Using the ADPC-IMS PicoFrit® Probe for the Thermo Ion Max™ Source

The ADPC-IMS PicoFrit® Probe

Empower your Thermo Finnigan™ Ion Max™ source with the sensitivity of nanospray using the ADPC-IMS PicoFrit® Probe from New Objective. A compact, cost-efficient solution to configuring your existing Ion Max source for nanobore LC-MS, the plug-and-play PicoFrit Probe design allows a fast and simple swap from the microbore ESI probe.

The PicoFrit® column is the ultimate proteomic platform. Spraying directly from the column to the MS inlet, PicoFrit columns minimize post-column loss, eliminate band broadening, and produce superior separations with femtomole sensitivity. Allowing flow rates five times below microbore probe capability, the PicoFrit contains an integral frit to reduce tip clogging and offer robust performance with high sensitivity.

Easily installed and adjusted, the ADPC-IMS PicoFrit® probe is powered by New Objective’s proven HV contact system. Enclosed within the probe and fitted with an interlock device compatible with the Ion Max™ source design, the PicoFrit Probe maximizes safety in both installation and operation. Once installed, the PicoFrit probe allows continuous Z-adjustment to produce the fine ion stream and optimized signal critical to nanobore LC. Additional tip configuration is possible by angular adjustment of the tip holder after installation.

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.

Never use this product with defective, damaged, or faulty equipment. Serious injury or death could result.

WARNING: Only qualified personnel should use this product. Provide a safe workplace equipped with all necessary safety equipment.

CAUTION: 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.
**PicoFrit® Probe Overview**

Figure 1 displays key components of the PicoFrit® Probe. The PicoFrit column is fitted with a MicroTight® union, positioned in the tip holder, and secured by tightening the union lock-down screw. The tip holder connects to the base of the PicoFrit probe by a Z-adjustment slide controlled by a handle located on the opposite end; this slide allows accurate tip depth positioning inside the Ion Max™ source. The exact angle at which the tip generates electrospray is determined by the tip angle control on the tip holder.

The base of the PicoFrit Probe contains a medial channel which allows a column and/or fused silica LC line to run along its length. The LC line and PicoFrit column are connected via a MicroTee union, allowing seamless flow to the tip. Two MicroTee pockets located 6.4 cm apart in the channel allow direct insertion of the MicroTee into the probe. The presence of two pockets facilitates use of variable column lengths; the MicroTee is inserted in the pocket closest to the tip (Position A) for short columns and in the rear pocket (Position B) for longer columns. When inserted in Position B, excess column length is threaded through two notches at the side of the base.

The probe cover attaches to the base via the cover screw. The cover also contains an interlock pin which, once installed, triggers activation of the probe’s enclosed high voltage system. The HV contact is located on the side opposite the Z-adjustment slide handle and connects to a voltage source.

**WARNING:** Do not defeat any mass spectrometer system software or hardware safety interlocks.

![Diagram of PicoFrit® Probe](image-url)
Probe Configuration

CAUTION: 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.

While not utilized as a plumbed union in this application, the MicroTight® union collars the end of the PicoFrit® column nearest the tip and allows its secure installation in the tip holder.

Preparation of the MicroTight® Union
1) Carefully trim a new green MicroTight® sleeve to a length of 14 mm
2) Remove the ferrule at one end of the MicroTight union and insert the green sleeve into the ferrule.
3) Slide the MicroTight sleeve through the union and into ferrule on the opposite side.
4) Tighten the fitting just enough to prevent unwanted movement of the sleeve. Do not overtighten.
5) Rotate each ferrule into the union until snug.

WARNING: It is very easy at this point to break the tip. Take care not to touch the tip to any surface.

Preparation of the PicoFrit® Column
1) Remove the ProFrit® column from its protective packaging according to the directions in Technical Note PF-4 “Handling PicoFrit Columns”.
2) Slide the distal, or non-tip, end of the column through the sleeve protruding from the fitting and union assembly.
3) Pull the column through the sleeve until the tip protrudes just a few millimeters from the sleeve.
4) Tighten the fitting around the column until no longer loose.
5) Seat the union in the tip-holder of the PicoFrit Probe and secure it in place with the union lock-down screw.

Plumbing the MicroTee
The MicroTee joins the transfer line to the PicoFrit® and supplies the high voltage.
1) Unscrew the ferrule and cap from the MicroTee. Orient the MicroTee as shown in Figure 2 so the platinum electrode is facing away from the user and the setscrews are visible.

WARNING: Do not loosen the setscrews or remove the electrode cap, as this may damage the electrode. The solvent will not become charged, and an electrospray will not form.
2) Thread the end of the PicoFrit® tubing through a green MicroTight® sleeve, which is used for assembly with 360 µm OD tubing. Make sure the PicoFrit does not extend past the tubing sleeve end that will be inserted into the MicroTee. Thread the sleeved PicoFrit through the fitting nut and a black MicroFerrule, as shown in Figure 3.

3) Cleave the end of the PicoFrit after the tubing is threaded through the sleeve, nut, and ferrule according to the lengths recommended in Table 1. Instructions for cleaving fused silica may be found in Technical Note FS-1 “Cleaving Fused-Silica Tubing”.

4) Slip the end of the tubing through the right post of the MicroTee, as viewed in Figure 4A, until the tubing and sleeve seat against the bottom ledge inside the post, as shown in Figure 4B.

5) Screw the nut finger-tight onto the MicroTee.

6) Insert the distal end of the fused-silica transfer line through a green MicroTight sleeve and then through the nut and the black MicroFerrule, as shown in Figure 5A.

7) Carefully trim the end of the transfer line.

8) After trimming, insert the assembly back into the MicroTee, seat the transfer line, ferrule, and sleeve against the PicoFrit, and finger-tighten the nut, as shown in Figure 5B.

9) Gently pull on the tubing ends to ensure the connection is tight. Check for leaks by running solvent through the tubing at the expected operating pressure. If leaks appear, solvent collects at the exposed ends of the sleeves.

<table>
<thead>
<tr>
<th>PicoFrit Column Bed Length (cm)</th>
<th>MicroTee Pocket A Cut to Length (cm)</th>
<th>MicroTee Pocket B Cut to Length (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15</td>
<td>---</td>
</tr>
<tr>
<td>5</td>
<td>15</td>
<td>---</td>
</tr>
<tr>
<td>10</td>
<td>15</td>
<td>22</td>
</tr>
<tr>
<td>15</td>
<td>20</td>
<td>27</td>
</tr>
<tr>
<td>20</td>
<td>20</td>
<td>32</td>
</tr>
</tbody>
</table>

**TABLE 1: Recommended PicoFrit column trim lengths and MicroTee pocket positions**

CAUTION: Do not overtighten fittings. Overtightened fittings contribute highly to clogging, as it can cause fused silica to crack, introducing shards into the line.
The PicoFrit® Probe contains two pockets designed for holding the MicroTee. Position A, the pocket closest to the tip, allows for short columns. As shown in Figure 6, position excess column length at the front of the probe to allow Z-adjustment.

For longer columns, seat the MicroTee in Position B with excess column length threaded through the two notches to create a loop (Figure 7). The flexible diameter of the resulting loop facilitates Z-adjustment of the tip holder.

With the tip, MicroTee, and PicoFrit column properly positioned, connect the top and base of the probe with an Allen wrench using the cover screw, as shown in Figure 8. Properly tightening the cover screw ensures the MicroTee seats securely and that the column moves freely when the slide is adjusted.

The PicoFrit Probe is now ready for use in your Thermo Finnigan™ Ion Max™ source.
Installing the PicoFrit® Probe into the Ion Max™ Source

1) Before swapping probes, deactivate the auxiliary and sheath gas and remove the fittings from the source. Refer to your instrument operator manual for additional information on this procedure.

2) Disconnect the high voltage cable from the contact on the existing ESI probe and remove from source.

WARNING: Turn off or disconnect all power prior to performing any service or any devices attached to it to avoid potentially lethal electrical voltages.

3) Retract the Z-adjustment slide of the PicoFrit® Probe to avoid breakage.

4) Position the tip holder towards the probe port of the Thermo Finnigan™ Ion Max™ probe. If extra column length extends from the front of the probe when seating the MicroTee in Position A, carefully roll the loop into the probe port to minimize pinching or stress. Figures 9 and 10 demonstrate proper technique.

5) Slowly guide the tip holder of the probe into the probe port of the Thermo Finnigan Ion Max source. Where contact with the tip can result in damage, monitor installation via the side window of the Ion Max.

6) Slide the metal interlock pin of the probe cover into the side slot of the ESI Interlock block.

7) Rotate the probe clockwise until it stops and slowly glide the probe forward into the source. The probe is now ready for fine adjustment.

Fine Adjustment of the PicoFrit Probe

1) Move the Z-adjustment slide located at the rear of the probe to control the distance of the tip to the MS inlet. Monitor tip position through the side window (Figure 11) of the Ion Max™ source to avoid breakage.

2) Turn the tip angle control on the tip holder to present the plume at the desired angle.

When ready to use, insert the high voltage cable into the HV contact as demonstrated in Figure 12.
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 achieving spray stability.

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.

An even plume of droplets (Figure 13) observed in the magnified image indicates an optimal spray. A direct linear stream can indicate the voltage is too high, while large droplets can form on the tip if voltage is too low.

Packing List

The PicoFrit® Probe is supplied with the following items:

1  PicoFrit® Probe (Part No. ADPC-IMS )
1  MicroTee (Part No. NOP-775)
1  MicroTight® union (Part No. NOP-720)
2  Additional tip holder screws
1  Allen wrench
1  Tech Note PF-7 “Using the ADPC-IMS PicoFrit® Probe for the Thermo Ion Max™ Source”
1  Tech Note FS-1 “Cleaving Fused-Silica Tubing”