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ICP-MS to Support Regulatory Limits and Industry Needs

In this issue, we present two regulatory developments that are enabled by ICP-MS. In the first article, we describe a new AOAC method that has been released for heavy metal analysis in cannabis and hemp products. And a second article describes an ion chromatography (IC) ICP-MS method that can be used as an alternative to HPLC-ICP-MS for routine analysis of trace hexavalent chromium (Cr(VI)) in drinking water.

Of the wide range of industries that use ICP-MS, semiconductor product development and manufacturing has among the most demanding requirements. Continuous advances in semiconductor technology require ever tighter control of impurities. Lower-level trace element analysis has been enabled by parallel developments in ICP-MS technology. The history and current performance of ICP-MS for semiconductor applications is discussed in a recent industry webinar and e-symposium reviewed in this issue.

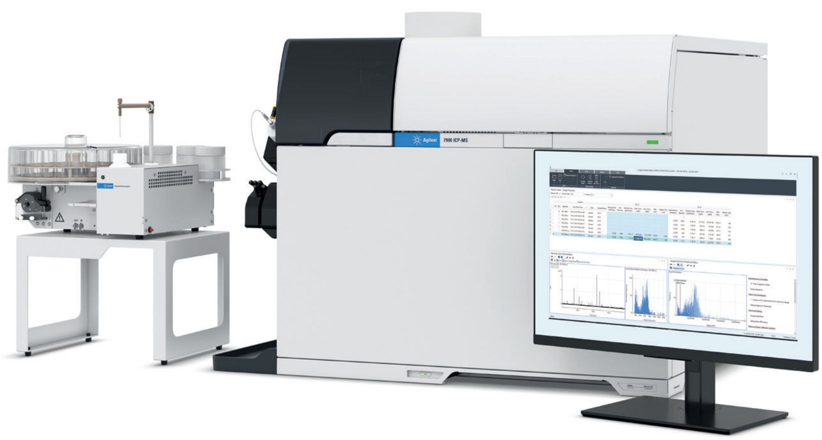


Figure 1. Agilent 7900 ICP-MS with the I-AS integrated autosampler for ultratrace analyses.

Steps Towards an Industry Standard Method for the Analysis of Cannabis and Hemp Samples by ICP-MS

Jenny Nelson, Craig Jones, Agilent Technologies, Inc. Sam Heckle, Leanne Anderson, CEM Corporation, USA

Cannabis industry requirements

The rapid rise in the availability and use of cannabis-based products for recreational and medical use has led to increasing need for robust analytical methods to ensure product quality and safety. However, the analysis of metals in cannabis and hemp products has been hampered by a lack of official methods in the industry. In August 2021, AOAC adopted a newly developed ICP-MS method for the analysis of heavy metals in various cannabis-based products as an Official Method of Analysis in First Action status (*1*). Reproducibility and performance of the new method will be tracked for up to two years and, if satisfactory, the new method will be recommended for Final Action status.

The new ICP-MS method can be used to determine arsenic (As), cadmium (Cd), mercury (Hg), and lead (Pb) in a variety of cannabis-derived products prepared using microwave digestion. Other elements can be added to the method following appropriate validation. The method is applicable to a comprehensive range of cannabis and hemp-based sample types, as listed in Table 1. In developing the new method, the aim was to create a robust and easy to use method that meets the AOAC Standard Method Performance Requirements (SMPR).

The new method is described in a recent webinar (*2*), which also highlights the importance of a fast and reliable sample preparation method for samples with varied matrices. Having one sample preparation method that works for all sample types is especially valuable for high throughput cannabis testing labs.

'Live' demonstration of sample preparation

During the webinar, Sam Heckle of CEM demonstrated how the MARS 6 microwave digestion system can digest up to 24 or 40 mixed cannabis and hemp samples in a single batch. Sam recommended the 24 place turntable for batches that include 'oil-type' samples. The sample digestion approach ensures complete digestion of varied cannabis and hemp sample types while also addressing the chemical stability of the target analytes.

Table 1. Cannabis and hemp-based sample types that can be analyzed using the new AOAC ICP-MS method.

Sample Category	Sample
Inhaled	Hemp flower
	Cannabinoid (CBD) vape oil
	Hemp isolate extract
Oral	Full spectrum softgel capsules
	Full spectrum tincture
	Isolate tincture
	CBD coffee grounds
	Hemp butter
	Hemp seed oil
	CBD beef jerky
	CBD hard candy
	CBD pineapple drink
Topical	Full spectrum balm
	Pain relief cream
	CBD balm
	CBD topical oil
	Hemp soap
Manufacturing	Hemp biomass and spent hemp biomass
	Trichomes
	CBD crude extracts, distillates, and isolates

Of the "big four" AOAC analytes, As, Cd, Hg, and Pb, Hg is not chemically stable in HNO_3 alone. Sample digests should therefore include HCl so stable Hg complexes are formed. But chloride ions from the HCl can combine to create polyatomic ion overlaps on several analytes – including As – in the ICP-MS spectrum. The ICP-MS must therefore include a simple and reliable approach to deal with these spectral overlaps using helium (He) mode in the collision/reaction cell (CRC).

Analysis of cannabis and hemp by ICP-MS

Development of the AOAC method was performed on an Agilent 7850 ICP-MS, and the same type of instrument was used in the work described in the webinar.

7850 ICP-MS features to ensure accurate data

The 7850 ORS[4] CRC was operated in He collision mode, which reduces common polyatomic interferences using kinetic energy discrimination (KED). He KED allows the same cell conditions to be used for the quantitative analysis of multiple elements in varied sample types, simplifying method development and routine operation.

The 7850 also addresses potential overlaps from doubly charged rare earth elements (REEs) using an automated 'half-mass correction' in ICP-MS MassHunter software (version 5.1 or later). The correction ensures accurate analysis of As, Se, and Zn in the presence of REEs (*4*).

To enable high and variable sample matrices to be analyzed routinely, the 7850 includes the Ultra High Matrix Introduction (UHMI) aerosol dilution system. Operating the 7850 at UHMI-4 provides robust plasma conditions that ensured internal standard (ISTD) signals were stable throughout an analysis lasting almost 24 hours, as shown in Figure 1. Details of the 7850 ICP-MS operating conditions and microwave digestion program are provided in the application note (*3*).

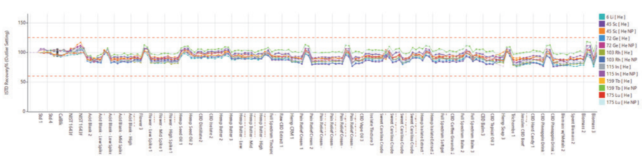


Figure 1. ISTD recoveries over almost 24 hours.

In addition to the quantitative analysis, the 7850 can use QuickScan data to provide IntelliQuant semiquantitative results. IntelliQuant results are based on a two-second full mass spectrum scan measured in He mode. The IntelliQuant data can be displayed as a Periodic Table heat map, as shown in Figure 2 for a softgel sample. The darker heat map color clearly indicates a relatively high concentration of Hg and Pb in the softgel sample.

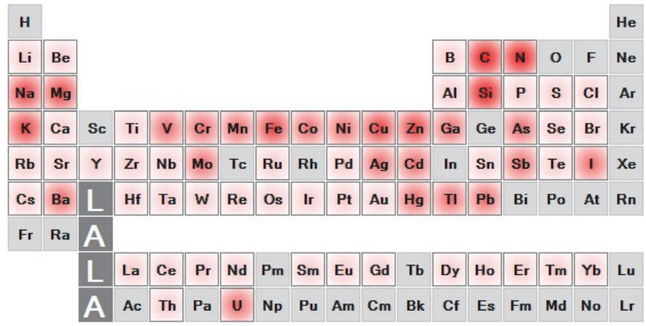


Figure 2. ICP-MS IntelliQuant heat map results for a softgel product.

As Jenny Nelson described in the recent webinar (*2*), the He mode IntelliQuant data allows isotope templates to be used for element confirmation. Confirmatory results are recommended in several regulated methods as they allow an independent check of the quantitative results. The presence of Hg and Pb in the softgel sample was confirmed from the good match between the spectrum peaks and the IntelliQuant templates (Figure 3).

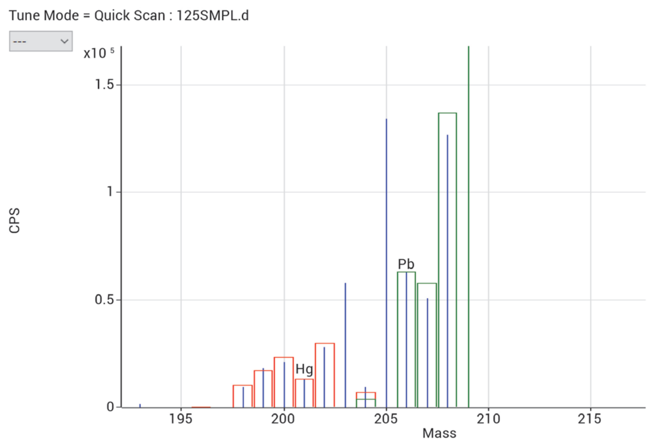


Figure 3. IntelliQuant isotope templates confirm presence of Hg and Pb (Tl at *m/z* 203 and 205 is not labelled). Peak at *m/z* 209 is Bi ISTD.

More information

1. AOAC Expert Review Panel (ERP) for Chemical Contaminants in Cannabis Methods, August, 2021 https://www.aoac.org/news/august-2021-analytical-methods-week-highlights/
2. Webinar link: https://cem.com/en/heavy-metals-in-cannabis-efforts-towards-an-official-aoac-method
3. J. Nelson *et al*, Agilent publication 5994-4080EN
4. T. Kubota, Agilent publication 5994-1435EN

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

Agilent Virtual Symposium on Advances in ICP-MS Performance for Semiconductor Applications

Abe Gutierrez, Bert Woods, Emmett Soffey, and Yan Cheung, Agilent Technologies, Inc.

Semiconductor industry status and trends

Semiconductors have been in the news recently due to chip shortages caused by surging demand for products such as consumer electronics, coupled with supply chain and manufacturing disruption during the pandemic.

Semiconductor manufacturers are also constantly striving to increase manufacturing yields and develop more advanced electronic devices with smaller scale, greater transistor density, higher speed, and lower power requirements.

ICP-MS has been an essential tool for semiconductor manufacturers and high purity chemicals suppliers for decades. In a September 2021 virtual symposium, Agilent ICP-MS specialists discussed how key innovations have enabled Agilent ICP-MS to continue to meet the evolving analytical requirements of the semiconductor industry.

A recording of the 3-hour symposium can be accessed at Agilent Semiconductor Virtual Symposium (on24) including sessions on:

- Chasing Zero: Ultra Trace Analysis by ICP-QQQ (starts approximately 4 minutes into the recorded webinar)
- History of ICP-MS and ICP-QQQ in the Semiconductor Industry (starts at ~45 minutes)
- Trace-Level Analysis of Non-Traditional Elements (starts at ~92 minutes)
- Measuring Nanoparticles (NPs) and Multi-Element NP Analysis by ICP-QQQ (starts at ~119 minutes)
- Q&A (starts at ~131 minutes)

- "Chasing zero". Via video link, Applications Specialist Bert Woods provides practical tips on controlling background contamination levels in the trace element laboratory and optimizing for low detection limits.

- "History of ICP-MS in the Semiconductor Industry". Bert is joined by Agilent Applications Specialist Yan Cheung and Product Specialists Abe Gutierrez and Emmett Soffey for a round-table discussion. These industry experts provide an insightful and entertaining perspective on the significant hardware developments that have enabled ICP-MS to continue to address the most challenging semiconductor applications.
- "Trace Level Analysis of Non-Traditional ICP-MS Elements and Nanoparticles Using ICP-QQQ". Abe and Bert show how ICP-QQQ improves detection of the "difficult" elements Si, P, S, and Cl and enables analysis of the smallest NPs.

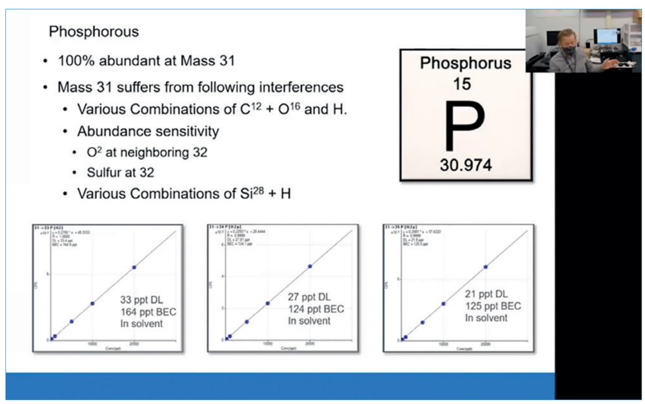


Figure 1. Phosphorus calibrations showing DL of 30 ppt in organic solvent using Agilent 8900 ICP-QQQ.

Single Particle ICP-MS Applications in Advanced Semiconductor Processes

Webinar review by Ed McCurdy and Jenny Nelson, Agilent Technologies, Inc.

Single particle analysis by ICP-MS

Charlie (Qilin) Chan, PhD, PMP, is an Advanced Research Specialist and corporate leader for elemental analysis technology at 3M Corporate Research Analytical Laboratory in St. Paul, Minnesota, US.

Charlie recently presented a fascinating webinar on how he uses single particle ICP-MS (spICP-MS) in applications related to advanced semiconductor processes. In the webinar, he provides an insight into how spICP-MS helps studies into the processes of ultrafiltration and chemical mechanical planarization.

Ultrafiltration

Ultrafiltration is a critical process in the production of ultrapure water and high purity process chemicals used in semiconductor manufacturing. Charlie showed spICP-MS data illustrating how retention efficiency for uniform particle sizes is drastically altered by the surface charges on the filter membrane and the particle. While size-exclusion retention of silica and gold nanoparticles is consistent with membrane pore size, overall retention is strongly affected by membrane-particle interactions (*1*).

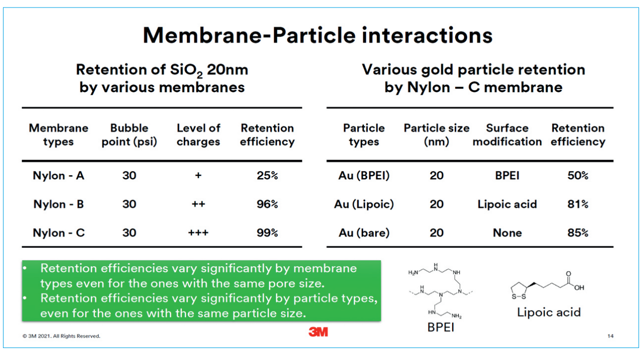


Figure 1. Slide image showing variation in SiO_2 particle retention efficiency for different membrane types and particle coatings. © 3M. Reproduced with permission.

Chemical Mechanical Planarization (CMP)

CMP is an essential step in the manufacturing of semiconductor chips. A CMP slurry containing silica, alumina, or ceria is placed on a rotating pad and the silicon wafer is pressed down onto the pad surface. CMP removes a precise amount of material and smooths the wafer surface between chip processing steps.

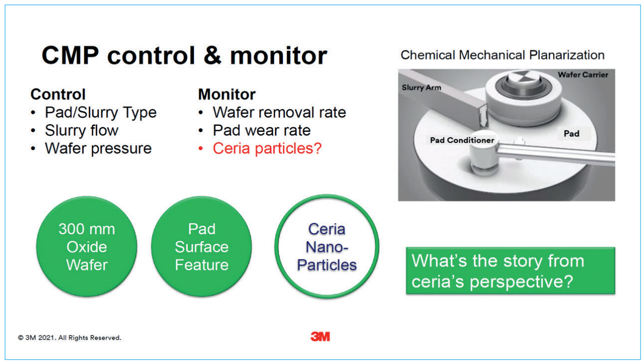


Figure 2. Slide image showing critical variables in the CMP process. © 3M. Reproduced with permission.

In the webinar, Charlie explains how he used spICP-MS to demonstrate that wafer processing caused a small but statistically significant change in ceria particle sizes (*2*).

These spICP-MS results enable a greater understanding of how ceria particles respond to CMP pad types and conditions such as slurry flow rate and wafer pressure.

The on-demand webinar recording is available at: Semiconductor and Specialty Chemical Industry

References

1. Q. Chan, M. Entezarian, J. Zhou, R. Osterloh, Q. Huang, M. Ellefson, B. Mader, Y. Liu, M. Swierczek, *J. Memb. Sci.* 599, **2020**, 117822
2. L. Zazzera, Q. Chan, J. Stomberg, A. Simpson, C. Loesch, D. LeHuu, D. Muradian, U.R.K. Lagudu, B. Mader, *ECS J. Solid State Sci. Technol.* 10, **2021**, 34009

Determination of Hexavalent Chromium in Drinking Water by Ion Chromatography (IC)-ICP-MS

Yan Cheung, Agilent Technologies, Inc., Jayesh Gandhi and Amy Furreness, Metrohm Inc., Lori Allen, Matthew Natschke, Hannah Tangen, and Chris LeValley, University of Wisconsin, Parkside, USA

Regulatory limits for chromium

Defining regulatory limits for chromium (Cr) can be problematic because the different chemical forms present very different health risks. Cr(III) is an essential trace nutrient and not a significant health concern, while Cr(VI) is toxic and carcinogenic (*1*) and should therefore be subject to strict regulation.

However, pH dependent species interconversion between Cr(III) and Cr(VI) can easily occur in samples before and during sample collection and preservation. A further issue is that species-specific methods for Cr(VI) analysis are not widely available in routine testing labs. Regulations for Cr(VI) include the California Office of Environmental Health Hazard Assessment (OEHHA) Public Heath Goal, which specifies a maximum concentration of 0.02 µg/L Cr(VI) in drinking water.

Determination of Cr(VI) using IC-ICP-MS

ICP-MS is an elemental analysis technique, typically used to quantify total concentrations. However, ICP-MS can easily be connected to a chromatography device – most commonly HPLC – to separate the different chemical forms before they are introduced to the ICP-MS, giving species-specific results. HPLC systems typically use stainless steel components, so measuring Cr species at trace levels requires these components to be replaced with inert materials, adding to the system cost.

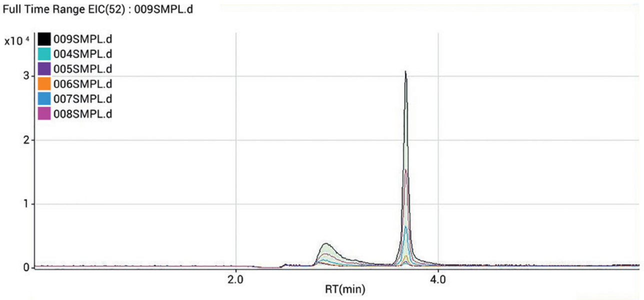


Figure 1. Overlaid chromatograms of Cr(VI) standards from 0.01 to 1 ppb using IC-ICP-MS (high standard at 10 ppb excluded for clarity).

A simple and inexpensive alternative is available using an ion chromatograph (IC) connected to the ICP-MS. A recent Agilent application note shows the performance of a new method for trace Cr(VI) analysis in drinking water (*2*). The new method used a Metrohm 940 Professional IC fitted with a Metrosep ASUPP4 250/4.0 column, connected to an Agilent 7800 ICP-MS.

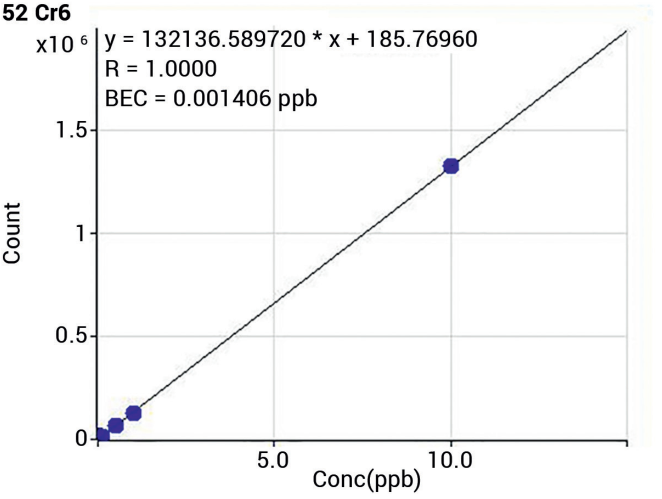


Figure 2. Calibration for Cr(VI) from 0.01 to 10 ppb using IC-ICP-MS.

Overlaid chromatograms for low-level Cr(VI) standards and a calibration for Cr(VI) are shown in Figures 1 and 2, respectively. Method validation was performed according to the guidelines in EPA Method 218.7. The method performance tests gave a Method Detection Limit (MDL) of 0.003 µg/L. The Minimum Reporting Limit (MRL) was confirmed at 0.020 µg/L, meeting the requirements for the California OEHHA Public Health Goal.

References

1. L. M. Calder, in: J. O. Nriagu and E. Nieboer, Eds., Chromium in the Natural and Human Environments, Wiley and Sons, New York, 1988, 215–229.
2. Yan Cheung *et al*, Agilent publication 5994-4295EN

Consumables News: Semiconductor Application-Specific Consumables and Easy-Fit ICP-MS Supplies

Gareth Pearson, Agilent Technologies, Inc.

Semiconductor application-specific supplies

Agilent works closely with users in the semiconductor industry to develop and optimize ICP-MS systems and consumables for the most demanding applications. Based on feedback from analysts, Agilent has developed a platinum-tipped version of our popular nickel-plated sampling cone and an O-ring free torch injector for the PFA inert sample introduction kits.

Agilent Pt-tipped, Ni-plated sampling cones are designed for routine analysis of corrosive chemicals including concentrated HNO_3, HCl, HF, 9.8% H_2SO_4, 1% H_3PO_4, and NH_4OH, and HF-digested Si. During testing on Agilent ICP-MS systems, the new Pt-tipped cones were found to:

- Resist etching of the copper base by aggressive acid matrices, maximizing cone lifetime.
- Reduce the need for cleaning and minimize the risk of damage from overcleaning.

Find out more at https://explore.agilent.com/semi-con

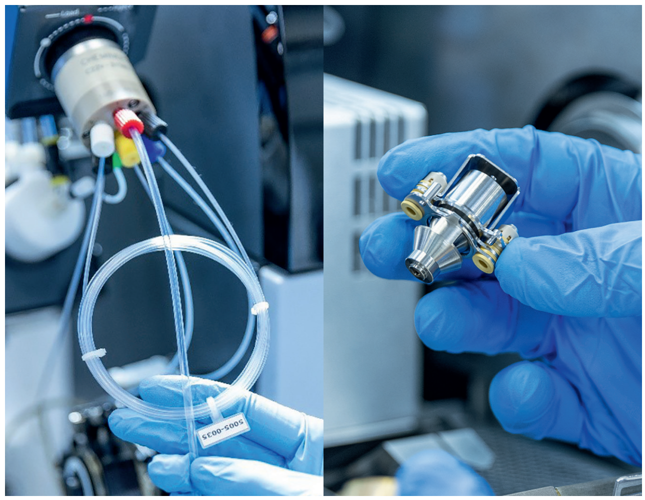
ICP-MS labs that run semiconductor applications – where the lowest background levels are required – or analyze samples that contain HF or other aggressive chemicals, typically use an inert sample introduction system. Agilent ICP-MS inert kits now contain an O-ring free torch, which makes it easier to remove the injector from the outer quartz torch body for maintenance. To learn more, download the flyer, 5994-3839EN.

Easy-fit, no-trim sample loops for ISIS 3

Agilent ICP-MS Journal 85 included an article on Easy-fit peristaltic pump tubing, which is pre-cut and flared for easy and consistent, leak-free fitting. The Easy-fit consumables range has now been extended to include pre-cut sample loops for the Agilent Integrated Sample Introduction System (ISIS-3) discrete sampling device.

The pre-cut, fixed-volume ISIS 3 loops ensure that methods are consistent and timing for the sample load, acquisition, and wash out steps is uniform, enabling quick and simple setup. There is no need for loop cutting and assembly or manual signal timing during method setup.

Learn more and order online



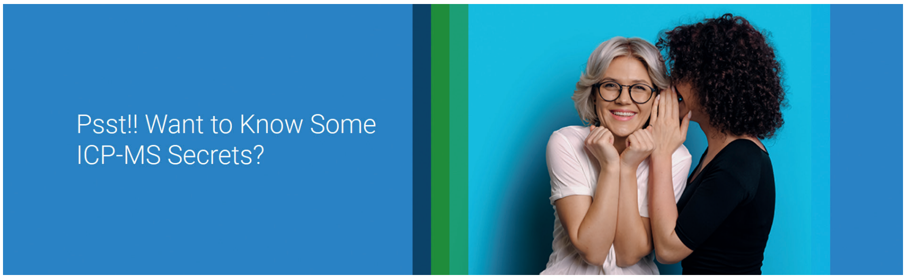
Maintenance-free, Easy-fit x-lens

Lens cleaning on Agilent ICP-MS systems is a quick and simple maintenance task as the lens is in front of the gate valve. Most routine labs clean the x-type Extract Lens and Omega Lens assembly every few thousand samples, depending on the sample types.

For labs that aim for 24/7 routine operation, Agilent can now offer an alternative approach with an optional, single-piece x-lens. The Easy-fit lens assembly is cost effective for routine applications, meaning it can be treated as a consumable. There's no need to clean the lens, just replace it to return your ICP-MS to peak performance.

Find the Easy-fit x-lens assembly for your ICP-MS model online

Demystifying ICP-MS: How to improve accuracy and minimize time-consuming and wasteful activities in ICP-MS analysis



Check out the four short videos starring Agilent ICP-MS expert Glenn Woods. Glenn uses simple language, humor, and analogies to explain four important ICP-MS functions that simplify routine analysis on the Agilent 7850 ICP-MS:

- Dealing with chemical instability issues in your samples
- How to dilute ICP-MS samples without using liquid diluent
- The secret to confirming a result without rerunning a sample
- How to do ICP-MS analysis from outside the lab

Link: https://explore.agilent.com/7850-icp-ms-tips-videos

Breaking news: Agilent commits to net-zero emissions of greenhouse gases by the year 2050. Learn more

Latest Agilent ICP-MS publications

- **Application note:** Determination of Heavy Metals in Cannabis and Hemp Products Following AOAC Method for ICP-MS, 5994-4080EN
- **Application note:** Ultrapure Process Chemicals Analysis by ICP-QQQ with Hot Plasma Conditions, 5994-4025EN
- **Application note:** Authenticating Rice by Elemental Profiling Using ICP-MS and Statistical Modeling, 5994-4043EN
- **Application note** *(updated)*: USP <232>/<233> and ICH Q3D Elemental Impurities Analysis, 5991-8149EN
- **Application note** *(updated)*: Determination of Trivalent and Hexavalent Chromium in Toy Materials, 5991-2878EN
- **Application brief:** Determining Compliance with Baby Food Heavy Metals Levels, 5994-3714EN
- **Technical overview:** Resolving REE^{2+} Overlaps on Arsenic and Selenium With Hydrogen Cell Gas, 5994-4071EN
- **Technical flyer:** Importance of Controlling Space Charge Effects in ICP-MS, 5994-3967EN

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