Handling PicoFrit® Columns

There are some important considerations in the handling and use of PicoFrit® columns that make them very different from other chromatography columns on the market today. PicoFrit columns are fabricated from 360 µm OD, polyimide-coated, fused-silica tubing, as shown in Figure 1. One end of the column, referred to here as the "tip end," has a specially tapered tip with an integral high-porosity frit. Behind the frit is the packed chromatography bed. There is no frit at the back end of the bed, only unpacked fused-silica tubing. Mobile phase flow must always be directed toward the tip. Reversing the flow may result in partial or complete unpacking of the chromatography bed.

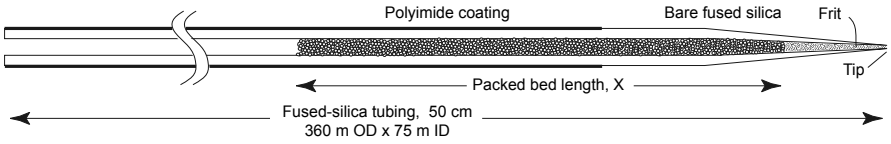

FIGURE 1 PicoFrit® Column

Packaging and Handling of PicoFrit® Columns

The PicoFrit® column is packaged as a loop, with the tip and distal ends protected by a sleeve of snug-fitting FEP (Teflon®) clear tubing, as shown in Figure 2. This sleeve keeps both ends of the column at the same pressure during shipping and storage. The loop should not be opened until the column is being prepared for installation and conditioning. It is a good idea to leave the FEP sleeve in place over the tip end during initial column conditioning and handling; it should not be removed from the tip end until the column is ready for mounting on the mass spectrometer. Note the label attached to the orange PEEK™ sleeve that slides along the length of the column. The arrow on the label points toward the tip end and direction of flow. The tip end may also be identified by the charred section of polyimide coating just before the tapered region of the tip. The columns ship filled with methanol and need to be conditioned with appropriate mobile phase for your gradient. Finally, since there is no distal frit, the distal end of the column bed may loosen with time. The bed will repack when the column is pressurized. For long-term storage, remove the column from the box and hang the loop vertically, with the FEP sleeve nearest to the floor.

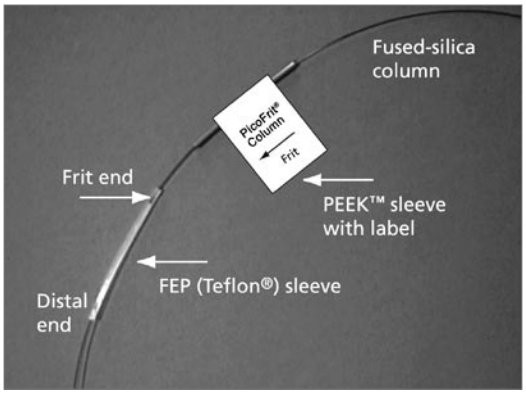

FIGURE 2 Packaging of a PicoFrit® Column

Removing a PicoFrit® Column from the FEP Sleeve

Care must be taken when removing the column from the FEP sleeve to prevent damaging the fragile tip and frit.

Do not bend the FEP sleeve while the tip is inside. The free-sliding PEEK™ sleeve will be used to push the FEP sleeve off the fused-silica tubing without damaging the tip.

Remove the distal end of the column from the FEP sleeve by either pulling it free of the sleeve or, preferably, by cleaving the fused-silica tubing near the terminus. See Technical Note FS-1, available on our Web site, for instructions on cleaving fused silica.

Slide the PEEK sleeve toward the tip end until it butts up against the FEP sleeve, as shown in Figure 3.

Orient the FEP sleeve vertically, with the tip facing the floor. This way, when the FEP sleeve slides free, it will fall to the ground without damaging the tip.

Push the PEEK sleeve against the FEP sleeve until the FEP sleeve falls off. This may take a great deal of force; do not be afraid to push hard. You will have to move the PEEK sleeve 3–5 mm. Figure 4 shows the column after the FEP sleeve has been pushed off.

Do not let the PEEK sleeve slide off the tip end. Instead, remove it by sliding it over the distal or back end of the column.

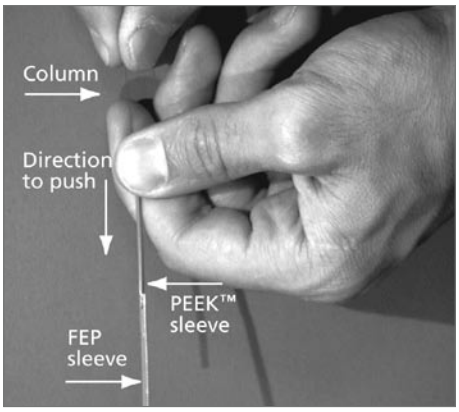

FIGURE 3 Sliding the PEEK™ sleeve

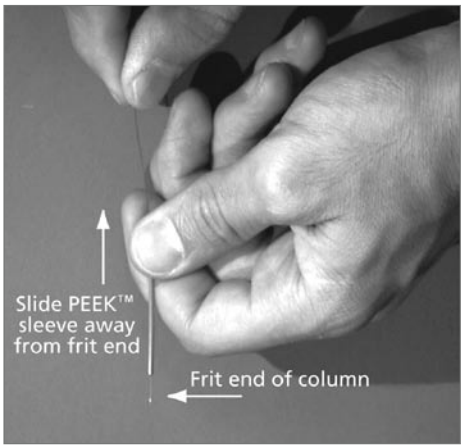

FIGURE 4 PicoFrit® column after the FEP sleeve has been removed

Storing PicoFrit® Columns

If you need to store a PicoFrit® column for reuse on a later date, we recommend the use of a MicroTight® union, fitting, and sleeves to protect the tapered end of the column. Letting the column dry out for an extended period of time may render it unuseable. Protecting the tip end will allow you to store the column in a solvent-filled vial, beaker, or centrifuge tube.

1. Slip the distal end of the PicoFrit column into the MicroTight sleeve, and oull through. DO NOT feed the column tip end first!
2. Pull the column further into the sleeve so that the tip is fully covered and protected by the sleeve. Tighten the fitting so that the column tubing is firmly held in place.
3. Place the column into a suitable vial or centrifuge tube and fill the container with suitable mobile phase. (The same composition used for column conditioning is recommended.) For best results, the column should be vertical, with the tip pointed sown. For long-term storage, the container should be covered with a suitable material to retard solvent evaporation.

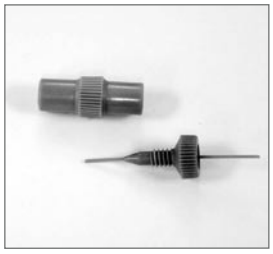

FIGURE 5 Union with sleeve

FIGURE 6 Column pulled through sleeve

FIGURE 7 Column tip pulled back into sleeve

FIGURE 8 Column tip pulled back into sleeve

Cleaving Fused Silica

Proper cleaving of fused-silica tubing is a critical but often overlooked operation in the preparation of emitters and columns prior to use. A flat, smooth cleave is essential for maintaining low dead volume connections with other sections of fused-silica tubing. It is also critical that cleaving does not generate flow-stopping particulate matter. Cleaving is best accomplished with a high-quality diamond chip or sapphire cleaving tool. New Objective's 1 mm wide diamond-blade cleaving tool, shown in Figure 1, has been selected to provide a consistent, flat cleave with a minimum of particulate generation. Inexpensive carbide scribing tools are not recommended, since they generally result in poor-quality (i.e., ragged) cleaved end faces that generate many fine particles.

WARNING: Handling of fused-silica tubing and emitters can result in serious personal injury, including skin and eye injury. Use safety glasses or goggles meeting ANSI Z87.1-1989 requirements or the equivalent. Puncture- and chemical-resistant gloves should be worn at all times.

Procedure

1. Place the tubing to be cut on a flat, clean surface and position the cleaving tool perpendicular to the tubing surface, as shown in Figures 10 & 11B. The long axis of the blade should be perpendicular to the tubing bore.
2. Press down gently (Figure 11B); DO NOT use a sawing motion when pressing the blade. You only need to nick the surface of the polyimide coating (Figure 11C). Be careful not to force the blade through the tubing, which will generate a ragged end and many particles (Figure 11D).
3. Pull gently on the tubing along its axis; it should easily separate at the point of contact. If it does not, repeat the procedure with a little more force. A typical cleave of 360 µm OD, 75 µm ID fused-silica tubing is shown in Figure 12. Residual surface irregularity is on average less than or equal to 10 µm.

Inspection of the distal end of the tip for particle contamination using a light microscope with transmitted light at 100x magnification is highly recommended. New Objective sells an accessory kit that contains all the high-quality tools (cleaver, special forceps, ruler, etc.) you will need to properly handle fused-silica emitters, columns, and tubing. Please see our catalog or Web site for a full description of our Micro Tool Kit (stock number TIP-KIT).

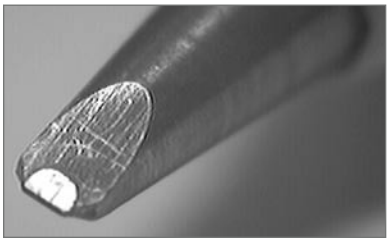

FIGURE 9 Close-up view of diamond-blade cleaving tool

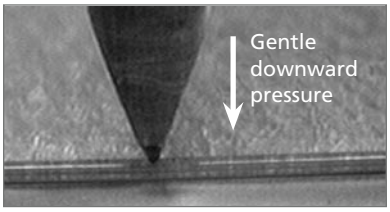

FIGURE 10 Cleaving tool in proper position

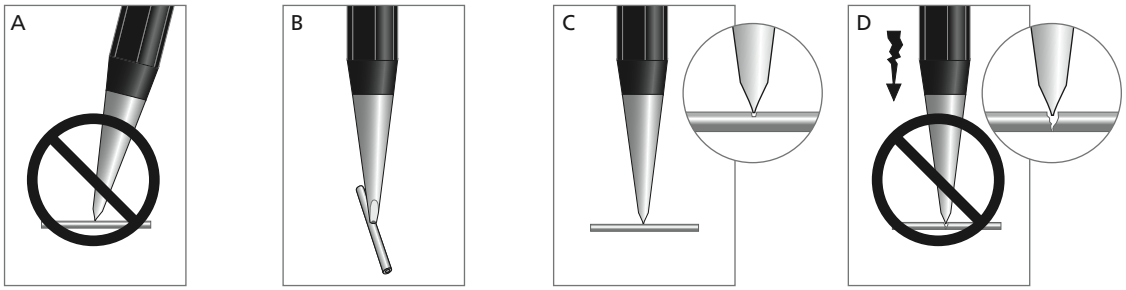

FIGURE 11 (A) Improper cutting angle (B) Align cleaving tool perpendicular to tubing (C) Press down gently, scoring tubing (D) Too much downward pressure will crush tubing, producing particles that can cause tubing to clog

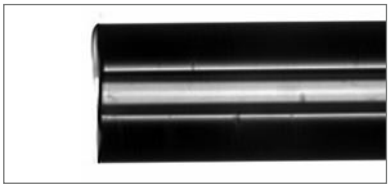

FIGURE 12 Typical cleave. Polyimide coating was removed after cleaving for clarity of image.