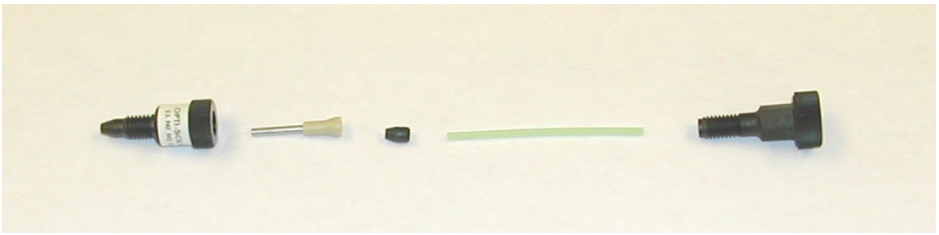
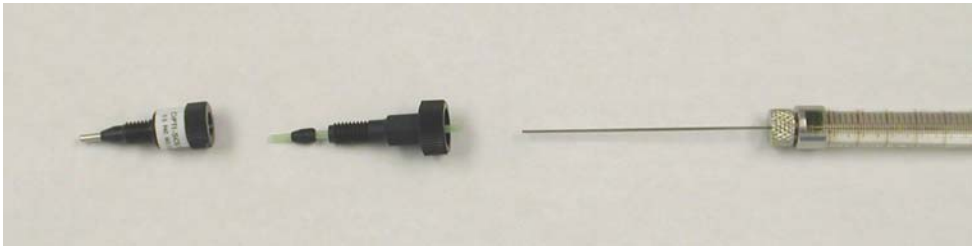
Technical Bulletin G0520 - Filtration for Manual Injections

The filter on the CapNMR™ probe was designed for inline protection to prevent particles greater then 2 micron from getting into the probe. However, a crude solution that has a lot of particulates could block the filter rather quickly. There is an option to filter the sample at the point of injection. The Opti-Solv Filter from Optimize Technologies (Part# 10-04-03389) is recommended for this use. [Optimize Technologies (800) 669-9015] The filters are rather expensive at $195 for a 5-pack but the filters are very robust and are easily back-flushed and regenerated (see below for directions). *This part number is not in their catalog or on their website.* They create this filter for another chromatography supply company but are readily purchased directly from Optimize by anyone.



Assemble the Optimize Filter so the metal filter stem is inside the black filter fitting. For the syringe connection, use the standard F-287 black nut and ferrule with a F-247 green sleeve for the syringe fitting.



The filter then gets screwed directly in the injection port.

The filter stem floats inside the fitting and should fall into place. Pushing the needle into the fitting just before it is tightened down can ensure that the stem is seated at the bottom of the port

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Once the filter is secured, place the needle and its black nut, ferrule, and green sleeve fitting into the filter.

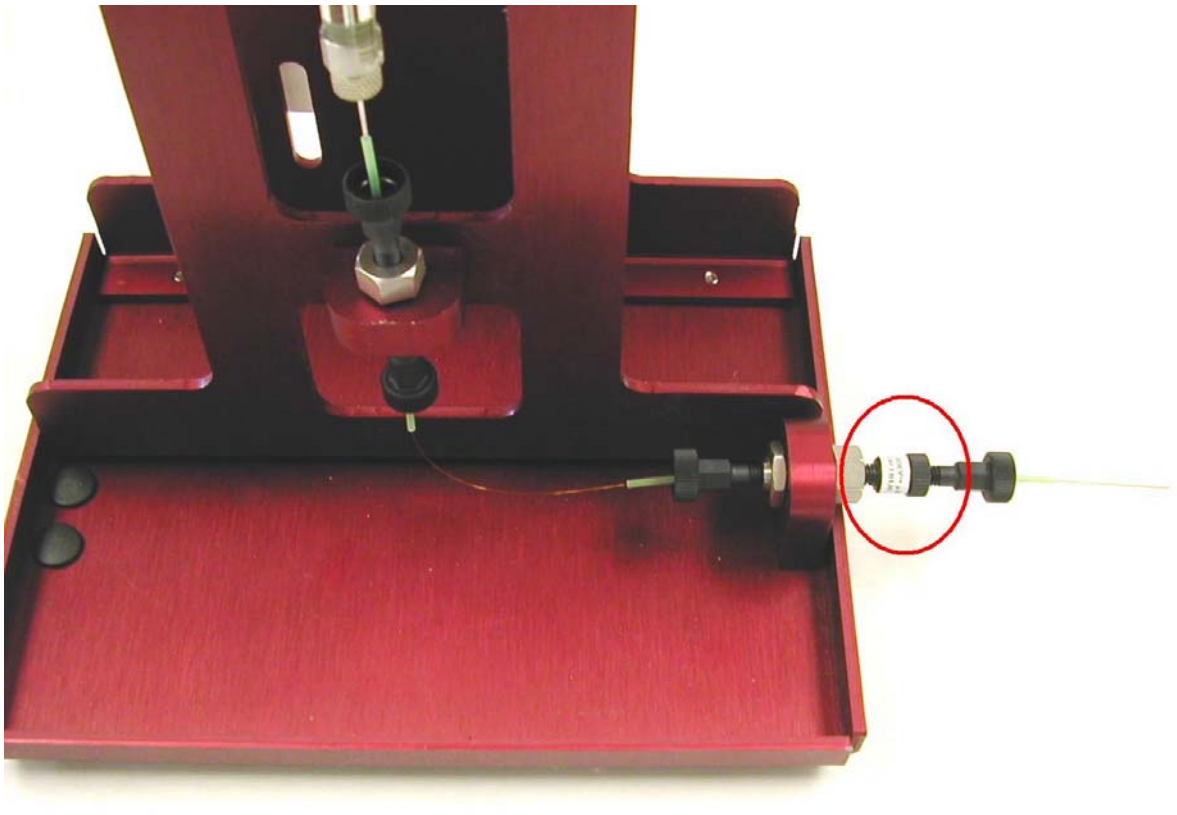
Important: When tightening the black nut, hold onto the filter so it doesn't rotate further. This ensures the fitting is tightened inside the filter.

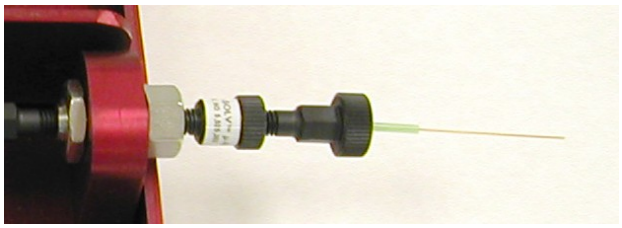
The result should look like this.

'Back-Flushing' the Filter

There are two methods to back-flush or regenerate the filter.

Method 1:
The first method is to use the injector platform and simply remove the filter and place on the outlet side with a small length of capillary (~2-3 inches) using a F-287 black nut and ferrule with a F-242 for capillary users or F-247 for FEP tubing users to guide the waste away from filter body.



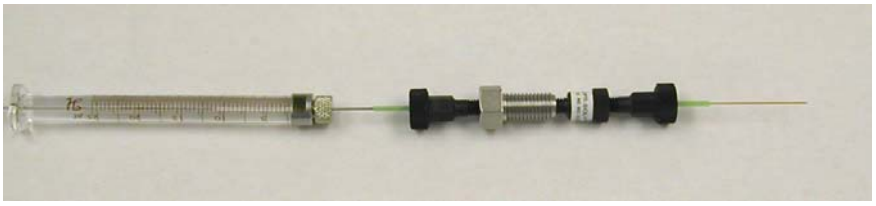


This allows the filter to be flushed in the opposite direction of injections so the particulates get flushed off the filter and out to waste. Once the filter is in place, use your syringe as if you were doing a normal injection and push 50-100 uL of solvent through the filter to flush particulates from the screen. For compounds with difficult solubility, like proteins, you may want to consider rinsing with multiple solvents like 50 uL water then 50 uL acetonitrile then 50 uL DMSO.

Method 2:

There is a second method that does not use the manual injector platform but requires the purchase of an extra Valco bulkhead union. The benefit to this method is the tubing does not have to be taken off the side of the injection platform so there is no risk of contamination from dirt or particulates or damaging the tubing tip when reconnecting the probe.

Assemble a Valco bulkhead union (Part # ZBU1XHC) Upchurch F-247 green sleeve with the F-287 back nut and ferrule for the injection side. The outlet side will consist of the Optimize filter and an Upchurch F-247 green sleeve and F-334N tan nut and Teflon ferrule with a link of FEP tubing (Upchurch Part # 1684). Slide the sleeve, black nut and ferrule over the needle. (This is the same assembly used for sample injections.) Screw the Optimize filter into the backside of the union. The F-334N nut, Teflon ferrule and F-247 sleeve should be assembled and tightened inside the union body before connecting it to the filter for the first time.



Tighten the black nut down to secure the syringe and attach the tan nut and FEP link to the back of the filter as a waste tube. Simply push 50-100 uL of solvent through the filter to flush particulates from the screen. For compounds with difficult solubility, like proteins, you may want to consider rinsing with multiple solvents like 50 uL water then 50 uL acetonitrile then 50 uL DMSO.