

## This is How you ICP-MS: Mastering the Art of Cone Performance

This article offers advice on best ICP-MS analytical performance and guides you through instrument maintenance issues. In particular, the article will focus on the interface cones.



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### Introduction

Focusing in on the interface region, where the cones sit, analyte ions are produced within the plasma which is at very high temperatures and atmospheric pressure. These ions must be transmitted into a mass spectrometer, which needs to operate at very low pressures. First, we sample these analyte ions through the first cone, (the sampling cone), before they enter a low-pressure interface region, where the ions expand and are extracted by a combination of a second cone (the skimmer cone) and extraction lenses.

Figure 1 is a schematic of the interface region. The purpose of the extraction lenses and sampling cones is to get the analyte ions through to the reaction cell, and then into the quadrupole in the mass spectrometer. As well as transitioning from atmospheric pressure to very low pressure, it is important to exclude any photons or neutral species that would contribute to background signal, and only transmit positively charged analyte ions through to the mass spectrometer.

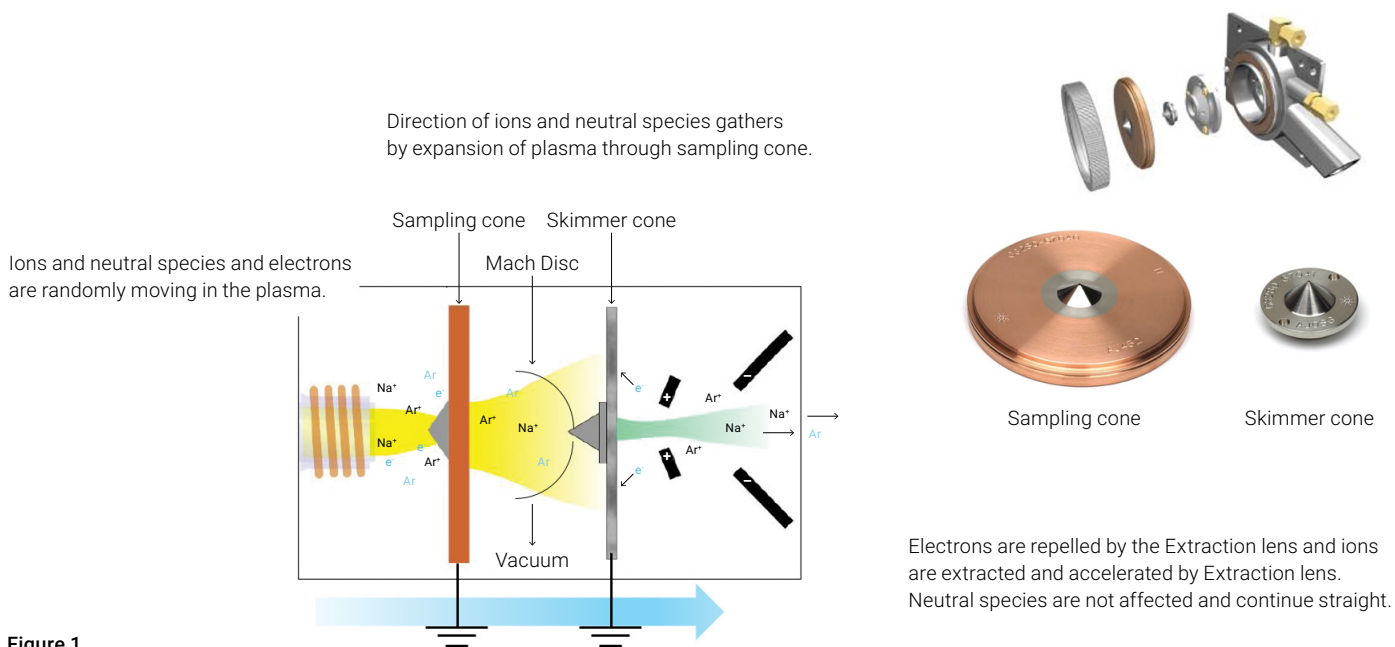


Figure 1.

Agilent provides several software tools to optimize this ion transmission.

**Startup procedure:** this automatically takes you through optimizing the plasma parameters within the ICP, such as setting the axis of the torch, the nebulizer gas flow, and tracking the performance of the instrument under standard conditions. This provides a performance report, which is valuable information that can be tracked over the lifetime of your ICP-MS to provide a history, and to easily identify when things start going off-track.

**Auto tune for lens and extraction parameters:** this removes operator variations and ensures consistent performance from day to day, so the ion lens extraction provides best performance for your ICP. We typically recommend that you select to run all of these startup procedures. Pulse/analog factors are usually set within your individual methods, because they need to be updated for the elements within that method. We then offer tuning both globally for the instrument and within each batch, so within each method that you're going to run.

## Interface Cones

Let's look more closely at the interface cones. These are key to the performance of the ICP-MS, so should be regularly inspected, focusing particularly on the orifice. Agilent offers a handy magnifier tool for this purpose. This magnifier is illuminated, and provides 10 times magnification, together with a measuring scale. You need to check that the orifice is clear, that it's still circular, and that the dimensions remain correct. The sampler cone should be one millimeter in diameter. If it's clogged, it should be cleaned, and if it's enlarged, it's come to the end of its useful life and should be replaced.

Some common interface cone issues can occur from mishandling or poor use. The cones themselves are very fragile, particularly the tip on the skimmer cone, which comes to a very fine point, so poor handling will cause problems. The tip should not be placed in contact with any surface during cleaning, removal, and reinstallation into the instrument.

The correct skimmer base for the skimmer cone must be used – the key thing to remember here is the material that you're using. Nickel skimmer cones need to use a stainless steel skimmer base, and this is the default for an x-lens system. If you're using platinum cones, you need a brass skimmer base. This is Agilent's default for the semiconductor configuration instruments. It allows control of the tip temperature to prevent overheating, and ensures that the matrix deposits in a uniform manner on the tip.

There is a balance to be struck here as the cones should be maintained to ensure their performance, but they should not be cleaned any more than necessary. This is because any cleaning of the cones will reduce their lifetime. You must, focus on the tip of the cone, especially the condition of the orifice – there is certainly no need to clean/polish the face of the cone back to its original condition. The appearance of the cone face is essentially unimportant, but we do need to ensure that the orifice is the correct dimension, is clear, and is the correct shape (Figure 2).

Once new cones have been installed, or existing ones cleaned, they then need to be conditioned. This is recommended as it will reduce drift due to initial deposition of the sample matrix onto the clean cone surface. You're looking for an equilibrium with the matrix on the surface of the cone – a thin layer can lead to improved sensitivity because it lowers the background level, particularly with nickel and copper.

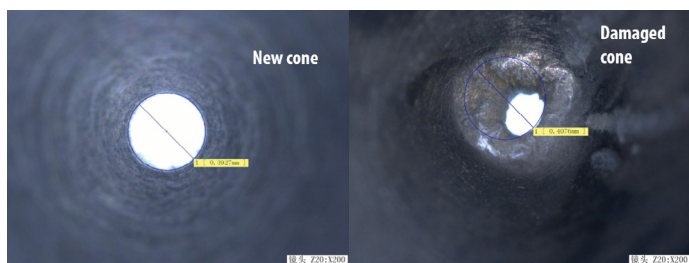


Figure 2.

Agilent's default conditioning recommendation is to aspirate a 50 ppm calcium solution. This can be made up in 1% nitric acid, and then aspirated for approximately 10 minutes. You should then follow with a rinse solution, for example with 1% nitric acid, for a further 10 minutes, and that will sufficiently condition the cone.

Practically, if you're running the same kind of sample matrix each day, you'll be able to aspirate the typical matrix for your sample, for around 15 minutes, which can be performed as part of the instrument warmup. Ignite the plasma, aspirate a sample rather than just rinse, and then follow this after the initial warmup with a blank or rinse solution for 10 minutes – this will condition your cone specifically for your analysis.

A third option is for environmental laboratories, where you can aspirate an interference-check solution ([6020 Interference Check Solution A part no. 5188-6526](#)). This should be diluted 10 times by volume with ultrapure water, and then aspirated for up to 30 minutes. The recommendation is to use general purpose plasma condition with auto tuning and no gas mode. Follow this with a 5% nitric acid rinse for 10 minutes.

As mentioned, make use of the instrument warm-up time, so you're not adding additional delay into your analysis. Figure 3 shows some nicely conditioned cones – this is the condition they should be in at the start of your analysis.

So why and when do we need to clean the cones? It has been mentioned already that you shouldn't over-clean the cones as this will reduce their effective life. When you see a reduction in sensitivity, poor long-term precision, or any elevated background due to your matrix, or nickel, or copper from the cone, that's typically the time to do some maintenance.

You may also notice changes to the interface vacuum. Typically, if the cones start getting clogged, or the orifice becomes blocked, the interface vacuum may change from its normal level, again indicating some maintenance is required. If the cones appear as shown in Figure 4 (compare with Figure 3), this indicates there's too much matrix deposited, particularly around the orifice, and this needs to be cleared.

Remember, when you're cleaning cones, the aim is not to get them back to 'as-new' condition – you're aiming for cones as shown in Figure 3, with a uniform deposit of matrix on the surface.

Agilent recommends a step-by-step procedure for cleaning the interface cones. Routinely, often all that is required is to ultrasonicate the cone in pure water. We offer cotton swabs, which have a fine tip at the end to allow cleaning of the back side of the cone and to make sure that orifice is clear. You simply use these cotton swabs with water, and then ultrasonicate the cones in pure water for at least five minutes (20 minutes is typical), and then repeat as required. A good rule of thumb is to check that the water remains clean after sonication.

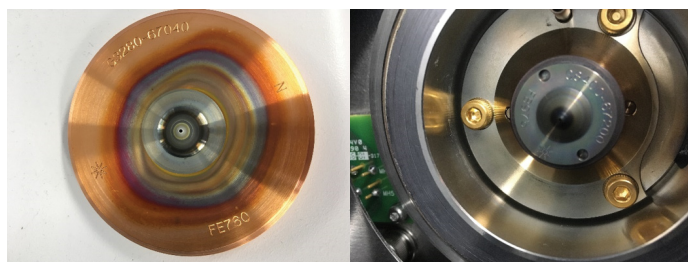


Figure 3.

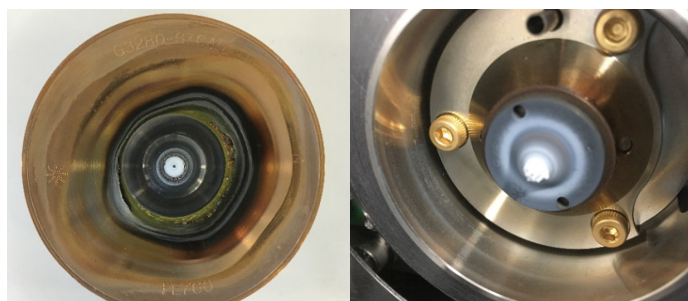


Figure 4.

Next, and only if your application requires it, you can clean with a 2% Citranox solution. We recommend you sonicate for only 2-3 minutes, rinse the cone with pure water, then ultrasonicate in pure water for around five minutes to make sure all the Citranox residue is removed. For additional information visit [www.agilent.com/en/promotions/icp-ms-resource](http://www.agilent.com/en/promotions/icp-ms-resource).

At that stage, most cones should be clean and back in use. For more severe contamination, after reinstalling the cone and checking sensitivity and performance for your analysis, we recommend a more aggressive cleaning with 2% nitric acid. We don't ultrasonicate or soak the cones in acid because you can pit the surface of the cone from acid attack. Instead, dip a cotton swab in 2% nitric acid and use it to clean both sides of the cone. Then rinse the cone in pure water, ultrasonicate it for a few minutes in pure water, and then repeat that procedure to ensure any remaining acid residue is removed. Once the cones are clean and before reinstalling them, check the condition of the graphite gasket that sits behind the sampler cone – if it's deformed or torn, replace it.

Use the skimmer cone removal tool to install and tighten the skimmer cone – then refit the sampling cone with the retaining ring, which should only be hand-tight. The best way to make sure the installation is correct is when you ignite the plasma, it transfers happily to analysis mode. Also, understand the typical interface pressure on your system, and check this to confirm that the interface is running correctly.

We've undertaken an in-depth study on the performance of our genuine Agilent cones for the Agilent 7900 ICP-MS using the x-lens configuration (these being the nickel sampler and the nickel skimmer cones), and to benchmark and compare with other cone manufacturers. You can find the [full article here](#), but some of the main findings are highlighted below.

Figure 5 shows a comparison of the sampler and skimmer cone weights. You can see the Agilent cones on the left-hand side, in comparison with cones from other manufacturers. The main takeaway message is that the cones are all different, and they can be grouped by manufacturer. These differences may or may not create issues with performance, but it certainly indicates different manufacturing methods for the cones, and that they're not within the Agilent specification.

Figure 6 shows a sensitivity comparison with automated startup procedures and auto tuning for low-matrix conditions. Brand new cones out of the box are shown in the top plot, and we can see that the Agilent cones (in blue) outperform third-party cones. The bottom plot in Figure 6 shows results after the conditioning procedure described earlier for environmental labs using the ICS standard. Agilent cones are really designed for these procedures, and provide the best sensitivity.

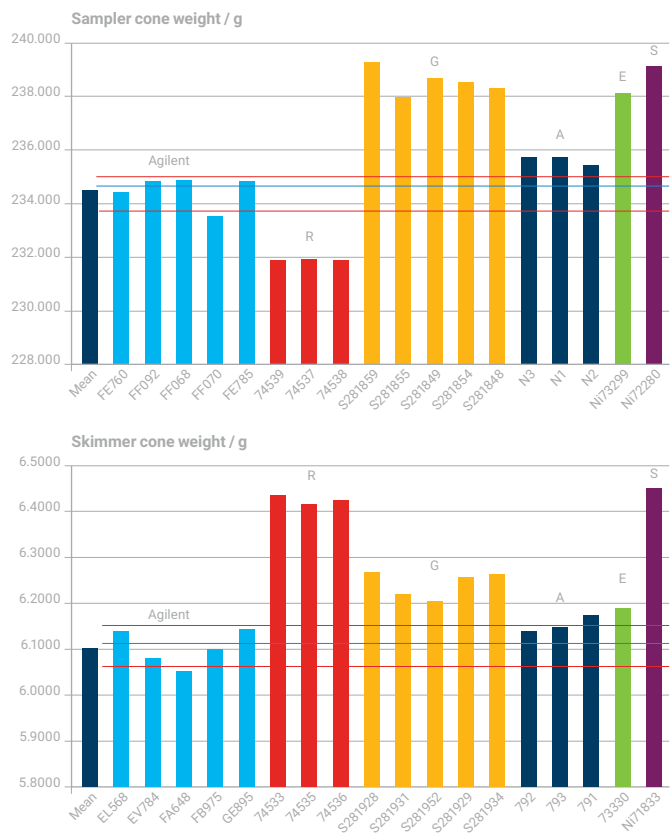


Figure 5.

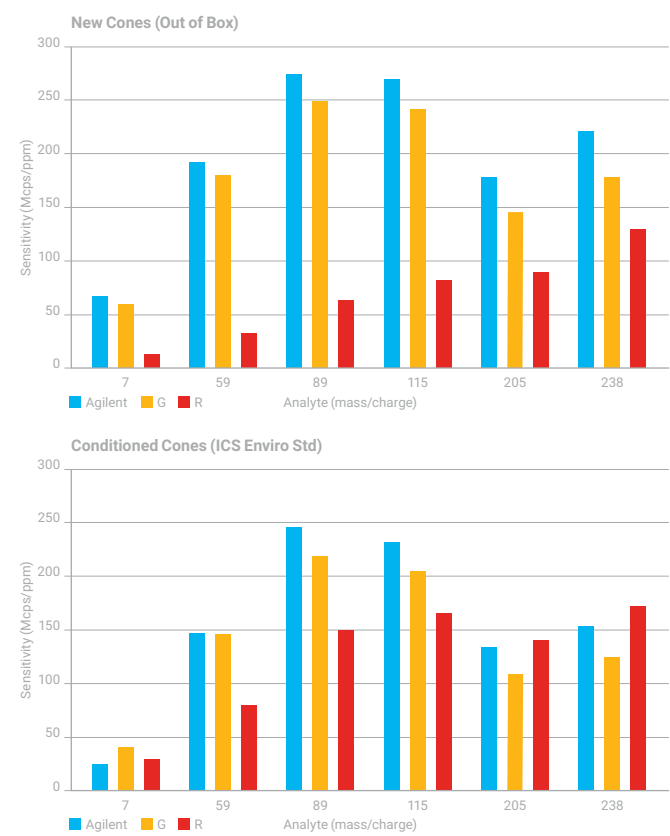


Figure 6.

We also investigated background. The background is very important, together with sensitivity, for Background Equivalent Concentration (BEC) levels. Figure 7 shows a comparison between a genuine Agilent cone for a full-mass scan versus the test sample for third-party cones. The blue lines indicate the levels that would be considered natural variation, and values above the upper level indicate an increased background at specific masses for the test cone. This was an example in no-gas mode, and it clearly shows that there's a number of masses that give a higher background with third-party cones.

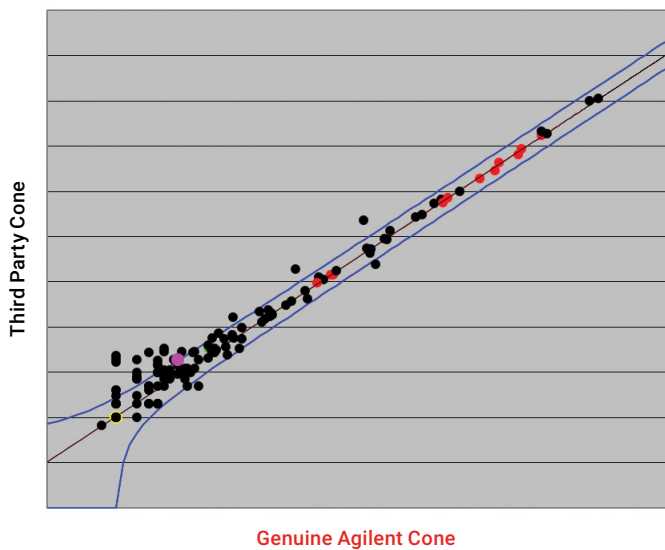


Figure 7.

Similar results are seen in helium mode and using aerosol dilution, where for different manufacturers there is an elevation of background across the mass range, and between different manufacturers the same masses being an issue across those third-party cones – this affects the BECs you'll find for your analysis.

Next, we looked at stability, both short-term (20 minutes), and long-term. All of the Agilent test cones generally meet the required specifications; one exception being for long-term stability with lithium, which is the most difficult element. Many of the third-party manufacturers suffer with lithium first, but there are a lot of failures across the board in terms of stability. Figure 8 highlights the worst-case scenario – on the left-hand side, you can see a typical two-hour stability trace expanded to show any variation, with the values plotted here in percent RSD. We typically expect less than 3% RSD over this time period. In the right-hand side, you can see significant instrument drift from a third party cone. All cones were treated in exactly the same way, and conditioned with our recommended procedure for environmental labs, highlighting that you need to take more care when using a third-party cone.

Moving back to the selection of the right type of cone for your application (Table 1), Agilent offers several different materials to suit your particular needs. The default material is nickel in sampler and skimmer cones, for the x-lens systems, which are suitable for most common applications. Nickel gives good thermal and chemical resistance, and provides the most economical operation.

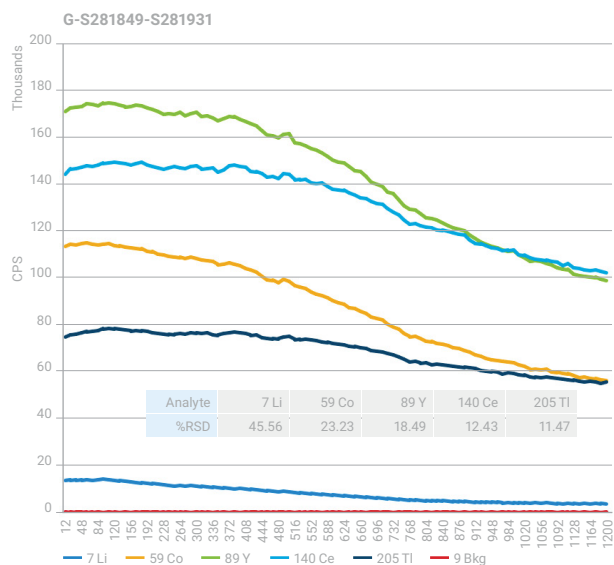
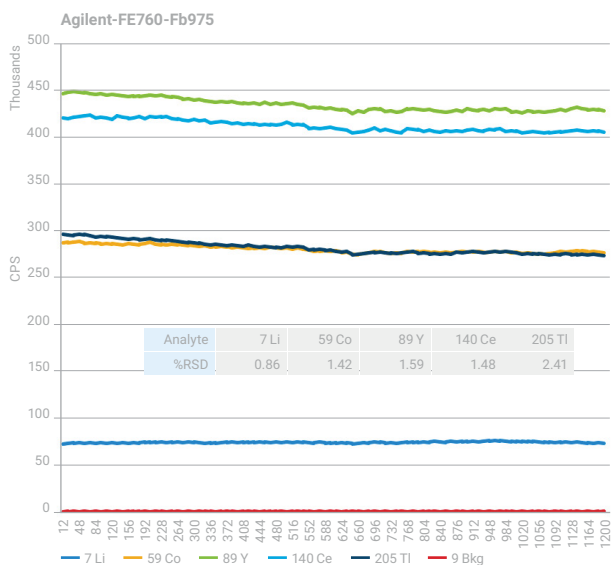


Figure 8.

Table 1.

| Type of Cone                      | For which Model ICP-MS?   | Skimmer Base Required | Recommended Applications   |
|-----------------------------------|---|-----------------------|--|
| Nickel sample/skimmer cones       | Standard on 7500a/i/c/ce/cx, 7700x/e, 7800/7900 and 8800/8900 with x-lens   | Stainless Steel       | Suitable for most common applications. Good thermal and chemical resistance. Provides most economical operation. Typically use 3-5/year (based on ~350 samples/day)  |
| Nickel plated sampling cone       | Optional for all 77/78/7900 and 88/8900 models  | –                     | For samples containing > 0.5 % HCl, or for routine operation with (U)HMI with max. aerosol dilution ratio  |
| Platinum sample/skimmer cones     | Standard on 7500/cs, 7700s, 7900 with s-lens, and 8800/8900 semicon configuration. Optional for all other models. | Brass                 | Required for analysis of aggressive acids (esp. HF) and when O <sub>2</sub> /Ar option gas is used for analysis of organic solvents. Use sample cone with larger 18mm insert for high viscosity and high boiling point acids e.g. H <sub>2</sub> SO <sub>4</sub> or H <sub>3</sub> PO <sub>4</sub> |
| Platinum skimmer with Copper base | Standard on 7700s, 7900, 8800/8900 semicon configuration and 8900c  | Brass                 | Recommended for the lowest LODs and for higher matrix samples. Typically use 1-2/year (based on ~350 samples/day)  |
| Platinum skimmer with Nickel base | Standard on 8900m   | Brass                 | Recommended for organics analysis  |



Skimmer cone, Nickel



Skimmer base, Stainless Steel



Skimmer cone, Platinum



Skimmer base, Brass

The sampler cone itself has a copper base, and we recommend customers move to the nickel-plated copper base if they are routinely analyzing high-chloride matrices or using the aerosol dilution with HMI-plasma modes, which produces a hotter plasma. This gives you extra resistance to these conditions, and will give you a better lifetime for your sampling cones.

Agilent also offers platinum sampler and skimmer cones. These are default for the s-lens systems and semi-conductor instruments. We require these for analysis of aggressive acids, especially hydrofluoric acid, and we also highly recommend this for organics analysis when you're using Oxygen-option gas to burn off the carbon load on the plasma.

Also available is a high-end platinum cone with a large insert, an 18 mm insert compared with the 12 mm standard insert, which is recommended for very aggressive acids, such as sulfuric and phosphoric, due to the extra resistance. Typically, Agilent offers the copper base as the standard configuration, but also offers a platinum skimmer with a nickel base for organics analysis.

Recently introduced are three cone care packages. These are designed to provide everything required for maintaining the cones, including two sampler cones – you can choose from the standard nickel cones, which have a copper base, nickel-plated cones, or platinum cones. The kit includes a magnifier tool to help with cone maintenance and inspection, a pack of the cotton swabs, and gaskets for the sampler cones. More details can be found at [www.agilent.com/cs/library/flyers/public/5991-8673\\_icpms\\_conecarekit\\_flyer.pdf](http://www.agilent.com/cs/library/flyers/public/5991-8673_icpms_conecarekit_flyer.pdf)

Agilent offers a recycling program for our platinum cones. This not only includes both cones, but also the torch shield, so you can return any used platinum cones to Agilent for a credit against future purchases of cones and torch shields. You can find more details at [www.agilent.com/chem/Ptcone](http://www.agilent.com/chem/Ptcone)

## Tips & Resources

Finally, following are some tips, tricks, and resources to help you with your maintenance procedures, and for getting the best performance out of the ICP-MS.

**Recommended end of day procedure:** Follow the steps below...

1. Aspirate acid rinse solution for a few minutes before shutting off the plasma. This helps to prevent sample deposition inside the nebulizer after the run
2. Extinguish the plasma and switch off the chiller
3. Remove the sample capillary from the rinse, start the pump again and pump any remaining rinse solution from the spray chamber
4. Release the pressure bars on the pump tubing and remove the bridges from the securing slot. Ensure the tubes are no longer stretched over the pump rollers
5. Empty waste vessel
6. Close the current worksheet – leave Mass Hunter S/W running
7. Leave mains power on. This keeps instrument in stand-by mode (ensures fastest start-up)

**Maintenance schedule:** The maintenance schedule is a daily check on the pressure on the gas lines, you will get alerts in the software if they fall below specified levels, but make sure you've got a good control on your gas supply; check the peristaltic pump tubing, roll it between your fingers, make sure it's not flattened and is still elastic; visually check the glassware and all the connections within the sample introduction system, and have a look at the exterior of your sample cone, which you can see by opening the cover of the instrument, to make sure it has a good appearance, and there's not a high level of matrix deposited on the tip. As frequently as required, you need to replace peristaltic pump tubing (typically every week). Check the torch, check the recirculator, which can often be forgotten, and also check the rotary oil pump for the interface vacuum; check the oil level and the condition of the oil in terms of its color, and maintain it as required.

**Early maintenance feedback:** Agilent has software prompts to help with maintenance – early maintenance feedback (EMF). The EMF window shows usage of various components and predicts when to perform maintenance. This is typically used for the foreline pump, for which we recommend an oil change every six months, but this is fully customizable, so you can set limits once you've got used to your system (Figure 9).

You can also make use of the user log which, if you've got multiple users, can be a convenient tool where you input what maintenance was done, and it automatically logs time and date, and that goes into the instrument history.

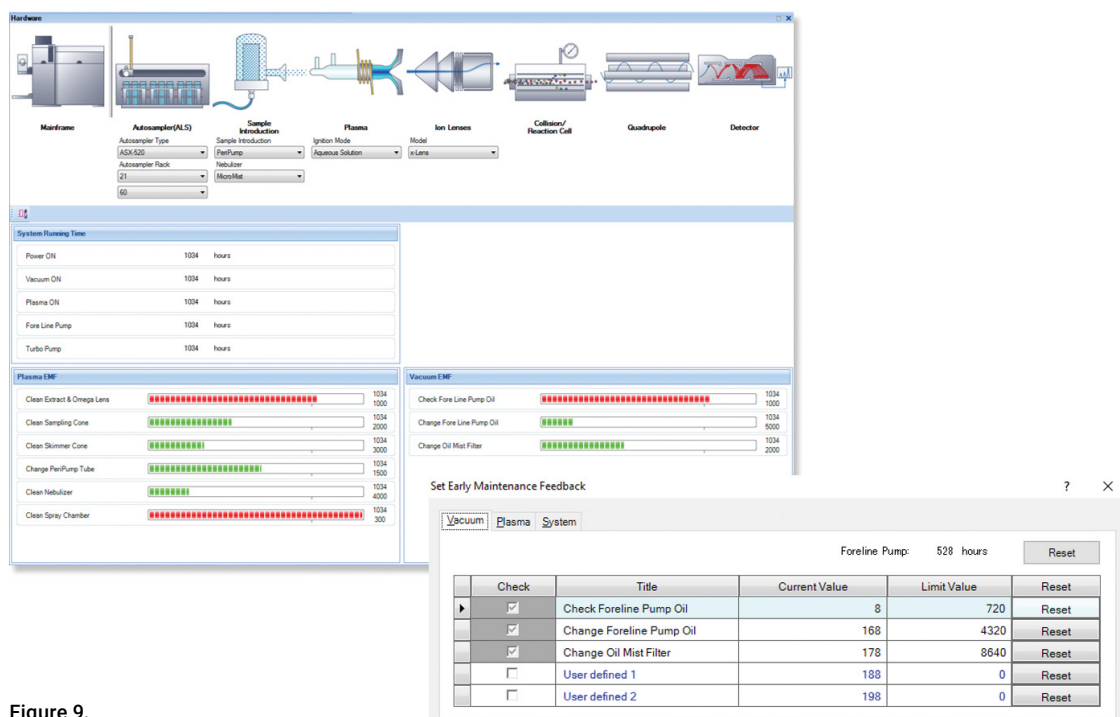


Figure 9.

**Key consumables for ICP-MS:** to ensure you avoid any downtime make sure you have replacements for any glassware, e.g. torch, spray chamber, nebulizer (because they can be broken during replacement), tubing connectors, and interface cones. Don't forget anything associated with the autosampler (sample tubes, racks, probes and transfer tubing), or if you're using the switching valve system (ISIS system), keep the tubing and connectors for this as well.

To simplify your ordering and make sure you've got these essential supplies to hand, Agilent produce customizable and basic kits for the typical components needed for 12 months operation of your ICP-MS. There are kits for different models, and you can choose supplies to suit the configuration of your ICP-MS as well, including what type of cones you require. You can find more information at [www.agilent.com/cs/library/brochures/ICP-MS\\_Supplies\\_Kit\\_5991-5006EN\\_Brochure.pdf](http://www.agilent.com/cs/library/brochures/ICP-MS_Supplies_Kit_5991-5006EN_Brochure.pdf)

Agilent also produces a complete range of inorganic and metallo-organic standards for atomic spectroscopy, so whether you're working with AAS, MP-AES, ICP-OES or ICP-MS, there is a full range, details of which can be found online at [www.agilent.com/en/product/chemical-standards](http://www.agilent.com/en/product/chemical-standards) or check out our standards catalog at [www.agilent.com/cs/library/catalogs/public/5991-5678EN\\_Chemical\\_Stdns\\_Catalog\\_LR.pdf](http://www.agilent.com/cs/library/catalogs/public/5991-5678EN_Chemical_Stdns_Catalog_LR.pdf). This includes original tuning solutions and wavelength calibration solutions recommended to check your instrument performance.

In addition, we 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. These are available for on-line ordering through Agilent's Ultra Scientific division at [www.ultrasci.com/components/customstandard](http://www.ultrasci.com/components/customstandard). Custom products are manufactured to the customer's specific requirements, then are qualified and certified by our team of expert chemists in our ISO 9001, 17025, and Guide 34 accredited facility.

## Agilent ICP-MS Online Resource Library

The focus of this article is education, to ensure you get the best out of your instrument and don't suffer from problems. As part of this educational effort Agilent has developed a dedicated web page – ICP-MS Online Resource Library at [www.agilent.com/en/promotions/icp-ms-resource](http://www.agilent.com/en/promotions/icp-ms-resource) – where you'll find links to videos which guide you through maintenance procedures, plus tips and tricks, and other literature that's going to assist you with making sure that your ICP-MS runs trouble-free.

## Summary

Most of the failures with an instrument occur in the sample introduction area, so ensure you pay attention to your interface cones, pump tubing, drain, torch, spray chamber and nebulizer, to achieve good day-to-day instrument performance. Below you'll find a list of resources which will help you navigate your ICP-MS work most effectively:

[Agilent Atomic Spectroscopy YouTube Channel](#)

[ICP-MS parts and supplies](#) (On-line Store)

[Agilent ICP-MS application notes](#)

[Agilent ICP-MS Quick Reference Guide](#)  
(lists most common consumables items)

[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 recorded webinars for atomic spectroscopy](#)

Agilent also produces the quarterly Agilent ICP-MS Journal ([www.agilent.com/en-us/newsletters/icpmsjournal](http://www.agilent.com/en-us/newsletters/icpmsjournal)), which has real-world applications, updates, and it's a very useful source of information for ICP-MS users. You can subscribe by clicking on the blue link above.

Finally, know that Agilent expert staff are on hand if you need any assistance, specific advice about your application, or details on your instrument. There's also the Agilent University ([www.agilent.com/en/technology/agilent-university](http://www.agilent.com/en/technology/agilent-university)), particularly for training new staff, or new users in this technology. On offer is preventative maintenance on all Agilent instruments to make sure they're in the best operating conditions, and we can offer method and application consulting specific to your needs.



## About the Author

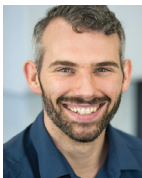
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Dr Pearson graduated from the University of Hull, UK in 2003 with a M.Chem. in Chemistry with Analytical Chemistry & Toxicology. He went on to complete a Ph.D. using ICP-MS in 2007 with the title "Elemental Speciation and Miniaturised Sample Introduction Studies for ICP-MS".

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Currently, Dr Pearson is the ICP-MS Supplies Product Manager for Agilent Technologies based at the Spectroscopy Technology Innovation Centre in Melbourne, Australia.

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Printed in the USA, April 11, 2019  
5994-0860EN

