



Turbo VTM Ion Source

Operator Guide



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AB Sciex Pte. Ltd.
Blk33, #04-06 Marsiling Industrial Estate Road 3
Woodlands Central Industrial Estate, Singapore 739256

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Operational Precautions and Limitations

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Note: Before operating the system, carefully read all of the sections of this guide.

This section contains general safety-related information. It also describes potential hazards and associated warnings for the system and the precautions that should be taken to minimize the hazards.

In addition to this section, refer to [Glossary of Symbols](#) for information about the symbols and conventions used in the laboratory environment, on the system, and in this documentation.

Operational Precautions and Hazards

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



WARNING! Ionizing 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, Ionizing Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Discontinue use of the ion source if the ion source window is cracked or broken and then contact a SCIEX Field Service Employee (FSE). Any toxic or injurious materials introduced into the equipment will be present in the source exhaust output. Exhaust from equipment should be vented from the room. 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 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.

Operational Precautions and Limitations



WARNING! Ionizing Radiation Hazard, Biohazard, Electrical Shock Hazard, or Toxic Chemical Hazard. In the event of a chemical spill, review product Safety Data Sheets for specific instructions. 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 local regulations 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.

Chemical Precautions



WARNING! Ionizing Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Determine whether decontamination is required prior to cleaning or maintenance. The customer must decontaminate the system prior to cleaning or maintenance if radioactive materials, biological agents, or toxic chemicals have been used with the system.



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



WARNING! Biohazard, Toxic Chemical Hazard. Connect the drain tubing to the mass spectrometer and the source exhaust drain bottle properly, to prevent leaks.

- Determine which chemicals have been used in the system prior to service and regular maintenance. Refer to the *Safety Data Sheets* for the health and safety precautions that must be followed with chemicals. SCIEX *Safety Data Sheets* can be found at sciex.com/tech-regulatory.
- Always wear assigned personal protective equipment, including powder-free neoprene or nitrile gloves, safety glasses, and a laboratory coat.

- Work in a well-ventilated area or fume hood.
- Avoid ignition sources when working with flammable materials, such as isopropanol, methanol, and other flammable solvents.
- Take care in the use and disposal of any chemicals. Potential risk of personal injury if proper procedures for handling and disposing of chemicals are not followed.
- Avoid skin contact with chemicals during cleaning and wash hands after use.
- Make sure that all exhaust hoses are connected properly and that all connections are functioning as designed.
- Collect all spent liquids and dispose of them as hazardous waste.
- Comply with all of the local regulations for the storage, handling, and disposal of biohazardous, toxic, or radioactive materials.

System Safe Fluids

The following fluids can safely be used with the system.

CAUTION: Potential System Damage. Do not use any other fluid until confirmation is received from SCIEX that it does not present a hazard. This is not an exhaustive list.

- **Organic Solvents**
 - MS-grade acetonitrile; up to 100%
 - MS-grade methanol; up to 100%
 - Isopropanol; up to 100%
 - HPLC-grade or higher water; up to 100%
 - Tetrahydrofuran; up to 100%
 - Toluene and other aromatic solvents; up to 100%
 - Hexanes; up to 100%
- **Buffers**
 - Ammonium acetate; less than 1%
 - Ammonium formate; less than 1%
 - Phosphate; less than 1%

Operational Precautions and Limitations

- **Acids and Bases**
 - Formic acid; less than 1%
 - Acetic acid; less than 1%
 - Trifluoroacetic acid (TFA); less than 1%
 - Heptafluorobutyric acid (HFBA); less than 1%
 - Ammonia/ammonium hydroxide; less than 1%
 - Phosphoric acid; less than 1%
 - Trimethylamine; less than 1%
 - Triethylamine; less than 1%

Laboratory Conditions

Operating Conditions

The system is designed to operate safely under these conditions:

- Indoors
- Altitude: Up to 2 000 m (6 560 feet) above sea level
- Ambient temperature: 5 °C (41 °F) to 40 °C (104 °F)
- Maximum relative humidity: 80% for temperatures up to 31 °C (88 °F), decreasing linearly to 50% at 40 °C (104 °F)
- Mains supply voltage fluctuations: $\pm 10\%$ of the nominal voltage
- Transient overvoltages: Up to the levels of Overvoltage Category II
- Temporary overvoltages on the mains supply
- Pollution degree: Pollution Degree 2

Performance Specifications

The system is designed to meet specifications under these conditions:

- An ambient temperature of 15 °C to 30 °C (59 °F to 86 °F)
Over time, the temperature must remain within a range of 4 °C (7.2 °F), with the rate of the change in temperature not exceeding 2 °C (3.6 °F) per hour. Ambient temperature fluctuations exceeding the limits might result in mass shifts in spectra.
- Relative humidity from 20% to 80%, non-condensing

Equipment Use and Modification



WARNING! Electrical Shock Hazard. Do not remove the covers. Removing the covers might cause injury or malfunctioning of the system. The covers need not be removed for routine maintenance, inspection, or adjustment. Contact a SCIEX Field Service Employee (FSE) for repairs that require the covers to be removed.



WARNING! Personal Injury Hazard. Use SCIEX-recommended parts only. Use of parts not recommended by SCIEX or use of parts for any purpose other than their intended purpose can put the user at risk of harm or negatively impact system performance.

Use the mass spectrometer and ion source indoors in a laboratory that complies with the environmental conditions recommended in the *Site Planning Guide* for the mass spectrometer.

If the mass spectrometer and ion source are used in an environment or in a manner not prescribed by the manufacturer, then the protection provided by the equipment might be impaired.

Unauthorized modification or operation of the mass spectrometer and ion source might cause personal injury and equipment damage, and might void the warranty. Erroneous data might be generated if the mass spectrometer and ion source is operated either above or below the recommended environmental conditions or operated with unauthorized modifications. Contact an FSE for information on servicing the system.

The Turbo V™ ion source can be used for either electrospray ionization (ESI) or atmospheric pressure chemical ionization (APCI).

The TurbolonSpray® probe is used for ESI mode operation. The APCI probe is used for APCI mode operation.

Applications for the ion source include qualitative method development and qualitative and quantitative analysis.

Ionization Modes

ESI Mode

ESI produces gas phase ions of analytes in a sample by applying a high voltage to the sample effluent flowing through a needle. With the aid of heated gas flow, ESI produces singly and multiply charged ions in a relatively mild condition so that it is suitable for a wide range of compounds including small molecules, such as drugs or pesticides, and larger molecules, such as peptides, proteins and other biopolymers. The sensitivity depends on the chemical properties of the analyte, the gas flow rate, the temperature, the voltage, and the mobile phase composition.

The ESI technique is mild enough to be used with labile compounds, such as peptides, proteins, and thermally labile pharmaceuticals. It functions with flow rates from 5 µL/min to 3000 µL/min and it vaporizes 100% aqueous to 100% organic solvents.

Refer to [Electrospray Ionization Mode](#).

APCI Mode

The APCI mode is suitable for:

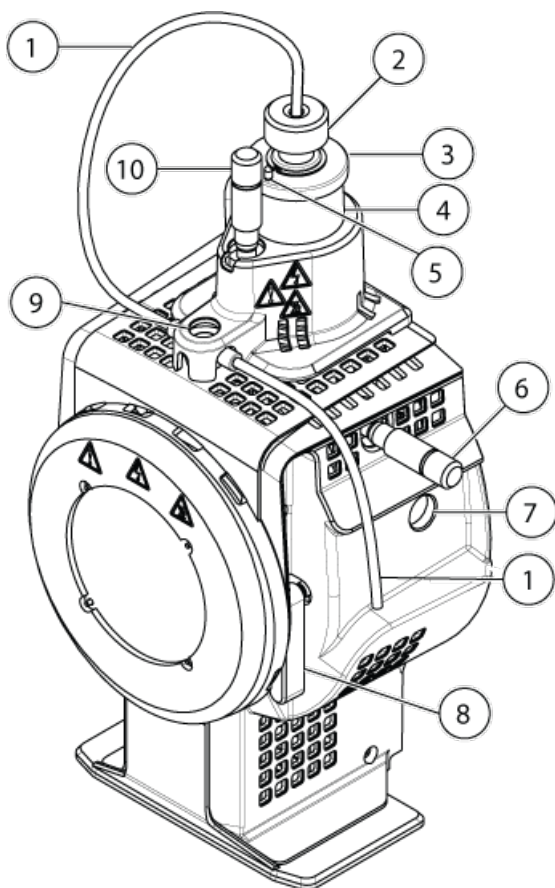
- Ionization of compounds that do not readily form ions in solution. These are usually non-polar compounds.
- Creation of simple APCI spectra for LC-MS/MS experiments.
- High-throughput analyses of complex and dirty samples. It is less sensitive to ion suppression effects.
- Rapid sample introduction by flow injection with or without an LC column.

The APCI technique can be used for volatile and labile compounds with minimal thermal decomposition. The rapid desolvation and vaporization of the droplets and entrained analyte minimizes thermal decomposition and preserves molecular identity for ionization by the corona discharge needle. Buffers are readily tolerated by the ion source without significant contamination and the flash vaporization of the sprayed effluent allows up to 100% water to be used. The probe can accept the entire effluent, without splitting, at flow rates from 200 µL/min to 3000 µL/min (through a wide-bore column).

Refer to [APCI Mode](#).

Ion Source Components

Figure 2-1 Ion Source Components



Item	Description	Primary Materials
1	Sample tubing from a sample supply device	Red PEEK tubing
2	Electrode adjustment nut	Stainless steel
3	Retaining ring	Stainless steel
4	Probe tower	Stainless steel
5	Corona discharge needle adjustment screw	PEEK tubing

Ion Source Overview

Item	Description	Primary Materials
6	Micrometer used to position the probe on the horizontal axis for ion source sensitivity adjustments	Glass
7	Window port	Stainless steel
8	One of two source latches that secure the ion source to the mass spectrometer	Stainless steel
9	Grounding union. It is located below the ion source cover.	Stainless steel
10	Micrometer used to position the probe on the vertical axis for ion source sensitivity adjustments	Aluminum bronze

Probes

The TurbolonSpray[®] and APCI probes provide a range of capability for testing samples. Select the probe and method most suitable for the compounds in the sample.

Table 2-1 Ion Source Specifications

Specification	TurbolonSpray [®] Probe	APCI Probe
Temperature range	From ambient temperature to 750 °C, depending on liquid flow	From 50 °C to 750 °C, depending on liquid flow
Liquid flow inlet	40 µL/min to 1000 µL/min	200 µL/min to 2000 µL/min
Gas 1 / Gas 2	Refer to the <i>Site Planning Guide</i> for the mass spectrometer.	

The software for the mass spectrometer identifies the installed probe and enables the corresponding user controls. All of the data acquired using the ion source is identified with an abbreviation representing the probe used to acquire the data (TIS for the TurbolonSpray[®] probe and HN for the APCI probe).

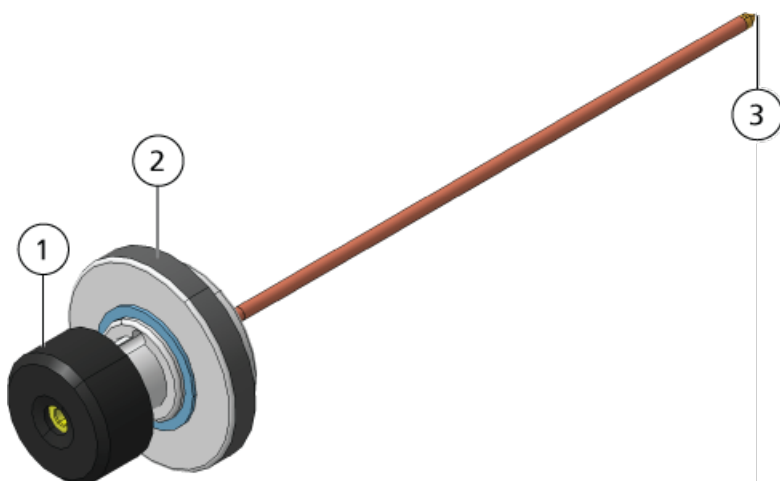
TurbolonSpray[®] Probe

The TurbolonSpray[®] probe consists of 0.012 inch outside diameter (o.d.) stainless steel tubing. It is located centrally with the two turbo heaters placed at a 45 degree angle to each side. Samples introduced through the TurbolonSpray[®] probe are ionized within the tubing, by the application of high voltage (IonSpray[™] voltage). Then they are nebulized by a jet of hot, dry, ultrahigh purity (UHP) nitrogen gas from the turbo heaters, creating a mist of small, highly-charged droplets. The combination of the IonSpray[™] effluent and the heated dry gas from the turbo sprayer is projected at a 90 degree angle to the ion path. Refer to [Principles of Operation — Ion Source](#).



WARNING! Puncture Hazard. Be careful when handling the electrode. The tips of the electrodes are extremely sharp.

Figure 2-2 Parts of the TurbolonSpray[®] Probe



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

APCI Probe

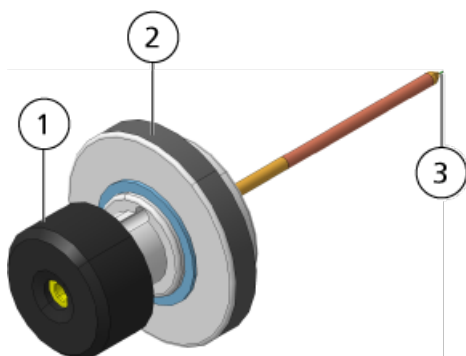
The APCI probe consists of 100 μm inside diameter (i.d.) (0.004 inch) stainless steel tubing surrounded by a flow of nebulizer gas (Gas 1). The liquid sample stream is pumped through the sprayer, where it is nebulized in a ceramic tube containing a heater. The inside wall of the ceramic tube can be maintained at a temperature range of 100 $^{\circ}\text{C}$ to 750 $^{\circ}\text{C}$ 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](#).



WARNING! Puncture Hazard. Be careful when handling the electrode. The tips of the electrodes are extremely sharp.

Figure 2-3 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

Gas and Electrical Connections

Gas and low- 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.

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.
- A turbo heater has failed.

- The ion source has over-heated.

Source Exhaust System



WARNING! Ionizing Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Make sure that the source exhaust system is connected and functioning, to safely remove sample vapor exhaust from the laboratory environment. Emissions from the equipment must be exhausted into the general building exhaust and not allowed to exhaust into the workspace of the laboratory. For requirements for the source exhaust system, refer to the *Site Planning Guide*.



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



WARNING! Ionizing Radiation Hazard, Biohazard, or Toxic Chemical Hazard. If an LC system is used with the mass spectrometer, and if the source exhaust system is not functioning properly, then shut down the LC system until functionality of the source exhaust system has been restored.



WARNING! Fire Hazard. Do not direct more than 3 mL/min of flammable solvent into the ion source. Exceeding the maximum flow rate can cause solvent to accumulate in the ion source. Do not use the ion source if the source exhaust system is not enabled and functioning when the ion source and the probe are properly installed.

Note: Make sure that all of the exhaust tubing is securely connected to reduce the chance of equipment exhaust entering the room.

An ion source produces 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 does not operate unless the source exhaust system is operating.

Ion Source Overview

A vacuum switch installed in the source exhaust sense circuit measures the vacuum in the source. If the vacuum in the source rises above the set point while the probe is installed, then 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 information about the ventilation requirements for the source exhaust system, refer to the *Site Planning Guide*.

Note: The exhaust system should be periodically inspected to make sure that the exhaust line is intact and that no exhaust is leaking into the room.

Ion Source Installation

3



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 windows on the side and front of the ion source.

When the ion source is installed, the software recognizes the ion source and shows the ion source identification.

CAUTION: Potential System Damage. Do not lift or carry the ion source with one hand. The ion source is designed to be lifted or carried using the molded grips on each side of the ion source.

Required Materials

- Ion source
- TurbolonSpray[®] probe
- (Optional) APCI probe
- Red PEEK tubing (0.005 inch bore)

Prepare for Installation



WARNING! Puncture Hazard. Be careful when handling the electrode. 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 electrode adjustment nut on the probe to move the electrode tip inside the electrode tube.

For optimum stability and performance, the electrode tip should extend between 0.5 mm and 1.0 mm from the end of the probe. Refer to [Optimize the TurbolonSpray® Probe Position](#) or [Optimize the APCI Probe Position](#).

Install the Probe

Prerequisite Procedures

- [Remove the Ion Source](#)



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



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

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.

CAUTION: Potential System Damage. Make sure that the corona discharge needle tip is turned away from the aperture if the TurbolonSpray® probe is in use.

The probe is not pre-installed in the ion source. Always remove the ion source from the mass spectrometer before exchanging probes.

Note: 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. Make sure that the corona discharge needle tip is pointed away from the curtain plate aperture. Refer to [Adjust the Position of the Corona Discharge Needle](#).
2. Insert the probe into the tower. Align the hole on the probe with the corona discharge needle adjustment screw at the top of the ion source. Refer to [Ion Source Components](#).
3. Gently push the probe down until the contacts engage with those in the tower.
4. 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 finger-tight.
5. For the APCI probe only, make sure that the corona discharge needle tip is pointed toward the curtain plate aperture. Refer to [Adjust the Position of the Corona Discharge Needle](#).

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! Ionizing 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](#).

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



WARNING! Pinching Hazard. When installing the ion source, be careful not to pinch fingers between the ion source and the vacuum interface.

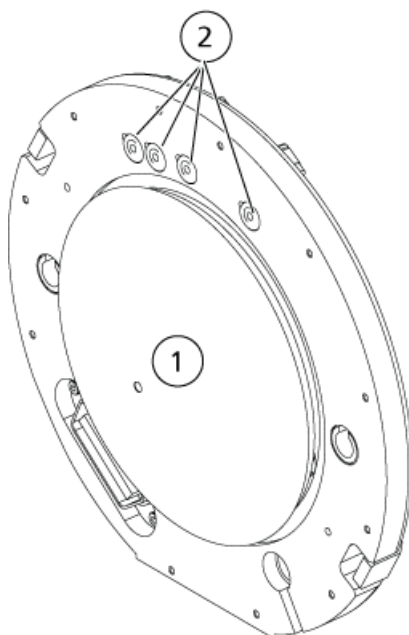
CAUTION: Potential System Damage. Do not let the protruding electrode tip or the corona discharge needle touch any part of the ion source housing, to avoid damaging the probe.

Note: 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.

Prerequisites

- Make sure that all of the O-rings are present on the vacuum interface.

Figure 3-1 O-rings on the Vacuum Interface



Item	Description
1	Curtain plate
2	O-rings

1. Make sure that the ion source latches on either side of the ion source are pointing up in the 12 o'clock position. Refer to [Ion Source Components](#).
2. Align the ion source with the vacuum interface, making sure that the guide pins 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.

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

4. Connect the red PEEK tubing from the sample supply device to the other side of the grounding union on the ion source.

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 of the connections are tight enough to prevent leaks. Do not over-tighten.

Inspect for Leaks



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

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

Ion Source Optimization

4



WARNING! Ionizing 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 flammable solvent into the ion source. Exceeding the maximum flow rate can cause solvent to accumulate in the ion source. Do not use the ion source if the source exhaust system is not enabled and functioning when the ion source and the probe are properly installed.



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

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

When optimizing ion source-dependent parameters, introduce the sample at a flow rate that will be used during sample analysis, using either Flow Injection Analysis (FIA) or Tee-infusion as the method of sample introduction. Optimize the position of the ion source before optimizing the ion source-dependent parameters.

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, and gas and voltage parameters to maximize the signal-to-noise ratio and signal stability.

Refer to [ESI Mode](#) or [APCI Mode](#).

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, through a syringe pump, 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 only be used for ion path optimization and MS/MS fragment selection.

The liquid sample stream is delivered to the ion source by an LC pump. The sample can be injected directly into the mobile phase using flow injection analysis (FIA) or tee infusion, through a syringe pump (not supplied), or through a separation column using a loop injector or autosampler.

Flow Rate

Sample flow rates are determined by the LC system or syringe pump. The TurbolonSpray[®] probe supports flow rates from 5 $\mu\text{L}/\text{min}$ to 3000 $\mu\text{L}/\text{min}$. The APCI probe supports flow rates from 50 $\mu\text{L}/\text{min}$ to 3000 $\mu\text{L}/\text{min}$.

TurbolonSpray[®] Probe Optimization



WARNING! Ionizing Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Make sure that the source exhaust system is connected and functioning 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! Fire Hazard. Do not direct more than 3 mL/min of flammable solvent into the ion source. Exceeding the maximum flow rate can cause solvent to accumulate in the ion source. Do not use the ion source if the source exhaust system is not enabled and functioning when the ion source and the probe are properly installed.



WARNING! Ionizing 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.

Ion Source Optimization

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

Note: To keep the system clean and at its optimum performance, adjust the probe position when changing the flow rate.

Tip! It is easier to optimize signal and signal-to-noise with flow injection analysis than with on-column injections.

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.

Flow Rate and Ion Source Temperature

The sample introduction flow rate and the sample solvent composition affect the optimal TurbolonSpray® probe temperature. A higher flow rate or a higher aqueous content have a higher optimal temperature.

The TurbolonSpray® probe is often used with sample flow rates of 5 µL/min to 1000 µL/min. The heat is used to increase the rate of evaporation which improves ionization efficiency, resulting in increased sensitivity. Extremely low flow rates of high organic solvent usually do not require increased temperatures. Refer to [Source Parameters and Voltages](#).

Set Up the System

1. Configure the HPLC pump to deliver the mobile phase at the required flow rate. Refer to [Source Parameters and Voltages](#).
2. Connect the grounding union on the ion source to an HPLC pump, through an injector equipped with a 5 µL loop, or to an autosampler.
3. If an autosampler is being used, then configure the autosampler to perform multiple injections.

Prepare the System

1. Start the Analyst® or Analyst® TF software.
2. On 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 the sample flow and sample injection.

Set the Starting Conditions

1. In the Tune Method Editor, make sure that the correct **Scan Type** and appropriate compound parameters are selected.
2. On the Source/Gas tab, type a starting value for **Ion Source Gas 1 (GS1)**.

For LC pumps, use a value between 40 and 60 for Gas 1.

3. Type a starting value for **Ion Source Gas 2 (GS2)**.

For LC pumps, use a value between 30 and 50 for Gas 2.

Note: Gas 2 is used with higher flow rates typical with an LC system and in conjunction with increased temperature.

4. In the **IonSpray Voltage (IS)** or **IonSpray Voltage Floating (ISVF)** field, type the value appropriate to the mass spectrometer.

Table 4-1 IS Parameter Values

Mass Spectrometer	Starting Value
3500, 4000, 4500, 5000, 5500, 5500+, 6500, and 6500+ systems	4500
TripleTOF [®] systems	5500

5. In the Curtain Gas (CUR) field, type the value appropriate to the mass spectrometer.

Table 4-2 CUR Parameter Values

Mass Spectrometer	Starting Value
3500, 4000, and 4500 systems	20
5000, 5500, and 5500+ systems	25
6500 and 6500+ systems	30
TripleTOF [®] systems	20 to 25, depending on the flow rate

6. Type **45** in the **Collision Energy** field.
7. Start acquisition.

Optimize the TurbolonSpray[®] Probe Position



WARNING! Ionizing 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. The tip of the electrode is extremely sharp.

After the probe is optimized, it needs only minor adjustment. If the probe is removed, or if the analyte, flow rate, or solvent composition change, then repeat the optimizing procedure.

Refer to [Ion Source Components](#).

1. Look through the window of the ion source housing to view the position of the probe.
2. Monitor the signal or signal-to-noise of the analytes in the Analyst[®] or Analyst[®] TF software.
3. Use the horizontal micrometer to adjust the probe position in small increments to achieve the best signal or signal-to-noise ratio.

The probe can optimize slightly to either side of the aperture.

Tip! Adjust the horizontal micrometer setting to direct the liquid spray from the TurbolonSpray[®] probe away from the aperture to prevent contamination of the aperture; to prevent piercing of the Curtain Gas[™] flow, which can create an unstable signal; and to prevent electrical shorting due to the presence of the liquid.

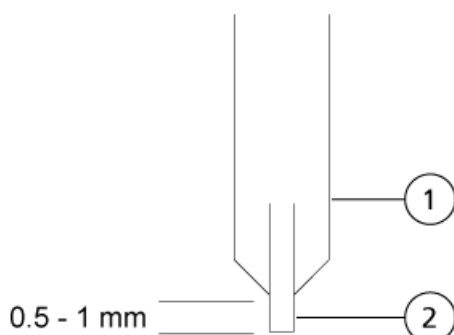
4. Use the vertical micrometer to adjust the probe position in small increments to achieve the best signal or signal-to-noise ratio.

Note: The vertical position of the probe depends on the flow rate. At lower flow rates, the probe should be closer to the aperture. At higher flow rates, the probe should be farther from the aperture.

5. Adjust the black electrode adjustment nut on the probe to move the electrode tube in or out of the probe. Typically, the optimum protrusion of the electrode is 0.5 mm to 1.0 mm beyond the end of the probe.

The optimal setting for the electrode tip 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.

Figure 4-1 Electrode Tip Extension Adjustment



Item	Description
1	Probe
2	Electrode

Optimize Source and Gas Parameters and Voltage

Optimize Ion Source Gas 1 (nebulizer gas) for best signal stability and sensitivity. Ion Source Gas 2 (heater gas) aids in the evaporation of solvent, which helps to increase the ionization of the sample.

Too high a temperature can cause premature vaporization of the solvent at the TurbolonSpray[®] probe tip, especially if the probe protrudes too far, which results in signal instability and a high chemical background noise. Similarly, a high heater gas flow can produce a noisy or unstable signal.

Use the lowest IonSpray[™] voltage possible without losing signal. Focus on signal-to-noise and not just signal. If the IonSpray[™] voltage is too high, then a corona discharge can occur. The discharge is visible as a blue glow at the tip of the TurbolonSpray[®] probe. This will result in decreased sensitivity and stability of the ion signal.

1. Adjust **GS1** and **GS2** in increments of 5 to achieve the best signal or signal-to-noise ratio.
2. Increase the value in the **CUR** field until the signal begins 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.

3. Adjust **IS** or **ISVF** in increments of 500 V to maximize signal-to-noise.

Optimize the Turbo Heater Temperature

The optimal heater temperature is compound-dependent, flow rate-dependent, and mobile phase composition-dependent. The higher the flow rate and the higher the aqueous composition, the higher the optimized temperature.

When optimizing the source temperature, make sure that the ion source equilibrates to the new temperature setting.

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

APCI Probe Optimization



WARNING! Ionizing Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Make sure that the source exhaust system is connected and functioning 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! Fire Hazard. Do not direct more than 3 mL/min of flammable solvent into the ion source. Exceeding the maximum flow rate can cause solvent to accumulate in the ion source. Do not use the ion source if the source exhaust system is not enabled and functioning when the ion source and the probe are properly installed.



WARNING! Ionizing 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 are not controlled by the software, then do not leave the mass spectrometer unattended while in operation. The liquid stream from the HPLC system can flood the ion source when the mass spectrometer goes into Standby mode.

Note: The minimum flow rate supported by the APCI probe is 200 µL/min. Refer to [APCI Probe Parameters](#) for a complete list of APCI probe parameters.

Tip! It is easier to optimize signal and signal-to-noise with flow injection analysis than with on-column injections.

Note: When using the APCI probe, make sure that the corona discharge needle is pointing toward the aperture.

Set Up the System

1. Configure the HPLC pump to deliver the mobile phase at the required flow rate. Refer to [Source Parameters and Voltages](#).
2. Connect the grounding union on the ion source to an HPLC pump, through an injector equipped with a 5 µL loop, or to an autosampler.
3. If an autosampler is being used, then configure the autosampler to perform multiple injections.

Prepare the System

1. Start the Analyst[®] or Analyst[®] TF software.
2. On 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 the sample flow and sample injection.

Set the Starting Conditions

1. In the Tune Method Editor, make sure that the correct **Scan Type** and appropriate compound parameters are selected.
2. Type **30** in the **Ion Source Gas 1 (GS1)** field.
3. In the Curtain Gas (CUR) field, type the value appropriate to the mass spectrometer.

Table 4-3 CUR Parameter Values

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

Table 4-3 CUR Parameter Values (continued)

Mass Spectrometer	Starting Value
6500 and 6500+ systems	30
TripleTOF [®] systems	20 to 25, depending on the flow rate

4. Type **1** in the **Nebulizer Current (NC)** field.
5. On the Compound tab, in the **Declustering Potential (DP)** field, type **100**.
6. Type **45** in the **Collision Energy** field.
7. Start acquisition.

Optimize Gas 1 and Curtain Gas[™] Flow

1. Adjust **GS1** in increments of five to achieve the best signal or signal-to-noise ratio.
2. 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.

Adjust the Position of the Corona Discharge Needle

Required Materials

- Insulated flat-bladed screwdriver



WARNING! Electrical Shock Hazard. Follow this procedure to avoid contact with the high voltages applied to the corona discharge needle, curtain plate, and turbo heaters.

When using the APCI probe, make sure that the corona discharge needle is pointing toward the aperture. When using the TurbolonSpray[®] probe, make sure that the corona discharge needle is pointed away from the aperture.

1. Use an insulated flat-bladed screwdriver to rotate the corona discharge needle adjustment screw on the top of the needle.
2. Look through the glass window to make sure that the needle is aligned with the tip facing the aperture.

Optimize the APCI Probe Position



WARNING! Ionizing 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.

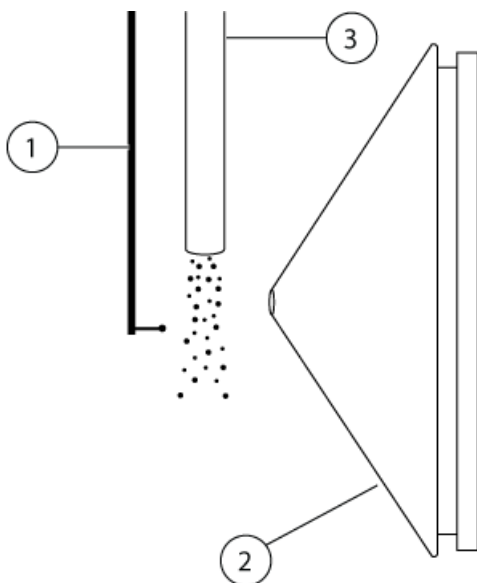


WARNING! Puncture Hazard. Be careful when handling the electrode. The tip of the electrode is extremely sharp.

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 the probe position 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 4-2 Sprayer Nozzle Position



Item	Description
1	Corona discharge needle
2	Curtain plate
3	APCI probe

Ion Source Optimization

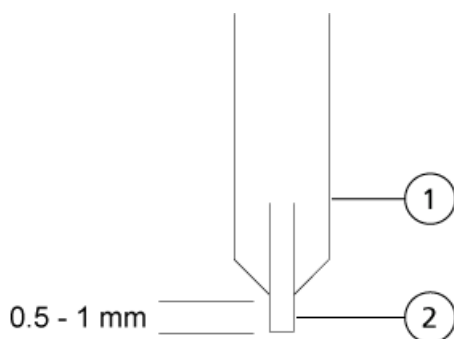
1. Use the previous horizontal and vertical micrometer settings or set them to 5 as a starting position.

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

2. Monitor the signal or signal-to-noise of the analytes in the Analyst® or Analyst® TF software.
3. Use the horizontal micrometer to adjust the probe in small increments to achieve the best signal or signal-to-noise ratio.
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 nut on the probe to move the electrode tube in or out of the probe. The electrode tip should protrude between 0.5 mm and 1.0 mm from the end of the probe.

The optimal setting for the electrode tip 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.

Figure 4-3 Electrode Tip Extension Adjustment



Item	Description
1	Probe
2	Electrode

Optimize the Nebulizer Current

The ion source is controlled by current and not by voltage. Select the appropriate current for the acquisition method, regardless of ion source selection position.

- Start with a **NC** value of 3 and then increase or decrease it to achieve the best signal or signal-to-noise ratio.

The nebulizer current applied to the corona discharge needle usually optimizes between 1 μA and 5 μA in positive mode. If no changes in signal are observed when the current is increased, then leave the current at the lowest value that provides the best signal or signal-to-noise ratio.

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.

Optimization Tips

Optimization of the ion source minimizes the need for cleaning of the ion source and vacuum interface components.

- Use the highest temperature possible when optimizing compounds. A temperature of 700 °C is common for many compounds. High temperatures help keep the ion source clean and reduce background noise.
- Use the highest Curtain GasTM flow rate (CUR) possible without decreasing the signal. This helps to:
 - Prevent penetration of the Curtain GasTM flow, which can produce a noisy signal.
 - Prevent contamination of the aperture.
 - Increase the overall signal-to-noise ratio.
- Adjust the horizontal micrometer setting to direct the liquid spray from the probe away from the aperture to:
 - Prevent contamination of the aperture.
 - Prevent piercing of the Curtain GasTM flow, which can create an unstable signal.
 - Prevent electrical shorting due to the presence of the liquid.To do so, use the vertical micrometer to move the probe up.
- Use the lowest IonSprayTM voltage possible without losing signal. Focus on signal-to-noise and not just signal.

Ion Source Maintenance

5

The following warnings apply to all of the 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 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! Ionizing Radiation Hazard, Biohazard, Electrical Shock Hazard, or Toxic Chemical Hazard. In the event of a chemical spill, review product Safety Data Sheets for specific instructions. 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, Ionizing Radiation Hazard, Biohazard, or Toxic Chemical Hazard. Discontinue use of the ion source if the ion source window is cracked or broken and then contact a SCIEX Field Service Employee (FSE). Any toxic or injurious materials introduced into the equipment will be present in the source exhaust output. Exhaust from equipment should be vented from the room. Dispose of sharps following established laboratory safety procedures.

CAUTION: Potential System Damage. Do not lift or carry the ion source with one hand. The ion source is designed to be lifted or carried using the molded grips on each side of the ion source.

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
- Cleanliness of the samples and sample preparation techniques
- 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

- | |
|--|
| <ul style="list-style-type: none"> • 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 |
|--|

Recommended Maintenance Schedule

The following table provides a recommended schedule for cleaning and maintaining the ion source. Refer to the *Parts and Equipment Guide for Ion Sources and Mass Spectrometers* for a list of consumable and spare parts.

Ion Source Maintenance

Tip! Perform maintenance tasks regularly to make sure that the mass spectrometer is performing optimally.

Contact a Qualified Maintenance Person (QMP) to order consumable parts and for basic service and maintenance requirements. Contact a SCIEX Field Service Employee (FSE) for all other service and maintenance requirements.

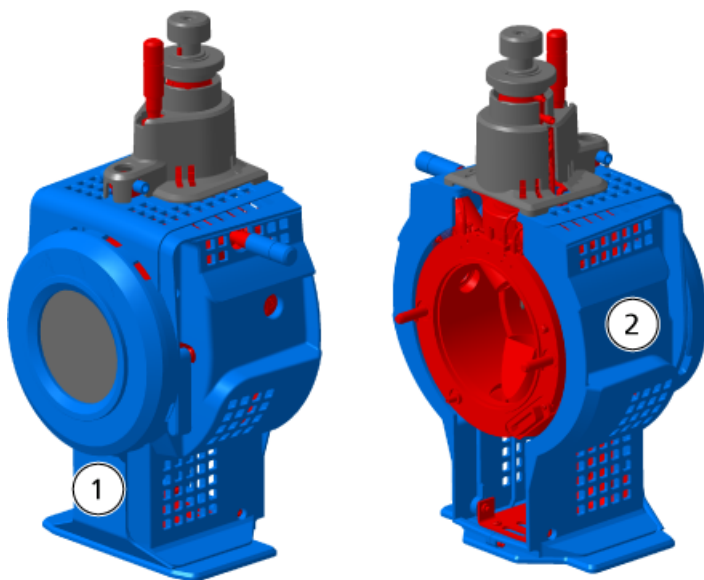
Note: For part numbers, refer to the *Parts and Equipment Guide* for the mass spectrometer.

Table 5-1 Ion Source Maintenance Tasks

Component	Frequency	Task	For more information...
TurbolonSpray [®] and APCI probes	As needed	Inspect and replace	Refer to Remove the Probe and Install the Probe .
TurbolonSpray [®] and APCI electrodes	As needed	Inspect and replace	Refer to Replace the Electrode .
Corona discharge needle	As needed	Replace	Refer to Replace the Corona Discharge Needle .
Turbo heater	As needed	Replace	Contact the local QMP or FSE.
Sample tubing	As needed	Replace	Refer to Connect the Ion Source Tubing .

Ion Source Handling

Surfaces of the ion source become hot during operation. [Figure 5-1](#) shows surfaces that are cooler (blue and gray) and surfaces that remain hot for an extended period of time (red). Do not touch the red surfaces shown below while using or removing the ion source.

Figure 5-1 Ion Source Hot Surfaces (Red=Hot, Gray=Warm, Blue=Handle with Care)

Item	Description
1	Front
2	Back

Remove the Ion Source

Note: (3500, 4500, 5500, 5500+, 6500, 6500+, and TripleTOF[®] systems) Nitrogen continues to flow at a rate of 5.3 L/min 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. Wait at least 30 minutes for the ion source to cool.
5. Disconnect the sample tubing from the grounding union.

Ion Source Maintenance

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.

Note: Be careful not to lose the O-rings that are installed on the vacuum interface.

8. Put the ion source on a clean, secure surface.

Clean the Ion Source Surfaces

Prerequisite Procedures
<ul style="list-style-type: none">• Remove the Ion Source



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.

- Wipe the surfaces of the ion source with a soft, damp, cloth.

Clean the Probe

Flush the ion source periodically, regardless of the type of compounds sampled. Do this by setting up a method in the Analyst[®] or Analyst[®] TF 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[®] or Analyst[®] TF software do the following:
 - a. Create an MS method.
 - b. Set **TEM** between **500** and **600**.
 - c. Set **GS1** and **GS2** to at least **40**.
 - d. Set **CUR** to the highest setting possible.
4. Wait until the **TEM** setpoint is reached.
5. Make sure that the probe and sample tubing are flushed thoroughly.

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](#)

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



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. The tip of the electrode is extremely sharp.

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

Note: After replacing the electrode, evaluate the effect of the change on system performance.

Ion Source Maintenance

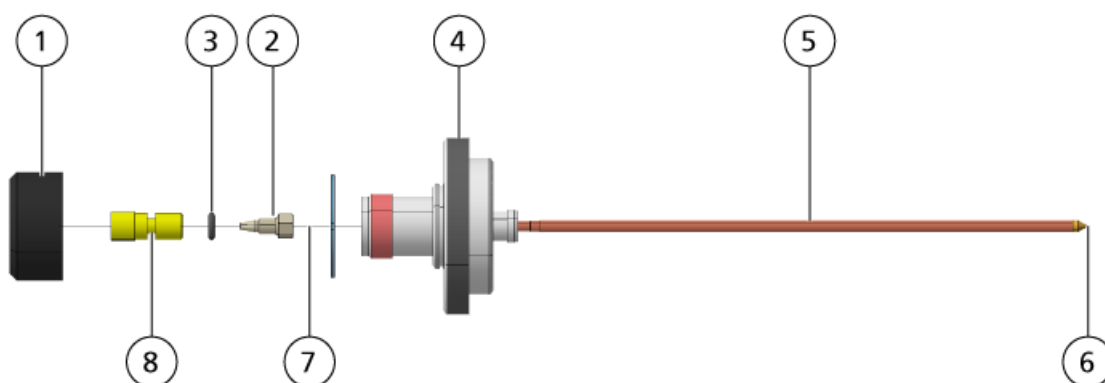
Prerequisite Procedures

- [Remove the Ion Source](#)
- [Remove the Probe](#)

This procedure applies to both probes.

1. Remove the electrode adjustment nut and then remove the electrode.
2. Holding the probe with the tip pointing downwards, so that the spring remains inside the probe, install a sample fitting in the PEEK union, tightening until it is finger-tight.

Figure 5-2 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. Pull the PEEK union and the attached electrode tube from the probe.
4. Remove the sample fitting from the PEEK union.

5. Use the 1/4 inch open-ended wrench to remove the retaining nut that holds the electrode tube in the PEEK union.
6. Remove the electrode tube from the retaining nut.
7. 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.

8. Tighten the retaining nut.

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

9. Make sure that the spring is still inside the probe and then tighten the electrode adjustment nut.
10. 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.
11. Install and then tighten the electrode adjustment nut.
12. Install the probe. Refer to [Install the Probe](#).
13. Install the ion source on the mass spectrometer. Refer to [Ion Source Installation](#).
14. Connect the sample tubing. Refer to [Connect the Ion Source Tubing](#).
15. Adjust the electrode tip extension. Refer to [Optimize the TurbolonSpray[®] Probe Position](#) or [Optimize the APCI Probe Position](#).

Replace the Corona Discharge Needle



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. Handle the needle with care. The tip of the needle is extremely sharp.

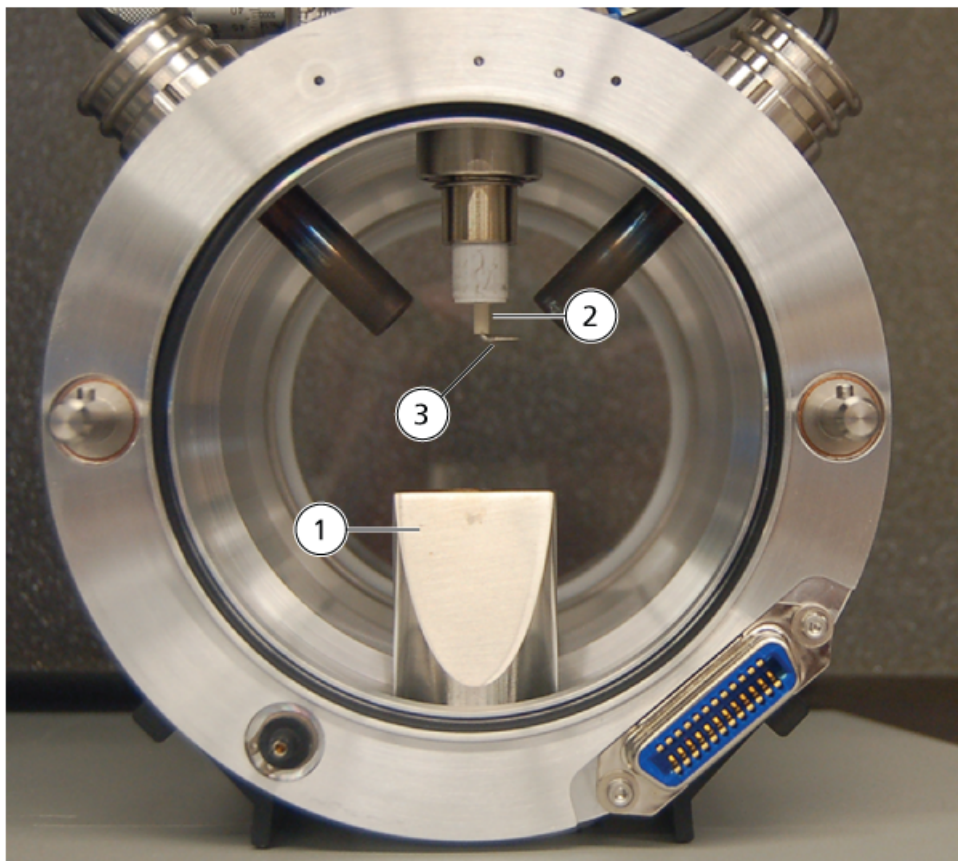
Prerequisite Procedures
<ul style="list-style-type: none"> • Remove the Ion Source • Remove the Probe

If the corona discharge needle tip becomes corroded, it might not be removable by hand. In that case, cut off the needle tip to remove it, then replace the entire corona discharge needle.

Ion Source Maintenance

1. Rotate the ion source so the open side is accessible.

Figure 5-3 Corona Discharge Needle



Item	Description
1	Exhaust chimney
2	Ceramic sleeve
3	Corona discharge needle tip

2. While holding the corona discharge needle adjustment screw (refer to [Ion Source Components](#)) between the thumb and forefinger of one hand and the corona discharge needle with the other hand, rotate the corona discharge needle tip counter-clockwise to loosen and gently remove the tip.
3. Gently pull the corona discharge needle down through the exhaust chimney to remove it.

4. Insert the new needle through the exhaust chimney into the ceramic sleeve as far as possible.
5. Holding a new tip between the thumb and forefinger of one hand and the corona discharge needle adjustment screw with the other hand, rotate the corona discharge needle tip clockwise to install the tip.
6. Insert the probe and then install the ion source on the mass spectrometer. Refer to [Ion Source Installation](#).

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.

Prerequisite Procedures

- Stop the sample flow and make sure that any remaining gas has been removed through the source exhaust system.
- [Remove the Ion Source](#).

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

1. Disconnect the sample tubing from the probe and the grounding union.
2. Replace the sample tubing with an appropriate length of tubing, cut with a proper tubing cutter. Refer to [Connect the Ion Source Tubing](#).
3. Install the ion source. Refer to [Ion Source Installation](#).
4. Start the sample flow.

Storage and Handling



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

The environmental requirements for the storage and transport of the ion source:

- Ambient temperature between -30°C and $+60^{\circ}\text{C}$ (-22°F and 140°F)
- Atmospheric pressure between 75 kPa to 101 kPa
- Relative humidity not exceeding 99%

Ion Source Troubleshooting

6

Symptom	Possible Cause	Corrective Action
The Analyst® or Analyst® TF software reports that the mass spectrometer has gone to a Fault state.	<ol style="list-style-type: none">1. The probe is not installed.2. The probe is not connected securely.	<ol style="list-style-type: none">1. Install the probe. Refer to Install the Probe.2. Reinstall the probe:<ol style="list-style-type: none">a. Remove the probe. Refer to Remove the Probe.b. Install the probe, making sure to tighten the retaining ring securely. Refer to Install the Probe.
The Analyst® or Analyst® TF software indicates that a specific probe is in use, but a different probe is installed.	The F3 fuse is blown.	Contact an FSE.
The spray is not uniform.	The electrode is blocked.	Replace the electrode. Refer to Replace the Electrode .
Sensitivity is poor.	<ol style="list-style-type: none">1. The interface components (front end) are dirty.2. Solvent vapor or other unknown compounds are present in the analyzer region.	<ol style="list-style-type: none">1. Clean the interface components and then install the ion source.2. Optimize the Curtain Gas™ flow. Refer to Ion Source Optimization.
During testing, the ion source fails to meet specifications.	<ol style="list-style-type: none">1. The test solution was not prepared correctly.2. The mass spectrometer has not passed the installation tests.	<ol style="list-style-type: none">1. Confirm that the test solutions were prepared correctly.2. If the problem cannot be resolved, then contact the FSE to perform the installation tests.

Symptom	Possible Cause	Corrective Action
Background noise is high.	<ol style="list-style-type: none"> 1. The Temperature (TEM) is too high. 2. The heater gas flow rate (GS2) is too high. 3. The ion source is contaminated. 	<ol style="list-style-type: none"> 1. Optimize the temperature. 2. Optimize heater gas flow. 3. Clean or replace the ion source components, and then condition the ion source and front end: <ol style="list-style-type: none"> a. Move the probe to the furthest position from the aperture (vertically and horizontally). b. Make sure that the interface heater is On. c. Infuse or inject 50:50 methanol:water with a pump flow rate of 1 mL/min. d. In the Analyst[®] or Analyst[®] TF software, set TEM to 650, GS1 to 60, and GS2 to 60. e. Set the CUR flow to 45 or 50. f. Run for a minimum of 2 hours or preferably overnight for best results.

Ion Source Troubleshooting

Symptom	Possible Cause	Corrective Action
Ion source performance has degraded.	<ol style="list-style-type: none">1. The probe is not optimized.2. The sample was not prepared correctly or the sample has degraded.3. The sample inlet fittings are leaking.	<ol style="list-style-type: none">1. Optimize the probe. Refer to TurbolonSpray® Probe Optimization or APCI Probe Optimization.2. Confirm that the sample was prepared correctly.3. Verify that the fittings are tight and replace the fittings if leaks continue. Do not overtighten the fittings.4. Install and optimize an alternate ion source. If the issue persists, then contact an FSE
Arcing or sparks occur.	The position of the corona discharge needle is incorrect.	Turn the corona discharge needle toward the curtain plate, and away from the stream of heater gas. Refer to Adjust the Position of the Corona Discharge Needle .

Principles of Operation — Ion Source

A

Electrospray Ionization Mode

The probe is located centrally between the two turbo heaters, which are located at a 45-degree angle on either side of the probe. The combination of IonSpray™ effluent and the heated dry gas from the turbo heaters is projected at a 90-degree angle to the aperture in the curtain plate.

Only compounds that ionize in the liquid solvent can be generated as gas phase ions in the source. The efficiency and rate of ion generation depends on the solvation energies of the specific ions. Ions with lower solvation energies are more likely to evaporate than ions with higher solvation energies.

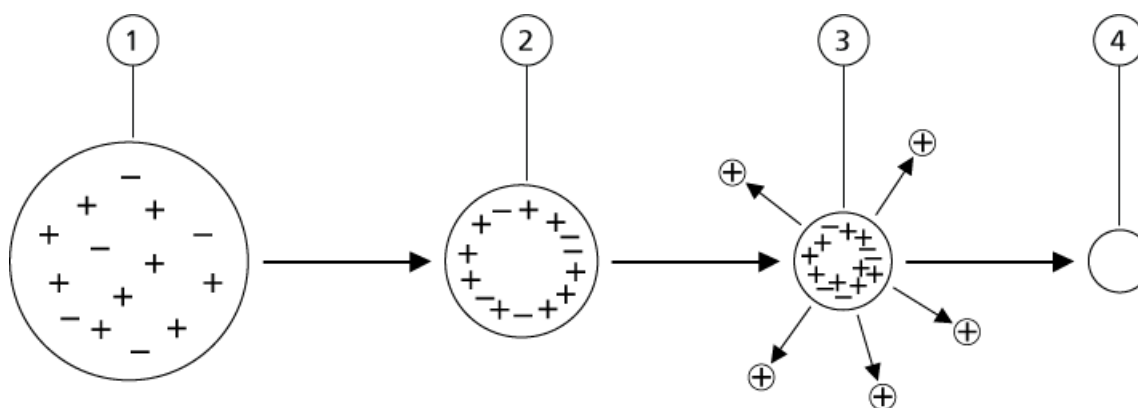
The interaction of the IonSpray™ voltage and the turbo heaters helps focus the stream and increases the rate of droplet evaporation, resulting in an increased ion signal. The heated gas increases the efficiency of ion evaporation, resulting in increased sensitivity and the ability to handle higher liquid sample flow rates.

A high-velocity flow of nebulizer gas shears droplets from the liquid sample stream in the IonSpray™ inlet. Using the variable high voltage applied to the sprayer, the ion source applies a net charge to each droplet. This charge aids in the droplet dispersion. Ions of a single polarity are preferentially drawn into the droplets by the high voltage as they are separated from the liquid stream. However, this separation is incomplete and each droplet contains many ions of both polarities. Ions of one polarity are predominant in each droplet, and the difference between the number of positively or negatively charged ions results in the net charge. Only the excess ions of the predominant polarity are available for ion evaporation, and only a fraction of these actually evaporate.

The probe can generate multiply-charged ions from compounds that have multiple charge sites, such as peptides and oligonucleotides. This is useful during analysis of high-molecular-weight species where the multiple charges produce ions of a mass-to-charge (m/z) ratio within the mass range of the mass spectrometer. This allows routine molecular-weight determinations of compounds in the kiloDalton (kDa) range.

As shown in [Figure A-1](#), each charged droplet contains solvent and both positive and negative ions, but with ions of one predominant polarity. As a conducting medium, excess charges reside at the surface of the droplet. As the solvent evaporates, the electrical field at the surface of the droplet increases due to the decreasing radius of the droplet.

Figure A-1 Ion Evaporation



Item	Description
1	Droplet contains ions of both polarities with one polarity being predominant.
2	As the solvent evaporates, the electrical field increases and the ions move to the surface.
3	At some critical field value, ions are emitted from the droplets.
4	Nonvolatile residue remains as a dry particle.

If the droplet contains excess ions and enough solvent evaporates from the droplet, a critical field is reached at which ions are emitted from the surface. Eventually, all of the solvent will evaporate from the droplet, leaving a dry particle consisting of the nonvolatile components of the sample solution.

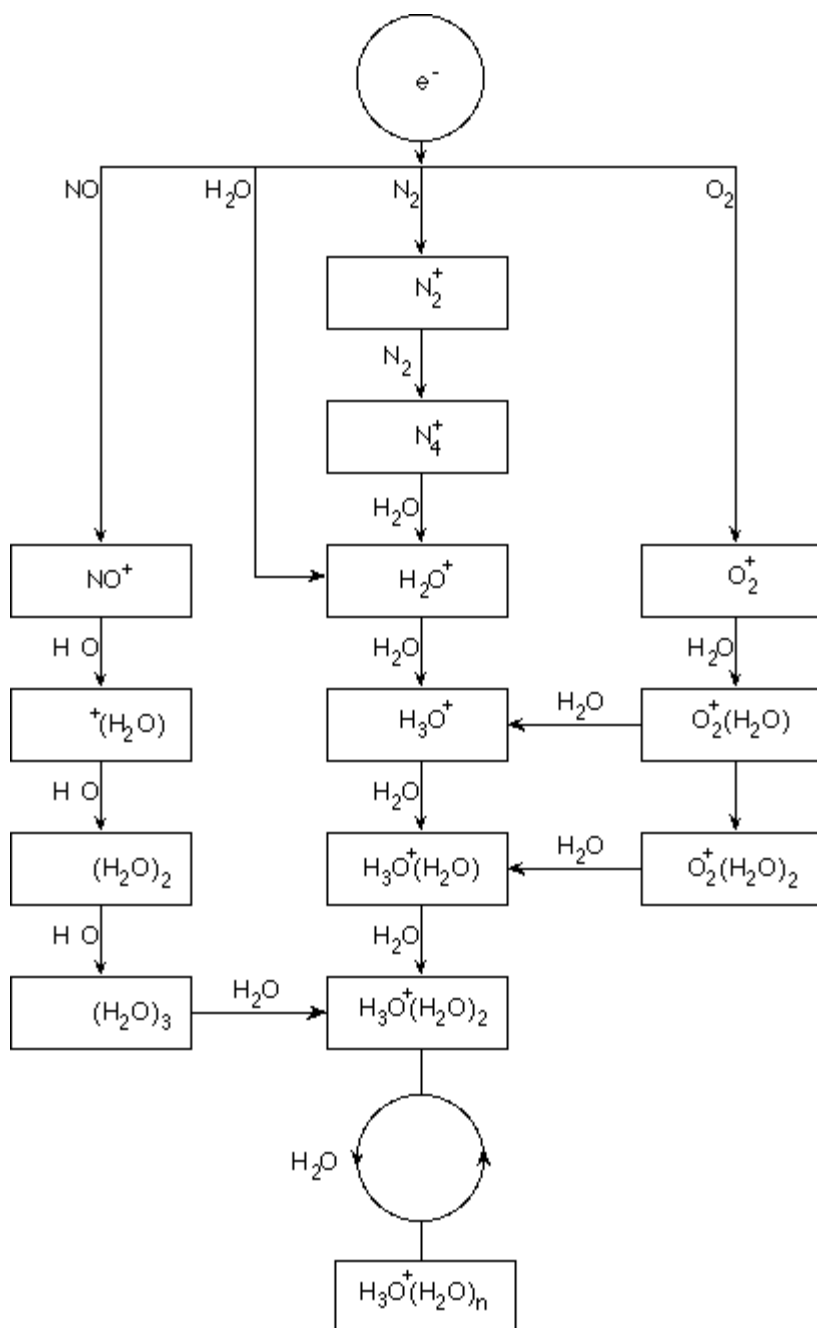
Because the solvation energies for most organic molecules are unknown, the sensitivities of any given organic ion to ion evaporation are difficult to predict. The importance of solvation energy is evident because surfactants that concentrate at the surface of a liquid can be detected very sensitively.

APCI Mode

The basis for past incompatibilities in linking liquid chromatography with mass spectrometry arose from difficulties converting relatively involatile molecules in solution in a liquid into a molecular gas without inducing excessive decomposition. The APCI probe process of gently nebulizing the sample into finely dispersed small droplets in a heated ceramic tube results in the rapid vaporization of the sample so that the sample molecules are not decomposed.

Figure A-2 shows the reaction flow of the APCI process for reactant positive ions (the proton hydrates, $\text{H}_3\text{O}^+[\text{H}_2\text{O}]_n$).

Figure A-2 APCI Reaction Flow Diagram



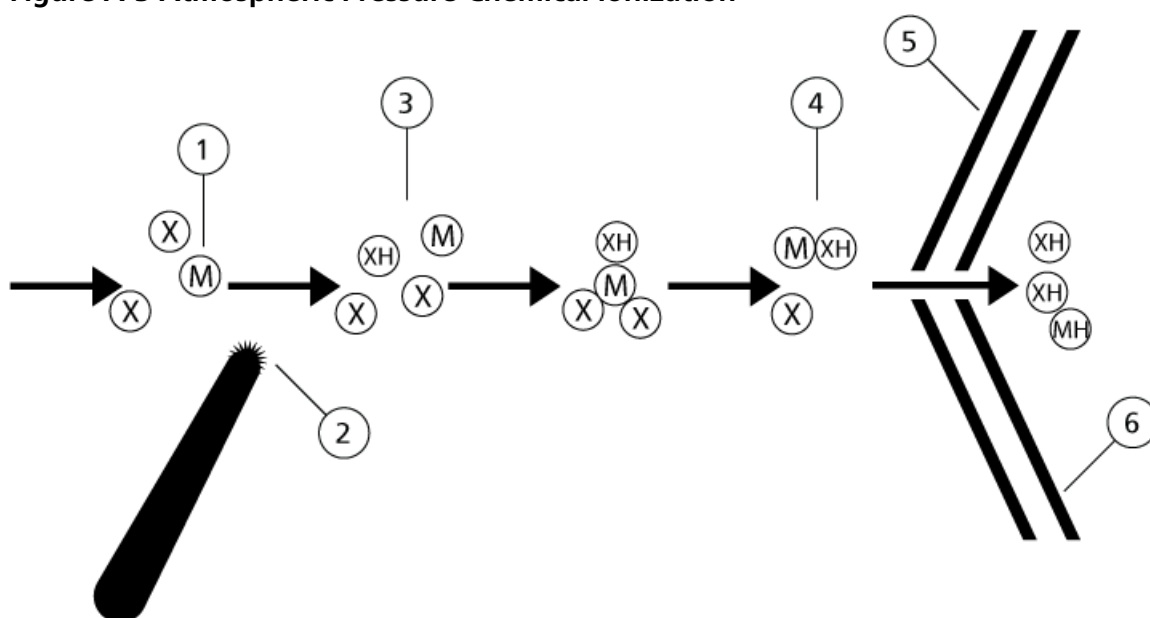
The major primary ions N_2^+ , O_2^+ , H_2O^+ , and NO^+ are formed by the electron impact of corona created electrons on the major neutral components of air. Although NO^+ is normally not a major constituent of clean air, the concentration of this species in the source is enhanced due to neutral reactions initiated by the corona discharge.

Principles of Operation — Ion Source

Samples that are introduced through the APCI probe are sprayed, with the aid of a nebulizer gas, into the heated ceramic tube. Within the tube, the finely dispersed droplets of sample and solvent undergo a rapid vaporization with minimal thermal decomposition. The gentle vaporization preserves the molecular identity of the sample.

The gaseous sample and solvent molecules pass into the ion source housing where the ionization by APCI is induced by a corona discharge needle connected to the end of the ceramic tube. The sample molecules are ionized by colliding with the reagent ions created by the ionization of mobile phase solvent molecules. As shown in [Figure A-3](#), the vaporized solvent molecules ionize to produce the reagent ions $[X+H]^+$ in the positive mode and $[X-H]^-$ in the negative mode. It is these reagent ions that produce stable sample ions when they collide with the sample molecules.

Figure A-3 Atmospheric Pressure Chemical Ionization



Item	Description
1	Sample
2	Primary ions are created in the vicinity of the corona discharge needle
3	Ionization produces predominantly solvent ions
4	Reagent ions react with sample molecules forming clusters
5	Curtain plate
6	Interface

x = solvent molecules; M=sample molecules

The sample molecules are ionized through a process of proton transfer in the positive mode and by either electron transfer or proton transfer in the negative mode. The energy for the APCI ionization process is collision-dominated because of the relatively high atmospheric pressure of the ion source.

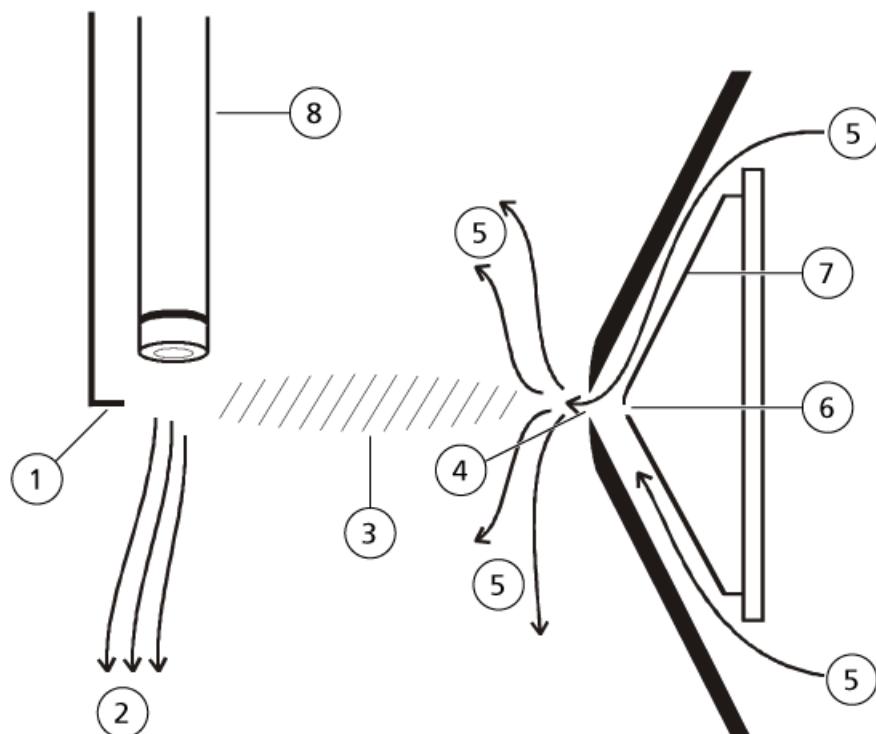
For reverse phase applications, the reagent ions consist of protonated solvent molecules in the positive mode and solvated oxygen ions in the negative mode. With favorable thermodynamics, the addition of modifiers changes the reagent ion composition. For example, the addition of acetate buffers or modifiers can make the acetate ion $[\text{CH}_3\text{COO}]^-$ the primary reagent in the negative mode. Ammonium modifiers may make protonated ammonia $[\text{NH}_4]^+$ the primary reagent in the positive mode.

Through collisions, an equilibrium distribution of certain ions (for example, protonated water cluster ions) is maintained. The likelihood of premature fragmentation of the sample ions in the ion source is reduced because of the moderating influence of solvent clusters on the reagent ions and the relatively high gas pressure in the source. As a result, the ionization process yields primarily molecular product ions for mass analysis in the mass spectrometer.

APCI Ionization Region

Figure A-4 shows the general location of the ion-molecule reactor of the APCI probe. The slanted lines indicate a wall-less reactor. A self-starting corona discharge ion current in the microampere range is created as a result of the electric field between the discharge needle and the curtain plate. Primary ions, for example, N_2^+ and O_2^+ , are created by the loss of electrons that originate in the plasma in the immediate vicinity of the discharge needle tip. The energy of these electrons is moderated by a number of collisions with gas molecules before attaining an energy where their effective ionization cross-section allows them to ionize neutral molecules efficiently.

Figure A-4 APCI Ionization Region



Item	Description
1	Discharge needle tip
2	Sample flow
3	Wall-less reactor
4	Curtain plate aperture
5	Curtain Gas™ supply
6	Orifice
7	Orifice plate
8	Ceramic tube

The primary ions, in turn, generate intermediate ions that lead to the formation of sample ions. Ions of the chosen polarity drift under the influence of the electric field in the direction of the curtain plate and through the gas curtain into the mass analyzer. The whole ion formation process is collision-dominated because of the relatively high atmospheric pressure of the APCI probe. Except in the immediate vicinity of the discharge needle tip, where the

strength of the electric field is greatest, the energy imparted to an ion by the electric field is small in comparison with the thermal energy of the ion.

Through collisions, an equal distribution of certain ions (for example, protonated water cluster ions) is maintained. Any excess energy that an ion may acquire in the ion-molecule reaction process is thermalized. Through collisional stabilization, many of the product ions are fixed, even though many subsequent collisions occur. The formation of both product ions and reactant ions is governed by equilibrium conditions at 760 torr (atmospheric) operating pressure.

The APCI probe functions as a wall-less reactor because the ions that pass from the source to the vacuum chamber and eventually to the detector never experience collisions with a wall—only collisions with other molecules. Ions are also formed outside the designated ion source, but are not detected and are eventually neutralized by interacting with a wall surface.

The temperature of the probe is an important factor for APCI probe operation. To preserve the molecular identity, the temperature must be set high enough to ensure a rapid evaporation. At a sufficiently high operating temperature, the droplets are vaporized quickly so that organic molecules are desorbed from the droplets with minimal thermal degradation. If, however, the temperature is set too low, the evaporation process is slower and pyrolysis, or decomposition, may occur before vaporization is complete. Operating the APCI probe at temperatures above the optimal temperature may cause thermal decomposition of the sample.

Source Parameters and Voltages

B

TurbolonSpray[®] Probe Parameters

The following table shows the recommended operating conditions for the TurbolonSpray[®] probe at three different flow rates. For each flow rate, the Curtain Gas[™] flow should be as high as possible. The solvent composition used for optimization was 1:1 water:acetonitrile. These conditions represent a starting point from which to optimize the probe. Using an iterative process, optimize the parameters using flow injection analysis to achieve the best signal or signal-to-noise for the compound of interest.

Table B-1 Parameter Optimization for the TurbolonSpray[®] Probe

Parameters	Typical Values			Operational Range
LC flow rate	5 µL/min to 50 µL/min	200 µL/min	1000 µL/min	5 µL/min to 3000 µL/min
Gas 1 (nebulizer gas)	20 psi to 40 psi	40 psi to 60 psi	40 psi to 60 psi	0 psi to 90 psi
Gas 2 (heater gas)	0 psi	50 psi	50 psi	0 psi to 90 psi
IonSpray voltage	5500	5500 V	5500 V	5500 V
Curtain Gas [™] supply	20 psi	20 psi	20 psi	20 psi to 50 psi
Temperature ¹	0 °C to 200 °C	200 °C to 650 °C	400 °C to 750 °C	Up to 750 °C
Declustering Potential (DP) ²	Positive: 70 V Negative: -70 V	Positive: 70 V Negative: -70 V	Positive: 70 V Negative: -70 V	Positive: 0 V to 400 V Negative: -400 V to 0 V
Probe vertical micrometer setting	7 to 10	2 to 5	0 to 2	0 to 13
Probe horizontal micrometer setting	4 to 6	4 to 6	4 to 6	0 to 10

¹ Optimum temperature values depend on the compound and mobile phase composition (higher aqueous content requires higher temperature). Zero (0) means no temperature is applied.

² DP values depend on the compound.

APCI Probe Parameters

Table B-2 Parameter Optimization for the APCI Probe

Parameter	Typical Value	Operational Range
LC flow rate	1000 µL/min	200 µL/min to 2000 µL/min
Gas 1 (nebulizer gas)	30 psi	0 psi to 90 psi
Curtain Gas TM supply	20 psi	20 psi to 50 psi
Temperature ³	400 °C	100 °C to 750 °C
Nebulizer Current (NC)	Positive: 3 µA Negative: -3 µA	Positive: 0 mA to 5 µA Negative: -5 mA to 0 µA
Declustering Potential (DP)	Positive: 60 V Negative: -60 V	Positive: 0 V to 300 V Negative: -300 V to 0 V
Probe vertical micrometer setting	4	Scale 0 to 13

Parameter Descriptions

Table B-3 Source-Dependent Parameters

Parameter	Description
Ion Source Gas 1 (GS1)	Controls the nebulizer gas for the TurbolonSpray [®] and APCI probes. Refer to Principles of Operation — Ion Source .
Ion Source Gas 2 (GS2)	Controls the heater gas for the TurbolonSpray [®] probe. The best sensitivity is achieved when the combination of temperature (TEM) and heater gas (GS2) flow rate causes the LC solvent to reach a point at which it is nearly all vaporized. To optimize GS2, increase the flow to obtain the best signal or signal-to-noise ratio if there is a significant increase in background noise. Too high a gas flow can produce a noisy or unstable signal. Refer to Principles of Operation — Ion Source .

³ Temperature value depends on the compound.

Source Parameters and Voltages

Table B-3 Source-Dependent Parameters (continued)

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 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>
Temperature (TEM)	<p>Controls the heat applied to the sample to vaporize it. The optimal temperature is the lowest temperature at which the sample is vaporized completely.</p> <p>Optimize in increments of 50 °C.</p>
Temperature (TEM) - TurbolonSpray® probe	<p>Controls the temperature of the heater gas in the TurbolonSpray® probe.</p> <p>The best sensitivity is achieved when the combination of temperature (TEM) and heater gas (GS2) flow rate causes the LC solvent to reach a point at which it is nearly all vaporized.</p> <p>As the organic content of the solvent increases, the optimal probe temperature decreases. With solvents consisting of 100% methanol or acetonitrile, the probe performance might optimize as low as 300 °C. Aqueous solvents consisting of 100% water at flows of approximately 1000 µL/min require a maximum probe temperature of 750 °C.</p> <p>If the temperature is set too low, then vaporization is incomplete and large, and visible droplets are expelled into the ion source housing.</p> <p>If the temperature is set too high, then solvent might vaporize prematurely at the probe tip, especially if the probe is set too low (5 mm to 13 mm).</p>
Temperature (TEM) - APCI probe	<p>Controls the temperature of the APCI probe.</p> <p>As the organic content of the solvent increases, the optimal probe temperature should decrease. With solvents consisting of 100% methanol or acetonitrile the probe performance might optimize at temperatures as low as 400 °C at flow rates of 1000 µL/min. Aqueous solvents consisting of 100% water set at flows of approximately 2000 µL/min require a minimum probe temperature of 700 °C.</p> <p>If the temperature is set too low, then vaporization is incomplete and large, and visible droplets are expelled into the ion source housing.</p> <p>If the temperature is set too high, then thermal degradation of the sample occurs.</p>

Table B-3 Source-Dependent Parameters (continued)

Parameter	Description
Nebulizer Current (NC)	Controls the current applied to the corona discharge needle in the APCI probe. The discharge ionizes solvent molecules, which in turn ionize the sample molecules. For the APCI probe the current applied to the corona discharge needle (NC) usually optimizes over a broad range (about 1 μ A to 5 μ A in positive mode). To optimize, start at a value of 1 and then increase to achieve the best signal or signal-to-noise ratio. If, when the current is increased, no changes in signal are observed, then leave the current at the lowest setting that provides the best sensitivity (for example, 2 μ A).
IonSpray Voltage Floating (ISVF) or IonSpray Voltage (IS)	Controls the voltage applied to the sprayer in the TurbolonSpray [®] probe, which ionizes the sample in the ion source. The parameter value depends on the polarity, and affects the stability of the spray and the sensitivity.
Interface Heater (ihe)	This parameter is always on for 3500, 4500, 5500, 5500+, 6500, 6500+, and TripleTOF [®] systems. The ihe parameter turns the interface heater on and off. Heating the interface helps maximize the ion signal and prevents contamination of the ion optics. Unless the compound the user is analyzing is extremely fragile, we recommend that the user heats the interface.

Probe Position

The position of the probe can affect the sensitivity of the analysis. Refer to [Ion Source Optimization](#) for more information on how to optimize the position of the probe.

Solvent Composition

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 50 mmol/L for negative ions. The concentration of organic acids is 0.1% to 0.5% by volume for the TurbolonSpray[®] probe and 0.1% to 2.0% by volume for the APCI probe.

Commonly used solvents are:

- Acetonitrile
- Methanol
- Propanol
- Water

Source Parameters and Voltages

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











Glossary of Symbols


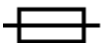









C

Note: Not all of the symbols in the following table are applicable to every instrument.











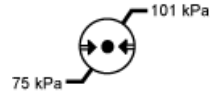

Symbol	Description
	Australian Regulatory Compliance Mark. Indicates the products complies with Australian Communications Media Authority (ACMA) EMC Requirements.
	Alternating current
A	Amperes (current)
	Authorized representative in the European community
	Biohazard
	CE Marking of Conformity
	cCSAus mark. Indicates electrical safety certification for Canada and USA.
	Catalogue number
	Caution Note: In SCIEX documentation, this symbol identifies a personal injury hazard.


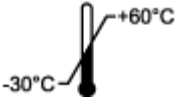





Glossary of Symbols

Symbol	Description
	China RoHS Caution Label. The electronic information product contains certain toxic or hazardous substances. The center number is the Environmentally Friendly Use Period (EFUP) date, and indicates the number of calendar years the product can be in operation. Upon the expiration of the EFUP, the product must be immediately recycled. The circling arrows indicate the product is recyclable. The date code on the label or product indicates the date of manufacture.
	China RoHS logo. The device does not contain toxic and hazardous substances or elements above the maximum concentration values, and it is an environmentally-friendly product that can be recycled and reused.
	Consult instructions for use.
	cTUVus mark for TUV Rheinland of North America.
	Data Matrix symbol that can be scanned by a barcode reader to obtain a unique device identifier (UDI).
	Environmental Hazard
	Ethernet connection
	Explosion Hazard
	Fire Hazard
	Flammable Chemical Hazard

Symbol	Description
	Fragile
	Fuse
Hz	Hertz
	High Voltage. Electrical Shock Hazard If the main cover must be removed, contact a SCIEX representative to prevent electric shock.
	Hot Surface Hazard
	In Vitro Diagnostic Device
	Ionizing Radiation Hazard
	Keep dry. Do not expose to rain. Relative humidity must not exceed 99%.
	Keep upright.
	Lacerate/Sever Hazard
	Laser Radiation Hazard
	Lifting Hazard

Glossary of Symbols

Symbol	Description
	Manufacturer
	Moving Parts Hazard
	Pinch Hazard
	Pressurized Gas Hazard
	Protective Earth (ground)
	Puncture Hazard
	Reactive Chemical Hazard
	Serial number
	Toxic Chemical Hazard
	Transport and store the system within 66 kPa to 103 kPa.
	Transport and store the system within 75 kPa to 101 kPa.
	Transport and store the system within 10% to 90% relative humidity.

Symbol	Description
	Transport and store the system within –30 °C to +45 °C.
	Transport and store the system within –30 °C to +60 °C.
	USB 2.0 connection
	USB 3.0 connection
	Ultraviolet Radiation Hazard
VA	Volt Ampere (power)
V	Volts (voltage)
	WEEE. Do not dispose of equipment as unsorted municipal waste. Environmental Hazard
W	Watts
	yyyy-mm-dd Date of manufacture

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