



PhotoSpray® Ion Source

Operator Guide



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Ion Source Overview

1

Atmospheric pressure photoionization offers an alternative method of introducing samples into the mass spectrometer. The PhotoSpray[®] ion source, much like the standard atmospheric pressure chemical ionization (APCI) probe and Turbo V[™] ion source, generates ions that represent the molecular composition of the sample.

From an applications point of view, this ion source provides a more versatile version of APCI, which can allow for greater sensitivity when optimized for specific compounds. The ion source also enhances LC-MS/MS detection of biological compounds and environmentally important compounds of low polarity.

Operational Precautions and Hazards

For regulatory and safety information for the mass spectrometer, refer to the safety guide or *System User Guide*.



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Use the ion source only if you have knowledge of and training in the proper use, containment, and evacuation of toxic or injurious materials used with the ion source.



WARNING! Puncture Hazard, Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Discontinue use of the ion source if the ion source window is cracked or broken and contact an SCIEX Field Service Employee. Any toxic or injurious materials introduced into the equipment will be present in the ion source and exhaust output. Dispose of sharps following established laboratory safety procedures.



WARNING! Hot Surface Hazard. Let the ion source cool for at least 30 minutes before starting any maintenance procedures. Surfaces of the ion source and the vacuum interface components become hot during operation.



WARNING! Toxic Chemical Hazard. Wear personal protective equipment, including a laboratory coat, gloves, and safety glasses, to avoid skin or eye exposure.

Ion Source Overview



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. In the event of a chemical spill, review product Safety Data Sheets for specific instructions. Stop the spill only if it is safe to do so. Make sure that the system is in Standby mode before cleaning a spill near the ion source. Use appropriate personal protective equipment and absorbent wipes to contain the spill and dispose of it following local regulations.



WARNING! Environmental Hazard. Do not dispose of system components in municipal waste. Follow established procedures when disposing of components.

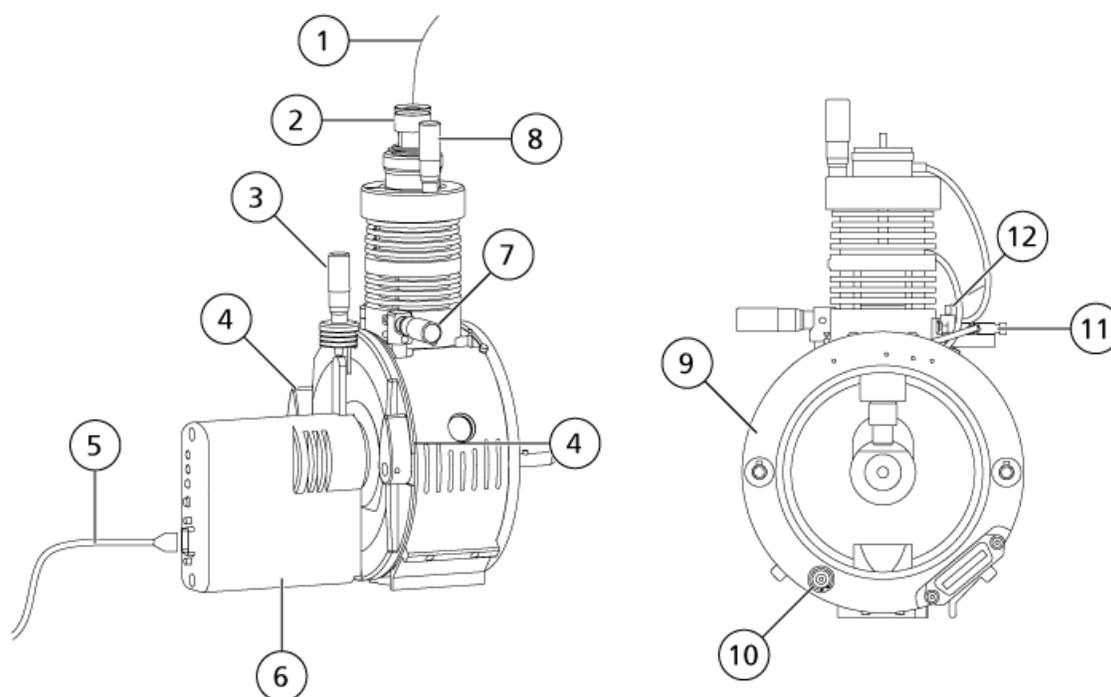


WARNING! Electrical Shock Hazard. Avoid contact with the high voltages applied to the ion source during operation. Put the system in Standby mode before adjusting the sample tubing or other equipment near the ion source.

Ion Source Components

This ion source uses a heated nebulizer to vaporize the sample. Following vaporization, the molecular constituents of the sample are ionized by the process of photoionization, induced by a continuous beam of ultraviolet radiation in the presence of a dopant within the ion source housing.

Figure 1-1 PhotoSpray Ion Source



Item	Description
1	Sample tubing
2	APCI probe
3	UV lamp micrometer used to set the vertical position for ion source sensitivity adjustments
4	Two source latches that secure the ion source to the mass spectrometer
5	Ion source cable to the mass spectrometer
6	UV lamp control module that houses the LED lamp status display and a 9-pin connector port for the ion source cable to supply electrical power and communication to the UV lamp
7	APCI probe micrometer used to position the probe on the horizontal axis for ion source sensitivity adjustments
8	APCI probe micrometer used to position the probe on the vertical axis for ion source sensitivity adjustments
9	Ion source housing plate that houses a high-voltage connector, ion source identity connector, and connection ports for ion source electrical power and gas supply
10	High voltage connection

Ion Source Overview

Item	Description
11	Dopant inlet used to attach the dopant supply line to introduce dopant to the nebulizer gas
12	Cable tie

Dopant Supply Line and Sample Tubing

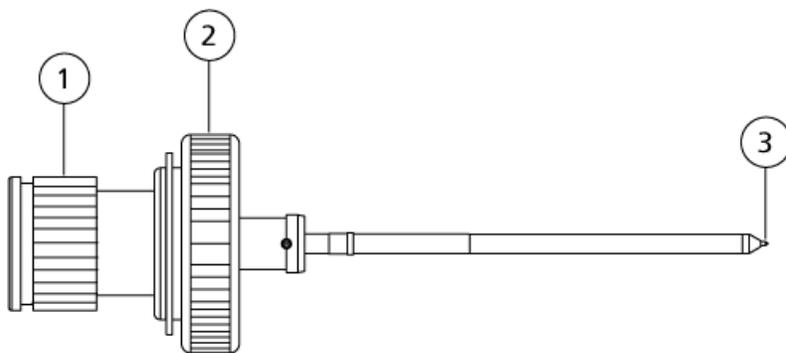
Manually connect a dopant supply line to the dopant inlet to introduce dopant to the nebulizer gas and manually connect the sample tubing to the probe. Refer to [Install the Ion Source Connections](#).

APCI Probe

The APCI probe consists of 100 µm inside diameter (i.d.) (0.004 inch) stainless steel tubing surrounded by a flow of nebulizer gas (Gas 1). The liquid sample stream is pumped through the sprayer, where it is nebulized in a ceramic tube containing a heater. The inside wall of the ceramic tube can be maintained at a temperature range of 100 °C to 750 °C and is monitored by the sensor embedded in the heater.

A high-velocity jet of nebulizer gas flows around the electrode tip to disperse the sample as a mist of fine particles. It moves through the ceramic vaporization heater into the reaction region of the ion source and then past the corona discharge needle where the sample molecules are ionized as they pass through the ion source housing. Refer to [Principles of Operation—Ion Source on page 41](#).

Figure 1-2 Parts of the APCI Probe

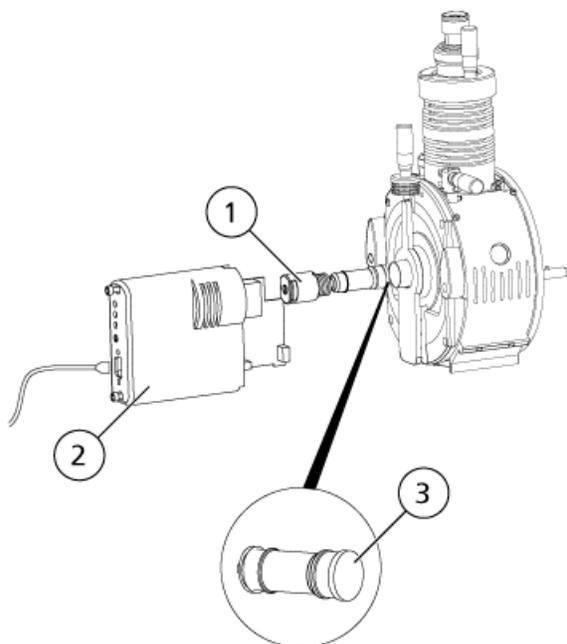


Item	Description
1	Electrode adjustment nut (black collar) that adjusts the extension of the electrode tip
2	Retaining ring that secures the probe in the probe tower
3	Electrode tip through which samples are sprayed into the sample inlet area of the ion source

UV Lamp

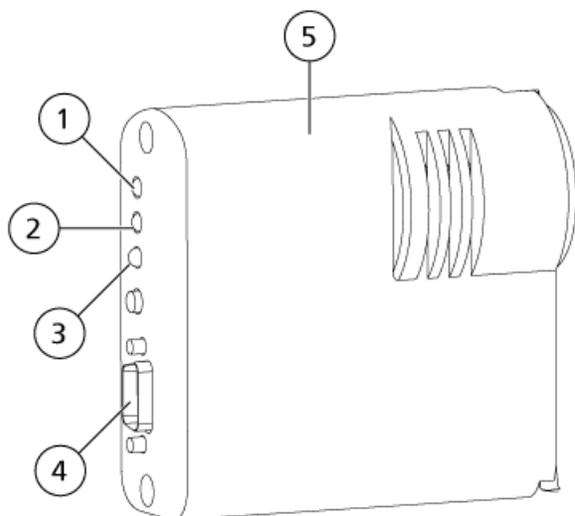
The krypton photoionization UV lamp is located within, and controlled by, the UV lamp control module. This UV lamp ionizes the dopant molecules within the incoming sample stream.

Figure 1-3 PhotoSpray Ion Source UV Lamp



Item	Description
1	Lamp holder
2	Lamp control module
3	UV lamp

Figure 1-4 Lamp Control Module and LED Status Display



Item	LED	Color	Description
1	Check	Red	Fault condition. The UV lamp could be on or off. Refer to Troubleshooting Tips on page 39 .
2	Normal	Green	The mass spectrometer is in Ready mode. The UV lamp is on.
3	Ignition	Yellow	The mass spectrometer is in Standby mode. The UV lamp is off.
4	—	—	9-pin connector: Additional low-voltage power and logic connections are made through the ion source cable that runs from the 25-pin sources connector on the mass spectrometer to the 9-pin connector.
5	—	—	Lamp control module

Gas and Electrical Connections

Gas and high-voltage electrical connections are provided through the front plate of the interface and connect internally through the ion source housing. When the ion source is installed on the mass spectrometer, all of the electrical and gas connections are complete.

Additional low-voltage power and logic connections are made through the ion source cable that runs from the 25-pin sources connector on the mass spectrometer to the 9-pin connector on the UV lamp power supply that is mounted on the ion source housing.

An ion source cable from the mass spectrometer connects to the UV (ultra violet) lamp control module to provide electrical power and communication from the mass spectrometer to the UV lamp. LED (light emitting diode) indicators on the UV lamp control module display the current status of the UV lamp.

When the ion source is installed on the mass spectrometer, most of the electrical and gas connections are made automatically. Refer to [Figure 1-1 on page 7](#) and [Figure 1-3 on page 9](#).

Ion Source Sense Circuit

An ion source sense circuit disables the high-voltage power supply for the mass spectrometer and the source exhaust system if:

- The ion source housing is not installed or is improperly installed.
- A probe is not installed.
- The mass spectrometer senses a gas fault.

Source Exhaust System



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Be sure to use the source exhaust system to safely remove sample vapor exhaust from the laboratory environment. For requirements for the source exhaust system, refer to the *Site Planning Guide*.



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Vent the source exhaust system to either an external fume hood or an external vent to prevent hazardous vapors from being released into the laboratory environment.



WARNING! Fire Hazard. Do not direct more than 3 mL/min of solvent in to the ion source. Exceeding the maximum flow rate can cause solvent to accumulate in the ion source. Make sure that the source exhaust system is working, to prevent flammable vapor from accumulating in the ion source.

All ion sources produce both sample and solvent vapors. These vapors are a potential hazard to the laboratory environment. The source exhaust system is designed to safely remove and allow for the appropriate handling of the sample and solvent vapors. When the ion source is installed, the mass spectrometer will not operate unless the source exhaust system is operating.

Ion Source Overview

A vacuum switch mounted in the source exhaust circuit measures the vacuum in the source. If the vacuum in the source rises above the set point while the probe is installed, the system goes into an exhaust fault (Not Ready) state.

An active exhaust system removes ion source exhaust (gases, solvent, sample vapor) through a drain port without introducing chemical noise. The drain port connects through a drain chamber and a source exhaust pump to a drain bottle, and from there to a customer-supplied exhaust ventilation system. For more information on the ventilation requirements for the source exhaust system, refer to the *Site Planning Guide*.

Contact Us

SCIEX Support

- sciex.com/contact-us
- sciex.com/support/request-support

Customer Training

- In North America: NA.CustomerTraining@sciex.com
- In Europe: Europe.CustomerTraining@sciex.com
- Outside the EU and North America, visit sciex.com/education for contact information.

Online Learning Center

- sciex.com/LearningPortal

Related Documentation

The guides and tutorials for the Analyst[®] software are installed automatically with the software and are available from the Start menu: **All Programs > SCIEX > Analyst**. A complete list of the available documentation can be found in the Help. To view the Help, press **F1**.

Documentation for the mass spectrometer can be found on the *Customer Reference DVD* for the mass spectrometer.

Documentation for the ion source can be found on the *Customer Reference DVD* for the ion source.

Technical Support

SCIEX and its representatives maintain a staff of fully-trained service and technical specialists located throughout the world. They can answer questions about the system or any technical issues that might arise. For more information, visit the SCIEX Web site at sciex.com.

Ion Source Installation

2



WARNING! Electrical Shock Hazard. Install the ion source on the mass spectrometer as the last step in this procedure. High voltage is present when the ion source is installed.

The ion source is connected to the vacuum interface and is held in position by two source latches. The interior of the ion source is visible through the tempered glass.

When the ion source is installed, the Analyst[®] software recognizes the ion source and shows the ion source identification.

Note: Always use a separate dopant LC pump. Using a syringe pump will greatly reduce the sensitivity of the ion source. In previous versions of the PhotoSpray[®] ion source, the dopant was introduced in a non-pressurized line, and a syringe pump was sufficient.

Required Materials

- Ion source
- APCI probe
- Ion source consumables kit

Prepare for Installation



WARNING! Puncture Hazard. Be careful when handling the electrode tube. The tip of the electrode is extremely sharp.

Tip! Do not discard the empty package. Use it to store the ion source when it is not in use.

- Adjust the black electrode adjustment cap on the probe to move the electrode tip inside the electrode tube.
- Make sure the dopant supply line and sample tubing are long enough to connect to the ion source with enough slack to move the fully connected ion source housing from the bench to the vacuum interface housing.
- Make sure the electrode tip of the probe is adjusted to specification. For optimum stability and performance, the electrode should extend between 0.5 mm and 1.0 mm from the end of the probe.

Ion Source Installation

This distance affects the shape of the spray cone, which affects the sensitivity of the mass spectrometer. Refer to [Adjust the Electrode Tip Extension on page 32](#).

Gas Connections Required

Make sure that the following gases are available:

- Clean zero air
- Ultrahigh purity (UHP) nitrogen

Inspect the following gas connections to the mass spectrometer:

- Curtain Gas™ supply is UHP nitrogen.
- Source exhaust gas supply is zero air.
- Gas 1/Gas 2 is UHP nitrogen.

Using UHP nitrogen for Gas 1/Gas 2 provides greater sensitivity than zero air.

Provide the following gas requirements for the ion source as shown in [Table 2-1](#).

Table 2-1 Gas Requirements

Component	Gas requirement
PhotoSpray® ion source	12 L/min UHP nitrogen

Install the Probe



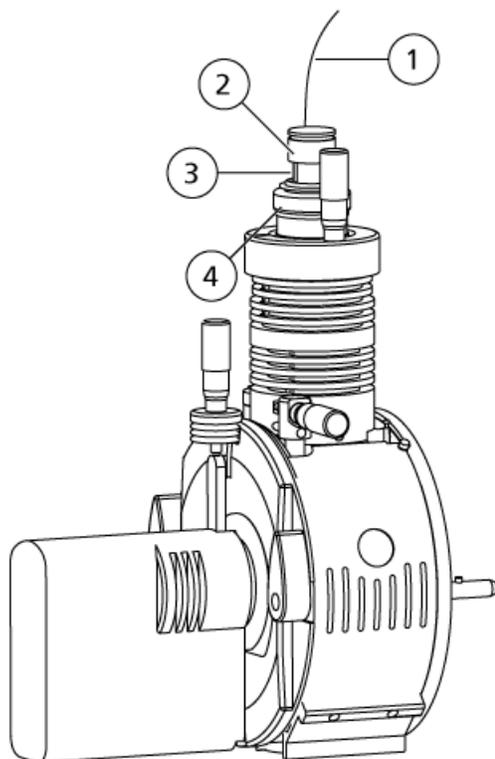
WARNING! Electrical Shock Hazard. Make sure that the ion source is completely disconnected from the mass spectrometer before proceeding.

The probe is not pre-installed in the ion source. Always remove the ion source from the mass spectrometer before exchanging probes. Refer to [Remove the Ion Source on page 29](#).

If the probe is not properly installed in the ion source then the high-voltage power for the mass spectrometer and source exhaust system is turned off.

1. Insert the probe into the tower. Refer to [Ion Source Components on page 6](#).
2. Gently push down on the probe so that the contacts engage with those in the tower.
3. Turn the retaining ring over the probe, push it down to engage its threads with the threads on the tower, and then tighten it until it is fully tightened. Tighten it using fingers only to avoid damaging the threads.

Figure 2-1 Sample Tube Connection



Item	Description
1	Sample tubing
2	Electrode adjustment nut
3	Probe
4	Retaining ring

4. Thread the sample tubing nut into the fitting at the top of the probe and then tighten until the sample tubing nut is finger-tight.

Install the Ion Source Connections

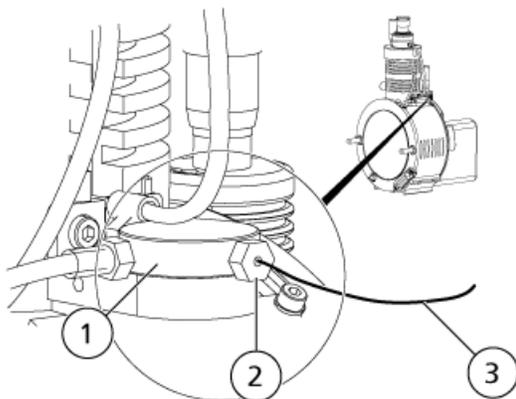
After the probe is installed, install the remaining ion source connections.

1. Install the dopant supply line in the dopant capillary.

Ion Source Installation

2. Thread the dopant tube nut in the union fitting and then tighten them until they are finger-tight. Refer to [Figure 2-2](#).

Figure 2-2 Dopant Supply Line

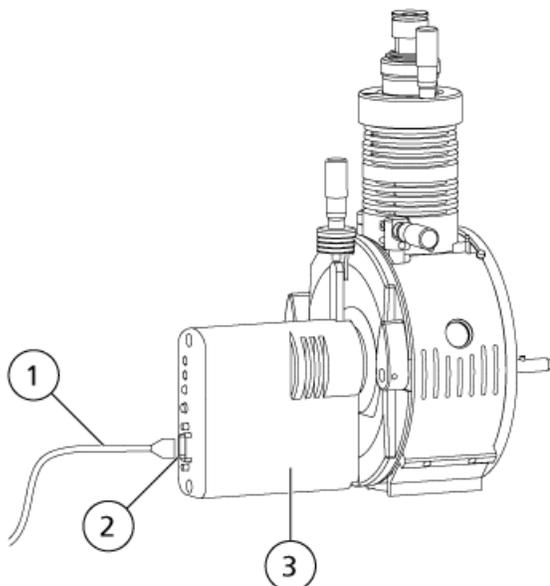


Item	Description
1	Union fitting
2	Dopant tube nut
3	Dopant supply line

CAUTION: Potential Equipment Damage: Do not disconnect the source cable from the ion source or mass spectrometer during operation.

3. Connect the source cable from the source connector on the I/O panel of the mass spectrometer to the 9-pin connector on the UV lamp control module. Install both clamping screws and then finger-tighten them. Refer to [Figure 2-3](#).

Figure 2-3 Source Cable Connector



Item	Description
1	Source cable to the mass spectrometer
2	9-pin connector
3	Lamp control module

Connect the Ion Source Tubing



WARNING! Electrical Shock Hazard. Do not bypass the grounding union connection. The grounding union provides grounding between the mass spectrometer and the sample introduction device.



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Make sure that the sample tubing nut is tightened properly before operating this equipment, to prevent leakage.

Refer to [Ion Source Components on page 6](#).

1. Insert a 30 cm piece of red PEEK tubing into the sample tubing nut.

Ion Source Installation

2. Install the sample tubing nut in the port at the top of the probe, and then tighten the sample tubing nut until it is finger-tight.
3. Connect the other end of the tubing to the grounding union on the ion source.

Install the Ion Source on the Mass Spectrometer



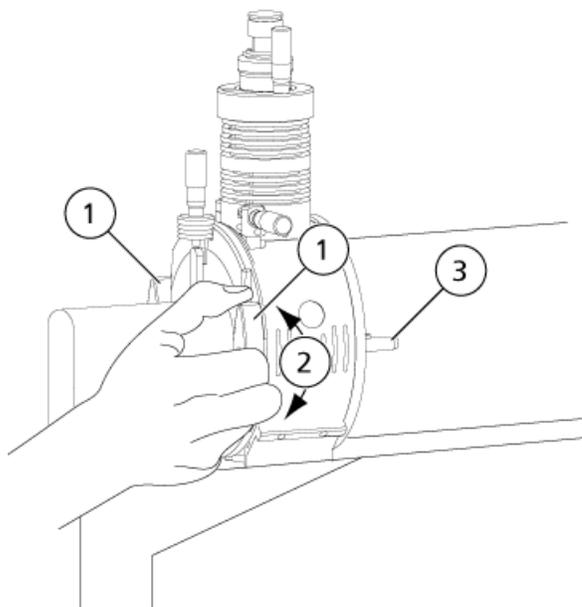
WARNING! Electric Shock Hazard. Install the probe in the ion source before installing the ion source on the mass spectrometer.

Tip! Use the correct orifice plate for the system for optimal performance. Do not use an orifice plate for another system. The model number for the system is etched into the orifice plate.

If the ion source probe is not properly installed, then the high-voltage power supply is not available.

1. Make sure that the source latches on either side of the ion source are pointing up in the 12 o'clock position. Refer to [Ion Source Components on page 6](#).
2. Align the ion source with the vacuum interface, making sure that the latches on the ion source are aligned with the sockets in the vacuum interface.
3. Push the ion source gently against the vacuum interface and then rotate the ion source latches down to lock the ion source into place.

Figure 2-4 Installing the Ion Source



Item	Description
1	Source latches
2	Open (12 o'clock) and closed (6:30)
3	Guide pins (2)

The mass spectrometer recognizes the ion source and then shows the ion source identification in the Analyst[®] software.

4. Connect the tubing from the sample supply device to the grounding union on the ion source.

Inspect for Leaks

Inspect fittings and tubing to make sure that there are no leaks.

Ion Source Optimization

3



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Use the ion source only if you have knowledge of and training in the proper use, containment, and evacuation of toxic or injurious materials used with the ion source.



WARNING! Fire Hazard. Do not direct more than 3 mL/min of solvent in to the ion source. Exceeding the maximum flow rate can cause solvent to accumulate in the ion source. Make sure that the source exhaust system is working, to prevent flammable vapor from accumulating in the ion source.



WARNING! Puncture Hazard, Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Discontinue use of the ion source if the ion source window is cracked or broken and contact an SCIEX Field Service Employee. Any toxic or injurious materials introduced into the equipment will be present in the ion source and exhaust output. Dispose of sharps following established laboratory safety procedures.

Note: If the IonSpray™ voltage is too high, then a corona discharge can occur. It is visible as a blue glow at the tip of the probe. A corona discharge results in decreased sensitivity and stability of the signal.

Optimize the ion source whenever the analyte, flow rate, or mobile phase composition changes.

Several parameters affect the performance of the source. Optimize the performance while injecting a known compound and monitoring the signal of the known ion. Adjust the micrometer, gas, and voltage parameters to maximize the signal-to-noise ratio and signal stability.

Sample Introduction

Method

The liquid sample stream is delivered to the ion source by an LC pump or by a syringe pump. If it is delivered by an LC pump, then the sample can be injected directly into the mobile phase using flow injection analysis (FIA) or tee infusion, or through a separation column using a loop injector or autosampler. If it is introduced by a syringe pump, then the sample is injected directly into the ion source. Infusion optimization can be used for ion path optimization and MS/MS fragment selection.

Flow Rate

Sample flow rates are determined by the chromatography system or by the volume of the sample available.

Sample Inlet Requirements

- Use appropriate analytical procedures and practices to minimize external dead volumes. The sample inlet transfers the liquid sample to the ion source inlet without loss and with minimal dead volume.
- Prefilter samples so that the capillary tubing in the sample inlets is not blocked by particles, precipitated samples, or salts.
- Make sure that all connections are tight enough to prevent leaks. Do not over-tighten.

APCI Probe Optimization



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Make sure that the mass spectrometer is properly vented and that good general laboratory ventilation is provided. Adequate laboratory ventilation is required to control solvent and sample emissions and to provide for safe operation of the mass spectrometer.



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Make sure that the electrode protrudes beyond the tip of the probe, to prevent hazardous vapors from escaping from the source. The electrode must not be recessed within the probe.

CAUTION: Potential System Damage. If the HPLC system connected to the mass spectrometer is not controlled by the software, then do not leave the mass spectrometer unattended while in operation. The HPLC system can flood the ion source when the mass spectrometer goes into Standby mode.

Refer to [APCI Probe Parameters on page 46](#).

CAUTION: It is easier to optimize signal and signal-to-noise with flow injection analysis or on-column injections.

Set Up the System

1. Configure the LC pump to deliver the mobile phase at the required flow rate. Refer to [Source Parameters and Voltages on page 46](#).
2. Connect the grounding union on the ion source to an LC pump, through .
3. the autosampler to perform multiple injections.

Run the Method

1. Start the Analyst[®] software.
2. In the Navigation bar, under **Tune and Calibrate** mode, double-click **Manual Tuning**.
3. Open a previously optimized method or create a method based on the compounds.
4. If the ion source has been allowed to cool, then do the following.
 - a. Set the **Temperature (TEM)** parameter to **450**.
 - b. Let the ion source warm up for 30 minutes.
The 30-minute warm-up stage prevents solvent vapors from condensing in the cold probe.
5. Start acquisition.
6. Start the sample flow and sample injection.

Set the Starting Conditions

1. Type **70** in the **Ion Source Gas 1 (GS1)** field.
2. Type **20** in the **Ion Source Gas 2 (GS2)** field.
3. In the **Curtain Gas (CUR)** field, type the value appropriate to the mass spectrometer.

Table 3-1 CUR Parameter Values

Mass Spectrometer	Starting Value
3200, 3500, 4000, and 4500 systems	20
5000 and 5500 systems	25
6500 and 6500 ⁺ systems	30

4. Set **Ion Transfer Voltage** to **800**.

Optimize Gas 1, Gas 2, and Curtain Gas™ Flow

1. Adjust **GS2** in increments of five to achieve the best signal or signal-to-noise ratio.
2. Adjust **GS1** in increments of five to achieve the best signal or signal-to-noise ratio.
3. Increase the **CUR** parameter until the signal starts to decrease.

Note: To prevent contamination, use the highest value for CUR possible without sacrificing sensitivity. Do not set CUR lower than 20. This helps to prevent penetration of the Curtain Gas™ flow, which can produce a noisy signal, prevent contamination of the aperture, and increase the overall signal-to-noise ratio.

Optimize the APCI Probe Position



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Make sure that the electrode protrudes beyond the tip of the probe, to prevent hazardous vapors from escaping from the source. The electrode must not be recessed within the probe.

CAUTION: Potential Equipment Damage: Use care when making adjustments to the probe. In some combinations of probe and UV lamp position, it might be difficult to adjust the position of the probe because of the position of the UV lamp, and vice versa.

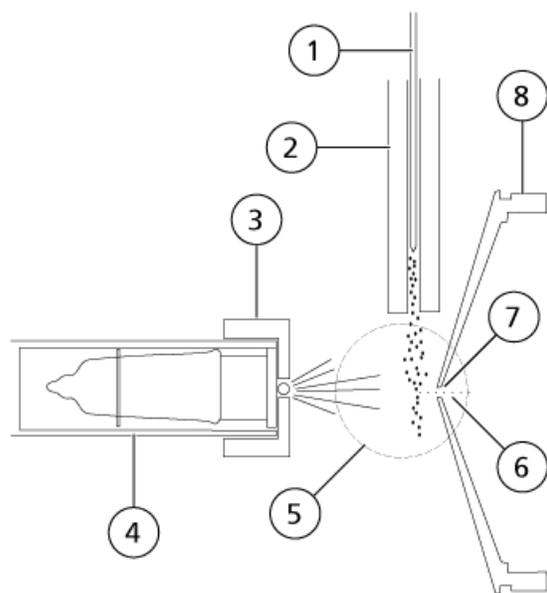
Make sure that the curtain plate aperture remains clear of solvent or solvent droplets at all times.

The position of the sprayer nozzle affects sensitivity and signal stability. Adjust probe sensitivity in small increments only. At lower flow rates, position the probe closer to the aperture. For higher flow rates, position the probe farther away from the aperture. After the probe is optimized, it needs only minor adjustment. If the probe is removed, or if the analyte, flow rate, or solvent composition changes, then repeat the optimization procedure.

Figure 3-1 shows the position of the probe relative to the UV lamp and curtain plate aperture.

Ion Source Optimization

Figure 3-1 APCI Probe Position



Item	Description
1	APCI probe
2	Heater
3	Ion deflector
4	UV lamp
5	Ionization region
6	Ions
7	Curtain plate aperture
8	Curtain plate

1. Use the previous micrometer as a starting position.

Note: To avoid reducing the performance of the mass spectrometer, do not spray directly into the aperture.

2. Use FIA or tee infusion to inject sample at a high flow rate.
3. Monitor the signal in the software.

4. Use the vertical micrometer to adjust the probe in small increments to achieve the best signal or signal-to-noise ratio.
5. Adjust the black electrode adjustment cap on the probe to move the electrode tube in or out of the probe. For optimum stability and performance, the electrode should extend between 0.5 and 1.0 from the end of the probe. Refer to [Adjust the Electrode Tip Extension on page 32](#).

Optimize the APCI Probe Temperature

The quantity and type of solvent affects the optimal APCI probe temperature. At higher flow rates, the optimal temperature increases.

- Adjust the **TEM** value in increments of 50 °C to 100 °C to achieve the best signal or signal-to-noise ratio.

Adjust the UV Lamp

CAUTION: Potential Equipment Damage: Use care when making adjustments to the UV lamp. In some combinations of probe and UV lamp position, it might be difficult to adjust the position of the probe because of the position of the UV lamp, and vice versa.

The position of the UV lamp relative to the curtain plate aperture affects sensitivity and signal stability. Always adjust the UV lamp in small increments.

To optimize the ion source settings for a given compound, try various UV lamp heights and optimize the ion transfer voltage at each new height setting to converge on the best setting for UV lamp height and ion transfer voltage.

The UV lamp adjustment depends on the compound. Repeat this optimization procedure if the analyte, flow rate, or solvent composition changes. After the probe is optimized, it needs only minor adjustment. If the probe is removed, or if the analyte, flow rate, or solvent composition changes, then repeat the optimizing procedure.

Note: The optimum ion transfer voltage is dependent upon the height of the UV lamp. There is only one optimum ion transfer voltage for a set UV lamp height and only one optimum UV lamp height for a given ion transfer voltage. After changing the UV lamp height, optimize the ion transfer voltage at each new height setting to converge on the best setting for UV lamp height and ion transfer voltage.

- Use the UV lamp vertical micrometer to adjust the vertical position to achieve the best signal or signal-to-noise ratio.

Optimization Tips

- Set the temperature at the lowest setting that results in the complete vaporization of the sample. The higher the liquid flow rate, or the higher the aqueous composition of the solvent, the higher the heater temperature required to vaporize the analyte. However, too high a temperature can cause thermal degradation of the compound.

Ion Source Optimization

- Use the highest Curtain Gas™ flow rate (CUR) possible without decreasing the signal. This helps to:
 - Prevent penetration of the Curtain Gas™ flow, which can produce a noisy signal.
 - Prevent contamination of the aperture.
 - Increase the overall signal-to-noise ratio.

For some difficult-to-ionize compounds, the signal intensity tunes sharply with Curtain Gas™ settings, often giving a sharp maximum at settings of 45 or 50. It is very important to tune the Curtain Gas flow when optimizing for the highest possible sensitivity.

Ion Source Maintenance

4

The following warnings apply to all maintenance procedures in this section.



WARNING! Hot Surface Hazard. Let the ion source cool for at least 30 minutes before starting any maintenance procedures. Surfaces of the ion source and the vacuum interface components become hot during operation.



WARNING! Fire and Toxic Chemical Hazard. Keep flammable liquids away from flame and sparks and use them only in vented chemical fume hoods or safety cabinets.



WARNING! Toxic Chemical Hazard. Wear personal protective equipment, including a laboratory coat, gloves, and safety glasses, to avoid skin or eye exposure.



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. In the event of a chemical spill, review product Safety Data Sheets for specific instructions. Stop the spill only if it is safe to do so. Make sure that the system is in Standby mode before cleaning a spill near the ion source. Use appropriate personal protective equipment and absorbent wipes to contain the spill and dispose of it following local regulations.



WARNING! Electrical Shock Hazard. Avoid contact with the high voltages applied to the ion source during operation. Put the system in Standby mode before adjusting the sample tubing or other equipment near the ion source.



WARNING! Puncture Hazard, Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Discontinue use of the ion source if the ion source window is cracked or broken and contact an SCIEX Field Service Employee. Any toxic or injurious materials introduced into the equipment will be present in the ion source and exhaust output. Dispose of sharps following established laboratory safety procedures.

This section contains general maintenance procedures for the ion source. To determine how often to clean the ion source or perform preventive maintenance, consider the following:

- Compounds tested

Ion Source Maintenance

- Cleanliness of the preparation methods
- Amount of time an idle probe contains a sample
- Overall system run time

These factors can cause changes in ion source performance, indicating that maintenance is required.

Make sure that the installed ion source is fully sealed to the mass spectrometer with no evidence of gas leaks. Regularly inspect the ion source and its fittings for leaks. Clean the ion source components regularly to keep the ion source in good working condition.

CAUTION: Potential System Damage. Use only the recommended cleaning methods and materials to avoid damaging the equipment.

Required Materials

- 1/4 inch open-ended wrench
- Flat-bladed screwdriver
- MS-grade methanol
- HPLC-grade deionized water
- Safety glasses
- Breathing mask and filter
- Powder-free gloves (nitrile or neoprene is recommended)
- Lab coat

Clean the Ion Source Surfaces



WARNING! Electrical Shock Hazard. Remove the ion source from the mass spectrometer before starting this procedure. Follow all electrical safe work practices.

Clean the surfaces of the ion source after a spill or when they become dirty.

1. Remove the ion source from the mass spectrometer.
2. Wipe the surfaces of the ion source with a soft, damp, cloth.

Clean the Probes

Flush the ion source periodically, regardless of the type of compounds sampled. Do this by setting up a method in the Analyst[®] software specifically for performing a flushing operation.

1. Change to a mobile phase that is 1:1 water:acetonitrile or 1:1 water:methanol.
2. Adjust the position of the probe so that it is as far from the orifice as possible.
3. In the Analyst[®] software do the following:
 - a. Set **TEM** between **500** and **600**.
 - b. Set **GS1** and **GS2** to at least **40**.
 - c. Set **CUR** to the highest setting possible.
 - d. Wait until the **TEM** setpoint is reached.
4. Inject mobile phase through the tubing and probe at 1 mL/min for 10 minutes to 15 minutes.
5. Make sure that the probe and sample tubing are flushed thoroughly.

Remove the Ion Source

Note: An additional 5.3 L/min of nitrogen flows when the mass spectrometer is off or the ion source is removed from the system. To minimize nitrogen gas consumption and to keep the mass spectrometer clean when it is not in use, leave the ion source installed on the mass spectrometer and leave the system on.

The ion source can be removed quickly and easily, without tools. Always remove the ion source from the mass spectrometer before performing any maintenance on the ion source or exchanging the probes.

1. Stop any ongoing scans.
2. Turn off the sample stream.
3. Type **0** in the **TEM** field, if the heaters are in use.
4. Let the ion source cool for at least 30 minutes.
5. Disconnect the sample tubing from the grounding union.
6. Turn the two source latches upward to the 12 o'clock position to release the ion source.
7. Pull the ion source gently away from the vacuum interface.
8. Put the ion source on a clean, secure surface.

Remove the Probe



WARNING! Electrical Shock Hazard. Remove the ion source from the mass spectrometer before starting this procedure. Follow all electrical safe work practices.

CAUTION: Potential System Damage. Do not let the protruding electrode tip or the corona discharge needle touch any part of the ion source housing, to avoid damaging the probe.

The probe can be removed quickly and easily, without tools. Always remove the ion source from the mass spectrometer before changing probes or performing maintenance on the probe.

Prerequisite Procedures

- [Remove the Ion Source on page 29](#)

1. Loosen the sample tubing nut and then disconnect the sample tubing from the probe.
2. Loosen the retaining ring that secures the probe on the ion source housing.
3. Gently pull the probe straight up out of the tower.
4. Put the probe on a secure, clean surface.

Replace the Electrode Tube



WARNING! Electrical Shock Hazard. Remove the ion source from the mass spectrometer before starting this procedure. Follow all electrical safe work practices.



WARNING! Puncture Hazard. Be careful when handling the electrode tube. The tip of the electrode is extremely sharp.

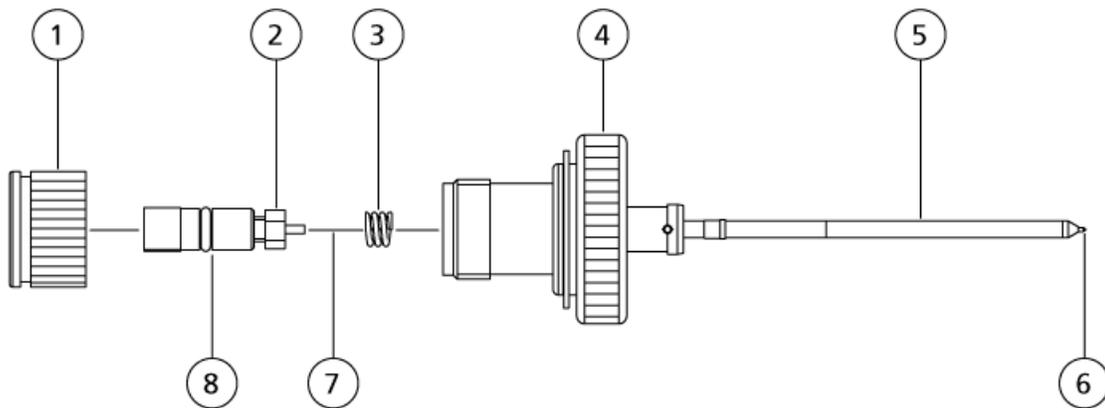
The probe contains an electrode tube. Replace the electrode tube when there is a decrease in performance.

Prerequisite Procedures

- [Remove the Ion Source on page 29](#)
- [Remove the Probe on page 30](#)

1. Remove the electrode adjustment nut.
2. Holding the probe with the tip pointing downwards, so that the spring remains inside the probe, pull the PEEK union and the attached electrode tube from the probe.

Figure 4-1 Probe, Expanded View



Item	Description
1	Electrode adjustment nut
2	1/4 inch retaining nut
3	Spring
4	Retaining ring
5	Sprayer tube
6	Electrode tip
7	Electrode tube
8	PEEK union

3. Use the 1/4 inch open-ended wrench to remove the retaining nut that holds the electrode tube in the PEEK union.

Ion Source Maintenance

4. Remove the electrode tube from the retaining nut.
5. Insert the new electrode tube into the retaining nut and then into the PEEK union.

Make sure that the electrode tube is inserted as far into the PEEK union as it will go. If there is a gap between the electrode tube and its seat inside the union, a dead volume may occur.

6. Tighten the retaining nut.

Do not cross-thread or over-tighten the retaining nut or the tubing might leak.

7. Make sure that the spring is still inside the probe and then tighten the electrode adjustment nut.
8. Align the electrode tube with the narrow opening in the sprayer tube and then insert the PEEK union and attached electrode tube into the probe. Be careful not to bend the electrode tube.
9. Install the probe. Refer to [Install the Probe on page 14](#).
10. Connect the sample tubing. Refer to [Connect the Ion Source Tubing on page 17](#)
11. Install the ion source on the mass spectrometer. Refer to [Ion Source Installation on page 13](#).
12. Adjust the electrode tip extension. Refer to [Adjust the Electrode Tip Extension on page 32](#).

Adjust the Electrode Tip Extension



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Make sure that the electrode protrudes beyond the tip of the probe, to prevent hazardous vapors from escaping from the source. The electrode must not be recessed within the probe.

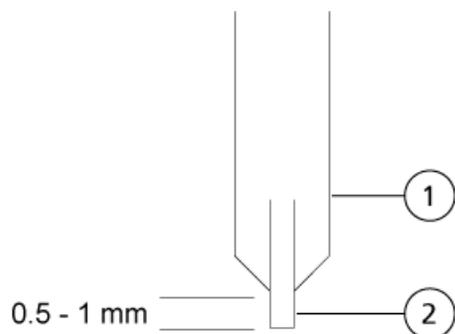


WARNING! Puncture Hazard. Be careful when handling the electrode tube. The tip of the electrode is extremely sharp.

Adjust the electrode tip extension for best performance. The optimal setting is compound-dependent. The distance that the electrode tip protrudes affects the shape of the spray cone, and the shape of the spray cone affects mass spectrometer sensitivity.

- Adjust the black electrode adjustment cap on the top of the probe to extend or retract the electrode tip. The electrode tip should protrude between 0.5 mm and 1.0 mm from the end of the probe.

Figure 4-2 Electrode Tip Extension Adjustment



Item	Description
1	Probe
2	Electrode

Clean the UV Optical Window



WARNING! Electrical Shock Hazard. Make sure that the ion source is completely disconnected from the mass spectrometer before proceeding.



WARNING! Hot Surface Hazard. Let the ion source cool for at least 30 minutes before starting any maintenance procedures. Surfaces of the ion source and the vacuum interface components become hot during operation.



WARNING! Toxic Chemical Hazard. Wear personal protective equipment, including a laboratory coat, gloves, and safety glasses, to avoid skin or eye exposure.



WARNING! Radiation Hazard, Biohazard, or Toxic Chemical Hazard. In the event of a chemical spill, review product Safety Data Sheets for specific instructions. Stop the spill only if it is safe to do so. Make sure that the system is in Standby mode before cleaning a spill near the ion source. Use appropriate personal protective equipment and absorbent wipes to contain the spill and dispose of it following local regulations.



WARNING! Puncture Hazard, Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Discontinue use of the ion source if the ion source window is cracked or broken and contact an SCIEX Field Service Employee. Any toxic or injurious materials introduced into the equipment will be present in the ion source and exhaust output. Dispose of sharps following established laboratory safety procedures.

CAUTION: Potential System Damage. Use powder-free gloves to handle the UV lamp. Fingerprints, under the high temperatures and strong UV intensity that come from operating the UV lamp, form corrosive compounds that etch the surface of the UV lamp and can cause it to break when it is turned on. When handling the UV lamp, keep the UV optical window dry and protect it from abrasion.

Use the following procedure to clean the UV optical window if it has become contaminated.

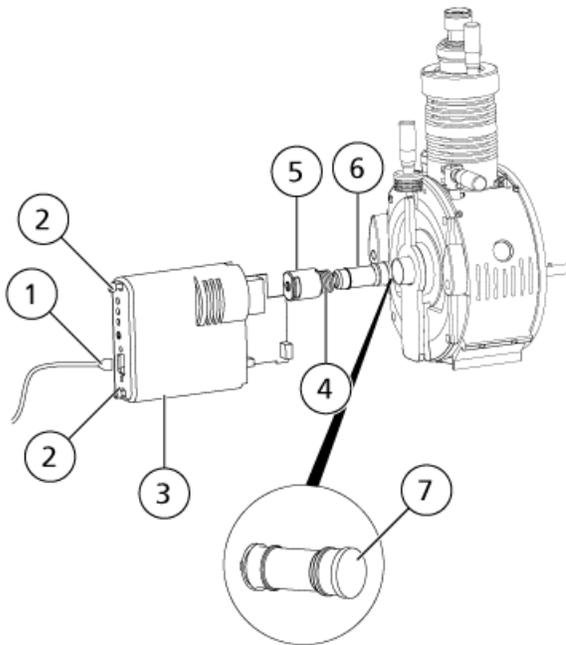
Prerequisite Procedures

- [Remove the Ion Source on page 29](#)

1. Remove both clamping screws and then disconnect the source cable from the 9-pin connector on the UV lamp control module. Refer to [Figure 4-3](#).
2. Remove the two 4 mm hex-head bolts.
3. Remove the UV lamp control module assembly from the ion source.
4. Push and turn the UV lamp holder, and then remove it from the ion source.
5. Remove the UV lamp bulb from the ion source.

Note: Handle the UV bulb with care to avoid breakage.

Figure 4-3 UV Lamp Components



Item	Description
1	Source cable to the mass spectrometer
2	4 mm screws

Ion Source Maintenance

Item	Description
3	Lamp control module
4	Compression spring
5	UV lamp
6	Lamp holder
7	UV optical window

- Using lint-free wipes or foam swabs, wipe the UV optical window with a 1:1 methanol:water mixture.
- When the UV optical window is clean, replace the UV lamp in the ion source.
- Twist the UV lamp holder into the ion source to secure the UV lamp bulb.
- Replace the two 4 mm hex-head bolts.
- Install the UV lamp control module assembly on the ion source.
- Connect the source cable to the 9-pin connector on the UV lamp control module.
- Thread in both clamping screws and then tighten them until finger-tight.
- Install the ion source on the mass spectrometer. Refer to [Ion Source Installation on page 13](#).

Replace the UV Lamp Bulb



WARNING! Electrical Shock Hazard. Remove the ion source from the mass spectrometer before starting this procedure. Follow all electrical safe work practices.



WARNING! Hot Surface Hazard. Let the ion source cool for at least 30 minutes before starting any maintenance procedures. Surfaces of the ion source and the vacuum interface components become hot during operation.

CAUTION: Potential System Damage. Use powder-free gloves to handle the UV lamp. Fingerprints, under the high temperatures and strong UV intensity that come from operating the UV lamp, form corrosive compounds that etch the surface of the UV lamp and can cause it to break when it is turned on. When handling the UV lamp, keep the UV optical window dry and protect it from abrasion.

Prerequisite Procedures

- [Remove the Ion Source on page 29](#)

The UV lamp bulb may need to be replaced after several months of continuous operation. Replace the bulb if it does not turn on when the user initializes the ion source. For troubleshooting information for the UV lamp, refer to [Troubleshooting Tips on page 39](#).

1. Remove both clamping screws and then disconnect the source cable from the 9-pin connector on the UV lamp control module. Refer to [Figure 4-3 on page 35](#).
2. Remove the two 4 mm hex-head bolts.
3. Remove the UV lamp control module assembly from the ion source.
4. Push and turn the UV lamp holder, and then remove it from the ion source.

Note: Handle the UV bulb with care to avoid breakage.

5. Replace the UV lamp.
6. Twist the UV lamp holder into the ion source to secure the UV lamp bulb.
7. Replace the two 4 mm hex-head bolts.
8. Install the UV lamp control module assembly on the ion source.
9. Connect the source cable to the 9-pin connector on the UV lamp control module.
10. Thread in both clamping screws and then tighten them until finger-tight.
11. Install the ion source on the mass spectrometer. Refer to [Ion Source Installation on page 13](#).

Replace the Sample Tubing



WARNING! Electrical Shock Hazard. Remove the ion source from the mass spectrometer before starting this procedure. Follow all electrical safe work practices.

Ion Source Maintenance

Use the following procedure to replace the sample tubing if it has a blockage.

Prerequisite Procedures
<ul style="list-style-type: none">• Stop the sample flow and make sure that any remaining gas has been removed through the source exhaust system.• Remove the Ion Source on page 29

1. Disconnect the sample tubing from the probe and the grounding union.
2. Replace the sample tubing with the same length of tubing used previously.
3. Install the ion source. Refer to [Ion Source Installation on page 13](#).
4. Start the sample flow.

Troubleshooting Tips

5

Symptom	Possible Cause	Corrective Action
UV lamp is in Check mode.	A short-circuit has occurred in PCB or UV lamp.	Make sure that the PCB tab is connected properly to the UV lamp spring or washer. Replace the UV lamp. Refer to Replace the UV Lamp Bulb on page 36 . If unable to resolve the issue, contact an FSE.
	The UV lamp control module unit is faulty.	Contact an FSE.
UV lamp stays in Ignition mode for more than 5 seconds after the mass spectrometer ready.	The control signal is missing. A source cable might be faulty.	Verify that source cable is correctly connected. If so, replace cable.
	The mass spectrometer is not ready.	Make sure that the mass spectrometer is ready. Restart the software.
	A latch might be open.	Verify that the latches are closed. Refer to Ion Source Sense Circuit on page 11 .
	There is a bad electrical contact in UV lamp housing.	<ol style="list-style-type: none"> 1. Remove the UV lamp. 2. Inspect for corrosion on the UV lamp seating or on the top electrode of the UV lamp. 3. Inspect the electrical connections to the UV lamp. 4. Reinstall the UV lamp. Refer to Replace the UV Lamp Bulb on page 36 .
	There is no UV lamp in the UV lamp control module.	Insert a UV lamp in the UV lamp control module.
	The UV lamp is not functioning.	Replace the UV lamp.

Troubleshooting Tips

Symptom	Possible Cause	Corrective Action
	The UV lamp control module unit is faulty.	Contact an FSE.
The lamp is in Ready mode, but no signal appears (flat line).	The ion transfer voltage is set below 500 V (in absolute value).	Set the Ion Transfer Voltage (IS) to a value greater than 500 V (in absolute value).
	The UV optical window is dirty.	Clean the UV optical window. Refer to Clean the UV Optical Window on page 34 .
More than one light is flashing.	The UV lamp bulb is defective.	Replace the UV lamp. Refer to Replace the UV Lamp Bulb on page 36 .
	The UV lamp control module is faulty.	Contact an FSE.
Signal is low.	The dopant supply system is faulty.	Verify the flow of dopant delivered to the dopant inlet tee.
	The Declustering Potential (DP) is not optimized.	Optimize DP to achieve the best signal or signal-to-noise ratio. The optimum values might be different from those found using other ion sources.
	The ion source UV lamp is not positioned properly.	Optimize the vertical position of the UV lamp.
	The APCI electrode is dirty or clogged.	Replace the electrode. Refer to Replace the Electrode Tube on page 30 .

Principles of Operation—Ion Source

A

Ionization Process

The basis for past incompatibilities of LC with mass spectrometry arises from difficulties of converting relatively involatile molecules (solvated in a liquid) into a molecular gas without inducing excessive decomposition. APCI and electrospray ionization (ESI) are two common methods of soft ionization.

The PhotoSpray[®] ion source is an evolution of the APCI technique. A UV lamp is used to initiate the ionization process, instead of the corona discharge used in the APCI.

Samples introduced through the heated nebulizer probe are sprayed with the aid of a nebulizing gas into the ionization region. Within the probe, the finely dispersed droplets of sample and solvent undergo a rapid vaporization with minimal thermal decomposition.

A dopant, usually toluene, is also vaporized in the nebulizer gas within the source chamber. When the vaporized mixture enters the ionization region, the UV radiation from the lamp ionizes the dopant molecules. The gaseous sample and solvent molecular ions are attracted from the ionization region by the potential difference between the probe and the curtain plate. Refer to [Figure A-2](#).

The photoions, formed in large quantity from the dopant, initiate a cascade of ion-molecule reactions involving solvent molecules. These ion-molecule reactions ultimately lead to the formation of the ionized analytes in the form $[M^+H]^+$ (by proton transfer) or $[M]^+$ (by charge exchange), depending on the nature of the analyte molecule. In negative ion mode, $[M^-H]^-$ ions are formed by proton abstraction.

The high collision rate that takes place at atmospheric pressure causes the charge on the photoion to be transferred efficiently to the analyte, if thermodynamic parameters are favorable. In general, proton transfer reactions tend to predominate in the PhotoSpray[®] ion source.

Thermodynamic ionization data for the most common organic compounds can be obtained from the NIST web site at <http://webbook.nist.gov/chemistry> (free of charge at time of publication).

Dopant Selection

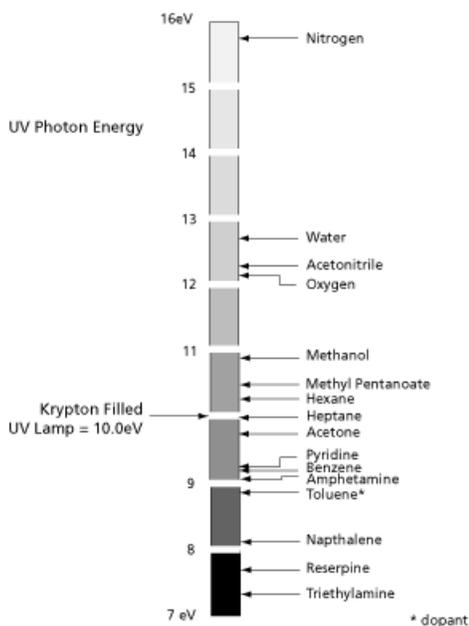
CAUTION: Potential System Damage. Do not use acetone as a dopant. It has the potential to cause deterioration of the gas tubing.

Photoion production is significantly enhanced by adding a dopant to the nebulizing gas (compared to the nebulizing gas without a dopant). The ideal dopant is selected for the following reasons:

Principles of Operation—Ion Source

- Its ability to undergo photoionization because of its favorable ionization energy (just below UV photon energy at 10.0 eV). Refer to [Figure A-1](#).
- The ease with which it can be continuously vaporized in the nebulizer gas stream.
- Its availability in high purity grade (preferably HPLC grade) and low toxicity.

Figure A-1 Ionization Energy Scale



Toluene (ionization energy of 8.83 eV) meets all these requirements, and it is the preferred dopant for most PhotoSpray[®] ion source applications. The relatively high recombination energy and low proton affinity of toluene causes the toluene photoion to react favorably with species present either by charge transfer, or by proton transfer in the ionization region.

Composition of Eluent and Modifiers

The eluent phase composition is an important parameter to consider when developing a method for the PhotoSpray[®] ion source. Immediately following photoionization, the reaction of photoions with solvent molecules opens a reaction channel by forming intermediate charged clusters. These clusters rearrange quickly and form protonated solvent clusters (with the release of deprotonated dopant molecules). In turn, these solvent clusters act as an intermediate step in the ionization of the analyte.

Solvent clusters are characterized by an association number that reflects the nature and structure of the solvent molecule. For example, three molecules of solvent are involved in the protonated clusters formed with a protic solvent like methanol ((CH₃OH)₃ H⁺), and two molecules of solvent in protonated clusters from a dipolar aprotic

solvent like acetonitrile ($(\text{CH}_3\text{CN})_2 \text{H}^+$). Final protonation of the analyte molecules occurs by proton transfer from these clusters.

Eluent

For reverse-phase chromatography, an eluent containing methanol and water provides the best sensitivity for most analytical species. The presence of acetonitrile in the eluent tends to reduce the signal strength for some compounds.

During normal-phase chromatography, when charge-transfer reactions predominate, an all-organic eluent can increase the analytical signal significantly. However, the use of reverse-phase eluents can also offer significant sensitivity gains. A mixture of iso-octane and 2-propanol work well in the PhotoSpray[®] ion source. Chlorinated solvents like methylene chloride and chloroform are also compatible with APPI ionization.

Modifiers

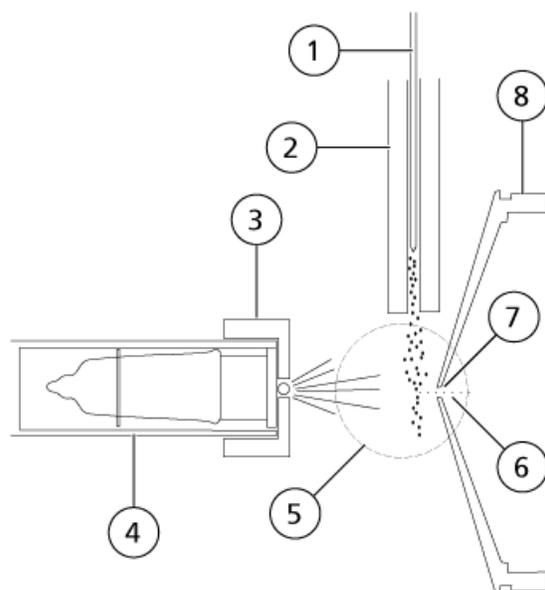
Modifiers such as formic acid, acetic acid, ammonium formate, and ammonium acetate can be used in the PhotoSpray[®] ion source, although they generally do not significantly enhance ion production. Typical concentrations of ammonium formate or ammonium acetate are from 2 mM to 10 mM for positive ions and from 2 mM to 100 mM for negative ions. The concentration of the organic acids is 0.01% to 6.0% by volume.

Other modifiers such as triethylamine, sodium phosphate, trifluoroacetic acid, and sodium dodecyl sulfate are not commonly used, except for specific applications, because they complicate the spectrum with their ion mixtures and clusters. They might also suppress the ion signal of the target compounds

Ionization Region

Figure A-2 shows the primary ionization region, indicated by the dashed circle, in the photoionization source. In this region, the UV light is irradiating from the lamp porthole at a right angle through the nebulizer gas stream and vaporized eluent stream.

Figure A-2 PhotoSpray Ion Source Chamber Cross Section



Item	Description
1	APCI probe
2	Heater
3	Ion deflector
4	UV lamp
5	Ionization region
6	Ions
7	Curtain plate aperture
8	Curtain plate

Photoion Production

Photoions are produced mostly from the dopant molecules in the gas phase of the nebulizer gas stream, from direct exposure to the beam of 10.0 eV photons emitted by the krypton lamp. The photoions (acting as primary ions) react in turn with solvent molecules to produce intermediate charged clusters. The final proton transfer reaction occurs between protonated solvent clusters and sample molecules and results in the formation of analytical ions. Another reaction path leading to formation of odd-electron ions from analyte molecules having both low ionization energy and low proton affinity involves a direct charge-transfer reaction from the photoion.

An equilibrium distribution for certain ions is maintained through collisions in the ionization region. Any excess energy acquired by an ion in the ion-molecule reaction process is thermalized. The formation of product and reactant ions is governed by equilibrium conditions at atmospheric pressure.

Photoion Travel

As the analyte ions are formed in the primary ionization region, they are forced towards the aperture by the application of an offset potential between the PhotoSpray[®] ion deflector and the mass spectrometer curtain plate. The optimum offset potential value is usually a few hundred volts, in absolute value, above that of the curtain plate (500 V relative to ground, the polarity being the same as the ions being analyzed). This is determined by both the nature of the analyte and the distance between the ion deflector and the curtain plate.

The ion source generates a total ion current that is lower than that of APCI. However, this ion source achieves high sensitivity partly because the ions formed in the field-free ionization region can be transferred with high efficiency to the mass spectrometer.

Sample Inlet Description

The sprayer probe consists of 0.012 inch o.d. stainless steel tubing and a sprayer surrounded by a flow of nebulizer gas. The liquid sample flow is pumped through the sprayer where it is nebulized into a ceramic tube that has an embedded heater. When the liquid sample is pumped into the ceramic tube, the sample and solvent are vaporized. The vapor then flows through the ceramic tube into the ionization region in the ion source housing.

The temperature of the heater determines the rate of sample vaporization and consequently the degree of thermal decomposition in the sample. The actual temperature of the sample and solvent does not exceed the vaporization temperature. In other words, there is no significant superheating of the liquid sample. However, increasing the temperature increases the rate of vaporization, which might induce thermal decomposition of the sample ions.

Source Parameters and Voltages

B

APCI Probe Parameters

Table B-1 shows the operating conditions for the ion source. These conditions represent a starting point from which to optimize the ion source. The solvent composition used for optimization was 1:1 water:acetonitrile. By an iterative process, the various settings are optimized using flow injection analysis to obtain the maximum signal-to-noise ratio for the compound of interest.

Table B-1 Parameter Optimization for the APCI Probe

Parameter	Typical Value	Operational Range
Lamp vertical micrometer setting	5	0 to 8.5
Nebulizer gas (GS1)	70*	20 to 90*
Lamp gas (GS2)	10*	10 to 40*
Curtain Gas (CUR)	10 to 20	25 to 50
Ion Transfer Voltage (IS)	1500	600 to 1500
Temperature (°C)	400	350 to 450
Dopant (toluene) (% of total flow)	10	10 to 15
Probe horizontal micrometer setting	5	0 to 10
Probe vertical micrometer setting	2	0 to 13

* If nitrogen is used for the nebulizer and UV lamp gas, rather than zero air, sensitivity improves.

Parameter Descriptions

Parameter	Description
Curtain Gas (CUR)	<p>Controls the flow of gas to the Curtain Gas™ interface. The Curtain Gas interface is located between the curtain plate and the orifice. It prevents ambient air and solvent droplets from entering and contaminating the ion optics, while permitting direction of sample ions into the vacuum chamber by the electrical fields generated between the vacuum interface and the spray needle. Contamination of the ion entrance optics thus reduces Q0 transmission, stability, and sensitivity, and increases background noise.</p> <p>Maintain the Curtain Gas flow as high as possible without losing sensitivity.</p>
Ion Transfer Voltage (IS)	<p>The optimum ion transfer voltage is dependent upon the height of the UV lamp. There is only one optimum ion transfer voltage for a set UV lamp height and only one optimum UV lamp height for a given ion transfer voltage. After changing the UV lamp height, optimize the ion transfer voltage at each new height setting to converge on the best setting for UV lamp height and ion transfer voltage.</p> <p>The values are typically significantly lower than those used with ESI. For example, the ion transfer voltage should be around 700 V to 800 V.</p>
Temperature (TEM)	<p>Heat is used to vaporize the sample and solvent sprayed into the ion source housing. If the temperature is set too low, then the sample vaporization is incomplete and solvent condensation might form in the housing. If the temperature is set too high, then it might induce thermal degradation of the sample.</p> <p>The optimal ion source temperature is affected by:</p> <ul style="list-style-type: none"> • The type of sample. The most significant factor is the solvent composition. The optimal probe temperature decreases as the organic content of the solvent increases. The probe performance might optimize as low as 300 °C with solvents consisting of 100% methanol or acetonitrile. Aqueous solvents consisting of 100% water (at flows of approximately 0.5 mL/min) require a minimum probe temperature of 425 °C. • The quantity of sample. The optimal temperature increases at higher flow rates. The ion source is normally used with sample flow rates of 0.2 mL/min to 0.5 mL/min, but it can be used with flows up to 1.5 mL/min. <p>Perform optimization in increments of 25 °C. The optimal temperature is the lowest setting that results in the complete vaporization of the sample.</p>
Ion Source Gas 1 (GS1)	<p>This gas is used to generate a fine aerosol spray. Optimize the gas setting for signal stability and sensitivity. Typical values range from 20 psi to 90 psi and depend on the sample flow rate. In general, a higher flow rate requires a higher GS1 setting.</p>
Ion Source Gas 2 (GS2)	<p>The UV lamp gas (GS2) is used to protect the optical window of the UV lamp. Typical values range from 10 psi to 40 psi.</p>

Solvent Composition

This section contains information about solvents and modifiers. The ion source is compatible with the mobile phases and volatile buffers typically used in LC-MS/MS, under both reverse phase and normal phase conditions.

The standard concentration of ammonium formate or ammonium acetate is from 2 mmol/L to 10 mmol/L for positive ions and 2 mmol/L to 100 mmol/L for negative ions. The concentration of the organic acids is 0.1% to 6.0% by volume.

Normal phase solvents are often used for non-polar compounds. Polar compounds are more likely to be soluble in the solvents used for reverse phase chromatography. The following chemicals are commonly used as normal phase solvents:

- Iso-octane
- Dichloromethane
- Chloroform

Commonly used solvents are:

- Acetonitrile
- Methanol
- Propanol
- Water

Use chemical modifiers in the solvent to optimize sampling conditions. In general, however, chemical modifiers do not help in the photoionization process.

Commonly used modifiers are:

- Acetic acid
- Formic acid
- Ammonium formate
- Ammonium acetate

The following modifiers are not commonly used because they complicate the spectrum with their ion mixtures and cluster combinations. They might also suppress the strength of the target compound ion signal:

- Triethyl amine (TEA)
- Sodium phosphate
- Trifluoroacetic acid (TFA)
- Sodium dodecyl sulfate

Dopant Composition and Flow Rate

CAUTION: Potential System Damage. Do not use acetone as a dopant. It has the potential to cause deterioration of the gas tubing.

Toluene is the most commonly used dopant. However, other appropriate compounds can also be used. Dopants should be of the highest possible quality, at least HPLC grade. Serious background ion problems occur with lower quality dopants.

In general, the optimum analytical signal is asymptotically approached at a dopant infusion rate corresponding to 10% to 15% of the total flow rate of the mobile phase (without splitting). The typical infusion rate is between 75 $\mu\text{L}/\text{min}$ to 150 $\mu\text{L}/\text{min}$. The value selected during method development is dictated by the signal strength and the required mobile phase flow.

Note: Always use a dopant LC pump. Using a syringe pump greatly reduces the sensitivity of the source. In previous versions of the PhotoSpray[®] ion source, the dopant was introduced in a non-pressurized line, and a syringe pump was sufficient.

Consumables and Spares

C

PN	Description
029031	Compression Spring (for UV lamp bulb). Refer to Replace the UV Lamp Bulb on page 36 .
1008006	Spring Cap (holds UV lamp bulb into ion source housing). Refer to Replace the UV Lamp Bulb on page 36 .
1002998	Photoionization Detector (UV lamp bulb). Refer to Replace the UV Lamp Bulb on page 36 .
1009515	Brass Washer (for UV lamp bulb). Refer to Replace the UV Lamp Bulb on page 36 .
025388	Electrode (for APCI probe). Refer to Replace the Electrode Tube on page 30 .
1009095	APCI Probe Assembly (complete probe). Refer to Install the Probe on page 14 .
1005596	Source Cable. Install the Ion Source Connections on page 15 .

Revision History

Document Number	Reason for Change	Date
D1000044558 A	First release.	April 2005
D1000044558 B	Added specifications.	September 2005
D1000044558 C	Applied new template. Updated for 5500 series of instruments.	October 2008
D1000044558 D	Rebranded.	April 2010
D1000044558 E	Applied new templated and added warnings.	October 2011
D1000044558 F	Updated for 4500 series of instruments.	March 2012
D1000044558 G	Updated for 6500 series of instruments.	July 2012
RUO-IDV-05-1461-A	Applied new template. Updated for the TripleTOF [®] systems. Removed reference to acetone as a dopant.	June 2014
RUO-IDV-05-1461-B	Added Caution about acetone. Removed references to TripleTOF [®] support.	June 2014
RUO-IDV-05-1461-C	Added support for the 6500 ⁺ series of instruments. Rebranded.	August 2015