

## Handling IntegraFrit™ Columns

There are unique considerations in the handling and use of IntegraFrit™ columns. IntegraFrit columns are fabricated from 360 µm OD, polyimide-coated, fused-silica tubing as shown in Figure 1. The frit end of the column has an integral high-porosity frit. The edge of the fused-silica tubing at the frit end has been polished flat. Behind the frit is the packed chromatography bed. There is no frit at the distal, or back, end of the bed, only unpacked fused-silica tubing.

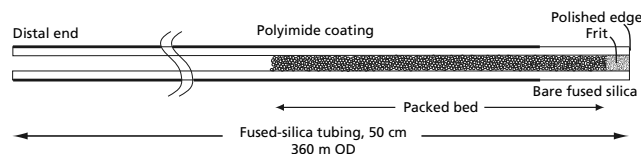


FIGURE 1 IntegraFrit™ Column

### Packaging

The IntegraFrit™ column is packaged as a loop, with the frit and distal ends connected and protected by a sleeve of snug-fitting FEP (Teflon®) tubing as shown in Figure 2. The clear FEP sleeve keeps the column hydrated with methanol during shipping and storage.

### Guidelines for Handling IntegraFrit™ Columns

- Never bend the clear FEP sleeve. Bending the sleeve will result in column failure.
- The column loop should not be opened until the column is being prepared for installation and conditioning.
- Mobile phase flow must always be directed towards the frit. Reversing the flow may result in partial or complete unpacking of the chromatography bed.
- For long term storage, remove the column from the box and hang the loop vertically with the FEP sleeve nearest to the floor.

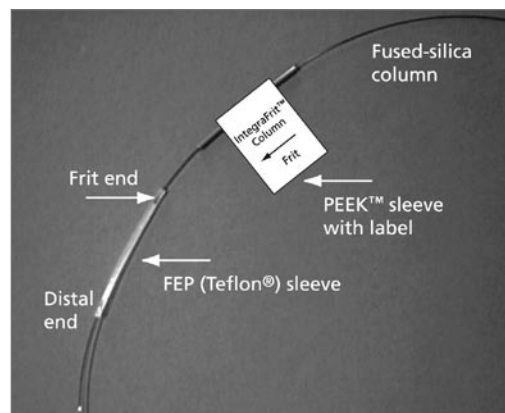


FIGURE 2 Packaging of an IntegraFrit™ Column

### Connecting to the IntegraFrit™ Column Outlet

You have two choices for connecting the outlet of the IntegraFrit™ column to your detector (UV, MS etc.):

- Remove the IntegraFrit column from the FEP sleeve and use a MicroTight® or similar union designed to connect two pieces of capillary tubing. To follow this option use the directions below, “Removing the FEP Sleeve from the IntegraFrit Column”.
- Leave the FEP sleeve in place on the column outlet and modify it for use as a direct-connect, zero dead volume (ZDV) union. To follow this option use the directions on the facing page, “Converting the FEP Sleeve to a ZDV Union”.

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

### Removing the FEP Sleeve from the IntegraFrit™ Column

Care must be taken when removing the FEP sleeve to prevent damaging the column. Bending the FEP sleeve can fracture the frit and render the column useless. Do not attempt to remove the sleeve by pulling it off the end of the column. Use the free-sliding PEEK™ sleeve to push the FEP sleeve off the fused-silica tubing in the following manner:

- Remove the distal end of the column from the FEP sleeve by either pulling it free of the sleeve, or (preferably) cleaving the fused-silica tubing near the terminus.
- Slide the PEEK sleeve towards the frit end until it butts up against the FEP sleeve as shown in Figure 3.
- In one hand, firmly grasp the fused-silica column. It is helpful to wrap some of the fused-silica tubing around the palm of your hand for a firm grip. The polyimide coating is quite durable and will withstand considerable handling.
- Orient the FEP sleeve vertically, frit facing down towards the floor. This way, when the FEP sleeve slides free, it will fall towards the ground without damaging the column.
- Using your other hand, push the PEEK sleeve against the FEP sleeve until the FEP sleeve falls off. This may take a LOT of force. Do not be afraid to push hard. You will have to move the PEEK sleeve about 3 to 5 mm. Figure 4 shows the column after the FEP sleeve has been pushed off.
- Remove PEEK sleeve by sliding it over the back end of the column.

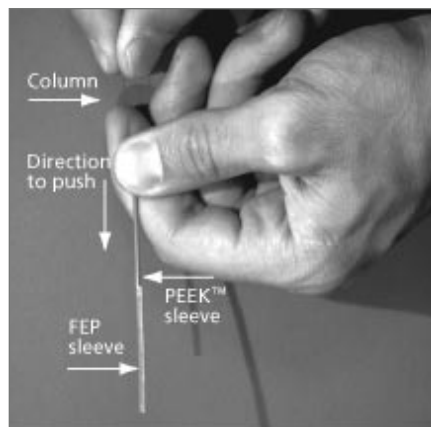


FIGURE 3 Sliding the PEEK™ sleeve

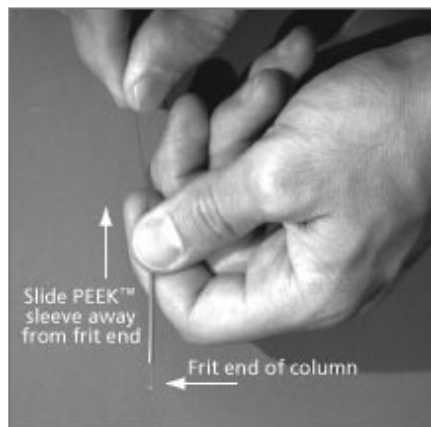


FIGURE 4 IntegraFrit™ column after the FEP sleeve has been removed

### Converting the FEP Sleeve to a ZDV Union

- Locate the fritted end of the column inside the FEP sleeve.
- Within the FEP sleeve there is a gap between the frit end and the distal end of the column. Measure 5 mm into this gap, towards the distal end of the column, from the end of the frit. Cut the FEP sleeve using a single edge razor blade or similar sharp cutting tool as shown in Figure 5A. Use caution because bending the FEP sleeve can fracture the frit and render the column useless. A properly cut FEP sleeve is shown in Figure 5B.
- Using a dissecting needle, such as that found in New Objective's Micro Tool Kit (order no. TIP-KIT), swage the inside of the cut FEP sleeve as in Figure 5C. The needle should come no closer than 2 mm to the frit. Do not contact the frit with the sharp end of the needle. Remove the needle from the sleeve.

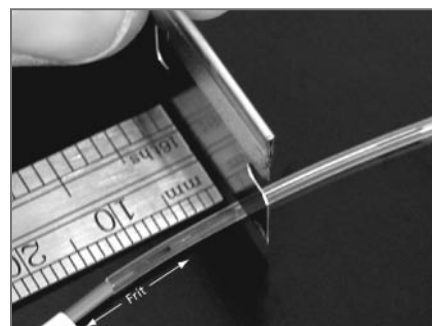


FIGURE 5A Cutting the FEP sleeve

- The sleeve is now ready for the insertion of a square cut length of fused-silica tubing or, for example, the distal end of a New Objective PicoTip®. Push the fused-silica tubing into the sleeve until the tubing contacts the end of the frit as in Figure 6.
- Inspection of a good connection in a FEP sleeve, now a union, under a light microscope is shown in Figure 7. This union is a true zero dead volume connection and will offer good chromatographic performance. A properly prepared FEP union will typically hold pressure to 200 psi or more.

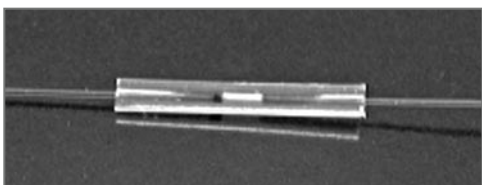


FIGURE 6 Properly loaded FEP union

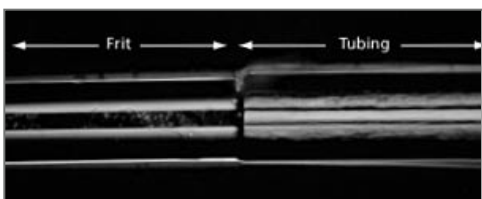


FIGURE 7 Close-up of a good connection

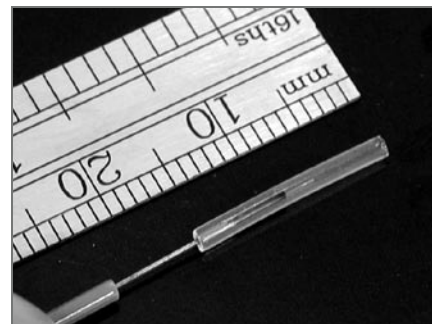


FIGURE 5B Properly cut sleeve

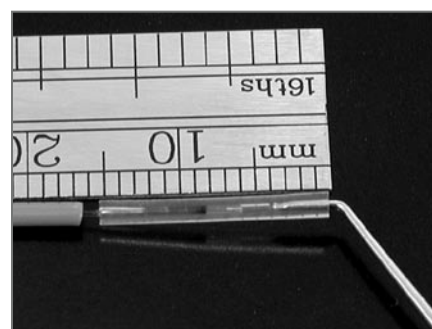


FIGURE 5C Swaging the sleeve

### *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 8, 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.

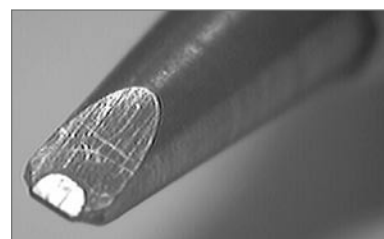


FIGURE 8 Close-up view of diamond-blade cleaving tool

**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 9 & 10B. The long axis of the blade should be perpendicular to the tubing bore.
2. Press down gently (Figure 10B); DO NOT use a sawing motion when pressing the blade. You only need to nick the surface of the polyimide coating (Figure 10C). Be careful not to force the blade through the tubing, which will generate a ragged end and many particles (Figure 10D).
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  $\mu\text{m}$  OD, 75  $\mu\text{m}$  ID fused-silica tubing is shown in Figure 11. Residual surface irregularity is on average less than or equal to 10  $\mu\text{m}$ .

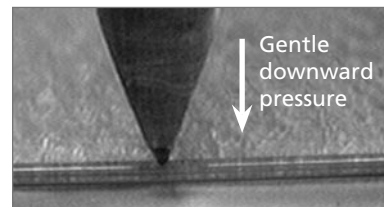


FIGURE 9 Cleaving tool in proper position

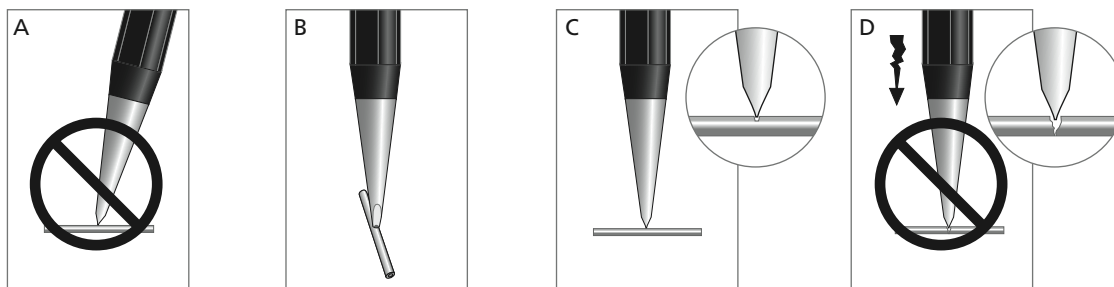


FIGURE 10 (A) Improper cutting angle (B) Align cleaving tool perpendicular to tubing (C) Press down gently, scoring tubing (D) Too much downward pressure will crush tubing, producing particles that can cause tubing to clog

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 accessory kit (stock number TIP-KIT).

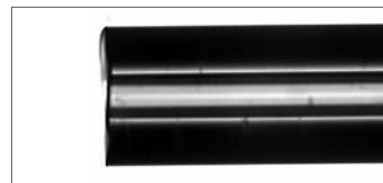


FIGURE 11 Typical cleave. Polyimide coating was removed after cleaving for clarity of image.

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