

Savillex Technical Note

Testing Savillex PFA Lab Bottles: ASTM D499I Vacuum Leak Test

Summary

A high integrity bottle seal is critical when using fluoropolymer bottles in a lab environment or as packaging for the shipment of high purity acids or samples. Savillex lab bottles feature a unique ferrule closure and are manufactured to produce a high precision injection molded lip and thread, which gives them excellent seal performance. To test the closure seal of Savillex lab bottles, three 500 mL PFA lab bottles were submitted to an independent testing lab for testing to ASTM Method D4991 (Vacuum Leak Test). All three bottles passed.

Importance of Bottle Seal Integrity

Fluoropolymer bottles (particularly PFA) can be found in virtually every trace metals testing lab. They are used for storing trace metal standard solutions and wash solutions used with inductively coupled plasma mass spectrometry (ICP-MS), where freedom from trace metal contamination is paramount. High purity acid manufacturers also use fluoropolymer bottles to ship high purity acid to labs worldwide,

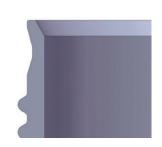
again for use with ICP-MS. When shipping acids, the seal performance of the bottle is critical from a safety aspect to prevent spillages and avoid contamination of



Savillex 500 ml PFA Lab Bottle

the contents. In addition, semiconductor testing labs send out empty, precleaned fluoropolymer bottles worldwide for their customers to fill with acid and return to them for analysis. Once again, a high integrity seal is critical for the above reasons. Sealing performance within the analytical lab is equally important to prevent contamination and avoid concentration changes due to evaporative losses, where fluoropolymer bottles are used to store isotopic standards prepared by weight.

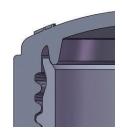
Savillex lab bottles are uniquely manufactured using a stretch blow molding process where the lip, thread, and neck of the bottle are injection molded. This gives much greater seal integrity than conventional fluoropolymer bottles, where the lip, thread, and neck are formed less precisely by extrusion molding. In addition, the Savillex bottle closure is molded from the same PFA material and grade as the bottle, with a ferrule style seal that locates in the angled bottle lip. A full 3.5 turns of thread engagement ensures that the closure is very firmly secured onto the bottle.



Savillex Bottle Neck - Lip Detail



Savillex Bottle Neck Closure

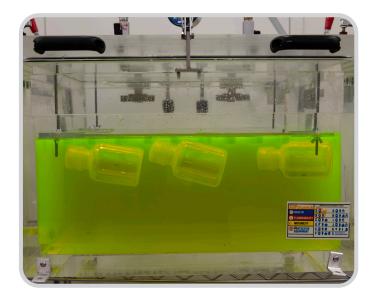


Savillex Bottle Closure - Detail

In burst test studies, Savillex lab bottles have been shown to withstand significantly higher pressure than conventional fluoropolymer bottles before leaking – to 90 psi and beyond. To demonstrate the seal integrity of Savillex lab bottles, they were submitted to an independent test lab for leak testing.

ASTM D4991 Vacuum Leak Test Method

The ASTM D4991-07 (2015) Standard Test Method for Leakage Testing of Empty Rigid Containers by Vacuum Method is a standard test method that covers the testing of empty rigid containers under differential pressure conditions such as those that can occur during air transport. It is an important and widely used test for containers used for shipping and is a good proxy for bottle seal integrity. Empty rigid containers are prepared by sealing the closures utilizing the manufacturer's recommended torque setting. The containers are then immersed in an ethylene glycol-water solution in a sealed, transparent test chamber. The chamber is then subjected to a gradually increasing partial vacuum. Bottles are shown inside the test chamber in the images below. During de-pressurization, the containers are observed for signs of leakage in the form of air bubbles leaking from the closure seal. Once testing is complete, each container is also inspected for the presence of fluid in the container. Passing containers show no signs of bubbles during depressurization and no signs of fluid leakage into the container.





Savillex PFA lab bottles shown immersed in ethylene glycol in test chamber

Savillex PFA Lab Bottles Pass ASTM D4991 Testing

Three 500 mL PFA lab bottles were sent to an independent testing lab (DDL, Eden Prairie, MN, USA) for testing to ASTM 4991. The bottles were capped, and the closures torqued to specification, then immersed in ethylene glycol-water solution inside the vacuum chamber. The vacuum chamber was sealed to prevent air migration into the chamber. A vacuum was then applied and slowly increased until the gauge read 28.16 in/Hg. The vacuum chamber was maintained at this setting for 30 minutes. After 30 minutes, the vacuum was turned off and released slowly. The bottles remained in the chamber at ambient pressure for several minutes before being removed and dried off. They were then inspected for any sign of leakage into the package.

Test Results

A test summary is shown below. All bottles passed the D4991 testing with no leaks observed. Note that the comment "liquid in threads" refers to immersion glycol liquid trapped in the threads between the bottle and closure and is not evidence of a leak.

Sample/Sub Sample	Pass	Fail	Comments
001-1	X		Liquid in threads
001-2	X		Liquid in threads
001-3	X		Liquid in threads
Summary			
Total	Pass	Fail	Failed

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Passing the ASTM D4991 test demonstrates that the design of the Savillex bottle thread, lip, and closure produces a high integrity seal that can be relied upon whether using the bottles in a lab environment or shipping high purity acids around the world.

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Conclusion

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The ASTM D4991 test method is an important method for testing containers for critical shipping applications. Although the test is a difficult one for standard containers, Savillex lab bottles easily passed. This is due to their ferrule-style closures, durable construction, and closure torque settings validated by CCIT.