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API LC/MS and LC/MS/MS IonSpray Ion Source Manual

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IonSpray Ion Source Manual

About This Manual

This manual contains the instructions required to operate the API IonSpray Ion Source.

Conventions



Within this manual, the following conventions are used:

WARNING! Indicates an operation that may cause personal injury if precautions are not followed.

CAUTION! Indicates an operation that may cause damage to the instrument if precautions are not followed.

NOTE: Emphasizes significant information in a procedure or description.

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Inlet Description

IonSpray (a technique developed at Cornell University), is an Atmospheric Pressure Ion Source in which pre-formed ions in solution are emitted into the gas phase without the application of any heat. In this way, quasi-molecular ions can be generated from very labile and high molecular weight compounds with no thermal degradation.

IonSpray is the technique by which a liquid sample is pumped by a liquid chromatography (LC) pump or syringe drive through the IonSpray Inlet, which is maintained at a high voltage, and is sprayed into the Ion Source creating a mist of highly charged droplets. Once inside the Ion Source, the droplets evaporate causing the ions to enter the gas phase by a low energy process called Ion Evaporation.

The IonSpray source consists of a low dead volume sprayer which is electrically isolated from the inlet fitting with fused silica capillary tubing through which the liquid flows. Up to 6000 VDC (positive or negative) can be applied to the sprayer from a power supply controlled from the data system keyboard. The IonSpray pneumatic nebulizer is fully articulatable which allows sample introduction in virtually any position. It has a built-in, low dead volume splitter for use when flow splitting is desired, making IonSpray directly compatible with most analytical and preparative high performance liquid chromatography (HPLC) columns (e.g., 0.18, 0.32, 0.5, 1.0, 2.1, 4.6 and 6.0 mm ID) and post column fraction collectors. IonSpray offers tremendous flexibility and is ideal for use in research environments as well as those requiring rugged and routine operation.

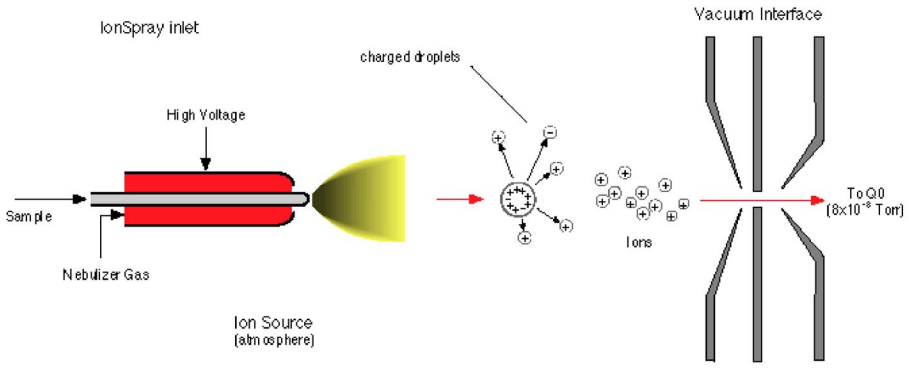


Illustration of IonSpray and Ion Source

IonSpray Features

- High sensitivity for biological compounds
- Produces molecular ions with no thermal degradation
- Low sample consumption
- Compatible with a variety of separation methods (e.g. LC, CE)
- Molecular weight determinations into the kDa mass range
- Fully articulatable for optimum performance
- Flow rates from 0.1 µL/min to > 1 mL/min

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When interfaced to our API System, IonSpray is an extremely sensitive and flexible ion source. It can be connected to a LC system to provide LC/MS, or to a syringe pump to allow small volumes of sample to be introduced directly to the MS at low flow rates.

Because the entire IonSpray process operates at ambient temperature, there is no temperature to optimize and no thermal decomposition to complicate the spectrum. Sprayer voltage and pressure optimize over a broad range thus optimum performance remains stable from one day to the next.

Ionization - IonSpray Theory

This section describes the theory of IonSpray, including the formation of charged droplets and the Ion Evaporation mechanism.

Droplet Generation and Charging

In the IonSpray Source, a high velocity flow of nebulizer gas shears droplets from the liquid sample stream. Using the variable high voltage applied to the sprayer, a net charge is applied to each droplet and aids in 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 the separation is incomplete, so each droplet contains many ions of both polarities. In each droplet the ions of one polarity are predominant, and the difference between the number of positively and 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 polarity and concentration of excess ions depends on the magnitude and polarity of the high voltage potential applied to the sprayer tip. For example, when a sample contains arginine in water/acetonitrile, and a positive potential is applied to the sprayer, the excess positive ions will be H^+ and MH^+ of arginine.

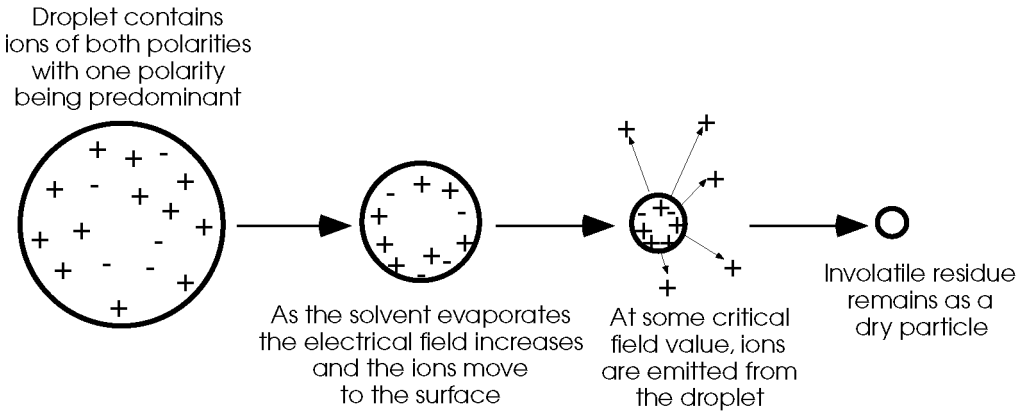
IonSpray can generate multiply charged ions from compounds which have multiple charge sites, such as, peptides and oligonucleotides. This is useful in the observation of high molecular weight species, where the multiple charges produce ions of a mass-to-charge (m/z) value within the mass range of the instrument, allowing routine molecular weight determinations of compounds in the kilodalton (kDa) range.

Ion Evaporation

Each charged droplet contains solvent and both positive and negative ions, with ions of one polarity being predominant. A simple view of the droplet as a conducting medium suggests that excess charges reside at the droplet surface. As the solvent evaporates, the electrical field at the surface of the droplet increases due to the decreasing radius of the droplet.

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

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Ion Evaporation

Only compounds which ionize in the liquid solvent can be generated as gas phase ions in the IonSpray Ion 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.

Given that the solvation energies for most organic ions are not known, the sensitivities of any given organic ion to ion evaporation is difficult to predict. An indication of the importance of solvation energy is provided by the observation that surfactants, which concentrate at the surface of a liquid, tend to be very sensitively detected.

IonSpray Components

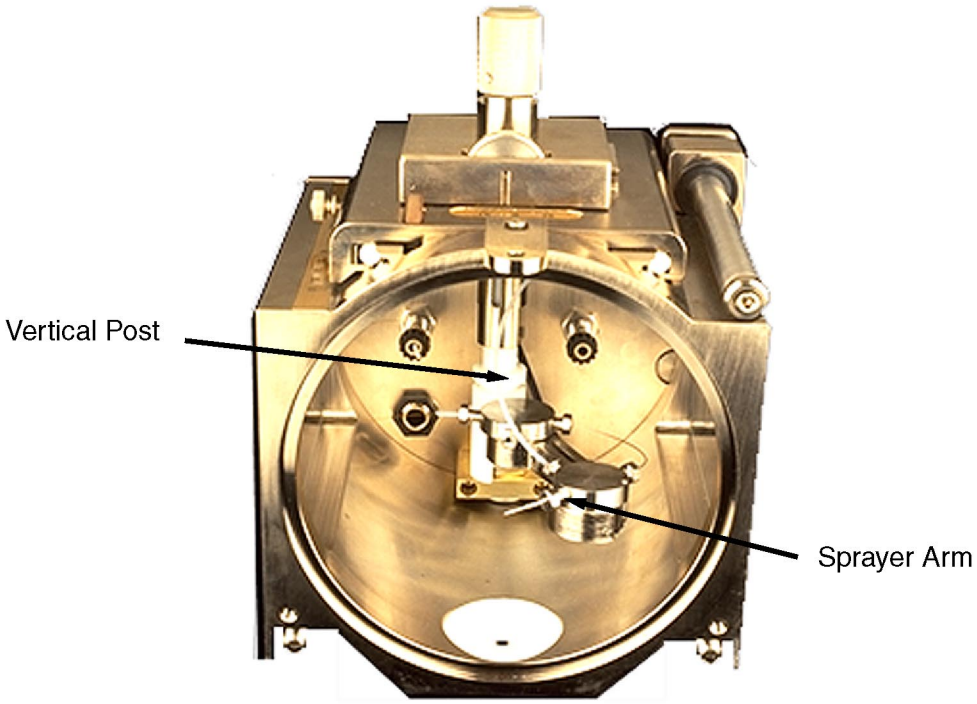
The IonSpray Source consists of a sprayer arm which is attached to the top of the Ion Source Housing using an electrically isolated mount.

The Ion Source sits above the Source Interlock Assembly which is attached to the Source Cover. The Source Interlock Assembly will disable the instrument's high voltages if a valid Ion Source is not properly installed.



WARNING! During normal operation, high voltages exist in the IonSpray source. Do not operate the instrument without the viewing glass in place on the casing of the IonSpray source.

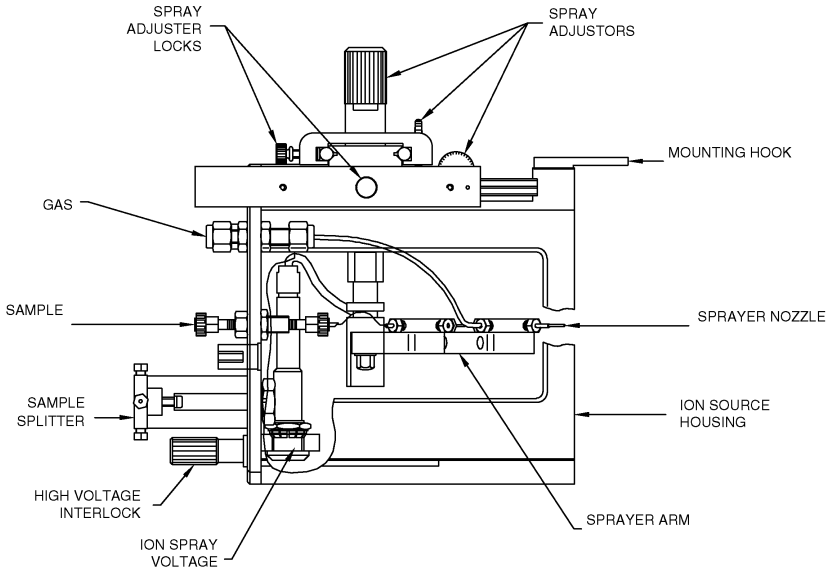
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The IonSpray Source

IonSpray Source Housing

The Ion Source housing mounts onto the front end of the Vacuum Chamber and is held in position by a mounting hook which fits over a pin on the Vacuum Interface. Two thumb screws anchored on either side of the Vacuum Interface thread into the Ion Source housing to position and secure it. Plexiglas windows on the front and back sides of the housing allow direct observation of the IonSpray Inlet.



IonSpray Source - Side View

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External connections to the IonSpray source are made through the bottom and Interface Connection Plate of the Ion Source housing. These attachments include the High Voltage connection for the sprayer, the LC Inlet and Splitter Outlet connections, and the Nebulizer Gas connection. As well, an Ion Source exit aperture mounted in the bottom of the housing allows sample vapors to be vented to a fume hood or exhausted via the Source Exhaust System.

The Ion Source and housing can be quickly, and easily removed without tools to provide convenient access to the Vacuum Interface. In addition, the Interface Connection Plate can be opened to provide access to the sprayer arm without removing the Ion Source from the instrument. Two thumb screws on the Interface Connection Plate hold the sprayer arm in position inside the Ion Source. Loosening the thumb screws allows the Interface Connection Plate and the entire IonSpray arm assembly to slide away from the Vacuum Interface and the Ion Source Housing. The assembly cannot be removed from the Source Housing completely, it slides from the housing just enough to provide access to the inlet plumbing.

There are high voltage interlocks that restrict access to the Ion Source while high voltages are engaged. The interlocks are activated when the Interface Connection Plate is opened or the Ion Source is removed. When activated the interlocks disable the instrument high voltages. The turbo pumps and the vacuum system are not affected by the source interlocks.



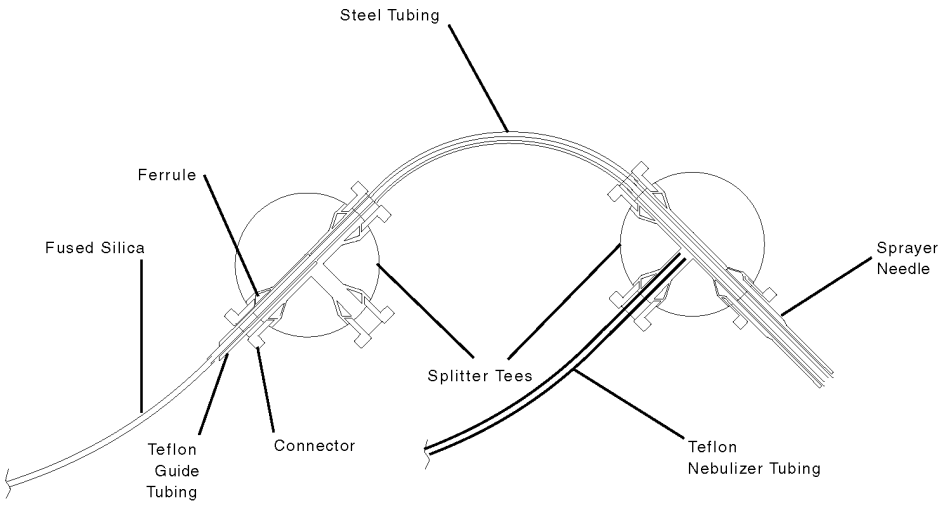
WARNING! Disconnect the IonSpray high voltage cable from the ion source by pulling on the connector, not the wire, before sliding the IonSpray inlet assembly from the housing.

IonSpray Arm

The Sprayer Arm is attached to the top of the Ion Source Housing using an electrically isolated mount. The IonSpray Arm is adjustable in three directions by use of the controls mounted on the top of the IonSpray housing.

- Side to side across the orifice. (X Axis)
- Up and down relative to the orifice. (Y Axis)
- In and out towards the orifice. (Z Axis)

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Sprayer Arm

The Sprayer Arm uses a two “Splitter Tee” design. The back Hold-Down Tee secures the sprayer fused silica and the electrode tube in place. The front Sprayer Tee also secures the electrode tube in place as well as providing the inlet for the nebulizing gas and holding the nebulizer tube.

The nebulizer tube at the end of the sprayer arm aims at an angle approximately 65° relative to the Vacuum Interface. There are three possible spray angle settings for the sprayer tee. The angle is set by positioning the sprayer tee indexing pin in one of the three locating slots in the sprayer arm. We recommend that you locate the sprayer tee in the middle position.

CAUTION! With the sprayer tee located in either of the outside positions, the sprayer angle relative to the Curtain plate is not optimum. It may result in the sprayer nozzle coming in contact with the curtain plate. Such contact, given the high voltages and the fragility of the sprayer nozzle, may cause damage to the Sprayer tip.

API users have successfully adapted the two-tee IonSpray design to provide a coaxial liquid sheath flow. This may be used when the liquid flow entering the source is below the optimum IonSpray flow rate (e.g., below 1 µL/min) or when the liquid composition is not ideal (e.g., 100% water). The coaxial sheath flow, or make-up flow, increases the total IonSpray flow or changes the composition of the liquid to more ideal conditions which may increase sensitivity.

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IonSpray Source Installation

Mounting the IonSpray Source



WARNING! The ion source voltage connection must be connected after the ion source is installed and the inlet assembly and interface connection plate are secured in position.

To install the IonSpray Ion Source:

1. Place the mounting hook on the Ion Source over the pin on the top of the Vacuum Interface and position the Ion Source over the Source Interlock Assembly.

CAUTION! Watch that the sprayer nozzle does not touch the Curtain Plate when mounting the Ion Source. The nozzle is fragile and can be damaged.

2. Turn the thumbscrews on both sides of the Vacuum Interface counter-clockwise until they are snug and the Ion Source is secure in position.
   Ensure that the Interface Connection Plate is snug against the Source housing, and the two thumbscrews which secure the assembly to the housing are tight.
3. Insert the male end of the Ion Source High Voltage (HV) connection into the high voltage connector through the bottom of the Ion Source Housing.
4. Insert the other end of the HV cable into the Instrument Bulkhead connector.
5. Connect the nebulizer gas tubing (Gas 1 Supply) from the Interface Panel to the Nebulizer fitting on the Interface Connection Plate.
6. Connect the sample flow to the source fitting LC INLET.

You have now installed the IonSpray source. Complete the following steps to remove the IonSpray source from the API instrument.

To remove the IonSpray Ion Source:

1. Stop all scans and place the instrument on **Standby** or **Overnight Quit**.



WARNING! High Voltage Risk. Remove the high voltage connector from the instrument prior to removing the high voltage connector from the ion source housing.

2. Disconnect the sample flow tubing from the source fitting LC INLET.
3. Disconnect the nebulizer gas tubing (Gas 1 Supply) from the Interface Panel and the Nebulizer fitting on the Interface Connection Plate.
4. Remove the male end of the Ion Source voltage connection from the High Voltage connector through the bottom of the Ion Source Housing.



WARNING! The ion source voltage must be disconnected by pulling on the connector, not the wire, before the connection plate and sprayer assembly can slide from the ion source housing.

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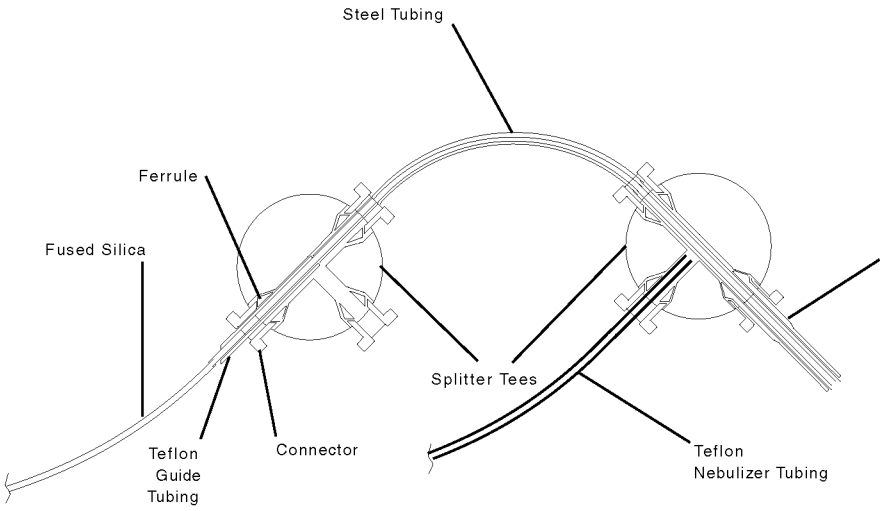
5. Turn the thumb screws on both sides of the Vacuum Interface clockwise until the Ion Source is loose.
6. Lift the mounting hook from over the pin on top of the Vacuum Interface and remove the Ion Source from the Source Interlock Assembly.

CAUTION! Watch that the sprayer nozzle does not touch the Curtain Plate when removing the Ion Source. The nozzle is fragile and can be damaged.

You have removed the IonSpray Ion Source from the instrument.

Installing the Fused Silica Sprayer

The sample is fed by an unbroken piece of fused silica tubing (typically 150 to 180 µm OD and 50 to 100 µm ID) from the zero volume connector on the Interface Connection Plate through the electrode tube to the tip of the sprayer. The silica tubing is fed through the hold-down tee attached to the sprayer arm. The hold-down tees primary function is to secure the silica tubing and sprayer electrode tube in position. It can be used as a sample splitter; however, another more accessible splitter is located on the outside of the Interface Connection Plate.



IonSpray- Sprayer Setup

The fused silica tubing should be replaced if the spray and/or the signal stability becomes poor. The frequency of this occurrence will depend on the amount and type of usage. The fused silica should also be changed to match the inner diameter of the tubing with the solvent flow rates being used.

To install the fused silica tubing:

1. Loosen the two thumbscrews that hold the Interface Connection Plate in place and slide out the sprayer arm assembly.
2. Loosen (but do not remove) the hold-down tee and zero volume connector nuts and remove any old fused silica tubing by pulling it out of the hold-down tee.

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3. Obtain a fresh piece of fused silica tubing (at least 30 cm long) and slide it through the hold-down tee nut until it protrudes from the end of the electrode tube tip.
4. Remove the zero volume connector nut and place the free end of the fused silica into the Teflon Tubing.
5. Ensure that the fused silica is flush with the connector nut Teflon Tubing and place in back into the zero volume connector. Finger tighten the nut and, if necessary, use a wrench to tighten the nut so that the fused silica is leak tight.
6. Adjust the protrusion of the fused silica relative to the electrode tube by manipulating it at the hold-down tee nut.

   NOTE: The position of the silica tubing in relation to the end of the electrode tube is a matter of preference, and a factor in ion source optimization. Initially, the silica tubing should be positioned flush with the electrode tube which itself protrudes about 1.0 mm past the nebulizer tube. As a matter of preference, the silica can be adjusted to be slightly inside or to stick out slightly past the electrode tube.

7. Once the fused silica is in position, tighten the hold-down tee nut so that the fused silica is secure and will not move easily.
8. Slide the Interface Connection Plate and sprayer arm assembly back into the source housing and tighten the two thumb screws.

High Voltage Connection



WARNING! Variable high voltages exist in the IonSpray inlet. Do not operate or service the instrument without the proper protective covers installed while voltages are applied.

High voltage (HV) from the Ion Source Power Supply, located on the HV Power Supply Board, is fed to the sprayer arm through a connector on the bottom of the Ion Source Housing. The High voltage wire lead is threaded directly to the sprayer arm from the connector. The variable voltage set by the Operator at the Applications Computer can range from 0 to ±6 kV at currents up to 100 µA. The applied voltage may be controlled either by its voltage or its current based upon the type of source which is installed. Using the IonSpray source, the applied high voltage is operator regulated by its voltage setting. The System Controller automatically sets the control mode based on the information received from the Source Interlock Assembly circuit board as to the type of Ion Source installed.

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IonSpray Optimization

IonSpray performance depends on the following factors:

- Sprayer Position
- IonSpray Voltage
- Nebulizer Gas
- Curtain Gas
- Declustering Potential and Focusing Potential Voltages
- Sprayer Fused Silica Line
- Sample Solvent Composition and Flow Rate
- Source Exhaust Pump

Optimum performance is relatively easy to achieve and little adjustment or tuning is required once the Source is optimized.

With experience, you will develop a personal optimization method which works best for you. In general, once the sprayer is set up and optimum factors have been determined, little or no readjustment of the values is required on a day-to-day basis if the same types of samples are analyzed. If the sprayer is removed, it is recommended that you re-optimize the sprayer and the gas flows after it is re-installed.

For tuning purposes, a compound with a known ion should be introduced either by continuous infusion or flow injection. The tuning compound should have characteristics similar to the sample to be analyzed during normal operation and should be introduced at the same liquid flow rate.

NOTE: The optimization procedures require the use of the Tune component of the Analyst software application. If you are not familiar with the Analyst application, refer to the Analyst Manual before proceeding with the optimization procedures.

Use Analyst to create a Q1 MI method, (for example, monitor the ions of interest with a 200 msec dwell time and a 5 minute duration). View the results in a chromatographic plot (intensity vs. time) and monitor the ion intensity to achieve a maximum value as you make iterative adjustments to the following factors which impact on the IonSpray efficiency.

Sprayer Position

CAUTION! When optimizing the Sprayer position make certain not to spray directly down the orifice. Spraying down the orifice may contaminate the Vacuum Interface and Vacuum Chamber ion optics components and could impact instrument performance.

CAUTION! To avoid possible damage to the sprayer tip, always view the sprayer tip through the side port when adjusting its position.

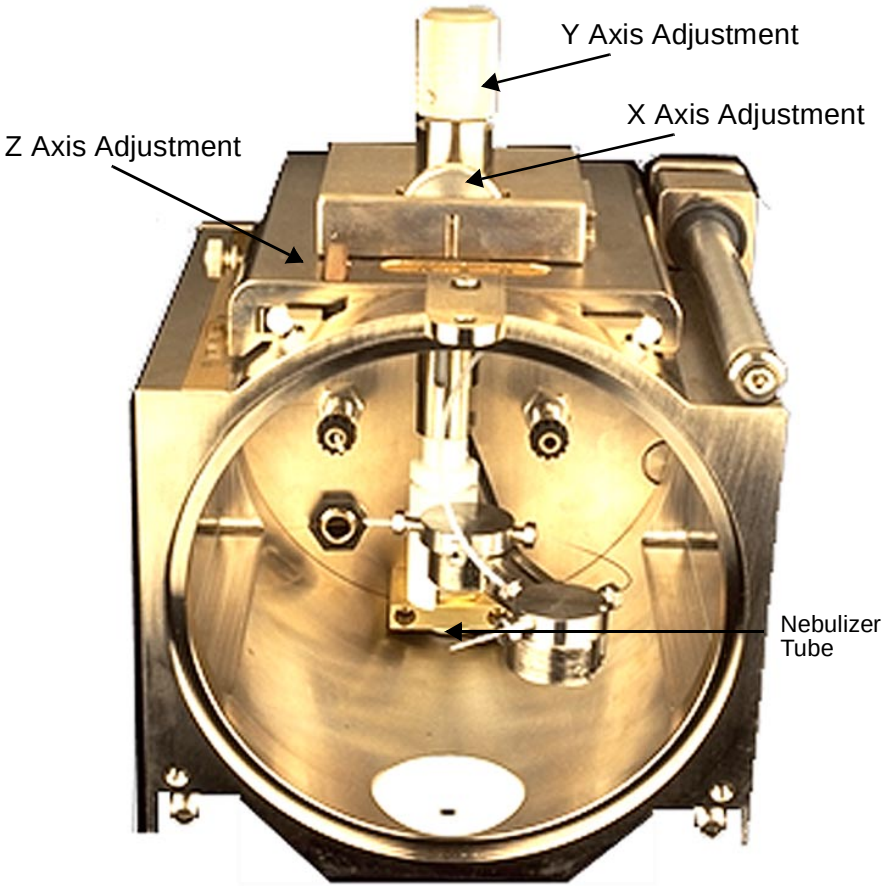
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The IonSpray Arm is adjustable in three directions by use of the controls mounted on the top of the IonSpray housing.

- Side to side across the orifice. (X Axis)
- Up and down relative to the orifice. (Y Axis)
- In and out towards the orifice. (Z Axis)

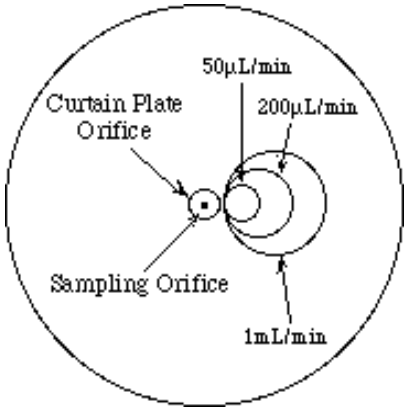
The sprayer should always be moved in small increments and checked to see that it is not spraying directly at the curtain plate orifice. Spraying directly down the orifice may cause severe instrument contamination and subsequent loss in performance. Looking through the opposite side window of the source down the axis of the sprayer will provide the best view of the spray trajectory.

In general, a good initial position should have the sprayer point 5 to 8 mm to the right edge of the curtain plate orifice when using low flow rates (below 50 µL/min). Higher flow rates will require the position to be further away from the orifice and optimized by flow injection analysis (FIA). Refer to the figure Spray Patterns at Different Flows. The curtain plate orifice should remain clear of solvent or solvent drops at all times.



The IonSpray Source

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Spray Patterns at Different Flows

IonSpray Voltage (IS)

The working range of the IonSpray voltage (IS) is typically 3500 to 6000 V in positive ion mode and -3000 to -5000 V in negative ion mode. When performing an infusion (e.g., 5 µL/min) optimization, the IonSpray voltage can be ramped in value to obtain the optimum value. For example, ramp the voltage from 3500 to 6000 V using a 50 V increment. Higher flow rates will require a flow injection analysis (FIA) optimization of the voltage.

NOTE: If the IonSpray voltage is set too high, a blue glow indicating corona discharge may be noticeable on the tip of the sprayer. Corona discharge may result in a decrease in ion stability. If this occurs, lower the voltage to minimize the discharge.

Nebulizer Gas (NEB)

The nebulizer gas (GAS 1) should be optimized for signal stability and sensitivity. This gas can be supplied from a bottle or gas generator of zero grade air or nitrogen at a pressure of 90 to 100 psi.

NOTE: The use of air instead of nitrogen is recommended when performing negative ion analyses since air will assist in reducing any corona discharge.

To optimize, increase and decrease the NEB gas setting over a wide range (typically 2 to 15) until the best stability and sensitivity is achieved.

Curtain Gas (CUR)

This gas can be supplied from a bottle or gas generator of ultra-high purity nitrogen (99.999%) at a pressure of 60 psi.

CAUTION! Do not operate the instrument with a curtain gas setting of less than 6 otherwise instrument damage may result.

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The main function of the curtain gas is to prevent the contamination of the ion optics. As a general rule, the Curtain Gas flow should be set as high as possible without reducing the signal significantly but never below a setting of 6. To optimize, start at a setting of 6 and increase until a loss in signal is observed.

NOTE: The IonSpray should be operated with the curtain gas setting adjusted to the highest flow rate possible without significant signal loss.

Declustering Potential (DP) and Focusing Potential (FP) Voltages

The Declustering Potential (DP) and Focusing Potential (FP) voltages control the amount of declustering and fragmentation in the orifice-skimmer region of the vacuum interface. These voltages should be set high enough to reduce chemical noise but low enough to avoid fragmentation. A good starting point is to set the Declustering Potential to 30V and the Focusing Potential to 300V. If an excessive amount of fragmentation is observed, these voltages can be reduced.

NOTE: The fragmentation energy of a compound is a function of its structure and molecular weight. Generally, lower molecular weight compounds require less energy and thus lower Declustering Potential and Focusing Potential voltages to induce fragmentation.

Sprayer Fused Silica Tubing

The fused silica tubing should be replaced if an unstable signal or no signal is observed as a result of a blockage in the line. The frequency of this occurrence will depend on the amount and type of usage. Before attempting to replace the fused silica, the following procedures should be considered:

In the event of an unstable spray:

1. Loosen the hold-down tee nut (but do not remove) such that the fused silica moves freely within the electrode tube.
2. Use a silica cutter to cut a fresh sprayer tip at the end of the silica tube.
3. Re-position the cut fused silica within the electrode tube and tighten the hold-down tee nut.

In the absence of spray (blocked Fused Silica):

1. Recut the tip of the sprayer line Silica as described above.
2. Recut the end of the fused silica at the point where it connects with the zero volume union on the Interface Connection Plate.

If the two procedures outlined above do not solve the stability or blockage problem, the procedure for the changing the fused silica tubing should be followed.

The fused silica can be changed to better match the inner diameter (ID) of the tubing with the solvent flow rates being used. For example, if a lower flow rate (below 50 µL/min) is required, a 50 µm ID fused silica can be used to reduce void volumes and increase spray stability. The 50µm ID silica can be used with flow rates as high as 200 µL/min, but an elevated back pressure may result. If the back pressure is too high or higher flow rates are

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required, then 75 or 100 µm fused silica should be used. In all cases, the outer diameter of the fused silica should be less than 190 µm in order to fit inside the electrode tube.

Sample Solvent Composition and Flow Rate

The sensitivity of the IonSpray source is affected by the composition and flow rate of the sample. In general, a high organic solvent content and low flow rates (50 µL/min and lower) produce the highest ion signal intensities. Commonly used organic solvents include acetonitrile, methanol and propanol.

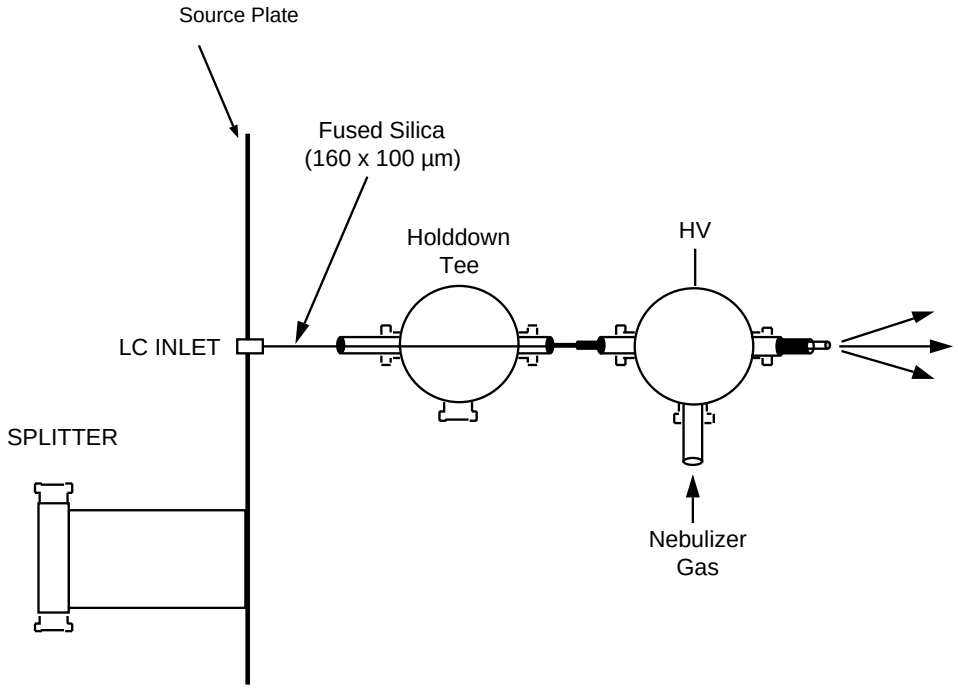
The addition of small amounts of acids or bases may also increase ionization efficiencies. For example, the addition of 0.05% to 0.5% acetic or formic acid to samples or LC mobile phases in positive ion mode is common. In negative ion mode, 1 to 20 millimolar ammonium formate or ammonium acetate is used. Mobile phase modifiers such as triethylamine (TEA), sodium phosphate, trifluoroacetic acid (TFA) and sodium dodecyl sulfate are not commonly used because they complicate the mass spectrum with their ion mixtures and cluster combinations. They may also suppress the strength of the target compound ion signal.

The IonSpray source can produce stable ion signals for a wide range of sample flow rates from less than 1 µL/min to greater than 200 µL/min. Operating at higher flow rates is possible although the sensitivity is usually poor. In these situations, reducing the sample flow entering the source by using a post-column splitter will dramatically improve the signal intensities. The IonSpray source has a built-in, low dead volume splitter for use when flow splitting is desired, making IonSpray directly compatible with most analytical and preparative HPLC columns and post column fraction collectors. The following sections describe how the IonSpray source can be configured to accommodate a direct coupling (with no splitting) and a high flow rate post-column splitting coupling.

Direct Coupling - No Splitting

In this configuration, the liquid sample tubing connects directly to the bulkhead fitting labeled LC INLET on the connection plate. The entire sample flow is fed directly by a continuous piece of fused silica from the Ion Source connection plate through the hold-down tee and the nebulizer tee to the tip of the sprayer. This is the standard IonSpray configuration.

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IonSpray Connections Without Sample Splitter

This configuration is most useful for the following types of experiments:

- LC/MS at 50 µL/min or less (using a 1 mm column), with the entire effluent going to the nebulizer.
- Flow Injection Analysis (FIA) under the above conditions.
- Direct infusion from a syringe pump using flow rates below 50 µL/min.

Flow rates as high as 200 µL/min can also be used successfully with a direct connection, although higher sensitivities would most likely be achieved with splitting (described in the next section).

Post-Column Splitting

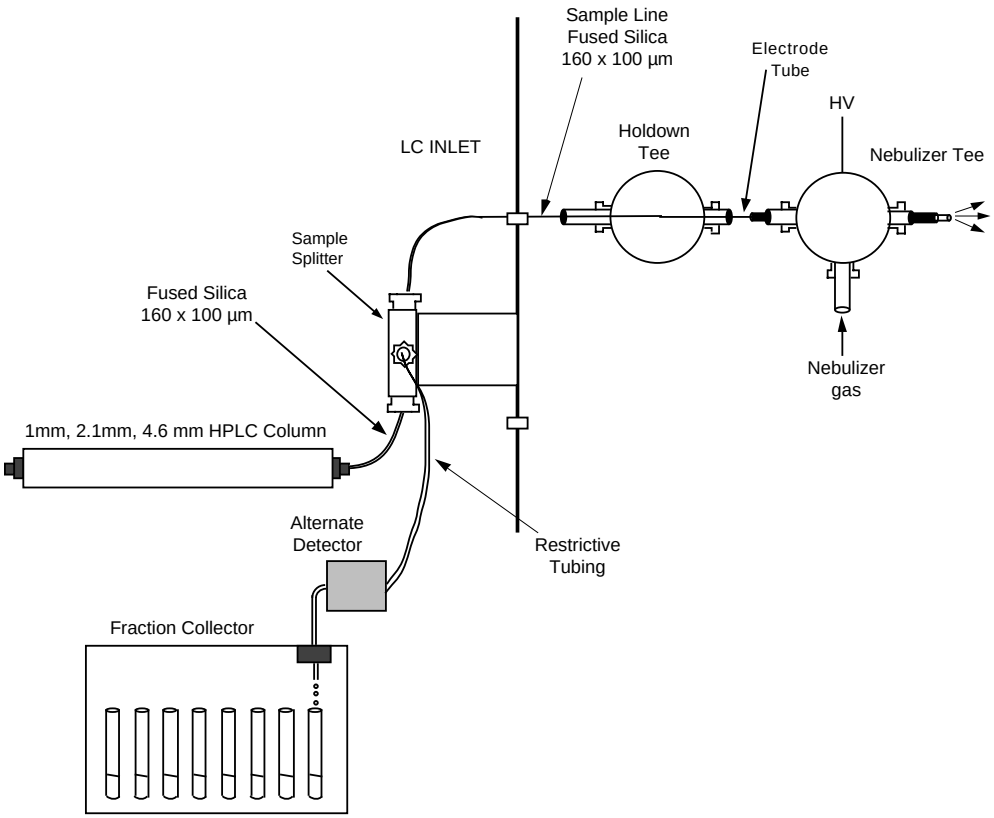
The IonSpray source has a low dead volume splitter conveniently attached to the front of the source connection plate for use when flow splitting is desired. The IonSpray source can operate with good performance at flow rates as high as 200 µL/min. However optimum performance is usually achieved with flow rates of 50 µL/min or less.

To split the sample flow:

1. Loosen the two thumb screws that hold the Interface Connection Plate in place and slide out the sprayer arm assembly.
2. Extend the sprayer fused silica tubing through the zero volume connector on the source plate and connect it to the top of the sample splitter tee.
3. Connect the sample flow or LC column outlet to the bottom of the sample splitter tee.
4. Connect a piece of restrictive tubing, fused silica or PEEK, to the last port on the splitter tee.

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The restrictive tubing is used to control the back pressure, and thus the sample split ratio. Changing the length and diameter of the restrictive tubing will change the split ratio.



IonSpray Connections Using the Splitter

While the exact split ratio is not critical, it can be determined by collecting and measuring the split flow incrementally as you shorten the restrictive tubing. It is possible to achieve split ratios of 10:1 or 20:1 with convenient lengths of restrictive tubing.

NOTE: The split flow can be connected to an alternate detector (e.g. UV detector) or used to collect fractions from LC peaks.

CE-MS Connection

The flow rates produced in conventional Capillary Electrophoresis (CE) separations are typically very low (e.g., below 200 nL/min). These flow rates are too low for the IonSpray source and therefore require a make-up flow to increase the total liquid flow into a range that is more suitable. There are a few methods for connecting a CE system to the IonSpray source. For a comparison of these methods refer to the paper entitled *Comparison of Liquid-junction and Coaxial Interfaces for Capillary Electrophoresis-Mass Spectrometry with Application to Compounds of Concern to the Aquaculture Industry* (Pleasance, S., Thibault, P., Kelly, J.; Journal of Chromatography 591(1.2) 1992 325-339).

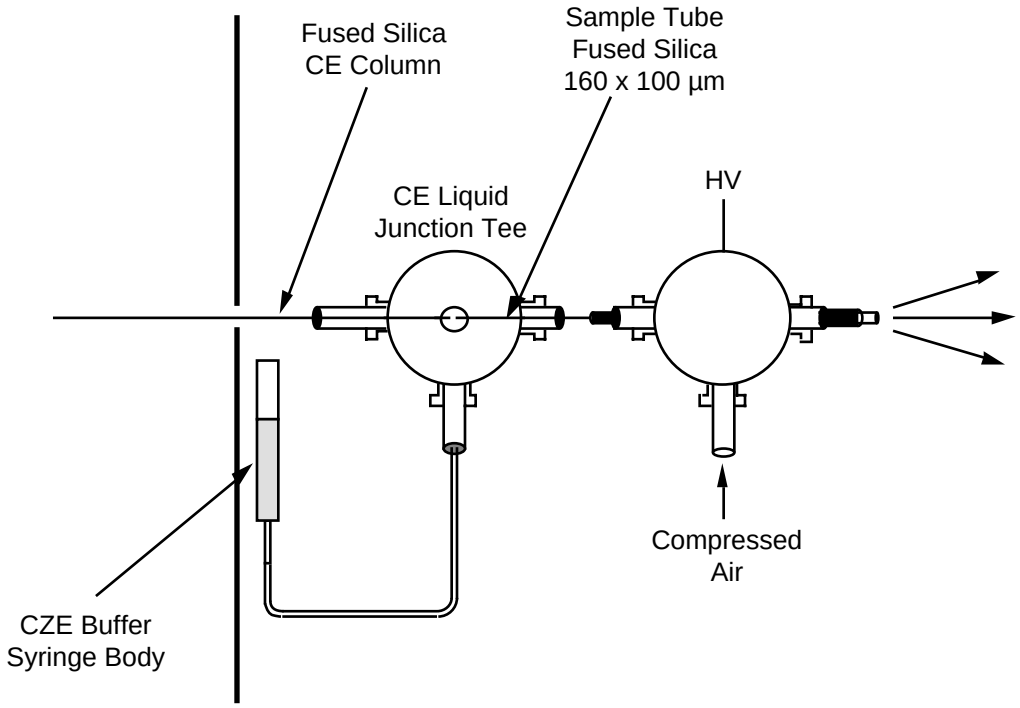
In order to use the Liquid Junction CE Interface, the IonSpray hold-down tee must be replaced with the optional Liquid Junction tee. The liquid junction tee has a small lens to allow accurate positioning of the CE column and the sample tubing connecting to the sprayer. For proper CE operation, the silica tubing from the CE column must connect

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directly to the Liquid Junction tee without interruption at the bulkhead connector (see IonSpray Connection to Capillary Electrophoresis figure). The reason for this is that the CE column (usually fused silica tubing) must maintain a potential difference between the CE power supply and the liquid junction tee. If the silica tubing is connected to the source plate, which is grounded, the potential difference may be interrupted.

At the liquid junction tee, the two ends of the silica tubes, the CE column and the sample tubing connecting the liquid junction output to the sprayer, must be aligned with a minimal gap of less than 100 µm. This may require removing the tee and positioning the column and connecting tubing under a microscope.

The CE Liquid Junction tee is connected to the make-up line and the CE make-up syringe mounted on a syringe pump. The syringe should be filled with a buffer solution that is conductive and contains some organic solvent (e.g., 1:1 water/methanol with 0.2% acetic acid) and set to flow between 2 to 10 µL/min. In order not to disturb the separation in the CE column, it is important to ensure that the CE column entrance and exit are at about the same height.



IonSpray Connection to Capillary Electrophoresis



WARNING! The make-up line should be grounded at the source plate. If not grounded the voltage at the make-up source may float as high as the IonSpray inlet voltage.

The usable CE current through the CE column is limited by the internal load resistor on the Ion Source High Voltage Power Supply Board, which supplies the voltage for the IonSpray. At a sprayer voltage of about 5kV a CE current of 100µA can be safely applied to the CE column. Running the Ion Source with CE currents exceeding the safe limit may cause a current overload on the ion source high voltage power supply module.

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CAUTION! It is important that the CE current through the column be maintained at a level which does not overload the Ion Source voltage supply on the High Voltage Power Supply module. CE currents in excess of 100µA may damage the instrument electronics.

Source Exhaust Pump

The source exhaust pump removes harmful solvents and chemicals from the source to an externally vented system such as a fume hood. The pump should be optimized for best sensitivity while maintaining the minimum source pump-out set point as indicated by the instrument status bar on the computer. Optimum sensitivity is usually obtained when the following method for adjusting the source pump is used:

- Reduce the source exhaust (by turning the control valve on the source cover counter clockwise,) until the fault light turns red on the computer status bar (there is a slight delay, about 5 seconds, in the computer read-back).
- Increase the source exhaust in small increments (by turning the control valve clockwise) until the red light is replaced with a “Ready” green light on the status bar.
- Increase the exhaust by another half turn (clockwise) on the control valve. Once the exhaust has been set, little or no further adjustments are required when changing sources, liquid flow rates or methods.



WARNING! The source exhaust pump must be vented to either an external fume hood, or external source.

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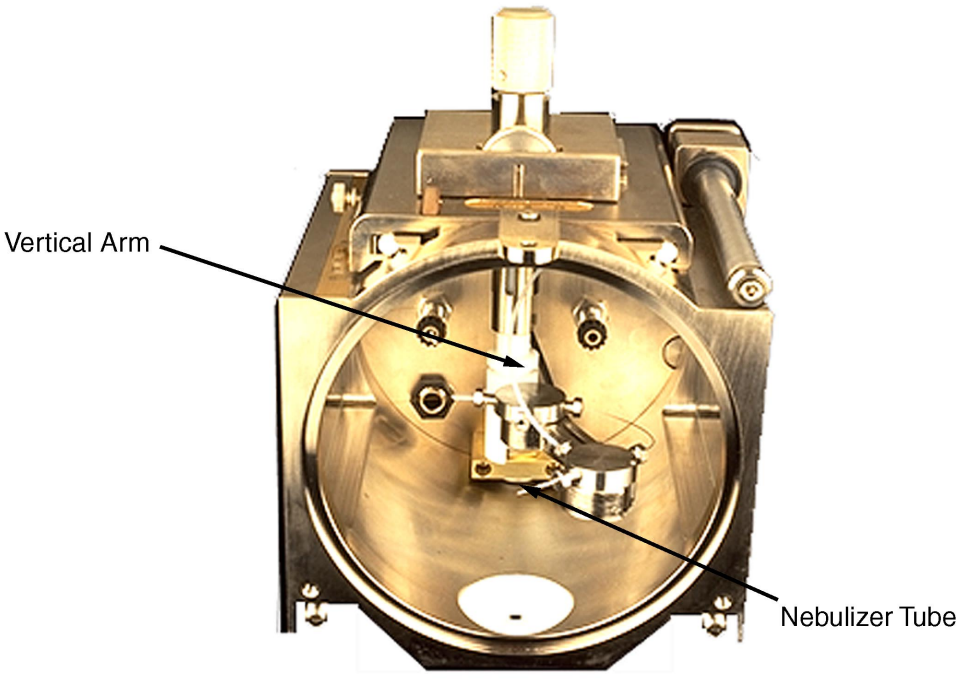
Appendix A: Arm Alignment

Alignment of the IonSpray Arm

The Sprayer Arm is attached to the top of the Ion Source Housing using an electrically isolated mount. The angle of the arm is factory set to provide the best range of movement while preventing the sprayer tip from coming into contact with the curtain plate. Use the following procedure to set the proper angle if the arm is moved or removed from the mounting post.

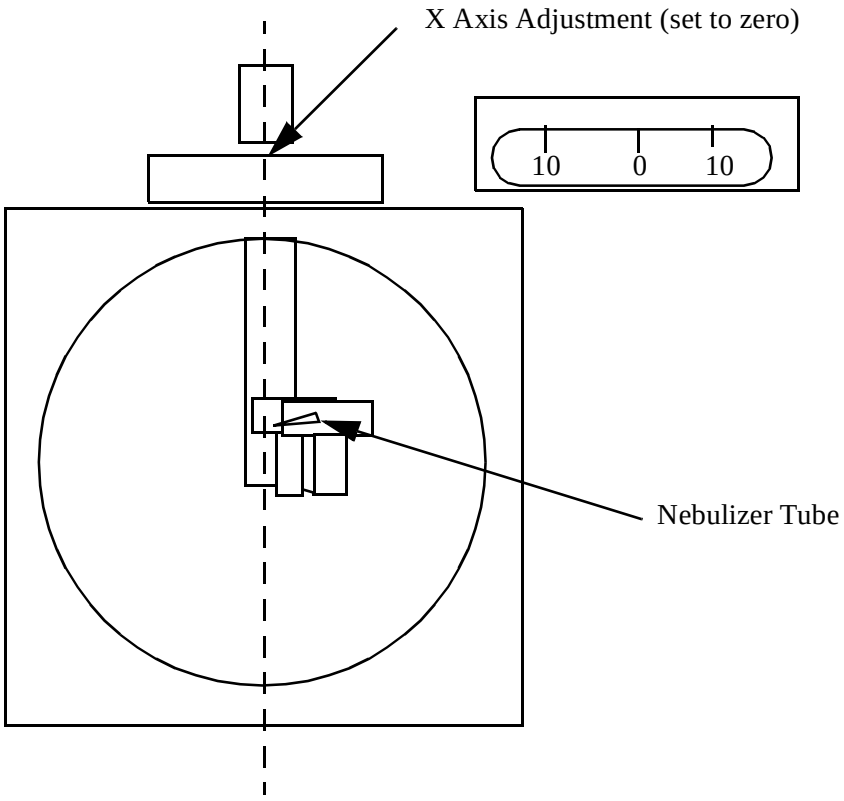
To set the proper angle:

1. Move the sprayer arm back along the Z axis such that the tip of the needle is flush with the front edge of the source.
2. Center the side-to-side (X axis) vernier at zero.
3. Using a ruler, measure the distance from the outside edge of the source to the sprayer needle. It should be approximately at the mid point of the source (7.6 to 7.9 cm from the outside edge of the source).
4. Adjust the angle of the arm if necessary. The M5 nut at the bottom of the vertical post may be loosened if the arm does not move smoothly.
5. Ensure that the M5 nut mounted at the bottom of the vertical post is tightened after the adjustment.



The IonSpray Source

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Alignment of the IonSpray Arm

Replacing The Electrode Tube

The electrode tube can degrade with time and continued use causing poor spray stability. This results from the sprayer end of the tube becoming corroded and uneven. If this occurs, the best and simplest solution is to replace the tube.

To remove the electrode tube:

1. Disconnect the high voltage lead from the bottom of the source housing by pulling on the connector, not the wire.
2. Loosen the two thumb screws that hold the interface connection plate and slide it back.
3. Loosen the hold-down tee nut that holds the sprayer fused silica in place and slide the silica tubing out of the electrode tube.
4. Loosen the hold-down tee and the sprayer tee nuts that hold the electrode tube in place and pull the tube out of the sprayer arm.

To install the electrode tube:

1. Slide one end of the electrode tube through the sprayer tee nut and ferrule so that it protrudes from the end of the nebulizer tube.
2. Gently bend the electrode tube and slide the other end through the hold-down tee nut and ferrule.

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3. Ensure that the electrode does pass through the hold-down tee ferrule (remove the nut to see the ferrule) and is pushed as far as possible into the tee.
4. Tighten the hold-down tee nut to secure the electrode tube in place.
5. Adjust the protrusion of the electrode tube from the nebulizer tube. It should protrude approximately 0.4 to 1.0 mm from the nebulizer tube.
6. Tighten the sprayer tee nut to secure the electrode tube in place.
7. Follow the steps outlined (in the IonSpray Source Installation section) to install the fused silica sprayer.

IonSpray Consumables: Part Numbers

PART	PART NUMBER
Fused Silica Tubing (160 OD x 100 µm ID)	011278
Electrode Tube	014991
Nebulizer Tube	015268
Electrode Tube Ferrule	011215
Teflon Tube	011555
PEEK Connectors	012627
PEEK Ferrules	012637

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