

Frit Kit



Instructions on the preparation of
silica caps for fused silica liquid
chromatography columns

Also: Addendum on column packing for LC/MS
using a pressure injection cell

Introduction

The Next Advance Frit Kit provides you with the proper reagents for preparing a fused silica plug or “frit”. These frits are used to plug the end of fused silica capillaries for liquid chromatography systems prior to column packing. Other applications may utilize fused silica plugs and may or may not be compatible with the reagents provided in this kit. Please consult with our Technical Support prior to using this kit for other applications as it may not be suitable for your use.

Prior to using this kit, make sure that you have all the reagents listed in the table below as well as the desired Next Advance pressure injection cell for loading your chromatography stationary phase after frit preparation.

Included in this Kit

1.0 mL Kasil[®] 1 – White-labeled 2.0 mL tube

1.5 mL Kasil[®] 1624 – Blue-labeled 2.0 mL tube

0.5 mL formamide – Yellow-labeled 2.0 mL tube

Cleaving tool

For MSDS go to msds.nextadvance.com

Instructions

1. Rinse the capillary with methanol. Let it dry completely.
2. In a small glass vial, pipette 300 μL Kasil[®] 1624 (potassium silicate). **OPTIONAL: Add 100 μL Kasil 1 and mix with Kasil[®] 1624. This step has been reported to improve the fidelity and strength of frits.**
3. Add 100 μL formamide. Mix well via pipette.
4. Briefly touch one end of the fused silica capillary to the Kasil / formamide solution to allow some of the mixture to draw into the capillary.
5. **NOTE:** Allowing the liquid to contact the capillary end for too long will draw too much solution into the capillary and create a large dead volume.
6. To cure the solution into a porous silica plug, place the capillary tubing into an oven at 100°C for four hours to overnight.
7. Remove the capillary and trim the excess frit to reduce the dead volume, leaving a 2-3mm frit.
8. The fritted capillary may be stored in a clean, dry place until you are ready to pack it.

References

- Maiolica A, Borsotti D, Rappsilber J (2005) Self-made frits for nanoscale columns in proteomics. *Proteomics* 5(15):3847-3850
- Myers M (2005) Nano-Lc/MS of attomole digested Glutamate Dehydrogenase (GDH) with the polypropylene nanospray nozzle, application note from Phoenix S&T, Inc.

Addendum: Packing Capillary Columns with a Pressure Injection Cell

These instructions are for packing typical, fused silica capillary columns, about 30 cm long.

Always make sure that connections are secure, all parts are properly tightened down, and that you understand how to work with pressurized gas safely. Always wear proper safety equipment.

Prior to column packing, you will need to either make your own frit at the end of the capillary or attach a microfilter to the end of the capillary.

Suspend the packing particles in a solvent, such as methanol, in a glass vial or microcentrifuge tube, and place a magnetic stir bar (part STRBR5X2) in the vial or tube.

Place the vial or tube in the pressure cell, and place the pressure cell on a magnetic stir plate to keep the particles suspended in the solvent.

Place the cover on the pressure injection cell and the capillary through the white plastic ferrule in the cover. Make sure that the cover is firmly bolted down. With the capillary in the correct place, tighten the nut to squeeze the ferrule so that it seals tightly around the capillary column. You can reuse the ferrules several times.

You will use a pressurized gas tank (also called gas cylinder), such as Helium, Nitrogen, Argon, or Dry Air, to force the packing particles into the capillary, and to pack tightly. Double check that all tubing and fittings are securely connected. Open the 3-way valve on the pressure injection cell to let the gas inside, and open the gas tank valve. Set the pressure regulator on the gas tank to 600 to 1000 psi (you may need to adjust the pressure based on the particle size of your packing material). Allow the column to pack for a few hours, and then close the valve on the gas cylinder. Then, let the pressure “leak” out through the capillary column for a few hours. Remove the capillary column, replace the solvent with 50% water, replace the capillary column and then pressurize again to pack tightly and then turn off the pressure and let the pressure leak out through the frit in the capillary column for several hours or overnight before closing the 3-way valve on the pressure injection cell. This prevents the particles from drawing backwards and is important for even packing. Your column is now ready.

