How to Achieve the Lowest Level of Metals Detection Using the Ultimate Clean Cup

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ABSTRACT

When it comes to achieving low detection capabilities for metals testing, laboratories know not only the importance, but also the difficulties associated with keeping a clean lab and clean testing supplies. This paper will discuss the new Ultimate Clean Digestion Cup from Environmental Express and how our lab has navigated through the obstacles of achieving low detection capabilities in metals digestions and analysis.

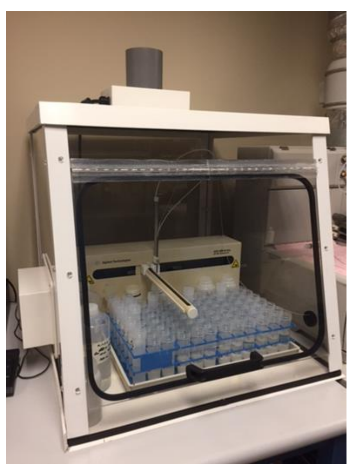
INTRODUCTION

Regulatory entities along with competitive markets are driving the demand for lower and lower detection levels of samples. When it comes to achieving low detection capabilities for metals testing, laboratories recognize not only the importance but also the difficulties associated with keeping a clean lab and clean testing supplies. You can achieve low detection capabilities with the new Ultimate Clean Digestion Cup from Environmental Express and a clean lab. Here's how our lab navigated through the obstacles to achieve low detection capabilities in metals.

PROCEDURE

To achieve and generate metals data at ultralow and trace levels (part-per-billion and part-per-trillion) you must first eliminate as much contamination as possible. The most obvious step in achieving a clean laboratory environment is to give it a deep cleaning. Dust is a major contributor of metals contamination, especially for Zinc (Zn). In our lab, we started by removing all unnecessary items from the lab. Then we reorganized all necessary lab items by placing them in designated drawers and cabinets. Next we wiped counter space, computers, instruments, and the fume hood. Finally, we vacuumed the floors versus sweeping to minimize airborne particulates. Mopping could be added to remove contaminants as well. It is a good idea to establish a regular cleaning schedule in your laboratory.

Once our lab was clean, we wanted to keep our sampling area free of contaminants, so we installed an AirLite™ benchtop enclosure system (item number SC801) over our autosampler (Picture 1). After new data was collected from the Agilent 7500cs ICP-MS system, it was compared to past data results. We noticed an improvement in the quality of the data from the decreased occurrence of random contamination spikes. Random spikes in the data mean that somehow the sample was contaminated from an outside source. This improvement drove our decision to replicate the same concept for our sample preparation area. (Picture 2). We designed, built, and installed a custom enclosure. Both of these AirLite enclosure units contain a High-Efficiency Particulate Air (HEPA) filter through which air is drawn by a small fan at the top of the unit. This maintained standards' purity and prevented us from contaminating samples during use. These three differences had a major impact toward achieving consistent lower detection levels.



Picture 1: AirLite™ Enclosure

Picture 2: Custom Enclosure

Our next target was to eliminate means of contamination from materials that encountered the samples. We stopped using glass throughout our sample preparation except the glass on the actual ICP-MS. It's important to do this because borosilicate glass has the potential to leach silicon (Si), boron (B), and sodium (Na) as well as absorb lead (Pb) and chromium (Cr). Glassware tends to exhibit "memory" effects from previous solutions.

It is also important to use standards that have not expired and ensure your standards are certified and carry a robust Certificate of Analysis (CoA) with information on background contaminants. Use high-purity acids: We use sub-boiled distilled hydrochloric (item number HP9030) and nitric (item number HP9040) acids.

A well-maintained clean deionized (DI) water system is crucial as it is used in every aspect of the laboratory, everything from sample prep to cleaning and soaking. Our water runs through an initial large-scale DI system off the main water supply and then through a benchtop DI water system, Thermo Scientific™ Barnstead™ MicroPure™ ultraviolet (UV) water purification system (item number W50132373). Periodically we tested our water at various points to ensure no contamination entered our system. We made it a practice in our lab to change the peristaltic pump sample intake tubing every other day to prevent build-up. In addition to this, we use a low-metals, prerinsed pump tubing which saves time because it rinses down much faster than traditional pump tubing during instrument setup. We only clean internal ICP-MS parts in our lab such as the spray chamber, torch, cones, etc., when it is recommended per the preventative maintenance schedule, or if we see contamination levels start to creep up on our blank results. We have found that soaking everything not in use, with a weak acid solution, is a crucial step to reducing contamination.

Preparing the calibration curve requires a steady hand, patience, and precision. The more steps that can be eliminated, and the less the calibration curve is handled, the more consistency can be achieved. We use dilutions and a balance to make our calibration curve. While making the curve, it only comes in contact with a single disposable pipette, a 50 mL polypropylene cup (item number UC475-NL), a 100 mL polypropylene cup (item number SC490) and a 50 mL perfluoroalkoxy (PFA) cup (item number SC545). Our calibration curve along with all our reagent blanks are made in the PFA cup. Our calibration curve is made up of four points within the 0 to 40 ppb range for minerals and six points within the 0 to 1 ppb range for all other elements. It is made daily to every other day to ensure optimal instrument calibration.

Sample preparation introduces many opportunities for contamination to occur if you are not attentive. In our lab, we make smaller batches of digestion solution. Depending on how many samples are prepared, a few batches may be made. Each time a new batch is made, it offers the potential for contamination. Originally, only one digestion solution blank was analyzed at the beginning of our entire sample run. Upon reviewing our data, we noticed elevated metals hits on groups of samples. These hits correlated with when a new digestion solution was made. Following this discovery, each time a new digestion solution is made we now run a blank in a PFA tube placed before the associated samples. The PFA tube acts as a control against our polypropylene cups. Notice the pattern in Chart 1 and Chart 2. In this instance the "contamination" can be traced to the digestion solution. While this proves that the contamination originated in the digestion solution and not the cup, it does not answer the million-dollar question, "Where did it come from in the first place?" This can be the most difficult question to answer when struggling with low-metals analysis. It took some investigation into our digestion method to shed some light on it.

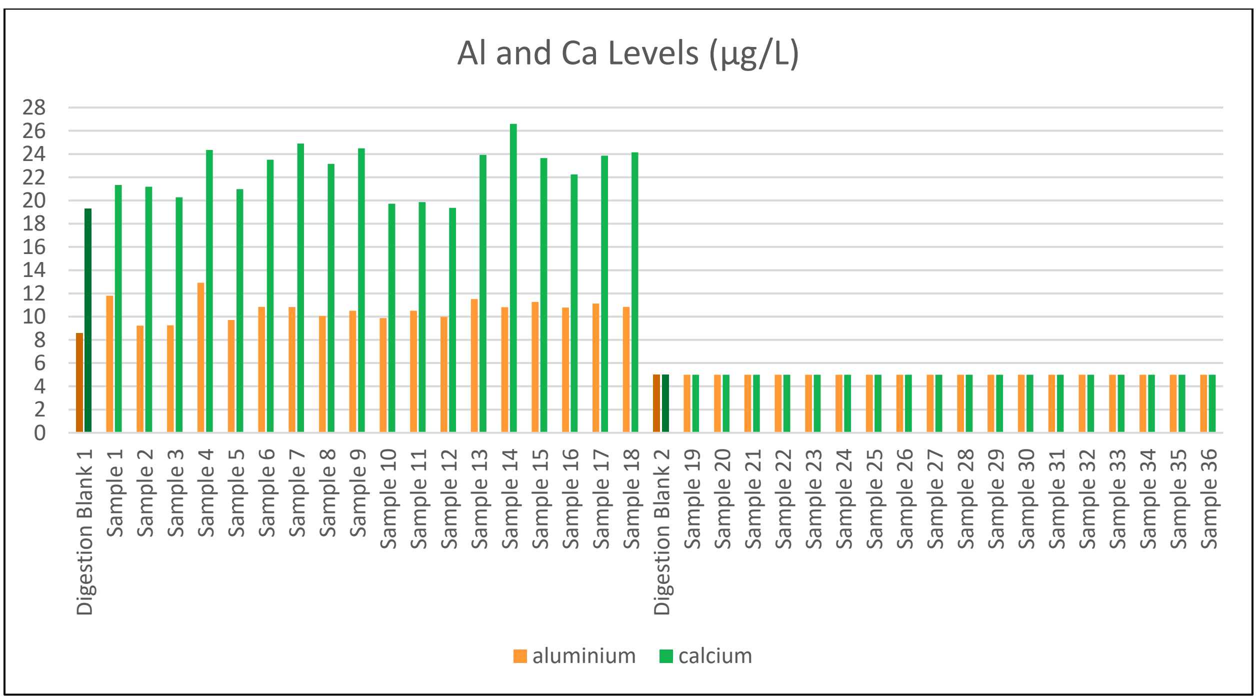


Chart 1: Detected Al and Ca (µg/L) levels in samples compared to the digestion blank.

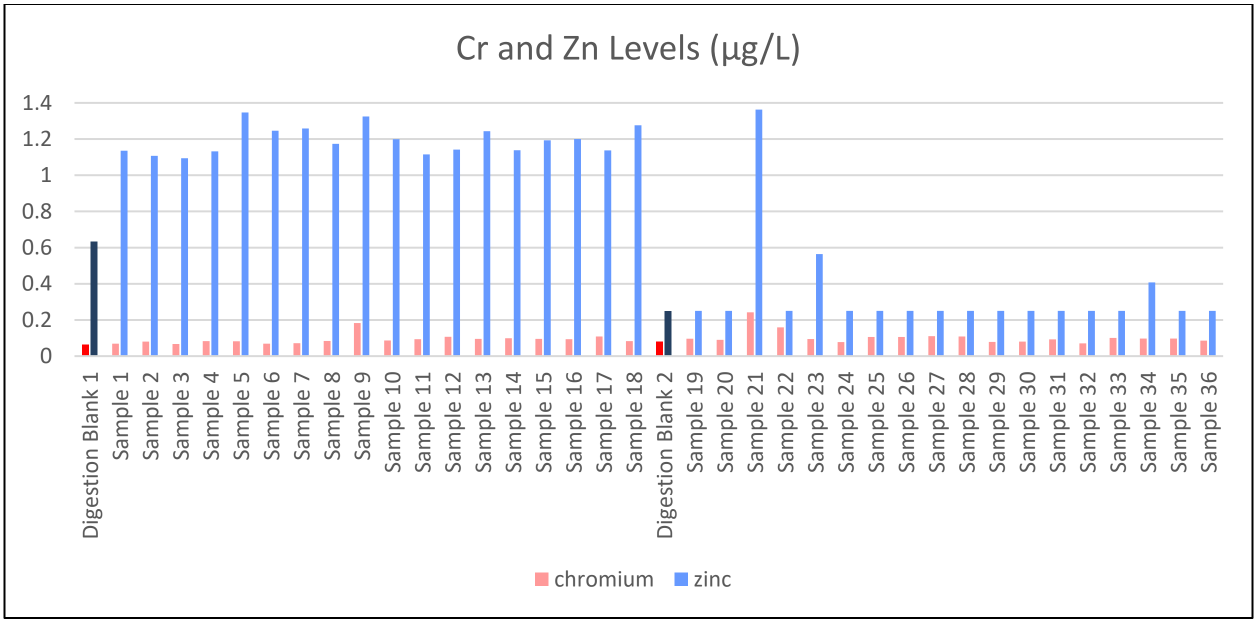


Chart 2: Detected Cr and Zn (µg/L) levels in samples compared to the digestion blank.

While we try to simulate typical United States Environmental Protection Agency (EPA) digestion methods as much as possible, our goal is to test our cups and caps for cleanliness. To do this, we performed our digestions with the caps loosely placed on top—much like using a watch glass or reflux cap. We found this practice provided the most rigorous digestion possible with our cups ensuring we were identifying all potential contaminants that may be present. Not only did this give us the best indication of what is present in our digestion cups but we were also able to achieve much cleaner and lower level results. This digestion procedure also gave us an answer to this source of contamination— the fume hood. Look at your fume hood and consider installing a HEPA filter so the air being drawn over your samples doesn't contaminate them. It is equally important to make sure your fume hood stays clean from dust, spills, and other chemicals.

In a clean lab, personal protective equipment (PPE) not only protects lab personnel from potential hazards in the lab but also protects the lab equipment and samples from lab personnel. Metals can be found in everything including deodorant, cosmetics, lotions, lint on clothes, and soil on shoes. Our lab personnel wear lab coats, safety glasses, pull free-flowing hair back, and remove as much jewelry as possible.

As in any lab, and especially a clean lab, gloves are imperative. We found that using vinyl gloves (item number EE8658022) instead of powder-free nitrile gloves provides a cleaner handling. Another practice we found to aid in cleanliness is to wash gloves with deionized water once the gloves are placed over your hands and before use. A finger dip test helped to confirm our observation as shown in Chart 3. Powder-blue nitrile gloves, fresh out of the box, showed contamination of 13 different elements while vinyl gloves, right out of the packaging, showed contamination of only three different elements. In both cases, rinsing the gloves with deionized water greatly decreased contamination.

	Powder-free blue nitrile gloves out of box (µg/L)	Powder-free blue nitrile gloves rinsed with deionized water (µg/L)	Vinyl gloves out of packaging (µg/L)	Vinyl gloves rinsed with deionized water (µg/L)
Sodium	83.59	5.252	301.4	<5
Magnesium	21.14	10.43	<5	<5
Aluminum	9.505	<5	<5	<5
Phosphorus	6.007	<5	<5	<5
Potassium	111.6	5.644	<5	<5
Calcium	6264	2627	35.75	15.27
Iron	7.907	<5	<5	<5
Zinc	259.1	135.1	15.85	1.902
Strontium	1.444	0.6905	<0.25	<0.25
Manganese	0.4559	0.192	<0.0625	<0.0625
Zirconium	0.1259	<0.0625	<0.0625	<0.0625
Lanthanum	0.0816	<0.0625	<0.0625	<0.0625
Cerium	0.1172	<0.0625	<0.0625	<0.0625

Chart 3: Comparison chart between the level of metals (µg/L) found in powder-free blue nitrile gloves and vinyl gloves directly out of the packaging and also rinsed with deionized water.

Whenever possible, we highly suggest you not run other tests in the same vicinity of your clean lab. These tests may produce fumes, leave a residue, create ash, etc., that could cause contamination. Another practice that may benefit a clean lab is the separation of making standards and prepping samples either in different hoods or different areas. This creates an environment where cross-contamination from splashes, spills, or mishandling is less likely.

The need and capability in achieving trace and ultra-trace detection levels drive a demand for contaminant-free products to run these tests. It only made sense for Environmental Express to develop the Ultimate Clean Cup (item number UCC000-50). These 50 mL digestion cups are specifically designed for trace metals analysis and carry the most robust certification on the market today. The Ultimate Clean Cup is certified for 68 elements at part-per-billion and part-per-trillion levels. The moment each cup comes off the mold, it is carefully handled to avoid common means of contamination. Our specially designed packaging ensures the Ultimate Clean Cup arrives ready to use in any clean environment.

CONCLUSIONS

Proper training is essential for lab personnel performing low metals analysis, as well as those working in a clean lab environment. Not only is it necessary to understand what the best practices are but also the reasoning and repercussions behind those practices. Contaminant-free products will not benefit a lab if clean lab procedures and techniques aren't followed. When we have new lab employee, we start them in the prep role to learn the basics. Fundamentals are key and the data produced can only be as good as your preparation. Ultimately, each individual lab needs to develop its own system of best practices and develop standard operating procedures to fit its clean lab needs. The Ultimate Clean Cup is a product that has taken years of study, in both the manufacturing and laboratory process, to perfect. Our technical staff has experienced many contamination failures and has solved the majority of them. We welcome your comments or questions.