50 µl zircon columns

Consumables:

- 1) Adtech Part # TR95; or BuyHeatShrink.com HSTFE4-0375-CL; %" diameter (9.525 mm) 4:1 shrink PTFE tubing. Estimated recovery width= 2.4mm (ID) and a 0.38mm wall thickness.
 - a) At 2.4mm ID, 50.0 µl is 1.1055 cm
 - b) At 9.525 mm ID, 1 ml (20 column volumes) is 1.4034 cm
- 2) SPC Technologies 1.5 mm fine grade PE (polyethylene) sheet (post treated hydrophilic) material reference number PE3515 works really well. I am currently trying Scientific Commodities (https://scicominc.com/) catalog number BB2062-50L.

Fine Grade Porous Polyethylene Sheet				
Thickness / mm	Material Reference Number	Max Pore Size / μ	Mean Pore Size / μ	Mean Air Permeability m ³ /m ² /min @2" Water Gauge
0.6 ± 0.06	PE3506	69	53	9.8
1.0 ± 0.08	PE3510	58	47	7.9
1.5 ± 0.10	PE3515	51	45	6.7
2.0 ± 0.10	PE3520	49	43	5.5
2.5 ± 0.10	PE3525	47	42	4.5
3.0 ± 0.10	PE3530	45	39	3.6
4.5 ± 0.15	PE3545	41	30	2.1
6.0 ± 0.20	PE3560	41	30	1.7



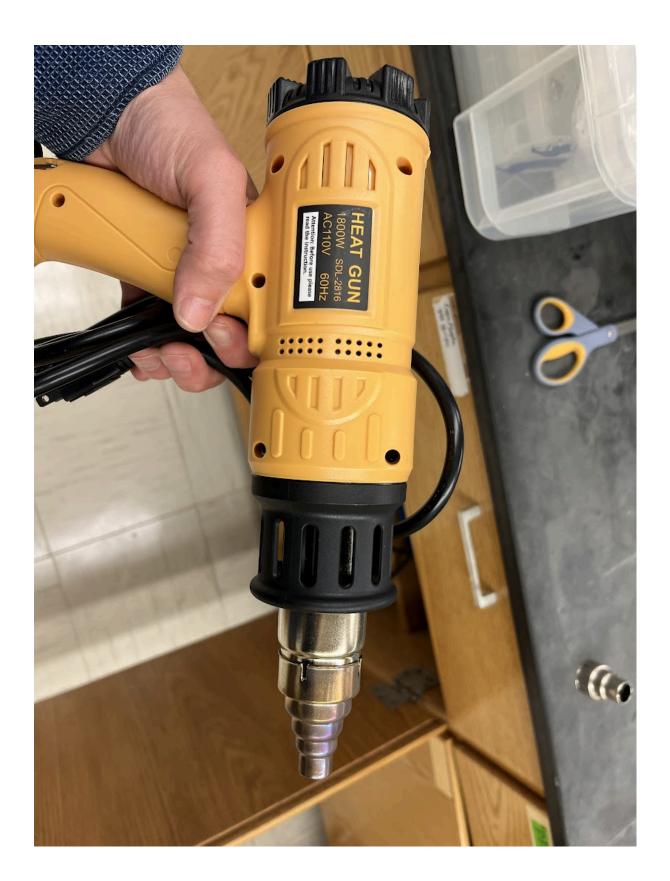


3) 10 mm diameter PTFE rod (at least 1 meter)

- 4) 0.094 inch diameter PTFE rod (McMaster Carr part number 84935K84), the minimum length order is plenty.
- 5) Liquid nitrogen

Other parts

- 1) Table top bench grinder
- 2) Sandpaper
- 3) Heat gun with high heat output. SDL 2816 SEEKONE 1800 W (~1200 F) gun barely works. Must put the column inside the gun. (see photo below for gun and correct fitting)
- 4) Wide top container for LN2
- 5) Clean 2.5 mm hole punch (e.g., https://www.harborfreight.com/leather-punch-tool-57177.html)



Instructions

1) Grind the end of a long (>1 m) 1 cm diameter PTFE rod so that it is slightly narrower than 10 mm for at least 3 cm of the end so that the 9.525 mm shrink tubing can slide

- onto it. Using a combination of a table-top grinding wheel and sandpaper, form a "squashed" hemisphere on the end that will become the shape of the bottom of the column reservoir. Smooth the end with sandpaper and clean it as best you can you don't want grit caught in the reservoir when you shrink it.
- 2) Cut a 3.5 cm length of TR95 shrink tubing. Slide it onto the end of a long (1 m) 1 cm diameter PTFE rod that has one end ground slightly narrower and is gently tapered so that the tubing can be snugly pulled onto the rod, and so that the bottom of the reservoir is tapered. Slide 1.5 cm of the tubing onto the rod this will be the reservoir volume.
- 3) Set a head gun on its back so that the gun is pointed upwards, and turn it on.
- 4) Heat shrinking:
 - a) Start the column shrinking by putting the reservoir portion in the heat stream to contract it around the rod, making it stay in place. This should be done with the rod **perpendicular** to the heat gun barrel. Keep the rod rotating to ensure that it contracts smoothly around the rod.
 - b) Rotate the rod so that it is parallel to the heat gun barrel (holding the rod far above the heat gun so as not to burn your hands) and place the shrink PTFE in the centre of the heat stream, while rotating the rod. Ensure that the entire tip of the column shrinks and that it is not at an angle.
 - c) Once the entire column is shrunk, plunge it immediately into a reservoir of liquid nitrogen to cool.
- 5) Punch a 2.5 mm diameter frit out of the SPC 1.5 mm PE sheet and clean as described below or as desired. Cut a flat edge on the 0.094 inch PTFE rod with a sharp blade, on a length a few inches long. Press the frit in from the top using the flat edge of this tubing. Since it is close to the same diameter as the column stem, the frit should not rotate or be crushed.
 - a) Use a 50 µl volume of water from a pipette to adjust the height of the frit to ensure that the column volume is correct.d.
 - b) Trim the end of the column with a razor. Cut at an angle to assist drop removal.

200 µl Pbc columns

Consumables:

- 1) Adtech Part # TR158/R; 5/8" diameter (15.9 mm) 4:1 shrink PTFE tubing. Estimated recovery width= 4.5mm (ID) and a 0.38mm wall thickness.
 - a) At 4.5 mm ID, 200 µl is 1.258 cm
 - b) At 15.9 mm ID, 4 ml (20 column volumes) is 2.021 cm
- SPC Technologies 1.5 mm fine grade PE (polyethylene) sheet (post treated hydrophilic) material reference number PE3515
- 3) 1.8 cm diameter PTFE rod
- 4) 4 mm diameter PTFE rod (maybe McMaster Carr part number 9660K41)

Other parts

- 1) Table top (bench) grinder
- 2) Sandpaper
- 3) Heat gun with high heat output.
- 4) Wide top container for LN2
- 5) Clean 4.5 mm hole punch

Instructions

- 1) Grind the 1.8 cm diameter PTFE rod similar to that above
- 2) Cut a 4.5 cm length of TR158/R shrink tubing. Slide it onto the end of the long PTFE rod, about 1.5 cm down the tube as the reservoir. Shrink as above.
- 3) Punch a 4.5 mm diameter frit out of the SPC 1.5 mm PE sheet. Push the frit in through the top of the column using the flat edge of a 4 mm diameter PTFE rod.
 - c) Use a 200 µl volume of water from a pipette to adjust the height of the frit to ensure that the column volume is correct. Push the frit with 4 mm PTFE rod.
 - d) Trim the end of the column with a razor. Cut at an angle to assist drop removal.

Cleaning

Soak the frits and the columns in 2D 6 M HCl for two days prior to insertion into the columns. Clean the PTFE holders in decon or RBS solution in an ultrasonic, rinse, and then soak in analytical grade HCl.

Column holders

Cut a 20 mm by 60 mm rectangle from a 1.5 mm thick PTFE sheet.

Drill a 5 mm hole, 10 mm (centre) from one end. Tap a M5 thread.

Drill a 10 mm hole, 15 mm (centre) from the other end. Tap a M10 thread on the inside, then a M12 thread, so it will slide (not thread) onto a M10 threaded rod.