



Application Note

Evaluation of Particulate Retention and Solvent Extractable Properties of Pall Acrodisc® Syringe Filters with Nylon Membrane

Introduction

In many laboratories, the need to consistently generate high quality data means that laboratory managers and technicians need to ensure their instruments are performing optimally around the clock. Filtration of samples and mobile phase prior to analysis helps increase the lifespan of chromatography columns, reduces overall instrument wear, and removes any particles that may interfere with the chromatogram.

Accurate, reproducible data depends upon proper HPLC column performance. Injection of samples containing particulates will eventually block the column inlet and column packing, thereby causing high column back-pressure and shortening the normal service life of the column¹. In fact, plugging of the HPLC column by particulate matter is the most frequent cause for column failure encountered by analysts. Sample filtration using syringe filters with 0.45 µm (HPLC) or 0.2 µm (UHPLC) pore size membrane, is a time effective and easy to implement method to protect the column. However, filters with the same nominal rating can vary drastically in their capacity to provide column protection.¹

In addition to sample filtration, mobile phase filtration through a 0.45 µm or 0.2 µm filter disc is also important to extend the life of the column, pump, injector, and other components from premature wear. Without filtration, accumulation of particulates cause high system pressure, shifted retention times, and poor peak shape and separation. Pall offers Nylaflor™ membranes in a 47 mm disc filter format as well as the SolVac® filter holder to help provide a particle free mobile phase.

Nylon is a hydrophilic membrane that is the industry standard for general use aqueous samples. It can be found in many laboratories performing different types of analyses including pharma laboratories using it in small molecule drug discovery for dissolution and formulation studies.

Nylon Extractables

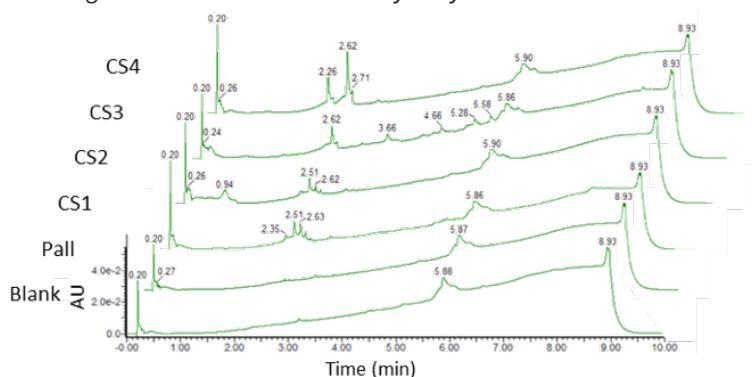
A property that sets Nylon membranes apart from many other polymeric filter media is that the nylon base material is intrinsically hydrophilic whereas the base material for most other polymeric media is hydrophobic and has to be made hydrophilic during membrane manufacture. Nylon's intrinsically hydrophilic nature eliminates the need for surfactants or wetting agents, which are chemicals that could extract from the membrane during sample preparation. However, nylon is not the material of choice for all purposes, as it does bind protein and has some limitations regarding compatibility with acids and organic solvents.

Although nylon does not require the use of surfactants or wetting agents during production, extractables stemming from the polymeric resins, solvents, pore formers, housing materials, and other chemical components utilized during device manufacturing still can be an area of concern if they leach into the sample during filtration. The cleanliness and chemical compatibility of a syringe filter directly affects data quality. Extractable materials can jeopardize analytical results through extraneous peaks and coelution and using an incompatible syringe filter can negatively impact critical data.

To determine if solvent extractables were present in the 0.2 μm Acrodisc syringe filter with Nylon membrane, extractions were carried out with methanol as the solvent followed by UHPLC analysis of the filtrates. Only the first mL of filtrate per filter was collected to enhance the ability to detect extractable materials. Potential variability was minimized by collecting the first mL filtrates of three syringe filters in an HPLC autosampler vial.

Figure 1

*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 Acquity \diamond UPLC \diamond 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 a cetonitrile with a flow rate of 0.6 mL/min and a column temperature of 35 $^{\circ}\text{C}$. 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. **Results may vary***



As shown in Figure 1, the Pall Acrodisc syringe filters with nylon membrane provide a clean chromatogram following extraction with methanol. All other tested commercial samples show several unidentified compounds leaching from the devices indicated by the additional peaks in the chromatograms at 214 nm. The presence of these additional peaks greatly complicates data analyses and reporting, requiring the analyst to identify these compounds and exclude them as originating from the sample. With the Pall Acrodisc syringe filters with nylon membrane having little to no effect on the sample, the analyst can be confident that the data generated is accurate without being compromised by the presence of unwanted chemicals.

Particulate Retention with Nylon Membrane Filters

Retention efficiency has a strong relationship to column life extension. Therefore we determined the ability of 0.45 μm and 0.2 μm Acrodisc syringe filters with nylon membrane to retain polystyrene latex beads by filtering suspensions of beads with average diameters of 0.46 μm and 0.24 μm , respectively. This was achieved by passing through each syringe filter a 3 mL volume of a 0.05% (w/w) polystyrene latex bead (Sigma) suspension in 0.1% Triton \diamond X-100 (Sigma). For each filter type, a total of five test pieces were evaluated. The bead concentrations before and after filtration were determined spectrophotometrically and used to calculate the latex bead retention efficiency.

Table 1

Latex sphere retention of syringe filters with 0.2 µm and 0.45 µm pore size ratings. Results may vary.

Replicate	Retention Efficiency (%)	
	0.2 µm	0.45 µm
1	90.8	92.2
2	81.6	92.1
3	89.0	92.0
4	75.4	91.6
5	88.9	91.8
Average	85.1	91.9
Std Dev	6.5	0.2

Table 1 shows the latex sphere retention of syringe filters with 0.45 µm and 0.2 µm pore size ratings. By injecting a cleaner sample into your LC system you extend the working life of the column, resulting in more instrument operation time.

Conclusions

The choice of whether to filter is an easy one to make. The benefits that filtration provide to the instrument and data are clear. However, the choice of which filter is the right filter requires more consideration. For general aqueous applications, nylon is often used, however we have shown that there can be significant variation between commercially available nylon membranes. The presence of additional peaks in the samples filtered with commercially available nylon membranes show that different chemistries were used during the manufacturing process. The benefits gained from the column protective properties are lost due to the extra time and cost associated with data manipulation and analysis. In some circumstances presence of the unwanted additional peaks can result in product quarantine.

Pall's Acrodisc syringe filters with nylon membrane:

- Provide excellent column protection
- Shows no detectable contaminants following extraction with methanol

References


1. Analytical Technical Guide, Pall Laboratory



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