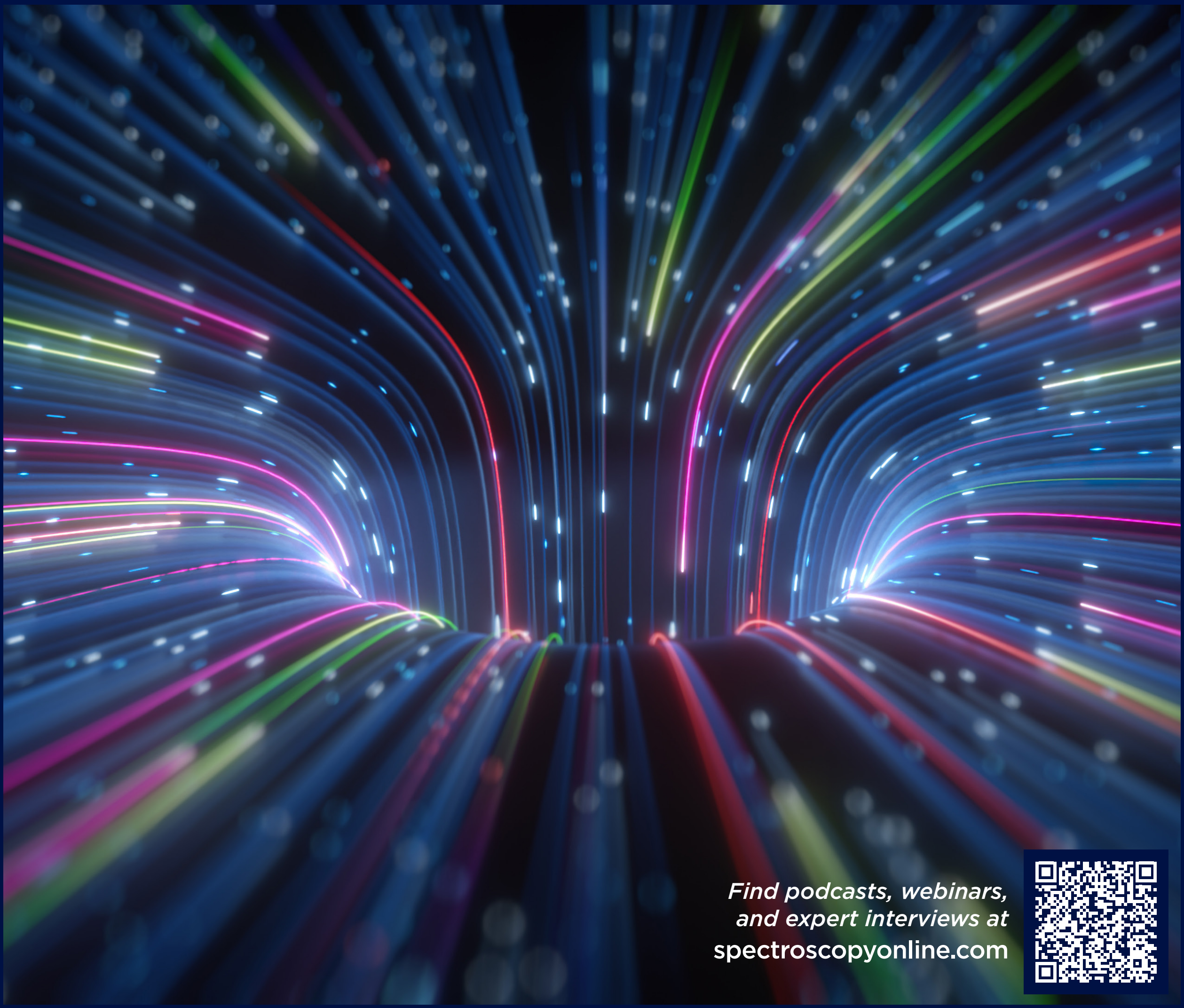
SEPTEMBER 2025

Spectroscopy®

SOLUTIONS FOR MATERIALS ANALYSIS



ADVANCES IN ICP-MS BEST PRACTICES

Supplement

TABLE OF CONTENTS

Spectroscopy

FEATURES

6 **Best Practices for Optimizing ICP-MS Analytical Methodology: Impact of the Evolving Application Landscape**
Robert Thomas

11 **Unlocking ICP-MS Accuracy and Productivity with Microwave Digestion**
Michael Coppola and Guilio Colnaghi

19 **The Role of the Nebulizer in ICP-MS: Design Considerations, Selection Criteria, and Optimization Guidelines**
Justin Masone

29 **Enhancing ICP-MS Efficiency with an Innovative, Low-Maintenance Nebulizer**
Sergei Leikin and Mary Kay Amistadi

38 **Behind the Signal Drop: Distinguishing Between Cone and Sample Introduction Issues in ICP-MS**
Genette Alcaraz, Elizabeth Ranalli, and Sergei Leikin

44 **Silent Saboteurs: Hidden Contaminants, Impurities, and Artifacts in Trace Element Analysis**
Patricia Atkins

September 2025

Cover Art: NeoLeo - stock.adobe.com
captivating spectrum lines

Like @SpectroscopyMagazine on Facebook

Follow @SpectroscopyMag on X

Join the Group @SpecGroup on LinkedIn

Subscribe to our newsletters for practical tips and valuable resources

LISTEN ONLINE

ANALYTICALLY SPEAKING
PODCAST
LC|GC | Spectroscopy

www.SpectroscopyOnline.com/
analytically-speaking-podcast

ALSO AVAILABLE ON
Apple Podcasts, SoundCloud, Google Podcasts

Spectroscopy® (ISSN 0887-6703 [print], ISSN 1939-1900 [digital]) is published January, February, March, April/May, June, July/August, September/October and November/December (8 times a year) by MultiMedia Healthcare LLC, 259 Prospect Plains Rd Unit H Monroe Township NJ 08831-3820. *Spectroscopy* is distributed free of charge to users and specifiers of spectroscopic equipment in the United States. Spectroscopy is available on a paid subscription basis to nonqualified readers at the rate of: U.S. and possessions: 1 year (8 issues), $106.00; Canada/Mexico: 1 year, $135.00; International: 1 year (8 issues), $210.00. Periodicals postage paid at Trenton, NJ 08650 and additional mailing offices. POSTMASTER: Send address changes to Spectroscopy, P.O. Box 457, Cranbury NJ 08512-0457. PUBLICATIONS MAIL AGREEMENT NO. 40612608, Return Undeliverable Canadian Addresses to: IMEX Global Solutions, P. O. Box 25542, London, ON N6C 6B2, CANADA. Canadian GST number: R-124213133RT001. Printed in the U.S.A.


CEM
BLADE™
The Future of
Microwave Digestion
Learn more at cem.com/blade
BLADE
Methods
Run
Data
BLADE
Microwave Digestion System
CEM

Spectroscopy

AN MJH life sciences® BRAND

EDITORIAL

Senior Vice President, Content
Allie Karas, MA
akaras@mjhlifesciences.com

Vice President, Content
Alicia Bigica
ABigica@mjhlifesciences.com

Associate Editorial Director
Caroline Hroncich
CHroncich@mjhlifesciences.com

Executive Editor
Jerome Workman
JWorkman@mjhlifesciences.com

Managing Editor
John Chasse
JChasse@mjhlifesciences.com

Senior Editor
Will Wetzel
WWetzel@mjhlifesciences.com

CUSTOM PROJECTS

Senior Custom Content Manager
Jeanne Linke Northrop
JLinke@clinicalcomm.com

Custom Content Specialist
Megan Manzano
MManzano@mjhlifesciences.com

Custom Content Specialist
Shannon Stolz
SStolz@mjhlifesciences.com

CREATIVE

Creative Director
Melissa Feinen
MFeinen@mjhlifesciences.com

Associate Art Director
Chrissy Bolton
cbolton@mjhlifescience.com

PUBLISHING/SALES

Executive Vice President, Healthcare and Industry Sciences
Brian Haug
BHaug@mjhlifesciences.com

Group Publisher
Stephanie Shaffer
SShaffer@mjhlifesciences.com

Senior Account Manager
Timothy Edson
TEdson@mjhlifesciences.com

National Account Manager
Gerard Onorata
GOnorata@mjhlifesciences.com

CONTENT MARKETING

Senior Virtual Program Manager
Lindsay Gilardi
LGilardi@mjhevents.com

Digital Production Manager
Sabina Advani
SAdvani@mjhlifesciences.com

OPERATIONS

Print & Circulation Director
Jonathan Severn
JSevern@mjhlifesciences.com

Print Coordinator
Amber Immordino
AImmordino@mjhlifesciences.com

Reprints
Stephanie Shaffer
SShaffer@mjhlifesciences.com

CORPORATE

Chairman & CEO
Mike Hennessy Jr

Chief Financial Officer
Neil Glasser, CPA/CFE

Chief Operating Officer
Beth Buehler

Chief Brand Officer
Silas Inman

Chief Data Officer
Terric Townsend

Executive Vice President, Global Medical Affairs & Corporate Development
Joe Petroziello

Senior Vice President, Human Resources & Administration
Shari Lundenberg

Senior Vice President, Mergers & Acquisitions, Strategic Innovation
Phil Talamo

Executive Creative Director
Jeff Brown

Founder
Mike Hennessy Sr
1960 - 2021

485F US Highway One South, Suite 210
Iselin, NJ 08830 • (609) 716-7777

Editorial Advisory Board

MANUSCRIPTS: To discuss possible article topics or obtain manuscript preparation guidelines, contact the managing editor at JChasse@mjhlifesciences.com. Publishers assume no responsibility for safety of artwork, photographs, or manuscripts. Every caution is taken to ensure accuracy, but publishers cannot accept responsibility for the information supplied herein or for any opinion expressed.

SUBSCRIPTIONS: For subscription information: *Spectroscopy*, P.O. Box 457, Cranbury, NJ 08512-0457; email.customerservice@mjhlifesciences.com. Delivery of *Spectroscopy* outside the U.S. is 3–14 days after printing.

CHANGE OF ADDRESS: Send change of address to *Spectroscopy*, P.O. Box 457, Cranbury, NJ 08512-0457; provide old mailing label as well as new address; include ZIP or postal code. Allow 4–6 weeks for change. Alternately, send change via e-mail to email.customerservice@mjhlifesciences.com for address changes or subscription renewal.

C.A.S.T. DATA AND LIST INFORMATION: Contact Stephanie Shaffer, (774) 249-1890; e-mail: SShaffer@mjhlifesciences.com

Reprints: Contact Stephanie Shaffer, e-mail: SShaffer@mjhlifesciences.com

INTERNATIONAL LICENSING: Contact Kim Scaffidi, e-mail: KScaffidi@mjhlifesciences.com

CUSTOMER INQUIRIES: Customer inquiries can be forwarded directly to MJH Life Sciences, Attn: Subscriptions, 2 Clarke Drive, Suite 100, Cranbury, NJ 08512; e-mail: email.customerservice@mjhlifesciences.com

Best Practices for Optimizing ICP-MS Analytical Methodology: Impact of the Evolving Application Landscape

Robert Thomas

I'm honored to be the editor of this special ICP-MS supplement for *Spectroscopy* magazine. It gives me the opportunity to focus on what I believe is largely being ignored in the open literature—educational material that can be easily understood and used by novices or someone who has limited experience in using the technique and wants to get the most out of their instrument. We see a multitude of applications that promote the high-performance capabilities of inductively coupled plasma–mass spectroscopy (ICP-MS) using triple quads and all the innovative bells and whistles that vendors like to sell, but there are very few publications that make life easier for the ICP-MS practitioner who just has a standard system and wants to optimize their analysis.

So, I have asked five companies I've collaborated with over the years to contribute articles to this special issue that focus on best practices for optimizing ICP-MS analytical methodology and, in particular, developing instrument components and accessories to deal with difficult sample matrices. Some of the topics covered include improving microwave digestion, maximizing sample delivery, improving detection capability, reducing contamination, minimizing interferences, and extending cone lifetime. In addition, I have also taken the opportunity to look at the application landscape since the technique was first commercialized in 1983 to get a better understanding of how ICP-MS practitioners have adapted these best practices to meet the diverse and varied demands of their sample workload.

Over 40 Years Old

The commercial ICP-MS marketplace has just celebrated its 40th anniversary. At the 1983 Pittsburgh Conference in Atlantic City, New Jersey, Sciex introduced the Elan 250 quadrupole-based ICP mass spectrometer. It was another 12 months before its joint venture with PerkinElmer was announced, but the event was the beginning of the meteoric rise of ICP-MS as the dominant multielement technique used for ultra-trace elemental analysis. Many vendors and instruments designs have come and gone over that time, but single quadrupole technology still dominates the instrumental landscape with approximately 80% of the marketplace.

Today, there are approximately 2000 ICP-MS installations worldwide every year, comprising of single quadrupole systems, triple/multi quads, time of flight (TOF) and magnetic sector instruments performing a wide variety of applications, including routine, high-throughput multielement analysis, isotopic fingerprinting, single particle and cell studies, and elemental speciation coupled with high performance liquid chromatography (HPLC), to name a few (1). As more and more laboratories invest in the technique, the list of applications is getting significantly larger and more diverse. The most common ones include environmental monitoring, geochemical, metallurgical, petrochemical, petroleum, food, agricultural, clinical, toxicology, semiconductor, industrial, rare earth, energy, nuclear, pharmaceutical, and cannabis, but every year it seems that a new market has woken up and realized the potential of its capabilities.

Market Segments

As a result of the widespread use and acceptability of ICP-MS, the cost of commercial instrumentation has dramatically fallen over the past 25 years. When the technique was first introduced, $250,000 was a fairly typical amount to spend for a single quadrupole-based system, whereas today one can purchase one for under $150,000. Although it can cost a great deal more to invest in a "triple quadrupole" collision or reaction cell instrument, most laboratories that are looking to invest in the technique should be able to justify the purchase of an instrument without price being a major concern.

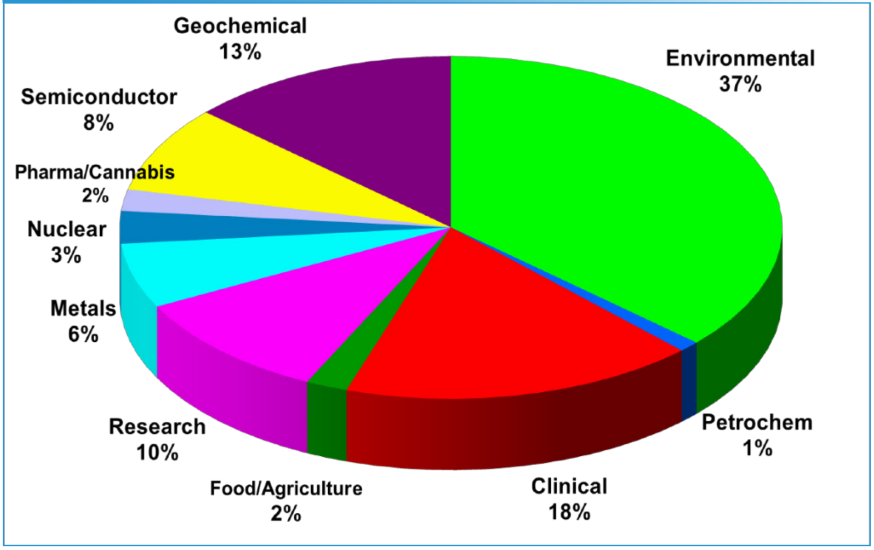


FIGURE 1: Major market segments being addressed by ICP-MS on a global basis.

One of the benefits of this kind of price erosion is that, slowly but surely, the atomic absorption (AA) and ICP-optical emission spectroscopy (OES) user community are being attracted to ICP-MS, and as a result, the technique is being used for more and more diverse application areas. Figure 1 shows a percentage breakdown of the major market segments being addressed by ICP-MS on a worldwide basis today.

Three points should be emphasized here. First, the data can be significantly different on a geographical or regional basis, because of factors such as a country's commitment (or lack of it) to environmental concerns or the size of a region's electronics or nuclear industry, for example. Second, many laboratories carry out more than one type of application, and as a result can be represented in more than one market segment. Finally, the research market segment has been listed as a separate category to show the instruments that are being used in an academic environment or for non-routine applications. However, many universities, federal organizations, or corporate R&D groups might be using their instrumentation for research purposes in a particular application segment. For these reasons, these data should only be considered an approximation for comparison purposes.

Performance Capability

Without question, extremely low detection capability, combined with increased sample throughput, have been the main incentives for analytical chemists to invest in ICP-MS. The technique is now considered a mature tool that is being used for the routine, high-throughput analysis of complex samples with very little or no sample preparation. In the early days, it was used by academic institutions and large corporate R&D laboratories. Nowadays, ICP-MS instrumentation is also being used by contract laboratories in the routine analysis of environmental, pharmaceutical and biomonitoring applications, among others. This has been made possible by greater automation on the sample preparation side and seamless method development tools, which have resulted in far less operator expertise required to run them. It is also worth pointing out that stricter regulations are requiring lower and lower detection limits (DLs), particularly in the semiconductor, biomonitoring, food and beverage market segments. In addition, for the past five years, the rapid growth in the legal cannabis industry has meant cultivators and processors have to ensure the safety of consumer products by meeting strict state-based limits for contaminants like heavy metals. So, the drive toward lower detection limits is just one factor, whereas the other is the analytical needs and demands of the customer. For example, the electronics industry is faced with the enormous challenge of "chasing zero," so manufacturers must measure the lowest level of elemental impurities in process chemicals and electronic devices to ensure the highest performance of their products.

Analytical Challenges

As ICP-MS has become widely applicable in a variety of application areas, it has become more readily accessible to startup laboratories. Part of this is the development of turnkey methods for food, environmental, biomonitoring, geochemical, pharmaceutical, and cannabis applications for inexperienced and novice users, which will be critical for the growth of this technique in these market segments.

Important considerations include ease of use, seamless software, low maintenance, and the ability to handle different matrices, which give the analyst confidence in the results being generated and ensure they get the correct answer the first time and every time. The user community expects these qualities, particularly in high-throughput laboratories. They want to run the instrument for extended periods with minimum maintenance and short downtime while simultaneously running multiple samples, many containing high concentrations of mineral acids. It is important to emphasize that this kind of sampling environment is not really instrument friendly. When samples are being run that contain high concentrations of mineral acids and dissolved solids, it will take its toll on the instrument's sample introduction components. Therefore, the instrument must be rugged enough to handle this kind of sample matrix, without contributing significantly to the routine maintenance of the system, while at the same time delivering high-quality data, day in and day out.

Therefore, ruggedness is critically important when designing an ICP-MS instrument. However, this is just one side of the analytical challenge. As mentioned previously, the semiconductor industry is the one application area where this kind of ruggedness is expected, but the lowest possible detection limits are also required. Just 15 years ago, 10 ppt was the guideline for elemental impurities in semiconductor process chemicals; today, the industry is requiring 1-2 ppt. This puts unique demands on the design of any instrument used for this kind of analysis.

Consequently, with the push for lower and lower detection capabilities, it is becoming a major challenge not only to develop instrumentation that has high analyte sensitivity and extremely low background noise, but also to have an ultra-clean lab and sample preparation environment that sets the stage for the detection of elements at such low levels. Both factors are absolutely critical. The trend towards ease of use has meant that operators have less time to be involved with the analytical procedure and therefore are often not versed in the nuances of working in the ultra-trace environment and won't necessarily recognize issues which require user intervention, should they arise. For that reason, instruments have to be rugged, and robust in the face of difficult and complex sample matrices but using preventative maintenance procedures should be able to diagnose and mitigate potential problems before they actually happen.

Best Practices

So, with this as background information, here are a series of articles covering "best practices" from five different companies who specialize in the fields of sample preparation, sample introduction and sample delivery to help ICP-MS practitioners better deal with their diverse and varied sample matrices and workloads.

Unlocking ICP-MS Accuracy & Productivity with Microwave Digestion

Michael Coppola and Guilio Colnaghi, Milestone Inc.

Achieving high accuracy and productivity in ICP-MS analyses begins with optimized sample preparation. Microwave digestion provides significant advantages for laboratories analyzing diverse matrices, enabling precise elemental recovery, lower detection limits, faster throughput, and reduced contamination risk. This article discusses pre-analytical best practices, recent innovations, and other productivity tools to consider throughout the workflow, explaining how these factors collectively enhance analytical accuracy and data integrity. Real-world case studies will highlight how laboratories can implement these strategies to strengthen their ICP-MS methods and overcome common sample prep challenges.

The Role of the Nebulizer in ICP-MS: Design Considerations, Selection Criteria, and Optimization Guidelines

Justin Masone,
Glass Expansion

This article examines best practices for optimizing nebulizer selection and performance in ICP-MS. It provides an educational overview of nebulizer design evolution, highlighting the benefits of various designs and their impact on aerosol characteristics, such as droplet size and distribution. Aerosol diagnostic data will be incorporated to demonstrate how both nebulizer design and operating conditions influence these characteristics, and how this relates to sensitivity, precision, and overall analytical performance. Finally, we explore modern aerosol dilution and aerosol filtration techniques, demonstrating their role in enhancing nebulizer aerosol quality to maximize instrument performance.

Enhancing ICP-MS Efficiency with an Innovative, Low-Maintenance Nebulizer

Sergei Leikin, Texas Scientific
Mary Kay Amistadi,
University of Arizona
Laboratory for Emerging Contaminants

Robust ICP-MS analysis of complex matrices—especially those containing even small particulates and high salt levels—remains a significant challenge due to frequent clogging of conventional concentric nebulizers. Commonly recommended filtration or centrifugation steps are labor-intensive, increase costs and extend turnaround time. To overcome these limitations, an innovative nebulizer featuring a robust non-concentric design with a relatively large sample channel internal diameter was employed. This design provides good resistance to clogging and improved tolerance to tough matrices. Results from over two years of continuous operation across a wide range of challenging sample types showed a substantial increase in ICP-MS throughput. Overall analytical efficiency was significantly enhanced through a combination of factors: elimination of nebulizer blockages, removal of time-consuming filtration or centrifugation steps, and a substantial reduction in nebulizer maintenance.

Behind the Signal Drop: Distinguishing Between Cone and Sample Introduction Issues in ICP-MS

Liz Ranalli and Genette Alcaraz, Spectron Inc.
Sergei Leikin, Texas Scientific

Reduction in signal intensity or analytical precision is the most common performance issues ICP-MS users are faced with. Troubleshooting these problems can be costly since distinguishing between cone degradation and issues related to the sample introduction system is often challenging—even for experienced analysts. A rational, step-by-step approach to diagnosing and resolving the common ICP-MS performance issues is proposed. It includes practical guidance on inspection criteria and maintenance protocols to determine when to clean, refurbish, or replace interface cones. The proposed approach is intended to reduce instrument downtime and operational costs by extending the cone lifetime and minimizing unnecessary replacement.

Silent Saboteurs: Hidden Contaminants, Impurities, and Artifacts in Trace Element Analysis

Patti Atkins,
Antylia Scientific

Accurate and reproducible elemental quantification by ICP-MS demands rigorous control of all sources of contamination and matrix interference. Trace-level analytes are particularly susceptible to false positives, signal suppression, and spectral overlap originating from suboptimal reagents, environmental background, and instrumental components. This article provides a comprehensive examination of contamination and interference pathways that compromise ICP-MS data quality. Specific focus is given to elemental impurities in high-purity acids and water, contributions from sample preparation vessels and labware, and the chemical and physical behaviors of polyatomic and isobaric interferences. We further address the under-appreciated risks posed by residual organics and microbiological contamination in reagents and diluents, which can alter plasma behavior or introduce matrix effects. Practical mitigation strategies—including best practices in reagent selection, lab hygiene, and reference material validation—are discussed to support high-accuracy measurements in regulated and ultra-trace analytical environments.

Robert Thomas is the principal scientist of Scientific Solutions, a consulting company that serves the educational needs of the trace element user community. He has worked in the field of atomic and mass spectroscopy for more than 50 years, including 24 years for a manufacturer of atomic spectroscopic instrumentation. Rob has led the heavy metals task force on the American Chemical Society (ACS) Committee on Analytical Reagents (CAR) for the past 25 years. As well as being the Atomic Perspectives (AP) column editor, he also serves on the editorial advisory board of Technology Networks and recently accepted a position of Assistant Adjunct Professor of Chemistry, at the University of North Dakota. Rob has written over 100 technical publications, including a 15-part tutorial series on ICP-MS, in addition to authoring six textbooks on the fundamental principles and applications of ICP-MS. His most recent book published in October 2023 is entitled, Practical Guide to ICP-MS and Other Atomic Spectroscopy Techniques: A Tutorial for Beginners. Rob has an advanced degree in analytical chemistry from the University of Wales, UK, and is also a Fellow of the Royal Society of Chemistry (FRSC) and a Chartered Chemist (CChem). ●

FEATURED ARTICLE

Unlocking ICP-MS Accuracy and Productivity with Microwave Digestion

Michael Coppola and Giulio Colnaghi

Achieving high accuracy and productivity in inductively coupled plasma–mass spectroscopy (ICP-MS) analyses begins with optimized sample preparation. Microwave (MW) digestion provides significant advantages for laboratories analyzing diverse matrices, enabling precise elemental recovery, lower detection limits, faster throughput, and reduced contamination risk. This article discusses pre-analytical best practices, recent innovations in MW design, and other productivity tools to consider throughout the workflow, explaining how these factors collectively enhance analytical accuracy and data integrity. Real-world case studies will highlight how laboratories can implement these strategies to strengthen their ICP-MS methods and overcome common sample prep challenges.

Inductively coupled plasma–mass spectroscopy (ICP-MS) is widely regarded as one of the most powerful analytical techniques for the detection of trace and ultratrace elements across diverse sample types. However, the technique's ability to provide highly accurate and reproducible data depends significantly on the quality of the sample preparation method employed. Among the various techniques available for pre-analytical sample processing, microwave-assisted acid digestion has become the method of choice in many analytical laboratories due to its ability to effectively break down complex matrices while minimizing contamination and preserving analyte integrity (1,2).

Traditional sample digestion methods, such as open-vessel hot plate or block digestion, have historically posed significant limitations in terms of time consumption, risk of contamination, operator variability, loss of volatile elements, and insufficient digestion of challenging matrices. See Table I for a comparison of hot block vs microwave (rotor-based) technology.

These limitations have led to inaccuracies in elemental recovery, particularly when analyzing samples with high organic content or resistant mineral structures.

Fundamentals of Microwave Digestion

In contrast, microwave digestion utilizes sealed pressure vessels and precise temperature control to accelerate the breakdown of samples in strong acids. The result is a faster, more complete digestion process with minimal loss of volatile elements and significantly improved reproducibility. In high-throughput laboratories and regulated environments such as food safety, environmental monitoring, and pharmaceutical quality control, these advantages translate into measurable gains in productivity, data integrity, and compliance (3).

This article explores the key factors that contribute to successful microwave digestion for ICP-MS workflows. It highlights best practices in pre-analytical preparation, recent innovations in system design, and practical tools to reduce contamination and improve throughput. Real-world examples and performance benchmarks demonstrate how adopting modern digestion strategies can enhance accuracy and reproducibility while streamlining lab operations.

Microwave digestion systems operate on the principle of dielectric heating, where microwave energy is absorbed by polar molecules in acid mixtures, resulting in rapid temperature and pressure increases within sealed vessels. This facilitates efficient sample breakdown and the dissolution of matrix components into a homogenous solution suitable for elemental analysis via ICP-MS.

TABLE I: Hot block vs microwave (rotor) technology			
Feature	**Microwave (Rotor)**	**Hot Block**	**Preferred Technology**
Heating	Direct microwave, fast & uniform	Conductive, slower, uneven	Microwave (rotor)
Speed	Completes digestions quickly	Slow heat transfer	Microwave (rotor)
Temp. Control	Real-time fiber optic/IR	Less precise, external source	Microwave (rotor)
Pressure Control	High-pressure (up to 100 bar)	None	Microwave (rotor)
Throughput	24–44 samples/hr	Lower, block-size dependent	Microwave (rotor)
Quality	Complete digestion, fewer interferences	Incomplete, risk of false positives	Microwave (rotor)
Reagent Use	~50% less	Higher due to evaporation	Microwave (rotor)
Contamination Risk	Closed vessels, minimal loss	Open system, higher risk	Microwave (rotor)
Safety	Monitored pressure/temp	Higher acid fume risk	Microwave (rotor)
Versatility	Broad sample range	Limited to clean matrices	Microwave (rotor)
Maintenance	Low, self-cleaning	Higher, frequent cleaning	Microwave (rotor)
Operational. Cost	Lower	Higher	Microwave (rotor)
Labor	Lower, automated	Higher, manual	Microwave (rotor)
ROI	< 1 year	Poorer ROI	Microwave (rotor)

Overcoming Sample Types and Matrix Challenges

Microwave digestion is suitable for a wide variety of matrices, from easier to digest matrices such as biological tissues, foodstuffs, and environmental samples (soil, water, sediments), to more challenging materials such as polymers, industrial materials, metal alloys and ceramics. Each matrix presents unique challenges in terms of digestion efficiency and contamination risk. For instance, high-fat foods and biological fluids require careful optimization to avoid incomplete digestion, carryover contamination, or exothermic reactions. Samples such as metal alloys and ceramics are particularly challenging to digest, as they require aggressive acid mixtures, and high temperature for extended time periods. Depending on the sample types and throughput needs, laboratories typically employ one of two main microwave digestion platforms: rotor-based, closed-vessel systems or single reaction chamber (SRC) technology, as seen in Figures 1–3 (2,4). Rotor-based systems use various rotors to match specific applications, such as medium-pressure rotor systems suited for high throughput environmental samples, high-performance rotor systems for cannabis and food applications, and high-pressure rotors, which are best used for more challenging matrices such as rocks and metal alloys. An example of a rotor-based microwave digestion system is shown in Figure 2, along with different rotors shown in Figure 3.

FIGURE 1: Single reaction chamber (SRC) microwave technology.

FIGURE 2: Rotor-based microwave technology.

Single-Reaction Chamber (SRC) Technology

For laboratories with particularly challenging organic or inorganic matrices to digest, that are looking to improve throughput and safety as well as increase automation and reduce labor time and costs, SRC technology is another option worth considering. Comparison table of capabilities of rotor-based technology vs SRC technology is illustrated in Table II.

SRC technology was developed to overcome inherent limitations of rotor-based systems, such as limited time at maximum temperature for challenging samples, the need to assemble and disassemble vessels rapidly, batching matrices of similar reactivities, vessel cleaning and limited throughput capa-

TABLE II: Capabilities of rotor-based technology vs single-reaction chamber (SRC) microwave technology

Feature	Rotor-Based	SRC Technology
Run any mixture of samples together (mixed batch)	No	Yes
System automation	Once operator presses start, microwave program commences, then rotor air-cooled. Individual reaction vessels must be manually depressurized by operator.	Once operator presses start, microwave program commences, then system cooled with external chiller and automatically depressurized.
Vessel assembly/disassembly	Multiple components requiring torque wrench	Uses vials with loose-fitting caps – No assembly/disassembly required
Vial/vessel types available	TFM	Quartz, TFM, Disposable glass
Maximum operating temperature	240 °C	280 °C
Maximum pressure	100 bar	199 bar
Sample mass capacity for polymers/chemicals	~0.25 g+ (sample dependent)	~0.5 g+ (sample dependent)
Minimum reagent volume	5 mL	No minimum reagent volume
Initial investment	$$	$$$
Consumable cost	$$	$
Labor cost	$$	$
Running cost	$$	$

bilities. The basic principles and workflow of SRC technology is illustrated in Figure 4.

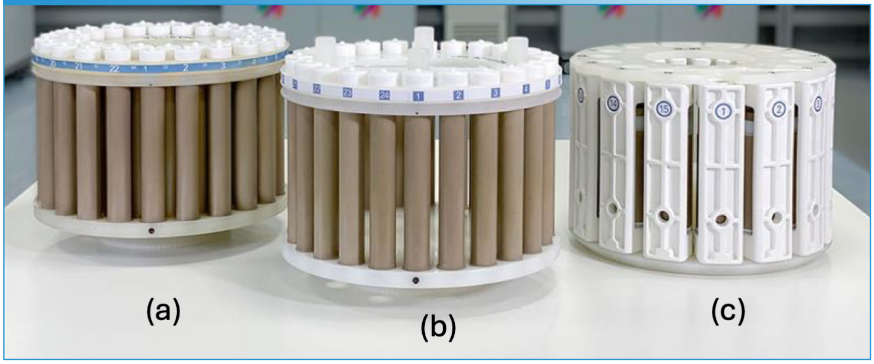


FIGURE 3: Different rotor systems designed for (a) high throughput; (b) high performance; and (c) high pressure.

Microwave Digestion Procedure

A standard digestion procedure involves weighing a representative sample into a clean, chemically inert digestion vessel. Concentrated acids, most commonly nitric acid (HNO_3) alone or in combination with hydrochloric acid (HCl) or hydrogen peroxide (H_2O_2), are added to promote matrix dissolution. Depending on the sample type and elements of interest, other acids such as sulfuric (H_2SO_4), phosphoric (H_3PO_4), hydrofluoric (HF), fluoroboric (HBF_4), or others may be used. Sealed vessels are placed into the microwave system, where the digestion process follows a precisely controlled temperature ramp. For easier-to-digest samples, temperatures typically reach 180–220 °C within a 15–30-min cycle. More difficult matrices may require temperatures up to 280 °C, maintained for 30 min or longer to ensure complete digestion.

Advanced microwave systems incorporate real-time temperature and pressure sensors, allowing for dynamic feedback control and reproducible conditions across multiple vessels. Rotor-based systems and SRC technology, further enable simultaneous digestion of different sample types under uniform conditions, reducing the risk of batch failure. With rotor-based closed-vessel microwave digestion systems, samples of similar reactivities must be run together. However, when utilizing SRC technology, all samples are at the same temperature and pressure conditions, allowing for full mixed-batch capability.

Contamination Controls

Contamination is a major concern in trace elemental analysis. High-purity reagents, Class A glassware, and rigorous cleaning protocols are critical. Many labs employ clean chemistry tools, such as automated acid purification systems (sub-boiling distillation apparatus) as shown in Figure 5 (5).

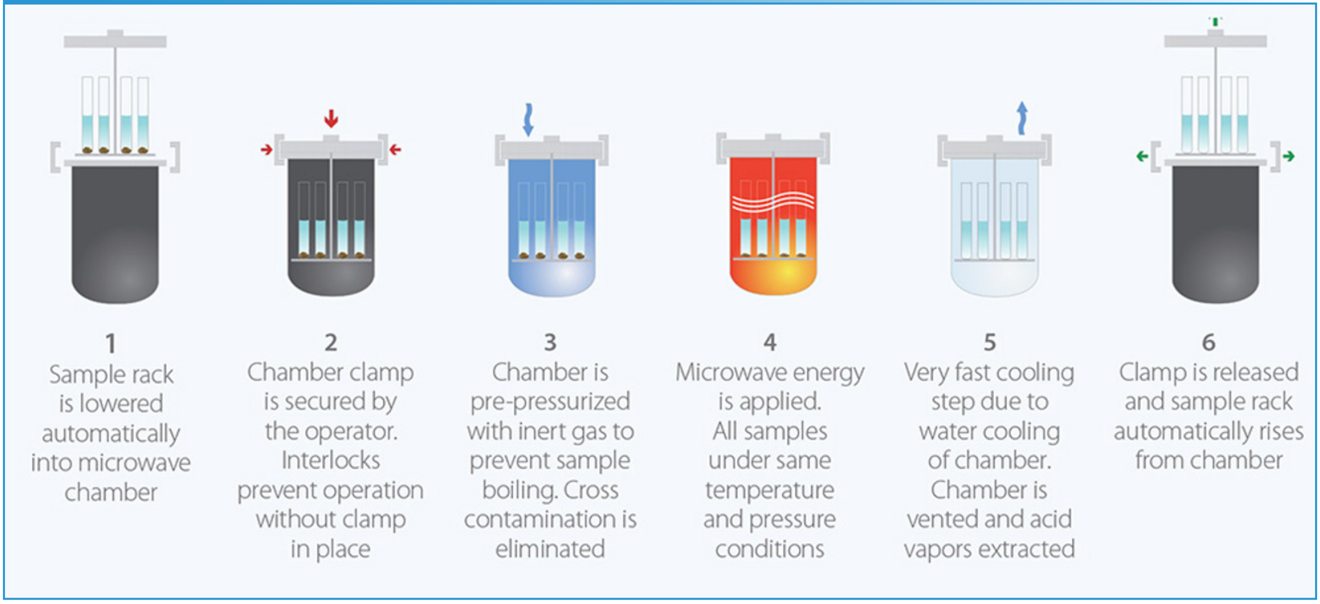


FIGURE 4: Basic principles and workflow of single reaction chamber technology.

Steam Cleaning Systems

Additional useful tools to reduce contamination are acid steam cleaning systems and non-contact reagent dispensers, which minimize exposure, reduce variability, and improve overall laboratory workflow. A commercially-available system is shown in Figure 6.

As mentioned, rigorous cleaning protocols are an essential aspect of the laboratory workflow for any laboratory looking to achieve trace metal detection limits. There are conventional techniques, such as acid baths and acid soaks, as well as using the microwave to clean vessels, but these are not as efficient and effective as acid steam cleaning. With acid steam cleaning, vessels to be cleaned are placed into a glass vat containing HNO_3. The chamber is closed, and the steam cleaning process commences. After about two hours, all vessels are clean and ready to be used.

When working at trace or ultra-trace levels, the use of high-purity or ultra-high-purity acids is critical to minimize contamination and ensure analytical accuracy. Commercial ultrapure reagents represent a significant laboratory expense, often exceeding $1000 USD per liter, and may be subject to extended supplier lead times. In-house sub-boiling distillation offers an effective solution to both supply chain challenges and cost constraints. As a result, laboratories can convert reagent-grade acids into ultrapure quality at a fraction of the commercial price, achieving cost reductions of up to 90% while maintaining the required analytical purity.

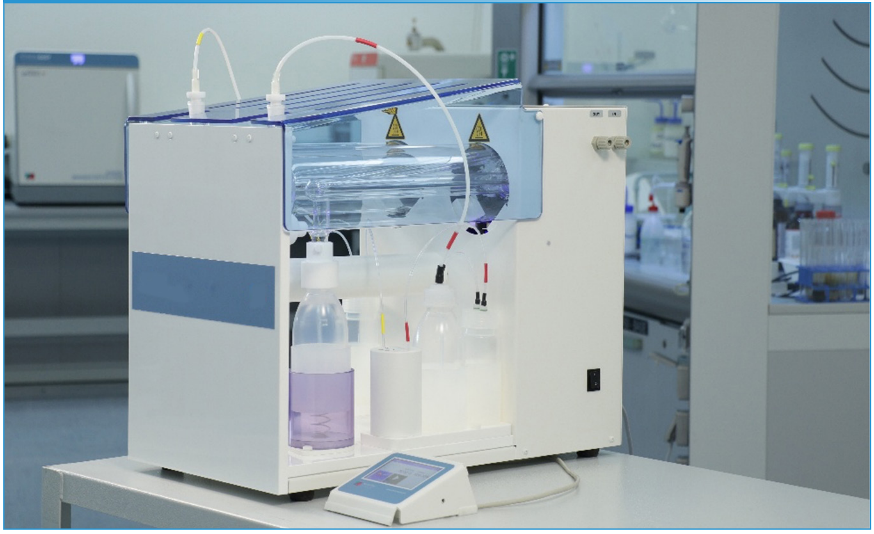


FIGURE 5: Sub-boiling distillation apparatus for acid purification.

Automated Dosing and Dispensing Systems

Dosing reagents into digestion vessels is still one of the most tedious and unpleasant steps in the laboratory, as it requires a significant amount of labor time and cost and exposes the operator to concentrated acids. This step is typically done manually and, as a result, has a number of drawbacks, including safety concerns, human error, and high risk of contamination. Addressing this task is key for laboratories wanting to improve lab safety and sample prep workflow.

One such tool is an automatic dosing and dispensing station. which provides enhanced safety with a hands-off approach—no acid handling or exposure to acid fumes. An automated dosing and dispensing station is shown in Figure 7.

This approach represents a reliable, fully automated way of dispensing acids, which is compatible with a wide range of vials and vessels. Its touchscreen interface streamlines operation, while automation reduces labor and boosts productivity. Constructed from anticorrosive polypropylene, it features a magnetic safety door, integrated fume filtration, and compatibility with up to six reagents, including hydrofluoric acid. Automatic line flushing simplifies acid changes, and a dedicated drain collects waste into a carboy. Real-time reagent and waste monitoring further enhances safety, efficiency, and ease of use.

FIGURE 6: A commercially available acid cleaning system.

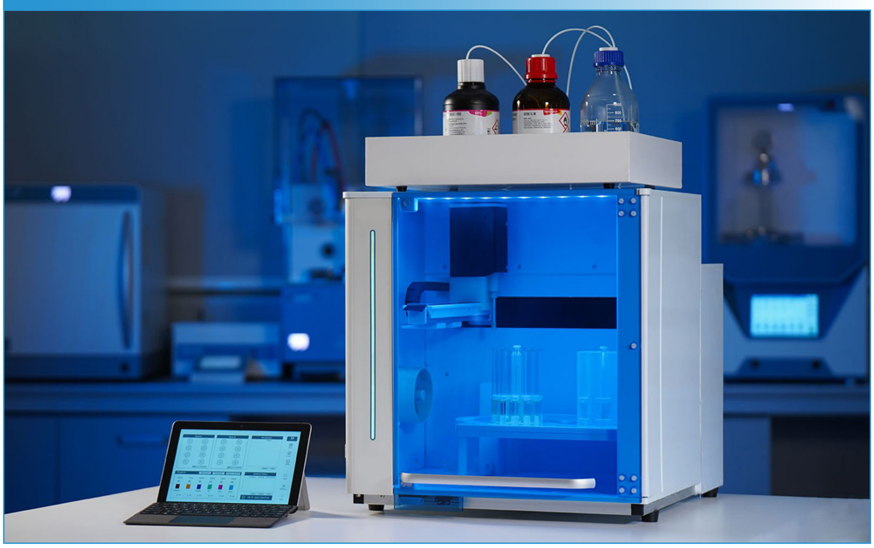


FIGURE 7: A fully automated acid dosing/dispensing system.

Microwave Digestion and ICP-MS Compatibility

Post-digestion handling is just as critical as the digestion process itself. Samples are typically cooled to room temperature, then diluted with ultrapure water to a known volume in Class A volumetric flasks. The final solution must be free of particulates and stable enough to prevent analyte loss or carryover. Vessel materials (PTFE and quartz, for example) should be selected based on chemical compatibility and the detection limits required for specific elements.

When done correctly, microwave digestion provides a robust foundation for ICP-MS measurements with minimal interference, ensuring compliance with regulatory methods such as EPA 3015, EPA 3051, and *USP* <232/233>.

Case Studies

To exemplify the real-world capability of microwave digestion and contamination control systems, here are two examples of contract labs that are realizing the benefits of these technologies.

Aerospace Materials Testing Laboratory

Dirats Laboratories, a leading contract laboratory in advanced elemental analysis for industries such as aerospace, defense, and electronics, specializes in the digestion and analysis of challenging materials including superalloys, ceramics, and composites. Seeking to improve safety, consistency, and turnaround times, they replaced traditional fusion and open-vessel methods with a single reaction chamber microwave digestion system. This upgrade reduced acid use, improved safety, and cut turnaround times by nearly 50% while achieving complete elemental recovery. Paired with an automated acid steam cleaning system, Dirats now operates a fully integrated, contamination-free workflow that safeguards ultra-trace accuracy and enhances laboratory efficiency (5).

Food & Nutraceutical Contract Laboratory

SGS (formerly Chemical Solutions/CSL), a leading contract laboratory based in Harrisburg, PA, specializes in metals, minerals, and elemental species analysis for over 400 clients across diverse industries. With a monthly workload of roughly 4000 samples and demanding turnaround times as short as same-day, CSL relies on a robust suite of microwave (MW) technologies, including four SRC digestion systems, two rotor-based microwaves, and two acid steam cleaning units, to meet extraordinary throughput and analytical precision requirements. The SRC's ability to process mixed sample types at uniform high temperature and pressure ensures complete digestion, even for volatile elements, reducing re-runs and improving recoveries. The rotor-based system provides efficient high-throughput digestion for uniform sample batches, while the acid cleaning system maintains contamination-free labware without consuming valuable instrument time (6).

Conclusion

MW digestion has become an indispensable tool in modern ICP-MS workflows, offering laboratories a reliable, efficient, and reproducible means of preparing samples for trace elemental analysis. The technology addresses longstanding challenges associated with traditional digestion methods, including inconsistent recovery, contamination risks, incomplete digestion and limited throughput.

By adopting closed-vessel microwave digestion systems and adhering to best practices in sample handling, labs can significantly enhance data quality and operational efficiency. The method supports compliance with globally recognized standards while accommodating the rigorous demands of industries such as food safety, environmental testing, and pharmaceutical manufacturing.

Looking ahead, continued innovation in digestion vessel materials, automation, and the integration of additional pre-analytical workflow solutions will further elevate the role of microwave digestion in analytical sciences (3). Laboratories that invest in these advancements will be well-positioned to meet evolving regulatory expectations, enhance productivity, and maintain data integrity, even in the most challenging analytical scenarios.

References

(1) Microwave Digestion for Agriculture Analysis. *Spectroscopy Online*, May, 2025 https://www.spectroscopyonline.com/view/microwave-digestion-for-agriculture-analysis

(2) Microwave Digestion for Battery Materials. *Spectroscopy Online*, April, 2025. https://www.spectroscopyonline.com/view/pre-analytical-workflow-for-lithium-ion-batteries

(3) Innovations in Microwave Sample Preparation: Total Workflow Solutions. *Milestone website*. December, 2024. https://www.milestonesci.com/wp-content/uploads/2024/12/LX_Milestone_Microwave-Sample-Prep_eBook_12.24.pdf

(4) SRC Technology eBook – Spectroscopy. *Milestone website*. August, 2018. https://www.milestonesci.com/wp-content/uploads/2019/01/SRC-Technology-EBook-Spectroscopy-Milestone-August-2018-b.pdf

(5) Lab Profile: Dirats Laboratories. *Milestone website*. June, 2025. https://www.milestonesci.com/wp-content/uploads/2025/06/Lab-Profile-Dirats-06.2025-Final.pdf

(6) Lab Profile: Chemical Solutions (SGS). *Milestone website*. August, 2019. https://www.milestonesci.com/wp-content/uploads/2025/08/Chemical-Solutions-Lab-Profile-USREV012819.pdf

Milestone Products Mentioned in this Article

Figure 1: Single Reaction Chamber Microwave Digestion System–ultraWAVE 3

Figure 2: Rotor-based Microwave Digestion System–ETHOS UP

Figure 3: Sample Holders for ETHOS UP High throughput–MAXI 44 High performance; MAXI 24 HP; High pressure–SK 15 ET

Figure 5: Sub-boiling Distillation–duoPUR

Figure 6: Steam Cleaning–traceCLEAN

Figure 7: Automated Acid Dispensing–easyFILL

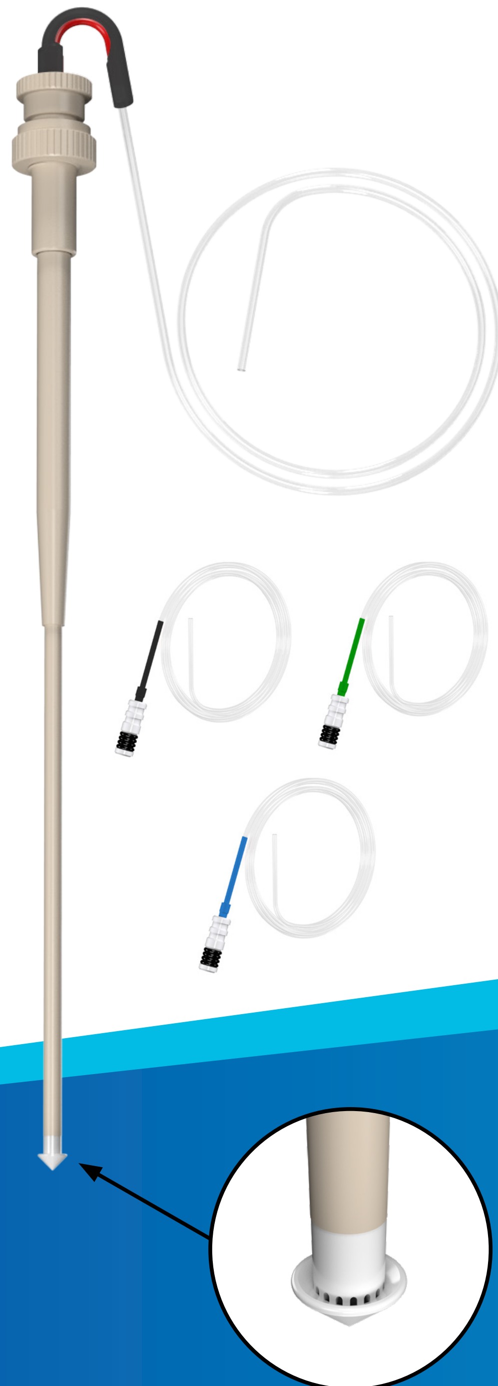
Michael Coppola serves as a product manager at Milestone Inc, directing product strategy, development, and market growth initiatives. He holds a bachelor's degree in chemistry and physics from Central Connecticut State University. Before joining Milestone, Coppola worked in the polymer chemistry sector as a product manager, where he led efforts to commercialize nanotechnology-based additives for the paint and plastics industries. He is a named inventor on more than 60 patents and publications related to materials science, chemical formulation, and applied analytical technologies.

Giulio Colnaghi has been working at Milestone since 2003. He is a chemist and started his career at Milestone as an application specialist, focusing on new method development and addressing specific needs in sample preparation. Later, he transitioned to Area Manager for most countries in Latin America and the eastern part of Europe, and then for the U.S. Since 2018, he has held the position of Marketing Manager at Milestone headquarters in Bergamo, Italy. During his career at Milestone, Giulio has worked with hundreds of laboratories and chemists to help them in overcoming daily challenges in elemental analysis by optimizing their workflow and methods. Today, Giulio aims to support worldwide laboratories to enhance their efficiency using state-of-the-art technologies.

GLASS EXPANSION
Quality By Design

Guardian Autosampler Probe

Glass Expansion is pleased to release the Guardian autosampler probe. The unique design of the robust tip—which combines drip-resistance and built-in particle filtering—helps to prevent cross-contamination during probe movement and blockages in your nebulizer and capillary tubing.



Benefits

- Robust tip design eliminates crushed and damaged tips due to misalignment.
- Drip-resistance prevents cross contamination of samples, especially with oils.
- Built-in particle filtering holds back particulates from blocking the line.
- Completely inert design, Ceramic, PEEK and PTFE construction.
- Interchangeable UniFit™ sample lines available in various IDs (e.g. 0.3, 0.50, 0.75 & 1.0mm)
- Designed to suit Teledyne Cetac®, Agilent®, PerkinElmer®, Shimadzu®, Aim Lab and Thermo Fisher Scientific™ Autosamplers.

Scan the QR Code to learn more about the Guardian Autosampler Probe:

The Role of the Nebulizer in ICP-MS: Design Considerations, Selection Criteria, and Optimization Guidelines

Justin Masone

Nebulizers play a pivotal role in the sample introduction systems of inductively coupled plasma mass spectrometry (ICP-MS), a technique that has revolutionized elemental analysis since its development in the early 1980s. Primarily designed for liquid samples, which constitute the majority of ICP-MS applications, nebulizers convert solutions into fine aerosols that can be transported to the plasma for desolvation, atomization, ionization and detection. However, the sample introduction system—encompassing the nebulizer and spray chamber—has long been regarded as the "Achilles' heel" of ICP-MS (1). This stems from its inherent inefficiency, where typically only 1-2% of the sample reaches the plasma, with the remainder lost to waste. Despite these limitations, advancements in nebulizer technology have significantly mitigated such issues, contributing to improved transport efficiencies and analytical outcomes.

The core function of a nebulizer is to deliver liquid samples into the plasma, where intense heat/energy—around 6000-10,000 K—atomizes and ionizes analytes for detection. Early designs focused on basic aerosol generation, but modern iterations prioritize precision, durability, and compatibility with challenging matrices. This evolution has been driven by the need to handle diverse samples, from trace-level environmental waters to high-matrix industrial effluents, while minimizing matrix interferences and maximizing signal stability. By understanding nebulizer mechanics, analysts can optimize parameters like gas flow rates and sample uptake, directly influencing aerosol quality and instrument performance.

The Importance of Nebulizer Droplet Size and Transport Efficiency

At the most fundamental level, effective nebulization is characterized by smaller droplet sizes and narrower distributions, as these factors determine how much of the sample reaches the plasma without overloading it—the plasma can only tolerate limited liquid loading before cooling or extinguishing it. Ideally, the sample introduction system should deliver 40 µL/min of water to the plasma or less, and exceeding 80 µL/min can extinguish the plasma (2). Additionally, if droplets are too large, they will not desolvate completely, significantly reducing plasma temperature and causing some elements to be released before others. This results in a higher signal for easily-ionizable elements (such as Na, K, and Al, for example) as compared to poorly-ionizable elements (such as Hg, As, and Se, for example). Thus, consistent droplet size is critical for accurate analysis. While all nebulizers generate a wide distribution of droplet sizes, nebulizers which generate a more consistent and narrower distribution will lead to better sensitivity and precision, as droplets over about 10 µm do not fully vaporize.

It is important to differentiate between what is called the primary aerosol and the tertiary aerosol. The primary aerosol is the aerosol that is produced from the nebulizer (as seen in Figure 1), whereas the tertiary aerosol is the aerosol that exits the spray chamber (and transfer tube, if used) and is injected into the plasma. As previously mentioned, all nebulizers produce a wide range of droplet sizes, and spray chambers are necessary to remove these larger droplets, with the smaller droplets going to the plasma and the larger droplets going to waste. This ratio of the amount of sample volume entering the plasma to the amount that is initially aspirated by the nebulizer is referred to as transport efficiency and is typically estimated by measuring the volume of waste draining from the spray chamber versus the total sample uptake volume.

Evolution of Nebulizer Designs

Nebulizers fall into two main categories: pneumatic and ultrasonic. Pneumatic nebulizers, the most common for ICP-MS, use the mechanical force of a gas flow to generate the sample aerosol, and will be the focus of this article. The early models of pneumatic nebulizers that were developed in the 1970s to support ICP-OES instrumentation achieved modest efficiencies but struggled with matrix effects and blockages. Over time, innovations in materials, fluid dynamics, and manufacturing have led to more robust and efficient designs.

FIGURE 1: Close-up of the primary aerosol produced at the tip of a concentric nebulizer (image provided by Glass Expansion).

Concentric Nebulizers

Concentric nebulizers represent a cornerstone of this evolution, featuring a central sample capillary encased in a glass sheath for coaxial flow

These nebulizers produce fine, uniform aerosols with small mean droplet diameters, ideal for high-sensitivity ICP-MS work. The earliest designs featured a heated and drawn inner glass capillary, which produced an excellent mist but resulted in a fragile, narrowing tip, prone to plugging by particles, as well as suboptimal relative standard deviations (RSDs) because of inconsistent concentricity and internal diameters (ID), increased glass porosity, and vibration issues caused by a high-velocity gas flow around the thin capillary. These issues are further exacerbated by similar inconsistencies in the forming of the nebulizer shell. Modern designs aim for a consistent ID along the capillary, as is seen with the VitriCone technology (Glass Expansion). This unique and revolutionary design involves machining precision-bore, thick-walled glass tubing, ensuring a uniform sample channel that resists clogging and provides high precision. Modern designs also use more advanced manufacturing methods to form the nebulizer jet nozzle, ensuring precise shape and structure.

Early designs of concentric nebulizers were prone to plugging and salting up with high-total dissolved solids (TDS) samples. This occurs as dissolved solids precipitate at the tip due to gas expansion and lower pressure. However, manufacturers have since developed improved designs for high-TDS samples, which feature recessed capillaries that move the salt precipitation zone away from the gas/liquid mixing zone, with some designs featuring a "self-washing nozzle," which uses the washing action of the dispersing droplets to further prevent crystal growth.

Benefits of Concentric Nebulizers

- Produce the finest aerosol at a variety of gas and sample flow rates, contributing to excellent reproducibility, transport efficiency, and stability.
- Can self-aspirate due to the natural Venturi effect, which can offset the pulsing effects of the peristaltic pump to maintain maximum precision.
- Are cost-effective, especially when made from glass.
- Easy to clean and maintain with convenient tools.

Potential Drawbacks of Concentric Nebulizers

- Can result in blockages from particulates or salts if the correct concentric nebulizer or accessories are not selected, as there are specific designs for different types of sample matrices.

Cross Flow Nebulizers

Cross Flow nebulizers, an early alternative to concentric nebulizers, intersect the sample and gas streams at right angles, offering robustness for high-salt samples. They can accommodate larger sample capillaries, but this results in a coarser aerosol with a wider droplet size distribution, leading to higher RSDs and limited suitability for ICP-MS (3).

Benefits of Cross Flow Nebulizers

- Offer larger liquid capillaries as compared to concentric designs, which allow larger particulates to pass through without plugging.

Potential Drawbacks of Cross Flow Nebulizers

- Provide a coarser mist than concentric nebulizers.
- Produce a wider range of droplet sizes and lower stability in the plasma
- Require higher liquid flow rates, typically limited to ICP-OES.

V-Groove Nebulizers

V-Groove nebulizers emerged to tackle high-particulate loads, as they have parallel gas and liquid paths, channeling liquid onto the gas orifice. They evolved based on non-plugging concepts and feature unrestricted sample paths that handle unfiltered slurries, with particulate tolerance exceeding 150 µm in some cases. While they can be effective for complex matrices, they produce larger droplet sizes with greater variability, negatively impacting precision. With the advancements in concentric nebulizers designed for particulates, slurries, and dissolved solids, in additional to accessories such as humidifiers, in-line sample filters, and autosampler probes with integrated filters, the V-groove is often not the optimal choice if precision and sensitivity are of utmost concern (4).

Benefits of V-Groove Nebulizers

- Particles cannot plug the liquid capillary, as there is no liquid capillary restriction, and it can have a large ID.
- Can handle unfiltered samples.

Potential Drawbacks of V-Groove Nebulizers

- Produces a mist with much larger droplets than other designs.
- Greater variation in droplet sizes as compared to concentric nebulizers.
- Cannot self-aspirate via the Venturi effect to offset the pulsing effects of the peristaltic pump.

Parallel Path Nebulizers

Parallel Path nebulizers were developed in the 1990s and deliver liquid adjacent to the gas orifice, with separate streams for the liquid and the gas. The sample is drawn into the gas stream by induction caused by the low pressure around the gas stream. Enhanced Parallel Path nebulizers were developed in the early 2000s, and, instead of simply placing the liquid near the gas stream and relying on induction, they feature a "spout," which protrudes into the gas stream (5).

Benefits of Parallel Path Nebulizers

- Separate gas and liquid channels with a large bore sample path that eliminates particulate blockages

Potential Drawbacks of Parallel Path Nebulizers

- Produces a wide range of droplet sizes.
- Cannot self-aspirate via the Venturi effect to offset the pulsing effects of the peristaltic pump.

Flow-Blurring Nebulizers

Flow-Blurring nebulizers are a recent development, similar in function to modern concentric nebulizer designs, operating with more turbulent gas-liquid interaction. The nebulizer has a central liquid capillary with surrounding gas orifices, resulting in highly turbulent mixing of the gas and liquid and creating an aerosol, in a process called "flow blurring" (6).

Benefits of Flow-Blurring Nebulizers

- High tolerance to particulates and dissolved solids while still maintaining sufficient sensitivity and precision

Potential Drawbacks of Flow-Blurring Nebulizers

- Increased optimization often required, as performance depends on precise gas and liquid flow ratios.
- Operate with very high backpressure—may not be recommended for high-throughput valve systems, and can accentuate noise from the peristaltic pump.
- While often touted as a "universal" nebulizer, they may not perform as well when compared to dedicated concentric nebulizers optimized for specific sample matrices.
- Cannot self-aspirate via the Venturi effect to offset the pulsing effects of the peristaltic pump.

Impact of Nebulizer Design on Aerosol Characteristics and Analytical Performance

Nebulizer design and operating conditions critically affect performance, specifically sensitivity, precision, and overall analytical accuracy. In general, a higher concentration of smaller droplets leads to improved sensitivity, and a more consistent droplet size is paramount for accurate and precise results.

While the most important factor in maximizing nebulizer performance is simply choosing the optimal nebulizer for your analysis, the operating parameters (for example, gas and liquid flow rates) still have a significant effect on performance. How these two parameters affect nebulization can be explained by understanding the Sauter mean diameter ($D_{3,2}$) – the average measure of particle size—and the Nukiyama and Tanasawa equation used to calculate it (7).

$$D_{3,2} = \frac{585}{V}\left[\frac{\sigma}{\rho}\right]^{0.5} + 597\left[\frac{\eta}{(\sigma\rho)^{0.5}}\right]^{0.45}\left[\frac{10^3 Q_l}{Q_g}\right]^{1.5}$$

in which:

- $D_{3,2}$ = Sauter mean diameter (µm)
- V = velocity difference between gas and liquid (m/s)
- σ = surface tension (dyn/cm)
- ρ = liquid density (g/cm^3)
- η = liquid velocity (Poise or dyn.s/cm^2)
- Q_l = volume flow rate, liquid (cm^3/s)
- Q_g = volume flow rate, gas (cm^3/s)

For water or aqueous matrices, surface tension (**σ**), liquid density (**ρ**), and viscosity remain constant. The droplet size, then, is inversely proportional to the velocity difference of the gas and liquid (**V**); that is, droplet size (**$D_{3,2}$**) decreases as the gas velocity increases and the liquid velocity (**η**) decreases. In practice, the smallest primary aerosol droplets are produced by using high argon gas flow and pressure, combined with low liquid flow and pressure.

The effect of lowering the sample flow rate can be seen in Figure 2 (it should be noted that while droplet size is an important factor for sensitivity, it is still dependent on the absolute amount of sample injected into the plasma). Three different nebulizers were compared using a constant gas flow rate and adjusting the sample uptake rate. Lowering the sample uptake rate for concentric designs increases the percent of droplet sizes less than 10 µm.

The same conditions were used to compare the effect that lowering the sample uptake rate has on the mean droplet size, which is shown in Figure 3:

Similar effects can be seen when holding the sample uptake rate constant and adjusting the gas flow rate. Increasing the nebulizer gas flow rate increases the percent of droplet sizes less than 10 µm. This can be seen in Figure 4.

And likewise with the mean/average droplet size, shown in Figure 5.

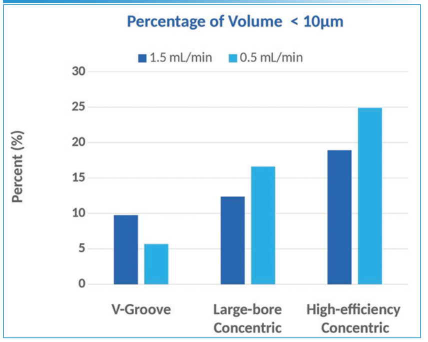


FIGURE 2: Effect of sample uptake rate on droplet size distribution.

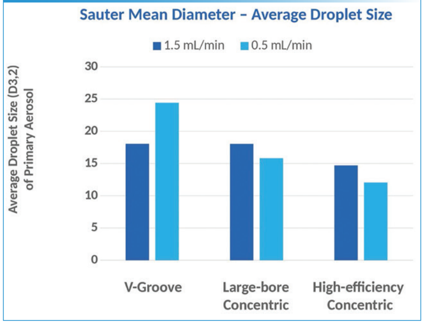


FIGURE 3: Effect of sample uptake rate on mean droplet size.

Aerosol Filtration

In addition to modern advancements in nebulizer designs, new techniques have been developed to improve aerosol quality post nebulization, which helps in maximizing instrument performance.

One such design is the Jet Vortex Interface (JVI Glass Expansion), which works in conjunction with the existing "Make-up" or "Dilution/Auxiliary" gas option on an ICP. Aerosol filtration accessories like the JVI provide precise control of the aerosol droplet size transported to the plasma, which, for high matrix samples, results in extended lifetimes of the torch, injector, and cones by a reduction in sample deposition, which also serves to prevent signal drift, in addition to reduced matrix effects in general. Aerosol filtration accessories enable the analyst to optimize the analysis for a variety of sample matrices (such as brines or volatile organics), as well as performance criteria (such as controlling for transport efficiency and signal intensity). The impact of JVI gas flow rate on droplet size is shown in Figure 6.

Aerosol filtration can also be utilized to "fine tune" the plasma conditions to create a more robust plasma for high matrix samples by reducing the oxide ratio to well below 1%. As a consequence, a more robust plasma results in higher sensitivity and, because of reduced matrix deposition, improved analytical stability, as shown in Figure 7.

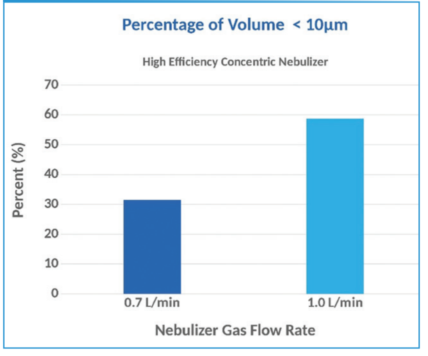


FIGURE 4: Effect of gas flow rate on droplet size distribution.

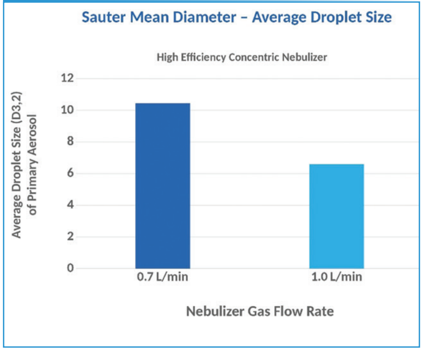


FIGURE 5: Effect of gas flow rate on mean/average droplet size.

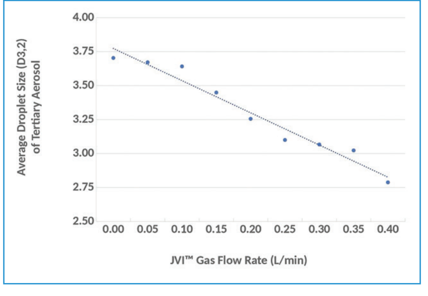


FIGURE 6: Effect of JVI gas flow rate on mean droplet size.

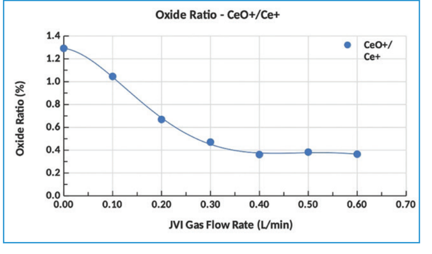


FIGURE 7: Effect of JVI gas flow rate on oxide ratio.

Nebulizer Selection Criteria

The choice of a nebulizer depends on several criteria, including:

- Reproducibility
- Sensitivity
- Precision
- Material (glass, PEEK (polymer), PFA (polymer), ceramic, etc.)
- TDS tolerance
- Particulates tolerance
- Hydrofluoric acid (HF) tolerance
- Purity
- Robustness
- Sample delivery method (self-aspirating vs. pumped)
- Cost

While the best advice is to consult with your instrument manufacturer or sample introduction supplier, some general guidance is offered in Table I

TABLE I: General performance guidance of nebulizers discussed in this article

Nebulizer Type	Material	Precision	TDS Tolerance	Particulates Tolerance	HF Tolerance	Purity	Self-Aspirating	Cost
Standard concentric	Glass	High	Good	Moderate	No	Good	Yes	Low
High-solids concentric	Glass	High	High	Moderate	No	Good	Yes	Moderate
High efficiency concentric	Glass	High	Moderate	Moderate	No	Good	Yes	Moderate
Large bore concentric	Glass	High	Moderate	High	No	Good	Yes	Moderate
PFA concentric	PFA	High	Moderate	Moderate	Yes	Excellent	Yes	High
PEEK concentric	PEEK	High	High	Moderate	Yes	Good	Yes	Moderate
V-Groove	Ceramic	Moderate	High	High	Yes	Good	No	High

(8). Similar charts, with more specific and detailed information, are often available from the manufacturer or supplier.

Nebulizer Maintenance

Regular cleaning is crucial for maintaining nebulizer performance and extending nebulizer lifespan. A good daily protocol is to always start and finish the use of a nebulizer by nebulizing a mildly acidic blank solution followed by deionized water for a couple of minutes. This ensures that sample deposits or crystals do not form inside a nebulizer when the solvent inside the nebulizer dries out.

If the sample capillary becomes blocked, tools like the Eluo (Glass Expansion) can be used for safe backflushing. For stubborn salt deposits, soaking the nebulizer in a 25% Fluka RBS-25 (Sigma Aldrich) solution for 24 h, and then flushing with warm water, followed by ethanol, is effective (9).

Note: *Never attempt to eliminate a blockage by inserting a wire or probe into the capillary—this may damage the nebulizer.*

Always handle the nebulizer tip with gloves to prevent detrimental effects from body oils. Be sure to store nebulizers in their specially-designed containers to prevent physical damage or contamination. Lastly, never put a nebulizer in an ultrasonic bath, as this can dislodge the sample capillary and damage the nebulizer.

Conclusion

Nebulizers remain a fundamental sample introduction component for ICP-MS. The diverse range of available nebulizer designs, coupled with an understanding of each design's impact on aerosol characteristics and analytical performance, enables analysts to select an optimal nebulizer for the wide variety of sample types and analytical challenges often encountered in today's laboratories. Ongoing advancements in nebulizer design and performance, and the integration of supplementary techniques such as aerosol filtration, continue to enhance the sensitivity, precision, accuracy, and robustness of ICP-MS. Lastly, proper nebulizer selection and dedicated maintenance are the key to maximizing instrument performance and minimizing instrument downtime.

References

(1) Browner, R. F.; Boorn, A. W., Sample Introduction: the Achilles' Heel of Atomic Spectroscopy? *Anal. Chem.* **1984,** *56* (7), 786A-798A. DOI: 10.1021/ac00271a718

(2) Beauchemin, D. *Sample Introduction Systems in ICP-MS and ICP-OES*; Elsevier, 2020.

(3) PerkinElmer product description. https://shop.perkinelmer.com/en-CA/product/gemtip-crossflow-ii-nebulizer/01tPs00000AcugnIAB

(4) Meinhard nebulizers product description: https://www.meinhard.com/Shop-by-Product/Nebulizers/High-Particulate-Nebulizers/MEINHARD-VGroove-One-Touch-Nebulizer-Agilent-ICP/

(5) Burgener nebulizers product description: https://www.burgener.com/products-nebulizers/p/peek-mira-mist-ip

(6) Agilent product description: https://www.agilent.com/store/productDetail.jsp?catalogId=G8010-60293

(7) Nukiyama, S.; Tanasawa, Y. An Experiment on the Atomization of Liquid by Means of an Air Stream. *J. Jpn. Soc. Mech. Eng.* **1937,** *40*, 128-135. DOI: 10.1299/jsmemagazine.40.243_453

(8) ICP Nebulizer Selection and Maintenance Application Note from Glass Expansion Website https://www.geicp.com/site/images/application_notes/AppSpotlight100608.pdf

(9) ICP Nebulizer Maintenance Application Note from Glass Expansion Website https://www.geicp.com/site/images/application_notes/ICPNebulizer_Maintenance_Prolong_Performance_and_Lifetime.pdf

Justin Masone is the Sales Manager for Glass Expansion in the Americas. Prior to joining Glass Expansion, Justin worked as a Sales Representative and Product Specialist for instrumentation manufacturers, as well as an Analyst in an environmental lab. Justin received his degree in Chemistry from Johns Hopkins University in 2010 and has spent his entire career in the field of elemental spectroscopy.

Rapid Washout of Boron Using Glass Expansion's Peltier Cooled Cyclonic (PCC) Spray Chamber on Agilent® 7900 ICP-MS

Author: Randy Mercurio

Abstract

This application note explores the efficient and rapid washout of boron (B) from the sample introduction system of Agilent's 7900 Inductively Coupled Plasma Mass Spectrometer (ICP-MS) instrument using Glass Expansion's Peltier Cooled Cyclonic (PCC) spray chamber. The combination of these technologies enhances analytical performance by minimizing memory effects and improving signal stability, particularly in the analysis of trace levels of boron in complex, real-world sample matrices.

Introduction

Boron analysis poses challenges using ICP-MS due to its tendency to form memory effects and interfere with subsequent analyses. This occurs when residual boron from previous samples adheres to the spray chamber walls, leading to carryover and inaccurate measurements in following samples. Along with a few other elements, such as Hg, Pb and Sb, B is notorious for causing memory effects. Sometimes, even relatively advanced ICP-MS instrumentation can struggle to meet basic QC requirements when it comes to analyzing challenging elements like these in real-world samples. For example, when analyzing for boron using the standard Scott style spray chamber, washout times can be extensive. This is primarily attributed to the large surface area of the Scott-style spray chamber, where the time needed to sufficiently get boron levels to an acceptable level can take valuable analysis time.

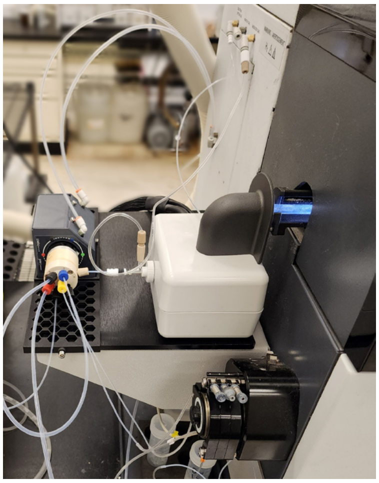
Whether analyzing difficult real-world samples with high boron content, or if low detection limits are hindering efficient rinse out times requiring repeated rinse blank analyses, Glass Expansion's Peltier Cooled Cyclonic Spray Chamber (PCC) Kit outfitted with a quartz spray chamber can quickly get boron down to acceptable levels in rinse blanks.

Glass Expansion's Tracey™ cyclonic spray chamber design provides the best sensitivity, reproducibility and lowest memory effects. Additionally, the Helix Constant Torque (CT) locking screw and seal creates an optimal nebulizer interface that significantly reduces dead volume around the nebulizer, assisting the rapid washout.

PCC Spray Chamber Design

The PCC spray chamber by Glass Expansion is a high-performance accessory designed to replace standard Scott style spray chambers on ICP-MS instruments. It features a unique cyclonic design that creates a centrifugal force within the chamber. This force helps minimize the residence time of sample aerosols, reducing their interaction with the chamber walls and consequently, lowering memory effects. Furthermore, the PCC Kit incorporates Peltier cooling technology, which actively cools the spray chamber, further reducing aerosol dwell time and enhancing washout efficiency. It offers several advantages over the standard Scott-style spray chamber, particularly when analyzing troublesome elements like B.

Figure 1. PCC Kit (P/N KT-1212Q) Installed on Agilent® 7900 ICP-MS.



Key Advantages of the PCC Spray Chamber

1. Improved Washout Efficiency:

- The distinctive design of the cyclonic spray chamber minimizes washout time, especially with highly concentrated samples containing challenging elements such as boron.
- Compared to the standard Scott-style spray chamber, the PCC significantly reduces the time required to reach baseline levels after analyzing boron-rich samples.

2. Helix CT Interface:

- All Glass Expansion cyclonic spray chambers feature the Helix CT locking screw and seal.
- This interface ensures a consistent inert PTFE seal against the nebulizer, preventing overtightening and ensuring a gas-tight seal during nebulizer installation.
- The Helix CT design provides unparalleled, reproducible day-to-day analytical performance, minimizing memory effects and improving sensitivity.

3. Interchangeable Materials:

The PCC Kit allows easy interchangeability between different spray chamber materials:

- **Borosilicate Glass:** Cost-effective option for routine analyses not requiring low-level boron measurements.
- **Quartz:** Required for precise low-level boron detection.
- **PFA:** Ideal for applications with a hydrofluoric acid matrix and ultra-trace ICP-MS analyses (paired optimally with Glass Expansion's DuraMist DC Nebulizer).
- The interior of the Tracey PFA spray chamber features the proprietary Stediflow surface treatment, improving wettability and efficient drainage.

4. Fast and Simple Installation/Operation:

- The PCC Kit connects directly to the existing electronics and water-cooling system.
- Use existing software and methods to operate.
- A convenient mounting bracket ensures swift installation on the Agilent® 7900 and 8900 ICP-MS systems.

5. Jet Vortex Interface (JVI™) – Aerosol Filtration:

- The JVI is illustrated in Figure 2, and is included in the PCC Kit to easily utilize your existing method settings for Make-Up or Dilution/Option Gas.

Experimental Setup

A high-throughput environmental laboratory analyzing particularly difficult samples with high boron concentrations was looking for a solution to their excessive rinse out times to ensure quality data and improve their sample throughput. The lab was using an Agilent 7900 ICP-MS with a standard Scott-style spray chamber, ISIS-3 switching valve, and SPS 4 Autosampler on two different instruments. Glass Expansion equipped both of the lab's 7900 ICP-MS instruments with the PCC Kit (KT-1212Q) to demonstrate the advantages (see Figure 1).

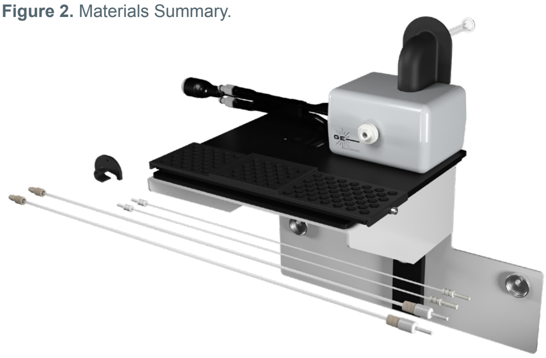
The PCC Kit operates using the instrument's existing software, communication port and chiller cooling ports, so installation and operation were a breeze.

A challenging, real-world, aqueous sample from a remediation site, containing 4.3 mg/L of boron was used to collect washout analysis data, comparing the Scott and PCC Kit, by running 10 consecutive rinse blanks after the sample was analyzed. The rinse blanks and the autosampler rinse station were composed of 2% nitric acid and 1% hydrochloric acid. The sample and 10 consecutive blanks were first analyzed using their standard Scott-style setup. This same analysis was then repeated using the PCC Kit. The collected rinse blank data was compared using isotope 11 of B in H_2 mode. The details of the equipment and material used are summarized in Table 1 and Figure 2.

Table 1. Materials Summary

Materials Summary	
ICP-MS Instruments	Agilent 7900 with ISIS 3 Switching Valve & SPS 4 Autosampler
Spray Chamber	Glass Expansion Quartz PCC Kit (KT-1212Q)
Nebulizer	Glass Expansion MicroMist DC Nebulizer (A13-1-UM04)
Sampler Cone	Glass Expansion Ni Plated (AT7701-Ni/Ni)
Skimmer Cone	Glass Expansion Ni Skimmer Cone (AT7902X-Ni)

Figure 2. Materials Summary.



P/N KT-1212Q
PCC Quartz Spray Chamber

P/N A13-1-UM04
MicroMist DC Nebulizer 0.4mL/min

JVI™

P/N AT7701-Ni/Ni
Nickel Plated Sampler Cone

P/N AT7902X-Ni
Nickel Skimmer Cone

Standard Agilent Performance Reports were analyzed after instruments were warmed up for thermal stability of the interface. A total of 15 performance reports from two separate instruments operating the same PCC kit configuration were used to create averages of the key data summarized in Table 2. The performance reports span a time period of more than 3 months and include data immediately after changing cones.

Table 2. Performance Report Averages [n=15]

Instrument Performance Report		
Average Nebulizer Gas Flow (L/min) = 1.038		
JVI Dilution Gas Flow (L/min) = 0.00		
H_2 & He Gas Flows (mL/min) = 0.00		
Mass	**Sensitivity (cps)**	**RSD%**
^{7}Li	6385	2.481
^{89}Y	30735	2.223
^{205}Tl	21436	2.447
Oxide 156CeO/Ce140 (<1.8%)	1.301	
Doubly Charged (<2.5%)	1.297	

This lab was operating under EPA Methods 200.8 and 6020, so each day after the instrument performance reports were analyzed they also analyzed an EPA Tune Check Report for H_2 and He modes to ensure the operating conditions of the nebulizer and JVI gas flows met these method criteria, which are different from the instrument performance reports in Table 2. Tables 3 and 4 illustrate the average of 16 EPA Tune Check Reports from 2 different instruments for the H_2 and He modes, respectively.

Table 3. H_2 Mode US EPA 200.8/6020 Tune Check Report Average [n-16]

US EPA 200.8/6020 Tune Check Report [H_2]		
Nebulizer Gas Flow (L/min) = 0.85		
JVI Dilution Gas Flow (L/min) = 0.15		
H_2 Gas Flow (mL/min) = 3.5		
Mass	**Sensitivity (cps)**	**RSD%**
^{9}Be	972	1.497
^{24}Mg	11245	2.083
^{25}Mg	1609	2.207
^{26}Mg	1982	1.974
^{59}Co	25236	0.899
^{115}In	84931	0.735
^{206}Pb	17030	0.871
^{207}Pb	14342	0.898
^{208}Pb	35324	0.868

Table 4. He Mode US EPA 200.8/6020 Tune Check Report Average [n-16]

US EPA 200.8/6020 Tune Check Report [He]		
Nebulizer Gas Flow (L/min) = 0.85		
JVI Dilution Gas Flow (L/min) = 0.15		
He Gas Flow (mL/min) = 4.5		
Mass	**Sensitivity (cps)**	**RSD%**
^{9}Be	87	2.615
^{24}Mg	1266	1.486
^{25}Mg	184	2.021
^{26}Mg	232	1.962
^{59}Co	20333	1.333
^{115}In	24991	1.464
^{206}Pb	11164	1.370
^{207}Pb	9399	1.357
^{208}Pb	23286	1.361

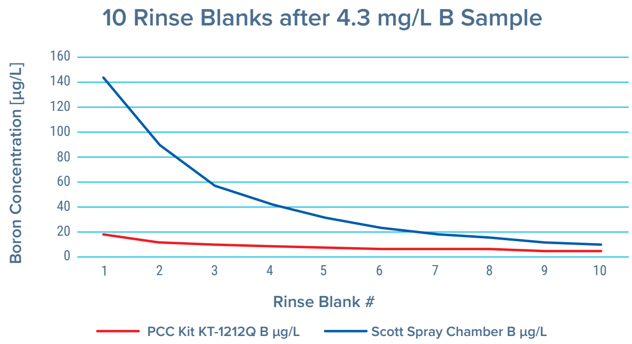
Results and Discussion

This lab's reporting limit for B was 10 µg/L, and it frequently encountered samples with boron levels on the order of 4.3 mg/L, making it difficult to rapidly rinse out these relatively high concentration samples. After analyzing a 4 mg/L B sample, it would normally take the Scott-Style spray chamber 10 rinse blanks, taking a total of 31 minutes to analyze the sample and then washout out the sample introduction system to get the background level below the reporting limit. In comparison, the PCC Kit was able to analyze the same sample and washout to <10 µg/L in 12 minutes. Table 5 contains this comparative data set to illustrate the washout efficiency of the PCC Kit. And Figure 3 graphs this data showing the <10 µg/L reporting limit was achieved on the 4th rinse blank using the PCC Kit, as opposed to the 10th rinse blank after the Scott-style spray chamber. This decreased the number of necessary rinse blanks from 10 to 4, saving 6 rinse blanks. In this example, this is a 61% improvement in B washout time, resulting in a much higher throughput and much lower potential for any carryover.

Table 5. Boron washout data for Scott-Style Spray Chamber vs PCC Kit

	PCC Kit (KT-1212Q)		**Scott Style Spray Chamber**	
	11 B [H2]		**11 B [H2]**	
Time Stamp	**Conc. [ppb]**	Type	**Conc. [ppb]**	Time Stamp
7:35 PM	4294.897	Sample	4376.533	2:49 PM
7:38 PM	18.655	Blank 1	141.146	2:52 PM
7:41 PM	12.491	Blank 2	89.449	2:55 PM
7:44 PM	10.483	Blank 3	57.566	2:58 PM
7:47 PM	**9.49**	Blank 4	42.028	3:01 PM
7:50 PM	8.183	Blank 5	31.679	3:04 PM
7:53 PM	7.505	Blank 6	24.263	3:08 PM
7:57 PM	7.083	Blank 7	19.126	3:11 PM
8:00 PM	6.706	Blank 8	15.771	3:14 PM
8:03 PM	5.366	Blank 9	11.902	3:17 PM
8:06 PM	5.026	Blank 10	**9.851**	3:20 PM

Figure 3. Comparison of B washout after 4.3 mg/L sample using 10 consecutive rinse blanks.



When only considering the combined sample uptake and washout times for routine sample analysis, this particular lab noted that it was able to save about 40 to 60 seconds per sample, depending on the analysis type. Saving these 40 to 60 seconds was accomplished by reducing unnecessary capillary length from the autosampler probe capillary to the nebulizer and utilizing the faster washout times of the PCC kit. Additionally, by reducing the number of rinse blanks from 10 to only 4 following high concentration B samples, the lab was able to regain up to 24 minutes of billable analysis time for each highly concentrated B sample. With the faster washout of the PCC Kit and shortened sample path, the sample analysis time range was reduced from 3.66—4.0 minutes down to a range of 3.0—3.25 minutes. Gaining these 18 to 24 minutes from one highly concentrated boron sample enables the analysis of about 6 to 8 more samples just from eliminating excessive rinses.

Summary of Key Benefits of Using a PCC Kit for Boron Analysis

1. Faster Washout Times:

- Compared to traditional Scott-style spray chambers, the PCC significantly reduces boron washout times, and in this case nearly 10x faster.

2. More Efficient Workflow:

- Less reruns from unanticipated carryover or poor RSDs.

3. Improved Signal Stability:

- Reduced memory effects lead to more consistent and reliable boron measurements, and longer analysis times with less analytical drift.

4. Higher Quality Data:

- Less data flags on instrument blanks, method blanks, duplicates, dilutions, and other QA/QC samples.

5. Increased Sample Throughput:

- Faster uptake and washout times enable shorter total sample acquisition times, thereby increasing sample throughput.

Conclusion

The Glass Expansion PCC Spray Chamber provides a significant advantage for ICP-MS users performing boron analysis. This application note demonstrated Glass Expansion's Quartz PCC Kit's effectiveness in reducing boron washout times when using a standard Scott style spray chamber on Agilent's 7900 ICP-MS. This environmental laboratory saw immediate results and was able to recapture profitable analysis time by eliminating unnecessary consecutive rinse blanks to more rapidly bring boron to baseline levels after highly concentrated samples. Moreover, sample uptake and rinse out times were reduced by shortening capillary lengths and taking advantage of the PCC Kit's fast stabilization times. Compared to the standard Scott-style spray chamber, Glass Expansion's uniquely designed cyclonic spray chamber minimizes washout time with elements known to cause memory effects, such as B, Hg, Pb, and Sb. The benefits of investing in the PCC Kit are many, making it a wise choice for labs analyzing elements that cause memory effects. For more information on Glass Expansion's PCC Kit, please visit our webpage: *Glass Expansion's Peltier Cooled Cyclonic Spray Chamber for Agilent® ICP-MS.*

Customer Feedback

"For our particular application, this PCC kit was a great improvement. On specific cases that required four or more blank runs to bring the boron level to baseline now we can do it with one or two blank runs. This allowed us to increase sample throughput by about 50%." — Specialty Chemicals Manufacturer – USA

FEATURED ARTICLE

Enhancing ICP-MS Efficiency with an Innovative, Low-Maintenance Nebulizer

Sergei Leikin and Mary Kay Amistadi

Robust ICP-MS analysis of complex matrices—especially those containing even small particulates and high salt levels — remains a significant challenge due to frequent clogging of conventional concentric nebulizers. Commonly recommended filtration or centrifugation steps are labor-intensive, increase costs and extend turnaround time. To overcome these limitations, an innovative nebulizer featuring a robust non-concentric design with a relatively large sample channel internal diameter was employed. This design provides good resistance to clogging and improved tolerance to tough matrices.

Results from over two years of continuous operation across a wide range of challenging sample types showed a substantial increase in ICP-MS throughput.

Overall analytical efficiency was significantly enhanced through a combination of factors: elimination of nebulizer blockages, removal of time-consuming filtration or centrifugation steps, and a substantial reduction in nebulizer maintenance.

What is an inductively coupled plasma–mass spectrometry (ICP-MS) analyst's worst nightmare? Spending hours preparing a large batch of samples and setting up an overnight run - only to return in the morning and find the sequence stopped after just a few samples, an empty progress bar and a familiar diagnosis: a blocked nebulizer.

Although ICP-MS is a well-established analytical technique, maintaining high efficiency and reliability in routine operation remains challenging. In many research and commercial laboratories, sample nature and complexity can vary daily, while expectations for high productivity and rapid turnaround are constant. When samples contain complex matrices or even small amounts of particulates, clogging of conventional concentric nebulizers occurs frequently (1), disrupting analyses and compromising data quality. A common recommendation: prevention is preferable to corrective action. That is why routine standard operating procedures often include filtration and/or centrifugation steps - measures that, while effective, are time-consuming and add significant costs to the analytical process.

To address these limitations, a nebulizer with an innovative design (2) has been employed and its potential to streamline high-throughput ICP-MS workflows without compromising analytical performance was assessed.

Principle of Operation and Experimental Setup

Compared to inductively coupled plasma–optical emission spectroscopy (ICP-OES)—which commonly operates at sample uptake rates of 1.0 to 2.0 mL/min except for some cases like high volatile organics—modern ICP-MS instruments work at much lower rates, typically between 0.100 and 0.400 mL/min. To achieve efficient performance at these low flow uptake rates, nebulizers for ICP-MS are designed with reduced internal diameters of the sample channel, typically ranging from 100 to 270 µm (3) (Figure 1 indicated by red area), which increases their susceptibility to clogging.

For challenging samples, the general recommendation is to use robust, blockage-resistant, non-concentric nebulizers with a large sample channel ID, accepting some trade-off in sensitivity and precision. A larger capillary ID can cause inconsistent sample delivery at low uptake rates. While such compromised performance may be acceptable for certain applications, it is often unsuitable for the majority, as ICP-MS is specifically designed to achieve the lowest possible detection limits.

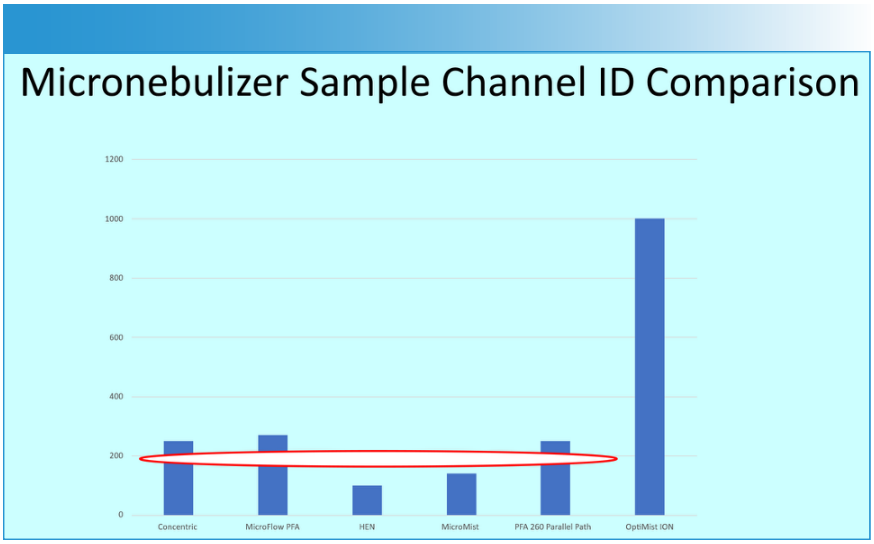


FIGURE 1: Sample channel ID comparison: nebulizers designed for low sample flow rates in ICP-MS.

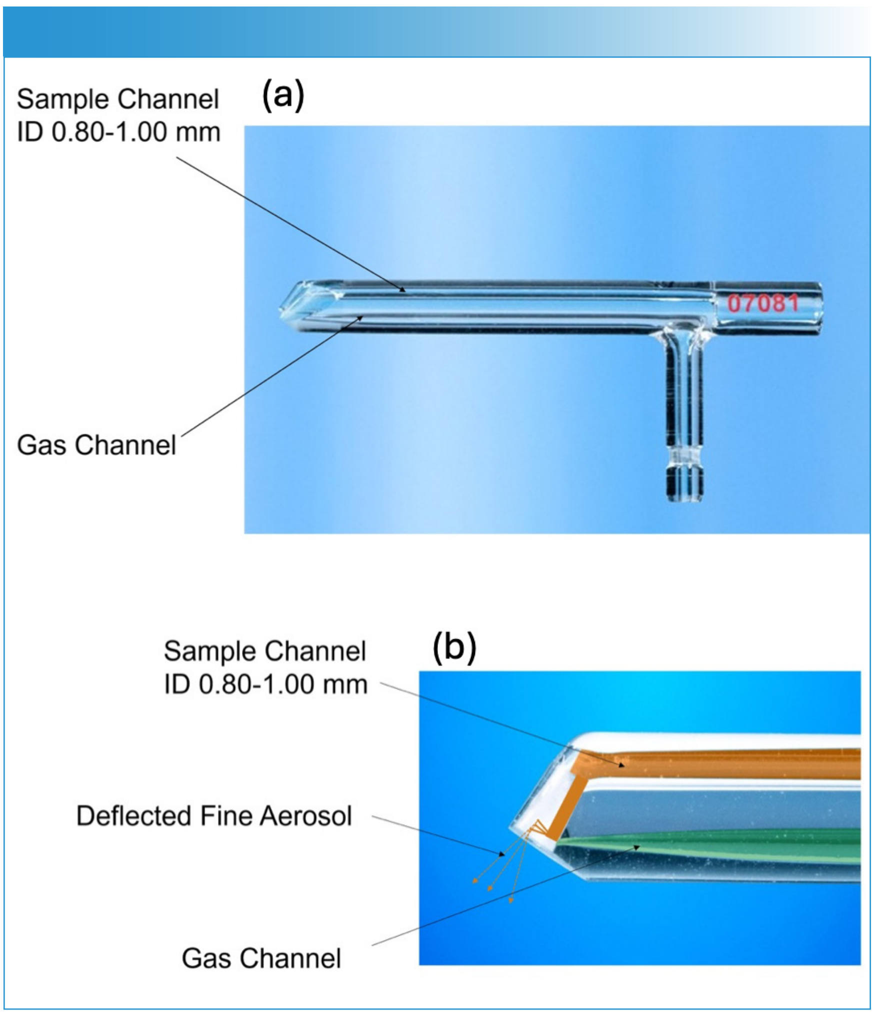


FIGURE 2: (a) OptiMist ION, and (b) aerosol creation diagram at the tip of the nebulizer.

The nebulizer (OptiMist ION, Texas Scientific Products, patent pending) employs a rugged non-concentric design, with gas and sample channels kept completely separate throughout its body. Its relatively large sample channel - approximately 0.75 mm in internal diameter (ID) - offers good clogging resistance and enables reliable performance with demanding sample matrices. Efficient aerosol generation is accomplished by positioning an impact surface near the gas orifice, integrated into a cover plate that forms a small chamber (Figure 2). The gas jet, directed at this surface under an optimized angle, produces a pressure drop that ensures stable sample delivery, even at low uptake rates.

Since fine aerosol is deflected by the impact surface, the nebulizer should be oriented at an angle to the spray chamber. An interface with Scott Double Pass spray chamber was accomplished by the specially designed End Cap supplied with a nebulizer. Figure 3 shows the nebulizer set up in two ICP-MS systems (a) Scott-double pass spray chamber of an Agilent 8900 and (b) cyclonic spray chamber of an Thermo ICAP RQ.

Applications and Results

The analytical work was carried out at the Arizona Laboratory for Emerging Contaminants (ALEC), which provides analytical support to researchers from five colleges at the University of Arizona.

The samples submitted to ALEC range from environmental - drinking and wastewaters and soil acid extracts to agricultural (plant tissues), biological (wood) and clinical - including undigested whole blood, serum, urine, and digested animal tissues. A few representative examples are shown on Figure 4

Wide sample varieties handled by ALEC that often may change daily are summarized in Table I.

Whole Blood and Serum

The nebulizer was first evaluated for direct analysis of whole blood and serum—two of the most challenging sample types for ICP-MS. Blood is a complex matrix containing inorganic salts, proteins, erythrocytes (red blood cells), and leukocytes (white blood cells). During sample dilution, protein coagulation and cell precipitation can occur, potentially impacting analysis (Figure 4). Although alkaline dilution is often preferred over acid dilution to reduce precipitation, we intentionally selected an acid-based dilution to test the nebulizer under the most demanding conditions, as this approach produces a higher load of particulate matter. The dilution factor was also kept to a minimum. Analyses were performed using an Agilent 7700 ICP-MS equipped with a collision/reaction cell.

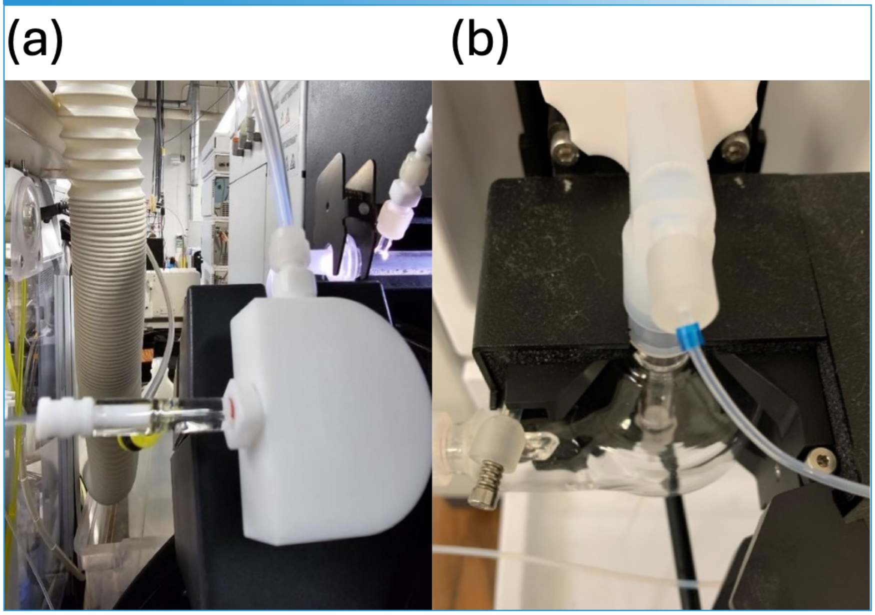


FIGURE 3: Nebulizer setup in two different ICP-MS instruments (a and b - refer to text for details).

Accuracy

For serum samples, a 2% v/v nitric acid solution containing ethanol and Triton X was used for dilution. Despite visible precipitation, no nebulizer blockages occurred. Whole blood samples were prepared by 1:20 dilution with a solution containing 0.5% v/v nitric acid, 0.05% Triton X, and 1.5% butanol, which produced only minor precipitation. In both cases, 25 elements relevant to biomonitoring were analyzed. Due to the complexity of the blood matrix, complete matrix-matching calibration is often used to maximize accuracy, typically by adding a "base" blood to all calibration standards (4). However, this approach is limited by the availability of sufficiently pure blood and requires time-consuming prescreening or quantification of contaminants via the method of standard additions.

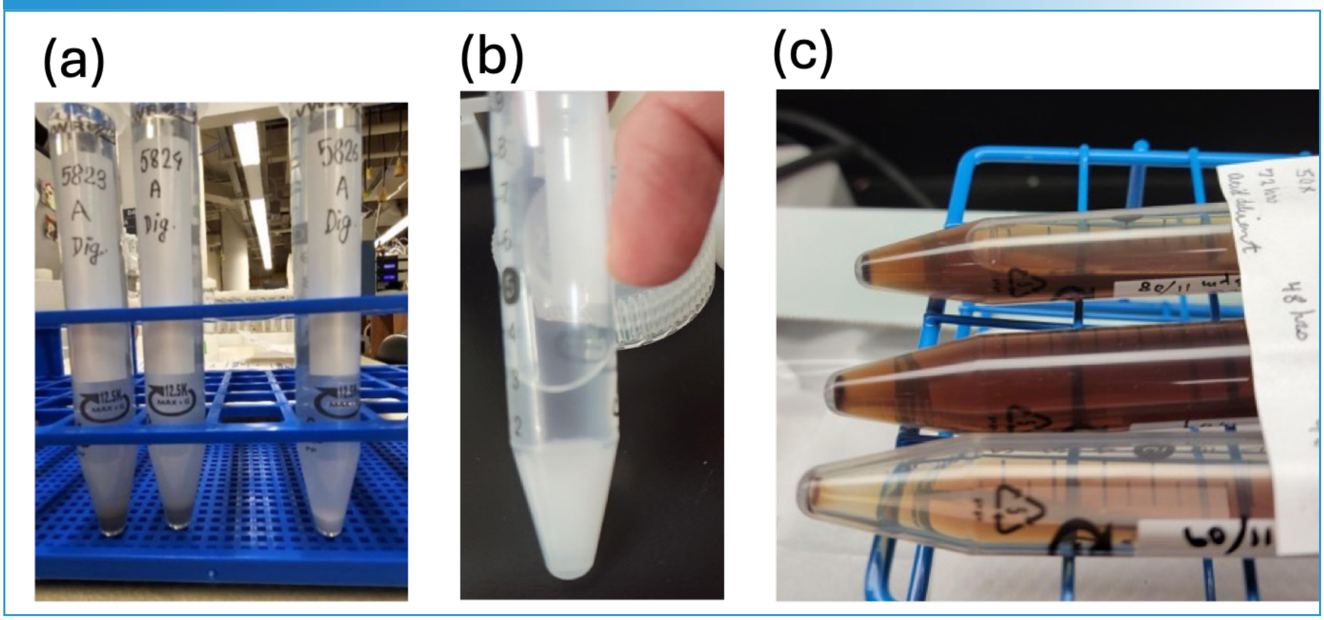


FIGURE 4: Examples of the samples handled by ALEC Lab: (a) soil acid extracts; (b) wood core digests; (c) whole blood at 1:20 Dilution in 0.5% HNO_3, 0.05% Triton X, 1.5 % butanol.

To address these limitations, we evaluated published whole blood composition data (5) and developed a partial matrix-matching approach. Synthetic calibration standards were prepared by adding key matrix elements—particularly those with significant effects on analytical signals, such as Ca, Na, and K—at concentrations representative of typical whole blood. For a 1:10 dilution, the synthetic calibration composition was (ppm): Na–90, K–85, S–70, Fe–25, Ca–3.0. This calibration strategy was tested using certified reference blood samples from the Institut National de Santé Publique du Québec (INSPQ), Canada. Results are summarized in Fig. 5.

TABLE I: Snapshot of ALEC sample logbook

Sample Type	Sample Type	Sample Type
Dairy sludge dry and cake	Dry biosolids	Insects
Resin	NaOH cell digests	Scorpions in yellow bag
Acid extracts	Breast milk 2022	Mine tailing extracts
Sunscreen	Fish feed	Ghost wipes
Wood digests	Sugar/water	Urines
Basalt leachate	Cell pellets	Furniture finish
Serum/CSF/ blood digests	Brines and waters	Rhamnolipid solutions
Plants	Tissue/serum/cells	Mollusks, feathers, fur

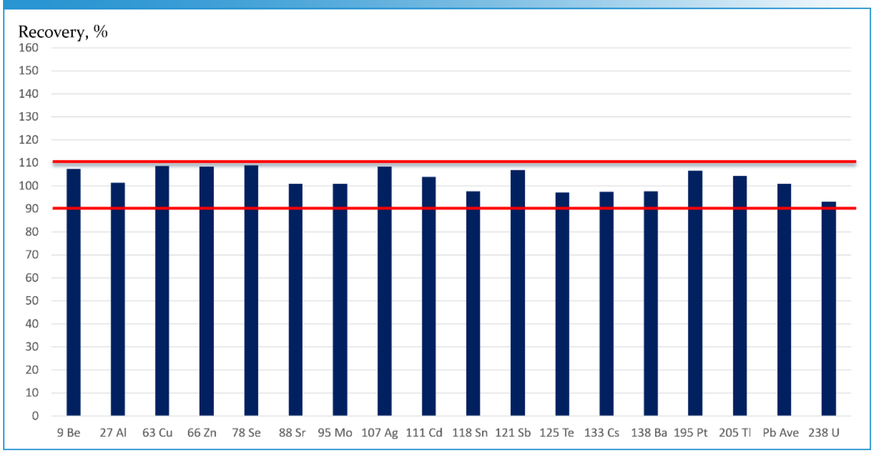


FIGURE 5: Accuracy data for whole blood CRM QM-B-Q1313 without complete matrix matching.

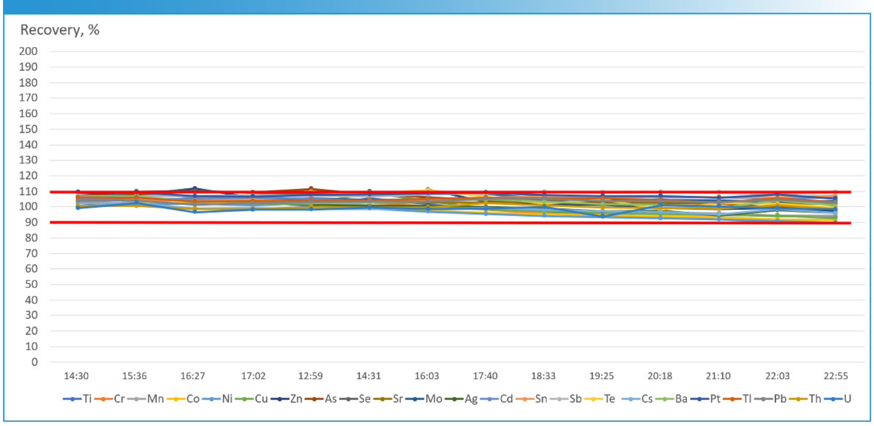


FIGURE 6. Calibration Stability over 12.5 Hours running whole blood samples.

Long-Term Stability

A long-term stability test was performed over two days by analyzing whole blood samples every 4.5 minutes (Fig. 6). After the study, the condition of the sample introduction components (a), (b), (c) and the ICP-MS interface (c), (d) was examined. As expected, the sample introduction parts, and interface cones were significantly affected by this challenging matrix. In contrast, the nebulizer remained in good operating condition, with no visible sample deposits (Fig. 7).

Routine Maintenance

To evaluate routine maintenance requirements, the nebulizer was operated continuously—purposely without any cleaning—while monitoring analytical performance. It is worth noting that trouble-free operation lasted almost three years (34 months). During this time, 31,500 challenging samples, many with high particulate content, were analyzed successfully without pre-filtration or centrifugation.

The first sign of reduced performance appeared after 34 months, when a slight loss of efficiency and increased backpressure were observed. The nebulizer's performance was fully restored by soaking in gently pre-heated laboratory detergent. It should be noted that the nebulizer was operated without an argon humidifier, and the large sample load eventually contributed to minor salt buildup around the gas orifice, as indicated by the elevated backpressure.

Initial evaluations of nebulizer performance (2) demonstrated efficiency and precision comparable to concentric nebulizer supplied with commercially available ICP-MS instruments. Relative sensitivity and method detection limits (MDLs) for both nebulizers are compared in two different ICP-MS system (Figures 8a [Agilent 7500] and 8b [Thermo ICAP

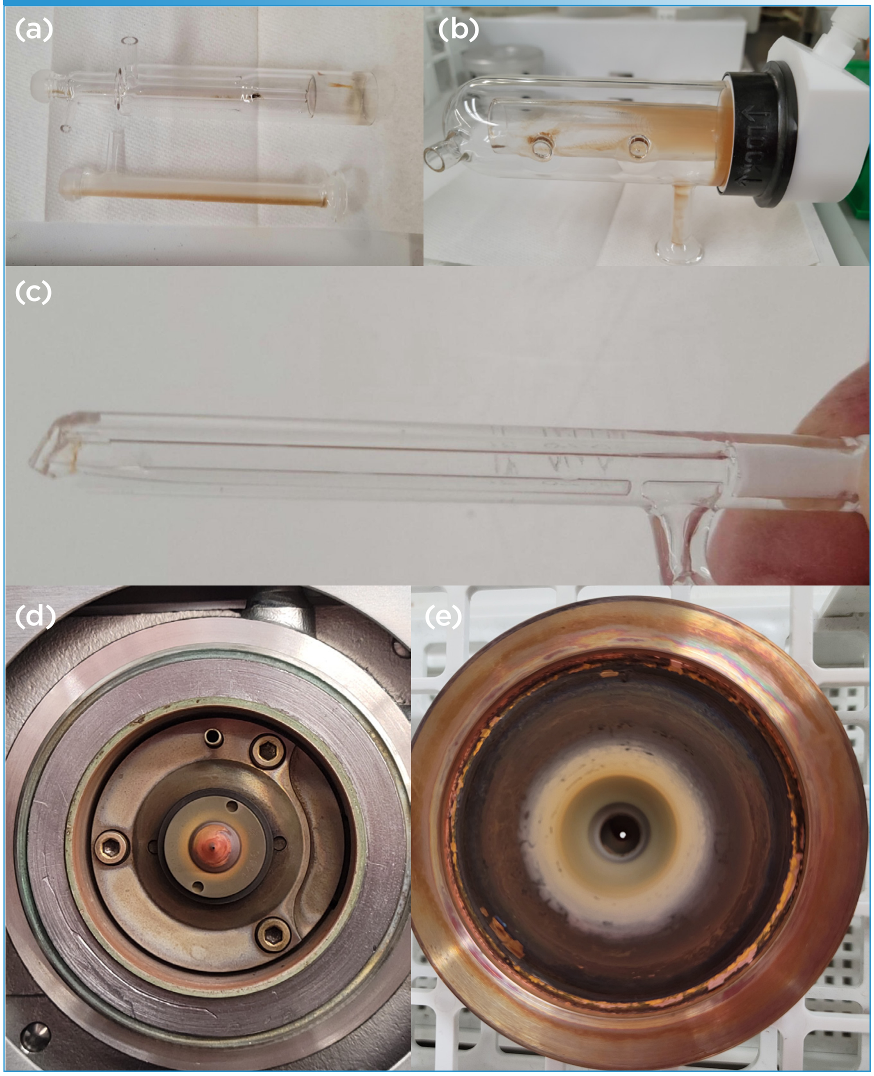


FIGURE 7: Sample Introduction Conditions after 2 days exposure to whole blood samples showing sample deposit on the (a) torch, adapter, (b) spray chamber, (c) clean nebulizer, and (d and e) interface cones.

RQ]). MDLs were determined according to the United States Environmental Protection Agency–Contract Laboratory Program. (USEPA CLP) protocol by analyzing a solution containing 1 ppb of target analytes and several ppm of Na, K, Mg, Ca, and Fe. The standard deviation from seven replicate measurements was multiplied by 3.14 to estimate the MDLs.

Further evaluation was carried out using arsenic speciation—an application where sensitivity is critical, as some species, particularly monomethylarsonic acid (MMA), can occur near the detection limit. That is why efficiency of the nebulizer is an important contributing factor. These experiments were performed on an Agilent 8900 triple-quadrupole ICP-MS in mass shift mode, using O_2 (75 As to 91 AsO) on the PRP-X100 anion exchange column with 12 min acquisition time and 50 µL injection volume. The samples were prepared using the CDC Method DLS-3000.15-03 for Urine Arsenic Speciation with eluents containing 10 mM NH_4 carbonate and TRIS buffer plus 4% MeOH at pH 8.6 and 10 mM NH_4 carbonate and TRIS buffer plus 4% MeOH and 15 mM NH_4SO_4 at pH 8.0. An example of chromatogram in urine sample where MMA content is less than 2% of total arsenic and close to the minimum detectable level is shown on Figure 9a. A calibration curve focused near the detection limit, along with a sample containing 90 ppt MMA (just above the 80 ppt standard), is shown in Figure 9b.The MDL for MMA under these conditions was estimated at 30 ppt in solution, based on 3σ from six replicate measurements of the lowest calibration standard (50 ppt).

Conclusions

Implementing an innovative nebulizer design in routine laboratory ICP-MS operations resulted in a substantial increase in analytical efficiency. Over nearly three years of continuous use across a wide range of challenging sample types, the system processed 31,500 samples without requiring nebulizer maintenance or cleaning.

This performance was achieved through the elimination of nebulizer blockages, removal of time-consuming filtration or centrifugation steps, and a significant reduction in maintenance requirements. Downtime common with concentric nebulizers—often due to frequent blockages—was eliminated, freeing operator time for higher-value tasks.

Importantly, these efficiency gains offer measurable cost savings, increased sample throughput, and improved reliability of ICP-MS analyses. Elimination of disposable filters and reduced cleaning contributes to additional cost reduction.

The approach is broadly applicable to commercial, regulatory, and research laboratories handling complex or variable matrices, including environmental, clinical, and industrial samples. Based on the demonstrated stability and performance, this technology shows strong potential for adoption in high-throughput laboratories worldwide.

References

(1) Thomas, R. *Practical Guide to ICP-MS and Other Atomic Spectroscopy Techniques*, 4th Edition; CRC Press, 2023. Chapter 3: Sample Introduction, p. 16.

(2) Leikin, S. Analytical Performance Evaluation of a New Nebulizer for ICP-MS Analysis of Dif-

ficult Samples. *Winter Conference on Plasma Spectrochemistry, Abstracts*; 2020.
(3) Todoli, J-L; Mermet, J-M. *Liquid Sample Introduction in ICP Spectrometry: A Practical Guide;* Elsevier, 2011.
(4) Jones, D. R. et al. Analysis of Whole Human Blood for Pb, Cd, Hg, Se, and Mn by ICP-DRC-MS for Biomonitoring and Acute Exposures. *Talanta* **2017,** *16*, 114–122. DOI: 10.1016/j.talanta.2016.09.060
(5) Heitland, P.; Koster, H. D, Human Biomonitoring of 73 Elements in Blood, Serum, Erythrocytes and Urine. *J. Trace Elem. Med.Biol.* **2021,** *64*, 126706. DOI: 10.1016/j.jtemb.2020.126706

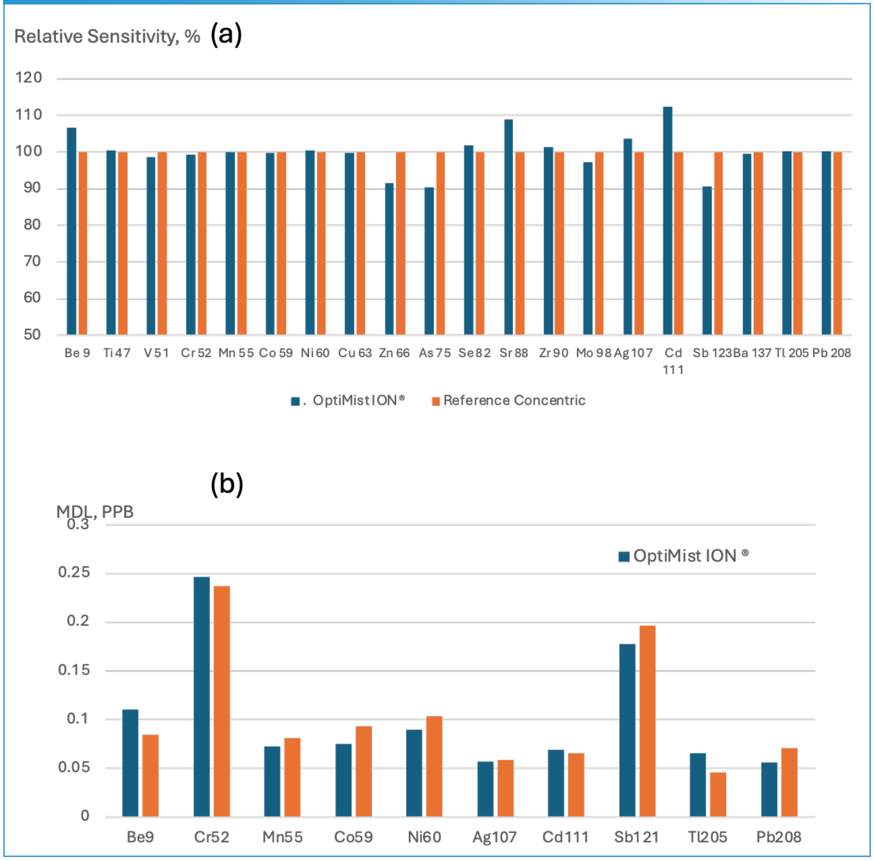


FIGURE 8: (a) Relative sensitivity and (b) method detection limits (MDL) compared with a concentric micro-nebulizer designed for low sample uptake rates (MDL data courtesy of Bonner Analytical Lab). Experiments were carried out on two commercially available ICP-MS systems (see text for details).

Dr. Sergei Leikin is a co-founder and Managing Director of TSP (Texas Scientific Products) – a supplier of high quality consumables and innovative sample introduction systems for ICP Atomic Emission and Mass Spectrometers. Prior to starting the TSP he worked as an Applications Laboratory Manager for premier instrument manufacturers of both the ICP-OES, ICP-MS and Microwave Digestion equipment.

Analytical chemist with an extensive scientific background, Sergei utilizes his 38 years of experience in atomic spectroscopy to help solve every day real-world material analysis problems.

At TSP Dr. Leikin directs the new product and business development while keeping the focus on what he likes the most – problem solving.

Sergei received a Master's Degree in Optical-Electronic Systems and a PhD in Analytical Chemistry.

Mary Kay Amistadi earned M.S. in soil chemistry from Pennsylvania State University. Currently serving as research laboratory manager for the Arizona Laboratory for Emerging Contaminants (ALEC) at University of Arizona in Tucson, providing method development and analytical services in ICP-MS.

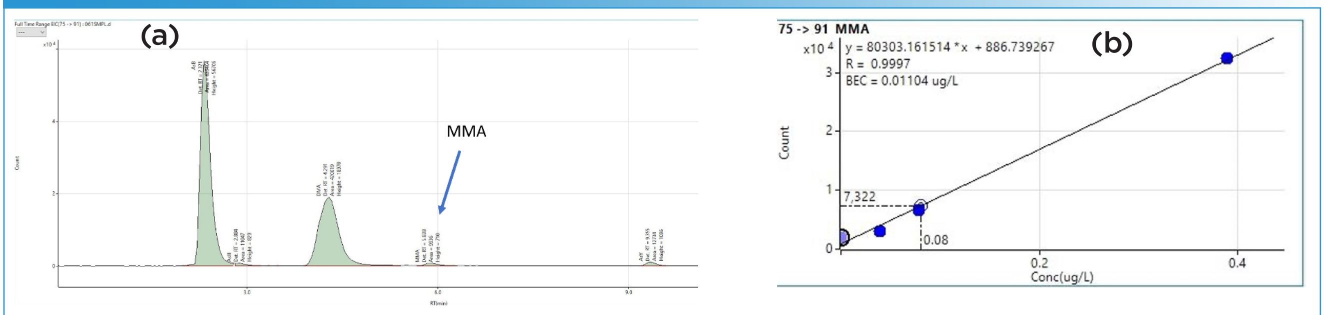


FIGURE 9: Detection of arsenic as MMA in urine carried out using an anion exchange column coupled with an ICP-MS system fitted with the nebulizer described in this study (a) chromatogram (b) calibration curve near the detection limit showing a 90 ppt sample (just above 80 ppt standard)

Redefining the Limits of Uptime & Throughput in Water Analyses with the NexION 1100 ICP-MS

Ruth Merrifield – Global Product Manager, ICP-MS
Liyan Xing – Senior Application Scientist, ICP-MS
PerkinElmer U.S. LLC

When conducting trace elemental analyses of waters, regardless of the type of laboratory you're part of, you are dealing with human or environmental health and are therefore subject to regulatory limits that reduce exposure to toxic levels of metals. For all laboratories concerned with following regulated or validated methods, having reliable instrumentation that is easy to maintain is paramount. Often, these labs must deal with a wide range of sample matrices, requiring minimal instrument downtime with maximum productivity.

This two-part study demonstrates the robustness and stability of PerkinElmer's **NexION® 1100 ICP-MS** (Figure 1) through the analysis of drinking water samples in Standard mode following U.S. EPA Method 200.8 (Part 1) as well as the analysis of simulated natural water samples using Collision/KED mode (Part 2).

For the more challenging analysis of simulated natural water with 0.25% total dissolved solids (TDS), 5200 samples were run over a 26-day period. Routine maintenance was conducted every 1400 samples, taking no more than 10 minutes to complete. This resulted in less than 1% of the overall time spent on routine cleaning while allowing the instrument to be running analytical methods for 92% of the total experimental time (Figure 2), demonstrating the robustness and stability of the NexION 1100 ICP-MS, for all types of water samples.

The operating conditions used in both sets of analyses are summarized in Table 1.

Part 1: Drinking Water in Standard Mode – U.S. EPA 200.8

Here, the NexION 1100 ICP-MS was used to analyze drinking water samples in Standard mode (no gas), in accordance with U.S. EPA Method 200.8. A total of 26 elements were measured, with 21 required following Method 200.8, and 5 additional elements were included for information purposes. Instrument performance was assessed in terms of detection limits, analytical accuracy, and robustness.

Detection Limits:

The NexION 1100 ICP-MS achieved IDLs and MDLs well below

Figure 1. PerkinElmer NexION 1100 ICP-MS

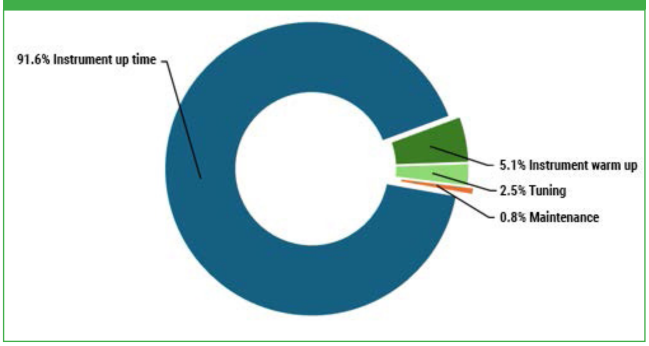


Figure 2. Percentage of overall time used for maintenance and optimization activities versus sample analysis (instrument productivity) on the NexION 1100 ICP-MS over 26-day project.

Method 200.8 requirements (Figure 3A), ensuring reliable trace to ultra-trace analysis. Its unique Extended Dynamic Range (EDR) electronic dilution capability increases linear dynamic range to 10^{14}, allowing the analysis of both high- and low-concentration elements in a single analytical run, without a cell gas. This dual capability streamlines workflows by enabling both trace and major element determinations in a single run.

Stability & Accuracy:

Stability was confirmed by the consistent recoveries of both internal standards and continuous calibration verification (CCV) samples during a 10-hour run of mid-hardness tap water (Figures 3B-C).

TABLE 1. NexION 1100 ICP-MS Parameters and Operating Conditions

Instrument Component/Parameter	Type/Value
ICP-MS	NexION 1100 (PerkinElmer)
Nebulizer	MEINHARD® Glass
Spray Chamber	Quartz Cyclonic with AMS (All Matrix System) Gas Port
Torch	One-Piece Quartz, 2 mm Injector
Interface	Standard Nickel Sampler and Skimmer Aluminium Hyper-Skimmer
Peripump Tubing	Carrier: Orange/Green (0.38 mm i.d.) ISTD: Orange/Green (0.38 mm i.d.) Waste: Gray/Gray Santoprene (1.30 mm i.d.)
Operating Conditions	**Type/Value**
RF Power	1600 W
Plasma Gas Flow	15 L/min
Auxiliary Gas Flow	1.2 L/min
Nebulizer Gas Flow	Optimized for $CeO^+/Ce^+<1.5\%$
Sample Uptake Rate (Carrier and IS combined)	0.24 mL/min
AMS Gas Flow (for natural waters only)	0.1 mL/min (~2X dilution)
Data Acquisition	**Type/Value**
Sweeps	20
Dwell Time	25-100 ms
Replicates	3
Autosampler and HTS	**Type/Value**
Autosampler	S23 (PerkinElmer)
Sample Loop Size	1.0 mL
Autosampler Probe	1.0 mm i.d.
Flush Delay	6 s
Read Delay	30 s
Probe Rinse	5 s
Wash	30 s
Peripump Speed	80 rpm

Accuracy was verified through the analysis of certified reference materials (CRMs) and spiked water samples, with recoveries within ±10 percent for all target elements (Figure 3D).

Part 2: Simulated Natural Waters Leveraging Collision/KED Mode

In this part of the study, He Collision/KED mode was leveraged on the NexION 1100 ICP-MS for the analysis of 5200 simulated natural water samples with 0.25% TDS over a 26-day period. A total of 24 elements were measured. Instrument performance was assessed in terms of detection limits, analytical accuracy, and robustness.

Detection Limits:

The method detection limits (MDLs) were measured at the end of each day to ensure that they were well within reporting limits. Figure 4A shows that the MDLs for each element do not significantly change over the 26 days of analysis, demonstrating that the NexION 1100 ICP-MS produces stable, reliable results.

Stability & Accuracy:

In this case, a continuous calibration verification (CCV) was run every 10 samples, along with internal standards for each injection as well as the analysis of a certified reference material (CRM) and matrix spike at the beginning, middle and end of the samples. The stability of the CCVs can be seen for over 5000 samples (Figure 4C). All CCVs are well within the +/- 20% allowable for a natural water method. Typical internal standard graphs, matrix spikes and CRM values from before and after a routine cleaning procedure can be seen in the cutout (Figures 4B-D), again demonstrating that all values are well within the allowable limits for these types of samples.

Conclusion

By combining exceptional sensitivity, accuracy, and long-term stability, PerkinElmer's NexION 1100 ICP-MS, with a host of proprietary technologies engineered with the user's efficiency in mind, offers a robust and reliable solution for laboratories tasked to meet the stringent requirements for trace-elemental analysis of water samples – from the simpler drinking water all the way to the more challenging natural water ones – while also necessitating fast turnaround. This is thanks to a range of proprietary technologies that come together to deliver accurate, repeatable results with minimal downtime. These include:

Extended Dynamic Range is an electronic dilution offering the highest linear dynamic range on the market – up to 14 orders of magnitude – providing the capability to run high- and low-concentration elements in the same run. Applied per-mass, this approach extends the linear range or attenuates high signals, reducing detector load and prolonging detector life.

High Throughput System is a flow injection module that helps speed up sample-to-sample time and reduces carryover by injecting into the system only the required amount of sample necessary, keeping the probe clean and eliminating contact between the sample and the peripump tubing.

Peltier-cooled Spray Chamber, like

the PC3X, provides enhanced stability and performance through temperature control.

All Matrix Solution online gas dilution allows for easy analysis of up to 35% total dissolved solids without excessive manual dilutions.

Triple Cone Interface and Quadrupole Ion Deflector: The Triple Cone Interface, featuring a unique Hyper-Skimmer cone and wide-aperture design, delivers a focused, stable ion beam with minimal clogging. Combined with the Quadrupole Ion Deflector, it eliminates the need for reaction cell maintenance.

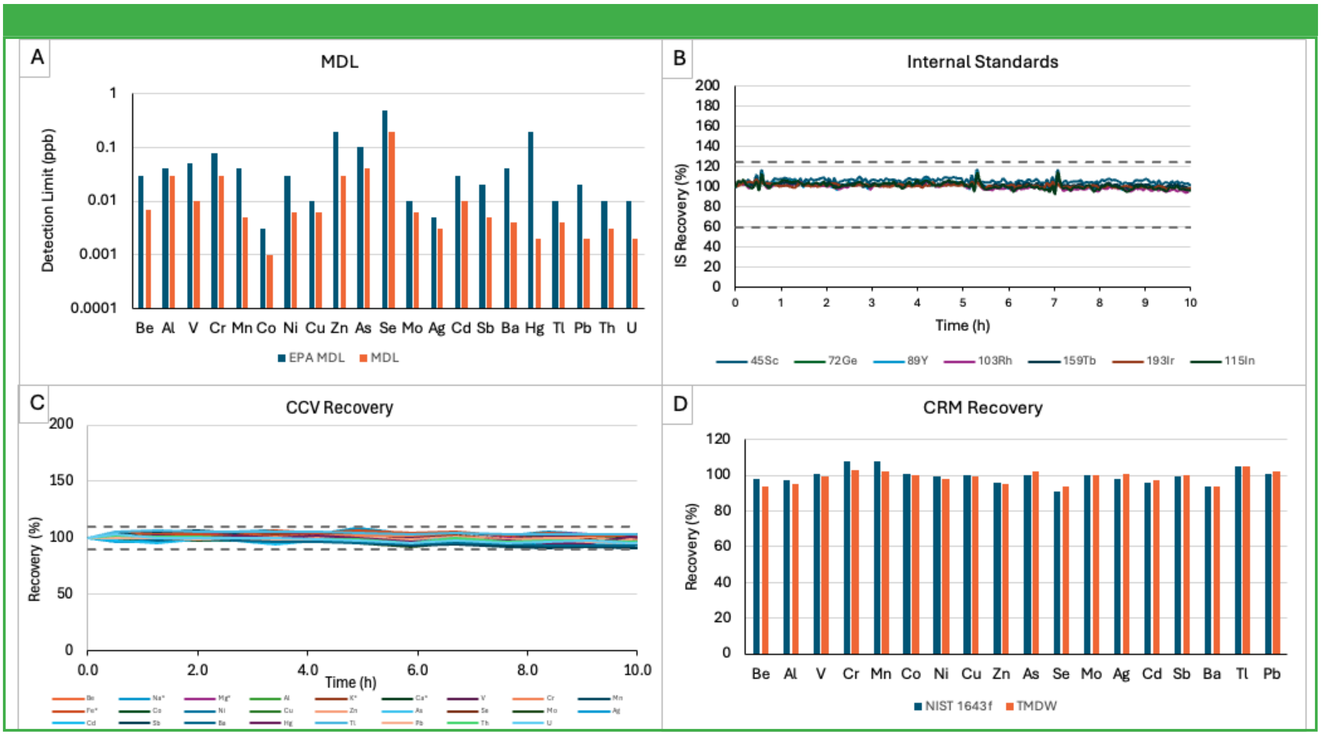


Figure 3. A) MDL, B) Internal standard recovery, C) CCV recovery, and D) CRM recovery for drinking water using 200.8 methodology.

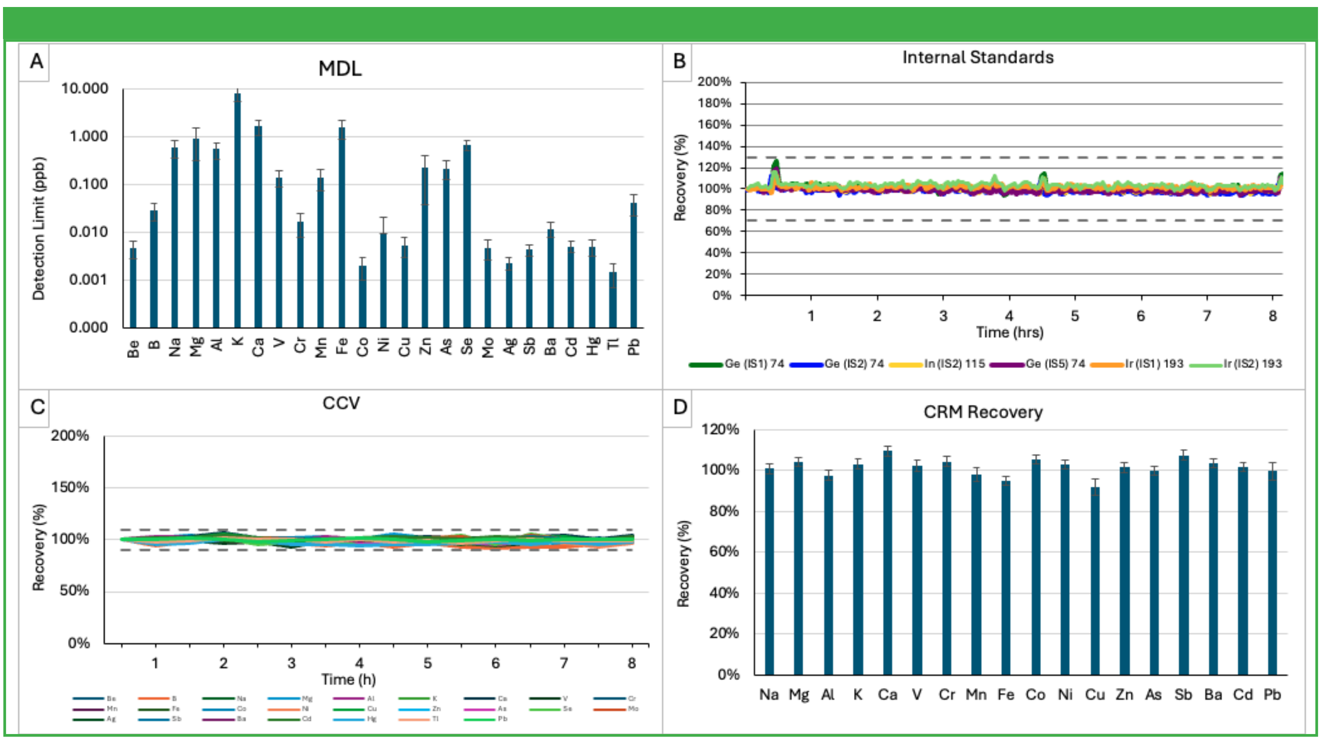


Figure 4. A) MDL, B) Internal standard recovery, C) CCV recovery, and D) CRM recovery for natural waters using Collision/KED methodology.

FEATURED ARTICLE

Behind the Signal Drop: Distinguishing Between Cone and Sample Introduction Issues in ICP-MS

Genette Alcaraz, Elizabeth Ranalli, and Sergei Leikin

Reduction in signal intensity or analytical precision is the most common performance issues inductively coupled plasma mass spectroscopy (ICP-MS) users are faced with. Troubleshooting these problems can be costly since distinguishing between cone degradation and issues related to the sample introduction system is often challenging—even for experienced analysts. A rational, step-by-step approach to diagnosing and resolving the common ICP-MS performance issues is proposed. It includes practical guidance on inspection criteria and maintenance protocols to determine when to clean, refurbish, or replace interface cones. The proposed approach is intended to reduce instrument downtime and operational costs by extending the cone lifetime and minimizing unnecessary replacement.

"My ICP-MS was working perfectly yesterday, overnight run went well, but today the sensitivity is down, and instrument can't pass the tuning routine."

Inductively coupled plasma mass spectroscopy (ICP-MS) analysts who run the analysis daily are familiar with this situation. Performance issues can vary but typically are narrowed down to either the loss of sensitivity or degradation of the precision or the combination of both. In addition, there might be a problem with the long-term stability when QC check samples are out of the acceptable range and instrument software calls for recalibration.

ICP-MS is a complicated system that includes multiple components which must work in concert. Still, over 95% of the performance problems are related to either the sample introduction system or the interface region—the sampler and skimmer cones. What's more frustrating for an analyst than replacing interface cones—only to learn the real problem was with the nebulizer, spray chamber, peristaltic pump tubing, or the pump itself? That is why distinguishing between the sample introduction system and the interface when troubleshooting ICP-MS performance issues is a challenging task, even for an experienced analyst, and requires a rational, step-by-step approach.

Approaching the Problem

Rather than immediately focusing on the sample introduction or interface region, it is best to first view the problem from the perspective of the application. While modern ICP-MS instruments are designed to handle a wide range of matrices—from drinking water to industrial waste or high total dissolved solids (TDS) brines—the impact on internal components can vary dramatically. The presence of calcium—even at a few hundred ppm—in complex matrices can quickly lead to material accumulation on the sampler and skimmer cones and its inspection makes common sense.

Moreover, since sample deposition on the cones is unavoidable when working with complex matrices, a common recommendation is to 'condition' new or freshly cleaned cones by aspirating a calcium-containing solution for approximately 10 min prior to calibration. In cases where the sample matrix is stable and predictable, using the actual sample solution to condition the cones for a few minutes is often sufficient. When samples with elevated TDS are analyzed, salt buildup in the nebulizer is a common concern. A straightforward way to assess this is by applying nebulizer gas at 1.0 L/min and monitoring the pressure, all without plasma ignition. Elevated pressure is a sign that the nebulizer requires cleaning to restore its performance.

The next logical step is to review instrument data, as it often provides the most reliable insight—particularly when considered alongside application-specific factors. The standard tuning procedure generates a performance report that includes sensitivity across the mass range, as well as oxide and doubly charged ion levels. If the argon gas or tuning solution is confirmed to be clean, elevated ratios may point to cone wear or contamination. Elevated interface pressure (such as reduced vacuum quality) is another indicator that the cones may require inspection or maintenance.

When neither application-specific factors nor instrument performance data clearly identify the issue, the next logical step is to inspect the sample introduction system in the following sequence:

- **Nebulizer:** Test nebulizer backpressure with gas flow through the nebulizer only; Aspirate water and check aerosol visually.
- **Peristaltic pump, Tubing and Connections**: Check tubing wear, roller tension, and flow consistency. Inspect for leaks, kinks, or blockages.
- **Spray chamber:** Check for large liquid droplets buildup and inconsistent draining.
- **Torch and injector:** check for the sample deposit on the tip or inside the torch injector.

It should be noted that routine maintenance recommended by ICP-MS manufactures and described in multiple publications may prevent the vast majority of the performance issues (1).

Nebulizer

Modern ICP-MS instruments are standardized to operate with a small sample uptake rate in the range 0.100-0.400 mL/min. Concentric nebulizers usually employed for their high efficiency and good precision are prone to blockages due to their tiny sample capillary and especially if not really clean solutions are analyzed (2).

As mentioned earlier, a nebulizer backpressure test without having to light the plasma is quick and a simple way to diagnose the blockage.

If backpressure is normal and within the nebulizer manufacturing specification, a look at the aerosol mist while aspirating deionized (DI) water on the dark background is another simple, quick, and efficient way to diagnose the nebulizer functionality (Figure 1).

If these tests are still non-conclusive, trying a "backup" nebulizer with a known-good operational condition should help to narrow down the problem.

FIGURE 1: Observing the nebulizer mist on the dark background.

Frequent nebulizer blockages is an indication of the wrong nebulizer type used for the application. If high-total dissolved solids (TDS) samples make up a significant portion of the laboratory workload, an argon humidifier is a must-have to prevent salt buildup around the nebulizer orifice.

Peristaltic Pump and Tubing

Worn out or flattened peristaltic pump tubing is quite often overlooked. Proactively replacing the sample and internal standard tubing on a regular basis is a simple yet effective preventive measure since it will help to focus on the other components of the sample introduction system. The frequency of tubing replacement depends on the sample throughput and type of matrices. It varies from daily to once every few days provided the tubing clamps of the peristaltic pump are released at the end of the run.

Any connections within the sample introduction path are highly recommended to check. Even a tiny leak or presence of air bubbles can affect the data precision.

Spray Chamber

The most common issue is inconsistent draining of the spray chamber. On modern ICP-MS instruments, where spray chambers are usually hidden behind Peltier cooling blocks, checking for steady drain flow is a straightforward way to identify possible issues.

If inconsistency is observed, it is worth to disassemble the Peltier cooler and look at the spray chamber conditions. Presence of accumulated large water droplets on the surface is a certain sign that cleaning is needed.

Torch and Injector

Finding the sample deposit either on the tip of the torch injector or inside the injector usually is not an issue with clean samples, but quite common when challenging matrices are analyzed. Direct analysis of oils or whole blood after dilution with appropriate solvent is just one example where sample deposition is expected to happen and the torch needs to be looked at closely.

Plasma Appearance

A plasma truly can be worth a thousand words. Its appearance serves as a quick and effective diagnostic tool. Flickering or the loss of the darker central channel—where the aerosol normally travels—often indicates a problem in the sample introduction system. Similarly, an unusually bright or misshaped plasma may indicate a leak.

The step-by-step diagnostics of ICP-MS performance issues is summarized in Figure 2.

If sample introduction system appears to be functioning properly, cone assessment becomes the next logical step.

Diagnosing and Restoring ICP-MS Performance: When to Clean, Refurbish, or Retire Interface Cones

This section outlines best practices for assessing cone condition, cleaning appropriately, and determining when platinum refurbishment or full replacement is warranted—all of which play a key role in restoring analytical performance and minimizing disruptions.

Visual Indicators of Cone Degradation

Sampler and skimmer cones serve as the critical interface between the plasma and the mass analyzer. Over time, their performance can degrade due to matrix buildup, physical wear, or damage. Cones should only be removed for inspection when baseline performance has declined, and upstream sample introduction components have been ruled out. Performance decline with no recovery after cleaning is a strong indicator that physical degradation has exceeded user maintenance thresholds and additional action is needed.

Key visual indicators of cone-related degradation are shown in Figure 3 (3).

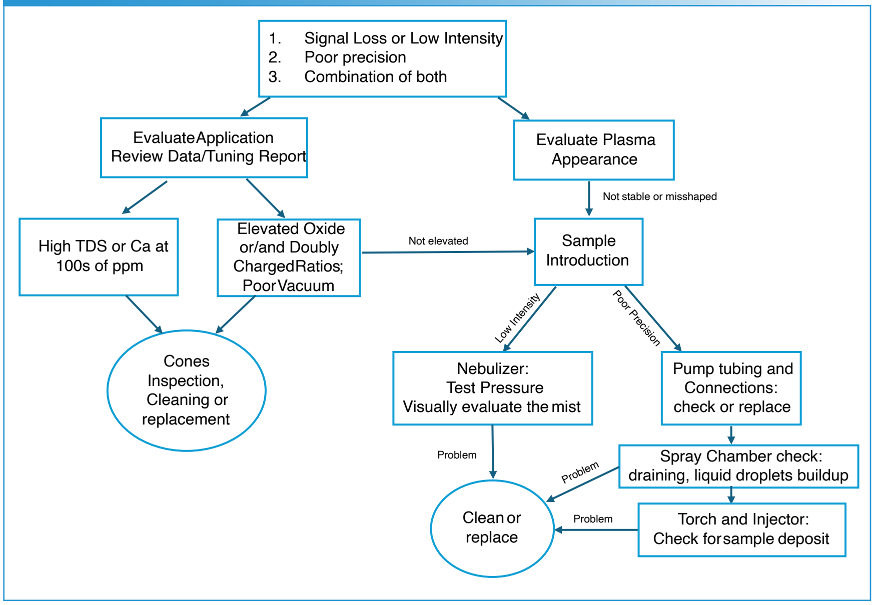


FIGURE 2: ICP-MS troubleshooting diagnostics: Sample introduction relative to cones.

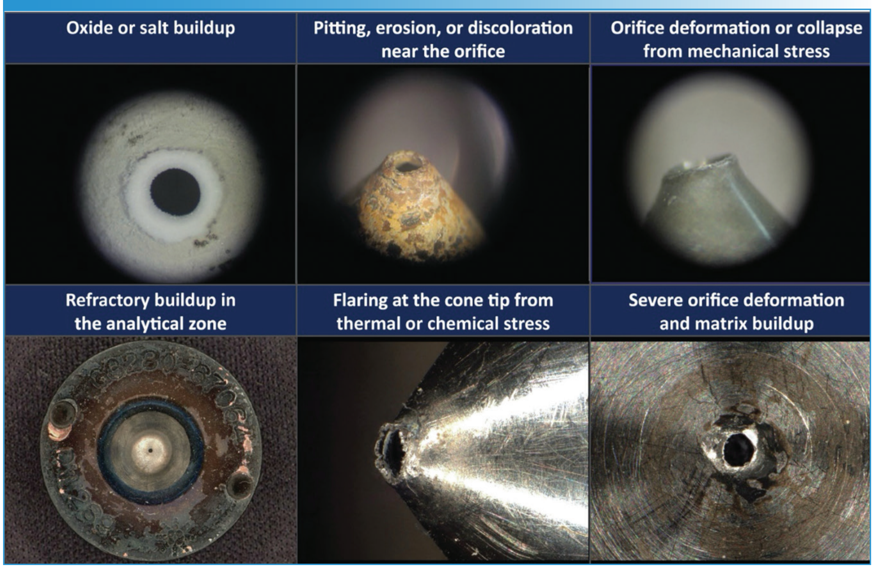


FIGURE 3: Different examples of ICP-MS cone degradation (x100-x150 magnification).

Cone Cleaning Guidelines for Optimal ICP-MS Performance

Proper cleaning is the first line of defense against signal degradation, but overly aggressive or incorrect methods can damage cones and reduce their lifespan. To maintain consistent performance and preserve cone integrity, the following cleaning protocols are recommended.

When to Clean

- Signal intensity drops or becomes unstable
- Background levels increase or become noisy (show high relative standard deviation [RSD])
- You observe deposits near the orifice

Gentle Cleaning Step

- Gently wipe the cone surface using a cotton swab with pure deionized water
- Rinse thoroughly with deionized water
- Place the cone in cleaning fixture with inserts or similar container or beaker
- Sonicate for 2-5 min in 1:20 Citranox Solution (Alcanox, Inc.) at 30 °C
- Rinse with deionized water
- Place the cone in cleaning fixture or place in sonicator
- Sonicate for 5 min in fresh deionized water at 30 °C
- Let air dry or blow dry with filtered compressed air or nitrogen if available

If gentle cleaning has not cleared the orifice of the cone proceed with enhanced cleaning.

Enhanced Cleaning Step

All Cones

- Place the cone in cleaning fixture with reservoir or similar container or beaker
- Soak in 1:20 Citranox solution for 2 h and then sonicate 30 °C for the last 15 min
- Rinse with tap water
- Place the tip of the cone only in the insert cup with reservoir or similar

container or beaker containing 1-2% HNO_3 for 10 min (Always use a cleaning fixture or modified set up to ensure the Ni or Cu base are fully protected, ensuring only the Pt tip is exposed to the acid environment).
- Rinse with tap water
- Return to cone cleaning fixture with insert cups or similar container or beaker for another 10-min soak
- Rinse with tap water
- Rinse and Return to cone cleaning fixture with insert cups or place in sonicator. Sonicate 30 °C in deionized water for 15 min
- Replace deionized water and repeat sonication
- Let air dry or blow dry with filtered compressed air or nitrogen if available

For Platinum Tips Only

- Place the cone in cleaning fixture with reservoir or similar container or beaker
- Soak in 1:20 Citranox solution for 2 h and sonicate 30 °C for the last 15 min
- Place the tip of the cone only in the insert cup with reservoir or similar container or beaker containing up to 5% HNO_3 for 10 min (Always use a cleaning fixture or modified set up to ensure the Ni or Cu base are fully protected, ensuring only the Pt tip is exposed to the acid environment).
- Rinse with tap water
- Return to cone cleaning fixture with insert cups or similar container or beaker for another 10-min soak
- Rinse with tap water
- Rinse and Return to cone cleaning fixture with insert cups or place in sonicator. Sonicate 30 °C in deionized water for 15 min
- Replace deionized water and repeat sonication
- Let air dry or blow dry with filtered compressed air or nitrogen if available

These cleaning procedures help preserve cone geometry and surface finish, extending cone life and ensuring optimal performance post cleaning, as shown in Figure 4.

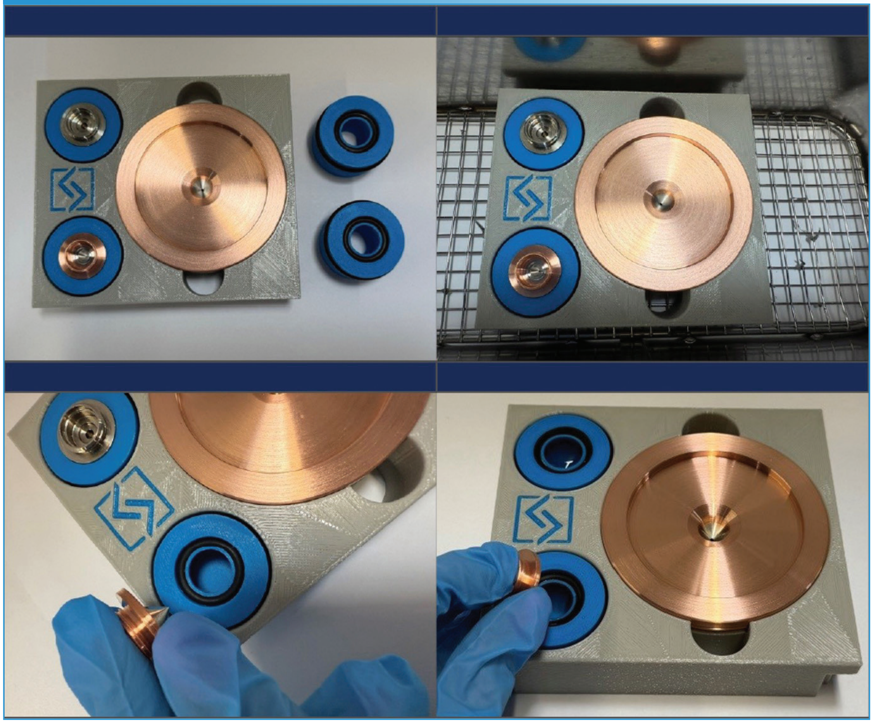


FIGURE 4: Cone cleaning fixture with Insert and reservoir cups shows an example of proper cleaning techniques for cones to ensure an extended life span.

When to Refurbish

If cleaning fails to restore performance, a platinum cone may be a candidate for refurbishment. Spectron's platinum cone refurbishing service uses precision techniques to:

- Remove embedded material or occlusions
- Reshape the orifice to restore correct geometry
- Inspect and verify dimensional tolerances

Depending on usage and condition, platinum cones can often be refurbished multiple times, extending their life by a factor of three to four. The images in Figure 5 show successful restoration outcomes for cones that would otherwise have been discarded due to orifice damage and partial or full occlusion.

Refurbishment is particularly beneficial when damage is from mechanical impact (for example, dropped cones) or heavy deposition, but enough material remains for reworking.

When to Retire a Cone

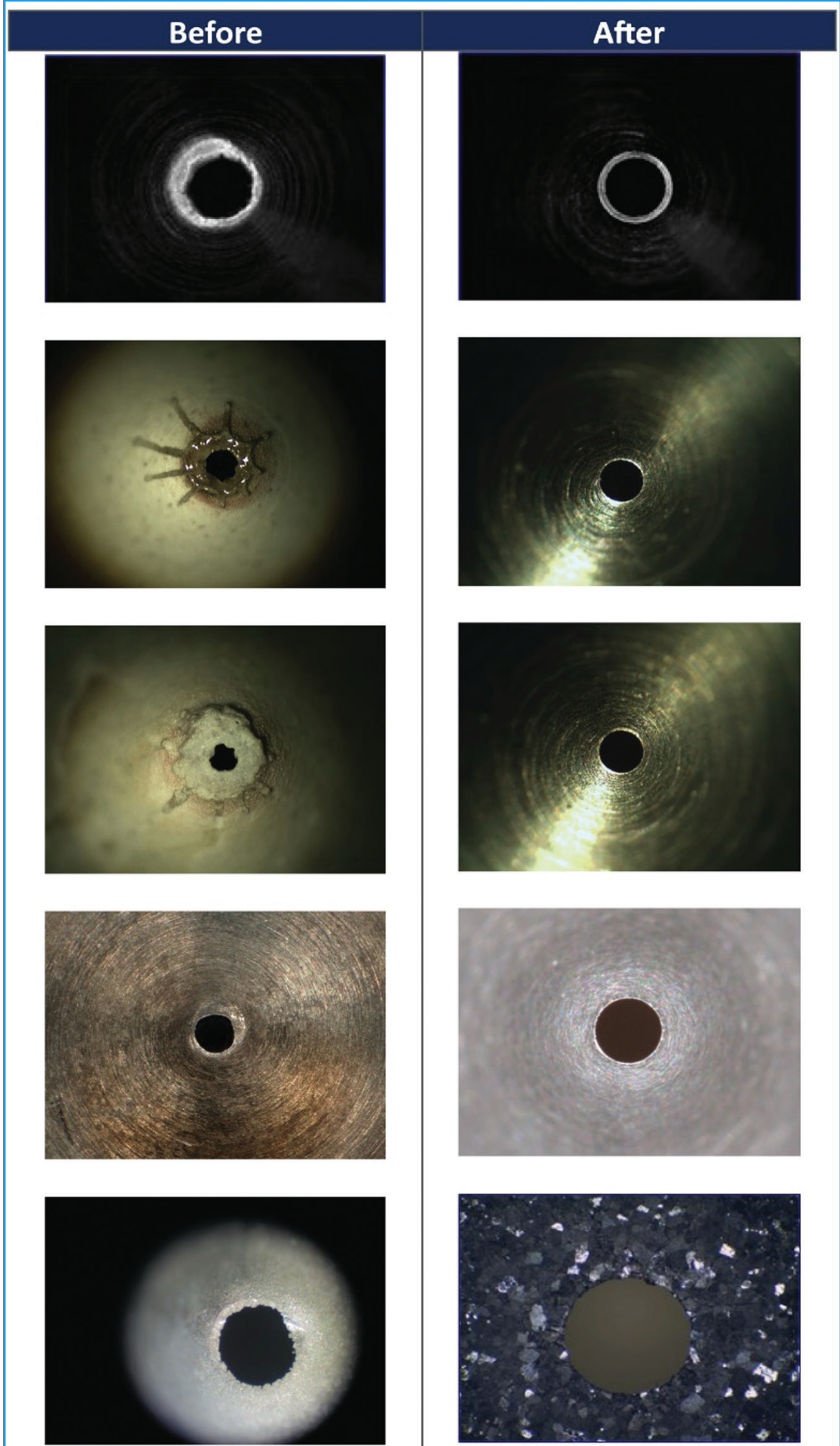
Not all platinum cones are refurbishable. A cone should be retired when:

- The material is brittle, porous, or annealed
- There is severe orifice collapse or cracking
- The cone lacks sufficient material to reform within manufacturer specifications

Spectron offers platinum credit for unusable cones through our reclaim program, issuing credit based on the average market value of the platinum alloy recovered. This adds value while supporting sustainability and waste reduction.

Conclusion

Extending cone life and optimizing ICP-MS performance requires a combination of structured diagnostics and well-informed maintenance decisions. Once upstream sample introduction components are ruled out, visual inspection, proper cleaning, and professional refurbishment, when appropriate, can restore analytical performance without unnecessary replacement. Figure 6 is a flowchart



Platinum cone damaged from mishandling and chemically degraded beyond repair

Platinum cone severely annealed with brittle material

FIGURE 5: (Left column) before, and (right column) after successful restoration of platinum ICP-MS cones and examples of material or damage that cannot be restored. (x100-x150 magnification).

to better understand cone-related performance issues.

Key takeaways

- Before reaching out to any components (sample introduction or cones) evaluate the analysis from the analytical application standpoint, which may lead to identifying the most likely component to inspect.
- Analyze the performance data available by checking the tuning report. Certain data may guide to the root of the performance issues
- If clean samples are run and there are no signs in the data directly pointing out to cones, start with sample introduction
- Evaluate plasma appearance as a quick and effective diagnostic tool
- After front-end causes are ruled out, turn to cone inspection
- Use metal-specific cleaning protocols to avoid introducing new performance issues
- Consider refurbishing platinum cones as a sustainable, cost-effective alternative to replacement
- Retire cones when material integrity or orifice geometry cannot be restored

By adopting these best practices, ICP-MS users can reduce downtime, maintain data quality, and extend the working life of high-value components.

References

(1) Thomas, R. The Importance of Routine Maintenance in ICP-MS. *Spectroscopy* **2024,** *39* (s9), 24–31. DOI: 10.56530/spectroscopy.pk7184n1

(2) Thomas, R. *Practical Guide to ICP-MS and Other Atomic Spectroscopy Techniques*, 4th Edition; CRC Press 2023. Chapter 3: Sample Introduction, Pages 11-21, 2023

(3) Neufeld, L. ICP-MS Interface Cones: Maintaining the Critical Interface between the Mass Spectrometer and the Plasma Discharge to Optimize Performance and Maximize Instrument Productivity. *Spectroscopy* **2019,** *34* (7), 12-17. https://www.spectroscopyonline.com/view/icp-ms-interface-cones-maintaining-critical-interface-between-mass-spectrometer-and-plasma-discharge

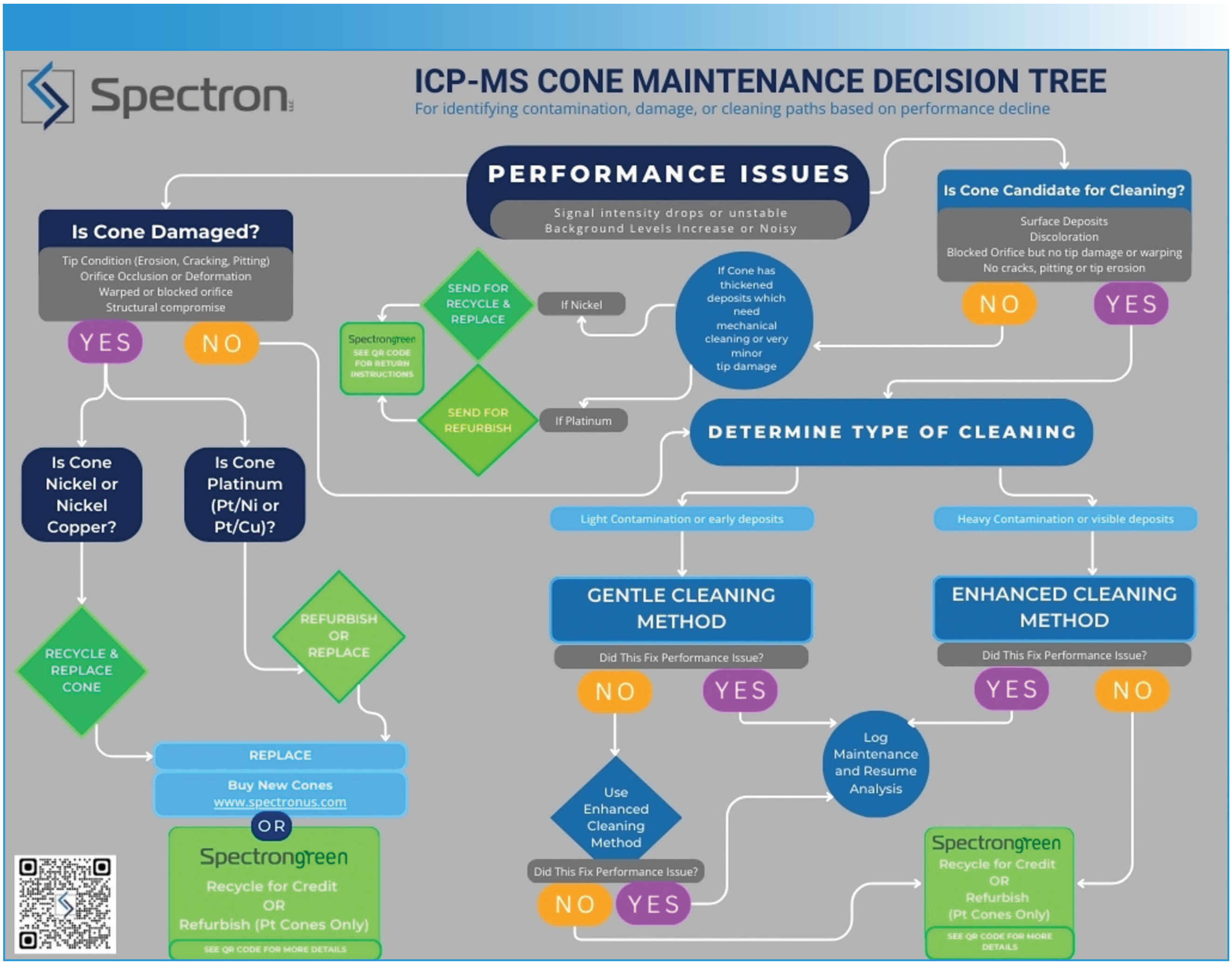


FIGURE 6: ICP-MS cone maintenance decision tree.

Genette Alcaraz is the CEO of Spectron LLC, a precision technical manufacturing company specializing in scientific consumables for ICP-MS as well as aerospace, and defense products. With over 20 years of experience in scientific manufacturing, she has worked both with and for large OEMs and is known for her results-driven leadership and commitment to continuous improvement. Genette fosters extremely successful business partnerships and builds customer relationships that are tailored to meet the changing needs of Spectron's customers, products and marketplace while maintaining high integrity and best-in-class quality.

Liz Ranalli is the Sales Director at Spectron LLC, where she has spent the past eight years supporting customers with ICP-MS cone solutions and technical sales. She previously spent a decade at Oxford Instruments in their atomic force spectroscopy division, managing consumable product lines and service operations. With nearly two decades of experience in scientific instrumentation and consumables, Liz values building strong partnerships and supporting the scientific community she serves.

Sergei Leikin is a co-founder and Managing Director of TSP (Texas Scientific Products) – a supplier of high quality consumables and innovative sample introduction systems for ICP Atomic Emission and Mass Spectrometers. Prior to starting the TSP he worked as an Applications Laboratory Manager for premier instrument manufacturers of both the ICP-OES, ICP-MS, and Microwave Digestion equipment.

Analytical chemist with an extensive scientific background, Sergei utilizes his 38 years of experience in atomic spectroscopy to help solve every day real-world material analysis problems.

At TSP Dr. Leikin directs the new product and business development while keeping the focus on what he likes the most – problem solving.

Sergei received a Master's Degree in Optical-Electronic Systems and PhD in Analytical Chemistry .

Silent Saboteurs: Hidden Contaminants, Impurities, and Artifacts in Trace Element Analysis

Patricia Atkins

As trace-level elemental analysis evolves, contamination has become the primary threat to data quality, particularly in techniques such as inductively coupled plasma–mass spectroscopy (ICP-MS). This article explores the multifaceted sources of laboratory contamination, including impurities in high-purity acids and water, leachables from labware and polymers, environmental background, and biological contamination such as microbes and biofilms. Special attention is given to emerging contaminants such as microplastics, their interactions with trace metals, and their dual role as carriers and substrates for microbial growth. The article also addresses the increasing complexity of polyatomic and isobaric interferences in contaminated systems, outlining strategies for mitigation and emphasizing the role of certified reference materials (CRMs). The discussion culminates in best practices for ensuring data fidelity in high-stakes environments such as environmental, food safety, pharmaceutical, and semiconductor laboratories.

Advances in inorganic and elemental analysis have revolutionized our ability to detect increasingly lower concentrations. However, with heightened sensitivity comes a corresponding rise in susceptibility to contamination. Contamination levels once considered analytically negligible now pose significant sources of error, particularly in regulated environments such as environmental monitoring, food safety, semiconductor manufacturing, and pharmaceutical quality control.

As the analytical capabilities of inductively coupled plasma–mass spectroscopy (ICP-MS) and other systems improve, laboratories must elevate their awareness of potential contamination sources and interference mechanisms. The notion of a "clean laboratory" has evolved beyond laminar flow hoods and powder-free gloves to encompass a complex understanding of chemical, physical, and biological contributors to analytical error.

Certified reference materials (CRMs) remain essential tools in modern trace elemental analysis, providing the accuracy and traceability needed for high-confidence results. Yet even the most rigorously validated CRMs cannot compensate for contamination introduced during the analytical process. In a world where parts-per-billion (ppb) and parts-per-trillion (ppt) are the new norm, unrecognized contamination—often from seemingly trivial sources—can exert a disproportionately disruptive effect. Despite advanced instrumentation, laboratories frequently encounter unexplained interferences, out-of-spec results, or questionable recoveries, many of which trace back not to instrument drift or poor calibration, but to subtle, unrecognized contamination.

A striking historical example of such contamination occurred in the field of oceanographic research. For decades, global ocean surveys reported seawater concentrations contaminated by trace metals leaching from ship structures and sampling equipment. This issue inflated readings of iron, copper, and zinc—distorting models of nutrient cycling and even influencing environmental policy decisions. This prompted a paradigm shift toward "trace-metal clean" sampling techniques and stringent contamination controls at sea. The economic and scientific consequences of revising decades of data were substantial (1).

Closer to the laboratory bench, documented studies have revealed equally impactful contamination pathways. For instance, routine barcoded labels on sample tubes have been shown to introduce measurable amounts of zinc into trace metal analyses (2), while minor lapses in glassware or pipette cleaning have elevated sodium and calcium backgrounds by orders of magnitude (3). Such contamination not only degrades data quality, but can directly impact environmental assessments, regulatory decisions, and public health responses.

Laboratory chemists and quality professionals often first suspect their instrument or their standards when results deviate from expectations. A common concern raised is that a standard is "too high" in a particular element—only to discover upon closer review that contamination arose from the water used to dilute it, the gloves used to handle the vial, the pipette tips, or even airborne fibers settling into sample containers. These instances highlight the insidious nature of trace contamination—it often masquerades as calibration error or standard deviation.

As detection limits continue to decline, the vulnerability of analytical results to contamination increases exponentially. At this level of sensitivity, even a fraction of a microliter of contaminated rinse water or a few micrograms of improperly handled plasticware can derail entire datasets.

In this article, we present a comprehensive examination of contamination pathways that threaten trace elemental analysis—from acids and water, to labware, environmental dust, and even microbiological residues. We also address silent, emerging contaminants like microplastics and microbial activity, which are rarely considered yet increasingly relevant.

Acids, Reagents, and Inorganic Impurity Profiles

Inorganic trace analysis begins—and can often be undermined—by the quality of the acids and reagents used throughout the sample preparation process. Whether for digestion, dilution, rinsing, or matrix matching, acids such as nitric acid (HNO_3), hydrochloric acid (HCl), hydrofluoric acid (HF), and sulfuric acid (H_2SO_4) are ubiquitous in elemental laboratories. These acids, while often labeled as "trace metal grade" or "ultrapure," may still harbor elemental contaminants at levels ranging from parts per trillion to parts per billion. For techniques like ICP-MS that are capable of measuring at or below the ppt level, even sub-ng/L concentrations of iron, lead, uranium, or boron can introduce background signals, obscure true analyte levels, or inflate detection limits.

TABLE I: Grade categories for acids in inorganic analysis

Grade Category	Example Grade Names	Typical Use	Possible Metal Impurities
Reagent	ACS, AR, Lab Grade, Technical	General lab use	ppm to high ppb
Trace	Trace Metal Grade, TraceSELECT, Instra-Analyzed, ROMIL-SpA	ICP-OES, environmental methods	10–100 ppb
Ultra-trace	Optima, Suprapur, ULTREX, ROMIL-UpA, EL Grade	ICP-MS, semiconductor, isotope work	1–10 ppt, some <1 ppt

The challenge lies in the fact that not all high-purity labels mean the same thing across suppliers or production methods. For example, "reagent grade" nitric acid may contain dozens of elemental impurities in the low ppb to mid-ppb range, while "ultrapure" sub-boiling distilled acid may offer impurity profiles down in the low ppt or even sub-ppt range. Unfortunately, cost and procurement constraints sometimes force laboratories to use lower-grade acids for cleaning or rinsing, unknowingly introducing background contamination into even their highest-priority samples. A single reagent lot with elevated boron, tin, or sodium can affect a month's worth of environmental or semiconductor samples. Different grades of acids used in trace element analysis are shown in Table I.

Each acid even in pure forms can be susceptible to elemental contamination which can interfere with analysis. (Table II) For example contamination in nitric acid often stems from the nitrate source, reactor vessel materials, and packaging. In *reagent grade*, iron (Fe), lead (Pb), boron (B), sodium (Na), and zinc (Zn) are commonly introduced from metal reactors, leaching during storage in unlined drums, and atmospheric contact during bottling. *Trace grade* nitric acid is typically processed using plastic-lined equipment or non-metal systems and stored in high-purity high-density polyethylene (HDPE) containers, which reduces Fe, Sn, and Pb to the low ppb range. *Ultratrace grade* nitric acid is purified by sub-boiling distillation in cleanrooms using quartz or fluoropolymer systems, minimizing contamination to the 1–10 ppt level for elements like U, B, and Na.

Hydrochloric acid contamination arises from chlorine gas production equipment, reactor vessel linings, and container materials. *Reagent grade* HCl may contain Fe, Zn, Ca, and Pb due to corroded stainless steel systems, PVC tubing, or residual chlorinated byproducts. *Trace grade* HCl is filtered and stabilized to reduce Zn, Fe, and Na, with packaging in virgin plastic minimizing external contamination. For *ultratrace grade,* purification techniques such as sub-boiling distillation and inert gas bottling help limit impurities like Fe, Zn, and Na to the ppt level, with storage exclusively in fluorinated ethylene propylene (FEP) and perfluoroalkoxy alkane (PFA) containers to prevent leaching.

Contaminants in sulfuric acid originate from the raw sulfur feedstock and legacy production equipment. *Reagent grade* H_2SO_4 often contains Fe, Pb, and As from older lead-lined towers, vanadium catalysts, and unrefined SO_2 sources. *Trace grade* sulfuric acid is refined to eliminate ore-based metals like Cu and Zn, typically through distillation and filtration. *Ultra-trace grade* acid is produced using high purity SO_3 in clean synthesis systems and stored in non-leaching containers to suppress Na, Pb, and Fe levels to ppt concentrations.

Due to its aggressive nature, HF readily leaches contaminants from nearly all materials it contacts. In *reagent grade,* impurities such as Ca, Si, Fe, and Al are introduced from fluorospar sources, reactor linings, and HDPE containers. *Trace grade* HF is packaged under inert conditions and stored in high-purity plastic to limit leaching of Na and Ca, while filtration reduces particulate metals. For *ultra-trace grade*, sub-boiling distillation and PFA storage are used to reduce Si, Na, and Ca to single digit ppt levels, avoiding any exposure to glass or atmospheric moisture.

In addition to acid matrices, many laboratories rely on metal-containing reagents for sample stabilization or derivatization—such as zinc acetate, iron chloride, or magnesium nitrate. These compounds are often assumed to be sufficiently pure for use without verification. However, because these materials are often manufactured for industrial or pharmaceutical use, they may contain heavy metal contaminants (such as Cu, Pb, Ni, and Tl) that were not removed or tested for in production. Without validation of incoming reagent purity—or adequate use of blank subtraction—these impurities may distort calibration curves, spike recoveries, or internal standard behavior.

For best practices, laboratories should use sub-boiling distilled acids for critical applications, screen new reagent lots by ICP-MS before use, and utilize separate acid lots for cleaning and analysis. Glass- or fluoropolymer-bottled acids should be selected based on analyte risk (avoid borosilicate containers for B, Si, and Na testing). For critical applications such as semiconductor, pharmaceutical, or environmental compliance work, the cost of ultrapure acids is a necessary investment in data integrity.

TABLE II: Elemental impurities in common acids

Acid	Common Impurities (Reagent)	Common Impurities (Trace)	Common Impurities (Ultra-trace)
Nitric acid	Fe, Pb, B, Na, Zn, Cu, Ca	Fe, Pb, B, Zn, Sn, Na	B, Fe, Na, Pb (<10 ppt)
Hydrochloric acid	Fe, Pb, Zn, Ca, Al	Fe, Zn, Pb, Ca, Na	Fe, Zn, Na (<10 ppt)
Sulfuric acid	Fe, Pb, As, Na, Mg, Cu	Fe, Pb, Na, As, Zn	Na, Fe, Pb (<10 ppt)
Hydrofluoric acid	Si, Ca, Al, Fe, Mg, Na	Si, Ca, Na, Fe	Si, Na, Ca (<10 ppt)
Perchloric acid	Na, Fe, K, Ca	Na, Fe, Ca, Zn	Na, Fe, (<10 ppt)

TABLE III: Types of laboratory water and their definitions (4)

Water Type	Resistivity (MΩ·cm at 25°C)	TOC (ppb)	Silica (mg/L)	Sodium (µg/L)	Chloride (µg/L)	Recommended Use
Type I	≥ 18.0	≤ 50	≤ 0.005	≤ 1	≤ 1	ICP-MS, ultratrace metal analysis
Type II	≥ 1.0	≤ 50	≤ 0.01	≤ 5	≤ 5	AAS, ICP-OES, general trace analysis
Type III	≥ 0.1	N/A	≤ 0.05	≤ 50	≤ 50	Glassware rinsing, feed to Type I units

Water: The Universal Solvent and Contaminant Carrier

Water is the single most used reagent in any analytical laboratory, serving as a solvent for sample preparation, dilutions, rinse solutions, blanks, and calibration standards. Despite its ubiquity, water is often underappreciated as a critical contamination vector. In trace-level analysis, even sub-ppb concentrations of trace metals in water can lead to significant errors—masking analyte signals, inflating blank values, or leading to poor recoveries.

Even high-resistivity water systems (18.2 MΩ·cm) can fail to remove metal ions entirely. Common trace elements found in poorly maintained or overloaded systems include sodium (Na), calcium (Ca), potassium (K) from glassware, laboratory air, or ion exchange beds; Boron (B), silicon (Si) from borosilicate glass or reverse osmosis (RO) membranes; iron (Fe), zinc (Zn), copper (Cu) introduced via corroded stainless plumbing or fittings and lead (Pb), cadmium (Cd) from legacy building plumbing, stagnant water loops. The appearance of these contaminants can be intermittent, seasonal, or equipment-dependent—making them harder to diagnose without routine monitoring. For example, in one case, a laboratory using Type I water for lead testing discovered blanks containing 20–30 ppt Pb. Investigation revealed deterioration in a deionization (DI) resin cartridge, which had been used past its exchange capacity, leading to leaching of metals back into the system (4-6) (Table III).

The choice of water purification technologies can impact the potential contaminant found in the laboratory since each purification process is developed around a specific set of targets but may allow critical contaminants to pass through into the water (Table IV).

Plastics, Plasticizers, and Piping Materials and Inorganic Analysis

Plastic materials are ubiquitous in the inorganic laboratory environment. They are widely used as we have seen in water filtration and purification systems in addition to use as storage containers, storage tanks, values, tubing, and filters. Not all these plastics are inert to the inorganic environment and can leach particles or trace contaminants especially if exposed to changes in pH, temperature over periods of time. Many nonfluorinated plastics use metal catalysts while obviously the fluoropolymers contain fluorine. (Table V).

Organic Contaminants

Many water sources have the potential for a wide range of contamination issues as we have seen but most of the issues, we have spoken about to this point have been inorganic contaminants. But there are organic contaminants which can influence and effect inorganic analysis. Many plastics in the laboratory are made of polymers mixed with additives such as plasticizers, slip agents and coatings which due

TABLE IV: Water purification technologies and limitations

Technology	Removes	Limitations
Reverse osmosis (RO)	Particulates, most ions, organics	Boron, silicates, and some small ions poorly removed
Deionization (DI)	Charged species (cations/anions)	No removal of organics, bacteria, or neutral species
Ultrafilters (UF)	Bacteria, colloids	Do not remove dissolved ions
UV oxidation	Organics, microbes	Does not remove metals directly
Sub-boiling distillation	Volatile impurities and metals	High purity but slow and costly
Mixed-bed polishers	Finishing step for ultrapure systems	Must be replaced routinely to avoid leaching contamination

TABLE V: Plastic materials and elemental contaminants

Polymer	Typical Lab Use	Elements Detected	Max Total ppm
Polystyrene (PS)	Disposable Petri dishes, pipette trays	Na, Ti, Al	4
TFE	Tubing, ultratrace labware	Ca, Pb, Fe, Cu	19
Low-Density PE (LDPE)	Bottles, wash bottles, tubing	Ca, Cl, K, Ti, Zn	23
Polycarbonate (PC)	Filter holders, sample containers	Cl, Br, Al	85
Polymethyl pentene (PMP)	Volumetric labware	Ca, Mg, Zn	178
FEP	High-purity containers and tubing	K, Ca, Mg	241
Borosilicate glass	Glassware, beakers, flasks	Si, B, Na	497
Polypropylene (PP)	Centrifuge tubes, beakers, containers	Cl, Mg, Ca	519
High-Density PE (HDPE)	Bottles, drums, container liners	Ca, Zn, Si	654

to their nature can produce interferences for inorganic analyses. Plastic products can also shed plastic particles which interfere with inorganic analyses (Table VI).

These additives, while essential for the functionality and durability of polymers, can leach into reagents, blanks, and samples under common laboratory conditions. Studies have demonstrated that phthalates like di(2-ethylhexyl) phthalate (DEHP), found in PVC and pipette tips, can migrate into aqueous samples, especially when exposed to solvents or elevated temperatures. This leaching can lead to matrix modifications that suppress or enhance ionization in techniques such as ICP-MS, resulting in skewed calibration curves or inaccurate quantification. Similarly, thermal desorption analyses have revealed the diffusion of plasticizers from labware into solution phases, while stabilizers such as butylated hydroxytoluene (BHT) and flame retardants can contribute to complex polyatomic interferences in the plasma. Most notably, inorganic contaminants like antimony—used as

TABLE VI: Plastic types and organic contaminants

Polymer	Typical Lab Use	Polymer Additives / Organic Contaminants	Typical Organic Contaminant Levels
Polystyrene (PS)	Disposable Petri dishes, pipette trays	Styrene monomer, antioxidants	Styrene: <1 ppm
TFE	Tubing, ultratrace labware	Perfluorinated additives (minimal organics)	Negligible
Low-Density PE (LDPE)	Bottles, wash bottles, tubing	Antioxidants, slip agents, UV stabilizers	BHT: 0.1–1 ppm
Polycarbonate (PC)	Filter holders, sample containers	Bisphenol-A (BPA), flame retardants	BPA: 0.01–0.2 ppm
Polymethyl pentene (PMP)	Volumetric labware	Antioxidants, processing aids	0.1–1 ppm
FEP	High-purity containers and tubing	Fluoropolymer stabilizers (minimal organics)	Negligible
Borosilicate glass	Glassware, beakers, flasks	Trace organics from alkali leaching	Trace siloxanes
Polypropylene (PP)	Centrifuge tubes, beakers, containers	Phthalates, slip agents, antioxidants	Phthalates: 0.1–5 ppm
High-Density PE (HDPE)	Bottles, drums, container liners	Antioxidants, plasticizers	BHT, Irganox: 0.5–2 ppm

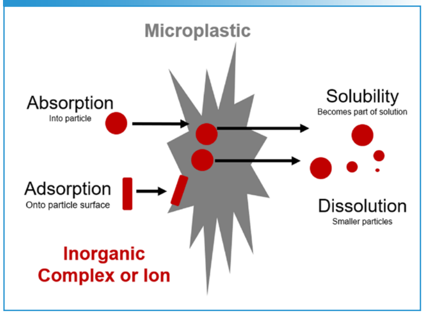


FIGURE 1: Microplastic and inorganic complex or metal interactions.

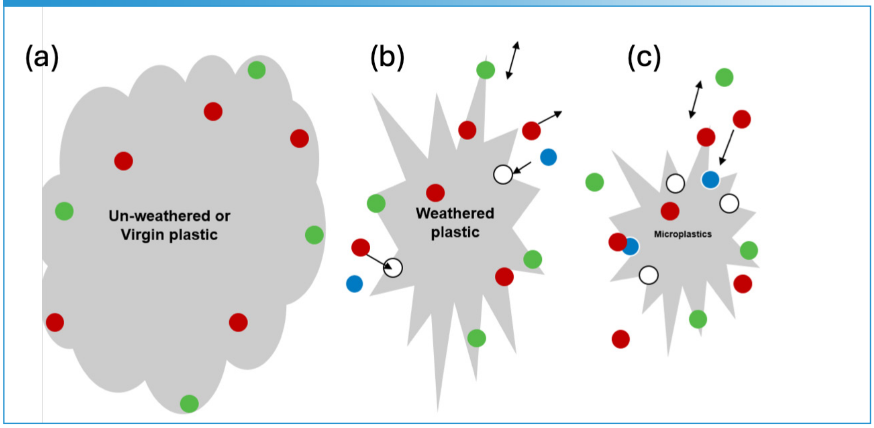


FIGURE 2: Weathering and Contamination processes of plastic particles. (a) Unweathered plastic polymer with embedded additives: (red) inorganic, or (green) organic plasticizers. Few sites for binding. (b) Weathered plastic allows for the release of chemicals (red & green) and opens binding sites (white), which allows binding of metals (red), and microbes (blue). (c) Microplastic particles continue to release and bind chemicals and attract microbes into biofilms, which can also bind metals and other chemical compounds.

a catalyst in polyethylene terephthalate (PET) plastics—have been shown to leach into bottled water, raising Sb levels well above background and potentially masking or mimicking trace-level target analytes. These findings emphasize the critical need to scrutinize not only the elemental purity of reagents and water but also the composition of the labware in contact with analytical systems, especially in ultratrace applications.

Dangers of Microplastics

A newer danger to health and safety has grown in interest over the last five years – microplastics. Microplastics are minute particles of plastics shed from plastic articles or the breakdown products of deteriorating plastic components. The dangers posed by microplastics have been undergoing increased study and are a great cause for health concerns in the scientific community. Another avenue to be discussed is the danger of microplastics in the laboratory setting and how they might impact analyses.

The shedding of micro- and nanoplastics from laboratory plasticware represents a subtle but growing threat to the accuracy and reliability of inorganic trace elemental analysis. As analytical detection limits reach into the parts-per-trillion range, even nanogram quantities of foreign material can disrupt results. Laboratory plastics—such as polyethylene (PE), polypropylene (PP), and polyvinyl chloride (PVC)—can degrade under common lab conditions including friction, acid exposure, ultraviolet (UV) light, and heat. This degradation leads to the release of micro- and nanoscale particles, often invisible to the naked eye, which can carry both inherent polymer contaminants and adsorbed environmental metals into critical analytical workflows (Figure 1).

These micro and nanoplastic particles can be a substrate for chemical adsorption to the particle surface by contaminants or may carry inks that have been adsorbed to the plastic surface. The metal or chemical species can also be absorbed by the particle allowing for later release as the particles enter an environment which allows for dissolution or increased solubility such as an in a situation of an acidic reagent or aqueous sample preparation.

Microplastics can act as dynamic reservoirs for trace elements in laboratory and environmental systems, particularly when influenced by surface weathering, pH conditions, and microbial colonization. (7) Pristine plastics exhibit low sorption potential for metals due to their hydrophobic, nonpolar surfaces. However, environmental and laboratory weathering—through UV exposure, oxidation, or mechanical abrasion—creates surface roughness and introduces polar functional groups that dramatically enhance metal-binding capacity. This effect is amplified in the presence of biofilms, where microbial extracellular polymeric substances (EPS) serve as additional chelating agents, concentrating metals like lead, zinc, cadmium, and copper on the plastic surface. Sorption behavior is also highly pH-dependent: at low pH, surface protonation suppresses metal adsorption, while neutral to slightly basic pH (6–8) enhances uptake, especially for cationic metals. These findings underscore the complex interplay between physical degradation, microbial activity, and chemical speciation in determining how microplastics influence trace metal distribution and behavior in analytical and natural systems. The weathering and contamination processes of plastic particles is exemplified in Figure 2.

Studies such as those by Mintenig and associates (8) and Lear and coauthors (9) have demonstrated that microplastics from common laboratory materials—including sample tubes, tubing, and filtration systems—can contaminate ultrapure water systems and blanks. These plastic particles may introduce exogenous elements such as heavy metals, zinc, aluminum, or antimony (used as catalysts or stabilizers in polymer manufacture) and organic residues like phthalates or bisphenol-A. Furthermore, nanoplastics can act as carriers for trace metals by adsorbing analytes onto their surface, leading to under-recovery or misidentification during ICP-MS analysis (10,11). As noted by Koelmans and associates (12), these microplastic-derived contaminants can mimic sample-related species or alter plasma behavior, complicating interpretation. In high-purity laboratories, where every source of contamination must be scrutinized, the integrity of plasticware is now recognized as a critical component of quality assurance, especially in cleanroom or ultra-trace environments. The potential contamination associated with different polymer materials is shown in Table VII.

A part of the danger of microplastics comes from their chemical composition but another part of the danger of contamination comes from their structure and ability to become a substrate for contamination to accumulate like complexes, dust, or microbes. Metals can accumulate in microplastics through four primary methods, the first as a component of their polymer structure, process formation, or in the form of chemical additives. The second method is acting as a site for electrostatic interactions between charged surfaces and ionized metals. In weathered plastics or in certain plastic additives there can be complexing with additives or chemical groups like carbonyl and carboxyl groups to attach metal substrates. Finally, biofilms can accumulate on the surface of plastics becoming chelators or exchange surfaces changing metal affinities.

TABLE VII: Contamination issues associated with different polymer materials

Source / Component	Polymer Type	Mechanism of Shedding	Contamination Risk
Polypropylene centrifuge tubes	Polypropylene (PP)	Friction, aging, autoclaving	Nanoplastics, Zn, Ca, organics
PE/PP pipette tips	Polyethylene (PE), Polypropylene (PP)	Mechanical wear, extrusion defects	Microplastic particles, organics
Plastic wash bottles	Polyethylene (PE)	Squeezing, chemical exposure	Shedding of PE particles, Zn
Tubing (PVC, silicone, PE)	PVC, PE, Silicone	Flexing, pressure stress	Plasticizers, microplastic fragments
Sample containers (LDPE, HDPE)	HDPE, LDPE	Long-term contact, temperature cycling	Additive leaching, PE fragments
Filter housings & capsules	Polyethersulfone (PES), Nylon	Filter membrane degradation	Membrane particles, adsorbed metals
RO/DI storage tanks	PE, PP	UV exposure, long residence time	Leaching of PE fragments, biofilms
Gloves (nitrile, latex, vinyl)	Nitrile, Vinyl	Abrasion, particle sloughing	Synthetic particles, trace organics
HEPA-filtered air (fibers)	Synthetic fiber residues	Airborne fiber fallout	Deposits on surfaces and vessels
Packaging films, plastic liners	Polyethylene, Polypropylene	Tearing, static charge attraction	Particles from liner residues

Missing the Microbes?

Microplastics not only pose chemical contamination risks in the laboratory but also serve as ideal substrates for microbial colonization and biofilm development. Due to their high surface area, hydrophobic nature, and persistent structure, plastic particles—particularly polyethylene (PE), polypropylene (PP), and polyvinyl chloride (PVC)—are prone to rapid microbial attachment when exposed to moist or aqueous environments.

Studies have shown that microplastics can harbor diverse microbial communities, including environmental bacteria, fungi, and even pathogenic species. In aquatic and laboratory systems alike, these particles function as "microbial rafts," enabling transport and proliferation of biofilm-forming organisms (13). Once established, these biofilms can bind trace metals from the surrounding environment, creating localized zones of elevated elemental concentration and posing a risk to trace-level inorganic analysis. Further, microbial metabolism within these biofilms can alter redox conditions, solubilize otherwise inert elements, or generate metabolic byproducts that interfere with techniques like ICP-MS. Recent research has extended this concern into controlled environments: Li and co-authors (14) demonstrated that polypropylene and polyethylene microplastics introduced into high-purity water systems supported dense microbial biofilms within days, even in filtered and UV-treated loops. These findings emphasize the importance of controlling both physical and biological contamination from microplastic residues in ultratrace laboratories.

Microorganisms—including bacteria, fungi, and microbial byproducts—can have surprising and disruptive impacts on inorganic analytical techniques such as ICP-optical emission spectoscropy (OES), ICP-MS, and ion chromatography (IC) (15–17). These effects are especially relevant in high-sensitivity laboratories focused on trace element detection, where even minor contamination or signal suppression can compromise results. Biofilms on water systems can accumulate metals like iron, copper, manganese, and zinc which can shed into water plants leading to signal spikes, drift, and false positives. Microbes can metabolize compounds producing compounds that reduce recoveries. Organic acids, chelators (such as siderophores), and exopolysaccharides can complex or sequester metals, altering their speciation or preventing proper ionization. Some microbes' oxide, reduce or

Table VIII: Microbial species contamination and inorganic techniques

Microbial Species / Group	Type/Form	Contamination Mechanism	Inorganic Disruption Pathways	Affected Techniques
***Pseudomonas* spp.**	Biofilm-forming gram-negative bacteria	Biofilms in UPW systems, metal uptake and chelation via siderophores	Chelation of Fe, Cu, Zn; pH shifts; metal ion retention	ICP-MS, IC, ICP-OES
***Bacillus* spp.**	Spore-forming bacteria	Spore particulate contamination, phosphate, and sulfate production	Sulfate/phosphate precipitation; matrix complexity	ICP-OES, IC
***Staphylococcus* spp.**	Skin-associated bacteria	Shedding and secretion of salts, skin oils	Surface contamination, spectral background	ICP-MS, Gravimetry
***Sphingomonas* spp.**	Waterborne biofilm bacteria	Biofilm formation in tubing; production of organophosphates	Sequestration of phosphate and chelation of trace metals	ICP-MS, IC
***Aspergillus* spp.**	Airborne fungal spores	Spores shed in lab air; biosorption of Al and Si	Al and Si release; fungal enzymes bind trace metals	ICP-MS, Gravimetry
***Penicillium* spp.**	Mold spores and hyphae	Spore deposition in lab; produces chelators and mycotoxins	Chelation of Zn, Cu, Fe by mycotoxins; pH alteration	ICP-OES, IC, Weighing
***Cladosporium* spp.**	Airborne environmental fungus	Environmental spores: binds transition metals	Binds Fe, Mn, Zn; alters sample mass	ICP-MS
***Nocardia* spp.**	Actinobacteria, filamentous	Biofilm-mediated degradation of lab plastics, metal adsorption	Metal leaching from plastic; sorption of analytes	ICP-MS, Cleanroom studies
***Mycobacterium* spp.**	Slow-growing actinobacteria	Persistent in filters, slowly releases fatty acids and metals	Modifies TOC and adsorption profile of filters	ICP-MS, Spectrophotometry
***Synechococcus* spp.**	Photosynthetic cyanobacteria	Releases organic acids and produces nitrates/nitrites	Interferes with anion chromatography; alters pH, ionic strength	IC, ICP-MS
***Chlorella* spp.**	Green algae	Matrix effect from pigments and cell debris; complexation of ions	Matrix effects in ICP-MS from chlorophyll and silica	ICP-MS, IC
***Nitrosomonas* spp.**	Nitrifying bacteria	Produces nitrite, nitrate via ammonia oxidation	Increases nitrate background in IC; alters N speciation	IC, ICP-MS
***Thiobacillus* spp.**	Sulfur-oxidizing bacteria	Produces sulfuric acid and sulfates via H_2S oxidation	Increases sulfate background in IC and ICP-MS	ICP-OES, IC
***Saccharomyces* spp.**	Yeast	Produces ethanol and acetates; acidic conditions alter speciation	Shifts solution pH; interferes with ICP sensitivity	ICP-MS, pH-sensitive assays
Escherichia coli	Gut-associated gram-negative bacteria	Produces endotoxins and polyamines; adsorbs Mg, Ca, Fe	Sequesters divalent cations; causes baseline drift	ICP-MS, IC, pH-sensitive prep
Viral contamination	Virus particles	Nucleic acid contamination alters conductivity; binds trace metals	Increases background signal in ICP-MS, alters ion matrix	ICP-MS, Conductivity, TOC

methylate metals transforming them to different forms which can impact speciation. The particles themselves can clog nebulizers or create matrix effects. Some microbes can produce IC analyte targets like nitrates, sulfates or phosphates and invalidate IC results (Table VIII).

Polyatomic and Isobaric Interference in Contaminated Systems

Spectral interference, particularly polyatomic and isobaric overlap, remains one of the most difficult analytical artifacts in ICP-MS. Polyatomic interferences arise when multiple atoms or molecules combine in the plasma to form a species with the same mass-to-charge ratio as the analyte of interest. Common examples include $ArCl^+$ overlapping with As^+ at *m/z* 75, or ArO^+ overlapping with Fe^+ at *m/z* 56. These interferences become even more complex in contaminated environments.

TABLE IX: Possible ICP-MS interferences or contributing contamination

Interfered Analyte	Interference Ion	Possible Contamination Sources
As^+ (m/z 75)	$^{40}Ar^{35}Cl^+$	HCl in lab reagents, microplastic chlorinated residues possible
Fe^+ (m/z 56)	$^{40}Ar^{16}O^+$ (ArO^+)	Laboratory air, water (dissolved O_2), possible microbial metabolism
Mn^+ (m/z 55)	$^{40}Ar^{15}N^+$, $^{39}K^{16}O^+$	Possible microbial byproducts (nitrifying bacteria), residual NO_3^+ in water
V^+ (m/z 51)	$^{35}Cl^{16}O^+$, PO^+	Microbial phosphates, degraded plasticizers, lab detergents
S^+ (m/z 32)	$^{16}O_2^+$, $^{14}N^{18}O^+$	Microbial activity, sulfur-containing organics in microplastics. O_2/NO species; corrected via O_2 mass-shift to SO^+ (48).
Ti^+ (m/z 48)	$^{48}Ca^+$, $^{32}S^{16}O^+$, $^{36}Ar^{12}C^+$	Possible contamination from chlorinated plastics (PVC), lab air particles, bottle cap liners
Ti^+ (m/z 52)	$^{52}Cr^+$	Cr in tap water, leached from lab valves or acid residue

When microplastics are introduced—either from environmental contamination or from labware—the resulting organics and additives can combust or incompletely ionize, producing complex polyatomic ions (such as $C_2H_3^+$, CCl^+, and CN^+) that affect lighter elements such as Ti, V, Cr, and Mn. Moreover, microbial contamination introduces nitrogen-, sulfur-, and phosphorus-containing compounds which can result in overlapping species such as NO^+, PO^+, or SO^+ (Table IX).

Furthermore, bacterial biofilms and organic-rich matrices can modulate plasma temperature, increasing the likelihood of incomplete ionization or new adduct formation. This variability means that even well-tuned instruments can yield poor recoveries or inaccurate readings if contamination is not tightly controlled.

The Critical Role of Certified Reference Materials in Trace Analysis

Certified reference materials (CRMs) are foundational to high-accuracy elemental analysis and regulatory compliance, especially in ultratrace work by ICP-OES, ICP-MS, and related techniques. However, not all CRMs are created equal. The most reliable CRMs are produced using multiple, orthogonal methods of verification—typically including gravimetric preparation, wet chemical analysis (for example, titration or complexometry), and instrumental confirmation via ICP-OES or ICP-MS. This multimodal validation ensures not only that the target analyte concentration is accurately reported, but that interferences and impurities are fully characterized.

CRMs of the highest caliber begin with the highest-purity raw materials—either commercially available at semiconductor or trace analytical grade, or further purified in-house through recrystallization, distillation, or sublimation. These materials must be free from trace-level interferences that could compromise the final value assignment. The value of a CRM increases significantly when elemental impurities are not only measured, but certified, allowing analysts to subtract known interferences or adjust their calibration accordingly.

Equally critical is the purity of the reagents—particularly water and acids—used in CRM production. ASTM Type I water systems must be routinely validated for microbiological, organic, and elemental contamination. Ultrapure nitric and hydrochloric acids used in CRM dissolution or dilution should be sub-boiling distilled and verified by ICP-MS to ensure blank levels are below the analyte reporting limits. Even the materials used for packaging and storage—bottles, caps, seals—should be pre-screened to avoid leaching of metals, plasticizers, or microbial residues.

By controlling and validating each input—chemical, procedural, and physical—CRM producers can provide standards that not only meet ISO 17034 and ISO 17025 requirements (18,19), but also inspire confidence in the laboratory data they support. In an era of single-digit parts-per-trillion reporting, the difference between a good CRM and a great one can define the reliability of an entire analytical method.

CRMs are essential to validating accuracy and controlling for contamination. However, their effectiveness depends on their appropriate application. Laboratories should:

- Select CRMs that match the matrix and concentration of their samples.
- Use first and second source CRMs from independent providers to verify accuracy.
- Regularly qualify reference materials against in-house blanks and previously validated standards.
- Avoid diluting CRMs with low-purity acids or water, which can introduce bias.
- Document lot numbers, storage conditions, and expiration dates as traceability tools.

Importantly, CRMs should not be assumed infallible. If repeated contamination appears in blank-corrected CRM runs, the contamination may lie in dilution, sample preparation, or the laboratory environment—not the standard.

Best Practices and Conclusion

Maintaining trace-level integrity in elemental analysis requires discipline, awareness, and a strong contamination control program. Key best practices include:

- Always use reagent-grade or ultrapure acids and ASTM Type I water.
- Validate all labware for elemental blank levels before use.
- Limit use or type of plastics where metals, microplastics, or plasticizers may leach.

- Control airflow and human presence in clean areas.
- Implement microbiological testing of UPW systems and sample prep areas.

The invisible nature of most contamination makes it easy to overlook—but the cost to data integrity can be enormous. As detection capabilities become increasingly sensitive, so too must our understanding of these silent saboteurs. Only through active control, monitoring, and education can laboratories ensure the validity and trustworthiness of their elemental data.

References

(1) Bruland, K. W.; Lohan, M. C. Controls of Trace Metals in Seawater. In *Treatise on Geochemistry*; Elderfield, H., Ed.; Elsevier: Oxford, 2003; Vol. 6, pp 23–47. https://doi.org/10.1016/B0-08-043751-6/06105-3

(2) Sun, D.; Sealy, D.; Truong, D.; Konforte, D. Barcoded Labels as Potential Source of Zinc Contamination in Trace Metal Testing. *J. Mass Spectrom. Adv. Clin. Lab.* 2025, 36, 9–10. https://doi.org/10.1016/j.jmsacl.2025.02.003

(3) A Common Sense Laboratory Guide to Reducing Errors and Contamination in ICP and ICP-MS Analysis. *Spectrosc. Suppl.* 2010, Special Issues-11-01-2010. https://www.spectroscopyonline.com/view/common-sense-laboratory-guide-reducing-errors-and-contamination-icp-and-icp-ms-analysis

(4) ASTM International. *ASTM D1193-06: Standard Specification for Reagent Water*; ASTM International: West Conshohocken, PA, 2006.

(5) Inorganic Ventures. Trace Analysis Guide: Environmental Contamination. https://www.inorganicventures.com/trace-analysis-guide/environmental-contamination (accessed Sept 24, 2025).

(6) U.S. Environmental Protection Agency. *Method 200.8, Revision 5.4: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma–Mass Spectrometry (ICP-MS)*; Office of Research and Development: Cincinnati, OH, 1994. https://www.epa.gov/sites/default/files/2015-08/documents/method_200-8_rev_5-4_1994.pdf

(7) Liu, S.; Huang, J.; Zhang, W.; Shi, L.; Yi, K.; Yu, H.; Zhang, C.; Li, S.; Li, J. Microplastics as a Vehicle of Heavy Metals in Aquatic Environments: A Review of Adsorption Factors, Mechanisms, and Biological Effects. *J. Environ. Manage.* 2022, 302, 113995. https://doi.org/10.1016/j.jenvman.2021.113995

(8) Mintenig, S. M.; Int-Veen, I.; Löder, M. G. J.; Primpke, S.; Gerdts, G. Identification of Microplastic in Effluents of Wastewater Treatment Plants Using Focal Plane Array-Based Micro-Fourier-Transform Infrared Imaging. *Water Res.* 2017, 108, 365–372. https://doi.org/10.1016/j.watres.2016.11.015

(9) Lear, G.; Kingsbury, J. M.; Franchini, S.; Gambarini, V.; Maday, S. D. M.; Wallbank, J. A.; Weaver, L.; Pantos, O. Plastics and the Microbiome: Impacts and Solutions. *Environ. Microbiome* 2021, 16 (1), 2. https://doi.org/10.1186/s40793-020-00371-w

(10) Hildebrandt, L.; Nack, F. L.; Zimmermann, T.; Pröfrock, D. Microplastics as a Trojan Horse for Trace Metals. *J. Hazard. Mater. Lett.* 2021, 2, 100035. https://doi.org/10.1016/j.hazl.2021.100035

(11) Binda, G.; Spanu, D.; Monticelli, D.; Pozzi, A.; Bellasi, A.; Bettinetti, R.; Carnati, S.; Nizzetto, L. Unfolding the Interaction between Microplastics and (Trace) Elements in Water: A Critical Review. *Water Res.* 2021, 204, 117637. https://doi.org/10.1016/j.watres.2021.117637

(12) Koelmans, A. A.; Besseling, E.; Shim, W. J. Nanoplastics in the Aquatic Environment. Critical Review. In *Marine Anthropogenic Litter*; Bergmann, M., Gutow, L., Klages, M., Eds.; Springer: Cham, 2015; pp 325–340. https://doi.org/10.1007/978-3-319-16510-3_12

(13) Zettler, E. R.; Mincer, T. J.; Amaral-Zettler, L. A. Life in the "Plastisphere": Microbial Communities on Plastic Marine Debris. *Environ. Sci. Technol.* 2013, 47 (13), 7137–7146. https://doi.org/10.1021/es401288x

(14) Li, J.; Liu, H.; Chen, J. P. Microplastics in Freshwater Systems: A Review on Occurrence, Environmental Effects, and Methods for Microplastics Detection. *Water Res.* 2018, 137, 362–374. https://doi.org/10.1016/j.watres.2017.12.056

(15) Wang, Q.; Zhang, Y.; Zhou, Y.; Li, Z.; Wang, J.; Chen, H. Effects of Biofilm on Metal Adsorption Behavior and Microbial Community of Microplastics. *J. Hazard. Mater.* 2022, 424, 127340. https://doi.org/10.1016/j.jhazmat.2021.127340

(16) Jing, R.; Kjellerup, B. V. Biogeochemical Cycling of Metals Impacted by Microbial Mobilization and Immobilization. *J. Environ. Sci.* 2018, 66, 146–154. https://doi.org/10.1016/j.jes.2017.04.035

(17) Lee, Y.-W.; Ong, S.-K.; Sato, C. Effects of Heavy Metals on Nitrifying Bacteria. *Water Sci. Technol.* 1997, 36 (12), 69–74. https://doi.org/10.2166/wst.1997.0432

(18) International Organization for Standardization. *ISO 17034:2016: General Requirements for the Competence of Reference Material Producers*; ISO: Geneva, Switzerland, 2016.

(19) International Organization for Standardization/International Electrotechnical Commission. *ISO/IEC 17025:2017: General Requirements for the Competence of Testing and Calibration Laboratories*; ISO: Geneva, Switzerland, 2017.

Patricia Atkins is the Senior Global Product Manager for Analytical Reference Materials for Spex-NSI-HPS-Chiron having started as a R&D Scientist at Spex over 15 years ago. She is a graduate of Rutgers University in NJ and was laboratory supervisor for Ciba Specialty Chemicals. Patricia conducted research and managed an air pollution research group within Rutgers University Civil and Environmental Engineering Department. She has been involved with many industry focused advisory & regulatory organizations including AOAC, ACIL, Emerald Conference, Cannabis Science Conference, ASTM and NACRW. She is a frequent presenter at conferences and a published author with her work appearing in various journals and trade publications including Spectroscopy, LCGC and Cannabis Science and Technology where she was a columnist for analytical issues in cannabis testing.