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New Developments in Automated Cleaning of PTFE, Glass, and Quartz Components Used in Ultratrace Analysis

by Robert Richter

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Advances in modern analytical instrumentation during the past decade allow sub–parts-per-billion determinations to be made on a routine basis. The analyte levels in the blank may be significant when compared with the sample, and these levels must then be subtracted from the measurement. Therefore, the total uncertainty of the analysis involves the uncertainty of both the blank and the sample. Because blank measurements are usually made near the instrument detection limit, the uncertainty of the blank measurement is usually greater than the uncertainty of the sample measurement. The accuracy of the result could therefore be obscured by the uncertainty of the blank measurement. The inability to control the analytical blank is often the primary source of error and the limiting factor for trace analysis.

Figure 1. TraceClean automated acid reflux system.

Analytical chemists take numerous precautions to ensure the lowest possible response for the analytical blank, including using high-purity acids and reagents for sample preparation, powder-free gloves, lint-free protective clothing, rigorous cleaning of all sample preparation and instrument components, and so forth. Despite all of these precautions, many chemists still have problems controlling the analytical blank. Their problems usually result from the way the sample preparation and instrument components (such as polytetrafluoroethylene [PTFE] bottles, volumetric flasks, glass and quartz ICP and ICP-MS spray chambers, torches, sample and solution containers, and microwave vessels) are cleaned and stored.

The most common procedure for cleaning components for trace-metal analysis is hot-acid leaching (1, 2). The components are placed in a large beaker filled with a 1:1 or 1:4 nitric acid/water solution. The beaker is placed on a hot plate and heated at ~80 °C for 1–4 h. After cooling, the components are removed, rinsed, and allowed to dry.

The first potential problem arises from the acid and container used for cleaning. The acid used is usually reagent-grade quality because using high-purity, double-distilled acid for cleaning is not economical. This poses a problem when the metal concentration of the cleaning acid is higher than that of the samples being analyzed. The beaker used for cleaning can also add trace-metal contamination to the cleaning solution. Furthermore, the cleaning acid must be changed after every cleaning to ensure the lowest blank levels.

The second potential problem arises from the postleaching processing. The clean components are removed from the acid bath with the aid of plastic tongs or thick protective gloves. Trace-metal contamination can be deposited onto the clean components if the tongs and gloves have not been thoroughly cleaned. After removal, the components must be thoroughly rinsed to remove any surface contamination introduced by the cleaning acid. The rinse water must be of a high purity and be kept free of contamination to ensure the best results. After rinsing, the clean components are allowed to air dry before use.

Finally, airborne contamination deposited during the air drying process is one of the major sources of contamination for trace-metal analysis. Airborne contamination can result from rust in the hood used to process the samples, dust in the laboratory ventilation system, nearby samples being prepared for analysis, the chemist, and other sources. The only truly effective way to control airborne contamination is to prepare samples in a cleanroom, and for most analytical labs this is not an option. Therefore, Milestone (Monroe, CT) has developed the TraceClean system to address these problems.

The TraceClean system (Figure 1) uses sub-boiling distillation to clean various sample preparation and instrument components.

Figure 2. Twenty-position component rack with PTFE-based glass rods.

Figure 3. The TraceClean system includes a microprocessor controller for unattended operation.

Sub-boiling distillation is the same technique used to prepare high-purity acids used for analysis. This means components are not contaminated by the acid used to clean them. Another important feature of the system is that the cleaning and drying of the components occurs in a sealed container, minimizing the amount of airborne contamination that can be deposited on the components and keeping them clean until they are needed.

The system was recently tested at DuPont's Jackson Laboratory (Deepwater, NJ). This article compares the cleaning of tetrafluoromethoxy (TFM) microwave digestion vessels and high-purity quartz inserts using the TraceClean system with the traditional method of cleaning these components.

INSTRUMENTATION

A first-generation TraceClean system was used for this study. It consisted of a glass distillation container that holds ~750 mL of acid, a 20-position cleaning rack (Figure 2), an automated elevator assembly to insert and remove the components from the distillation container, an electrical heating plate, and a built-in exhaust fan. The cleaning process is microprocessor-controlled (Figure 3), allowing users to program the cleaning time and temperature.

Cleaning efficiency study. The soil samples for this study were prepared in an Ethos Plus Microwave Labstation (Milestone), which consisted of the microwave unit, a 10-position segmented rotor, temperature and pressure controls, and high-purity quartz inserts. The digestion process was microprocessor-controlled, allowing users to set precise heating profiles for sample decomposition. An ELAN-6000 ICP-MS system (PerkinElmer Sciex, Norwalk, CT) in standard configuration, equipped with a J.E. Meinhard (Santa Ana, CA) concentric nebulizer, was used for analysis.

METHODS

Soil digestion. A certified soil sample was used for this study. Table I lists the concentrations of the elements of interest. EPA Method 3051A was used to digest 0.50 g of the soil sample. The digestions were performed using standard PTFE microwave vessels and high-purity quartz inserts. After the microwave vessels and quartz vessels were cleaned, digestion blanks were prepared using the same acids and procedure.

Microwave cleaning. High-temperature microwave cleaning has proven to be the most effective way to clean sample preparation and instrument components (3). This method is effective because the use of high-purity acids and closed vessels minimizes the potential for contamination. Each microwave vessel and quartz insert was cleaned by adding 10 mL of Optima (Fisher Scientific, Pittsburgh, PA) nitric acid and 3 mL of Optima hydrochloric acid, then microwave heating them for 20 min at 180 °C. After cooling, the cleaning acid was discarded, and the microwave vessels and quartz inserts were rinsed with high-purity water and allowed to dry.

Cleaning. The TraceClean distillation container was filled with ~750 mL of reagent-grade nitric acid. The microwave vessels, caps, and quartz inserts were loaded into the cleaning rack, lowered into position, and the cleaning program was started. When the cleaning program was complete, the vessels and quartz inserts were removed and immediately used for preparation of digestion blanks.

RESULTS AND DISCUSSION

TraceClean cleaning process. Sub-boiling distillation has been shown to be the method of choice for acid purification (3, 4). The key to this method is vaporization without vigorous boiling. Vaporization prevents aerosolized particles from contaminating the acid. The TraceClean system uses the same sub-boiling process for cleaning sample-preparation and instrument components.

The lower reservoir of TraceClean's distillation container holds ~750 mL of concentrated acid. When the cleaning rack is lowered into position, the PTFE bottom plate seals off the lower reservoir, with the exception of the vapor ports. The standard cleaning rack has 20 vapor ports fitted with hollow PTFE-coated glass rods. The microwave vessels and quartz inserts are placed over the glass rods. When the lower reservoir is heated, the puri-

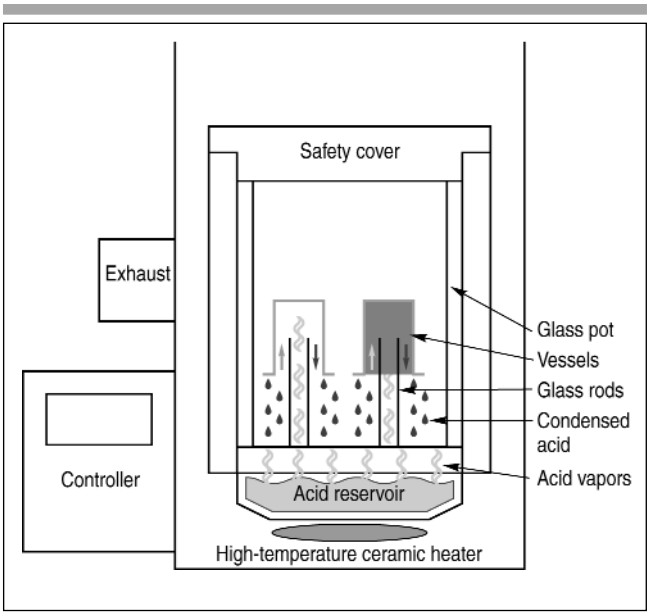

Figure 4. Schematic of the TraceClean's sub-boiling acid cleaning process.

fied acid vapor travels up through the glass rods and condenses on the vessels and inserts, removing the surface contamination (Figure 4). This method of cleaning is superior to the traditional method for the following reasons:

- The 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 dry when the cleaning process is complete. This eliminates the need for rinsing and air drying.
- 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.

Evaluation of TraceCLEAN cleaning efficiency. Digestion blanks prepared in the microwave vessels and quartz inserts after cleaning with the TraceClean and the microwave were analyzed for selected trace-metal contamination by ICP-MS. Tables I and II show the soil concentrations of the selected contaminants, the instrument detection limits, and method blank results. Table III gives a side-by-side comparison of the digestion blank values obtained with microwave cleaning and cleaning with the TraceClean system. The TraceClean produces lower blank values, especially for the common laboratory contaminants Al, Mg, Na, Cu, and Zn, for both types of materials. Note that operation of the TraceClean system and analysis of samples inside a clean room environment will result in significantly lower blank values.

Table I. Concentrations of the elements of interest in the soil sample.

Element	Sample Concentration
Al	6.53%
Mg	2.89%
Na	1.14%
Fe	2.89%
Ni	21 μg/g
Co	10 μg/g
Cu	114 μg/g
Cr	47 μg/g
Cd	41 μg/g
Tl	2.5 μg/g
Pb	1162 μg/g
Zn	350 μg/g

CONCLUSION

The TraceClean system has proven to be superior to traditional high-temperature microwave cleaning. The TraceClean's unique and highly efficient subboiling cleaning process will soon make the traditional hotplate and microwaving cleaning methods obsolete. The system can be used to clean a wide variety of laboratory items including PTFE bottles, volumetric flasks, glass and quartz ICP and ICP-MS spray chambers and torches, and sample and solution containers. The use of an automated elevator assembly for raising and lowering the components to be cleaned reduces the analyst's exposure to concentrated acids and awkward movements that could result in a chemical burn or spilling of concentrated acid. In addition, acid consumption is substantially reduced (the sub-boiling process allows the acid to be used for as long as six months) and analysts don't have to handle large quantities of acid on a daily basis. This acid washing system is highly recommended for any lab that wants to perform trace-metal analysis without the additional cost and overhead associated with operating a cleanroom.

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Table II. ICP-MS detection limit and level of contamination in the blank (ng/L).

Element	Detection Limit*	Method Blank**
Al	200	257 ±21
Mg	196	196
Na	121	⩽ 121
Fe	474	⩽474
Ni	55	⩽55
Co	56	⩽56
Cu	52	91 ±12
Cr	85	⩽85
Cd	72	⩽72
Tl	261	⩽261
Pb	57	⩽57
Zn	876	⩽ 876

*Calculated from the standard deviation of 10 blank measurements. **Error expressed as one standard deviation ($n = 3$).

Table III. Comparison of high-temperature microwave cleaning vs. TraceClean (ng/L).

	TFM PTFE Microwave Vessel		Quartz Microwave Insert	
Element	Microwave*	TraceClean*	Microwave*	TraceClean*
Al	287 ± 46	258 ± 24	398 ± 28	327 ± 18
Mg	289 ± 22	232 ± 15	441 ± 56	347 ± 26
Na	⩽ 121	⩽ 121	1190 ± 350	608 ± 67
Fe	⩽ 474	⩽ 474	⩽ 474	⩽ 474
Ni	⩽ 55	⩽ 55	⩽ 55	⩽ 55
Co	⩽ 56	⩽ 56	⩽ 56	⩽ 56
Cu	144 ± 39	117 ± 12	170 ± 15	109 ± 9
Cr	⩽ 85	⩽ 85	176 ± 57	⩽ 85
Cd	⩽ 72	⩽ 72	⩽ 72	⩽ 72
Tl	⩽ 261	⩽ 261	⩽ 261	⩽ 261
Pb	⩽ 57	⩽ 57	⩽ 57	⩽ 57
Zn	995 ± 80	⩽ 876	1640 ± 1000	1005 ± 124

*Error expressed as one standard deviation ($n = 3$)