CUSTOMER INTERVIEW Maximizing Value in Metals Recovery

We took time to talk with Adam Currie, Laboratory Manager at Siltech PMR in Melbourne, Australia, to understand how plasma-based techniques such as ICP-OES and MP-AES, and the Agilent OneNeb Series 2 Nebulizer, are being used and how this is providing benefits for their precious metals applications.

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Tell us about your ICP-OES applications at Siltech?

Our biggest application for ICP-OES is determining the purity of precious metal samples for our clients. Starting with 0.5-1.0 g sample of Au, Pt or

Pd, we digest this in 10-20% Aqua Regia and dilute to a final volume of 100 mL. We analyze this digest looking for 30 elements, and then determine purity by deducting the concentration of the found elements from an assumed 100%. This allows us to determine the purity of metal - this is a common technique for precious metals and allows us to distinguish lower purity from 99.999% purity for example. We also run the same type of analysis on silver dissolved in HNO₂.

The third type of analysis is what we call 'boil out' solutions. This is the end of the bullion fire assay process in which a silver prill containing Pt and Pd is dissolved in HNO, and analyzed for its Pt/ Pd content. We primarily use our Agilent MP-AES for this analysis - this is equipped with an Agilent OneNeb Series 2 nebulizer.

How important is the nebulizer in allowing you to complete your applications?

Very important. The transition between high salt

content and silver nitrate solutions means it can be common to have a cross over. This can cause insoluble silver chloride to drop out in the sample tubing and block the nebulizer.

How has the switch from a glass concentric nebulizer to the Agilent OneNeb nebulizer worked for you? What are the key differences you have noticed?

We started using a glass concentric nebulizer, but that was some time ago now. We then moved on to the OneNeb Series 1 nebulizer, before

upgrading to the current Agilent OneNeb Series 2 nebulizer. One major issue we noticed with the glass concentric nebulizer was that when blockage occurred it would easily break if any attempt to unblock it was made. We no longer have this challenge using the OneNeb.

What are your priorities when considering the purchase of a nebulizer?

I like the idea that you can detach and replace parts of the nebulizer. Expense is always an issue, so replacing individual components is cheaper, just like having a demountable torch where components can be removed and replaced separately. The OneNeb Series 2 nebulizer meets all of these priorities.

Have you noticed any difference in the longevity of your OneNeb compared with other nebulizers?

I've only been using the Agilent OneNeb Series 2 nebulizer for a short time, but already predict it will be around longer than the traditional glass concentric versions.



Adam received his degree in Applied Chemistry from RMIT in Melbourne, before moving into precious metals analysis. *He joined Siltech, where he set up and heads the precious* metals laboratory. Siltech PMR Pty Ltd specializes in the

treatment and disposal of film processing waste. It is one of the only fully licensed facilities in Australia with the ability to treat these kinds of wastes and products. Siltech holds EPA licenses for the treatment of chemicals and the production of silver, gold and platinum group metals and uses an Environmental Management System based on ISO14001.

IMPROVING SENSITIVITY & PRECISION The Benefits of Selecting the Right Nebulizer

We also took the time to talk with Eric Vanclay, Spectroscopy Supplies Product Manager at Agilent Technologies in Melbourne, Australia, to understand more about why the OneNeb is so different to other nebulizers on the market.

Adam outlined a number of advantages of the OneNeb. Are there any other benefits of working with the OneNeb?

The OneNeb Series 2 nebulizer is exclusive to Agilent. Adam outlined some of the advantages, but it also provides many other advantages compared with a conventional glass concentric nebulizer many users may be working with today. In addition to the robustness and greater tolerance to samples with high levels of total dissolved solids, the OneNeb provides higher sensitivity and improved precision. It also provides better long-term measurement stability. Of course, the biggest advantage for most users is that it is virtually indestructible – so even if dropped, the nebulizer will continue to operate. Most conventional nebulizers would need immediate replacement if the operator was unfortunate enough to drop one of those.

Can you explain how the OneNeb is able to provide these benefits?

Compared with a conventional glass concentric nebulizer, the OneNeb uses flow blurring nebulization, which creates highly turbulent mixing between the nebulizer gas flow and the sample. This creates a dense aerosol made up of extremely fine droplets with a narrow size distribution. Most droplets are less than 10 microns in diameter. This means more sample makes it into the plasma for higher sensitivity, and the sample breaks down easier, improving precision. This is also compatible with any liquid, and ensures efficient aerosol formation across a wide range of solution flow rates. In addition, with minimal pressure difference between the sample and gas at the exit, and a constant diameter sample capillary, nebulizer blockage is virtually eliminated.

Not everyone is working with the OneNeb today. Can you provide some guidance to help customers with nebulizer selection?

If the nebulizer the analyst is working with does not provide the performance they need, there are other options available. Generally, glass concentric nebulizers are designed for good tolerance to samples with low to medium total dissolved solids (TDS) levels. Laboratories working with higher acid concentrations and mixed acids that can include more aggressive acids required for proper dissolution and good stability of the analytes in solution, typically need a nebulizer that can handle larger particulates and the acid mixtures. Therefore, these laboratories will often require an inert nebulizer.

With a little background about the sample types you're running, and the sample preparation process, you can grasp the potential challenges you may face when analyzing your samples. If you need further guidance, Agilent has developed an ICP-OES nebulizer selection guide which is available from the Agilent website. This can help you select a nebulizer that will help you achieve optimum performance and reduce the risk of blockage and unplanned downtime.

Most users are working with conventional glass concentric nebulizers – and they'll suffer from a nebulizer blockage at some point. Can you provide some tips to help users overcome this issue?

Prevention is the most important thing. It's always good practice to thoroughly rinse the nebulizer between samples and especially at the end of analysis, using a rinse solution. Sample filtration prior to analysis can also be a good preventative step to help remove large particulates that can cause nebulizer blockages.

Once a nebulizer is blocked, the best way to remove the blockage is to use a dedicated nebulizer cleaning tool. Agilent offers a cleaning tool that is suitable for standard ICP-OES and ICP-MS glass concentric nebulizers. It's available as part number G3266-80020 and can be easily found by searching "Nebulizer cleaner" on Agilent.com. You can use this periodically to clean or backflush the nebulizer.

The most important tip is to avoid using a cleaning wire or an ultrasonic bath to clean or remove nebulizer blockages. Both will cause damage to the fine sample capillary and permanently damage the nebulizer.



Is there an easy way a user can diagnose a blocked nebulizer?

The first step is to run an instrument performance test, which will test the whole instrument using a standard set of conditions. Then examine the results. If sensitivity is down and the other performance indicators are in the normal range, you should suspect a nebulizer blockage. With Agilent's ICP Expert software for ICP-OES or MassHunter software for ICP-MS, you can use the nebulizer test to measure the backpressure and verify nebulizer status. Higher backpressure means the test will fail, indicating a likely blockage.

You can also remove the nebulizer from the spray chamber and visually inspect it. With the nebulizer gas flow on and water being pumped, you should see a consistent aerosol being formed. Little or no aerosol formation, or a lopsided aerosol can indicate a partial blockage of the nebulizer. With the nebulizer removed, check the condition of the tip and the internal sample capillary. Can you see any build-up or blockage, and is the tip broken or chipped? Basic troubleshooting like this can usually help you identify a nebulizer blockage issue.

Click here to view on-demand webinar for more 'Trusted Advice to Help Maximize your ICP-OES Instrument Performance and Uptime'



Eric Vanclay is the Spectroscopy Supplies Product Marketing Manager for Agilent Technologies, based in Melbourne, Australia. He graduated from Monash University in Melbourne, Australia with a Bachelor of Science degree majoring in

Chemistry in 1985. Eric joined Varian in 1988 and has worked in a number of roles including Atomic Absorption Product Specialist, Atomic Absorption Product Manager, Export Marketing Manager, European Field Sales/ Applications Specialist (atomic spectroscopy), 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 all of the atomic spectroscopy techniques.

10 BENEFITS OF THE Agilent OneNeb Series 2 Nebulizer

- 1. Eliminates downtime when switching applications and nebulizers
- 2. Inert: use with virtually any solution
- 3. Lower running costs: Virtually indestructible, even if dropped
- 4. Improve productivity: Reduces reporting limits and LODs, eliminating rework
- Confidence in results: Typical precision is <1% RSD
- 6. Higher throughput: Excellent long-term stability means longer runs
- 7. Less downtime: Minimize blockage with high TDS samples
- 8. Suitable for any ICP-OES
- 9. Hassle free: Replaces a conventional glass concentric nebulizer without adaptors or method changes
- 10. Reduced administration costs: Agilent can satisfy all of your supply needs