Caring for Turbo V™ Ion Source Electrodes

The Turbo V ion source requires smaller diameter electrodes when used with Eksigent microLC systems. The information and recommendations in this document are designed to guide you in the care and use of these electrodes. Follow the instructions below to help maximize electrode and column performance.

Prevent a Plugged Electrode

Follow the instructions below to prevent plugging the electrode on the ion source.

Be Careful with PEEKsil Tubing

- Never cut PEEKsil tubing.

  Cutting PEEKsil tubing can result in small particles of cut glass entering the flow path, leading to plugged tubing and electrodes. Use a shorter piece of tubing rather than cutting tubing.
- When connecting PEEKsil tubing:
  a. Connect the tubing on the end farther from the mass spectrometer first.
  b. Turn on the pump and allow liquid to flow through the tubing to flush out any particulate matter.
  c. Allow flow for ~30 seconds before making the next connection.
- Don’t overtighten connections to PEEKsil tubing.

  Overtightening can damage tubing and lead to plugged tubing. Instead, tighten fittings until finger-tight, turn on the pump and check for solvent at the fitting. If there is a leak, tighten the fitting about 1/16 turn at a time until there are no more leaks.

Prepare Clean Samples

The flow path can clog if samples contain too much particulate matter.

- Use HPLC- or MS-grade solvents at all times.
- Avoid the use of non-volatile salts and buffers such as CHAPS, phosphate, TRIS, HEPES and perchlorates. These additives can foul the electrospray source and mass spectrometer orifice.
- Avoid overloading the column with sample.
  - For 0.3 mm and 0.5 mm ID columns—use <12 µg of material
  - For 1 mm ID columns—use <50 µg of material
- AB SCIEX recommends pre-filtering samples with 0.45 µm pore filters to remove particulate contaminants which may cause clogging.
- If needed, centrifuge all samples at 10 000 RPM for 5 minutes to remove dust and particulates from the sample solution.

- If sample filtration or centrifugation is not sufficient, the following techniques can be used:
  - Protein precipitation (for biological samples)
  - Liquid-liquid extraction
  - Solid-phase extraction

Use a Guard Column or an In-line Filter

A guard column or filter can be used to remove particulates.

Install a Guard Column

To use a guard column, place it before the analytical column. Two columns are available from Eksigent. Both columns have a pressure limit of 5 000 psi.

- C8 guard column (PN 5028659)
- C18 guard column (PN 5028658)

Refer to *Installing an Eksigent Guard Column* (included with the guard column) for detailed installation instructions.

Install an In-line Filter

A filter, consisting of a filter housing (PN 200-00388) and a capsule (PN 200-00373), is available from Eksigent. The filter has a pressure limit of 5 000 psi.

Install the filter between the column and the electrode, using two 5 cm pieces of tubing. Select tubing based on flow rate:

- Flow rates >~20 µL/min—50 µm ID tubing (PN 205-00070)
- Flow rates <~20 µL/min—25 µm ID tubing (PN 205-00089)

Alternately, if there is no guard column in use, install the filter before the column, using 10 cm of PEEKsil tubing between the filter and the column.

Note: The filter capsule has no preferred orientation, but after the first use, do not change its orientation in the filter housing.

Limit the Temperature of the Electrode

Ionization efficiency is a function of LC flow rate, temperature, and gas flow in the ion source. Most of the analytes analyzed at flow rates between 5 µL/min to 60 µL/min are expected to generate highest sensitivity at temperatures below 450°C. High temperatures can boil the mobile phase at the electrode tip and lead to the accumulation of solids from mobile phase additives and samples at the electrode tip.

If your experiment requires a higher temperature, use a 65 µm electrode for the ion source (PN 5029342). The 65 µm electrode is stainless steel and conducts heat away from the tip better than the first generation 25 µm and 50 µm electrodes, which have a component made of PEEK near the tip.

Adjust the flow rate as needed to accommodate the larger diameter of the electrode.

Flush the Electrode at the End of the Batch

If the LC stops flowing while the ion source is hot, salts from the buffer or sample can crystallize and lead to a plugged probe.

1. In the Analyst® software, add a run to the end of the batch with no sample injection. Create a method with a lower ion source temperature and where flow from the LC system continues.

   Use the flow rate appropriate for your system:

   - High-flow configuration—20 µL/min
   - Low-flow configuration—10 µL/min

2. After 20 minutes or the ion source is cool enough to touch, stop the flow from the LC system.
3. (Optional) Remove the electrode and clean with water.

Clean a Plugged Electrode

If an electrode is plugged, the procedures below may remove the plug.

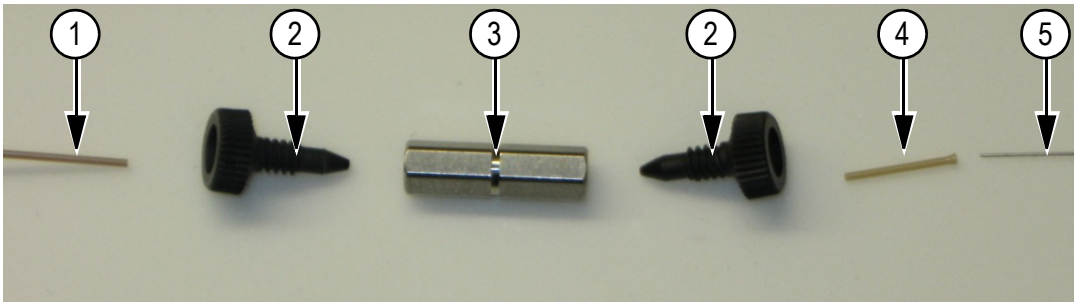
Backflush the Electrode

Flush the electrode using the pump.

Required Materials
• Stainless steel hex union (PN 5016413)
• Flared sleeve (PN 5025189)
• 2 PEEK fittings (PN 200-00342)

1. Remove the column from the flow path and connect the tubing with a union.
   a. Loosen the black PEEK fitting from the upstream end of the column.
   b. Remove the 1/32 inch OD PEEKsil tubing from the column and connect it to the stainless steel hex union (item 3 in Figure 1) using the fitting.

Figure 1 Components for Connecting the System to Backflush the Electrode



Item	Description
1	1/32 inch OD PEEKsil tubing
2	PEEK fittings
3	Stainless steel hex union
4	Flared sleeve
5	Electrode assembly

c. Insert the sleeve (item 4) into a black PEEK fitting. Insert the sleeve with the flared end of the sleeve at the rear of the fitting.

d. Insert the probe tip (item 5) into the sleeve using the flare as a guide.

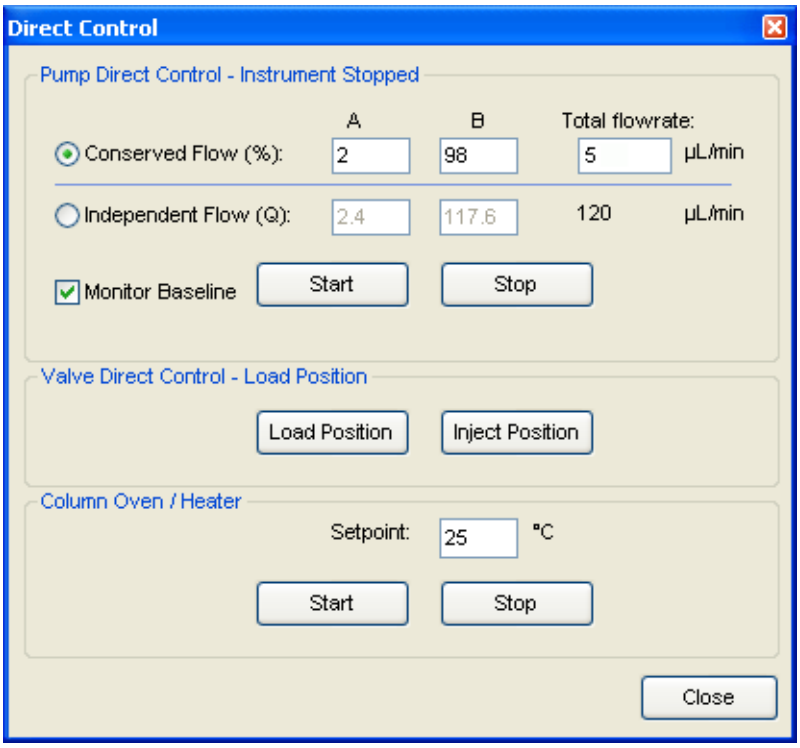
   The first time the sleeve is used, make sure that the sleeve is pushed as far as possible into the union. After the first use, the sleeve will remain in the fitting.

e. Tighten the fitting into the union so that the probe tip is snug.

2. Start the pumps.

   a. Select **System > Direct Control** in the Eksigent control software to open the Direct Control dialog.

Figure 2 Direct Control Dialog



b. Select **Monitor Baseline** to monitor pressure while backflushing.
c. Specify the flowrate to be **5 µL/min**.
d. Specify **A** and **B** so the solvent pumped is mostly organic.
e. Click **Start**.

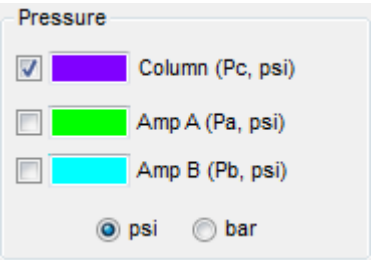
Caution: Potential System Damage: Do not allow the pressure to exceed 10 000 psi. Stop the pump if the pressure is greater than 10 000 psi.

3. Monitor the baseline in the Acquisition window.

   If necessary, configure the window so that Pc appears in the plot.

   a. Select **System > Appearance Settings**.
   b. In the **Appearance Settings** dialog, select the **Column (Pc, psi)** check box in the **Pressure** section (Figure 3).
   c. Click **OK**.

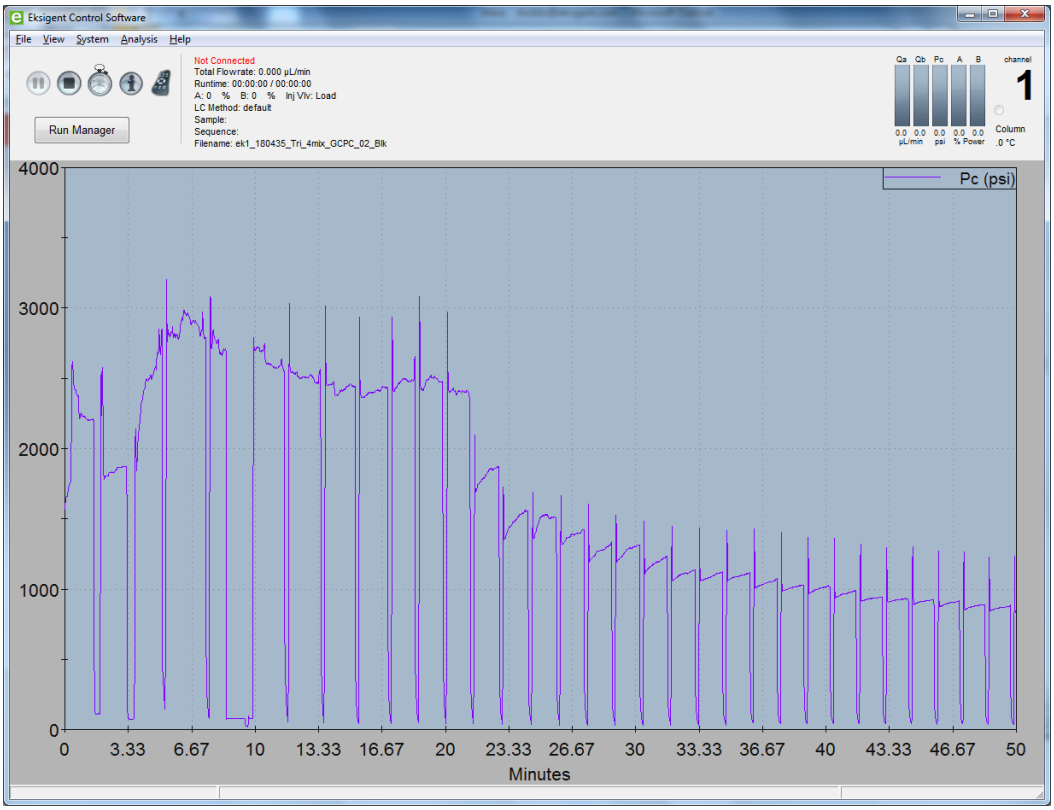
Figure 3 Appearance Settings Dialog—Pressure Section



4. Increase the flowrate, up to the maximum for the instrument's configuration (below) and pump until either the probe backpressure is reduced and stable or for 20 minutes, which ever comes first.
   - High-flow configuration—200 µL/min
   - Low-flow configuration—50 µL/min

Tip! Each time the pump restrokes, the Eksigent control software displays the Alerts log. The restrokes are expected and occur when the pump is empty and has to refill. Minimize the window to hide the log.

Figure 4 Acquisition Window, Showing Pressure Drop and Restrokes



The pump can be left unattended and be allowed to restroke repeatedly to dislodge the plug. The pressure fluctuations due to restroking or changing flow rate will aid in dislodging the blockage. If necessary the probe can be inserted into an ultrasonic bath while backflushing.

5. Click **Stop**.

   If the pressure dropped, the plug has been dislodged. Remove the union and reconnect the column into the flow path.

   If the pressure is still high, follow the procedure below to sonicate the electrode.

Sonicate the Electrode

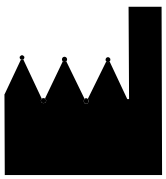
Sonicate the electrode only if backflushing did not remove the plug.

Required Materials
• Sonicator • Beaker • Acetone, methanol, ethanol, or warm water

1. Remove the electrode from the system.
2. Place the tip of the electrode in the beaker then add enough solvent to cover the tip and then a bit more. Sonicate for 10 minutes.
3. Replace the electrode in the source and backflush a second time.

Revision History

Revision	Reason for Change	Date
D5066063 A	First release of document.	May 2013
5028148	Replaced document number with part number.	June 2013
5030112	Updated part numbers for guard columns and electrodes.	October 2013
5031119 RUO-IDV-05-1182-A	Added instructions for replacing electrode emitter. Reorganized filter and guard column instructions to reflect best practices.	February 2014
5035371 RUO-IDV-05-1182-B	Updated introductory paragraph and title to indicate these instructions pertain to electrodes for the Turbo V ion source. Removed instructions for assembling and installing a guard column; referred user to document included with guard column. Removed instructions for replacing electrode emitter.	August 2014



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