Monthly

Clean the spray chamber and the nebulizer and, in the process, check all the other transfer tubing to make sure it is in good condition, replacing any components that are worn. Inspect the air filters on the instrument and chiller to make sure they are clean, removing any excess dust or dirt that has built up to ensure efficient cooling of both the instrument and the cooling water. The water filter on the instrument should be cleaned periodically, and you may need to change the argon filters. In many cases, these jobs will have been completed by an Agilent Field Service Engineer as part of a preventative maintenance program, so if you have a service contract, these tasks will be reduced and simplified.

Other common issues

Issues with plasma ignition are often dicussed. Generally, the most common cause for a problem with plasma ignition is an air leak in the sample introduction system – you want to check for any loose fittings, any damaged components or any fittings that perhaps were not fitted correctly on the instrument (particularly for older instruments). On current instruments, such as the 5000 Series ICP-OES, that is no longer an issue because connections are made automatically. For the same reasoning, check the positioning of the torch on older generation instruments, again just to make sure that it is in the right place.

Another common issue revolves around memory effects typically seen after measuring high concentrations (Figure 12). It is seen with a range of common elements including boron, mercury, molybdenum, strontium, and zinc where the first replicate on a low concentration sample is high, then the subsequent readings are much lower, leading to problems with precision. To solve this, make sure to use a matrix-matched acidified rinse solution and work with a suitable rinse time. It should be at least 30 seconds for most applications. If you are facing more challenges with this, there are other approaches you can use; for example, you can use smart rinse capability in the software to help you optimize and monitor the washout of a particular analyte. You could go to a switching valve, which will help improve the washout characteristics, or you could use a different type of spray chamber that has better washout characteristics.

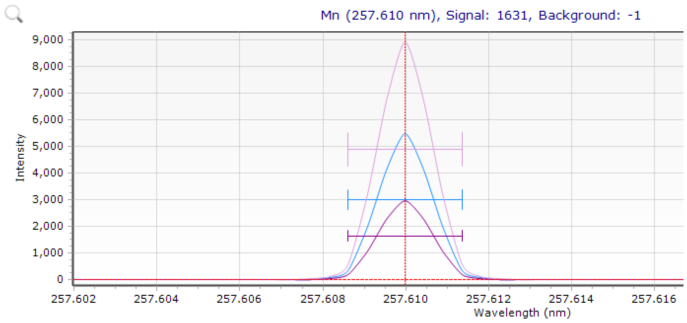


Figure 12. High intensity for the first replicate with more consistent results for subsequent readings indicates memory effects.

If you are working with an autosampler on your ICP-OES instrument, then you need to consider potential issues that can arise. For example, the need for a longer transfer tube connecting the two systems can give more problems such as a longer uptake time or problems with memory effects. Ideally, you want to enable the fast pump to minimize the uptake delay. Work with a suitable probe – if you are running samples with high levels of dissolved solids or more viscous samples, you need to use a wider bore probe on the instrument to make sure there's no blockage in the probe.

Also think about the chance of something happening to your samples whilst sitting on the autosampler waiting for analysis. This could be contamination from dust or dirt in the laboratory, or it could be evaporation of the sample leading to preconcentration of the sample. It could also be sample precipitating out in the sample vial while it is waiting for analysis. Think about these issues and adopt suitable approaches to address them.

Finally, Agilent recommends that users have spare sample introduction components to help them maintain operation of their ICP-OES instrument. Components such as spare tubing, torches, nebulizers, and spray chambers will enable you to keep your instrument running if you have a blockage or damage to some of those particular components. Having a spare on hand means you can replace that component and continue with your analysis while fixing the initial problem. Agilent offers a range of consumable kits to support ICP-OES instrumentation and this can be a convenient way to have essential supplies available to support routine operation of your instrument. These kits are available for the 5000 Series ICP-OES instrument (Table 1).

The Agilent atomic spectroscopy lineup

Figure 13 highlights Agilent's atomic spectroscopy instrumentation lineup. Driven by insights from customers from around the world, Agilent has created an innovative atomic spectroscopy portfolio – spanning instruments, consumables, standards, services, software, and more – to bring that confidence to your lab. Agilent offers a comprehensive range of instrumentation ranging from flame AA systems all the way through to high-end ICP-QQQ ICP-MS that provides MS/MS capability to enable unique control of interference removal in reaction mode. Regardless of the technique that you are working with, regardless of the type of analyses that you are doing, you can be confident that Agilent will have the products that you need for your particular application.

Table 1. ICP-OES operating supplies kits include the recommended supplies to support up to 12 months of routine operation of your instrument.

Components in the Operating Supplies Kit
1 Easyfit demountable torch with 2 spare outer tubes
4 packs pump tubing (for sample + waste)
1 pack int. standard pump tubing and connectors
SeaSpray nebulizer (U series)
1 pack of 0.75 mm id Unifit sample connectors for nebulizer
1 x Ezylok gas connector for nebulizer
Double pass spray chamber with torque-controlled nebulizer adaptor
1 pack of Unifit connectors for spray chamber drain
Spare torch clamp for the spray chamber ball joint socket
Spare pre-optic window (axial kit includes an extra window for the radial view)
Spare O-ring or washer for the pre-optic window
Capillary tubing for sample inlet
Nebulizer gas supply tubing
Drain tubing for spray chamber with 2 x barb connectors
Drain tubing for instrument spill tray
Replacement seal for spray chamber nebulizer inlet

Figure 13. Agilent's atomic spectroscopy instrumentation lineup.

Resources

- ICP-OES resource page
- Troubleshooting videos
- Agilent Community support forum
- Essential ICP-OES Instrument Supplies
- ICP-OES parts and supplies
- Agilent atomic spectroscopy application notes
- Agilent Spectroscopy consumables catalog
- Agilent high quality Inorganic and Metallo-Organic standards for Atomic Spectroscopy
- Agilent MSIS Technical Overview
- Agilent recorded webinars for atomic spectroscopy

Summary

This overview summarizes some of the challenges that you may be facing in your laboratory. We have highlighted how most of the potential causes for failure or unplanned downtime in your laboratory can be traced back to issues in the sample introduction system. So by focusing your maintenance in the sample introduction area, you can reduce that risk of unplanned downtime. We have also included some guidelines and maintenance procedures that you can use to set up standard operating procedures in your laboratory which will help prevent these types of issues from reoccurring.

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