Sample Preparation: The Forgotten Part of Method Development

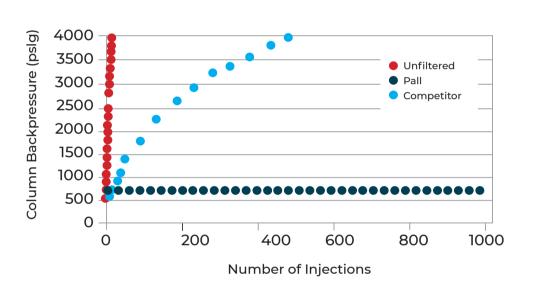
INTRODUCTION

High volume laboratories must keep instruments running while producing quality data. Using Pall Acrodisc® syringe filters as part of the sample preparation process makes this possible by preventing chromatography column blockages without contributing extractable materials. Pall Acrodisc syringe filters increase HPLC column life up to 52 times and UHPLC columns over 111 times compared to unfiltered samples. Additionally, Pall Acrodisc syringe filters, which include the most common membrane chemistries, combine high particulate retention with minimal release of extractable materials. This makes them ideal for a variety of chromatography and sample preparation applications, including GC, ICP and specialized MS and ion chromatography techniques.

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EFFECT OF FILTRATION ON HPLC COLUMNS

Effects of filters on HPLC column life following injections of unfiltered and filtered 0.05% latex sphere suspensions(1). With unfiltered samples, the column failed due to plugging after 19 injections. Samples passed through Competitor filters plugged the columns after 500 injections. No increase in backpressure was observed after 1000 injections of samples filtered with Pall Acrodisc® One syringe filters.



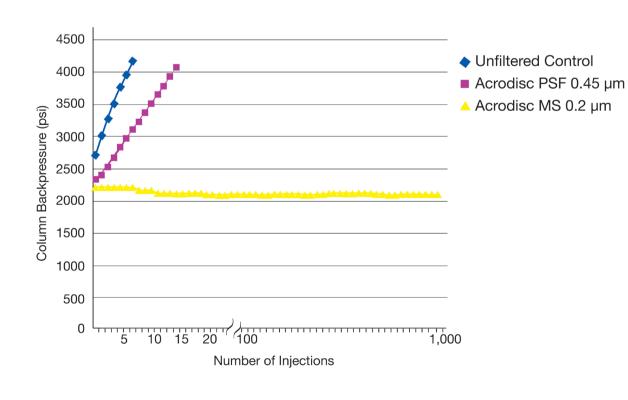


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UHPLC COLUMN LIFE EXTENSION

Columns last 111 times longer than without filtration

Figure 2
Effect of Filtration on UHPLC Column Life

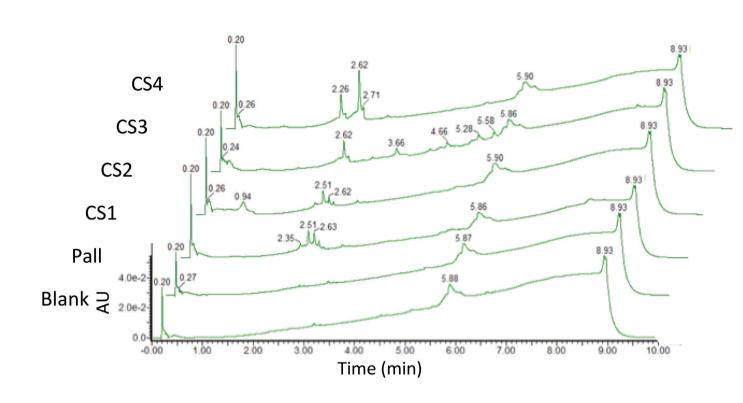


UHPLC COLUMN LIFE EXTENSION

A 0.05% microsphere suspension (average diameter 0.31 µm) in 0.002% Tritonu X-100 (prepared from a 10% w/w polymer stock), was filtered using either 0.2 µm Acrodisc MS syringe filters or 0.45 µm Acrodisc PSF syringe filters with nylon membrane. Injection of the unfiltered and the 0.45 µm filtered suspensions resulted in a rapid and significant increase in the backpressure of the Acquity UPLC column, after 9 and 16 injections, respectively. By contrast, column plugging was not observed even after 1,000 injections of the effluents from the Acrodisc MS syringe filters.

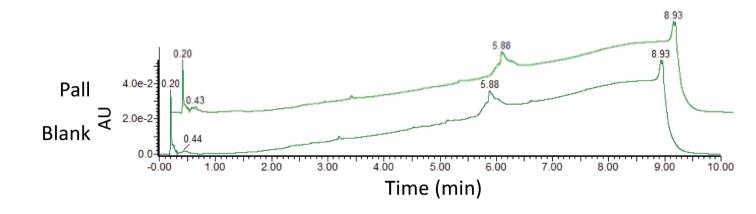
PALL ACRODISC SYRINGE FILTERS CONTRIBUTE MINIMAL EXTRACTABLE MATERIAL

Figure 3Chromatograms of Pall nylon extractables and competitive products



Solvent extractable properties of syringe filters equipped with 0.2 µm nylon membrane. Ten microliter injection volumes of the methanol solvent blank (Blank) and filtrates obtained with the Pall Acrodisc (Pall) or commercially available syringe filters (CS1-4) were analyzed using a Waters™ Acquityu UPLC H-Class system with a Tunable UV Detector and a 2.1 x 50 mm, 1.7 µm Waters Acquity UPLC BEH C18 reverse phase column under gradient conditions with a mobile phase consisting of water and acetonitrile with a flow rate of 0.6 mL/min and a column temperature of 35 °C. Initial conditions of 5% acetonitrile were held for 0.5 min, followed by a linear gradient of 5 - 100% acetonitrile over 6.9 min, and then to remain at 100% acetonitrile for 0.9 min. Data was collected at a wavelength of 214 nm.

Figure 4Chromatogram of extractables in Pall PTFE



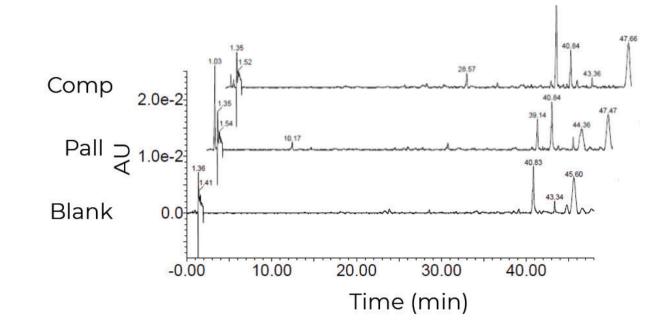
Solvent extractable properties of Pall Acrodisc syringe filters with 0.2 μ m hydrophobic PTFE membrane. Filtrate (Pall) and solvent blank (Blank) (10 μ L injection volume) were analyzed using a Waters Acquity UPLC H-Class system with a Tunable UV Detector and a 2.1 x 50 mm, 1.7 μ m Waters Acquity UPLC BEH C18 reverse phase column under gradient conditions with a mobile phase consisting of water and acetonitrile with a flow rate of 0.6 mL/min and a column temperature of 35 °C. Initial conditions of 5% acetonitrile were held for 0.5 min, followed by a linear gradient of 5-100% acetonitrile over 6.9 min, and then to remain at 100% acetonitrile for 0.9 min. Data was collected at a wavelength of 214 nm.

PALL ACRODISC SYRINGE FILTERS CONTRIBUTE MINIMAL EXTRACTABLE MATERIAL (CONTINUED)

Figure 5

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Chromatogram of extractables in Pall wwPTFE membrane



Solvent extractable properties of syringe filters equipped with 0.45 μ m hydrophilic PTFEmembranes (Pall and Competitor). The solvent, Methanol and acetonitrile, and solvent blank (Blank)were analyzed using a Waters Acquity UPLC system with a Diode Array Detector and a 2.1 x 50 mm, 4 μ m Waters Nova-Pak C18, 4.6 mm X 150 mm reverse phase column under gradient conditions with a mobile phase consisting of water and acetonitrile with a flow rate of 1 mL/min and a column temperature of 30 °C. Initial conditions of 5% acetonitrile were held for 3 min, then to remain at 100% acetonitrile for 91 min. The column was then equilibrated for 108 min at 5% acetonitrile before injection of the next sample. Data was collected at a wavelength of 214 nm.

ACRODISC MS (wwPTFE)

HDPE housing and WWPTFE membrane

- Extremely low extractables
- Excellent chemical compatibility

Certified for MS analysis

- Certificate of analysis included
- Testing by mass spectrometry
- Representative chromatograms in 50% MeOH in water and 100% MeOH

Figure 6 Competitive chromatograms in 50% MeOH

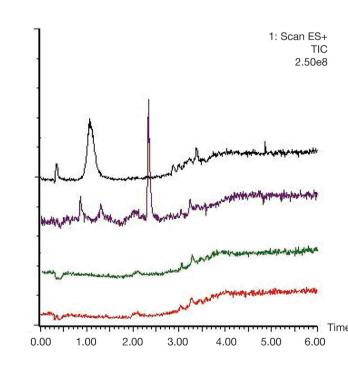
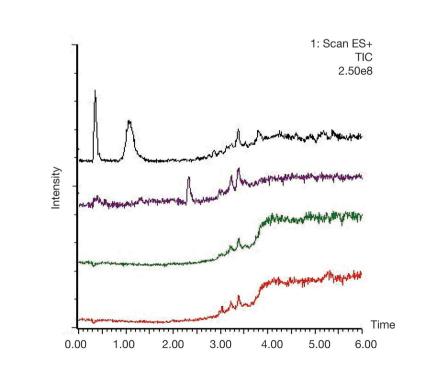


Figure 7 Competitive chromatograms in 100% MeOH



IC ACRODISC (SUPOR®)

Hydrophilic polyethersulfone

Phosphate

 Certified for low levels of common ions of interest: Chloride Nitrate Sulfate



Analysis of 4 Anions in 18 Megohm Water

	(mg/L)					
	CL.	NO ₃	PO ₄ 3-	SO ₄ ²⁻		
Sample 1	0.0	0.000	0.0	0.00		
Sample 2	0.0	0.000	0.0	0.00		

Concentration of 4 Anions in the Standard Solution

(119/1)				
CL.	NO ₃ -	PO ₄ 3-	SO ₄ ²⁻	
0.2	0.973	0.5	1.53	
0.2	0.951	0.5	1.5	
0.2	0.962	0.5	1.52	
	CL ⁻ 0.2 0.2	CL ⁻ NO ₃ ⁻ 0.2 0.973 0.2 0.951	CL ⁻ NO ₃ ⁻ PO ₄ ³⁻ 0.2 0.973 0.5 0.2 0.951 0.5	

Concentration of 4 Anions After Passing the Standard Solution Through the Test Filters

	(mg/L)					
	CL.	NO ₃	PO ₄ ³⁻	SO ₄ ²⁻		
Pall Laboratory	0.3	0.991	1.5	1.56		
Competitor 1	0.9	1.03	1.5	1.67		
Competitor 2	0.3	1.207	0.0	1.47		
Competitor 3	0.3	1.000	1.5	1.79		

Difference Between Table 2 and Table 3 for Test Filters

	(mg/L)				
	CL.	NO ₃ -	PO ₄ ³⁻	SO ₄ ²⁻	
Pall Laboratory	0.1	0.029	0	0.04	
Competitor 1	0.7	0.068	0	0.15	
Competitor 2	0.1	0.245	-1.5	0.05	
Competitor 3	0.1	0.038	0	0.27	

CONCLUSION

Filtration as part of the sample preparation process is a simple, easy and cost-efficient way to maximize the working life of your chromatography instrument.

Pall Acrodisc syringe filters remove column blocking particles from the sample, extending column life. Pall's range of membrane chemistries and application specific filters contribute minimal contaminating extractable materials, safeguarding your data.