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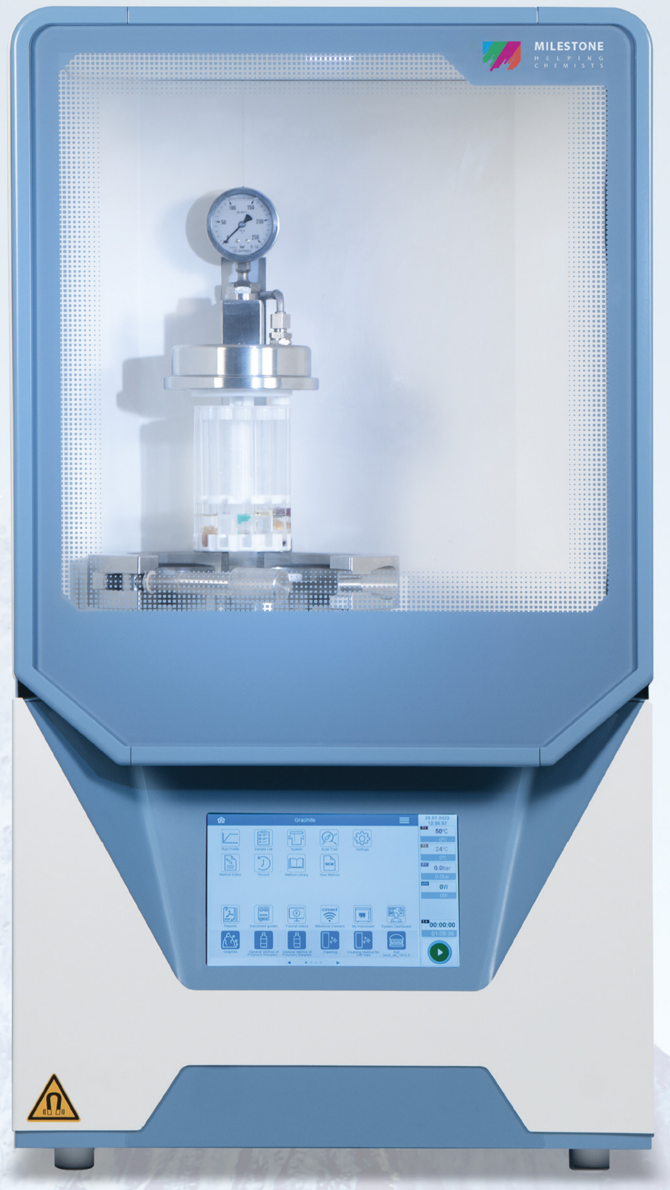

Milestone's new **ultraWAVE 3** is the latest generation of SRC technology. It provides all the benefits of its revolutionary predecessor, while taking sample prep productivity and performance to the next level. Several aspects of the system, such as no batching, less operating time, and faster digestions, reduce turnaround time and increase lab efficiency for elemental analysis.

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UPFRONT

Heavy Metal Fan?

ICP-MS confirms presence of metal particles in legal and illegal cannabis liquid vapes – raising alarm bells for more rigorous testing protocols

By Georgia Hulme

Since recreational cannabis was legalized in Canada in 2018, cannabis liquid vapes have risen in popularity. Although strict regulations require testing for chemical contaminants after the final step in the production process – during which contaminants could have been introduced – prolonged exposure to metal parts of the atomizer is often an overlooked hazard.

The regulations require testing occurs after the final step in the production process during which the contaminants could have been introduced or could be concentrated, whichever is later.

To dig into the issue a little deeper, researchers from Canada used a handful of analytical techniques to analyze metal contents in cannabis vape liquids (1). The total metal contents of 21 illegal and 20 legal electronic vaping devices were analyzed using inductively coupled plasma mass spectrometry (ICP-MS). We spoke to lead author, Zuzana Gajdosechova, who highlights that ICP-MS enabled "a low limit of detection and elemental specificity while successfully handling such a complex matrix."

Scanning electron microscopy with energy-dispersive X-ray spectroscopy and laser ablation ICP-MS were then used to image and identify the presence of metal particles. "The former method required minimal sample preparation and provided immediate visual confirmation, and the latter technique allowed us to access particles embedded deeper within the matrix," says Gajdosechova.

The study revealed that the metal particles may have originated from components in contact with the vape liquid. One legal and six illegal vapes contained high levels of lead, hugely exceeding the generally accepted limits of 0.5 µg g–1. The magnitude of exceedance varied between the analytes, but nickel levels were 900 times above the generally accepted tolerance limit in several of the illegal samples. Copper, zinc, and manganese were also present in metal particle form. Concentrations of cadmium, mercury, and arsenic were below the established limits used for cannabis products.

Going forward, the authors plan to investigate the specific source of metal contamination. However, Gajdosechova notes the opportunities for conducting cannabis research in Canada relative to many other countries, and hopes that her research will spark further studies of vape devices – to not only better understand their metal composition, but for the development of future vape construction standards.

Image Credit: Jeff W / unsplash.com

IN MY VIEW

Alternative Atomic Spectroscopy

Solution-cathode glow discharge is the answer to atomic spectroscopy's most frustrating traits.

Michael Webb is Associate Professor, Department of Chemistry, at the University of North Carolina Wilmington, USA.

> *"ICP torches of today may not look much different from Fassel's torch of five decades ago, but every step from sample introduction to signal processing has evolved."*

Atomic spectrometry is not a new field – in fact, its timeline can be measured in decades. It's been 19 decades since Henry Fox Talbot linked flame emission spectra to compounds of particular elements. Sixteen decades since Robert Bunsen and Gustav Kirchhoff began extensive studies of atomic absorption and atomic emission spectra. Ten since Niels Bohr linked atomic spectra to atomic structure. Six since Alan Walsh developed flame atomic absorption spectroscopy (FAAS) into a quantitative analytical technique. Five since Velmer Fassel and Stanley Greenfield introduced inductively coupled plasma optical emission spectrometry (ICP-OES). Even Sam Houk's initial ICP mass spectrometry (MS) work is nearly four decades old.

Despite this long history, ICP-OES and ICP-MS remain the preferred methods for many analyses, and outsiders to the field could be forgiven for thinking atomic spectroscopy was stagnant. That would overlook enormous progress. ICP torches of today may not look much different from Fassel's torch of five decades ago, but every step from sample introduction to signal processing has evolved, particularly through work that continues into identifying and reducing or correcting matrix interferences. Other authors would be better suited to bringing attention to this research. My interests are in a more radical direction – developing alternative atomic spectrometry sources.

The strengths of ICP-OES are well known – trace-level detection limits, long linear range, mild matrix effects, and good precision. Still, it has disadvantages. It requires around 15 L/min of Ar, 1.5 kW, water cooling, and a high-resolution spectrometer. Most sample introduction uses nebulizers that can be prone to clogging with high solid samples. ICP is not practical for on-site industrial or remote environmental monitoring. Solution-cathode glow discharge (SCGD)–OES, on the other hand, is well suited for such applications.

SCGD is one of a growing number of atomic spectrometry sources operating on similar principles. All owe their roots to Cserfalvi and Mezei's electrolyte cathode atmospheric glow discharge (ELCAD), which was introduced in 1993 but got surprisingly little attention at first. In addition to SCGD, sources related to ELCAD now include liquid sampling atmospheric pressure glow discharge, direct current atmospheric pressure glow discharge in contact with a flowing liquid cathode, and alternating current electrolyte atmosphere liquid discharge. These instruments have now been applied to tea, mineral water, brines, tuna fish, aquatic plant matter, oyster tissue, coal fly ash, groundwater, hepatitis-B vaccine, lake water, soil leachates, spruce needle leachates, colloidal silica, zirconium alloys, and simulated natural water. I'll focus on SCGD, but these other sources have similar advantages.

SCGD does not use any compressed gases, requires only about 70 W to maintain the plasma, does not require cooling, and can use a compact low-resolution spectrograph. 'Solution cathode' refers to the sample, which acts as one electrode of an atmospheric pressure glow discharge that is 3 mm tall and about 1 mm in diameter. With the sample directly in contact with the discharge, there is no nebulizer to clog. The power demands are so low that the instrument could conceivably run on batteries. ICP instruments use high resolution (~10 pm bandpass) to avoid spectral interferences, but SCGD has a relatively sparse spectrum and so can use lower resolution (~350 pm bandpass), while still avoiding most spectral interferences. A small, inexpensive spectrograph can accomplish this resolution while simultaneously covering a wide spectral range to allow multielemental analysis. SCGD-OES is capable of detection limits and precision similar to ICP-OES.

Of course, SCGD has its shortcomings. Most notably, matrix effects are more severe than with ICP (although generally less severe than with FAAS). The inadequacy can largely be overcome using standard addition calibration, but external standard calibration would usually be preferable. Studies are ongoing to identify and reduce or correct matrix interferences in SCGD.

When considered on the scale of atomic spectroscopy, SCGD and related methods are still youngsters. With more attention? I believe they will mature and play a complimentary role to ICP.

IN MY VIEW

The Beginner's Guide to ICP-MS

You've convinced your boss that your laboratory absolutely needs a shiny new quadrupole-based, inductively coupled plasma mass spectrometry (ICP-MS) system for trace element analysis. Now what?

By Robert Thomas

There are a number of excellent commercial ICP-MS systems on the market – all with very similar specifications – so how do you choose the one that best fits your application needs? How do you go about comparing the different designs, hardware components, and performance factors, all of which are of critical importance in the decision-making process?

First, it's very important to decide what your objectives are, particularly if you are part of an evaluation committee. You can have more than one objective, but they must be clearly defined. Every laboratory's application demands are unique, so it is important to prioritize before you begin the evaluation process. Capability, usability and reliability are the areas that I feel require particular focus, so let's take a closer look.

The major reason that the trace element community was attracted to ICP-MS over 30 years ago was its extremely low multielement detection limits. Other multielement techniques, such as inductively coupled plasma optical emission spectrometry (ICP-OES), offered very high throughput but could not achieve ultratrace levels. Even though graphite furnace atomic absorption (GFAA) spectrometry offered much better detection capability than ICP-OES, it did not offer the sample throughput. In addition, GFAA was predominantly a single-element technique and was therefore impractical for carrying out rapid multielement analysis.

These limitations quickly led to the commercialization of ICP-MS as a tool for rapid ultratrace element analysis. However, there are certain areas where ICP-MS is weak. For example, dissolved solids for most sample matrices must be kept below 0.2 percent; otherwise it can lead to serious drift problems. So in applying ICP-MS to real world samples, it's important to be aware of how different instrumental designs handle these limitations. There are a number of common performance metrics that can be used to measure the capability of an ICP-MS, including:

- Detection limit
- Sensitivity
- Accuracy/Precision
- Long-term stability
- Dynamic range
- Interference reduction
- Sample throughput

Once again, the importance of each metric is dependent on your laboratory's application needs. Is detection limit performance at the top of your list? Or perhaps the instrument will be used to generate revenue, in which case sample throughput is of greater importance.

Analytical performance is clearly a very important consideration; however, the vast majority of instruments in use today are being operated by technician-level chemists, who may have some experience in the use of AA or ICP-OES, but in no way could be considered ICP-MS experts. Therefore, the usability aspects might be competing with performance capability as the most important selection criteria, particularly if the application does not demand the ultimate in detection limits. Even though usability is dictated by the expertise of the operator, there are some factors that need to be considered. They include, but are not limited to:

- Ease of use
- Routine maintenance
- Sampling accessory compatibility
- Installation requirements
- Technical support and training.

> *"The major reason that the trace element community was attracted to ICP-MS over 30 years ago was its extremely low multielement detection limits."*

Good instrument reliability is taken for granted nowadays, but it has not always been the case. When ICP-MS was first commercialized, the early instruments were a little unpredictable, and quite prone to breakdowns. However, as the technique became more mature, the quality of instrument components, and hence the reliability, improved. You should therefore be aware of the instrument components that are more problematic than others. This is particularly true when a brand new instrument has been introduced or a model has had a major redesign. In the life cycle of a newly designed instrument, the early years might be more susceptible to reliability problems than when the instrument is more mature.

One final point: it's very important that you talk to real users in your application field; their experience – and even failures – can also guide you. For further help, you could read my book (1)

Reference

Robert. J. Thomas, "Practical Guide to ICP-MS: A Tutorial for Beginners", (3rd Edition; ISBN 978-1—4665-5543-3, CRC Press, FL).

SOLUTIONS PAGE

How to Enhance Your Sample Prep Workflow to Optimize Elemental Analysis

Integrated sample prep workflow for higher efficiency

As the performance of atomic spectroscopy techniques for elemental analysis has improved over the past several decades, there has been a concurrent need for improvements in sample preparation. While the sample digestion step appropriately draws the most attention in the preparation process, there are other important steps in the sample preparation workflow that also impact the outcomes of the laboratory.

The "total workflow" approach to sample preparation evaluates and addresses ways to improve key aspects of elemental analysis, such as lab throughput, data quality, costs, and safety. The sample preparation workflow incorporates all the major steps that are required to prepare the sample for elemental analysis, such as acid purification, automated reagent addition, vessel handling, microwave digestion, sample filtering, and labware cleaning.

This approach offers practical advice for preventing workflow disruptions, such as incomplete digestions or sample contamination, which can prevent a laboratory from meeting its overall performance, cost, and safety goals.

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Understanding the fundamental importance of effective microwave digestion for elemental analysis sample preparation

High-purity acid expenses for elemental analysis labs can consume a significant portion of operational budgets. In addition, accidental contamination, and supply chain challenges for obtaining high-purity acids can put a lab's operations at risk. Sub-boiling distillation enables in-house acid purification leading to cost savings and ensure an uninterrupted analysis workflow.

The reagent addition, or "dosing," step of the sample preparation process has traditionally been a tedious and laborious task within elemental analysis labs, especially when it involves concentrated acids. Adding an automated reagent provides greater consistency, while removing operators from exposure to harmful acid fumes and freeing them to perform safer and more value-added tasks.

Single Reaction Chamber (SRC) microwave digestion technology provides a step-change in sample prep performance and capabilities for elemental analysis. In the context of workflow, SRC's benefits include higher overall sample throughput, faster digestions for difficult samples, and reduced labor requirements. By digesting all samples, regardless of type or acid chemistry, at the same temperature and pressure, it works to avoid incomplete digestions that can lead to mass and optical spectral interferences, sample reruns, and increased instrument downtime.

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The importance of clean chemistry tools for atomic spectroscopy

Using acid baths and microwave systems for cleaning sample prep vessels and other types of labware can have a substantial impact on an elemental analysis lab's throughput, workflow, and efficiency. Automated acid-steam cleaning keeps your microwave system focused on digesting samples and removes the tedium and hazards of hand-cleaning labware from your staff.

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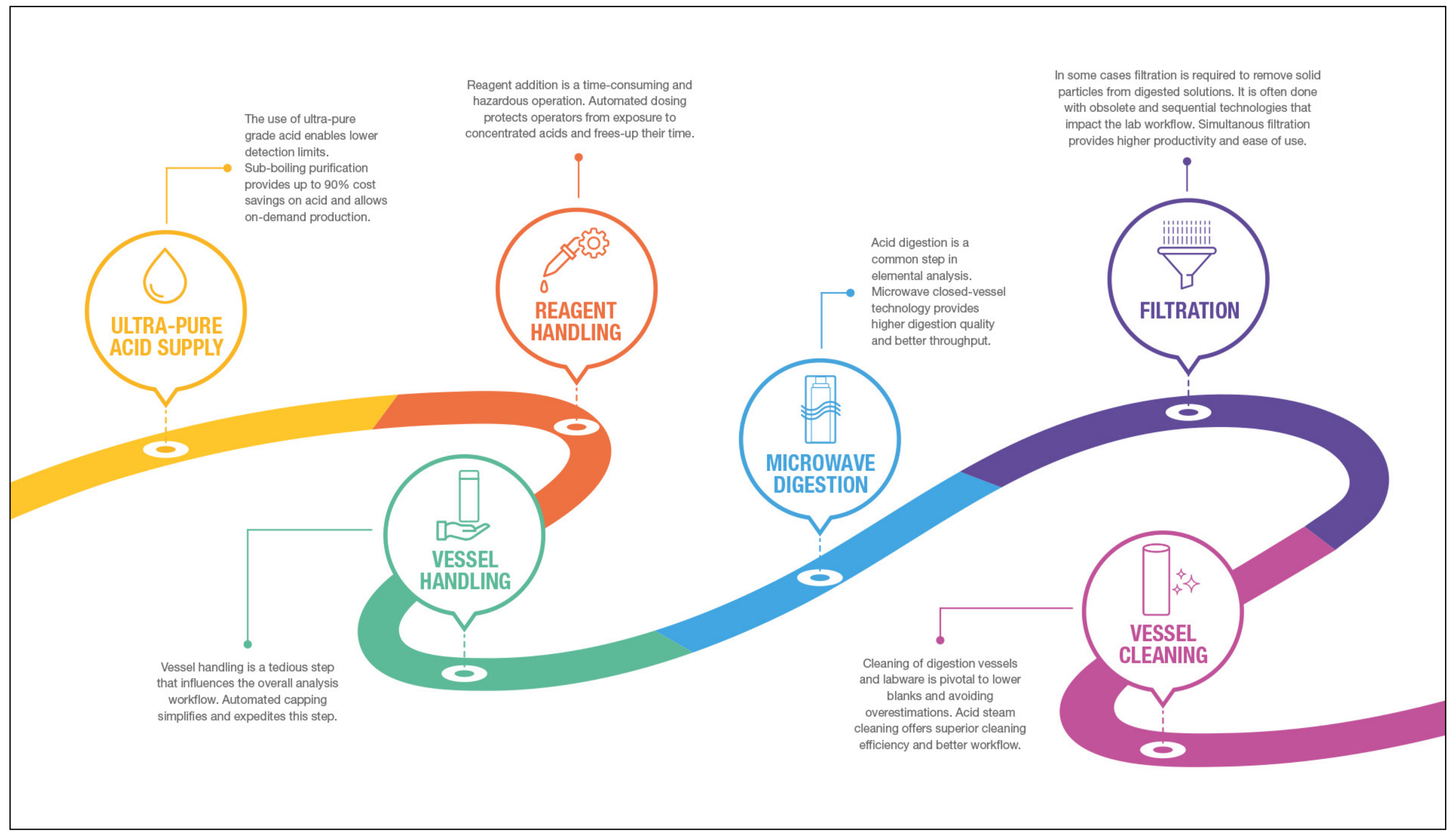

The use of ultra-pure grade acid enables lower detection limits. Sub-boiling purification provides up to 90% cost savings on acid and allows on-demand production.

Reagent addition is a time-consuming and hazardous operation. Automated dosing protects operators from exposure to concentrated acids and frees-up their time.

Vessel handling is a tedious step that influences the overall analysis workflow. Automated capping simplifies and expedites this step.

Acid digestion is a common step in elemental analysis. Microwave closed-vessel technology provides higher digestion quality and better throughput.

In some cases filtration is required to remove solid particles from digested solutions. It is often done with obsolete and sequential technologies that impact the lab workflow. Simultanous filtration provides higher productivity and ease of use.

Cleaning of digestion vessels and labware is pivotal to lower blanks and avoiding overestimations. Acid steam cleaning offers superior cleaning efficiency and better workflow.

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APPLICATION NOTE

Best Practices for Sample Preparation of Baby Food in Trace Metal Determination

An integrated, rugged, and greener sample preparation workflow for trace metal analysis

Food is the fuel for life, and keeping our body in balance requires a careful consideration of the quality and the quantity of our food choices. There are some elements that we must not ingest, such as arsenic, cadmium, lead, and mercury, which means it is critically important that they aren't present in foods – especially foods used to feed babies and infants (defined by the US Baby Food Safety Act of 2021 as "food intended for sale to children up to 36 months of age, including infant formula." To keep food safe for babies and infants, determining the concentrations of these elemental contaminants and increasing the information available about food composition are key.

According to the recent legislation, we must be able to measure concentrations of As, Cd, Hg, and Pb in levels from 2 to 15 µg/kg. Analytical chemists have developed powerful strategies for determining trace elements. Of course, modern instrumental methods, such as inductively coupled plasma mass spectrometry, are important for meeting these analytical demands. However, despite having proper instrumentation available, we need to strengthen the analyst's culture about working with trace concentrations and educate the community about contamination sources in typical analytical procedures. Here, sample preparation is a fundamental step for obtaining accurate and precise results in trace analysis. Some items to which we must pay close attention include:

- purity of reagents, as well as how they can be easily purified
- contamination and cleaning of laboratory materials
- sample preparation procedures that involve digesting high amounts of samples using low volumes of purified nitric acid
- analytical procedures with lower numbers of successive steps

All these points are covered in this e-book, where we demonstrate how optimized procedures can be successfully developed and applied to meeting the demands of a "closer to zero plan."

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SPOTLIGHT ON...

Technology

easyFILL Automated Reagent Dosing Station

Reagent addition is an inherent step of the sample preparation process that involves the handling of concentrated acids, leading to potential safety issues and time-consuming operation. easyFILL completely automates this step, saving operator time and reducing exposure to concentrated acids. It is fully compatible with commonly used concentrated acids, such as HCl, HNO_3, HF, H_2O_2.

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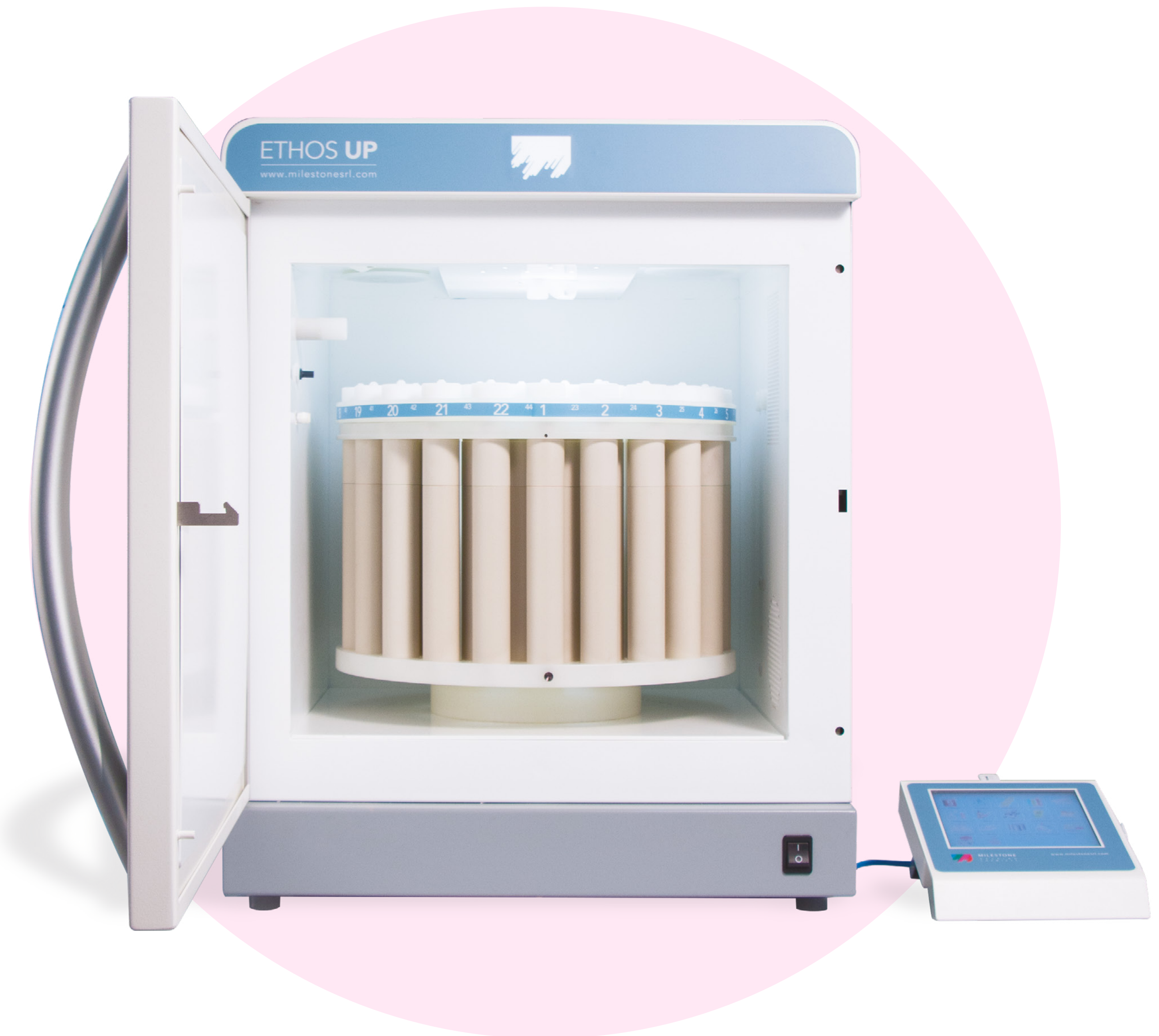

ETHOS UP Rotor-based Microwave Digestion System

ETHOS UP is the most advanced rotor-based system for microwave sample preparation ever manufactured. It offers a perfect integration of microwave hardware, user interface, and digestion sensors.

To complete the performance of the ETHOS, a suite of digestion rotors are offered to meet any digestion and throughout requirements for a wide selection of samples.

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New ultraWAVE 3 Single Reaction Chamber Microwave Digestion System

Thanks to its superior digestion capabilities that result from its higher temperature and pressure capabilities, ultraWAVE's unique SRC technology provides greater digestion efficiency.

The system's reduced handling and cleaning, as well as simultaneous sample processing, streamline the daily routine of the lab, increasing efficiencies.

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