Assembly of a packed-bed reactor ● **TIMING ~30 min**

(i) Gather two Swagelok stainless zero-volume-reducing unions having an outside diameter of 1/4-inch and lay the parts out as displayed in **Figure 8a**; see also **Supplementary Videos 6**–**9**.

(ii) Mount the seamless stainless-steel tubing into a bench vise and mark out a 10-cm portion (**Fig. 8b**). Place the metal tube cutter around the point to be cut, and tighten it. Rotate the metal tube cutter around the stainless-steel tubing, and at every ten turns, tighten the tube cutter further to ensure that the blade of the tube cutter remains in contact with the stainless-steel tubing (**Supplementary Video 6**).

Keep rotating the metal tube cutter around the stainless-steel tubing until the metal tube is cut. If the metal tubing is moving, then the tube cutters are too tight. In this case, loosen the tube cutters and tighten them less at every ten turns.

(iii) File both ends of the cut stainless-steel tubing flat using a metal file (**Supplementary Video 7**). Then, deburr the opening using a deburring tool to ensure that the inside surface is flush with the rest of the stainless-steel tube.

(iv) Unscrew the metal nut (component 0) from the union’s 1/4-inch ports (component 1) and insert a 10-μm stainless-steel frit. Push the frit to the bottom using tweezers, and then reattach the metal nut (**Fig. 8e**).

 **CRITICALSTEP** Inserting the frit into the 1/4-inch ports (component 1) at this stage avoids incorrect spacing in Step 5.

(v) To both ends of the stainless-steel tubing, add the Swagelok union parts in the order and orientation shown in **Figure 8c**. First, slide the metal nut (component 2) onto the tube with the threads pointing toward the end of the tube. Next, slide the ring component of the ferrule set (component 3) onto the tube, with the larger-diameter face pointing toward the threads of the metal nut. Then slide the cone component of the ferrule set (component 4) onto the tube, with the larger-diameter face pointing toward the ring component (component 3) and the metal nut (component 2). Finally, slide the Swagelok union (component 1) onto the tube, with the threads facing the ferrule set and the metal nut.

(vi) Compress the components and tighten them by hand in a clockwise motion to secure them. Mount the stainless-steel tubing into the bench vise and use 1/2-inch and 9/16-inch wrenches to tighten the nut into the Swagelok union further.

 **CRITICALSTEP** The connection must be tight; the packed-bed mixer is under pressure.

(vii) Unscrew the smaller metal nut (component 0) and lay the contained ferrule set out as shown in **Figure 8d**. Be careful not to lose the components inside, as they can sometimes slide out.

(viii) Cut 2 × 10-cm-long pieces of DuPont 0.02-inch-inner-diameter PFA tubing with a polymer-tube cutter to ensure that both ends of the tubing are flat. These will serve as the tubing connecting the packed-bed mixer to the other parts of the continuous flow system.

 **CRITICALSTEP** Use polymer-tube cutters to cut the PFA tubing to reduce the chance of the reactor leaking. Polymer-tube cutters provide a flush cut ensuring optimal sealing of the fitting.

(ix) To one end of the PFA tubing, add components 5, 6, and 7 as shown in **Figure 8f** (**Supplementary Video 8**). First, slide the metal nut (component 5) onto the PFA tubing, with the thread facing the end of the PFA tubing. Next, slide on the metal ring (component 6), with the larger-diameter side facing the threads of the metal nut and the smaller diameter facing the end of the PFA tubing. Finally, add the metal cone (component 7), with the larger-diameter side facing the threads of the metal nut and the metal ring and the smaller face pointing toward the end of the PFA tubing.

(x) Insert this end of the PFA tubing into the Swagelok union (component 1) and apply pressure while simultaneously finger-tightening. Mount the packed-bed reactor back into the bench vise and tighten the fittings using 9/16-inch and 5/16-inch wrenches (**Fig. 8g**).

**? TROUBLESHOOTING**

(xi) Repeat Step 51B(ix and x) on the other side of the stainless-steel tube to ensure that both sides of the packed-bed reactor now have PFA-tubing attachments.

(xii) To both ends of the PFA tubing, add a super flangeless nut and a ferrule set using the method previously described in Steps 2–7 (**Fig. 8i**).

(xiii) Unscrew the metal nut (component 2) from the Swagelok union (component 1) to reveal an opening into the stainless-steel tube.

(xiv) To create a funnel for loading, take a 3-ml disposable plastic syringe and completely remove the plunger. Insert the small end of the syringe into the stainless-steel tube so that it is held firm.

(xv) Add sand to the stainless-steel tube while simultaneously tapping the tube and the syringe with a spatula to ensure good packing and sand release (**Supplementary Video 9**). Other materials, such as stainless-steel chippings, can be used in the packed-bed reactor; however, the use of very small packing materials can cause a large pressure increase, leading to syringe-pump failure. As a general rule, use a material that will require ten units or more to span the diameter of the stainless-steel tubing. For example, if the internal diameter of the packed-bed reactor is 0.180 inch, then, sand particles with a diameter of ≤0.0180 inch should be used.

(xvi) Use the flat back end of a drill piece to pat down the sand to ensure complete and firm packing.

(xvii) Place your finger on top of the filled stainless-steel tubing and subject the surrounding nuts to compressed air to remove any loose sand.

 **CRITICALSTEP** Sand caught in the metal nut can lead to an inefficient seal.

(xviii) Screw the metal nut (component 2) into the Swagelok union (component 1) using 1/2-inch and 9/16-inch wrenches to ensure a tight fitting. The packed-bed reactor is now complete and ready to use (**Fig. 8h**). To change the material in the packed-bed reactor, repeat Step 51B(xii–xviii), ensuring that the sand is disposed of as solid waste and that the column is washed with the solvent of choice. Unpacking the column is best when the material contained within the column is dry.