Agilent 6200 Series Time-of-Flight LC/MS

Maintenance Guide

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Agilent Technologies

Notices

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Safety Notices

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In This Guide...

This guide contains information to install, use, maintain, and troubleshoot the Time-of-Flight LC/MS.

1 Basic Operation

This chapter contains procedures for the basic operation of your Time-of-Flight LC/MS.

2 Maintenance

This chapter contains procedures for the maintenance of your Time-of-Flight LC/MS.

3 Troubleshooting

This chapter describes troubleshooting steps to take when you have problems with the Time-of-Flight LC/MS hardware.

4 Reference

This chapter contains safety information and technical specifications for the Time-of-Flight LC/MS.

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1
Basic Operation

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This chapter describes procedures for the basic operation of your 6210 or 6220 instrument.

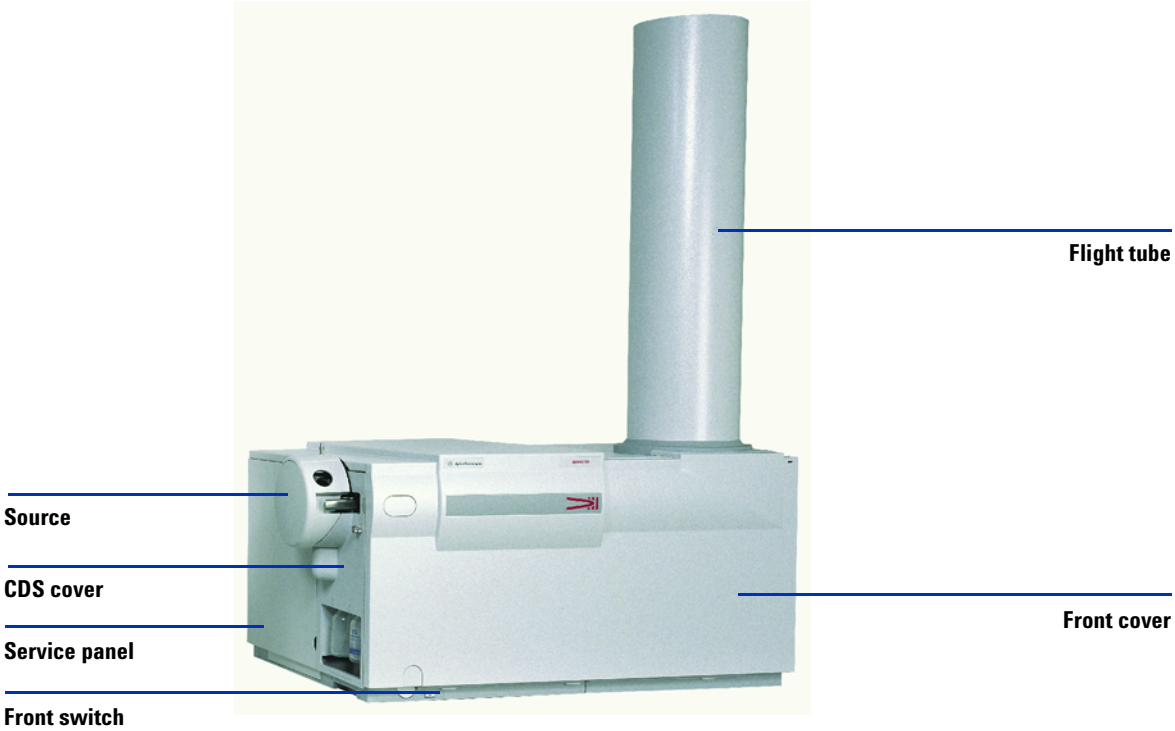


Figure 1 Time-of-Flight LC/MS covers. The main power switch is behind the service panel.

For more details, see the site preparation guide that is sent to customers before installation. If you move the instrument, you must follow and repeat the steps detailed in the site preparation guide.

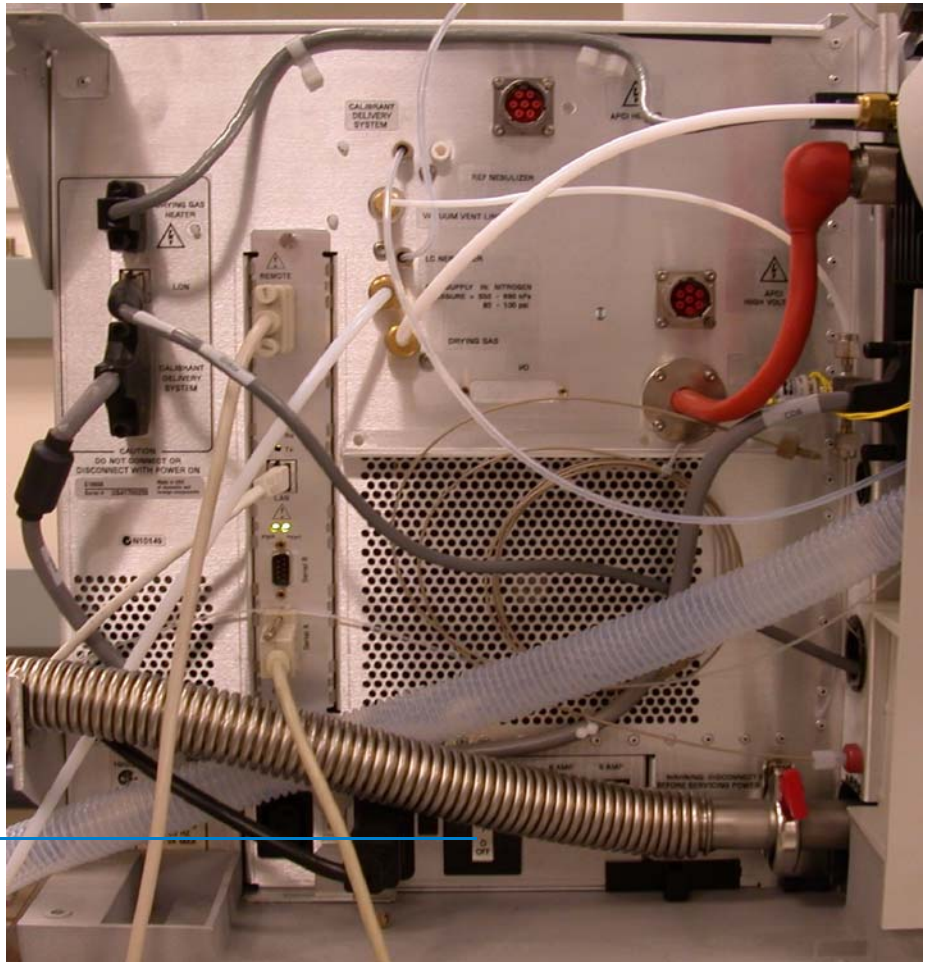
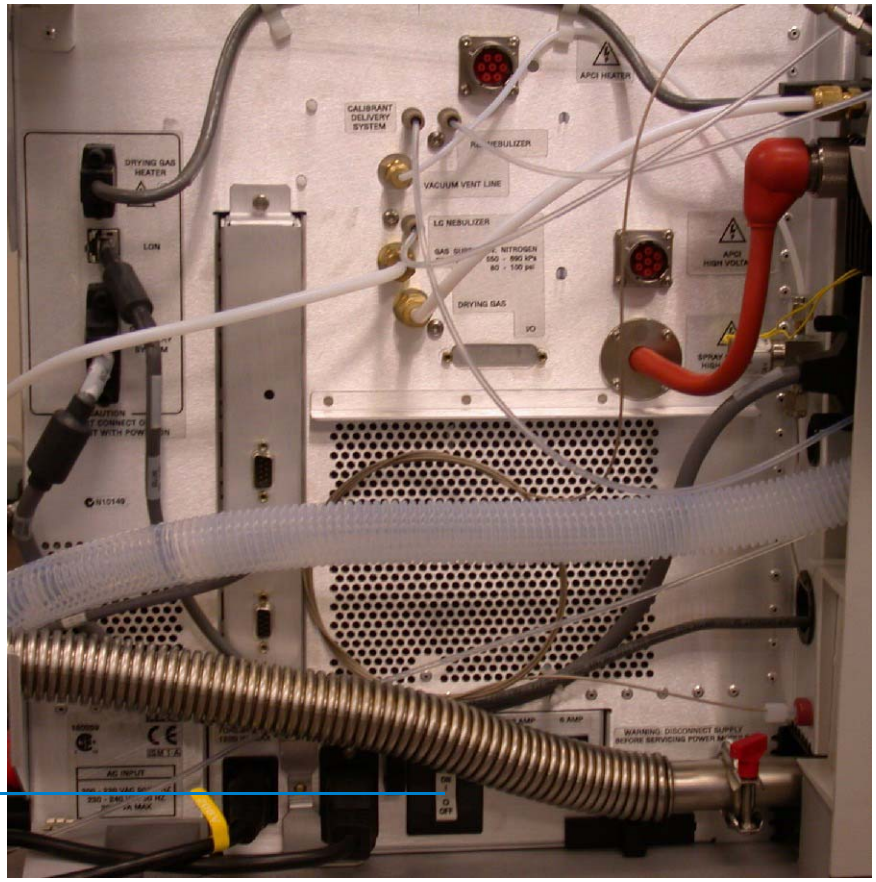


Figure 2 Connections behind service panel for 6210 systems equipped with SmartCard 3 processor board



Main Power Switch

Figure 3 Connections behind service panel for 6210 or 6220 systems equipped with SmartCard 4 or 5 processor board.

Starting and Shutting Down

This section describes how to start up your 6210 or 6220 and how to shut it down.

The 6210 or 6220 instruments have three standard modes of operation that you can select from the software.

On Mode

On is the fully operational status of the system. The instrument and the source are on. The MS TOF icon indicator is green, unless a method is running. When a method is running, the MS TOF icon indicator is blue.

To put an individual device in the On mode, right click the image of the device in the instrument pane and select **On**. To put all devices in the On mode, click the **On** button in the instrument status pane.

Standby Mode

In *Standby*, the source interface is on, the drying gas remains heated, and nebulizer flows are maintained. The source and ion optics voltages are turned off, and the mass spectrometer is not sending spectra to the PC. However, the TOF high voltage electronics are on.

For APCI and MMI sources, the vaporizer gas heaters are turned down.

This mode lets the instrument remain idle for periods lasting from minutes to days. You should place the instrument in Standby mode when it is not in use or when you want to change the ion source.

To put an individual device in the Standby mode, right click the image of the device in the instrument pane and select **Standby**. To put all devices in the Standby mode, click the **Standby** button in the instrument panel.

Off Mode

In *Off* mode, the source interface temperatures and voltages are turned off. The flight assembly high voltages are also turned off.

If a serious error occurs, such as the loss of drying gas or nebulizer gas, the instrument is set automatically to the Off mode. Simultaneously, the status indicator displays “Error” and the MS TOF icon turns red.

However, the vacuum system is still on and the software is running. When you put the system back to the On mode from the Off mode after an extended period of time, allow several hours for the flight assembly voltages to achieve optimal stability.

To put an individual device in the Off mode, right click the image of the device in the instrument pane and select **Off**.

Powered Off State

When the system is in the powered off state, the system is vented and the instrument is turned off to allow maintenance or to prepare for long periods when the instrument is not used.

NOTE

Before you move the instrument to another location, maintain it, or expect it to be idle for long periods of time, turn off both the front switch and the main power breaker switch behind the service panel.

To start the system in Standby mode

This procedure brings the system from the powered off state to Standby mode.

1 Check that nitrogen gas is present at the nitrogen port behind the service panel in the purity and pressure specified in the site preparation guide.

2 Check that the rough pump is connected to the 6210 or 6220 instrument.
   - Check that the rough pump power cable is connected to the **FORELINE PUMP** power receptacle behind the service panel of the 6210 or 6220 instrument.
   - Check that the rough pump has the correct voltage rating for your AC power (230 V or 208 V).
   - Check that the rough vacuum tubing is connected to the inlet part of the rough pump.
   - Check that the switch on the rough pump is in the On position.

NOTE A silicone rubber septum is placed between the spray shield and the capillary for shipment. Make sure that this rubber septum is removed if you are starting the system for the first time.

CAUTION Do not connect the drain bottle tubing to the rough pump, or the rough pump tubing to the drain bottle.

3 Check that all pump and spray chamber exhausts are vented outside the laboratory.

CAUTION Vent all exhausts external to the building where they cannot be recirculated by environmental control systems. Do not vent exhaust into your laboratory. The exhaust fumes from the vacuum pumps and spray chamber contains trace amounts of the chemicals you analyze. Health hazards include chemical toxicity from solvents, buffers, samples and pump fluid vapor, as well as potentially biohazardous aerosols of biological samples.

The oil mist filter must be installed on the rough pump, and the ballast valve must be closed. Open the ballast valve periodically (must be done on a weekly basis) to return any oil that has condensed in the oil mist filter to the pump as needed.

4 Turn on the power for the computer and monitor.

5 Turn on the main breaker behind the service panel.

6 Turn on the front switch of the 6210 or 6220 instrument.

This starts the pumping system and the electronics.

7 After Windows starts, start the TOF diagnostics program:

- For A.02.02, click **Start > All Programs > Agilent > TOF Software > Tools > TOF Diagnostics**.
- For B.01.03, click **Start > Programs > Agilent > MassHunter Workstation > Acq Tools > TOF Diagnostics**.

8 From the **Connection** pull-down menu, select **Connect** and click **OK.** See Figure 4 on page 17.

9 In the System Turn-on pane, mark the check boxes for **Pump Down** and **HV Condition**.

10 Click the **Start** button in the System Turn-on pane.

The system monitors the vacuum pressure. Once the vacuum pressure reaches 3 x 10^{-6} torr, the system will begin conditioning the high voltage electronics.

11 Once high voltage conditioning has completed, start the TOF acquisition program:

- For A.02.02, click **Start > All Programs > Agilent > TOF Software > MassHunter Workstation.**
- For B.01.03, click **Start > Programs > Agilent > MassHunter Workstation> Data Acquisition**.

The system will come up in Standby mode.

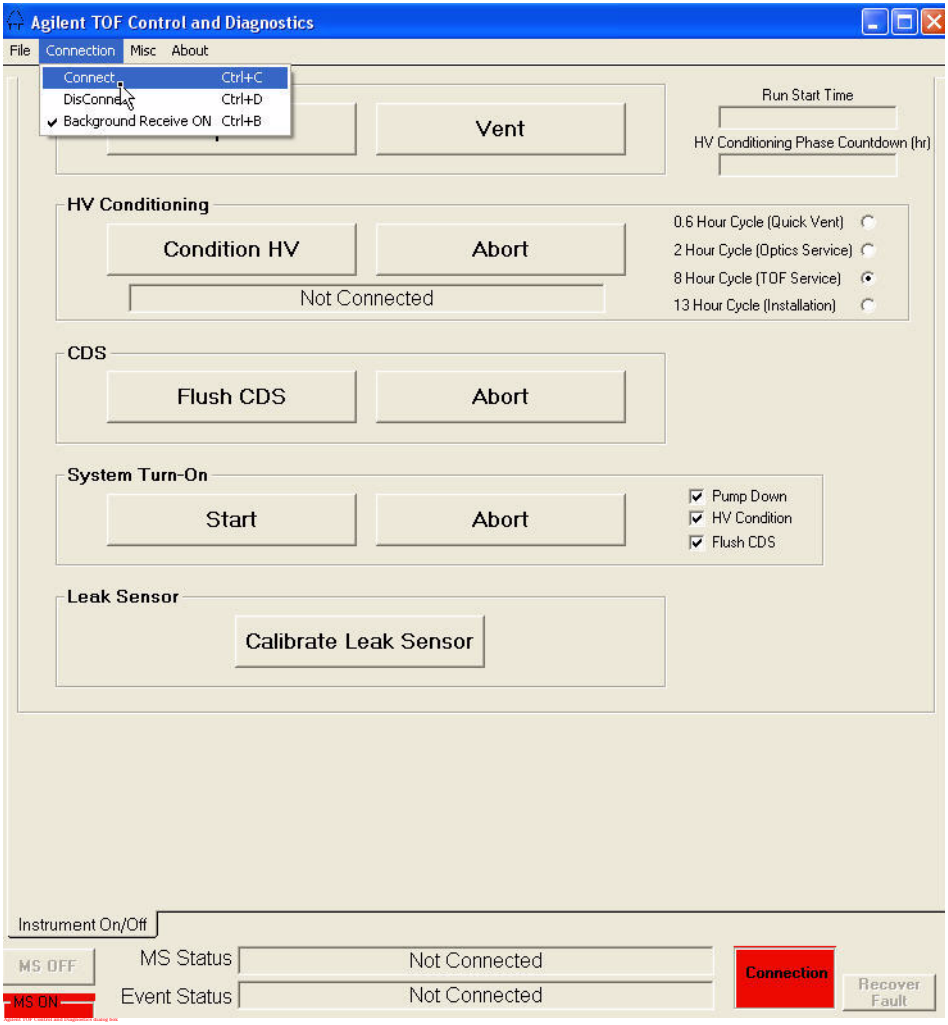


Figure 4 Click Connect from the Connection menu to connect to the TOF.

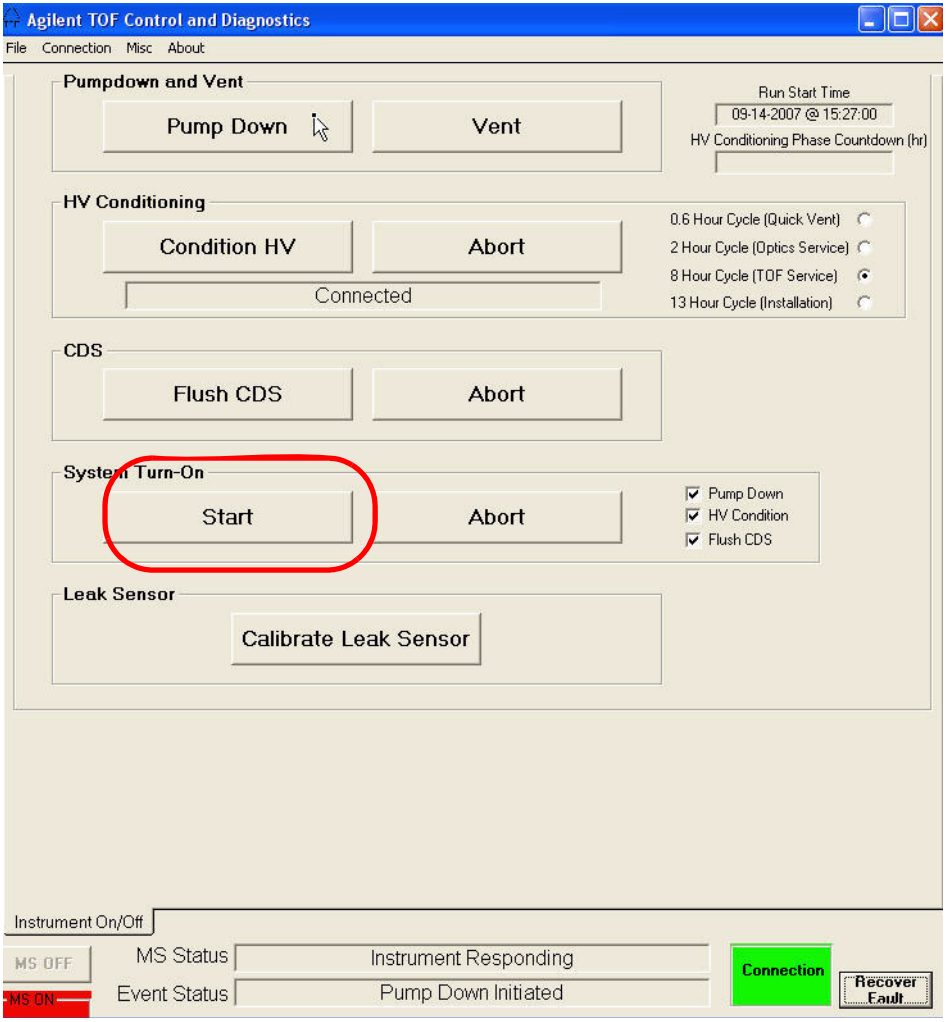


Figure 5 Click the Start button in the System Turn-On pane to start the system.

By default, the ESI source values are set to:

- Drying gas flow: 3.5 L/min.
- Drying gas temperature: 300 °C
- Nebulizing gas pressure: 20 psi.

NOTE You will not be able to set the flows while the TOF system is in a “Not Ready” state. The system will remain in the Not Ready state while the high vacuum pressure is above 3.5 X 10^{-6} torr.

12 Allow the TOF instrument to continue pumping down.

The Rough Vacuum pressure (pressure at rough pump) should be about 2 to 3.5 torr. The High Vacuum (pressure in the flight tube region) should be in the 10^{-6} torr range within two to three hours. The High Vacuum reading should be in the 10^{-7} torr range after pumping overnight to several days.

Leave the TOF system in Standby.

To change from Standby to On

Use this procedure when the Time-of-Flight LC/MS is in Standby mode and you need to bring it into the On mode.

Before you begin, check that:

- The Time-of-Flight LC/MS is in Standby mode

1 In the Time-of-Flight LC/MS software, click **On**.

   You can only activate the Standby mode or On mode if the ion source is mounted and closed. You can now select parameters for your measurement.

2

3 Turn on the LC and the TOF.

   Click **On** in **Status** pane to turn on the TOF system.

The instrument is now in the On mode.

To change from On to Standby

Put the Time-of-Flight LC/MS in Standby mode when you have completed your analysis of samples.

Before you begin, check that:

- You have completed tuning and data acquisition and you have saved all data as needed (described in later chapters).

- In the MassHunter Acquisition program, click **Standby**.

  The drying gas and nebulizer gas flows are maintained along with the drying gas temperature. The spray chamber high voltages are turned off in Standby mode. Voltages in the flight tube remain On in Standby mode to maintain stable operating voltages. The mass spectrometer stops generating spectra.

The system is now in Standby mode.

To shut down the instrument

You can turn off the Time-of-Flight LC/MS when the instrument will be idle for an extended time or when you must do maintenance.

Before you begin, check that you have completed tuning and data acquisition and you have saved all data as needed.

CAUTION When you choose Vent from the MS TOF shortcut menu, the Time-of-Flight LC/MS does not turn off. You must follow the entire shutdown procedure.

WARNING **The spray shield and related spray chamber components are likely to be hot. Do not touch them!**

1. Turn off the solvent flow.

   If you have analyte in any of the components in the sample delivery system, flush the delivery system with pure solvent before you continue.
2. Check that the ballast valve on the rough pump is closed.
3. In the TOF Software, right-click on the TOF icon and select **Vent**.

   When you vent the system, these parts are turned off: spray chamber high voltages, the drying gas heater, the nebulization flow, the detector, and other lens voltages. The drying gas flow is set to 3.5 L/min. If the APCI source is installed, the vaporizer heater is also turned off.

   The Rough Pump turns off when the turbo speed is below 20 percent.
4. Wait until the TOF instrument completely vents (reaching 760 torr) to ensure that the flight tube is filled with nitrogen.

   This protects the detector from moisture damage.
5. Turn off the front switch located in the lower left front corner of the instrument.
6. Turn off the main breaker located behind the service panel.
7. At this point, you can also shut down the PC.

The system is now shut down.

Changing Ion Sources

This section describes the steps to change from one ion source to another. First, you need to follow the set of steps to remove your current source. Then, you need to follow the steps to install the new source.

To remove an electrospray source

WARNING **The spray chamber operates at very high temperatures. Let the spray chamber cool before you continue.**

1. Put the instrument in Standby mode.
2. Slide open the nebulizer cover.
3. Disconnect the LC nebulizer tubing and LC nebulizing gas tubing.
4. Disconnect the Reference nebulizer tubing and Reference nebulizing gas tubing.
5. Open the spray chamber.
6. Plug the reference nebulizer gas outlet on the side of the instrument, under the service panel.
7. Remove the spray chamber by lifting it along the axis of its hinges.

To remove an APCI or APPI source

1. Put instrument in Standby mode.
2. Disconnect the APCI or APPI heater cable.
3. For APCI source only, disconnect the APCI high voltage cable.

WARNING **The spray chamber operates at very high temperatures. Let the spray chamber cool for 10 minutes before you continue.**

4. Disconnect the nebulizing gas tubing and LC tubing from the nebulizer.
5. Open the spray chamber.
6. Remove the spray chamber by lifting it along the axis of its hinges.

To remove a nano ESI source

WARNING **The spray chamber operates at very high temperatures. Let the spray chamber cool before you continue.**

1. Put instrument in Standby mode.
2. Remove the nanospray needle assembly from the source.
3. Clean the nanospray needle with 100% acetonitrile. You can also use any solvent that dissolves the last sample run.

CAUTION Do not let the sample recrystallize inside the needle, which leads to plugged needles. Follow the steps above to prevent plugged needles.

4. Disconnect the power cable from the lamp.
5. Open the spray chamber.
6. Remove the spray chamber by lifting it along the axis of its hinges.
7. Remove the capillary cap and drying gas diverter.
8. Install the standard capillary cap and spray shield. For the standard spray shield, the small drying gas exit hole should be oriented above the main hole, or entrance to the capillary cap.

To remove a dual nano ESI source

WARNING **Do not touch the top of the dual nanospray source or the capillary cap. They may be very hot. Let the parts cool before you handle them.**

1. Put instrument in Standby mode.
2. Remove the reference and LC nanospray needle assembly from the source.
3. Clean both nanospray needles with 100% acetonitrile. You can also use any solvent that dissolves the last sample run.

CAUTION Do not let the sample recrystallize inside the needle, which leads to plugged needles. Follow the steps above to prevent plugged needles.

4. Disconnect the power cable from the lamp.
5. Open the spray chamber.
6. Remove the spray chamber by lifting it along the axis of its hinges.
7. Remove the capillary cap and drying gas diverter.
8. Install the standard capillary cap and spray shield. For the standard spray shield, the small drying gas exit hole should be oriented above the main hole, or entrance to the capillary cap.

To install an electrospray source

1 Install the electrospray spray chamber.
2 Close the spray chamber.
3 Connect the nebulizing gas tubing and LC tubing to the analytical nebulizer.
4 Connect the nebulizing gas tubing and LC tubing to the reference nebulizer.

CAUTION Do not touch the nebulizer tip to any surface. The needle is not protected. If you damage the needle, you get bad system performance.

5 Load or create the desired analytical method.

   This will load the appropriate gas flows, temperature set points and fragmentor voltage. After the method is loaded, the source should be allowed to heat to its working temperature.
6 In the left side of the **MS TOF** tab, in the Method pane, select **ESI** as the **Ion Source**.
7 Click **Apply**.
8 Click **On** and let the source heat up to its working temperature.
9 Allow the source to heat to its working temperature.
10 Load **ESIAutotune.m**. Perform a checktune to verify the source is operational.
11 Load or create the desired analytical method.

   This will load the appropriate gas flows and temperature set points.

To install an APCI source

1. Close the ESI reference nebulizer gas tubing with a cap. A threaded plug is provided for this purpose.
2. Install the APCI spray chamber.
3. Close the spray chamber.
4. Connect the nebulizing gas tubing and LC tubing to the APCI nebulizer.
5. Connect the APCI heater cable and the APCI high voltage cable.
6. In the left side of the **MS TOF** tab, in the Method pane, select **APCI** as the **Ion Source**.
7. Click **Apply**.
8. Put the instrument in **On** mode.

   Let the source heat to its working temperature. Typically, this takes about 15 minutes.
9. Load **APCIAutotune.m**. Perform a checktune to verify the source is operational.
10. Load or create the desired analytical method.

    This will load the appropriate gas flows and temperature set points.

To install an APPI source

1. Install the APPI spray chamber.
2. Close the spray chamber.
3. Connect the vaporizer heater cable to the APCI heater connector which is located on the left side of the instrument chassis.
4. Connect the APPI power supply cable to the APPI power DB9 connector and screw it in.
5. Connect the other end of the cable to the SmartCard serial port. If SmartCard has more than one serial port, connect to the Serial B port.
6. Plug the APPI power supply into an AC outlet using the power cord supplied with the APPI interface kit.
7. Connect the 1/8-inch nebulizing gas tubing from the TOF instrument mainframe to the nebulizer gas fitting.
8. Connect the LC tubing to the nebulizer zero dead volume fitting.
9. In the left side of the **MS TOF** tab, in the Method pane, select **APPI** as the **Ion Source**.
10. Click **Apply**.
11. Put the instrument in **On** mode.
12. Allow the source to heat to its working temperature. Typically, this takes about 15 minutes.
13. Load **APPIAutotune.m**. Perform a checktune to verify the source is operational.
14. Load or create the desired analytical method.

    This will load the appropriate gas flows and temperature set points.

To install a nano ESI, dual nano ESI, or multimode ionization source

- Refer to the *User Guide* for the source.

2 Maintenance

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This chapter covers common maintenance tasks. Do these tasks when scheduled to reduce problems, prolong system life, and reduce overall operation costs. (See Table 1 on page 31.) Keep a record of system performance characteristics and maintenance operations so that you can more easily detect variations from normal operation and to take corrective action.

NOTE For information on maintaining an APPI, a nanospray, a dual nanospray, or a Multimode Ionization source, please refer to the *User's Guide* for that product.

Table 1 Suggested maintenance schedule.

Task	Daily	Weekly	Twice/ year	As needed
Flush sample path	X			
Clean spray chamber, spray shield, capillary cap, contacts and the tip of the corona needle (APCI)	X			
Check rough pump fluid level		X		

Table 1 Suggested maintenance schedule.

Task	Daily	Weekly	Twice/ year	As needed
Clean capillary			X	X
Replace rough pump fluid			X	X
Inspect hoses, power cords, and cables			X	
Empty the drain bottle			X[1]	
Clean cabinet				X
Replace nebulizer needle				X
Clean or replace entire capillary				X
Clean or replace skimmer				X
Replace Electron Multiplier				X
Replace gas conditioner			X	X
Replace corona needle			X	X
Clean Ion optics				X

1 The drain bottle filling rate depends on the type of the analysis. The filling rate is particularly higher if high-flow LC conditions are used together with the divert valve and the option “to waste” on the MS TOF Tune tab. Find out the appropriate interval for emptying the drain bottle.

Cabinet

The instrument cabinet requires very little maintenance. You should keep it clean. Every 6 months, inspect the power cord, cables and exhaust hoses.

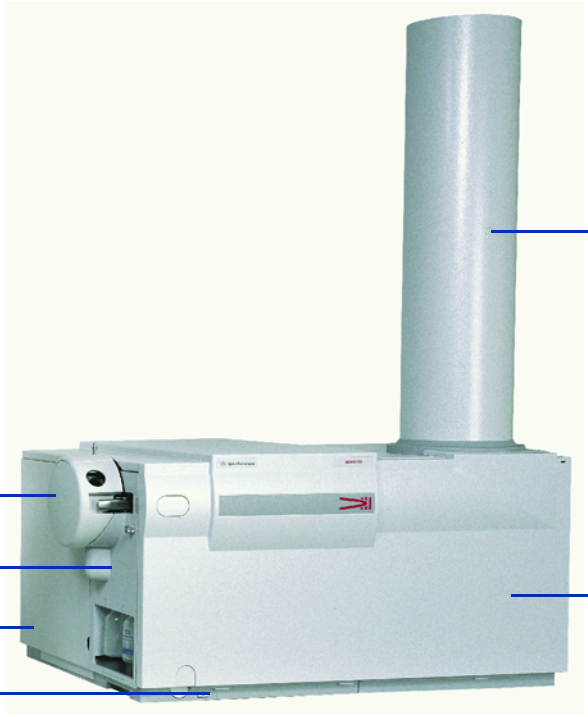

Figure 6 Instrument covers

To inspect hoses, power cords, and cables

Every 6 months, inspect the instrument. Check the hoses for bends, pinches or anything else that prevents flow. Check the power cord and cables for damage and make sure their connections are secure.

- Make sure the exhaust hose is securely connected to the outlet of the rough pump. Check for condensation at low points in the hose. It will help support the correct exhaust flow. Make sure the hose is not bent sharply.
- Make sure the drain hose is securely connected to the spray chamber and to the drain bottle. Do not allow fluid to collect at a low point in the hose before it reaches the drain bottle. Keep the drain bottle upright.
- Make sure the power cord is securely plugged into its outlet. Make sure the cord is not being pinched or bent by other equipment.
- Make sure all cables are securely plugged into their outlets. Make sure no cable is being pinched or bent by other equipment.
- Make sure the filters behind the service panel are not clogged. Clean them using a vacuum cleaner.
- Make sure the air inlet of the rough pump is not clogged or blocked by any other object (box, carton). Clean it using a vacuum cleaner.

To remove the covers

Before you begin, check that you have:

- Screwdriver
- Torx 10 driver (p/n 8710-1623)

1. Put the instrument in Standby mode.
2. Remove the spray chamber.
3. Remove the TOF front cover.

   Push on the latches to the left and right and pull the front cover forward approximately 45 degrees. Gently lift the cover off and set aside.
4. Remove the flight tube collar:
   - Pull the two halves of the rubber seal.
   - Slide it out of its groove.
5. The TOF top cover is hinged in back. Lift the top cover until the holding cable is taut.
6. Remove the CDS cover:
   a. Remove the CDS bottles (A and B).
   b. Use a Torx T-10 driver to loosen the captive screw near the spray chamber latch.
   c. Loosen the second captive screw in the lower panel between the two CDS bottles.
   d. Slide the CDS cover away from the TOF side panel and set aside.

To install the covers

Before you begin, check that you have:

- Screwdriver
- Torx 10 (p/n 8710-1623)

1. Install the CDS cover:
   a. Slide the CDS cover onto the TOF side panel. Be careful not to pinch the CDS liquid tubing lines.
   b. Tighten the captive screw in the lower panel between the two CDS bottles.
   c. Tighten the captive screw near the spray chamber latch.
   d. Install the CDS bottles (A and B).
2. Lower the TOF top cover until the standoffs are resting on the Electronics Tub cover.
3. Install the flight tube collar:
   - Slide the collar along the groove.
   - Push the two halves of the rubber seal onto their mounting posts.
4. Install the TOF front cover:
   a. With the front cover at 45 degree angle, insert the tabs at the bottom of the cover.
   b. Lift to a vertical position and secure the latches.

Vacuum system

This section lists procedures to maintain the vacuum system of the instrument. They should be performed according to the maintenance schedule or as indicated by instrument symptoms. This section includes the following procedures:

- To check the rough pump fluid level
- To check the oil mist filter
- To add rough pump fluid
- To replace the rough pump fluid

To check the rough pump fluid level

Check the level and color of the pump fluid weekly.

- Check the fluid level in the window of the rough pump. The fluid level should be between the marks for Max and Min (see Figure 7).
- Check that the color of the pump fluid is clear or almost clear with few suspended particles.
- If the pump fluid is dark or full of suspended particles, replace it.

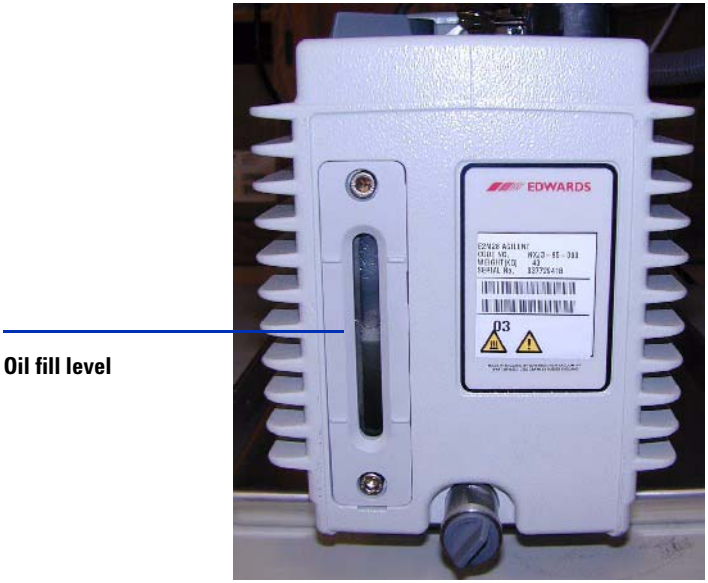

Figure 7 Fluid level window on the rough pump

To check the oil mist filter

Check the oil mist filter **weekly**.

- Check the oil mist filter.

  Check for any damage and if pump fluid has been collected in it.
- Check the oil mist filter for damage.

  If the oil mist filter is damaged, replace it.
- Check whether oil has collected in the bottom of the oil mist filter.

  If there is oil in the oil mist filter, open the gas ballast valve counterclockwise just enough to return the condensed oil back to the pump. Close the gas ballast valve clockwise.

NOTE When you close the ballast valve, you increase the efficiency of the pump. However, you lose oil to the mist filter if you don't recycle. Check the status of your oil mist filter at least once per week to ensure that it does not fill with oil. If you lose too much oil in the rough pump, the vacuum will not be maintained, and the TOF instrument will vent.

To add rough pump fluid

Add pump fluid when the pump fluid level is low. Before you begin, make sure you have:

- Funnel
- Gloves, chemical resistant, clean, lint free (p/n 9300-1751)
- Rough pump fluid (Inland 45 oil, p/n 6040-0834)

CAUTION Wear chemical-resistant gloves and safety glasses (goggles)

WARNING **Never add pump fluid while the pump is on.**

WARNING **The fill cap and pump may be dangerously hot. Check that the fill cup and pump are cool before you touch them.**

CAUTION Use only Edwards rough pump fluid (Inland 45 oil, p/n 6040-0834). Any other fluids can substantially reduce pump life and invalidates the pump warranty.

1. Vent and turn off the instrument.

   See "To shut down the instrument" on page 22.
2. Unplug the instrument power cord from the electrical outlet.

   Leave the power cord unplugged while you do this procedure.
3. Remove the fill cap on the rough pump (see Figure 8).
4. Add new pump fluid until the fluid level is near, but not over the maximum mark beside the fluid level window (see Figure 8).
5. Reinstall the fill cap.
6. Wipe off all excess oil around and underneath of the pump.
7. Reconnect the power cord.

8 Start up the instrument.

See "To start the system in Standby mode" on page 15.

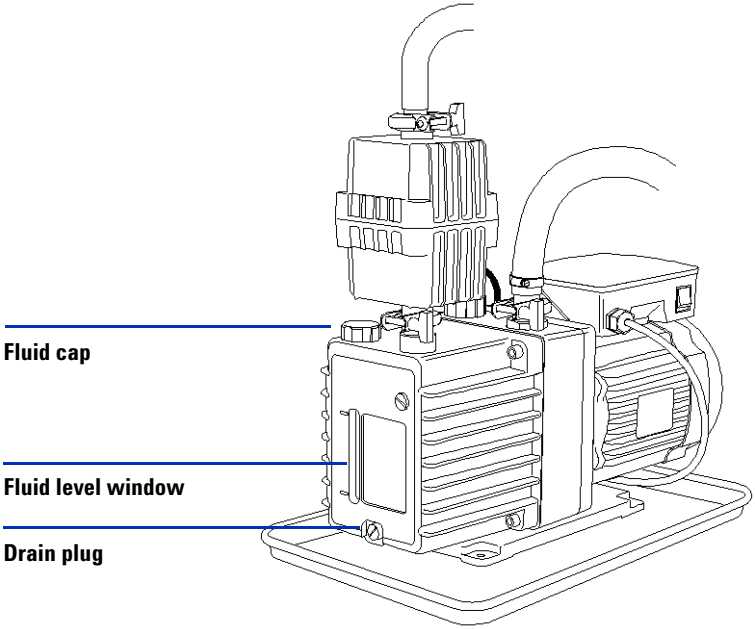

Figure 8 Rough pump.

To replace the rough pump fluid

Replace the pump fluid every six months. Replace it sooner if the fluid appears dark or cloudy.

Before you begin, make sure you have:

- Container for catching old pump fluid
- Funnel
- Gloves, chemical resistant, clean, lint free (p/n 9300-1751)
- Rough pump fluid (Inland 45 oil, p/n 6040-0834)
- Screwdriver, flat-bladed, large (p/n 8710-1029)

CAUTION Wear chemical-resistant gloves and safety glasses (goggles)

WARNING **Never add pump fluid while the pump is on.**

WARNING **The fill cap and pump may be dangerously hot. Check that the fill cap and pump are cool before you touch them.**

WARNING **Do not touch the fluid. The residue from some samples are toxic. Properly dispose of the fluid.**

CAUTION Use only Edwards rough pump fluid (Inland 45 oil, p/n 6040-0834). Any other fluids can substantially reduce pump life and invalidates the pump warranty.

1 Turn off the instrument.

See "To shut down the instrument" on page 22.

2 Unplug the power cord from the instrument

   Leave the power cord unplugged while performing this procedure.

3 Place a container under the drain plug of the rough pump (see Figure 8 on page 41).

4 Remove first the fill cap (see Figure 8 on page 41), then open the drain plug.

   Allow the fluid to drain completely.

5 Reinstall the drain plug.

6 Pour in new pump fluid until the fluid level is near, but not above the maximum mark beside the fluid level window (see Figure 8 on page 41).

7 Reinstall the fill cap.

8 Reconnect the power cord.

9 Start up the instrument.

   See "To start the system in Standby mode" on page 15.

10 After 30 minutes pump down, inspect the pump for leak.

   Inspect for leak after overnight pump down.

Electrospray Ion Source

This section describes the removal, disassembly, cleaning, and reassembly of the API-interface and the assemblies that make up the source.

To flush the nebulizer

When required Daily or at the end of each shift (or more often) to flush traces of samples and buffers out of the tubing, valves and nebulizer.

Tools required
- Acetonitrile, HPLC-grade or better
- Water, HPLC-grade or better

Parts required None

1 Make sure acetonitrile and water are two of the solvents installed in your liquid chromatograph.

NOTE This procedure applies to both electrospray and APCI nebulizer.

2 Set the liquid chromatograph to pump a mixture of 90% acetonitrile and 10% water at 2 ml/minute.

3 Pump this mixture through the nebulizer for 3 minutes.

NOTE This is a good general-purpose flushing mixture but you may need to adjust it based on the solvents, samples and buffers you are using. For example, a mixture of 50% acetonitrile and 50% water works well for removing salts.

To clean the electrospray spray chamber daily

When required Daily or at the end of each shift or anytime you suspect carryover contamination from one sample or analysis to another.

Tools required

- Cloths, clean, lint-free (p/n 05980-60051)
- Gloves, clean
- Isopropanol, reagent grade or better
- Mobile phase, current
- Wash bottle, clean
- Water, reagent-grade or better

Parts required None

NOTE Recent residue should be soluble in the mobile phase. If you are not sure what mobile phase was used recently, a mixture of 50% isopropanol and 50% water works well as a general cleaning solution.

1. Prepare the mobile phase you have been using.
2. Turn off the spray chamber.

WARNING **The electrospray spray chamber operates at high temperatures. Allow sufficient time to cool down before cleaning.**

3. Remove the electrospray nebulizer and the reference nebulizer.
4. Open the spray chamber (Figure 9).

Figure 9 Opened electrospray spray chamber

5 Rinse the interior of the spray chamber with the current mobile phase or with a mixture of isopropanol and water.

WARNING **Some mobile phases are dangerous. Use the degree of caution appropriate for the mobile phase being used.**

6 Wipe the interior of the spray chamber with a clean, lint-free cloth.

7 Rinse the area around the spray shield.

CAUTION Do not spray directly toward the tip of the capillary. This can cause pressure surges in the vacuum system.

8 Dampen a clean cloth with the mobile phase.

9 Wipe the spray shield and the area around the spray shield.

10 Close the spray chamber.

11 Reinstall the electrospray nebulizer.

WARNING Use the weekly cleaning procedure if symptoms of contamination persist or if the spray shield or capillary cap show significant discoloration that cannot be removed by the regular, daily cleaning.

To clean the electrospray spray chamber weekly

When required Weekly or whenever symptoms indicate that there is contamination in the spray chamber and normal daily cleaning does not correct the problem.

Tools required

- Abrasive paper, 4000 grit (p/n 8660-0827)
- Cloths, clean, lint-free (p/n 05980-60051)
- Cotton swabs (p/n 5080-5400)
- Gloves, clean
- Isopropanol, reagent grade or better
- Mobile phase, current
- Wash bottle, clean
- Water, reagent-grade or better

Parts required None

NOTE Recent residue should be soluble in the mobile phase. If you are not sure what mobile phase was used recently, a mixture of 50% isopropanol and 50% water works well as a general cleaning solution.

1. Turn off the spray chamber.

WARNING **The electrospray spray chamber operates at high temperatures. Allow sufficient time to cool down.**

2. Remove the electrospray nebulizer.
3. Open the spray chamber and remove it from the instrument.
4. Fill the spray chamber with clean mobile phase, or with a mixture of isopropanol and water.

Figure 10 Filling the spray chamber

WARNING **Some mobile phases are dangerous. Use the degree of caution appropriate for the mobile phase being used.**

5 Scrub the insulators and the interior of the spray chamber with a clean cotton swab.

6 Empty the spray chamber.

7 Reinstall the spray chamber on the instrument.

8 Remove the spray shield.

9 Use abrasive paper to gently clean the end of the capillary cap.

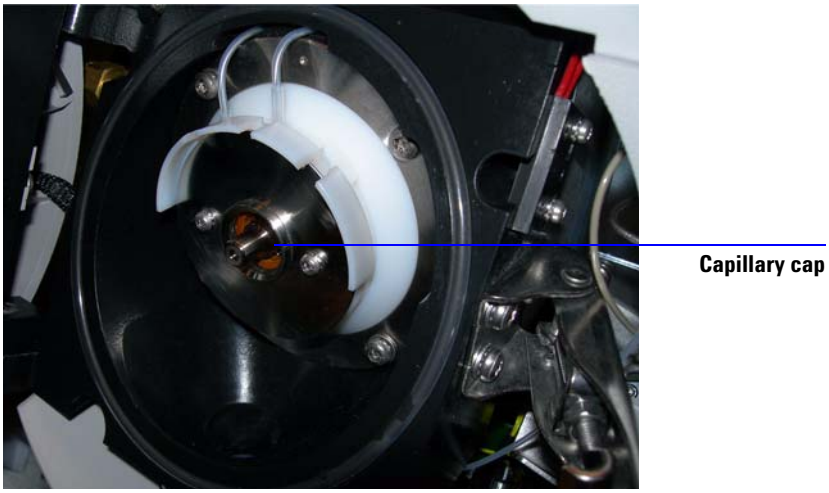

Figure 11 End of capillary cap

10 Dampen a clean cloth and wipe the end of the capillary cap.

11 Reinstall the spray shield.

12 Use abrasive paper to gently clean the spray shield.

13 Dampen a clean cloth and wipe the spray shield.

14 Rinse the area around the spray shield.

Figure 12 Rinsing the area around the spray shield

CAUTION Do not spray directly toward the tip of the capillary. This can cause pressure surges in the vacuum system.

15 Wipe the area around the spray shield.

16 Close the spray chamber.

17 Reinstall the electrospray nebulizer and the reference nebulizer.

To clean the spray shield and capillary cap with abrasives

Clean the spray shield and capillary cap with abrasives when symptoms of contamination persist after normal daily cleaning, or if the spray shield or capillary cap have significant discoloration or deposits that cannot be removed by regular cleaning

Before you begin, check that you have:

- Abrasive paper, 4000 grit (p/n 8660-0827)
- Cloths, clean, lint free (p/n 05980-60051)
- Gloves, clean, lint free (p/n 9300-1751)
- Isopropyl alcohol 99,5 %, reagent grade or better
- Water, HPLC grade or better

CAUTION Do not clean surfaces other than the spray shield and capillary cap with abrasives. Surfaces such as the spray chamber look similar but are softer metals or are plated by materials that are damaged when you clean them with abrasives.

WARNING **The electrospray spray chamber operates at high temperatures. Let the spray chamber cool before you begin.**

1. Mix a solution of 50 % isopropyl alcohol and 50 % water.
2. Shut down the instrument.

   See “To change from On to Standby” on page 21.
3. Open the spray chamber.
4. Remove the spray shield.
5. Remove the capillary cap.

CAUTION Do not twist off the capillary cap, or damage to the capillary may result.

6 Move the spray shield in a figure 8 pattern on the abrasive paper to clean the shield.

   Only the large flat surface needs to be abrasively cleaned unless there are obvious deposits elsewhere on the shield. The inner rim of the main hole in the shield may occasionally need cleaning also. You can use a cotton-tipped applicators and a mixture of isopropyl alcohol-water to clean this surface.

7 Clean the capillary cap by moving it in a figure 8 pattern on the abrasive paper.

   Only the end surface of the cap needs to be abrasively cleaned unless there are obvious deposits elsewhere on the cap. The inner rim of the hole in the cap my occasionally need cleaning also.

8 Dampen a clean cloth with the mixture of isopropyl alcohol and water.

9 Wipe the spray shield and capillary cap to remove any residual abrasive.

   Alternatively, the spray shield and capillary cap can be sonicated for 5 minutes in a 50:50 isopropyl alcohol:water mixture.

10 Reinstall the capillary cap.

11 Reinstall the spray shield.

12 Wipe the spray chamber.

13 Close the spray chamber.

To remove the electrospray nebulizer

When required When you need to access the nebulizer for maintenance.

Tools required Gloves, clean

Parts required None

NOTE Recent residue should be soluble in the mobile phase. If you are not sure what mobile phase was used recently, a mixture of 50% isopropanol and 50% water works well as a general cleaning solution.

Figure 13 Electrospray nebulizer

1. Shut off the flow of LC solvent.
2. Shut off the flow of nebulizing gas.
3. Slide back the plastic cover from over the nebulizer.
4. Disconnect the LC tubing and nebulizing gas tubing from the nebulizer.
5. Turn the nebulizer counterclockwise until it disengages from the retaining screws.
6. Gently lift the nebulizer out of the spray chamber.

WARNING **The tip of the nebulizer may be very hot. Allow it to cool before handling it.**

To replace the electrospray nebulizer needle in the analytical or the reference nebulizer sprayer

When required When the needle is plugged. Common symptoms of a plugged needle are increased LC back pressure or off-axis spraying or dripping from the nebulizer or when the reference nebulizer needle is not spraying.

Tools required

- Adjustment fixture (p/n G1946-20215)
- Gloves, clean
- Pliers, long nose (p/n 7810-0004)
- Wrench 3-mm, open-end (p/n 8710-2699)
- Wrench ¼-inch x 5/16-inch, open-end (p/n 8710-0510)

Parts required Nebulizer needle kit, Electrospray (includes needle, ferrule, and needle holder, p/n G2427A)

Figure 14 Electrospray nebulizer needle in needle holder

1. Install the nebulizer in the adjustment fixture.
2. Loosen the locknut next to the zero-dead-volume (ZDV) union.
3. Remove the union from the nebulizer.
4. Loosen the locknut of the needle holder.
5. Unscrew the needle holder and pull it out of the nebulizer.
6. Slide the non-tapered end of the needle through the new needle holder from the narrower side.

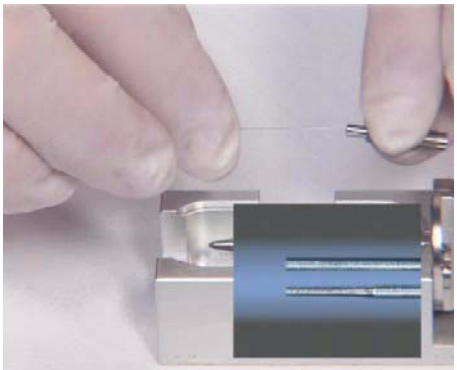

Figure 15 Sliding the non-tapered end of the needle through the new needle holder

7 Push a new ferrule, flat-side first, onto the needle.

8 Be sure the needle does not extend from the ferrule.

9 Reinstall the locknut and the union. Hand tighten the union.

10 Hold the needle holder steady with a 3-mm wrench. Tighten the union one-quarter to one-half turn to compress the ferrule.

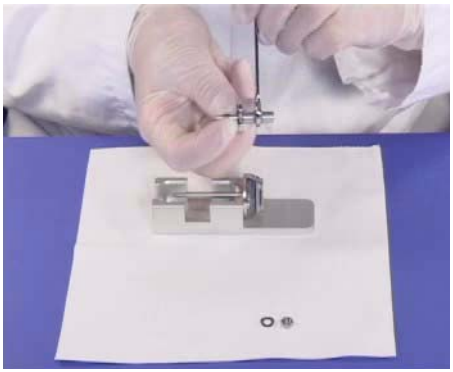

Figure 16 Tightening the union screw to compress the ferrule

11 Tighten the locknut against the union.

12 Pull carefully on the needle to ensure the needle is held firmly in place.

13 Replace locknut and washer.

14 Insert the needle into the nebulizer shaft.

CAUTION Take care when inserting the needle. The tapered end of the needle must pass through the restrictions in the nebulizer shaft. The tip of the needle can be damaged if excessive force is applied.

15 Adjust the electrospray needle position before reinstalling the nebulizer in the spray chamber.

NOTE Record this procedure in the Maintenance Logbook.

To adjust the electrospray nebulizer needle

When required After replacing the electrospray nebulizer needle or if symptoms indicate the needle is not correctly adjusted.

Tools required

- Adjustment fixture (p/n G1946-20215)
- Gloves, clean
- Magnifier (p/n G1946-80049)
- Wrench 3-mm, open-end (p/n 8710-2699)
- Wrench ¼-inch x 5/16-inch, open-end (p/n 8710-0510)

Parts required None

1 Install the nebulizer in the adjustment fixture.

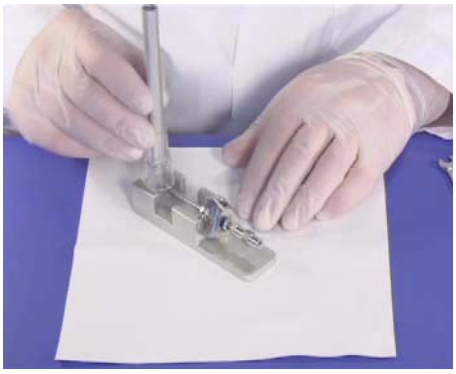

Figure 17

2 Loosen the needle holder locknut.

3 Position the magnifier so you can see the tip of the nebulizer.

4 Adjust the needle holder until the needle is even with the tip of the nebulizer.

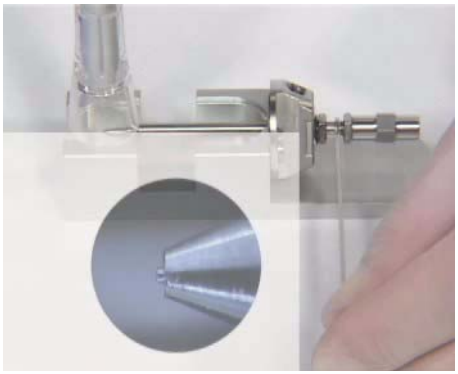

Figure 18 Adjusting the needle holder

5 Tighten the locknut. Make sure this does not change the position of the needle.

6 Remove the nebulizer from the adjustment fixture and reinstall it in the electrospray spray chamber.

7 Be very careful not to hit the tip of the nebulizer against anything. Any damage will have a large, negative effect on system performance.

NOTE Record this procedure in the Maintenance Logbook.

To reinstall the electrospray nebulizer

When required As necessary.

Tools required None

Parts required None

1 Insert the nebulizer part way into the spray chamber.

CAUTION Do not hit the tip of the needle as you insert the nebulizer. The tip of the needle is easily damaged.

2 Reconnect the nebulizing gas tubing to the nebulizer.
3 Finish inserting the nebulizer into the spray chamber.
4 Turn the nebulizer clockwise and lock it in place.
5 Reconnect the LC tubing the nebulizer.

CAUTION Do not overtighten the LC fitting. Overtightening the fitting can crush the tubing, creating a restriction.

6 Close the nebulizer cover.

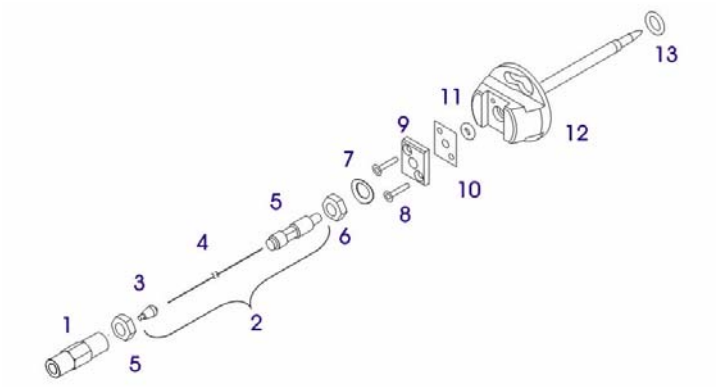

Figure 19 Exploded view of the nebulizer. See Table 2.

Table 2 ES Nebulizer Parts List

Item Number	**Part Description**	**Part Number**
	Electrospary Nebulizer	G1946-60098
1	Union, zero dead volume	0100-2234
2	ES needle replacement kit	G2427A
3	Ferrule, 0.010	G1946-20213
4	ES needle*	
5	Needle holder	G1946-20174
6	Hex nut	2740-0001
7	Curved spring washer	3050-1167
8	Screw, M2 x 0.4	0515-1602
9	Square Nut	G1946-20175
10	O-ring retainer	G1946-20202
11	O-ring, 2-005	0905-0082
12	ES nebulizer body†	
13	O-ring, 2-009	0905-1022

* Order complete ES needle replacement kit.

† Cannot be ordered separately. Order complete ES nebulizer assembly.

To remove the desolvation assembly

Remove the desolvation assembly when you need to gain access to the skimmer.

Before you begin, check that you have:

- Screwdriver, Torx 20 (p/n 8710-1615)
- Wrench, 1/2-inch x 9/16-inch, open-end (p/n 8710-0877)

WARNING **The spray chamber and desolvation assembly operate at very high temperatures. Let the spray chamber and desolvation assembly cool before you begin.**

NOTE The ion transport capillary is contained in the desolvation assembly and does not need to be removed.

1. Vent the instrument through the software.

   See "To shut down the instrument" on page 22.
2. Disconnect the instrument power cord from the electrical outlet.
3. Disconnect the LC tubing and nebulizing gas tubing from the nebulizer.
4. Open the spray chamber.
5. Remove the spray chamber by lifting it along the axis of its hinges.
6. Disconnect the drying gas tubing.
7. Disconnect the drying gas heater cable.
8. Disconnect the spray chamber high voltage cable.
9. Remove the CDS bottles.
10. Remove the CDS cover.
11. Disconnect the drain hose.
12. Remove the two retaining screws that keep the desolvation assembly attached to the support rods.
13. Slide the desolvation assembly off of the support rods.

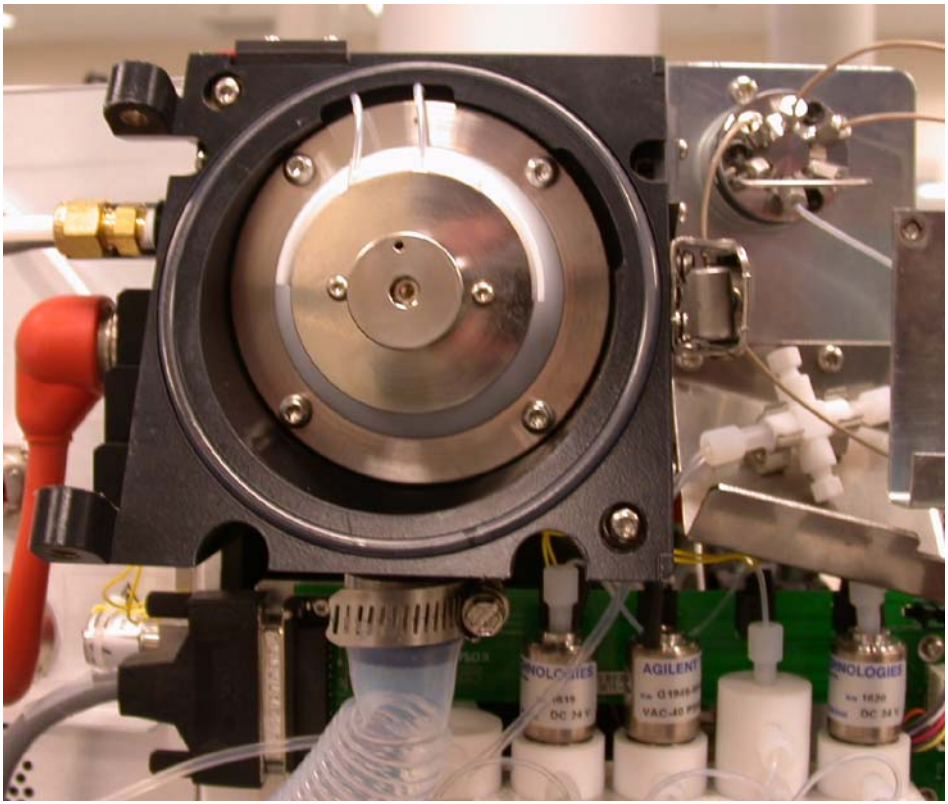

Figure 20 Desolvation assembly with cover removed

To clean the skimmer

Clean the skinner when you need to gain access to the skimmer.

Before you begin, check that you have:

- Cloths, clean, lint free (p/n 05980-60051)
- Gloves, clean, lint free (p/n 9300-1751)
- Isopropyl alcohol 99,5 %, reagent grade or better
- Water, HPLC grade or better

CAUTION The tip of the skimmer is delicate. Use care not to damage it.

CAUTION Do not clean the skimmer with strong acidic or basic solutions. Do not use excessive ultrasonic cleaning! Do not exceed 3 min in ultrasonic bath! This can damage the coating of the skimmer! Use a solution of isopropyl alcohol and water mixture with 0.5 % acetic acid to clean.

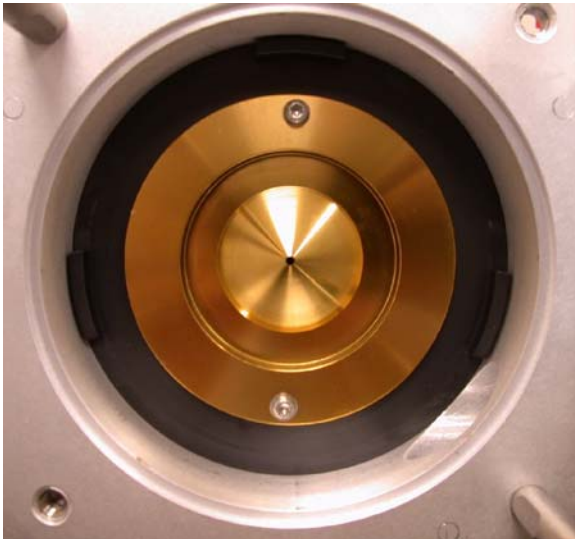

Figure 21 Skimmer

1 Vent the instrument through the software.

   See “To shut down the instrument” on page 22.

2 Disconnect the instrument power cord from the electrical outlet.
3 Open the spray chamber.
4 Remove the spray chamber by lifting it along the axis of its hinges.
5 Remove CDS bottles.
6 Remove the CDS cover.
7 Remove the desolvation assembly.
8 Dampen a clean cloth with the mixture of isopropyl alcohol and water.
9 Wipe the skimmer.
10 Reinstall the desolvation assembly.

To install the desolvation assembly

Before you begin, check that you have:

- Wrench, 1/2-inch x 9/16-inch, open-end (p/n 8710-0877)
- Screwdriver, Torx 20 (p/n 8710-1615)

1. Mount the desolvation assembly on the support rods and slide it back until it seals against the vacuum manifold.
2. Install the two retaining screws.
3. Reconnect the drain hose.
4. Replace the CDS cover.
5. Replace the CDS bottles.
6. Reconnect the spray chamber high voltage cable.
7. Reconnect the drying gas heater cable.
8. Reconnect the nebulizing gas tubing.
9. Reinstall the spray chamber and close it.
10. Reconnect the nebulizing gas tubing and LC tubing to the nebulizer.

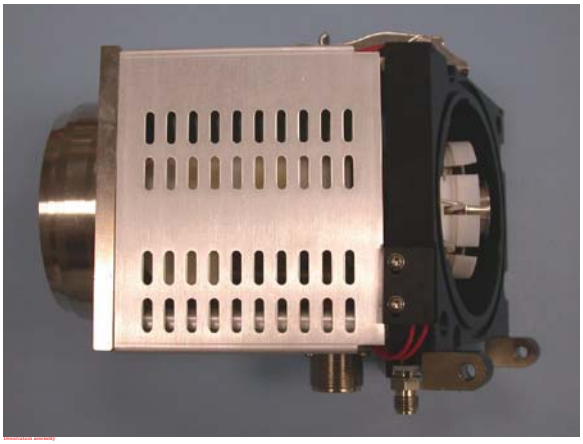

Figure 22 Desolvation assembly

To empty the drain bottle

Empty the drain bottle when the drain bottle nears full.

Before you begin, check that you have:

- Gloves, clean, lint free (p/n 9300-1751)

WARNING **Always treat the liquid in the drain bottle as hazardous. The liquid is undiluted solvent and samples.**

1. Check that there is no LC flow to the nebulizer.
2. Unscrew the top from the drain bottle.
3. Pour the contents into a hazardous waste container.
4. Reinstall the top on the drain bottle.

Figure 23 Drain bottle

To replace the gas conditioner

Replace the gas conditioner when the gas conditioner is saturated and chemical background appears and other sources of chemical background (e.g. solvents, contamination of the spray chamber) have been excluded. Also, when many ions are present even though no sample or solvent is flowing. This is most easily observed in APCI. In electrospray mode, solvent flow is generally required to generate ions and the ions formed depend on the composition of the solvent.

Replace the gas conditioner at least every 12 months.

Before you begin, check that you have:

- Wrench, 1/2 x 9/16 inch, open-end (p/n 8710-0877)
- Gas conditioner (filter) (BHT-4)

NOTE Place the gas conditioner cartridge in the correct flow direction and in a vertical position. Do not lay it in a horizontal position.

1. Shut off the nitrogen gas supply at its source.
2. Remove the old gas conditioner.
3. Connect the tubing from the nitrogen source to the inlet of the new gas conditioner.
4. Start the flow of nitrogen gas at its source.
5. Purge the gas for 5 minutes at the normal pressure setting.
6. Shut off the flow of nitrogen gas at its source.
7. Connect the tubing from the outlet of the gas conditioner to the instrument.
8. Start the flow of nitrogen at its source.

Ion Transfer Capillary

This section describes the steps to remove, clean and reinstall the ion transfer capillary.

To remove the capillary

When required When you need to clean or replace the capillary.

Tools required Gloves, clean

Parts required None

1. Vent the system.
2. Unplug the instrument power cord from the power outlet after venting is complete.
3. Open the spray chamber.

WARNING **The spray chamber operates at very high temperatures. Give the spray chamber time to cool before proceeding.**

4. Remove the spray shield.
5. Remove the capillary cap from the end of the capillary.
6. Carefully pull the capillary straight out of the desolvation assembly.

Figure 24 Pulling the capillary out of the desolvation assembly

CAUTION Pull the capillary straight out along its long axis. The capillary is glass, and you can break it by putting vertical or horizontal pressure on it.

To clean the capillary

When required When you observe decreased sensitivity and decreased signal stability.

Tools required

- Cloths, clean, lint-free (p/n 05980-60051)
- Cotton swabs (p/n 5080-5400)
- Gloves, clean
- Methanol, reagent grade or better
- Scissors
- Wash bottle, clean
- Water, reagent grade or better
- Wire (p/n 8180-2234)

Parts required None

1. Cut a 50 cm length of wire.
2. Dampen the interior of the capillary with the mixture of methanol and water.

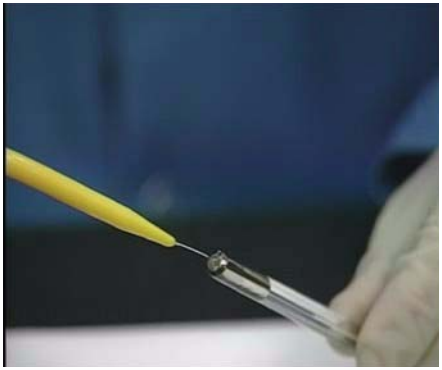

Figure 25 Dampening capillary interior with methanol and water

3. Hold the ends of the wire together and carefully push them through the capillary.
4. Pull the wire until only a small loop remains outside the capillary.

Figure 26 Pulling the wire through the capillary

5 Tear a very small piece of cotton from a cotton swab and place the cotton in the loop.

Figure 27 Placing cotton in the loop

CAUTION The piece of cotton must be very small. It must fit through the bore of the capillary. If the cotton is too large, it could cause the wire to break. If the wire breaks while the cotton is in the capillary, the cotton cannot be removed and the capillary is ruined.

6 Dampen the cotton with the mixture of methanol and water.

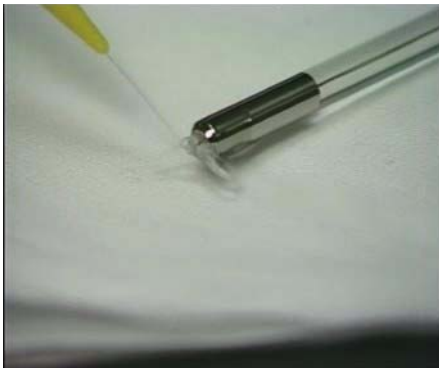

Figure 28 Dampening with methanol and water

7 Slowly and carefully pull the wire and cotton through the capillary.

8 Wipe the ends of the capillary with a clean lint-free cloth that is dampened with the methanol and water mixture.

9 Reinstall the capillary.

NOTE Record this procedure in the Maintenance Logbook.

To reinstall the capillary

When required After cleaning the capillary or when installing a new capillary.

Tools required Gloves, clean

Parts required None

1 Slide the capillary straight into the desolvation assembly. The capillary must be aligned correctly so that its end will fit into a fixed capillary cap inside the desolvation assembly.

Figure 29 Reinstalling the capillary

CAUTION Putting vertical or horizontal pressure on the capillary can break it.

2 Reinstall the capillary cap over the outer end of the capillary.

3 Reinstall the spray shield.

4 Close the spray chamber.

NOTE If a new capillary was installed, record this procedure in the Maintenance Logbook.

APCI Source

This section describes how to open and close the APCI source and maintain it. To learn how to install and remove the APCI source, see "To install an APCI source" on page 22 and "To remove an APCI or APPI source" on page 18.

To flush the APCI nebulizer

Flush the nebulizer daily, or at the end of each shift, to flush traces of samples and buffers out of the tubing, valves, and nebulizer. More often is acceptable.

Before you begin, check that you have:

- Methanol, HPLC grade or better
- Water, HPLC grade or better

This procedure applies to both electrospray and APCI nebulizer.

- If you have an LC:
  - **a** Check that methanol and water are installed in your LC.
  - **b** Set the liquid chromatograph to pump a mixture of 90 % methanol and 10 % water at 2 mL/min.
  - **c** Pump this mixture through the nebulizer for 3 minutes.
- If you do not have an LC:
  - **a** Connect a common syringe to the nebulizer, filled with a mixture of 90 % methanol and 10 % water.
  - **b** Pump this mixture through the nebulizer by gently pressing the syringe manually.

The methanol/water mixture is a good general-purpose flushing mixture but you may have to adjust it based on the solvents, samples, and buffers you use. For example, a mixture of 50 % organics and 50 % water works better for removing salts.

To clean the APCI spray chamber daily

When required Daily at the end of each shift or anytime you suspect carryover contamination from one sample or analysis to another.

Tools required

- Cloths, clean, lint-free (p/n 05980-60051)
- Gloves, clean
- Isopropanol, reagent grade or better
- Mobile phase, current
- Wash bottle, clean
- Water, reagent-grade or better

Parts required None

WARNING

The APCI spray chamber operates at high temperatures. Allow sufficient time to cool down.

Some mobile phases are dangerous. Use the degree of caution appropriate for the mobile phase being used.

CAUTION

Do not spray the mobile phase upward into the vaporizer.

Do not spray directly toward the tip of the capillary. This can cause pressure surges in the vacuum system.

NOTE

- Recent residue should be soluble in the mobile phase. If you are not sure what mobile phase was used recently, a mixture of 50% isopropanol and 50% water works well as a general cleaning solution.
- Use the weekly cleaning procedure if symptoms of contamination persist, or if the spray shield or capillary cap shows significant discoloration that cannot be removed by the regular daily cleaning.

1. Turn off the spray chamber.
2. Remove the corona needle.

3 Make sure the needle has cooled and then carefully clean it with abrasive paper.

4 Open the spray chamber.

Figure 30 Opened spray chamber

5 Rinse the interior of the spray chamber with the current mobile phase or with a mixture of isopropanol and water.

6 Wipe the interior of the spray chamber and the end of the vaporizer with a clean cloth.

7 Remove the spray shield.

8 Use abrasive paper to gently clean the end of the capillary cap.

9 Dampen a clean cloth and wipe the end of the capillary cap.

10 Reinstall the spray shield.

11 Use abrasive paper to gently clean the spray shield.

12 Dampen a clean cloth and wipe the spray shield.

13 Rinse the area around the spray shield.

14 Wipe the area around the spray shield with a clean cloth.

15 Close the spray chamber.

16 Reinstall the corona needle.

To clean the APCI spray chamber weekly

When required Weekly or whenever symptoms indicate contamination in the spray chamber and the normal daily cleaning does not correct the problem.

Tools required

- Abrasive paper, 4000 grit, 8660-0827
- Cloth, clean, lint-free, 05980-60051
- Cotton swabs, 5080-5400
- Gloves, clean
- Isopropanol, reagent grade or better
- Mobile phase, current
- Wash bottle
- Water, reagent-grade or better

Parts required None

1. Prepare the mobile phase you have been using.

   Recent residue should be soluble in the mobile phase. If you are not sure what mobile phase was used recently, a mixture of 50% isopropanol and 50% water works well as a general cleaning solution.
2. Turn off the spray chamber.
3. The APCI spray chamber operates at high temperatures. Allow sufficient time to cool down.
4. Remove the corona needle.
5. Make sure the needle has cooled and then carefully clean it with abrasive paper.
6. Open the spray chamber.
7. Rinse the interior of the spray chamber with the current mobile phase or with a mixture of isopropanol and water.

   Some mobile phases are dangerous. Use caution that is appropriate for the current mobile phase.

CAUTION Do not spray the mobile phase upward into the vaporizer.

8 Wipe the interior of the spray chamber and the end of the vaporizer with a clean cloth.
9 Remove the spray shield.
10 Use abrasive paper to gently clean the end of the capillary cap.
11 Dampen a clean cloth and wipe the end of the capillary cap.
12 Reinstall the spray shield.
13 Use abrasive paper to gently clean the spray shield.
14 Dampen a clean cloth and wipe the spray shield.
15 Rinse the area around the spray shield.

CAUTION Do not spray directly at the end of the capillary. This can cause pressure surges in the vacuum system.

16 Wipe the area around the spray shield with a clean cloth.
17 Close the spray chamber.
18 Reinstall the corona needle.

To remove the APCI nebulizer

When required When you need to access the nebulizer for maintenance.

Tools required
- Gloves, clean

Parts required None

Figure 31 APCI nebulizer

1. Shut off the flow of LC solvent.
2. Shut off the flow of nebulizing gas.
3. Disconnect the LC tubing and nebulizing gas tubing from the nebulizer.
4. Turn the nebulizer counterclockwise until it disengages from the retaining screws.
5. Gently lift the nebulizer out of the spray chamber.

WARNING **The tip of the nebulizer may be very hot. Allow it to cool before handling it.**

To replace the APCI nebulizer needle

When required When the needle is plugged. Common symptoms of a plugged needle are increased LC back pressure or off-axis spray from the nebulizer (difficult to see in an APCI system).

Tools required

- Adjustment fixture (p/n G1946-20215)
- Gloves, clean, lint-free (large, p/n 8650-0030; small, p/n 8650-0029)
- Pliers, long-nose (p/n 8710-0004)
- Wrench 3-mm, open-end (p/n G1946-20203)
- Wrench ¼-inch x 5/16-inch, 2 required (p/n 8710-0510)

Parts required Nebulizer needle kit, APCI (includes needle, ferrule, and needle holder, p/n G2428A).

1 Install the nebulizer in the adjustment fixture.

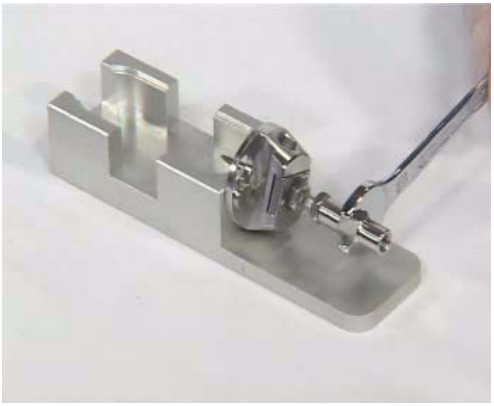

Figure 32 Installing the nebulizer in the adjustment fixture

2 Loosen the locknut next to the zero-dead-volume (ZDV) union.

3 Remove the union from the nebulizer.

4 Loosen the locknut of the needle holder.

5 Unscrew the needle holder and pull it out of the nebulizer.

6 Slide the non-tapered end of the needle through the new needle holder from the narrower side.

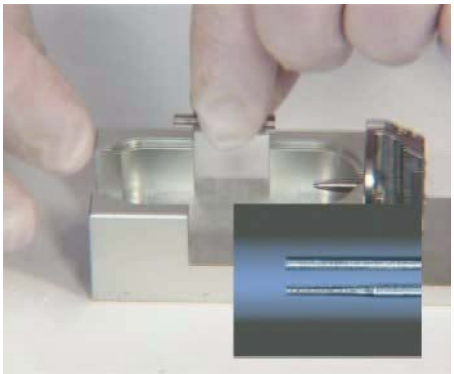

Figure 33 Sliding the non-tapered end of the needle through the new needle holder

7 Push a new ferrule, flat-side first, onto the needle.

8 Be sure the needle does not extend from the ferrule.

9 Reinstall the locknut and the union. Hand tighten the union.

10 Hold the needle holder steady with a 3-mm wrench and tighten the union one-quarter to one-half turn to compress the ferrule.

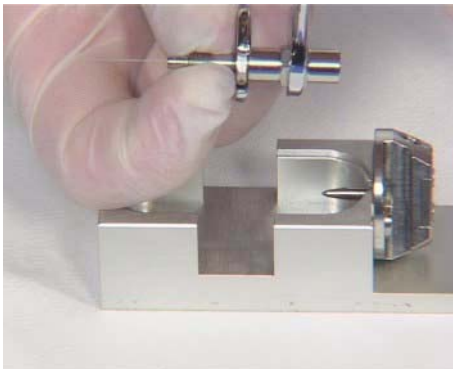

Figure 34 Tightening the union to compress the ferrule

11 Tighten the locknut against the union.

12 Pull carefully on the needle to ensure the needle is held firmly in place.

13 Replace locknut and washer.

14 Insert the needle into the nebulizer shaft.

CAUTION Take care when inserting the needle. The tapered end of the needle must pass through restrictions in the nebulizer shaft. The tip of the needle can be damaged if excessive force is applied.

15 Adjust the APCI needle position before reinstalling the nebulizer in the spray chamber.

NOTE Record this procedure in the Maintenance Logbook.

To adjust the APCI nebulizer needle

When required After replacing the APCI nebulizer needle or if symptoms indicate the needle may not be correctly adjusted.

Tools required

- Adjustment fixture (p/n G1946-20215)
- Gloves, clean
- Magnifier (p/n G1946-80049)
- Wrench 3-mm, open-end (p/n 8710-2699)
- Wrench ¼-inch x 5/16-inch, 2 required (p/n 8710-0510)

Parts required None

1 Install the nebulizer in the adjustment fixture.

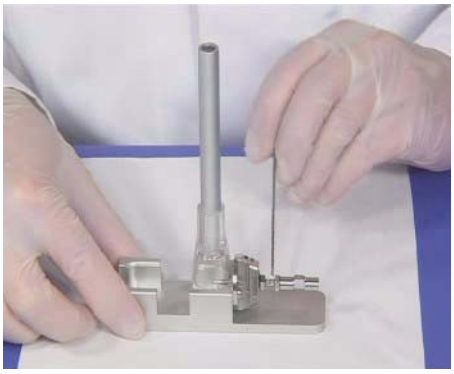

Figure 35 Installing the nebulizer in the adjustment fixture

2 Loosen the needle holder locknut.

3 Position the magnifier so you can view the tip of the nebulizer.

4 Adjust the needle holder until the needle is even with the tip of the nebulizer.

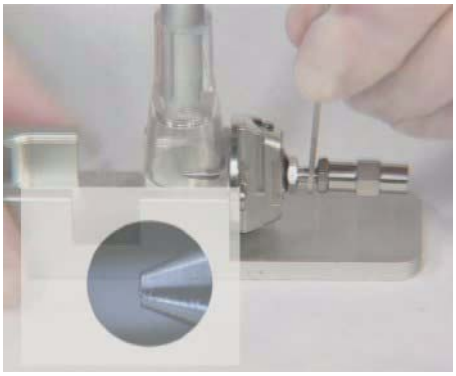

Figure 36 Adjusting the needle holder

5 Tighten the locknut. Make sure this does not change the position of the needle.

6 Remove the nebulizer from the adjustment fixture and reinstall it in the APCI spray chamber.

7 Be very careful not to hit the tip of the nebulizer against anything. Any damage will have a large, negative effect on system performance.

NOTE Record this procedure in the Maintenance Logbook.

To reinstall the APCI nebulizer

When required As necessary.

Tools required None

Parts required None

1. Insert the nebulizer into the spray chamber.
2. Turn it clockwise to lock it into place.
3. Reconnect the nebulizing gas tubing to the nebulizer.
4. Reconnect the LC tubing to the zero-dead-volume union.
5. Do not overtighten the LC fitting. Overtightening the fitting can crush the tubing, creating a restriction.

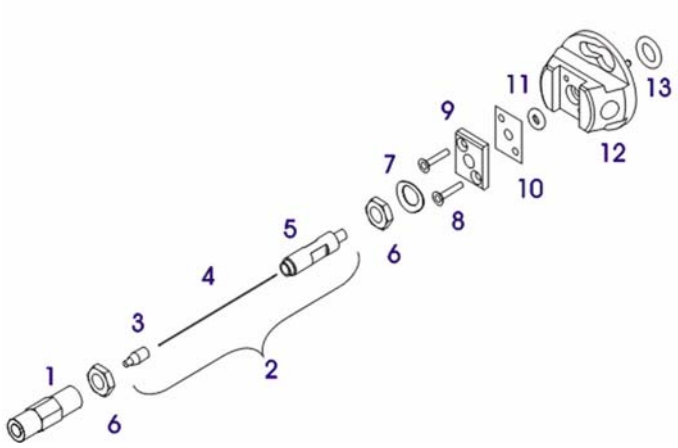

Figure 37 Exploded view of the APCI nebulizer. See Table 3.

Table 3 APCI Nebulizer Parts List

Item Number	Part Description	Part Number
	APCI Nebulizer	G1946-60037
1	Union, zero dead volume	0100-2234
2	APCI needle replacement kit	G2428A
3	Ferrule, 0.010	G1946-20213
4	APCI needle*	
5	Needle holder	G1946-20174
6	Hex nut	2740-0001
7	Curved spring washer	3050-1167
8	Screw, M2 x 0.4	0515-1602
9	Square Nut	G1946-20175
10	O-ring retainer	G1946-20202
11	O-ring, 2-005	0905-0082
12	APCI nebulizer body†	
13	O-ring, 2-009	0905-1022

* Order complete APCI needle replacement kit, G2428A.

† Cannot be ordered separately, order complete APCI nebulizer

To clean the corona needle

When required When you observe decrease sensitivity, decreased signal stability, and increase corona voltage during APCI operation.

Tools required

- Abrasive paper, 4000 grit (p/n 8660-0827)
- Cloths, clean, lint-free (p/n 05980-60051)
- Gloves, clean
- Isopropanol, reagent grade or better

Parts required None

1 Pull the corona needle assembly out of the spray chamber.

WARNING **The needle and related parts get very hot during operation. Make sure they have cooled before proceeding.**

Figure 38 Dirty APCI corona needle.

2 Fold a piece of abrasive paper over the base of the needle.

3 Pull and twist the abrasive paper along the needle and off the tip of the needle.

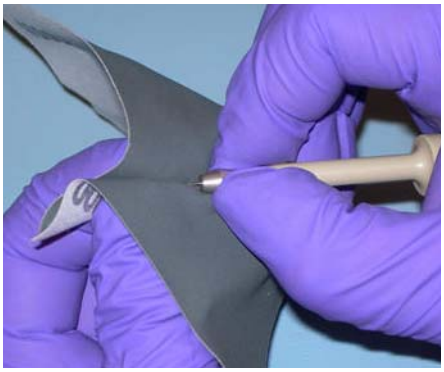

Figure 39 Pulling and twisting abrasive paper along the tip of the needle.

CAUTION Do not bend or blunt the tip of the needle, or it will decrease system performance. Sharpening the needle is not necessary.

4 Repeat steps 2 and 3 several times, or until the needle looks clean and shiny.

5 Starting at the base of the needle, wipe the needle with a clean cloth. The cloth can be dry or dampened with isopropanol.

Figure 40 APCI corona needle after cleaning.

6 Reinstall the corona needle assembly in the spray chamber.

To replace the corona needle

CAUTION Do not hit the tip of the needle against anything. It will bend the tip which will decrease system performance.

If this procedure fails to restore system performance, replace the corona needle.

When required When symptoms indicate poor corona needle performance and cleaning the needle does not restore performance.

Tools required
- Cloths, clean, lint-free (p/n 05980-60051)
- Gloves, clean
- Isopropanol, reagent grade or better

Parts required Corona needle (p/n G2429A)

1 Pull the corona needle assembly out of the spray chamber.

WARNING **The needle and related parts get very hot during operation. Make sure they have cooled before proceeding.**

2 Unscrew the needle collar from the corona needle shaft.

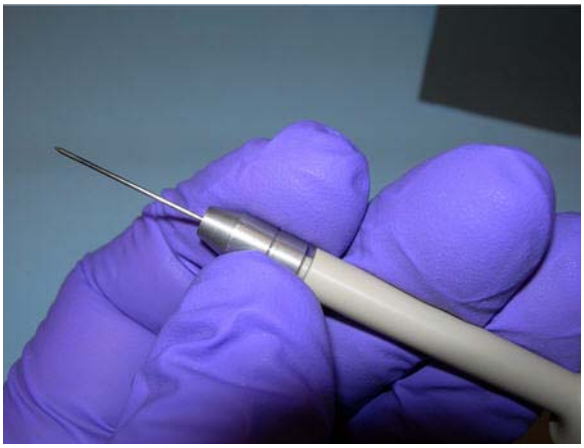

Figure 41 Unscrew the corona needle from the shaft.

3 Remove the old corona needle from the collar.

4 Install a new needle, with its integral ferrule, in the collar.

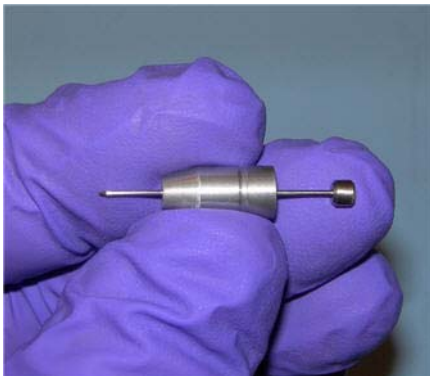

Figure 42 Replace the APCI corona needle.

5 Turn the collar onto the needle holder and tighten by hand.

6 Reinstall the corona needle assembly in the spray chamber.

CAUTION Do not hit the tip of the needle against anything. It will bend the tip which will decrease system performance.

NOTE Record this procedure in the Maintenance Logbook.

Multimode Source

This section describes how to clean and adjust the multimode source.

To clean the multimode source daily

When required

Daily or anytime you suspect carryover contamination from one sample or analysis to another, or when you must access the end cap and capillary cap for cleaning and inspection.

Tools required

- Gloves
- Wash bottle, clean

Parts required

- Abrasive paper, 4000 grit (p/n 8660-0827)
- Cloths, clean, lint-free (p/n 05980-60051)
- Cotton swabs (p/n 5080-5400)
- Mobile phase from the current method *or* clean isopropanol, reagent grade or better
- Water, reagent-grade or better

1 Turn off the spray chamber, nebulizer pressure, drying gas flow, drying gas temp, and vaporizer temp.

WARNING **Do not touch the multimode source or the capillary cap. They may be very hot. Allow the multimode source to cool down before you handle them.**

2 Remove the nebulizer and the APCI corona needle.

3 Remove the cosmetic cover. You will have to remove the thermocouple probe before you can wipe the spray chamber. Otherwise, leave the thermocouple intact.

4 Open the spray chamber.

5 Rinse the interior of the spray chamber using the wash bottle filled with the current mobile phase or with a mixture of isopropanol and water.

NOTE Recent residue should be soluble in the mobile phase. If you are not sure what mobile phase was used recently, a solution of 50% isopropanol and 50% water works well as a general cleaning solution.

WARNING **Some mobile phase are hazardous chemicals. Use the degree of caution appropriate for the mobile phase being used.**

6 Wipe the interior of the spray chamber with a clean, lint-free cloth.

WARNING **There are sharp edges inside the spray chamber such as the separator. Pay close attention when wiping the interior of the spray chamber.**

7 Rinse the area around the spray shield. Do not spray directly toward the tip of the capillary. This can cause pressure surges in the vacuum system.

8 Dampen a clean cloth with the mobile phase. Wipe the spray shield, field shaping electrodes and the area around the spray shield.

9 Replace the nebulizer and the APCI corona needle.

10 Install the thermocouple probe and adjust it so that it protrudes 15 mm from the inner spray chamber wall.

11 Replace the cosmetic cover.

12 Close the spray chamber.

NOTE Use the weekly cleaning process if symptoms of contamination persist, or if the spray shield or capillary cap show significant discoloration that can not be removed by the normal daily cleaning procedure.

To clean the multimode source weekly

The cleaning procedure for cleaning the multimode source weekly is similar to the daily procedure. The main difference is that the multimode source is removed from the instrument in the weekly procedure.

When required Weekly if the normal daily cleaning procedure is not sufficient

Tools required

- Gloves
- Wash bottle, clean

Parts required

- Abrasive paper, 4000 grit (p/n 8660-0827)
- Cloths, clean, lint-free (p/n 05980-60051)
- Cotton swabs (p/n 5080-5400)
- Mobile phase from the current method *or* clean isopropanol, reagent grade or better
- Water, reagent-grade or better

1. Remove the multimode source.
2. Fill the spray chamber with clean mobile phase, or with a mixture of isopropanol and water.

NOTE Recent residue should be soluble in the mobile phase. If you are not sure what mobile phase was used recently, a solution of 50% isopropanol and 50% water works well as a general cleaning solution.

WARNING **Some mobile phase are hazardous chemicals. Use the degree of caution appropriate for the mobile phase being used.**

3. Scrub the corona insulator and the interior of the spray chamber with a clean cotton swab.
4. Empty the spray chamber
5. Wipe the interior of the spray chamber with a clean, lint-free cloth

WARNING **There are sharp edges inside the spray chamber such as the separator. Be careful when wiping the interior of the spray chamber.**

6. Remove the spray shield. Use abrasive paper to gently clean the end of the capillary cap.
7. Dampen a clean cloth and wipe the end of the capillary cap.
8. Reinstall the spray chamber.
9. Use abrasive paper to gently clean the spray shield. Dampen a clean cloth and wipe the spray shield.
10. Rinse the area around the spray shield then wipe the area around the spray shield.
11. Reinstall the spray chamber on the instrument.
12. Replace the nebulizer and APCI corona needle.
13. Install the thermocouple probe and adjust it so that it protrudes 15mm from the inner spray chamber wall.
14. Replace the cosmetic cover.
15. Close the spray chamber.

To adjust the multimode nebulizer

When required When the needle is plugged. Common symptoms of a plugged needle are increased LC back pressure or off-axis spraying or dripping from the nebulizer.

Tools required

- Adjustment fixture (p/n G1946-20215)
- Gloves, clean
- Pliers, long nose (p/n 7810-0004)
- Wrench 3-mm, open-end (p/n 8710-2699)
- Wrench ¼-inch x 5/16-inch, open-end (p/n 8710-0510)

Parts required Nebulizer needle kit, Electrospray (includes needle, ferrule, and needle holder, p/n G2427A)

Figure 43 Electrospray nebulizer needle in needle holder

1. Install the nebulizer in the adjustment fixture.
2. Loosen the locknut next to the zero-dead-volume (ZDV) union.
3. Remove the union from the nebulizer.
4. Loosen the locknut of the needle holder.
5. Unscrew the needle holder and pull it out of the nebulizer.
6. Slide the non-tapered end of the needle through the new needle holder from the narrower side.

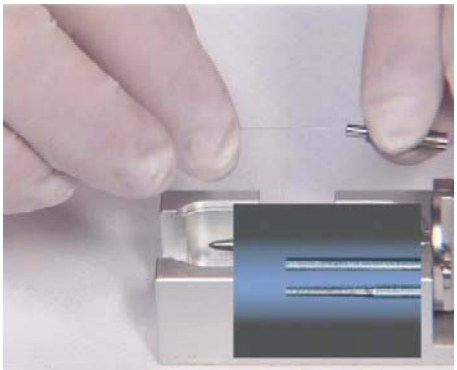

Figure 44 Sliding the non-tapered end of the needle through the new needle holder

7 Push a new ferrule, flat-side first, onto the needle.

8 Be sure the needle does not extend from the ferrule.

9 Reinstall the locknut and the union. Hand tighten the union.

10 Hold the needle holder steady with a 3-mm wrench. Tighten the union one-quarter to one-half turn to compress the ferrule.

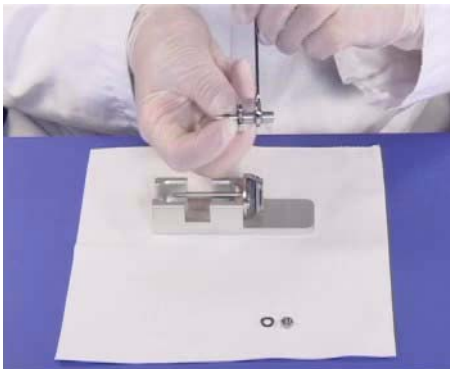

Figure 45 Tightening the union screw to compress the ferrule

11 Tighten the locknut against the union.

12 Pull carefully on the needle to ensure the needle is held firmly in place.

13 Replace locknut and washer.

14 Insert the needle into the nebulizer shaft.

CAUTION Take care when inserting the needle. The tapered end of the needle must pass through the restrictions in the nebulizer shaft. The tip of the needle can be damaged if excessive force is applied.

15 Adjust the electrospray needle position before reinstalling the nebulizer in the spray chamber.

NOTE Record this procedure in the Maintenance Logbook.

Divert Valve

When you do maintenance procedures on the divert valve:

- Check drain bottle in shorter intervals as usual!
- Do not bend any capillary line.
- The position of the divert valve can be set only in the On mode.
- Put the instrument in the Off mode.
- Remove the covers before you begin.
- Install the covers of the instrument after you finish.

Table 4 Divert Valve Connection (see Figure 46)

No.	Port	Connection	User action
1	Loop	loop to port 5	none (permanent connection)
2	Inlet	from CDS	none (permanent connection from calibrant delivery system)
3	Outlet	to the ion source	connect the flexible capillary to the ion source
4	Inlet	from the front inlet union behind the inlet cover	none
5	Loop	loop to port 1	none (permanent connected)
6	Waste	to the drain bottle	none

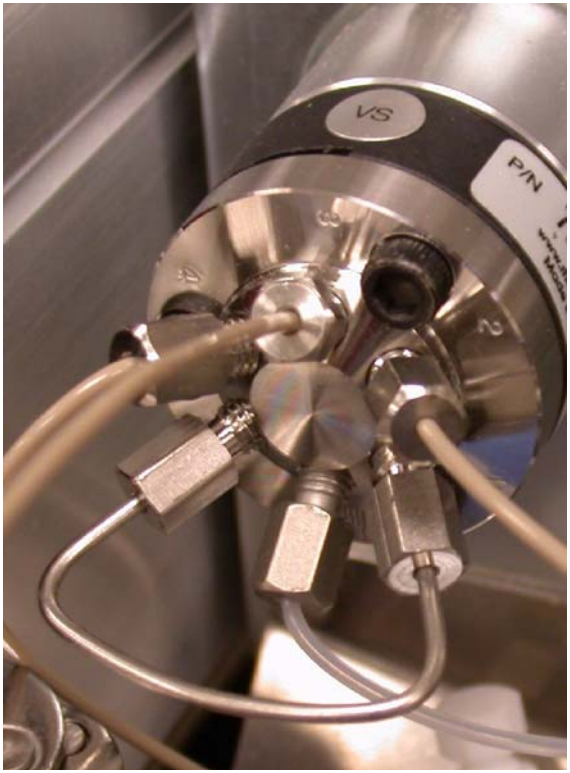

Figure 46 Divert valve with port position indicators

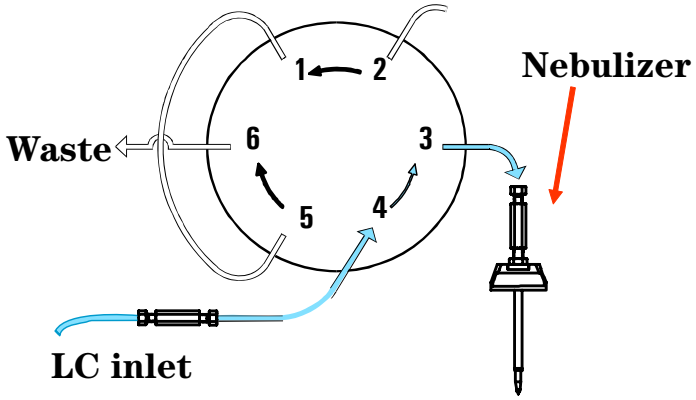

Figure 47 "LC -> MS" selected in the Tune tab of the Method pane

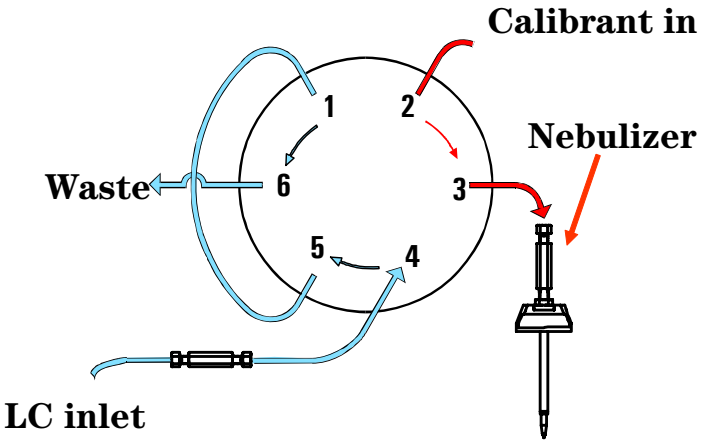

Figure 48 "LC -> Waste" or "Cal B" selected in the Tune tab of the Method pane

To replace the outlet capillary line

Replace the outlet capillary line when the outlet capillary line has been bent or is broken.

Before you begin, check that you have:

- Wrench, 1/4 inch
- Gloves, clean, lint free (p/n 9300-1751)

1. Check that there is no flow through the divert valve.
2. Disconnect the outlet capillary line from the ion source.
3. Unscrew the outlet capillary line from port 3.
4. Cut a new capillary line to the right length (35 cm, 13¾ inch).
5. Connect the capillary line to port 3.
6. Tighten the screw of port 3.
7. Connect the outlet capillary line to the ion source.

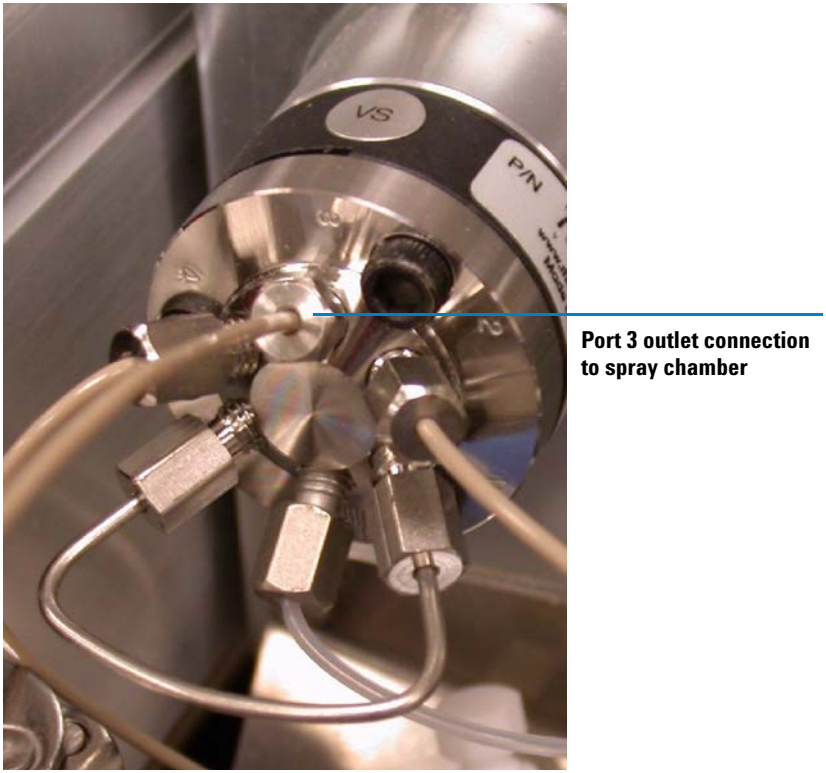

Figure 49 Divert valve outlet connection

To replace the inlet capillary line

Replace the inlet capillary line when the inlet capillary line has been bent or is broken.

Before you begin, check that you have:

- Wrench, 1/4 inch (p/n 8710-0510)
- Inlet capillary line
- Gloves, clean, lint free (p/n 9300-1751)

1. Check that there is no flow through the divert valve.
2. Disconnect the inlet capillary line from the inlet union *behind* the CDS cover.
3. Unscrew the inlet capillary line from port 4.
4. Cut a new capillary line to the right length (15 cm, 6 inch).
5. Connect the capillary line to port 4.
6. Tighten the nut of port 4.
7. Connect the capillary line to the inlet union behind the CDS cover and tighten it.

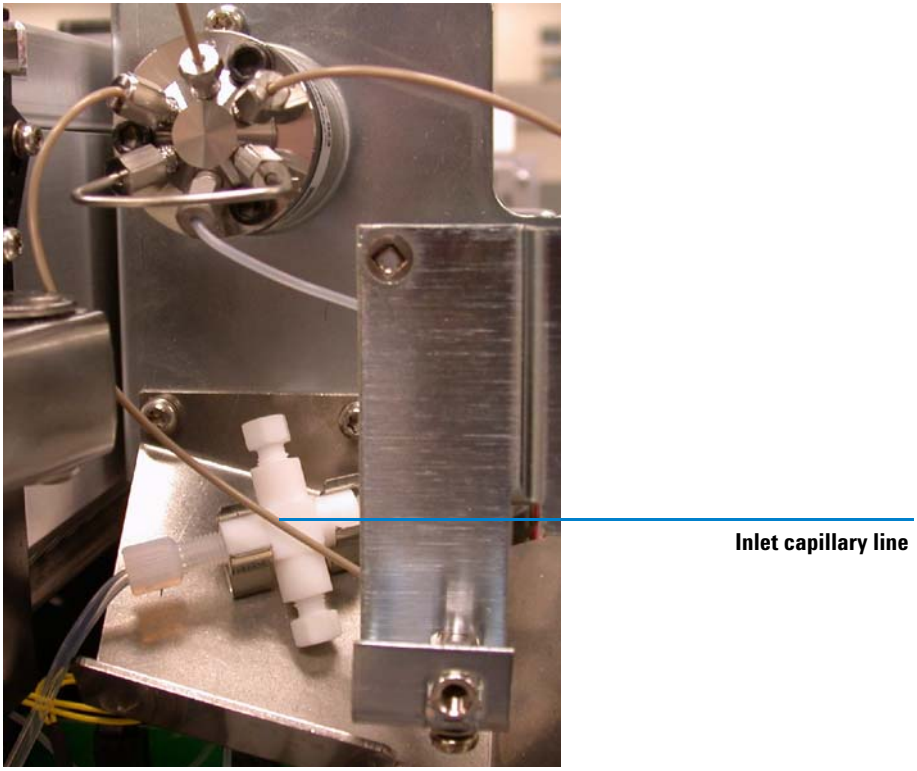

Figure 50 Divert valve inlet connection

To replace the waste capillary line

Replace the waste capillary line when the waste capillary line has been bent or is broken.

Before you begin, check that you have:

- Wrench, 1/4 inch (p/n 8710-0510)
- Gloves, clean, lint free (p/n 9300-1751)
- Waste line (p/n G1969-60086)

1. Make sure there is no flow through the divert valve.
2. Disconnect the waste capillary line from the waste tube.
3. Unscrew the waste capillary line from port 6.
4. Install new waste capillary line to port 6.
5. Connect the new capillary line to port 6.
6. Tighten the screw of port 6.
7. Connect the capillary line to the waste tube connection and tighten it.

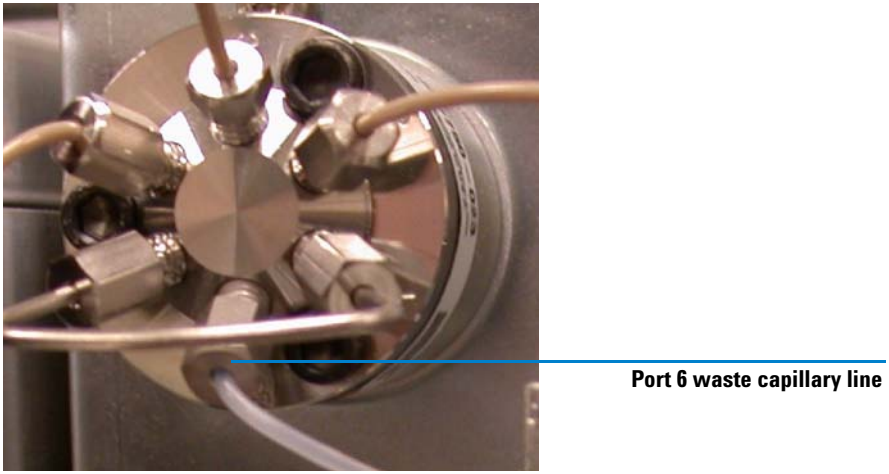

Figure 51 Divert valve waste connection

Manifold

This section describes how to open and close the manifold, and to clean the ion optics assembly.

To remove the manifold cover

1. Vent the instrument.

   See "To shut down the instrument" on page 22.
2. Disconnect the instrument power cord from the electrical outlet.
3. Remove the covers of the instrument.
4. Remove the retaining screws from the vacuum manifold cover.

   If the screws are difficult to remove, they were possibly incorrectly installed after the system was under vacuum. To fix the problem, turn on the instrument for a few seconds and remove the screws. Then turn off the instrument and disconnect the power cord.
5. Lift off the vacuum manifold cover.
6. Check the manifold O-ring for damage or dust.

   The O-ring must lie correctly in its slot, or the system will not pump back down.

Figure 52 Removing the vacuum plate from the manifold

To clean the ion optics assembly

Remove the ion optics assembly to clean octopole 1 and the skimmer. Do so when the signal to noise ratio is outside of acceptable parameters, and no more useful measurements are possible.

Before you begin, check that you have:

- Cloths, clean, lint free (p/n 05980-60051)
- Gloves, clean, lint free (p/n 9300-1751)
- Isopropyl alcohol 99,5 %, reagent grade or better
- Water, HPLC grade or better

CAUTION The octopole and the tip of the skimmer are delicate. Use care not to damage these parts.

CAUTION Wear gloves and a hair net to protect the vacuum system from hair and dust.

1. Mix a solution of 50:50 isopropyl alcohol:water.
2. Vent and turn off the instrument.

   See "To shut down the instrument" on page 22.
3. Removing the desolvation assembly.

   See "To remove the desolvation assembly" on page 61.
4. Open the manifold.
5. Use a pair of pliers to disconnect the cables from the octopole feed-through:
   a. Disconnect the left green cable.
   b. Disconnect the left black cable.
   c. Disconnect the black plug.

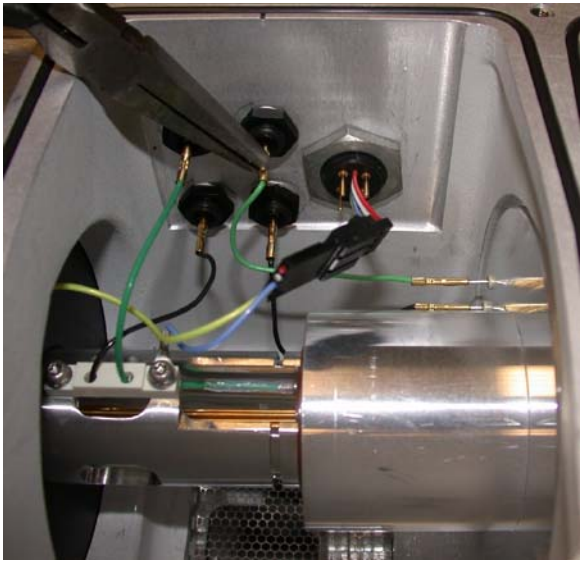

Figure 53 Disconnecting the cables of the octopole

6 Carefully remove the ion optics assembly out of the manifold.

Use care so that you do not damage the octopole.

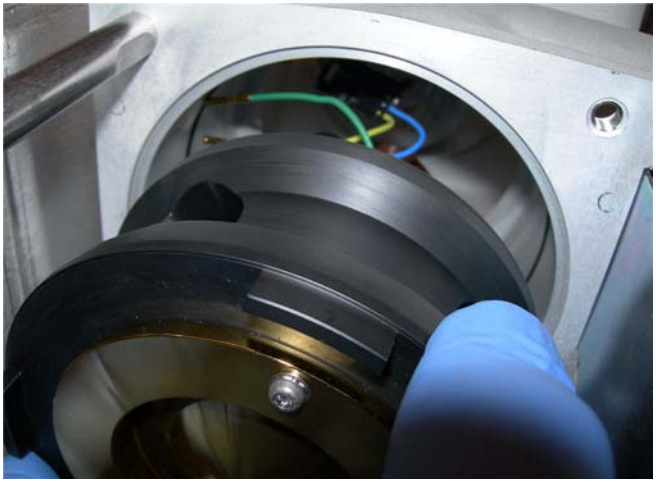

Figure 54 Removing the ion optics assembly.

7 Place the ion optics assembly on a clean flat surface.

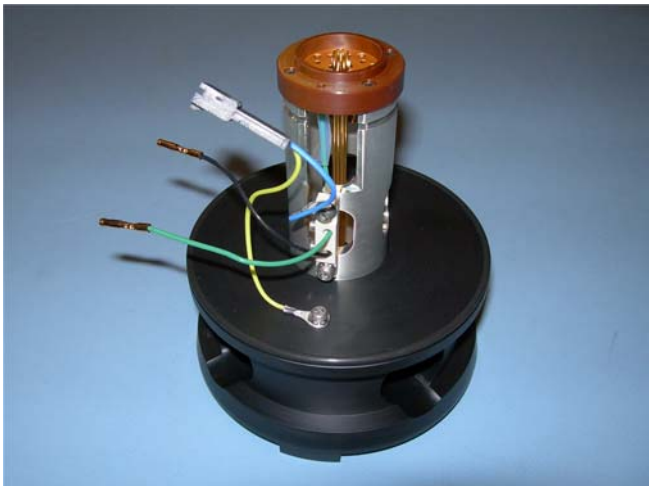

Figure 55 Ion optics assembly

8 Reinstall the vacuum manifold cover to protect the vacuum system from dust or particles.

Do not get dust or hair into the vacuum system.

9 Remove the skimmer from the ion optics assembly.

Figure 56 Removing the optics skimmer

10 Wipe the skimmer with a wet, lint-free cloth. Alternatively, after wiping the skimmer, remove the black O-ring on the skinner, then sonicate for approximately five minutes.

11 Holding the ion optics assembly, pour 50 mL of the 50:50 isopropyl alcohol:water solution over the octopole rods.

12 Allow the cleaned parts to dry.

Thoroughly dry all of the cleaned parts to shorten the pump down-time.

13 Remove the manifold cover.

14 Insert the octopole carefully into the manifold.

Do not damage the ion optics assembly. The cable strain relief should be at the top of the assembly. Align the pin of the octopole 2 assembly with the hole in the ion optics assembly.

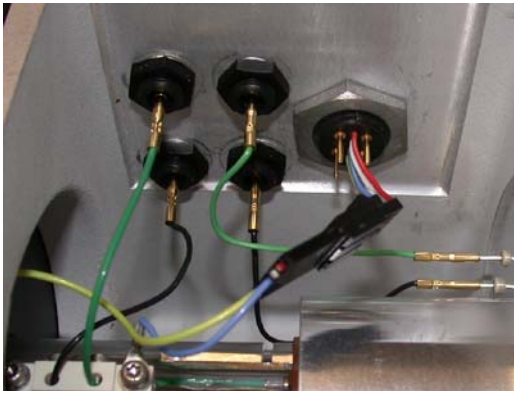

Figure 57 Octopole wiring

15 Connect the octopole cables to the manifold:

- **a** Connect the black plug.
- **b** Connect the black cable to the lower pin.

  Do not cross the black cables!
- **c** Connect the green cable to the upper pin.

  Do not cross the green cables!

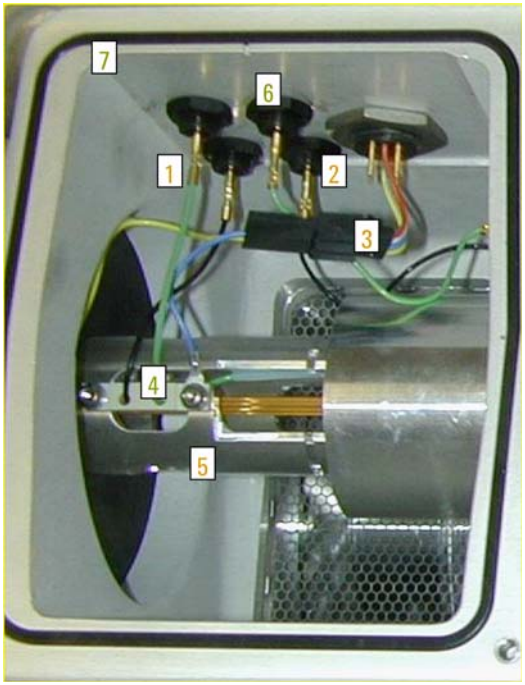

Figure 58 Ion optics cabling (see Table 5)

Table 5 TOF Octopole 1 Cabling Parts List

Item Number	Part Description	Part Number
1	Cable, Rear Octopole Green – Quantity 2	G1969-60820
2	Cable, Rear Octopole Black – Quantity 2	G1969-60821
3	Lens Cable Assembly	G1946-60070
4	Cable, Octopole Assembly	G1946-60080
5	Ion Optics Assembly	G1956-60341
6	H.V. Feedthrough (includes O-ring and nut) – Quantity 4 used	G1946-80013
	O-Ring,2-013, Fluorocarbon, Black	0905-0490
	Feedthrough Nut	2950-0144

Table 5 TOF Octopole 1 Cabling Parts List (continued)

Item Number	Part Description	Part Number
7	O-ring, Front Manifold	0905-1466
n/a	O-ring, 2-155, Fluorocarbon, Black (mounted inside the manifold)	0905-1467

16 Inspect manifold O-ring to ensure no damage to the O-ring.

17 Close the manifold. See "To close the manifold" on page 116.

18 Reinstall the desolvation assembly.

The pump-down procedure can now done. See "To start the system in Standby mode" on page 15.

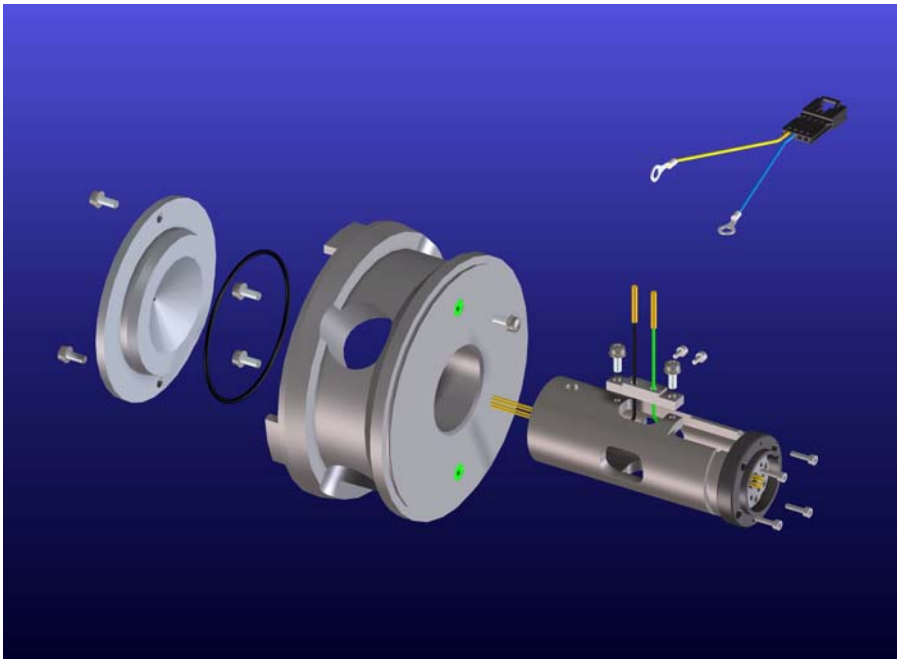

Figure 59 Exploded view of the ion optics assembly

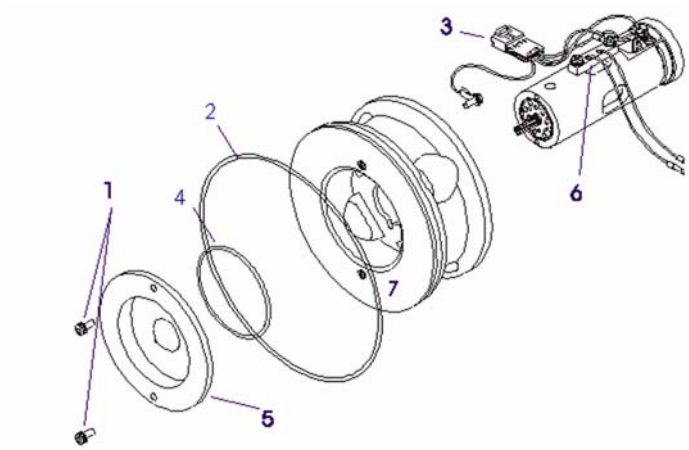

Figure 60 Ion optics parts exploded (see Table 6)

Table 6 Ion Optics and Octopole 1 Assembly Parts List

Item Number	Part Description	Part Number
	Ion Optics and Octopole 1 Assembly for TOF LC/MS	G1956-60341
1	Screw, M3 x 0.5 x 8 T10	0515-0372
2	O-ring, 2-153 (for Skimmer Spacer)	0905-1468
3	Octopole cable assembly	G1946-60080
4	O-ring, 2-033 (for Skimmer)	0905-1471
5	Skimmer, 2 mm Diameter	G1969-20302
6	Strain relief block	G1946-20172
7	H.T. Skimmer spacer	G1956-20303
	NOTE: Remove Lens 1 & 2 and replace 1.6mm I.D. Skimmer with 2.0mm I.D. Skimmer, G1969-20302	

To close the manifold

CAUTION Tighten the manifold screws only until they are tight. If you continue to tighten the screws after they are tight, you can get vacuum leaks.

NOTE You must install the retaining screws before the pump down the instrument. After you pump down the instrument, it can be difficult to install the retaining screws because the manifold cover may not be the correct position.

NOTE After you pump down the instrument, the retaining screws may be loose. Do not tighten them. Tightening them will make them difficult or impossible to remove later.

1. If you worked on any components in the manifold, make sure the electrical connections are correct.
2. Inspect manifold's O-ring for damage or dust. Lightly grease O-ring, if needed.
3. Position the manifold cover on the vacuum manifold.
4. Hand tighten the two retaining screws.
5. Reinstall the covers of the instrument.
6. The pump-down procedure can now be done.

   See "To start the system in Standby mode" on page 15.

3
Troubleshooting

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APCI 136

This chapter will help you troubleshoot problems that you might encounter while you operate your Time-of-Flight LC/MS.

NOTE For information on maintaining an APPI, a nanospray, a dual nanospray or a Multimode Ionization source, please refer to the *User's Guide* for that product.

Performance

This section describes conditions that you can see when you tune or acquire data. These conditions result in mass spectra that are not normal.

If you get no peaks

Many conditions in the API-electrospray LC/MS system can prevent peaks.

✔ Check that the solvent flows from the needle

If you are not sure, turn off the nebulization gas and then turn it back on. If there is no spray from the needle, or if only droplets appear, see "Flow" on page 125.

✔ Check that the divert valve setting is correct in the method.

Make sure that the divert valve setting in the MS TOF Data tab is set to **To Source**. If **To Waste** is selected, the sample and solvent flow streams are diverted to the drain bottle away from the source.

✔ Check that the electrospray high voltages are turned on.

Put the instrument in Standby mode then back to On.

✔ If you get the message "Spray Chamber Open", check that the spray chamber is closed.

✔ Check that the set values for the spray chamber are set correctly:

Table 7 Electrospray (approximate values)

	Positive ionization	Negative ionization
Capillary voltage	3,500 V	3,500 V
End plate offset	(set automatically to 500 V)	

Table 8 APCI (approximate values)

	Positive ionization	Negative ionization
Capillary voltage	3,500 V	3,500 V
End plate offset	(set automatically to 500 V)	
Corona current	4 µA	-20 µA

✔ Check that the currents in the high-voltage electrodes are correct.

Check that you have flow from the needle before you check currents. If you do not have a flow, you will not have a current.

Table 9 Electrospray

Electrode	Current
Capillary	8 nA to 200 nA
End plate	0.2 µA to 1.0 µA

Table 10 APCI

Electrode	Current
Capillary	50 nA to 500 nA
End plate	Approximately the same values as the Corona current value
Corona	Positive: 0 to 10 µA; typical 4 µA Negative: 0 to 40 µA; typical 10 µA

✔ Check for solvent effects.

The currents depend on the electrode voltage and the solvent. Aqueous solvents are more conductive and result in higher currents than non-aqueous solvents. Currents that are too high or unstable often indicate arcing or corona discharge that will greatly reduce or eliminate signal. Reduce the flow or the voltages. In negative ionization mode, oxygen in the nebulizing gas can help prevent unstable currents.

✔ Check to see if the cap is in place on the capillary entrance and the spray shield is correctly installed.

✔ Check for damage to the metal plating on the entrance end of the capillary:

1. Shut down the Time-of-Flight LC/MS.
2. Open the spray chamber.
3. Remove the spray shield and the cap on the capillary entrance.
4. Check the electrical continuity between the side of the capillary contact and the metal-plated entrance end of the capillary.
5. The measured resistance should be a few ohms or less. If the resistance is significantly higher, replace the capillary.
6. Check continuity between all three surfaces on the end of the capillary.
7. Check the *end*, the *side*, and the *beveled* surfaces. It is not unusual to have continuity to one or two of these surfaces but not the others because the metal plating at the corners is worn away or vaporized. This type of damage is not always visible.
8. Re-install the cap on the capillary entrance and the spray shield.

✔ Check the drying gas flow

Check the drying gas flow and temperature (in the Tune page). The drying gas flow should be between 3.5 and 12 L/min. depending on solvent flow. Make sure that the setting and the read-back values are correct. If the setting is correct, but the read-back value is too low, check the nitrogen delivery system.

The drying gas temperature set point should be between 150 °C and 300 °C depending on the solvent flow and composition. Make sure the temperature set point is correct and the actual temperature is at the set point.

See “If the nebulizer produces droplets” on page 127.

✔ Check that the fore and high vacuum pressures are within normal ranges.

Too much pressure can greatly decrease signal. If any of the pressures are outside the normal ranges, see “Pressure” on page 128.

If you get a low signal

If you can not achieve the specified intensity for the tuning compounds, or if your sample shows much lower-than-expected intensities, check these steps.

WARNING **Check that the parts are cooled before you handle them.**

- ✔ Run Autotune to update your tune parameters.
- ✔ Check the solution chemistry.

  Make sure the solvent is appropriate for your sample and for the type of data (positive or negative) you are acquiring.
- ✔ Check the sample

  Old samples, or samples that are not correctly stored, result in lower-than-normal signal.
- ✔ Check the spray shape

  Make sure the spray is a narrow, symmetrical plume. If the spray is off to one side, the needle tip is possibly damaged. Replace the nebulizer.
- ✔ Check the nebulizer pressure, drying gas flow and temperature

  For autotuning, make sure drying gas flow and temperature are at the correct set points. Drying gas flow should be 7 L/min. Temperature should be 300°C. Nebulizer gas pressure should be 15 psi.

  If you are not tuning, make sure you use the appropriate spray chamber parameters for your sample.
- ✔ Clean the capillary entrance and spray shield

  See "To clean the spray shield and capillary cap with abrasives" on page 51.
- ✔ Check the capillary for damage or contamination

  Put the system in Standby mode. Check for electrical continuity between all three surfaces on the entrance end of the capillary. Check the end, the side, and the beveled surfaces. It is not unusual to have continuity to one or two

of these surfaces but not the end. This is because the metal plating at the corners can be worn away or vaporized. This type of damage is not always visible.

✔ Clean the capillary

See “To clean the capillary” on page 70.

If you get an unstable signal

If the peak-to-peak reproducibility is poor, check these steps.

WARNING **Check that the parts are cooled before you handle them.**

✔ Check the spray shape

Make sure the spray is a fine, symmetrical plume. If the spray is off to one side, the needle tip is possibly damaged. Replace the nebulizer.

✔ Check the drying gas flow and temperature

Make sure drying gas flow and temperature are correct for the solvent flow and composition.

✔ Check the stability of the solvent flow

If you are using an LC, make sure the solvent is thoroughly degassed. Do not degas proteins and other samples that are damaged by ultrasonic degassing. Make sure the LC back pressure is steady. This indicates a steady solvent flow. Degassing also applies to infusion unless it would damage the sample.

✔ Make sure current in the high-voltage electrodes are correct and stable

Verify that there is a flow from the needle before you check current. If you have no current, you have no flow. See “If you do not get a flow” on page 125.

✔ Check the spray chamber voltage

Check the Tune page if the high voltage readings give values close to the set values. Check the capillary voltage. If you are running APCI, check the corona voltage also.

✔ Check the capillary for damage or contamination

See “To clean the capillary” on page 70.

✔ Clean the capillary.

See “To clean the capillary” on page 70.

If you get poor mass accuracy

- ✔ Check the ions used for tuning.

  Make sure the ions used for tuning span the mass range of the sample ions. Make sure they show strong, stable signals.

- ✔ Tune and recalibrate the mass axis.
- ✔ Check the ions being used for reference mass correction.
- ✔ Check the abundance for the ions being used for reference mass correction.
- ✔ Check for excessive vibration of the TOF.

  Excessive vibration can cause instability in the ion mirrors.

- ✔ Check the room temperature over a full day.

  If the room temperature varies too much, the electronic calibration can drift. This can affect the mass accuracy.

- ✔ Check the reference mass indicator in the MS-TOF icon. See the table below.

Green indicates that for the scan, all reference mass ions were found. Yellow indicates that one or more reference mass ion were not found, and red indicates that all reference mass ions were not found.

Green indicates that for the sample, all reference mass ions were found. Yellow indicates that one or more reference mass ion were not found, and red indicates that all reference mass ions were not found.

Green indicates that for the worklist, all reference mass ions were found. Yellow indicates that one or more reference mass ion were not found, and red indicates that all reference mass ions were not found.

Flow

Flow symptoms are symptoms related to the flow of solvent and sample through the needle assembly.

If you do not get a flow

Check that the nebulization gas is turned off before checking to detect flow.

If there is no flow from the needle into the interface after you turn on the syringe pump or LC, check the following:

✔ Check the LC pump settings

Make sure the LC is on. Make sure the pump is set to the correct flow and the specified bottle contains enough solvent.

✔ Check for LC error messages

A problem such as overpressure or a detected leak may cause the LC pump to turn off automatically.

✔ Check the tubing to make sure it is primed

The tubing between the LC and the interface may be empty. Disconnect the tubing from the LC and manually inject solvent through the tubing until it flows out of the nebulizer. If the solvent does not exit the nebulizer and you feel resistance, check for blockage.

✔ Check the LC pump for blockages

✔ Check the LC back pressure. If the back pressure is higher than normal, there may be a constriction or blockage in the solvent path. While solvent is being injected into the tubing (manually or by the pump), disconnect the tubing from the nebulizer. If there is flow from the tubing, the nebulizer is plugged. Replace the nebulizer needle. If there is no flow, continue to disconnect components until there is flow. The last component disconnected is plugged and should be replaced.

✔ Check the syringe pump, if present

Make sure the syringe pump is on and the pusher block is pressing against the syringe plunger. Make sure the syringe has not reached the end of its travel.

✔ Check the pump settings

Make sure pump settings match the syringe diameter and desired flow.

✔ Check the tubing to make sure it is primed

The tubing between the pump and the interface may be empty. Disconnect the syringe and manually inject solvent through the tubing until it flows out of the nebulizer. If the solvent does not exit the nebulizer and you feel resistance, check for blockage.

✔ Check the syringe pump for blockages

While solvent is being injected into the tubing (manually or by the pump), disconnect the tubing from the nebulizer.

If there is flow from the tubing, the nebulizer is plugged. Replace the nebulizer.

If there is no flow, continue to disconnect components until there is flow. The last component disconnected is plugged and should be replaced.

If the spray is not straight

If the spray that emerges from the needle is angled off to one side rather than straight, check the following:

✔ Check the condition of the needle.

Stop the solvent flow and remove the nebulizer. Use a magnifier to examine the tip of the nebulizer needle. Make sure it is not damaged. Very gently wipe off any deposits on the end of the nebulizer. If the spray is still not straight, there may be a partial blockage inside the tip of the nebulizer needle. Replace the nebulizer needle.

If the nebulizer produces droplets

If droplets rather than a spray are exiting the needle, check these steps. Be sure that the nebulization gas is turned off before checking to detect flow.

✔ Check the nebulizing gas

Make sure there is an adequate supply of nebulizing gas at adequate pressure.

Make sure the nebulizing gas pressure is set to the appropriate pressure for the LC flow rate.

Make sure the appropriate tubing is connected from the nebulizing gas supply to the interface.

✔ Check that the solvent system is ready

✔ Check the condition of the nebulizer

Stop the solvent flow and remove the nebulizer assembly. Use a magnifying glass to examine the tip of the nebulizer needle. Make sure it is not damaged. If the nebulizer needle is damaged, replace it (see the “Time-of-Flight LC/MS Maintenance Manual” for details).

✔ Check the electrospray high voltages

Verify that the settings are correct, the voltages are turned on, and the voltages are really present.

✔ Check the glass capillary for damage or contamination

✔ Check that the spray chamber is closed

Pressure

Abnormal pressure in the vacuum system is sometimes an indication of a problem with the Time-of-Flight LC/MS. In general, pressure readings can be divided into two groups:

- Readings seen when the system is open.

  System open means the capillary is uncapped; nebulizing gas, drying gas, and solvent (or sample) are flowing; and the drying gas heater is on.

- Readings seen when the system is closed.

  System closed means the capillary is capped with a rubber septum, and gases and solvent are not flowing.

The table below shows typical pressure readings. Because pressure is influenced by many variables, it is important that you familiarize yourself with the pressures that are typical for your instrument and your analyses.

Table 11 Normal pressure readings

	Pressure (in torr)	
	System closed (capillary capped)	**System open (capillary uncapped)**
• Rough vacuum pressure (Stage 1)	< 1	2 to 3
• High Vacuum (Stage 5)	$< 5 \times 10^{-7}$	< 2 x 10-6

If the Stage 1 pressure is too low – system open

When the system is open, the stage 1 pressure should be 2 to 3 torr. If the pressure is less than 1.5 torr, check the following:

✔ Check that the capillary is not capped.

Leaving the cap in place is a common cause of lower-than-normal pressure.

WARNING **Make sure spray chamber voltages are off and parts are cool before you remove the capillary cap.**

✔ Check the capillary for blockages or restrictions.

1. Vent and turn off the Time-of-Flight LC/MS.

   See "To shut down the instrument" on page 22.
2. Remove the capillary (see "To remove the capillary" on page 68).
3. Inspect the capillary for blockages or deposits (see "To clean the capillary" on page 70).
4. Reinstall the capillary (see "To reinstall the capillary" on page 73).
5. Start up the Time-of-Flight LC/MS again.

   See "To start the system in Standby mode" on page 15.

✔ Check whether the rough pump fluid was recently replaced.

Pumps with new pump fluid can perform better and create a lower-than-normal pressure. This is not a problem. The pressure will increase slowly with time as the pump fluid ages.

If the Stage 1 pressure is too high – system closed

With the system closed, the stage 1 pressure should be less than 1 torr. If it is greater than 1 torr, check these steps.

✔ Check the rubber septum on the capillary inlet.

A capillary cap that is not secure is the most common cause of this symptom.

✔ Check the fluid level and condition in the rough pump.

If needed, add or replace the pump fluid.

If the Stage 5 pressure is too high – system closed

If the system is closed, the Stage 5 pressure should be approximately 7×10^{-7} torr or lower. If the pressure is higher than 2×10^{-6} torr, check these steps:

✔ Check the rubber septum on the capillary inlet.

The rubber septum may not be securely installed. If this is the cause, the rough pump pressure (stage 1) will also be higher than normal.

✔ Check the capillary seals.

The capillary seals can leak. If this is the cause, the fore pressure (stage 1) will also be higher than normal. See those symptoms for more information.

✔ Check the fluid level in the rough pump.

The rough pump is possibly not working. If needed, add pump fluid.

✔ Check whether the TOF system was recently vented.

If so, check whether there is a leak at the O-ring that seals the analyzer cover to the vacuum manifold.

If the Stage 5 pressure is too high – system open

With the system open, the analyzer (rear) manifold pressure should be approximately 7 x 10^{-7} torr or lower. If the pressure is more than that, check these steps.

✔ Check the fluid level in the rough pump

The rough pump is possibly not working correctly. If needed, add pump fluid.

✔ Check whether the TOF system was recently vented.

If so, check if you have a leak in the O-ring that seals the analyzer cover to the vacuum manifold.

✔ Check for leaks.

Check the rough pump pressure readings. If the rough pump pressure is too high, you possibly have a leak.

Temperature

This section describes symptoms related to heating the drying gas.

If the drying gas does not heat

✔ Check if the drying gas heater is turned on.

Put the system in Off mode and then back to On mode.

✔ Check the gas pressure.

If the gas supply pressure is too low (< 80 psi), the heater is turned off automatically to protect the heater, and an instrument fault is generated.

If the drying gas heats but does not reach set point

✔ Wait a little bit longer.

At high gas flow rates, the time required for the temperature to reach its set point may take several minutes.

Divert Valve

For maintenance information, see “Divert Valve” on page 98.

“Divert valve connections” on page 134 illustrates the divert valve and its connections.

If you get no peaks in position “MS”

See (Figure 62 on page 134).

- ✔ Check if the capillary line is attached (outlet: plug 3 to ion source; inlet: plug 2 to front side inlet plug)
- ✔ Check if the capillary line is damaged (bent or broken). Replace it with a new one.

If when switched to waste there is no flow, or pressure is too high

- ✔ Check if the waste connection is broken (Figure 63 on page 135).

Replace the waste capillary line.

Figure 61 Divert valve connections

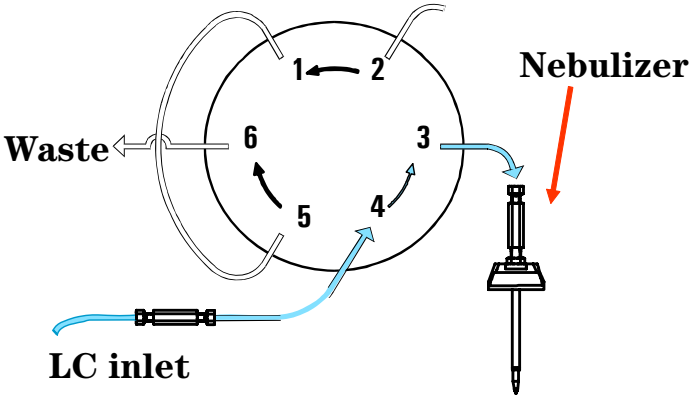

Figure 62 “LC -> MS” selected in the Tune tab of the Method pane

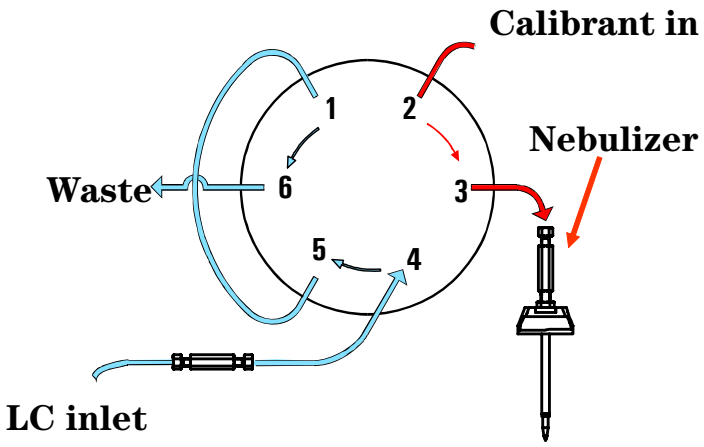

Figure 63 “LC -> Waste” or “Cal B” selected in the Tune tab of the Method pane

APCI

This section covers symptoms with the APCI.

If you do not get high voltages

✔ Check that the APCI high voltages are turned on

Put the instrument in Standby mode and back to On mode in the Status pane.

If you get the message “Spray Chamber Open”

The spray chamber is possibly not securely closed.

✔ Check that the spray chamber is closed and latched.

If you get a weak or no signal

The currents depend on the electrode voltage and the solvent. Aqueous solvents are more conductive and result in higher currents than non-aqueous solvents. Currents that are too high or unstable often indicate arcing or corona discharge that will greatly reduce or eliminate signal.

✔ Adjust the drying gas flow (optimum greater than 3.5 L/minutes) or the spray chamber voltages. In negative ionization mode, 1 to 5 percent oxygen in the nebulizing gas can help prevent unstable currents.

If you get no current

No current indicates a bad electrical contact.

✔ Check if the cap is placed on the capillary entrance, and the spray shield is correctly installed.

✔ Check for damage to the metal plating on the entrance end of the capillary.

1 Shut down the Time-of-Flight LC/MS.

"To shut down the instrument" on page 22

2 Remove the ion transport capillary.

See "To remove the capillary" on page 68.

3 Check the electrical continuity between the side of the capillary contact and the metal-plated entrance end of the capillary.

The measured resistance should be a few ohms or less. If the resistance is significantly higher, replace the capillary. Check continuity between all three surfaces on the end of the capillary. Check the end, the side, and the beveled surfaces. It is not unusual to have continuity to one or two of these surfaces but not the others. This is because the metal plating at the corners can be worn away or vaporized. This type of damage is not always visible.

4 Re-install the cap on the capillary entrance and the spray shield.

5 Restart the system.

See "To start the system in Standby mode" on page 15.

4
Reference

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This chapter contains reference information for the 6210 or 6220 TOF hardware.

NOTE For information on maintaining an APPI source, a nanospray source, or a dual nanospray source, please refer to the User's Guide for that product.

Safety Issues

Some of the procedures in this topic require access to parts of the 6210 or 6220 instrument while it is in Standby mode or shortly after it is turned off. If these procedures are not done correctly, they can be dangerous and may expose you to dangerous temperatures, voltages, and chemical hazards. This topic explains these potential dangers. Read it carefully.

High temperatures

Many parts in the 6210 or 6220 instrument operate at, or during operation reach, temperatures high enough to cause serious burns. These parts include, but are not limited to:

- Mechanical pumps
- Drying gas heater
- Drying gas
- APCI heater (vaporizer)
- Capillary and capillary cap
- Spray shield
- Any other parts that come in contact with the drying gas, such as the entire spray chamber

Most of these parts are normally covered or shielded. Even so, the covers can become hot. All contact with these parts, shielded or not, should be avoided.

WARNING **Many of these parts remain hot for long after the 6210 or 6220 instrument is shut down or turned off. Use extreme caution when you work on an instrument that has recently been turned off**

Hazardous voltages

Whenever the 6210 or 6220 instrument is not turned off and disconnected from the power supply, many parts use hazardous voltages. Parts that use hazardous voltages include, but are not limited to:

- Mechanical pumps
- Transformers and power supplies in the 6210 or 6220 instrument cabinet
- High voltage electrodes (capillary and end plate) in the spray chamber

- Drying gas heater
- APCI heater
- APCI corona needle
- Wiring and cables between these parts
- Octopole RF generator
- Multiplier cables
- HV mirror cables
- HV pulser cables

All of these parts are usually covered or shielded. As long as the covers and shields are in place, it would be difficult to accidentally contact hazardous voltages.

WARNING

Never remove any of the instrument covers while the 6210 or 6220 instrument is turned on and connected to a power source.

Do not interrupt the protective conductor inside or outside the instrument. Do not disconnect the protective earth terminal. Either of these actions can make the instrument dangerous. Intentional interruption is strictly prohibited.

Disconnect this equipment from the power source by removing all power cords from the power outlets when servicing.

Chemical residues

The API-electrospray interface only ionizes a portion of the sample and solvent. The majority of sample and solvent passes through the interface without being ionized. The vacuum pumps of the 6210 or 6220 instrument carry away the un-ionized sample and solvent, therefore the exhaust from these pumps can contain traces of your samples and solvents. Vent all pump exhausts to the outside or into a fume hood. Comply with your local air quality regulations.

WARNING

Vent all exhausts externally to the outdoors where they cannot be recirculated by environmental control systems. Do not vent exhausts into your laboratory. The exhaust fumes from the vacuum system and spray chamber will contain trace amounts of whatever chemicals you are analyzing. Potential health hazards include chemical toxicity to solvents, samples, buffers, and pump fluid vapor, as well as potentially biohazardous aerosols of biological samples. See the warning labels on the instrument. Comply with your local air quality regulations.

When you replace the pump fluid, use protective gloves and safety glasses. Avoid contact with the fluid.

Fluid drained from the spray chamber is composed of solvent and sample from your analyses. The fluid in the mechanical and diffusion pumps collects traces of the samples and solvents. In addition, unnebulized solvent and sample collects at the bottom of the spray chamber. Connect the drain on the bottom of the spray chamber to a closed container.

Handle and dispose all fluid with care appropriate for its chemical and/or biological content.

Handle all used pump fluid as hazardous waste. Dispose of used pump fluid as specified by your local regulations.

Protective equipment

Safety glasses and other protective equipment should be worn during all maintenance procedures.

Electrostatic discharge

All of the electronic assemblies in the 6210 or 6220 instrument contain components that can be damaged by electrostatic discharge (ESD). None of these are located in areas that should be accessed by the operator.

Power supply information

This equipment must be installed in an environment of Category II installation as defined in IEC 664. Ensure the supply voltage does not fluctuate more than ±10% of the rate voltage.

Cleanliness

Cleanliness and preventing accidental contamination during maintenance are very important. Contamination of the interior of the vacuum system or the sample path can affect the results of your analyses.

Always wear clean gloves when handling parts that come in contact with the sample path. Oil from your fingers is a difficult contaminant to remove.

When you set parts down, place them on clean, lint free cloths or clean aluminum foil, not directly on the laboratory bench. Keep parts covered so that dust does not collect on them.

If possible, maintain a separate set of tools that have been thoroughly cleaned. Use these tools only when working on clean assemblies.

WARNING **Do not leave the vacuum manifold of the 6210 or 6220 instrument open to the atmosphere. For example, if you remove the spray chamber for maintenance, cover the opening in the front of the manifold while you work on the spray chamber.**

Safety Symbols

NOTE

This symbol is placed on the product where it is needed for you to refer to the **manual** in order to understand a **hazar**d.

WARNING CAUTION

This symbol is placed on the product within the area where **hazardous voltage** is present or shock hazard can occur. Only trained service persons should perform work in this area.

WARNING

This symbol is placed on the product within the area where **hot parts and surfaces** are present. Allow the product to cool before performing work in this area.

WARNING

This symbol is placed on the product within the area where **biohazards** are present. Handle these areas with the respective care.

Technical Specifications

Size

Table 12 Agilent 6200 Series Dimensions

Height	Length	Width
48 cm	64 cm	62 cm
19 in.	25 in.	25 in.

Weight 108 kg

Current 15 A

Power consumption 2000 VA

Frequency 50 Hz / 60 Hz ± 3%

Current protector (Automatic cutout) 15 A

Operating Temperature 15°C to 35 °C (59 °F to 95 °F) (Analytical specifications will be met only within a temperature range of ± 3 °C (± 6 °F).

Operating Humidity 15% to 95% (non condensing at any temperature)

Operating Altitude < 4572 m (15 000 ft.)

Spare part numbers

Table 13 Agilent 6200 Series Parts

Description	Spare Part Numbers
Miscellaneous	
Abrasive mesh (micro-grit paper)	8660-0827
Adapter Tip, Valco	0100-2049
Capillary	59987-20040
Capillary cap	G1946-20302
Capillary cleaner	G1946-80054
Cloths, clean, lint free (15/pkg)	05980-60051
Gloves, clean, lint free	9300-1751
Inlet Filter Assembly	G1946-60180
Nitrogen gas purifier (1/4″ fitting)	BHT-4
Reserpine	G2423A
Rough pump oil, Inland 45, 1 liter	6040-0834
Skimmer	G1969-20302
Spray shield	G1946-20157
Spare Filter Elements	0100-2051
Teflon tubing, 1/4″ OD gas	G1946-80078
Tie wraps (10/pkg)	1400-0482
Tubing, 1/2″ ID Exhaust	G1946-80077
Tools	
Hex Wrench, 6 mm	8710-1839
Screwdriver, 11″	8710-1029
Screwdriver, Pocket	8730-0008

Table 13 Agilent 6200 Series Parts

Description	Spare Part Numbers
Screwdriver Torx T-10	8710-1623
Screwdriver Torx T-20	8710-1615
Torx Wrench, 6 mm	8710-1839
Tubing Cutter	8710-1930
Wrench, 3-mm, open-end	8710-2699
Wrench, 1/4" x 5/16"	8710-0510
Wrench, open-end 1/2" and 7/16"	8710-0806
Wrench, open-end 1/2" and 9/16"	8710-0877

Table 14 APCI spare parts

Description	Spare Part Number
APCI Corona needle	G2429A
APCI Nebulizer needle	G2428A
APCI source	G1947A
Nebulizer APCI assembly	G1946-60037
Nebulizer needle ferrule	G1946-20213
O-ring for Nebulizer	0905-1022

Table 15 Tuning and reference mixtures

Description	Spare Part Number
APCI / APPI Tuning Mixture	G1969-85010
ESI Tuning Mixture	G1969-85000
MMI Tuning Mixture	G1969-85020

Table 15 Tuning and reference mixtures

Description	Spare Part Number
TOF Reference Mass Solution	G1969-85001
ES-TOF Biopolymer Analysis Reference Mass Standard Kit	G1969-85003

For other parts list, see:

- Table 2, “ES Nebulizer Parts List,” on page 60
- Table 3, “APCI Nebulizer Parts List,” on page 86
- Table 5, “TOF Octopole 1 Cabling Parts List,” on page 113
- Table 6, “Ion Optics and Octopole 1 Assembly Parts List,” on page 115

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In This Book

This book contains the information to use, maintain, and troubleshoot your Time-of-Flight LC/MS hardware.

For more information on getting started with your system, see the *Agilent 6200 Series TOF LC/MS System Quick Start Guide*.

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