# Savillex Technical Note

## Lab Bottles for Ultratrace Metals Analysis

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## Summary

Trace metals analysis is typically performed by inductively coupled plasma mass spectrometry (ICP-MS), which has detection limits at the low ppt and sub-ppt levels for almost every measurable element. Therefore, elemental contributions from reagents (DI water, acids) and reagent storage containers must be strictly controlled in order not to impact ICP-MS data integrity. PFA fluoropolymer lab bottles are used to ship high purity acid and in every trace metals lab to store trace metal standards and rinse solutions. The use of fluoropolymer bottles with the lowest possible trace metal contribution is, therefore, as important as the purity of reagents used in the analysis.


*Savillex ISO Class 7 Cleanroom*

Savillex's lab bottles are manufactured from the highest purity grade of virgin fluoropolymer PFA resin in an ISO Class 7 cleanroom using unique stretch blow molding technology. As a result, they represent state-of-the-art in ultraclean fluoropolymer bottles for trace metals analysis. This technical note gives detailed information on bottle cleaning procedures along with high-resolution ICP-MS (HR-ICP-MS) analytical data for 63 elements in Savillex lab bottles following cleaning. The combination of unique molding technology and cleanroom manufacturing has enabled the production of fluoropolymer bottles that, once cleaned, have no elemental background contribution measurable by HR-ICP-MS.

## Stretch Blow Molding: Benefits for Bottle Cleanliness

Until now, fluoropolymer bottles have been produced by extrusion blow molding: molten polymer is extruded into the molding press, and pressurized air is injected via a tube which forces the molten polymer to conform to the shape of the tool, producing the bottle. Because the neck of the bottle is molded as the bottle is blown, the quality of the neck and thread is inferior to that of injection molding. After molding, the neck and lip must be machined (producing particles) to obtain a good seal with the closure (cap). Even then, a secondary seal or insert is often required inside the closure to produce a reliable long-term seal.

Savillex has taken a different approach, applying the newer stretch blow molding technology to fluoropolymer bottle manufacturing. Since no commercially available stretch blow molding machinery can handle the high temperatures required for fluoropolymer molding, Savillex designed and built stretch blow molding machinery in-house to produce lab bottles. Stretch blow molding is a two-step process. First, a "preform" is produced by high-precision injection molding. Next, the neck and thread of the finished bottle are molded in this step. The preform is essentially a miniature bottle with straight sides and a finished bottleneck and thread. The preform is then blown into the final bottle in the stretch blow molding step. Because the bottle lip and thread are injection molded, the seal quality of the finished bottle is excellent. No machining of the lip is needed, and no closure insert is required. In addition, the smooth inner neck and lip of the Savillex bottles allow smooth, drip-free pouring, improving safety and reducing contamination risk.

Unlike other fluoropolymer bottles, Savillex lab bottles and closures are made from the highest purity grade PFA resin. Only virgin resin is used. No reground or recycled waste fluoropolymer is ever added.

## Cleanroom Manufacture

The second benefit of stretch blow molding is that the process is cleaner, and the equipment is much smaller than extrusion blow molding, which allows it to be located inside a cleanroom. The preforms and closures are injection molded outside the cleanroom, but extensive precautions are taken to prevent contamination by airborne particulates, including the use of HEPA enclosures around the press, portable clean hoods, and anti-static devices. The preforms are blown into bottles inside an ISO Class 7 cleanroom with continuous particle count monitoring. The bottles are immediately capped, inspected, and then sealed in HDPE bags prior to leaving the cleanroom.

More information on stretch blow molding and bottles, in general, can be found at www.savillex.com.

*Savillex Lab Bottle Manufacturing in Class 7 Cleanroom*

## Bottle Cleaning Prior to Use

The combination of highest purity grade virgin resin, exhaustive precautions against contamination, and cleanroom manufacture enables the production of bottles with ultra-low metal content, even before cleaning. Nevertheless, bottles must be cleaned prior to first use in trace metal applications. The recommended cleaning strategy upon removal of the bottle from its HDPE bag is to rinse with DI water and fill the bottle with high purity 2% $HNO_3$/1% HF, replace the closure and maintain at 50°C for seven days to accelerate the removal of any residual contamination. Finally, the contents are discarded, and after a thorough rinse with DI water, the bottle is ready for use.

## Analysis of Bottle Trace Metal Content

To demonstrate the cleanliness of bottles following initial cleaning, three 500 mL Savillex PFA lab bottles were tested for trace metal contribution by an independent lab, using an acid extraction test followed by HR-ICP-MS analysis. The cleaning, extraction, and measurement methodology is given below.

**Cleaning Cycle:** On receipt, the bottles were removed from their bags and rinsed with DI water. They were then filled with a solution of high purity 2% $HNO_3$/1%HF in DI water. The closures were replaced, and the bottles were maintained at 50°C for seven days. This solution was discarded, and the bottles were rinsed well with DI water. This simulates the initial cleaning of new bottles prior to use.

**Extraction Cycle:** The bottles were then refilled with high purity 2% $HNO_3$/1%HF in DI water, closures were replaced, and the bottles were again maintained at 50°C for seven days. This extraction solution contains any trace elements that would be leached from the bottles on initial use after cleaning.

**Measurement:** The extraction solution was analyzed for trace metals as follows: an aliquot of the solution was evaporated in a preconcentration system to give a preconcentration factor of x125. The preconcentrated solution was analyzed for 63 elements using a Thermo Element2 HR-ICP-MS. All sample prep and analysis were performed inside a cleanroom. A reagent blank of high purity 2% $HNO_3$/1%HF (also preconcentrated x125) was measured. The elemental content of the extraction solutions in Table 1 is shown after correcting for the x125 preconcentration factor and is not blank subtracted.

## Table 1

Elemental concentrations in the acid extraction solutions from three 500 mL lab bottles. LR, MR, and HR denote the mass resolution used (low, medium, high). Bottle concentration data shown was not blank subtracted.

| Analyte | Blank | Bottle #1 | Bottle #2 | Bottle #3 | Analyte | Blank | Bottle #1 | Bottle #2 | Bottle #3 |
|---|---|---|---|---|---|---|---|---|---|
| (Resolution Used) | ppt | ppt | ppt | ppt | (Resolution Used) | ppt | ppt | ppt | ppt |
| Ag107(LR) | <20 | <20 | <20 | <20 | Na23(LR) | <10 | <10 | <10 | <10 |
| Al27(LR) | <20 | <20 | <20 | <20 | Nb93(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| As75(LR) | <8 | <8 | <8 | <8 | Nd142(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Au197(LR) | <5 | <5 | <5 | <5 | Ni58(MR) | <5 | <5 | <5 | <5 |
| B11(LR) | <20 | <20 | <20 | <20 | P31(MR) | <50 | <50 | <50 | <50 |
| Ba138(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Pb208(LR) | <0.5 | <0.5 | <0.5 | <0.5 |
| Be9(LR) | <2 | <2 | <2 | <2 | Pd106(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Bi209(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Pr141(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Ca44(LR) | 30 | 70 | 20 | 20 | Pt195(LR) | <5 | <5 | <5 | <5 |
| Cd114(LR) | <0.6 | <0.6 | <0.6 | <0.6 | Rb85(LR) | <1 | <1 | <1 | <1 |
| Ce140(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Re187(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Co59(MR) | <1 | <1 | <1 | <1 | Rh103(LR) | <0.2 | <0.2 | <0.2 | <0.2 |
| Cr52(MR) | <2 | <2 | <2 | <2 | Ru102(LR) | <0.8 | <0.8 | <0.8 | <0.8 |
| Cs133(LR) | <0.2 | <0.2 | <0.2 | <0.2 | Sb121(LR) | <2 | <2 | <2 | <2 |
| Cu63(MR) | <2 | <2 | <2 | <2 | Sc45(MR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Dy164(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Sm152(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Er166(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Sn120(LR) | <5 | <5 | <5 | <5 |
| Eu153(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Sr88(LR) | <0.2 | <0.2 | <0.2 | <0.2 |
| Fe56(MR) | <2 | <2 | <2 | <2 | Ta181(LR) | <2 | <2 | <2 | <2 |
| Ga69(LR) | <2 | <2 | <2 | <2 | Tb159(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Gd158(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Te125(LR) | <5 | <5 | <5 | <5 |
| Ge74(LR) | <1 | <1 | <1 | <1 | Th232(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Hf180(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Ti48(MR) | <1 | <1 | <1 | <1 |
| Ho165(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Tl203(LR) | <0.2 | <0.2 | <0.2 | <0.2 |
| In115(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Tm169(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| K39(MR) | <5 | <5 | <5 | <5 | U238(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| La139(LR) | <0.1 | <0.1 | <0.1 | <0.1 | V51(MR) | <1 | <1 | <1 | <1 |
| Li7(LR) | <1 | <1 | <1 | <1 | W184(LR) | <1 | <1 | <1 | <1 |
| Lu175(LR) | <0.1 | <0.1 | <0.1 | <0.1 | Y89(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Mg24(LR) | <2 | <2 | <2 | <2 | Yb174(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Mn55(MR) | <1 | <1 | <1 | <1 | Zn66(MR) | <5 | <5 | <5 | <5 |
| Mo98(LR) | <1 | <1 | <1 | <1 | Zr90(LR) | <0.1 | <0.1 | <0.1 | <0.1 |
| Na23(LR) | <10 | <10 | <10 | <10 | | | | | |

## Results

Examination of the reagent blank values in Table 1 shows that every element except for Ca (30ppt) was below the detection limit. Ca is challenging at low ppt level due to contamination in the sample prep stage. Concentrations reported in the three bottle extraction solutions show that only Ca was measured above the blank and in only one of the bottles. Therefore, it can be assumed that Ca measured in one of the bottles was due to contamination at the sample prep step. This data demonstrates that, after a simple initial cleaning procedure, Savillex lab bottles do not produce any elemental contamination measurable by HR-ICP-MS, even after preconcentration.

## Summary

The stretch blow molding technique enables fluoropolymer bottle manufacturing to be performed in a cleanroom and eliminates the need to machine the bottle lip or use a cap insert. When combined with extensive precautions to eliminate airborne contamination during manufacture and the use of the highest purity virgin fluoropolymer resin, it enables the production of ultraclean bottles. After cleaning, the Savillex lab bottles tested were found to contain no elemental contamination measurable by HR-ICP-MS. Their superior seal integrity and drip-free pouring make them uniquely suited for the most demanding ultra trace analysis applications and the storage of ultrapure reagents.