

Maximize Your ICP-OES Instrument Performance and Uptime

Tips, tricks and good advice for ensuring your ICP-OES instuments are optimized for best performance, and your methods and applications are robust and reliable.



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.

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Prevent Nebulizer Blockage

How can we 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 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'.

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

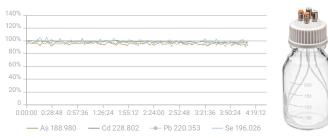


Figure 1.

the argon humidifier accessory. Long-term stability with < 2.5% precision over that full period of the test is achieved.

A third 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 realise the full benefits of filtration.

Now in addition to filtering of samples, there are also some things we should 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. We need to consider if we're working with the most appropriate digestion procedure. Are the analytes being quantitatively extracted and dissolved? In many cases the digestions that we work with may only be a partial extract and we may also face the potential that some volatile analytes may be lost during digestion. This is where we should take a certified reference material, preferably a solid certified reference material, through the sample preparation and analysis procedures. In this way we can check whether any



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.

loss of analyte occurs during the digestion steps. In addition, we 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, we've discussed strategies 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 do we clean and maintain those other critical components in the sample introduction system of the ICP-OES? The first component to consider is the spray chamber. The glass cyclonic spray chamber is probably the most common type of spray chamber that's in use today on an ICP-OES system and 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.

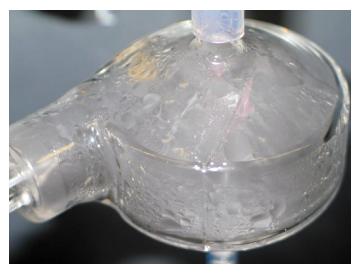


Figure 3.

The next component to consider is the torch for the ICP-OES instrument. To clean the torch of the Agilent 5100 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 2-3 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's 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.

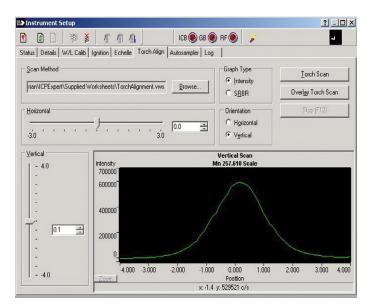


Figure 4.

Prepare Accurate Standards

Now let's think about another approach that we can use to get the best performance out of our instrument - how we prepare our calibration standards. For accurate quantification, we always need a standard of known composition to calibrate the instrument, so we can measure the unknown in your particular sample. The accuracy of your analysis is totally dependent on how well prepared your particular standards are. Any errors made or contamination introduced during preparation will lead to inaccurate results and other issues. This may lead to instrument downtime while trying to troubleshoot those issues. You might spend time preparing fresh standards and re-measuring samples and in doing so, run the instrument longer leading to faster or premature replacement of instrument supplies. If you're in an accredited laboratory, the worst-case scenario would be failure of an audit and loss of ISO lab accreditation. The stakes here are very high.

That's why we recommend that you work with certified reference materials when preparing calibration standards. Agilent offers such certified reference materials that are manufactured to ISO 9001 Guide ISO 34 and certified in an ISO 17025 testing laboratory (Figure 5). In addition, all raw materials and solvents are tested for impurities prior to preparation. The standards are traceable – they are certified using high-performance test protocols from the National Institute of Standards and Technology (NIST) in the United States, and all Agilent standards or certified reference materials are directly traceable back to the NIST 3100 Series of standard reference materials. The standards are contamination free because they are packaged in



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pre-cleaned, high-purity, high-density polyethylene bottles sealed to prevent any chance of contamination in shipment. Most have a shelf life of 18-24 months, supported by shortand long-term stability studies, and come with thorough confirmation. Impurity testing is achieved using an Agilent ICP-MS and actual impurity levels are reported for up to 68 trace impurities.

Single- and multi-element standards are available covering a range of different concentrations suitable for use with both atomic absorption, ICP-OES and ICP-MS. A comprehensive range of metallo-organic standards is also available for laboratories that, for example, measure oil or biodiesel samples. A range of tuning and calibration standards for both Agilent and PerkinElmer instrumentation is also available.

There are some simple steps you can take to improve the preparation of standards and to help you improve accuracy. First, make sure that the standards are still within the use-by date and you're working with calibrated pipettes and Grade A volumetric flasks. Periodically, it's good practice to verify the accuracy and reproducibility of these pipettes and make sure a series of small dilutions is performed rather than one large one, to achieve the best overall accuracy. Next, think about the concentration of your standards. Low concentration standards have a finite life so, if working at ppb and sub-ppb concentrations, it is recommended that you prepare new standards from a higher concentration stock every time you do an analysis. How are your standards being stored? Plastic vessels, particularly PFA or FEP vessels, provide much better stability but that is applicable only if we remember to stabilize our standards during preparation by adding extra acid.

In addition to preparing accurate standards we need to think about approaches that reduce the potential for contamination. Contamination can come from anything that comes into contact with the sample whether it's during storage, digestion, dilution or analysis. What are the key approaches? First, check the purity of the reagents this is where the certificate of analysis can really help. The certificate of analysis for an Agilent certified reference material highlights all potential impurities and actual concentrations reported for those particular impurities. This allows you to immediately see if the particular reagent or standard is likely to give you an issue with your analysis. What about other potential causes of contamination? There's the reagent water used in the laboratory - remember that plastic containers, FEP or PFA containers, are preferred because they can help to reduce the chance of contamination, particularly from borosilicate glass. Lots of analysts in the laboratory work with coloured pipette tips, but they can also introduce significant levels of contamination, particularly for zinc, cadmium, iron and copper. Consequently, it's best to work with natural tips to help you reduce that chance of contamination.

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 ID than the sample tubing to make sure that you're getting efficient removal of waste liquid from the sample introduction system (Figure 6). The tubing also needs to be resistant to the solvent that's in use. 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's 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're 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're 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.

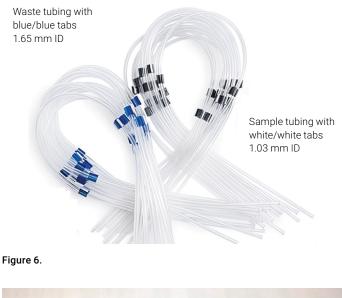




Figure 7.

In fact, if you've got 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't applied the correct pressure on the pump tubing. Remember not to overtighten the tubing – as long as it is pumping smoothly and efficiently that's all that's 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's an indication that you've got plugging in your sample introduction somewhere. Then we need to 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 our 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. We can then shut off the plasma after making sure that we pump out any remaining liquid from the pump tubing and from the sample introduction system. Next, we 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, let's 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 we recommend 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's 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's often the first thing to be forgotten – we haven't had the boost purge on long enough and so we miss some of the wavelengths in the UV region.

In addition to the wavelength calibration routine there are several other approaches that you can use to check the health of your particular instrument. With the current software that's available for Agilent ICP-OES instruments there's a visual monitor that tells you 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 for you 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. So this is a very 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 also be invaluable when you're performing diagnostics or troubleshooting. The gauges will tell you immediately whether or not there are any particular issues; for example, looking at the nebulizer backpressure you can see immediately whether you're starting to see any build up occurring in the nebulizer. As the build up or blockage starts to form, you'll 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'll see there is a nebulizer test that will give you the capability to test the performance of your nebulizer before you start your analysis.

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 5110 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.

To help you with quantification when you're using this tool Agilent has a range of calibration standards that are used with the Intelliquant procedure to provide improved accuracy.

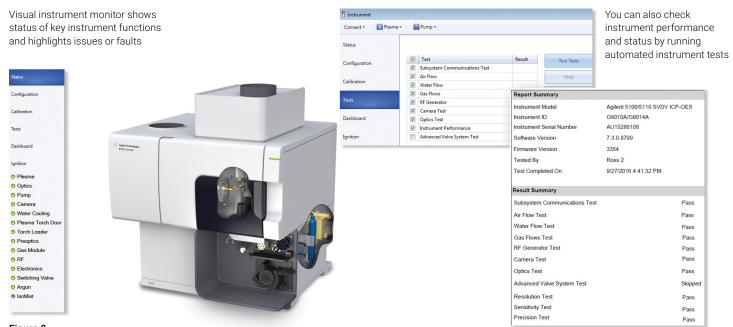






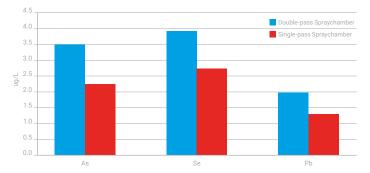
Figure 9.

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 Intelliguant 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 very simple thing that you can do is use a longer read time when you're 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 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 two-times improvement in detection limit capability (Figure 10).

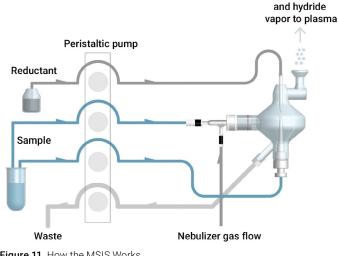
For elements that form hydrides, particularly elements such as arsenic, selenium and mercury, you can switch to a multi-mode sample introduction system (MSIS). This is a spray chamber that we use with ICP-OES and microwave plasma that gives us the capability for simultaneous determination of routine 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). We need a four-channel

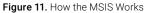




pump to enable us to work with this device. Alternatively, we can use a standalone peristaltic pump if we don't have a four-channel pump on the instrument. Sample is pumped through the nebulizer in the conventional way and we use that to do determinations of our routine elements. For hydride-forming elements we 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 vapour is then swept into the plasma along with the sample aerosol allowing simultaneous determination.

So, why would we do that? Well, the benefit is improved detection limit capability particularly for those challenging elements where we have generally low sensitivity; elements such as arsenic, selenium, antimony, 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. Sample aerosol





Routine Maintenance

Here are some recommended maintenance schedules to ensure you're 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's 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's 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's in good condition, replacing any components that are worn. Inspect the air filters on the instrument and chiller to make sure they're 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 the Agilent Field Service Engineer as part of a preventative maintenance programme so if you've got a service contract maybe these are tasks you won't need to do.

Other Common Issues

Issues with plasma ignition are often asked about. 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 weren't fitted correctly on the instrument (particularly for older instruments). On current instruments such as the 5100 Series ICP-OES that's 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's in the right place. Another common issue is if you've used the emergency stop button on the instrument you'll need to reset it manually otherwise it will inhibit ignition of the plasma.

Another common question revolves around memory effects typically seen when measuring high concentrations (Figure 12). We see it with a range of common elements including boron, mercury, molybdenum, strontium and zinc where the first replicate on a low concentration sample is high and 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're 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.

If you're 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're 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 pre-concentration of the sample. It could also be sample precipitating out in the sample vial whilst it's waiting for analysis. Think about these issues and adopt suitable approaches to address them.

Finally, we really recommend 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 5100 Series ICP-OES instrument (Figure 13) as well as for older generation instruments such as the 700 Series instruments.

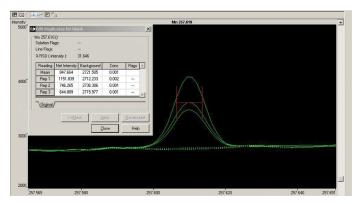


Figure 12.

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 0.75mm id Unifit sample connectors for nebulizer

1 x Ezylok gas connector for nebulizer

Twister spray chamber with Helix seal

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

1 pack of Helix seals for spray chamber nebulizer inlet

Figure 13.

Resources

ICP-OES resource page

Troubleshooting videos

Agilent PlasmaNet ICP-OES email support forum

5100/5110 ICP-OES quick reference guide

ICP-OES parts and supplies (On-line Store)

Agilent atomic spectroscopy application notes

Agilent Spectroscopy consumables catalog

Agilent high quality Inorganic and Metallo-Organic standards for Atomic Spectroscopy

Agilent supplies for PerkinElmer ICP-OES & ICP-MS systems catalog

Agilent MSIS Technical Overview

Agilent recorded webinars for atomic spectroscopy

Summary

Agilent ICP-OES instrumentation is just one of the Agilent atomic spectroscopy systems available – Agilent is able to provide you with an instrument for any type of application whether it's traditional atomic absorption, ICP-OES, ICP-MS (including the relatively new triple quadrupole ICP-MS), or microwave plasma systems.

In this presentation, we've tried to outline the particular challenges that you may be facing in your laboratory. We've 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've also included some guidelines and maintenance procedures that you can use to set up standard operating procedures in your laboratory which will help these types of issues from reoccurring.

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Eric graduated from Monash University in Melbourne, Australia with a BSc degree in Chemistry in 1985. He joined Varian in 1988 and worked in several roles including Atomic Absorption Product Manager, European Field Sales/Applications Specialist, **ICP-OES Product Manager and MP-AES** Product Manager. After the acquisition of Varian by Agilent Technologies, Eric took on the role of Spectroscopy Supplies Product Marketing Manager, with responsibility for field sales support, customer support, product development and marketing of the spectroscopy supplies. He has over 30 years' experience with atomic spectroscopy techniques and is based at Agilent's Spectroscopy Technology Innovation Centre in Melbourne, Australia.

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