



HOW TO USE A “TOTAL
WORKFLOW” SAMPLE PREP
APPROACH TO **OPTIMIZE**
ELEMENTAL ANALYSIS



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H E L P I N G
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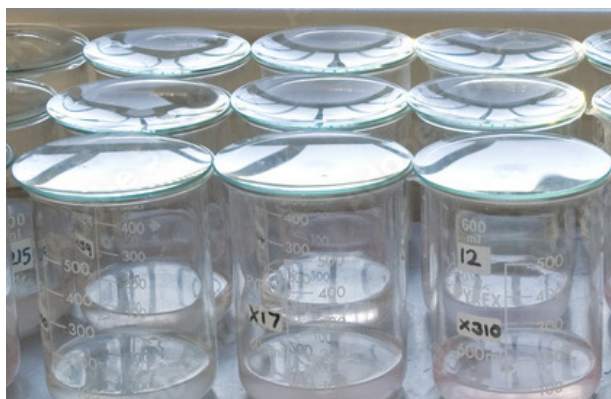
How to Use a “Total Workflow” Sample Prep Approach to Optimize Elemental Analysis

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1.

State-of-the-art elemental analysis workflow and related benefits

Over the years, laboratories have transitioned from flame atomic absorption (AA) and graphite furnace atomic absorption (GFAA) to ICP-OES and ICP-MS to increase productivity, improve data quality, and meet new regulations. At the same time, sample preparation has also evolved, moving from relatively rudimentary open-vessel digestion to advanced closed vessel digestion, marked by technologies like contactless temperature control and advanced vessel designs that allow for higher temperatures and pressures.



Heating block



Milestone ETHOS UP

The more recent introduction of the revolutionary Single Reaction Chamber technology moves the digestion process to a higher level. Overall,

these advances have led to improved digestion time, quality, blanks, and safety. 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, some of which may be unexpected in scope and scale. This ebook presents a “total workflow” approach to sample preparation and examines the ways to improve key aspects of elemental analysis such as lab throughput, data quality, costs, and safety via an optimization of the following steps:

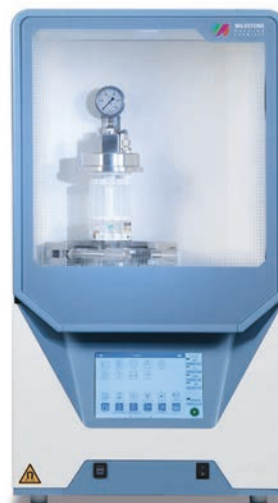
- Acid supply and quality
- Reagent addition
- Vessel handling
- Microwave digestion
- Sample filtering
- Labware cleaning

Just as importantly, it also offers practical advice for preventing workflow disruptions, such as incomplete digestions or sample contamination, that can prevent a laboratory from meeting their overall performance, cost, and safety goals. Optimizing each step of a lab’s sample prep workflow with various solutions from Milestone can have a meaningful impact on elemental analysis, minimize disruptions, and achieve better:

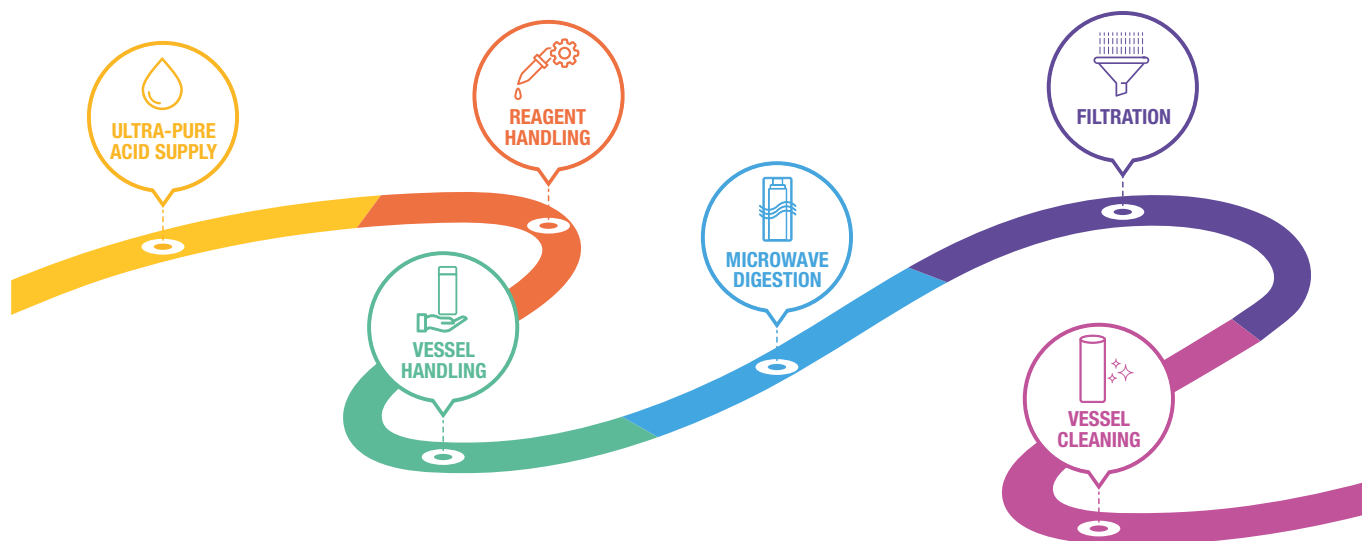
- Speed in sample processing, analysis turnaround time, and labware cleaning
- Quality by reducing detection limits, removing interferences, and improving analyte recovery and reproducibility
- Efficiency by optimizing operator time and cost per sample



Milestone ultraWAVE 1 & 2



Milestone ultraWAVE 3



Steps of the sample preparation workflow

VIDEO

Total Sample Prep Workflow



WEBINAR

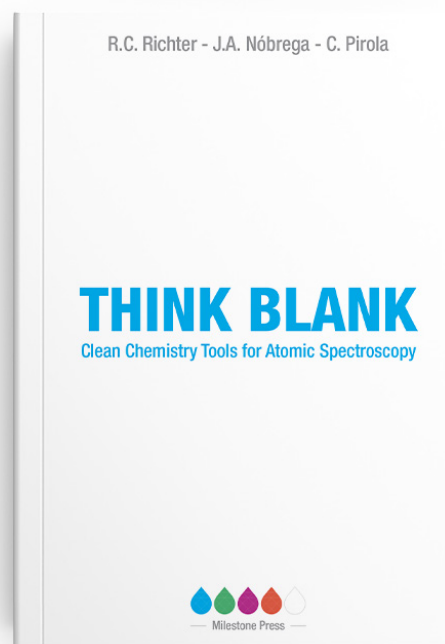
Total Sample Prep Workflow



2.

First things first: contamination sources

During preparation, samples can be contaminated by various elements, including from airborne particles such as dust or via ionic transfer. Solutions for this challenge include a small anti-static gun to neutralize the scale area, or a small ion fan that blows a gentle air current of positive and negative ions to remove charged particulates and reduce static charge. That said, humans are the main point of contamination in most labs, and hair, sweat, or makeup can introduce elements that can impact analytical outcomes. It is nearly impossible to completely eliminate contamination; the goal is to minimize it as much as possible. To that end, powder-free nitrile gloves are recommended to minimize skin oil and sweat contamination. Since gloves are only as clean as the last item touched, it's important to change them regularly. Avoiding cotton or linen lab coats, which can give off lint, is also recommended. Instead, use lint-free materials like nylon, polyester, or Tyvek™. Selecting the right container is critical for reagent purity, and prolonged storage of solutions prior to analysis is not recommended since different containers can carry different levels of contamination that can impact analytical results. Low contamination options include PTFE, FEP, and PFA. Other solutions include minimizing the surface area to volume ratio of the container, keeping the duration of contact between the solution and container surfaces as short as possible, pre-conditioning containers with acid leaching (ideally with acid vapors), and only opening bottles when necessary.



THINK BLANK book by Milestone

DOWNLOAD

“Think Blank” Book



3.

Addressing the lab acid supply and quality requirements

Use of ultrapure reagents is mandatory to work at trace and ultra-trace levels and being able to match the desired detection limits. Ultrapure reagents are typically an expensive line of the analysis costs, moreover the purchased reagent can be easily contaminated. As shown in the table below, contamination of an ultrapure water bottle was significantly higher after being open in a standard lab environment for eight hours. That said, no single purification method can remove all contamination, and

Element	Reference Bottle Freshly Prepared (ppt)	Range of four bottles used for routine preparation operations over 8 hr day (ppt)
Al	11	40 - 120
Ca	20	100 - 700
Cr	0.3	0.4 - 2
Cu	2	2 - 12
Fe	4	5 - 60
K	20	100 - 300
Mg	2	9 - 70
Na	10	100 - 1300
Ti	0.2	0.3 - 13
Zn	4	11 - 50

Influence of analyst on ultrapure water contamination

traditional distillation methods have significant drawbacks. For example, as the liquid is heated, contamination can be trapped in steam droplets and bubbles, which can then be transferred to the sample. As the primary reagent, the acid chosen is important; however, it's also a component that can be expensive, contaminated, or even impacted by supply chain issues. An effective approach to overcome this limitation is by adopting in-house sub-boiling acid purification, via the Milestone

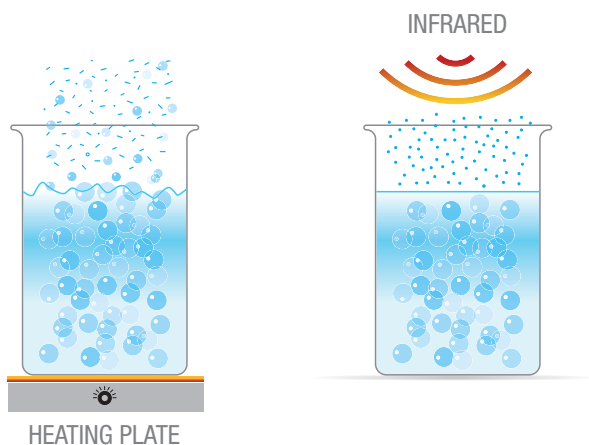
duoPUR system. duoPUR uses infrared heat to gently vaporize the surface liquid only, preventing the formation of aerosolized particles that can carry contamination. One study showed total contamination at 2.3 micrograms per liter using duoPUR, versus 220 micrograms per liter of contamination via traditional distillation.

The benefits of duoPUR:

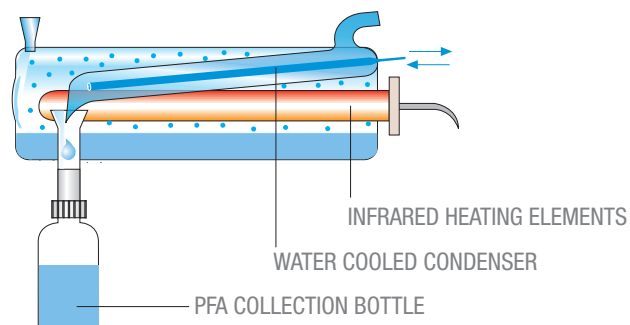
- Control of the acid supply: fresh, high-purity acid on hand or made fresh when needed
- Minimal reagent handling (loading and draining is automatic)
- High productivity, up to 400 mL per hour of purified acid
- High-purity quartz stills, which have no porosity or memory effect
- Up to 90% cost savings over commercial acids with incredibly high ROI



Milestone duoPUR



*Conventional vaporization
VS sub-boiling*



*Schematic of a sub-boiling
distillation process with duoPUR*

VIDEO

Introducing duoPUR



TECNOLOGY REPORT

On sub-boiling acid purification



WEBINAR

On sub-boiling acid purification



4.

How to enhance safety and efficiency in handling of concentrated acids

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, for example, 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.

Milestone's easyFILL is an automatic dosing station offering enhanced safety with a hands-off approach — no acid handling or exposure to acid fumes needed. It is reliable, consistent, and is compatible with a variety of vials and vessels. easyFILL's smart and intuitive software features a touch-screen terminal, and its automation decreases labor and improves productivity.

The dosing compartment is made of anticorrosive polypropylene, and the door is magnetic so it can't be opened while running. It is equipped with a dedicated filter, which pulls acid fumes out through an exhaust (connected to a lab's existing fume hood exhaust system) and captures any particulate contamination. easyFILL allows for the use of up to six reagents at a time, and has full reagent compatibility, including hydrofluoric acid. The system features automatic line flushing, which is convenient when changing acids, and waste is collected through a dedicated drain into a carboy for easy disposal. Reagents and waste are tracked in real time and the user is notified if any specific reagents require refilling or the waste carboy needs to be emptied. easyFILL comes pre-programmed for several vessels and vials, shown in the table on the next page.

The benefits of easyFILL:

- Enhanced safety: no direct contact with acids or exposure to acid vapors
- Improved workflow via automated processes and pre-saved programs
- High reliability with a limited risk of contamination and human error
- High flexibility, suitable for reagent addition and pre-dilution, and compatible with all current Milestone rotors and racks, as well as several popular autosampler racks



Milestone easyFILL

VIDEO

easyFILL in action



WEBINAR

On reagent handling



Rack type	Vessel vial diameter	Milestone MW system/ compatibility	Max. throughput (total n° of positions)
2 x 4	Ø 35-44 mm	SK-15 SR-15	16
4 x 6	Ø 25-31 mm	MAXI MAXI HP	48
3 x 7	Ø 30 mm	ICP autosampler	42
5 x 12	Ø 16 mm	ICP autosampler	120
Rack-15	Ø 16 mm	ultraWAVE	30
Rack-7	Ø 25 mm	ultraWAVE3	14
Rack-20	Ø 16 mm	ultraWAVE3	40
Rack-40	Ø 18 mm	ultraCLAVE	80
Rack-62	Ø 16 mm	ultraCLAVE	144

Examples of easyFILL's compatibility with digestion vessels , vials, and autosampler tubes

5.

Matching the lab requirements with an appropriate digestion process

Once the samples are placed in the digestion vessels and the acid has been added, it is time to perform the digestion process. Nowadays closed-vessel microwave digestion is the benchmark as it has many advantages, including:

- Superior digestion quality by allowing for higher temperatures and pressures
- Greater speed by allowing for fast digestion of multiple samples simultaneously
- Lower detection limits, as it is not open to the environment, requires lower acid usage, and eliminates sample cross-contamination

Rotor-based microwave systems dramatically improved the sample preparation process, however, there are still some challenges that need to be understood and addressed by the operators. Samples can be static-prone, and assembly (and disassembly) of digestion vessels is done manually, requiring operator time. There may also be contamination with NO_x inside the vessels over time. If the sample mass is too high for the rotor, there can be excessive venting that prohibits the sample from reaching the desired temperature and can result in loss of some elements. Some signs of incomplete digestion include yellowing, cloudiness, or unexplained sediment. There may be a need to re-run a sample due to incomplete digestions, exothermic reactions, or contamination, and this will have a negative impact on a laboratory's workflow. Milestone's ETHOS UP helps to mitigate many of these challenges. Like all rotor-based systems, it uses individual pressurized reactors processed in batches. Its large 70.5-L microwave cavity offers the highest level of safety and flexibility (with the ability to accommodate various types of rotors), and full traceability of samples.



Milestone ETHOS UP

ROTOR GUIDE

Choosing the right configuration



The rotor is the heart of the microwave and it's important to choose the correct type based on the sample type and productivity requirements of the lab. When working with closed vessels, it's important to safely ventilate any excessive gases. Milestone's patented vent-and-reseal safety mechanism gently and automatically releases pressure in case of over-pressurization. This improves the safety conditions of the lab by



Vent-and-reseal technology

eliminating the need for a pressure sensor and prevents loss of volatiles. Assembling and closing vessels can take a lot of time and can impact the quality of the digestion if done improperly. One solution is to use an automated capping station like easyCAP to simplify the process, remove the human factor, and ensure reliable vessel closure. The technology used to control the temperature



Milestone easyCAP

The Benefits of easyCAP:

- Higher productivity via reduced handling time and faster closing
- Greater digestion reliability by standardizing vessel closure
- Ergonomic
- Compact and affordable with a small footprint
- Compatible with MAXI-24 HP and MAXI-44 rotors

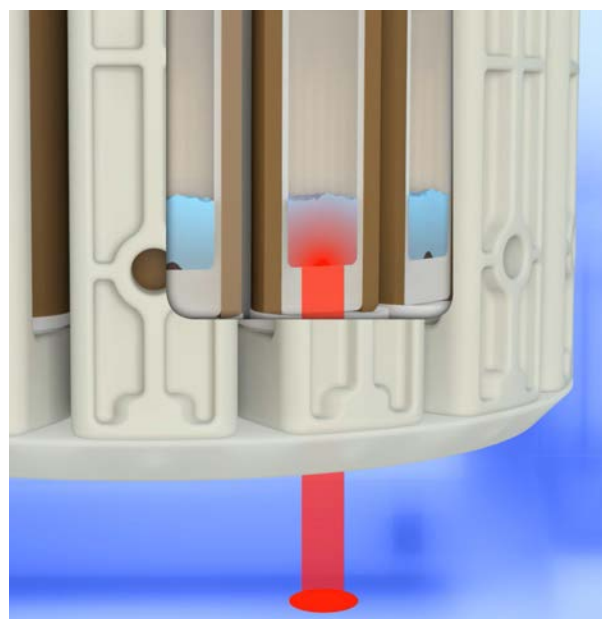
VIDEO

easyCAP in action



in the SK-15 and MAXI rotors is called easyTEMP. easyTEMP controls the temperature of every vessel as it passes over a contactless sensor (internal probes are not required). It provides accurate temperature feedback to ensure complete

digestion in all vessels and, what makes this technology unique, is that every vessel acts as a reference vessel—labs can see in real time the



easyTEMP technology

temperature of each vessel, with the hottest being the control to ensure high safety of operation. Another challenge that can cause delays in workflow is an exothermic reaction. Since easyTEMP is constantly monitoring the temperature of the samples, it can temporarily pause microwave output and cool the sample if temperatures increase too much. Once it reaches again the set profile, it will continue with the rest of the run.

VIDEO

easyTEMP temperature sensor



The benefits of easyTEMP:

- Direct temperature control in all vessels via a contactless sensor
- Accurate temperature feedback ensures complete digestion
- Every vessel acts as a reference vessel

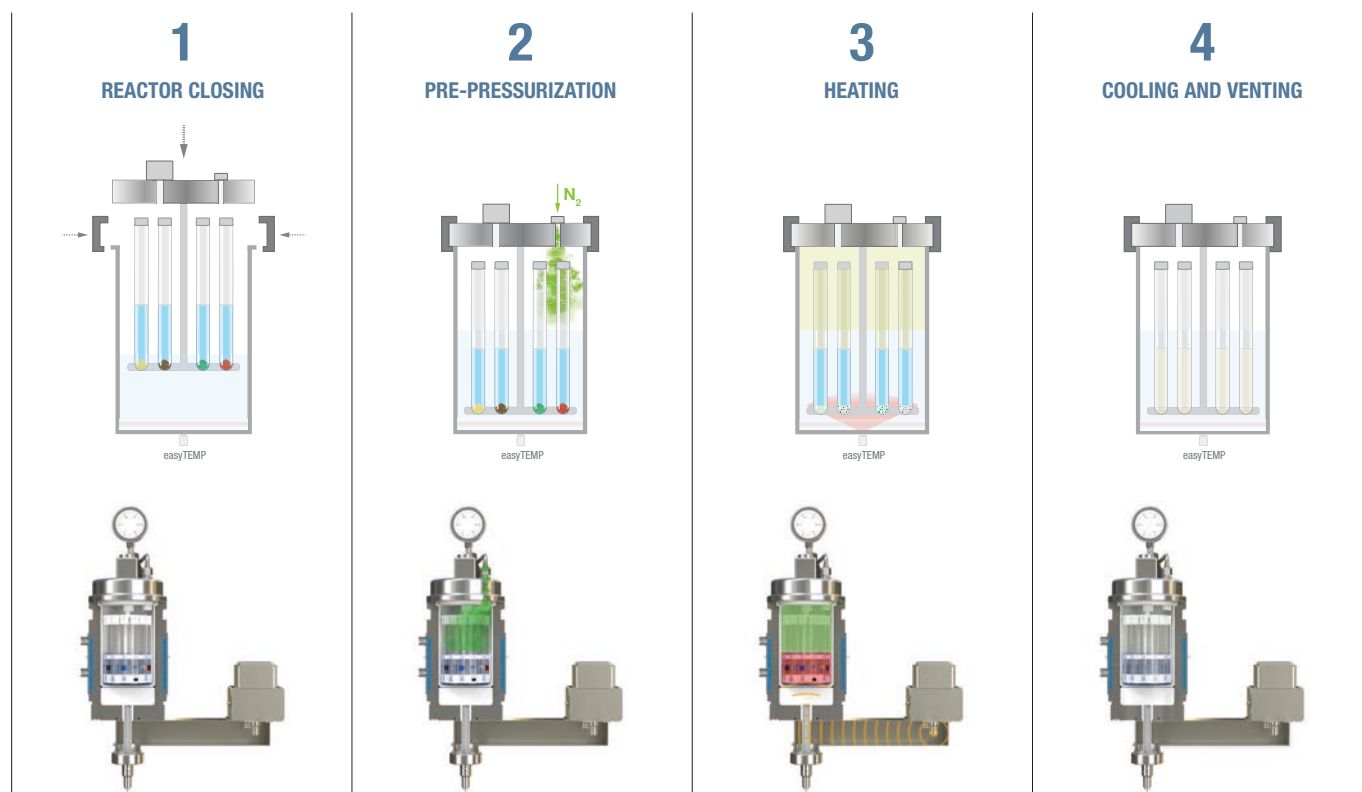
Rotor-based technology offers many benefits — higher digestion quality compared to a hot plate, rugged hardware, a wide library of methods, and multiple configurations — but it also presents various challenges. For example, it has a limited ability to process multiple sample types at the same time. It can also be time consuming, requiring intricate handling and regular replacement of digestion vessels. Thanks to Milestone’s single reaction chamber (SRC) technology, it’s possible to streamline this process. SRC technology is the latest revolution in microwave closed-vessel digestion. Instead of a microwave oven, it’s a pressurized one-liter stainless steel microwave reactor.

The process is straightforward: samples and reagents are loaded into vials and placed inside the cavity; the reactor is closed and hermetically sealed; and the system then automatically introduces nitrogen gas for internal pressurization (which also serves to close the caps). By ensuring



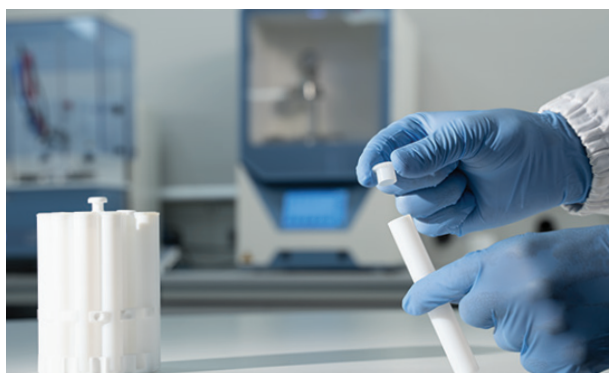
Diagram of Milestone Single Reaction Chamber technology

that the same pressure is present throughout the entire chamber and vials, an equilibrium is established. This means all the analytes stay within the solution in the vials and loss of elements is avoided. SRC also allows for much higher temperature and pressure conditions, reaching



Milestone SRC technology: principle of operation

up to 300 degrees C and 199 bar, allowing for stable and complex matrices to be completely digested. The result? Labs can run any sample, any chemistry, any volume, all in a single run for a significant increase in lab workflow. SRC then cools itself down with water surrounding the reactor, and the internal fumes are removed and collected to the exhaust system. Finally, the reactor can be opened, and the final solutions can be collected for analysis. The result is a shorter digestion run overall, thanks to rapid heating and cooling, and up to 47% reduction in operator time, thanks to the more user-friendly vials and racks construction. Milestone's ultraWAVE 3 elevates all the benefits of SRC. It offers a rugged stainless-steel chamber protected by a PTFE liner and cover, a complete corrosion resistant construction, and compatibility with any chemistry without volume restrictions or modifications. ultraWAVE 3 also has separate high-pressure lines for the nitrogen gas and gas removal, preventing any condensation droplets from entering the reactor.



Milestone loose fitting caps and disposable glass vials for SRC technology

These lines are then automatically flushed to minimize any potential contamination and prolong the lifetime of the tubes. The water-cooled magnetron on this unit is a new noiseless, high-efficiency system. It is independent from ambient temperature, offers a longer lifetime than conventional systems and greater efficiency regardless of the operational conditions. Cleaning also becomes unnecessary with SRC when using disposable glass vials, further reducing the overall sample preparation workflow. Vials in high-purity PTFE-TFM and quartz enable achievement of low detection limits and are ideal for ultra-trace elemental analysis.

The Benefits of ultraWAVE 3:

- Highest productivity, with a 20 - 50% increase per digestion run
- Racks are available with 7, 20, 27, and 40 positions
- Unparalleled usability, with improved handling and an integrated terminal and software
- Unmatched performance and safety, including contactless temperature control and a first-of-its-kind, water-cooled magnetron

VIDEO

Introducing ultraWAVE 3



TECNOLOGY REPORT

On Single Reaction Chamber technology



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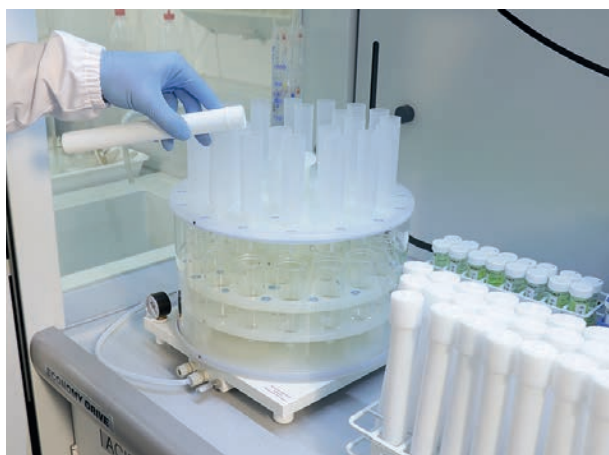
On Single Reaction Chamber technology



6.

Manual filtering made easy and fast

After a digestion, in some cases a sample filtration step is required, for example if a lab is performing an acid leach for elements of interest, or if a digestion leaves behind silicates or other insoluble species. The solution offered by Milestone is called SFS-24, ideal for both acid digestion and solvent extraction procedures. It draws the liquid out into a flask, trapping the solids in a filter. This is much faster than the manual approach — capable of filtering 24 samples at once. It is easy to use, is adaptable to standard evaporation vials, and has a small footprint.



Milestone SFS-24

VIDEO

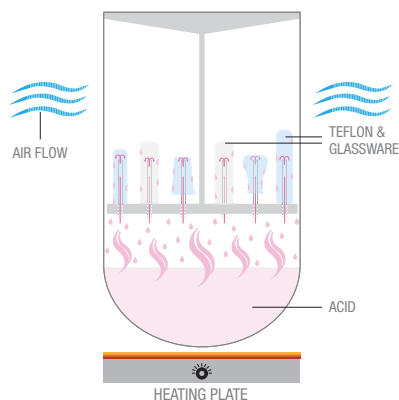
SFS-24 in action



7.

Efficient cleaning for greater detection limit and lab operations

At the end of any digestion, it's important to clean the vessels, with methods ranging from a simple deionized water rinse to deeper cleaning using an acid bath, microwave cleaning cycle, or acid steam cleaning. The downsides of acid baths include the risk of operators being exposed to acid fumes, high acid consumption, long cleaning time, and costly waste disposal. On the other hand, cleaning the vessels in the microwave is effective but lacks efficiency—plus, it impacts the lifetime of the microwave components. Thanks to Milestone's traceCLEAN, vessel cleaning can be an automated step. Acid is placed in the reservoir at the bottom of the system, and the components are placed on hollow rods. The acid is then heated, generating



Automated acid steam cleaning scheme

clean acid vapors, which pass through the rods and remove contamination. The dirty acid falls back into the reservoir and is redistilled for further cleaning. This whole process takes between one and two hours, after which the components can be rinsed with deionized water. traceCLEAN uses a closed, sealed container, and all the components are cool and dry when cleaning is complete. There is no need to soak, and no need to change acids. Plus, it does not require the use of a fume hood

(though it does require a ventilation source) and takes up a very small footprint.



Milestone traceCLEAN

The benefits of traceCLEAN:

- Improved efficiency, eliminating the need for acid baths and microwave cleaning runs
- Automated and convenient—just press play
- Suitable for all digestion vessels, glassware, and ICP accessories
- Safe, with no operator exposure to acid fumes
- User-friendly interface

VIDEO

Introducing traceCLEAN



TECNOLOGY REPORT

On acid steam cleaning



WEBINAR

On acid steam cleaning



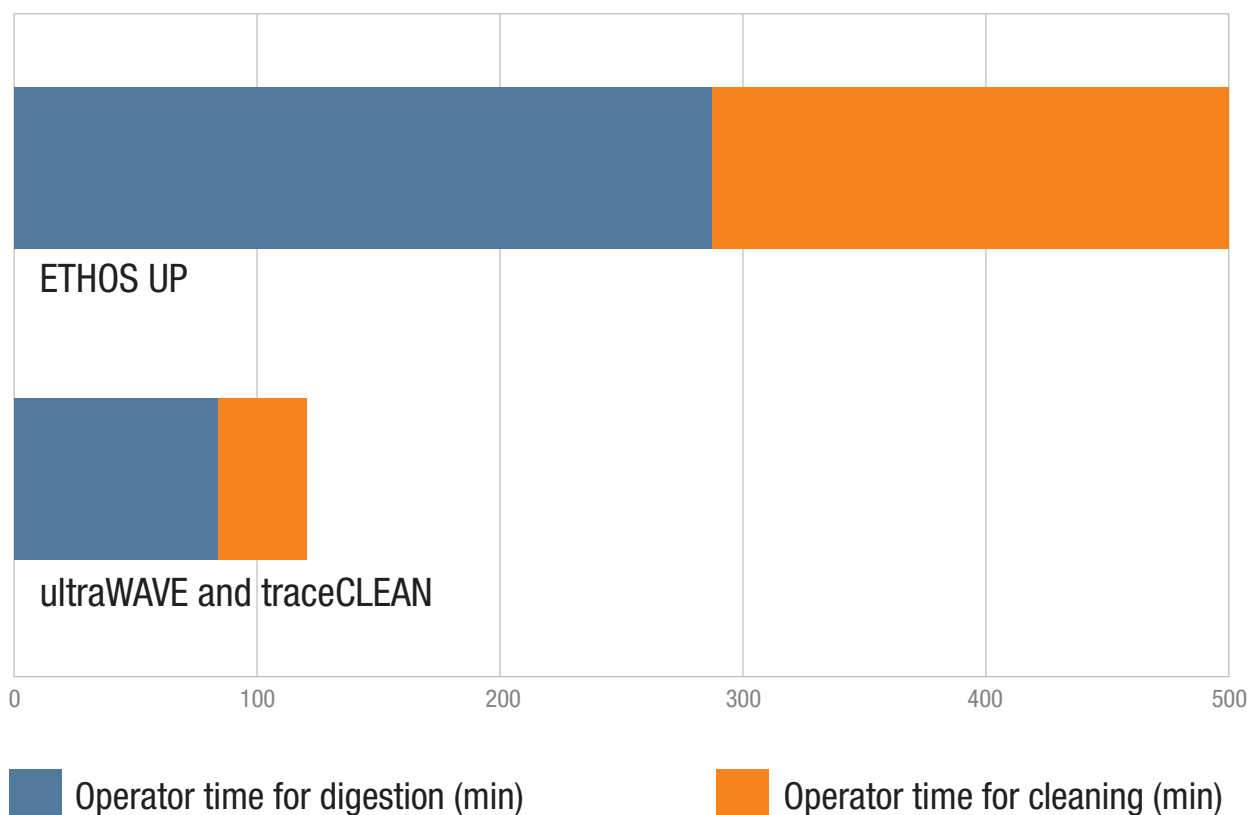
8. Conclusion

Two case studies clearly demonstrate the benefits of automating a workflow with Milestone solutions. The first study features a contract service lab, serving over 400 clients in the pharmaceutical, cosmetic, and dietary supplement industry. Their main application is elemental analysis in compliance with USP standards. This lab runs about 7,000 samples per month (or about 350 samples per day) and was seeking efficiency in digestion and cleaning. They chose to use four ultraWAVE systems, so they could run various samples and chemistries simultaneously; for cleaning, they chose two traceCLEAN systems.

This approach allows the lab to meet their productivity goal of 350 samples per day while requiring only 2 hours of operator time, whereas reaching this level of productivity using traditional (rotor-based) microwave systems and cleaning procedures would require 7.6 hours of operator time per day. Today, the same level of productivity could be achieved using just 3 ultraWAVE 3s, thanks to its greater sample capacity.

CASE STUDY

On a contract lab workflow



Comparison between operator time to run 350 samples/day

The second case study featured a lab operating in the food industry. Its workload is about 2,400 samples per month (or about 120 samples per day). This lab was looking for a solution to streamline its acid production, reagent additions, digestion, and cleaning. Three approaches were proposed, each with their own significant cost and time saving benefits. In conclusion, the benefits of using a total sample prep workflow approach

include increased throughput, lower costs — labor, acid, consumables, and acid waste disposable costs — extra digestion capabilities, reduced errors and contamination, fresh acid availability, and improved lab safety.

WEBINAR

On workflow case studies

<i>Equipment setup</i>	2 x ETHOS UP	1 x easyFILL 1 x ultraWAVE 3 1 x traceCLEAN	1 x duoPUR 1 x easyFILL 1 x ultraWAVE 3 1 x traceCLEAN
<i>Productivity</i>	120 samples/day	120 samples/day	120 samples/day
Daily labor and costs			
<i># of runs</i>	5	6	6
<i>Manual labor</i>	5.7 hours	2.4 hours	2.7 hours
<i>Ultrapure acid costs</i>	\$1677	\$1100	\$157
Annual labor and costs			
<i>Headcount</i>	0.71 FTE	0.30 FTE	0.34 FTE
<i>Ultrapure acid costs</i>	\$445k	\$275k	\$39k

Comparison of the labor and costs using different setups



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ELEMENTAL ANALYSIS:
**GET IT RIGHT
FROM THE START**

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