

Using PicoFrit® Columns With the Micromass® ZSpray™ Nanoflow™ Stage

New Objective's probe modification kit enables the use of PicoFrit® columns with your existing Micromass® ZSpray™ Nanoflow™ stage. Use of either self-pack or pre-packed PicoFrit columns is supported through a platinum wire electrode to establish pre-column, high-voltage contact. With their built-in emitters, PicoFrit columns allow you to avoid the tedious process of having to connect a nanobore column to an ESI tip, as well as eliminate clogged tips. The integral PicoTip® design assures you optimal chromatographic performance by eliminating post-column band broadening. You spray directly off of the end of the column and into your mass spectrometer.

Contents of the ADPC-MZS kit:

- PicoFrit® stage adapter (base plate, MicroTee™ and union mounting blocks)
- MicroTee™ with platinum wire electrode
- MicroTight® union, fittings and sleeves
- New Objective's diamond scribe



FIGURE 1
Assembled Adapter

WARNING: Electrospray ionization involves the use of potentially lethal, high-voltage electrical current. Observe all manufacturers' safety recommendations in the use of such equipment. No equipment modifications should be made except by trained personnel using methods approved by the manufacturer in accordance with all safety requirements. Installation of equipment should be performed by qualified personnel in accordance with all applicable electrical codes.

Removing the spray manifold block from the Micromass ZSpray™ stage

As shown in Figures 2A-C, the ADPC-MZS replaces the spray manifold block provided by Micromass for your nanoflow stage. Before mounting the ADPC-MZS, please review the manufacturers' safety instructions for turning off all source voltages and selecting the proper standby mode.

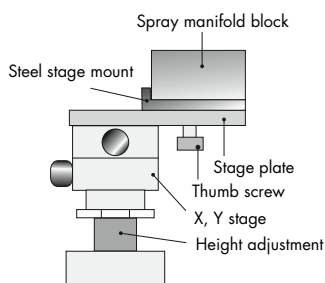


FIGURE 2A

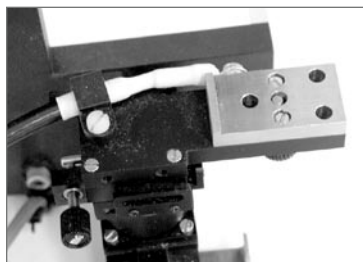


FIGURE 2B Remove spray manifold block

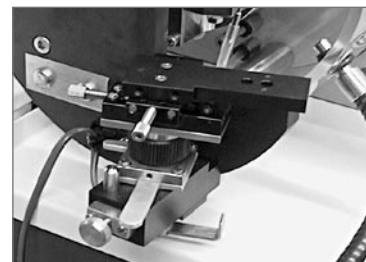


FIGURE 2C Remove steel stage mount and high-voltage lead

- 1) Put the system into Standby mode.
- 2) Rotate the stage into the Open position.
- 3) Remove all liquid and gas lines from the spray manifold block.
- 4) Loosen and remove the thumbscrew that holds the spray manifold block to the stage plate.
- 5) Using a precision screwdriver, loosen the two (2) 2 mm-thread screws from the top of the steel stage mount. You can then remove the steel stage mount from the top of the stage.
- 6) Disconnect the high-voltage lead from the side of the stage by loosening the mounting screw. The stage should now look like Figure 2C.

You are now ready to mount the ADPC-MZS base plate.

Mounting the ADPC-MZS base plate

Mount the ADPC-MZS base plate on the stage plate as shown in Figure 3.

- 1) Line up the ADPC-MZS base plate with the top of the stage plate.
- 2) Insert the screws into the counter-bored holes at the front of the ADPC-MZS base plate.
- 3) Using the 1.5 mm Allen wrench provided, screw the two 2 mm screws into place to securely hold the ADPC-MZS base plate on top of the stage plate.

Mounting the PicoFrit® Column on the ADPC-MZS base plate

- 1) Prepare the MicroTight® union as shown in Figure 4A by removing a fitting from one end. Slide the green MicroTight sleeve through the remaining fitting in the union as seen in Figure 4B. Tighten the fitting just enough to prevent unwanted movement of the sleeve. Replace the fitting on the other end of the union as in Figure 4C.
- 2) Place the union in the union mounting block as seen in Figure 4D. Tighten the set screw to hold the union firmly in place.
- 3) Remove the PicoFrit column from its protective packaging according to the directions that came with the column.
- 4) Slide the distal, or non-tip, end of the column through the sleeve protruding from the fitting and union assembly as in Figure 4E. Pull the column through the sleeve until the tip protrudes just a few millimeters from the sleeve as shown in Figure 4F. Tighten the fitting around the column until the column is no longer loose.
- 5) Place the union mounting block on the end of the ADPC-MZS base plate as in Figure 5.

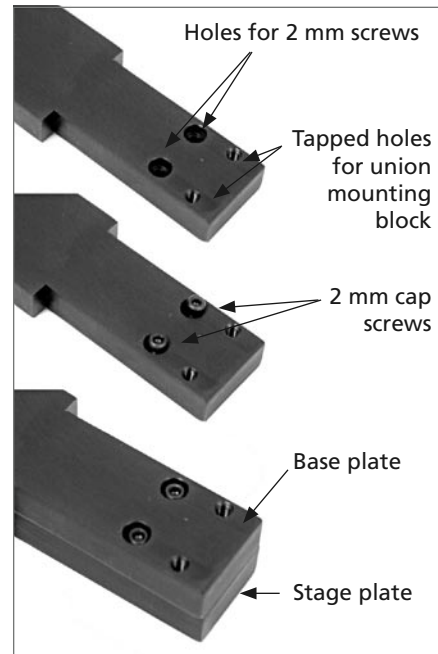


FIGURE 3 Mounting the ADPC-MZS plate onto the stage plate

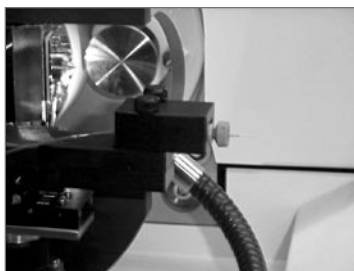


FIGURE 5 ADPC-MZS adapter mounted on nanoflow™ stage with a PicoFrit® column in place

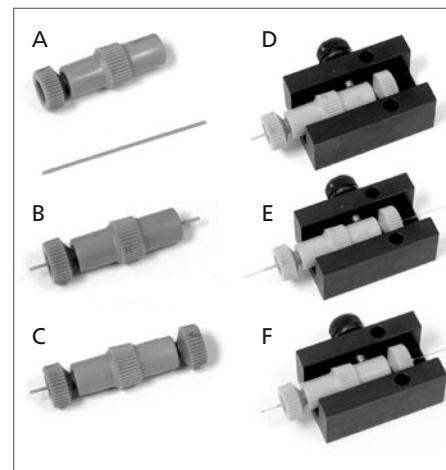


FIGURE 4(A-F) Placing the PicoFrit® column into the union and onto the union mounting block

WARNING: The transfer line that connects to the high-voltage MicroTee must be made of an electrically non-conductive material such as PEEK™ or fused silica. Do not use stainless steel, or any other electrically conductive material or risk of electrical shock is present. A proper electrical grounding point must also be supplied on the inlet side end of the transfer line. Do not attempt to modify, adjust, or remove any of the set screws of the MicroTee assembly.

Connecting the PicoFrit® Column to the MicroTee

- 1) Verify that the high voltage is turned off.
- 2) Locate a green MicroTight® sleeve and MicroTee. Unscrew the ferrule and cap from the MicroTee as shown in Figure 5A.
- 3) Slide the distal, or non-tip, end of the column through the sleeve. Then slide the column and sleeve through the cap and ferrule as shown in Figure 5B. Using the diamond scribe and the cleaving technique described in Technical Note FS-1, trim the distal end of the column so that the overall length of the column is 15-17 cm.
- 4) Insert the column, sleeve, and ferrule into the forward port of the MicroTee. While pushing the column tubing and sleeve flush with the inlet port, tighten the nut fingertight. You should not be able to pull out the column tubing with a moderate pulling force. If you can, re-seat the tubing and apply greater torque to the nut.
- 5) Mount the MicroTee assembly to the ADPC-MZS base plate, as shown in Figure 5C, using the 4-40 x 1/2" thumb screw provided.
- 6) Connect a fused-silica transfer line (50 mm ID tubing is supplied) to the back port of the MicroTee by repeating Steps 2 - 4. Connect the transfer line to your sample injection and mobile-phase delivery system through an electrical ground. Consult your manufacturer's literature to locate an appropriate grounding point. The pumping system should be capable of delivering flow rates in the range of 100-500 nL/min for optimal PicoFrit performance.

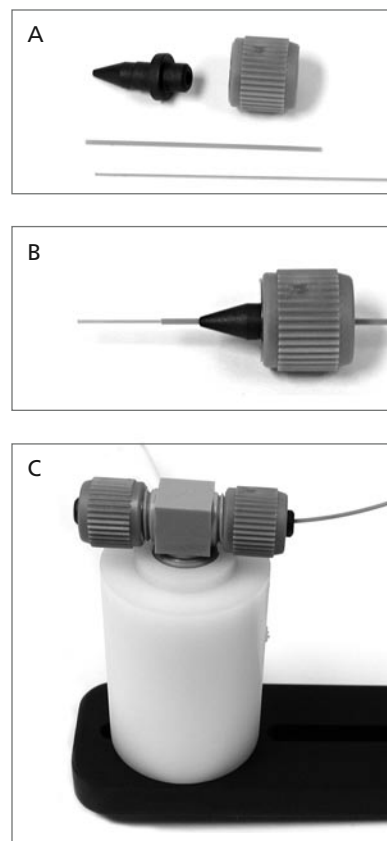


FIGURE 5 (A) Ferrule, cap, and sleeve ready for assembly with a PicoFrit® column or transfer line tubing (B) Ferrule in cap loaded with sleeve and column/transfer line tubing (C) PicoFrit column connected to the forward port of the MicroTee, mounted on the ADPC-MZS base plate

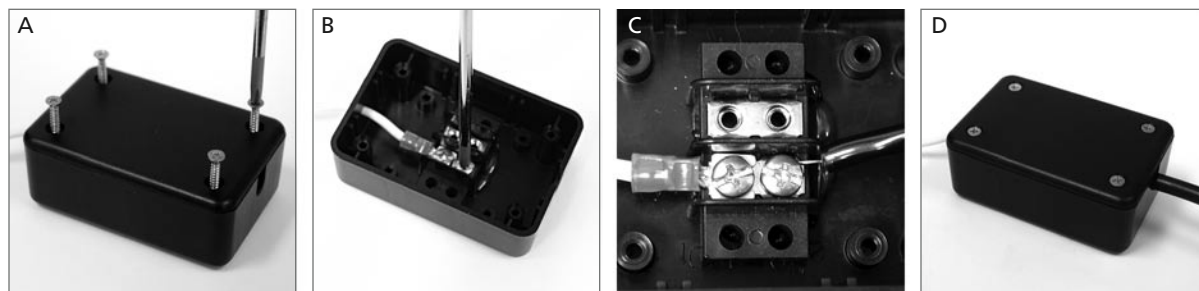


FIGURE 6 Connecting the high-voltage cable

Connecting the high-voltage lead to the junction box

- 1) Verify that the high voltage is turned off. Remove the four screws that hold the junction box lid in place, as shown in Figure 6A, and remove the junction box lid.
- 2) Loosen the unused screw on the terminal strip as shown in Figure 6B.
- 3) Place the cable high-voltage lead around the screw as in Figure 6C. You may have to gently bend and reshape the lead for a good fit under the head of the screw. (Do not over-bend as you may fatigue and break the lead.) Tighten the screw for a good fit.
- 4) Verify the tightness of the lead, replace the junction box lid, and all four cover screws as in Figure 6D.
- 5) You may use the hook-and-loop fastener provided to temporarily mount the junction box to the instrument. Mount the box in a position that will prevent unwanted tugging or pulling on the Micromass high-voltage cable. Route the adapter's high-voltage cable in a safe manner that will prevent damage to the cable insulation.

PicoFrit® Tuning Hints

Applied voltage is perhaps the most important parameter for stable, efficient operation.

NOTE: To prevent an arc or corona discharge never use a “turn-on” voltage above 500 V unless stable ESI has been previously established.

It is best to start tuning at a low voltage, under 1 kV, and increase the operating potential in 100 V increments until stable operation is achieved.

In low-flow ESI, it is important to recognize the interdependence of flow rate and the applied electric field. For a given tip size, stable ESI can occur over a wide range of flow rates but only over a narrow range of field strength (50 V or less). Raising the flow rate requires a higher field strength, and vice versa.

Best results are usually obtained by observing the magnified image of the spray pattern and tuning for an even ESI plume of droplets, as shown in Figure 7, rather than a directed linear stream.

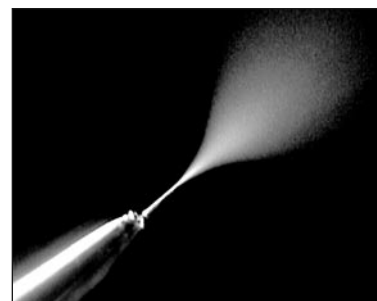


FIGURE 7 Stable ESI plume

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