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traceCLEAN Automated Acid Reflux Cleaning System assures best cleaning quality of your TFM, glass and quartz parts for ICP/ICP-MS accessories.

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A major consideration for any laboratory carrying out elemental determinations by ICP-OES and ICP-MS is the increasing demand to achieve lower detection limits. The analytical instrument is just one part of achieving the lowest limits of detection. Laboratories must also ensure their sample preparation procedure does not contribute any additional sources of contamination that will negatively impact their overall detection capability. The factors to consider when looking to minimize contamination and reduce blank levels while preparing samples for analysis by plasma spectrochemistry include equipment cleanliness, reagent purity, quality of materials, and the digestion procedure itself. In this technical note, we will examine different methods of cleaning microwave digestion vessels, instrument components, and labware.

APPROACHES FOR CLEANING EQUIPMENT

To achieve high-quality blanks when using microwave digestion, pre-cleaning of the vessels is critically important. It is insufficient to simply wash or soak vessel liners in dilute acid. The surface of PTFE is porous and will take up trace amounts of digest solution at elevated temperatures, especially as it “ages” with continued use. Running a blank in a vessel previously used to digest a sample,

without thorough cleaning, will lead to blank contamination and degraded detection capability.

There are two ways to thoroughly clean a microwave digestion vessel. The traditional method is to perform a cleaning run (with blank acid) in the microwave prior to each sample digestion run. However, the downside is that this significantly reduces the sample processing capacity of the microwave digester and puts additional wear and tear on the vessels, since it is done at high temperature and pressure. The alternative approach involves placing the vessels in a closed system containing high-purity acid vapors. There are many benefits of this technique over microwave cleaning runs or acid soaking, which are described below.

THE TRACECLEAN ACID REFLUX CLEANING SYSTEM

The traceCLEAN from Milestone is an automated acid reflux system that thoroughly and safely cleans microwave digestion vessel liners including TFM, PFA, glass, and quartz. The system is comprised of a heated acid reservoir with a metal-free cleaning chamber above it. The traceCLEAN is based on acid steam cleaning, so only high-purity acid vapor comes into contact with the items to be cleaned. Trace metal contaminants present in the cleaning acid remain in the lower reservoir and do not come in contact with the cleaned items. A built-in exhaust/cooling system prevents operator exposure to acid vapors, allowing the system to be placed outside the fume hood. User safety is ensured through the use of a dedicated interlock which allows for unattended running. A cleaning run typically takes about one hour, and cleaned items are cooled inside the system prior to removal, which prevents airborne contamination. In addition, the traceCLEAN can also be used for cleaning other accessories compatible with acid, including plasma torches and sample introduction components used with ICP-OES and ICP-MS instrumentation. A schematic of the system is shown in Figure 1. This method of cleaning is preferred to traditional acid soaking because:

- Trace-metal contamination found in the reagent-grade acid remains in the lower reservoir and does not come in contact with the component to be cleaned.
- The clean component does not remain in contact with the cleaning acid after the surface contamination is removed.
- The critical surfaces of the clean components are nearly dry when the cleaning process is complete, which reduces the need for rinsing and drying.

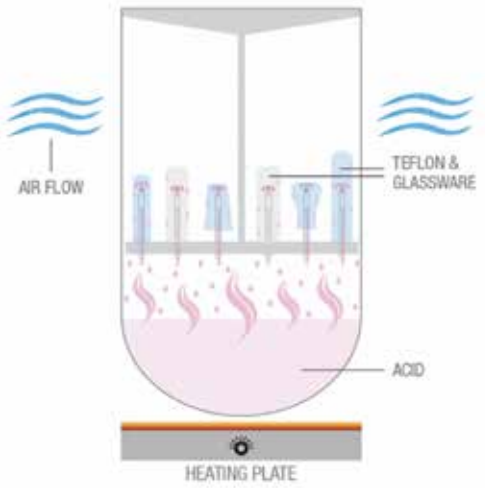

Figure 1: Schematic of the traceCLEAN acid reflux cleaning system.

- The cleaning process takes place in a sealed container, which minimizes airborne contamination and provides a clean environment for the components to be stored until they are needed.
- The clean components are not subjected to high pressure as with microwave cleaning, thus enhancing the component lifetime.
- Increased productivity as the microwave digestion system is used for running samples while the traceCLEAN is dedicated to cleaning vessel components.

CAPABILITY OF THE TRACECLEAN

The performance of the system is exemplified in Table 1, which is a comparison between acid steam cleaning of the traceCLEAN compared to conventional microwave cleaning (1). It shows the ICP-MS analysis of a suite of elements in digestion blanks of a soil sample prepared in microwave vessels with quartz inserts after cleaning with both the traceCLEAN and the microwave digestion system. The first two columns show the concentrations of elements in the soil sample, together with the instrument detection limits. The remaining columns give a side-by-side comparison of the digestion blank values obtained by microwave cleaning and cleaning with the traceCLEAN system. It can be clearly seen that the traceCLEAN produces lower blank values, especially for the common laboratory contaminants such as Al, Mg, Na, Cu, and Zn, for both PTFE-TFM and quartz vessels.

			TFM Teflon Vessel		Quartz Vessel	
Element	Soil Sample (Elemental Concentrations)	Achievable ICP-MS Detection Limit**	Acid Leaching (ng/L)*	traceCLEAN Steam Cleaning (ng/L)*	Acid Leaching (ng/L)*	traceCLEAN Steam Cleaning (ng/L)*
Al	6.53%	200	287±46	258±24	398±28	327±18
Mg	2.89%	196	289±22	232±15	441±56	347±26
Na	1.14%	121	<121	<121	1190±350	608±67
Fe	2.89%	474	<474	<474	±474	<474
Ni	21 mg/g	55	<55	<55	<55	<55
Co	10 mg/g	56	<56	<56	<56	<56
Cu	114 mg/g	52	144±39	117±12	170±15	109±9
Cr	47 mg/g	85	<85	<85	176±57	<85
Cd	41 mg/g	72	<72	<72	<72	<72
Tl	2.5 mg/g	261	<261	<261	<261	<261
Pb	1,163 mg/g	57	<57	<57	<57	<57
Zn	350 mg/g	876	995±80	<876	1640±1000	1005±124

***Detection limits calculated from the standard deviation of 10 blank measurements. *Error expressed as one standard deviation (n=3). (1)*

Table 1: Comparison between high temperature microwave cleaning and cleaning with the traceCLEAN after digesting a soil sample.

CONCLUSION

The traceCLEAN is a highly efficient steam cleaning system that is proven to be superior to traditional high-temperature microwave cleaning and acid leaching. The system can be used to clean a wide variety of laboratory items including microwave digestion vessels and vials, PTFE bottles, glass/quartz volumetric flasks, as well as ICP-OES and ICP-MS sample introduction components. In addition, acid consumption is significantly reduced, and analysts do not have to handle large quantities of highly corrosive acids on a daily basis. As a result, this acid washing system is highly recommended for any lab that wants to perform trace-metals analysis.

ABOUT MILESTONE

With over 50 patents and more than 20,000 instruments installed in laboratories around the world, Milestone has been widely recognized as the global leader in metals prep technology for the past 30 years. Committed to providing safe, reliable and flexible platforms to enhance your lab's productivity, customers worldwide look to Milestone for their metals digestion, organic extractions, mercury analysis and clean chemistry processing needs.

Robert Thomas is the principal of Scientific Solutions, a consulting company that serves the application and writing needs of the trace element user community. He has worked in the field of atomic and mass spectroscopy for more than 40 years and has written over 90 technical publications including a 15-part tutorial series on ICP-MS. He recently completed his fourth textbook entitled **Measuring Elemental Impurities in Pharmaceuticals**: A Practical Guide. He has an advanced degree in analytical chemistry from the University of Wales, UK, and is also a Fellow of the Royal Society of Chemistry and a Chartered Chemist. He has led the heavy metals, plasma spectrochemistry task force on the ACS Committee on Analytical Reagents.

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