thermo scientific

WHITE PAPER 72898

Evaluating filtration options for ion chromatography

Author
David G. Moore,
Thermo Fisher Scientific

Keywords
Filtration, particulates, ultrafiltration, microfiltration, dialysis

Did you know? Microfiltration is defined as utilizing filters with pore sizes between 0.1 and 10 µm. Ultrafiltration requires filter pore sizes <0.1 µm.[1]

Introduction

Ion Chromatography (IC) is a rugged technique with a high level of sensitivity, meaning that sample dilution is often the only sample preparation that is needed. However, because some sample matrices such as waste or surface waters, food and petroleum products can be extremely challenging, some form of sample preparation is necessary. The large diversity of matrices has led to many sample preparation options, and it can be difficult to identify the most appropriate technique. Here we will categorize the sample preparation challenges for samples with a high level of particulates, and the benefits and drawbacks of each solution available.

For typical environmental water analysis, the United States Environmental Protection Agency recommends point of collection filtration (US EPA 314.1; OASQA Sample Submission Procedures, ISO 5667-3:2018). This is a critical step in order to remove bacteria from the sample, which could otherwise metabolise (and alter the concentration of) ions such as nitrite, nitrate, orthophosphate, ammonium, and short-chain fatty acids. Since the samples have already been filtered at this level, further filtration is typically redundant.

ThermoFisher SCIENTIFIC

However, if filtration at the point of sample collection is not possible, then some form of particulate removal prior to introduction of the sample into the eluent stream is usually used to protect the downstream components. Additionally, some samples, particularly those high in iron, can form precipitates during storage even after initial filtration; secondary filtration prior to injection is necessary in such cases.

Offline sample filtration

Offline sample filtration is more labor intensive than inline alternatives. As such, labs may need to make judicious analysis of which samples need to be filtered and which don't, placing an additional training burdens on technicians. Labs that have high sample throughput should strongly consider using an alternative form of sample preparation.

Vacuum filtration

Vacuum filtration utilizes a filtration apparatus that applies a vacuum to assist in drawing a sample through a filter paper. It is no longer used for the majority of applications, that require sample filtration since the labor cost and sample volume requirements are too high.

Syringe filters

Syringe filters[2] are used extensively for the removal of particulates in ion chromatography samples. They are single-use, disposable filters that are attached directly to a luer-lock syringe after sample aspiration (Figure 1). Syringe filters don't require any special training, and don't require any additional instrumentation; they are also specifically referenced by EPA method 300.1.

Figure 1. Luer-lock syringe with syringe filter attached.

However, being single-use only, syringe filters can be costly and can contribute to a lot of waste. Additionally, there is a potential for them to burst when subjected to high pressure, as can be applied by technicians who are trying to filter high particulate samples.

Pre-injection inline filtration

Inline filtration allows for automation of filtration into the IC workflow without additional labor requirements. Inline filters can be used for multiple samples, but will eventually need to be replaced at a frequency that depends on the samples being filtered. Pre-injection filtration means that only filtered samples are introduced into the ion chromatograph, removing any possibility of system damage due to particulates.

Tangential flow microfiltration

Tangential flow microfiltration continually passes a high volume of sample across a filter membrane (for IC a 0.2 µm pore size filter is commonly used) and a negative pressure is applied on the other side of the filter to bring some of the sample across the membrane. Since the sample is flowing across the filter, rather than trying to push through it, heavily contaminated samples can be used without clogging the membrane.

Unfortunately, it is impossible to determine exactly how much filtrate will be generated for a given quantity of sample, and so a large sample volume must be used to be sure of completely filling the sample loop (including any necessary overfill). Even more importantly, it is necessary to run a water injection between every sample to guarantee that there is no sample carryover.

Stopped-flow dialysis

Stopped-flow dialysis utilizes the tendency for solutes to move down a concentration gradient to reach equilibrium. The sample and acceptor solution (ultrapure water) are placed on two sides of a semipermeable membrane and allowed to sit until equilibrium is achieved and the concentration of ions in the acceptor solution matches the concentration of the original sample. The acceptor solution is then injected directly into the IC. Stopped-flow dialysis can be used for highly contaminated samples that would clog typical filters.

Stopped-flow dialysis carries high set-up and instrumentation requirements. It is necessary to perform numerous experiments to determine the optimum transfer times from the dialysis membrane to the injection valve. The optimum dialysis time also varies depending on analyte concentration, sample characteristics, and membrane condition. Multiple additional pumps are required for sample and acceptor solution delivery. Lastly, sample carryover is possible and cannot be ruled out unless an analysis of a water blank is made between every sample injection.

Sample vial filter caps

Sample vial filter caps[3] (plunger caps with an integral filter) are installed in the top of an inert sample vial. The autosampler arm applies a sampling tube which forms a seal with the filter cap and pushes the cap into the vial, displacing the sample upwards through the filter and into the IC system (Figure 2).

Figure 2. Autosampler sample displacement filtration.

This approach is amenable for a wide range of samples, including those which are heavily loaded with particulates. Filtration occurs from the top down, so filtration efficiency is assisted by gravity which acts to deposit larger particles in the bottom of the tube; thus, the filter acts on the least contaminated part of the sample. Each sample has its own individual filter which eliminates sample carry over related to the filter.

Post-injection inline filtration

High pressure inline filters

High pressure inline filters[4] are low-volume (<1.5 µL) filters placed in the high-pressure flow path of the ion chromatograph, behind the injection valve. In this location, the filter serves to remove fine particulates from the sample and protects the downstream components from clogging due to particulate accumulation (Figure 3). The low volume permits filtration of just the injected sample volume, generally 10 to 25 µL, helping to prolong filter lifetime.

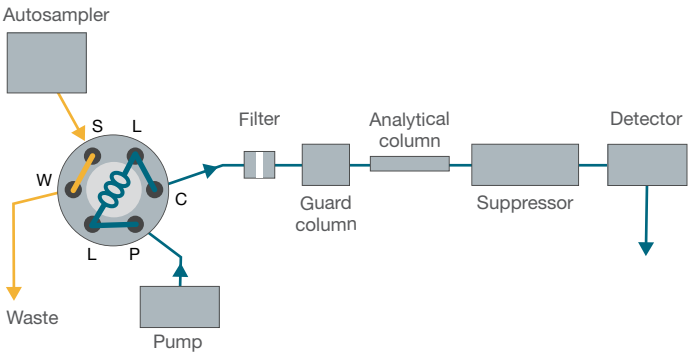

Figure 3. Inline high-pressure filtration schematic.

Inline filtration installed on the high-pressure side of the injection valve provides three major benefits:

- The filter is constantly exposed to eluent flow so sample carry over is eliminated
- The filter is reusable for many samples
- System pressure can be used to monitor filter condition.

Monitoring system pressure allows the user to ascertain when the time has come to service the filter. Increased system pressure implies that the filter frit is loading up with particulates and needs to be changed. Maintenance of the inline filter is very simple—one of the tubing connectors is removed, allowing that half of the filter housing to be unscrewed. The filter frit is replaced, the housing is screwed back together, and the tubing reinstalled. The system is ready for operation in minutes.

However, high-pressure inline filters are not suitable for very highly contaminated samples, where a pre-injection method is more suitable.

Dual inline filters with backflush

A filter backflush approach can be implemented where particulate loading of samples is heavier or concerns about sample carry over remain. Utilizing two filters, a 2-position 10-port high pressure valve, and an auxiliary high-pressure pump (Figure 4), this approach allows for filters to be backflushed with ultrapure water to eliminate carryover and increase filter lifetimes by up to a factor of 100×.

The effectiveness of the filtration backflush process can be monitored by observing the system pressure over a series of injections. A secondary method for monitoring filter effectiveness is to observe the pressure of the back-flushing stream. A low and stable pressure demonstrates once again that the filter is not clogging with particulates.

This approach allows for the analysis of samples that have a higher particulate load than can be used with a single inline filter. However, additional instrumentation is required, and this approach is still not recommended for extremely highly contaminated samples. For these samples, one of the pre-injection inline sample preparation techniques, such as the vial filter caps, should be used.

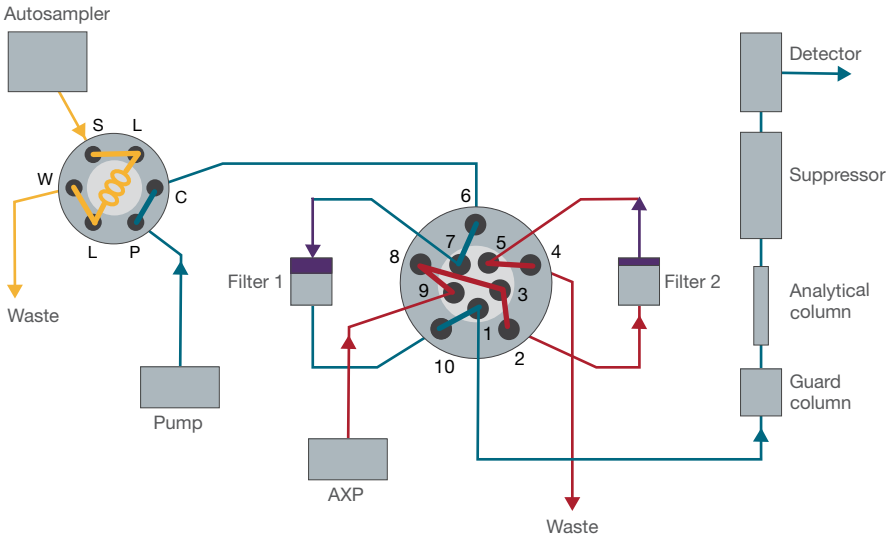

Figure 4. Inline high-pressure filtration schematic, dual filter with backflush.

Table 1. Thermo Fisher Scientific offers the widest choice of particulate removal capabilities, including offline, pre-injection, and post-injection options, ensuring that you can find the most suitable solution for your application.

High particulate removal technique		Summary	Maximum particulate contamination	
Offline	Vacuum filtration	Very high labor requirements	Medium	ThermoFisher SCIENTIFIC
	Syringe filters	No carry-over, high cost and labor requirements	Medium	ThermoFisher SCIENTIFIC
Pre-injection inline	Tangential-flow microfiltration	Labor saving, but potential for carryover and requires large sample volume and additional dual-channel pump	Medium	
	Stopped-flow dialysis	Labor saving, but potential for carryover and requires additional two dual-channel pumps	High	
	Sample vial filter caps	Labor saving and no carry-over, but single-use	High	ThermoFisher SCIENTIFIC
Post-injection inline	High-pressure inline filters*	Labor saving, low maintenance, no sample loss, and no additional instrumentation required	Low	ThermoFisher SCIENTIFIC
	Dual inline filters with backflush	Labor saving, no carry-over, no sample loss, additional valve and pump required	Medium	ThermoFisher SCIENTIFIC

* Filters can be placed before the injection valve if desired.

References

1. Encyclopedia of Separation Science, p. 196.
2. Available from Thermo Fisher Scientific at thermofisher.com/order/catalog/product/F2500-14.
3. Available from Thermo Fisher Scientific at thermofisher.com/order/catalog/product/038008.
4. Available from Thermo Fisher Scientific as a kit. For more information, see Technical Note 144.

Find out more at **thermofisher.com/competitiveIC**

WP72898-EN 1218M

ThermoFisher SCIENTIFIC