Purillex® Bottle Trace Metals Analysis

APPLICATION NOTE

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Savillex

APPLICATION NOTE

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ABSTRACT

Purillex bottle production

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 level for almost every measurable element. Elemental contributions from reagents (DI water, acids) and reagent storage containers therefore must be strictly controlled in order not to impact ICP-MS data integrity. PFA and FEP fluoropolymer bottles are used for the shipment of high purity acid and in every trace metals lab for the storage of trace metal standards and rinse solutions. The design and manufacture of fluoropolymer bottles with the lowest possible trace metal contribution is therefore as important as the purity of reagents used in the analysis.

Savillex's Purillex® PFA and FEP bottles are manufactured from the highest purity grades of virgin fluoropolymer resin in an Class 10,000 ISO 7 cleanroom using unique stretch blow molding technology. They represent the 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 Purillex PFA 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.

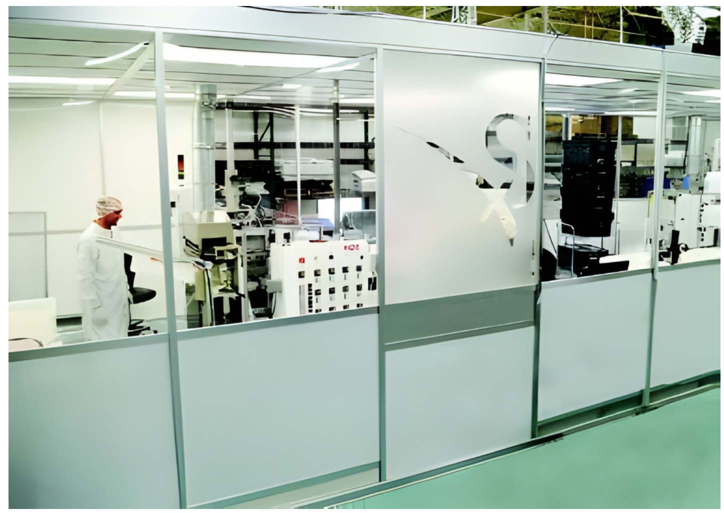
STRETCH BLOW MOLDING: BENEFITS FOR BOTTLE CLEANLINESS

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 injection molding. After molding, the neck and lip must be machined 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 technology of stretch blow molding to fluoropolymer bottle manufacturing. Since there is no commercially available stretch blow molding machinery that can handle the high temperatures required for fluoropolymer molding, Savillex designed and built stretch blow molding machinery in-house to produce PFA and FEP Purillex bottles. Stretch blow molding is a two step process. First, a "preform" is produced by high precision injection molding. The neck and thread of the finished bottle are molded in this step. Secondly, a preform is transferred to a cleanroom and is then blown into the final bottle in the stretch blow molding step. Because the bottle lip and thread are injection molded, seal quality is excellent. No machining of the lip is needed and no closure insert is required. Unlike other fluoropolymer bottles, Purillex bottles and closures are made from the highest purity grade fluoropolymer. Only virgin resin is used. No reground or recycled waste fluoropolymer is ever added.

CLEANROOM MANUFACTURE

A 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 portable clean hoods, clean enclosures and anti-static devices. Transfer from a portable clean hood direct to the cleanroom takes place in precleaned bins. The cleanroom is Class 10,000 ISO 7 with continuous particle count monitoring. After the preforms are blown into bottles, they are capped and sealed in HDPE bags prior to leaving the cleanroom.



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

BOTTLE CLEANING PRIOR TO USE

The combination of high purity virgin resin, exhaustive precautions against contamination and cleanroom manufacture enables the production of bottles with ultra-low metal content, even prior to cleaning. Nevertheless, Purillex 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 50°C for seven days to accelerate the acid removal of any residual contamination. The contents are discarded, and after a thorough rinse with DI water the bottle is ready for use.

TRACE METAL ANALYSIS OF PURILLEX BOTTLES

To demonstrate the cleanliness of Purillex bottles following initial cleaning, three 500 mL PFA Purillex 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 are provided on the next page.

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% HNO3/1%HF in DI water. The closures were replaced and the bottles maintained 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% HNO3/1%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 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 was performed in a cleanroom. A reagent blank of high purity 2% HNO3/1%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 ACID EXTRACTION SOLUTIONS FROM THREE 500 mL PURILLEX PFA BOTTLES

*LR, MR, and HR denotes mass resolution used. Bottle data shown was not blank subtracted.

Analyte (Resolution Used)	Blank (ppt)	Bottle #1 (ppt)	Bottle #2 (ppt)	Bottle #3 (ppt)
Ag107 (LR)	<0.3	<0.3	<0.3	<0.3
Al27 (LR)	0.8	<0.5	0.8	0.8
As75 (HR)	<10	<10	<10	<10
Au197 (LR)	<0.5	<0.5	<0.5	<0.5
B11 (LR)	40	20	40	30
Ba138 (LR)	0.03	0.06	0.06	0.1
Be9 (LR)	<0.6	<0.6	<0.6	<0.6
Bi209 (LR)	<0.2	<0.2	<0.2	<0.2
Ca44 (LR)	9	9	11	10
Cd114 (LR)	<0.2	<0.2	<0.2	<0.2
Ce140 (LR)	0.2	0.1	0.2	0.2
Co59 (MR)	<0.1	<0.1	<0.1	<0.1
Cr52 (MR)	0.9	<0.6	0.9	0.4
Cs133 (LR)	<0.01	<0.01	<0.01	<0.01

Analyte (Resolution Used)	Blank (ppt)	Bottle #1 (ppt)	Bottle #2 (ppt)	Bottle #3 (ppt)
Na23 (LR)	3	3	3	3
Nb93 (LR)	<0.01	<0.01	<0.01	<0.01
Nd142 (LR)	0.02	0.02	0.03	0.03
Ni58 (MR)	7	5	7	6
Pb208 (LR)	<0.08	0.133	0.168	<0.08
Pd106 (LR)	<0.04	<0.04	<0.04	0.05
Pr141 (LR)	<0.01	<0.01	<0.01	<0.01
Pt195 (LR)	<1	<1	<1	<1
Rb85 (LR)	<0.1	<0.1	<0.1	<0.1
Re187 (LR)	<0.2	<0.2	<0.2	<0.2
Rh103 (LR)	<0.2	<0.2	<0.2	<0.2
Ru102 (LR)	<0.1	<0.1	<0.1	<0.1
Sb121 (LR)	<0.09	<0.09	<0.09	<0.09
Sc45 (MR)	<0.04	<0.04	<0.04	<0.04

TABLE 1 - ELEMENTAL CONCENTRATIONS IN ACID EXTRACTION SOLUTIONS FROM THREE 500 mL PURILLEX PFA BOTTLES (CONTINUED)

Analyte (Resolution Used)	Blank (ppt)	Bottle #1 (ppt)	Bottle #2 (ppt)	Bottle #3 (ppt)
Cu63 (MR)	<0.5	<0.5	<0.5	<0.5
Dy164 (LR)	<0.01	<0.01	<0.01	<0.01
Er166 (LR)	<0.01	<0.01	<0.01	<0.01
Eu153 (LR)	<0.01	<0.01	<0.01	<0.01
Fe56 (MR)	2	<0.8	2	3
Ga69 (LR)	<0.3	<0.3	<0.3	<0.3
Gd158 (LR)	0.02	<0.02	0.03	0.03
Ge74 (LR)	<0.6	<0.6	<0.6	<0.6
Hf180 (LR)	<0.02	<0.02	<0.02	<0.02
Ho165 (LR)	<0.07	<0.07	<0.07	<0.07
In115 (LR)	<0.01	<0.01	<0.01	<0.01
K39 (MR)	<0.8	<0.8	<0.8	<0.8
La139 (LR)	<0.02	<0.02	<0.02	<0.02
Li7 (LR)	<0.1	<0.1	<0.1	<0.1
Lu175 (LR)	<0.01	<0.01	<0.01	<0.01
Mg24 (LR)	0.2	0.3	0.6	0.5
Mn55 (MR)	<0.1	<0.1	<0.1	<0.1
Mo98 (LR)	<0.1	0.1	0.4	1.0

Analyte (Resolution Used)	Blank (ppt)	Bottle #1 (ppt)	Bottle #2 (ppt)	Bottle #3 (ppt)
Sm152 (LR)	<0.02	<0.02	<0.02	<0.02
Sn120 (LR)	<1	<1	<1	v
Sr88 (LR)	<0.5	<0.5	<0.5	<0.5
Ta181 (LR)	<0.03	<0.03	<0.03	<0.03
Tb159 (LR)	<0.01	<0.01	<0.01	<0.01
Te125 (LR)	<0.3	<0.3	<0.3	<0.3
Th232 (LR)	<0.1	<0.1	<0.1	<0.1
Ti48 (MR)	2.5	0.5	5.4	2.4
Tl203 (LR)	<0.2	<0.2	<0.2	<0.2
Tm169 (LR)	<0.02	<0.02	<0.02	<0.02
U238 (LR)	<0.05	<0.05	<0.05	<0.05
V51 (MR)	<0.04	<0.04	<0.04	<0.04
W184 (LR)	0.4	0.1	0.5	0.4
Y89 (LR)	<0.01	<0.01	<0.01	<0.01
Yb174 (LR)	<0.01	<0.01	<0.01	<0.01
Zn66 (MR)	0.4	0.5	0.5	0.3
Zr90 (LR)	<0.01	<0.01	0.03	0.02

RESULTS

Examination of the reagent blank values in Table 1 shows the large majority of elements to be close to or below the detection limit. The only elements present in measurable amounts in the blank were B (40 ppt), Ca (9 ppt) and Ni (7 ppt). The concentrations reported in the three bottle extraction solutions show that there was no element measurable above the blank. This testing data demonstrates that, after a simple initial cleaning procedure, Purillex PFA bottles do not produce any elemental contamination measurable by HR-ICP-MS, even after preconcentration.

SUMMARY

The stretch blow molding technique enables final bottle manufacturing to be performed in a cleanroom and eliminates the need to machine the bottle lip or use a cap insert. Combining extensive precautions to eliminate airborne contamination during manufacture with the use of highest purity virgin fluoropolymer resin has enabled the production of ultraclean bottles. After cleaning, the Purillex PFA bottles tested were found to contain no elemental contamination measurable by HR-ICP-MS, thus making them uniquely suited for the most demanding ultratrace analysis applications and storage of ultrapure reagents.

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