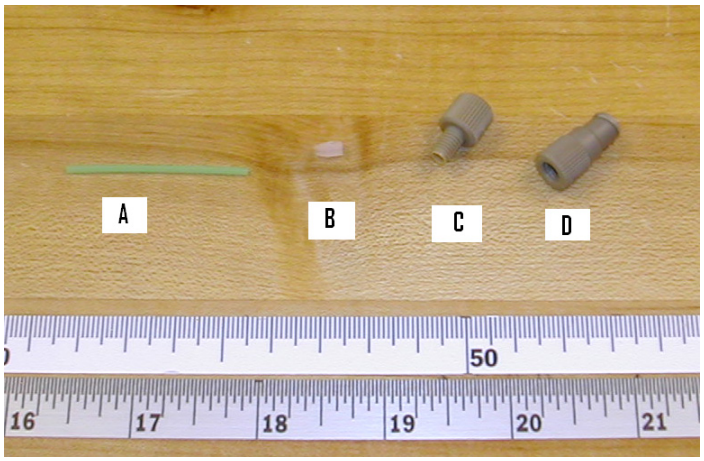
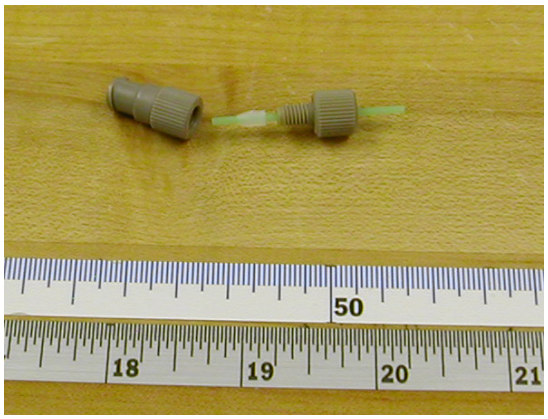
Technical Bulletin G0112: Fluidic Connections

Connecting to the Probe, Basic Rinsing Techniques, and Fluidic Connections including Putting Together Unions & Filters

Connecting the Syringe to Capillary



Part A – Large Green Sleeve	(F-242)
Part B – Ferrule	(F-331N)
Part C – Nut	(F-331N)
Part D – Luer adapter	(P-659)



Assembly as shown, the green sleeve can accommodate any capillary or PEEK tubing with an outer diameter (OD) of 360 µm or less.

There are two ways to connect syringes to capillary. The first connection (pictured on the right) consists of a F-331Nx nut & ferrule with a F-242 green sleeve. This is using with a P-659 luer

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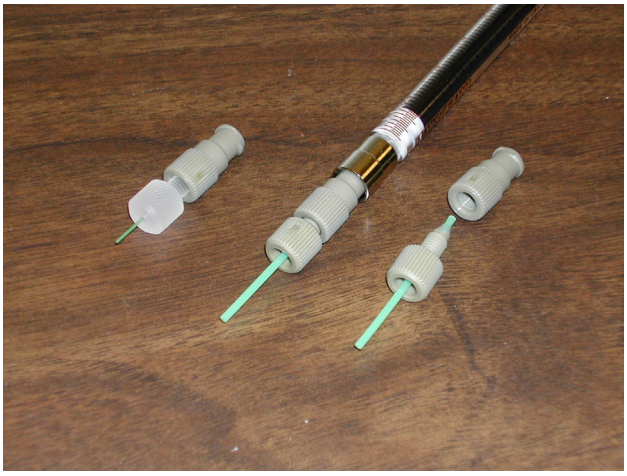
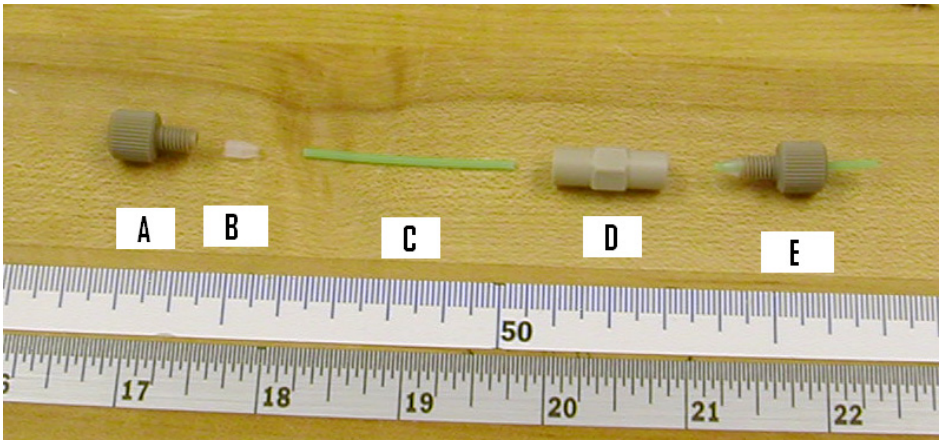
adapter, which is a twist-lock adapter for the gas tight Hamilton syringes. The second (pictures on the left) is the Kel-f M-110 adapter with a F-185 green sleeve. Along with the same P-659 luer adapter, both connections connect capillary to syringes *but the first connection is much more robust.*



Connecting Capillary to Capillary



Part A – Nut	(F-331N)
Part B – Ferrule	(F-331N)
Part C – Large Green sleeve	(F-242)
Part D – Union	(P-779-01)
Part E – Parts A-C assembled	

(Note: *For Syringe injection using this union* all fittings are seen as above except on side of union need F-247 sleeve instead of F-242 sleeve. The F-247 sleeve has a larger inner diameter, which will fit the needle tip.)

The capillary, once places through the green sleeve, should be pushing against the hard stop inside the union. As the nut and ferrule are tightened down, ensure that the capillary and sleeve maintain a solid interface with the hard stop in the center of the union.

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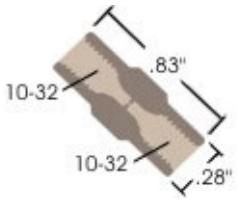
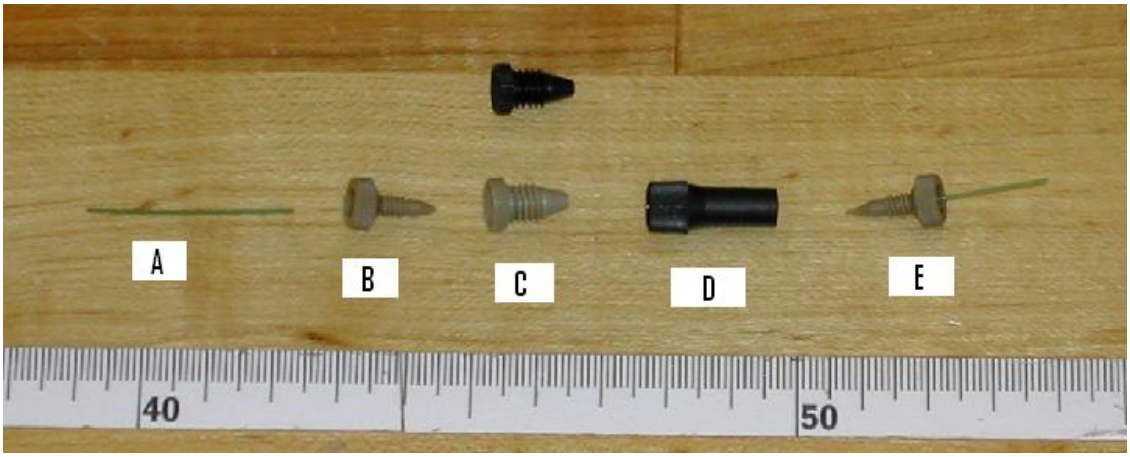


P-779-01 is the best fluidic union that we've found. It has a dead volume that is so small it is fluidically invisible which means it does not cause inline broadening.

Inline Capillary to Capillary Filters

Recommended filter – M-135 (on the right)

The two filters seen above are the M-520 (left) with the black end fittings and the M-135 (right) with the tan end fittings. The M-520 comes with a M-120 0.5 micron PEEK frit. However, unless you have a sample that is metal sensitive, you should use the M-135 with the tan M-130 end fitting which has a 1.0 micron s.s. screen which we've found to be just as effective as the frit but has a lower tendency to become blocked.



Part A – Small Green sleeve (F-185x)

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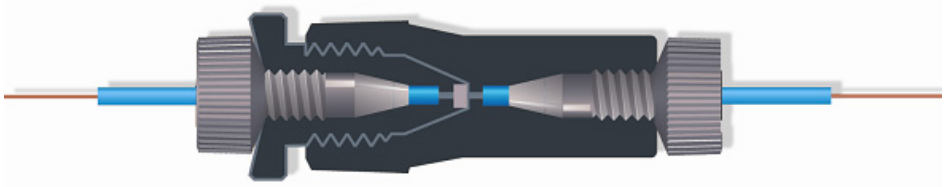
Part B – Fitting (F-125)
Part C – Filter end-fitting (M-130)
Part D – Filter Body (M-135)
Part E – Parts A-B assembled

(Note: *When ordering filters, the M-135 comes with several end fittings as well as both fittings so only a package of small green sleeves (F-185x) would need to be ordered to completely assemble the filter.*)

Insert the capillary through the F-185 sleeve and then fit through the F-125 fitting which has a built-in ferrule. Once the fitting is constructed, (see right side) the fitting should be screwed into the main body of the filter. If the thread does not fit, then a filter end fitting needs to be inserted. With the filter end-fitting is in place, both sides of the filter are symmetric. It is always a good idea to have the sleeve protruding from the fitting before it is inserted into the filter body. This ensures the sleeve is pushed back into correct position, which will give a zero dead volume connection. The same should be done for the capillary. Before you screw the fittings in, you should brush your fingers along the capillary and sleeve using the friction to push the capillary and sleeve tightly against the through-hole in the filter. See a completed diagram below.



Does direction matter? Yes, although the filters are symmetric, the fluid should always enter from the side that has the end fitting to make sure all the particulates are trapped. If fluid enters from the other side, particulates could get trapped in the tip of the capillary before entering the filter. If this occurs, even if the filter is changed, the clog remains in the capillary. Practically all these downfalls can be prevented by simply checking the capillary tips with the provided magnifying glass (at least 10x power) and cutting the tips when needed. See the procedure for cutting capillary below.

Using the Syringe Clamp and Vice

You now have a few custom designed apparatuses that are used for pushing solvent through the probe and for air pushes. The syringe vise was designed with a captured spring that can be used to apply the pressure to push solvent, or cleaning solution through the probe.

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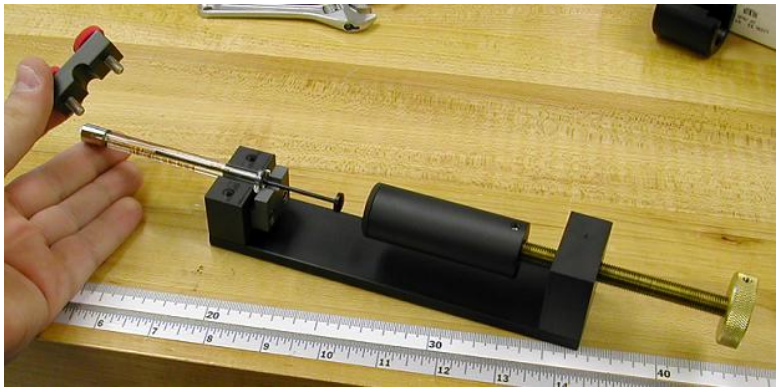
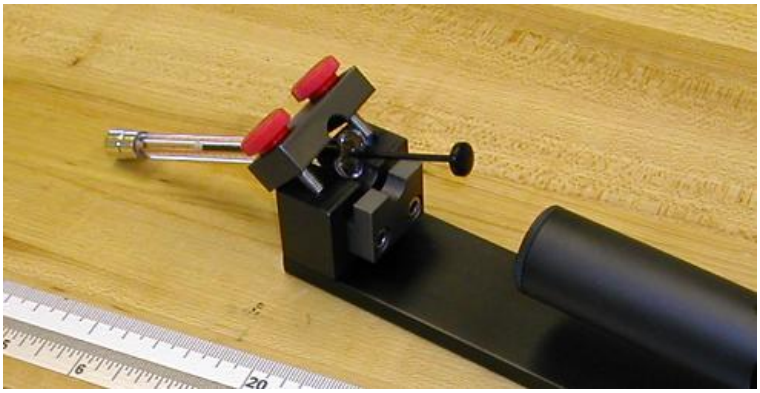
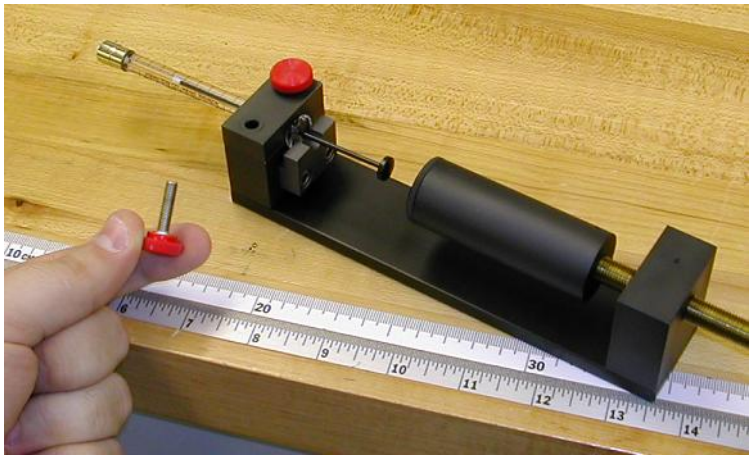


The syringe vise can work with 100 µL, 250 µL, 500 µL Hamilton gastight syringes. Just fill the syringe with the desired volume of solvent and place into the grove.



There are two screws and a bar with can get screws down on top of the syringe to keep it in place. Make sure that the little lip on the syringe is on the inside of the outer lip of the restraining lip to keep the syringe from moving in any directions.



Once the syringe is lined up, just screw the two screws in. Do not use a syringe larger then 500 uL, as the syringe body is too big.

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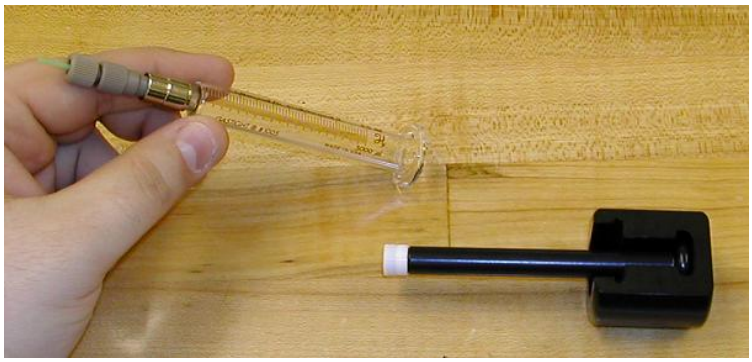
The syringe clamp can ONLY be used with the Hamilton gastight 5 mL syringe. This is used exclusively for the air push. Start by attaching the adapter with the capillary for the probe onto the syringe.



Place the end of the syringe plunger onto the clamp. It may need to be pushed in as we designed the clamp to hold the plunger. Once in the clamp, push the syringe onto the plunger and push all the way on.

The lip of the syringe can be rotated so the clamp actually holds the syringe tight while still applying force to the contents. This allows for an easy air push. The end of the capillary should be examined to make sure that air actually is indeed being pushed through.

Switching Solvent or samples

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When changing solvents, it is always a good idea to rinse with the same solvent as the sample is dissolved then an air push. Follow this by ~50 μL of acetone and another air push. This prevents any problems from some solvents being immiscible. If two immiscible solvents are pushed into the flowcell, just like oil and water, one of the solvents will form a droplet that will stay inside the flowcell until a miscible solvent gets pushed through, dissolves the solvent droplet and gets push out again. A perfect example is our own lineshape sample and our sucrose sample sets. Chloroform and water are immiscible and if mixed, the chloroform will usually ball up into a droplet inside the flowcell causing havoc on shimming.

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(Excerpt from the CapNMR Manual)

Appendix 7: Cutting and Trimming Fused Silica Capillary

You should have received a brief demonstration of how to handle fused silica capillary during your installation. Cut capillary to the approximate required length using a ceramic cutting stone. Then, trim the last centimeter or so of the tip with the Upchurch Precision Fused Silica Capillary Cutter, part #FS-315.

As a review, the sequence for trimming is:

- Push in the button on trimmer with your thumb and hold it down
- Position capillary within cutting wheel
- Tighten gently the gold wheel which holds the capillary in place with an o-ring
- Release the button
- Rotate the cutting wheel about one-half way around
- Push in the button
- Loosen the gold wheel which holds the capillary
- Slip the capillary out of place
- Run your index finger and thumb lengthwise along the capillary and crack the capillary
- Flick the tip with your finger, and check the tip with a magnifying glass

From time to time, clean the inside of the trimmer with water or alcohol. If the blade is damaged or indicates wear, replace it with Upchurch part #FS-315-02.

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