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APCI Heated Nebulizer

Source Manual

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API LC/MS and LC/MS/MS

APCI Heated Nebulizer Ion Source Manual

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APCI Heated Nebulizer Ion Source Manual

About This Manual

This manual contains the instructions required to operate the API Atmospheric Pressure Chemical Ionization (APCI) Heated Nebulizer 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.

Introduction

The Heated Nebulizer offers an alternative method of introducing samples to the API mass spectrometer. The Heated Nebulizer, much like the standard IonSpray source, generates ions representative of the molecular composition of the sample. Where the IonSpray source produces ions by the process of ion evaporation, the Heated Nebulizer vaporizes the sample prior to inducing ionization by a process called Atmospheric Pressure Chemical Ionization (APCI).

The Heated Nebulizer source produces ions by nebulizing the sample in a heated tube causing the finely dispersed sample drops to vaporize. This process leaves the molecular constituents of the sample intact. These molecules are ionized via the process of APCI, induced by a corona discharge needle, as they pass through the Ion Source chamber and into the interface region.

The following list outlines the features of the Heated Nebulizer ion source:

- Able to function with flow rates up to 2.0 mL/min and can handle the entire flow from a wide bore column without splitting.
- Able to vaporize a 100% aqueous mobile phase.
- Able to handle volatile mobile phase buffers.
- Able to vaporize volatile and labile compounds with minimal thermal decomposition.
- The simple APCI spectra is ideal for MS/MS.
- Capable of being used for rapid sample introduction by flow injection with or without an Liquid Chromatograph (LC) column.

Heated Nebulizer Components

The Heated Nebulizer Ion Source inlet is compatible with the PE SCIEX API 100 and API 300 Series Mass Spectrometers. The inlet requires that the Source Exhaust System of the API Mass Spectrometer be **ON** and operating to specification. If the Source Exhaust system is not working properly, the instrument power supplies are disabled. Further explanation of the Source Exhaust system is found in both the Operators' and Reference Manuals.

The Heated Nebulizer Ion Source consists of:

- Nebulizer vaporization chamber with replaceable quartz tube
- Heater with computerized temperature control and control circuit board
- Optional manifold bracket with pressure regulator and customer supplied sample injector(s)

Ion Source Temperature Range

- Probe temperature may be adjusted from 50° to 500°C

Liquid Chromatograph

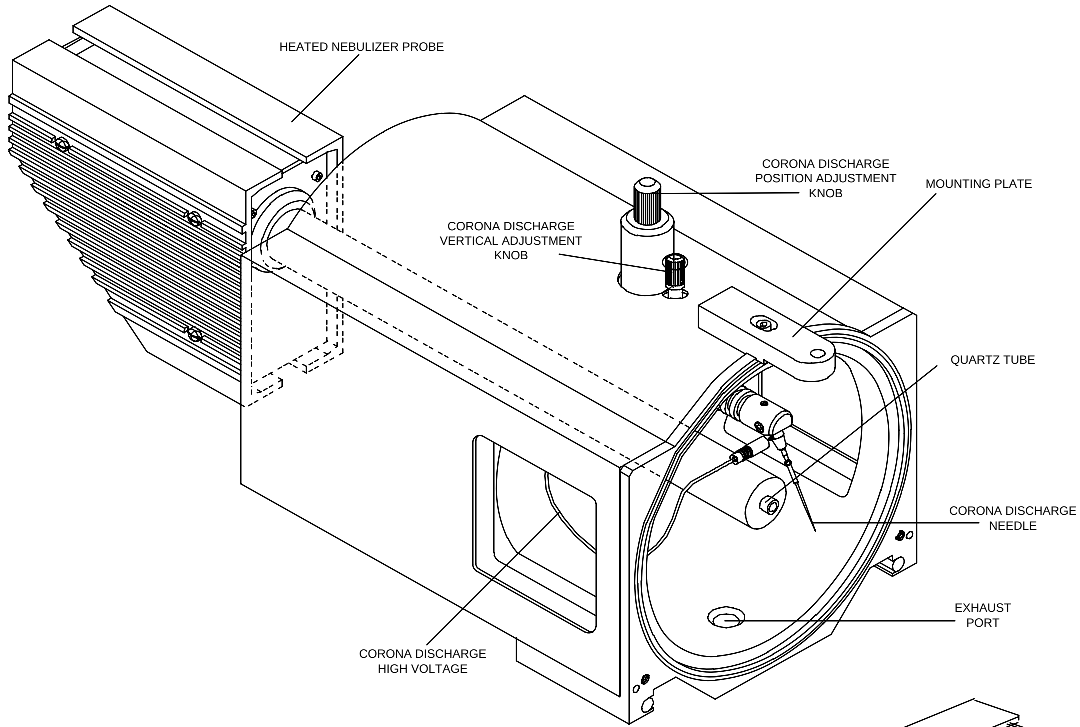
- Interfaces to any liquid chromatograph system

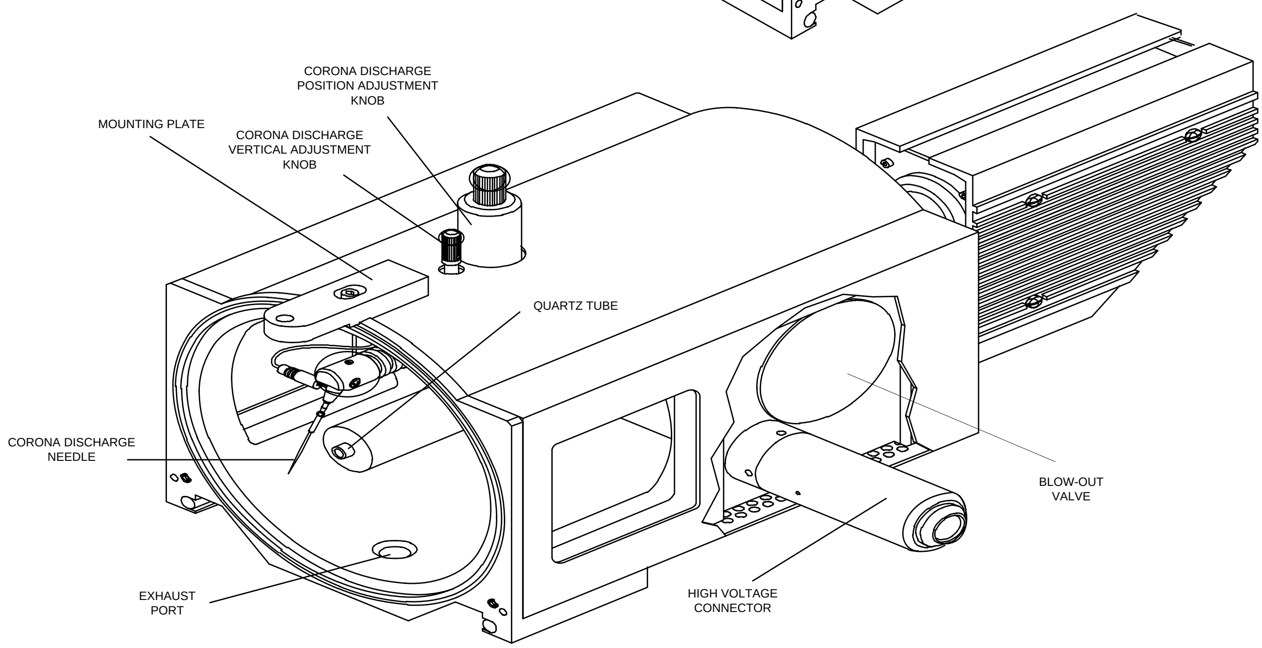
Auxiliary Gas [Gas1]

- Zero Grade Air or UHP nitrogen (99.999% purity) regulated to 90 psi

Nebulizer Gas [Gas2]

- Zero Grade Air or UHP nitrogen (99.999% purity) at 90 psi

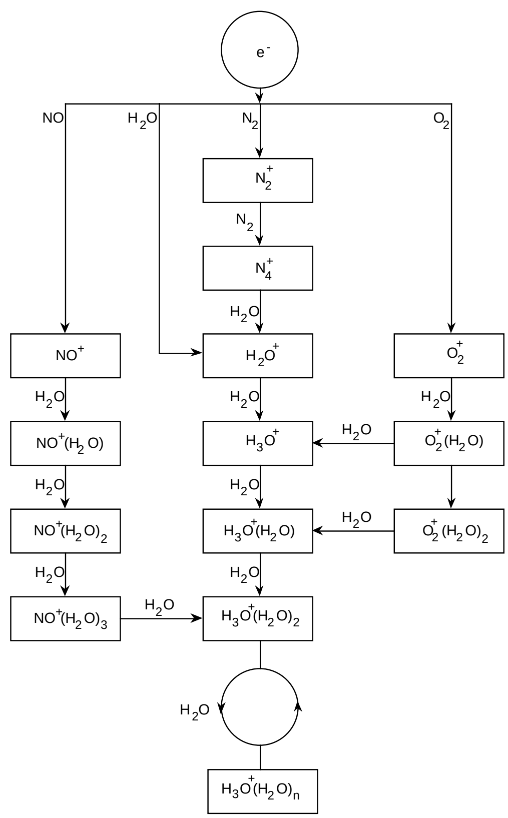

HEATED NEBULIZER PROBE
CORONA DISCHARGE POSITION ADJUSTMENT KNOB
MOUNTING PLATE
CORONA DISCHARGE VERTICAL ADJUSTMENT KNOB
QUARTZ TUBE
CORONA DISCHARGE NEEDLE
EXHAUST PORT
CORONA DISCHARGE HIGH VOLTAGE


CORONA DISCHARGE POSITION ADJUSTMENT KNOB
MOUNTING PLATE
CORONA DISCHARGE VERTICAL ADJUSTMENT KNOB
QUARTZ TUBE
CORONA DISCHARGE NEEDLE
EXHAUST PORT
HIGH VOLTAGE CONNECTOR
BLOW-OUT VALVE

Ionization Process

The basis for past incompatibilities of linking liquid chromatography (LC) with Mass Spectrometry (MS) arises from difficulties converting relatively involatile molecules solvated in a liquid into a molecular gas, without inducing excessive decomposition. The Heated Nebulizer process of gently nebulizing the sample into finely dispersed small droplets in a heated tube ensures rapid vaporization of the sample so that the sample molecules are not decomposed.

The following figure shows the reaction flow of the APCI process for reactant positive ions (the proton hydrates, $H_3O^+[H_2O]_n$). This sequence is derived from experimental results summarized by Huertas and Fontan[1]. The major primary ions N_2^+, O_2^+, H_2O^+ and NO^+ are formed by electron impact of corona-created electrons on the major neutral components of air. Although NO is normally not a major constituent of clean ambient air, the concentration of this species in the source is enhanced due to neutral reactions initiated by the corona discharge.



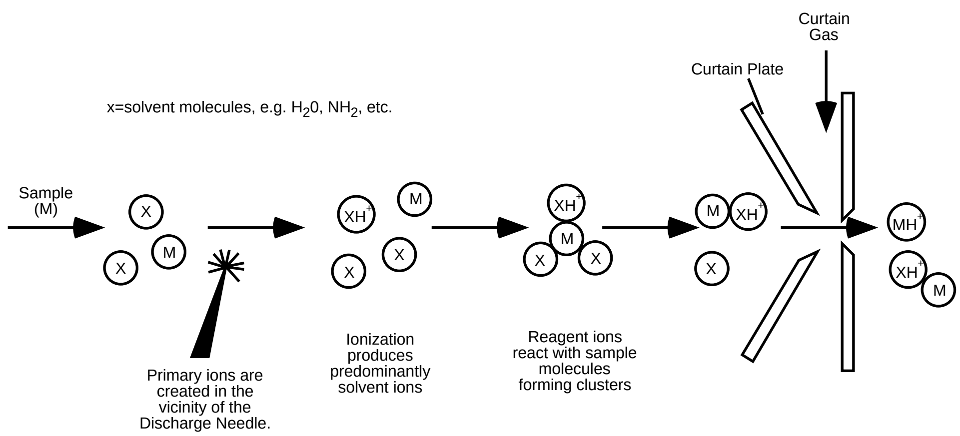
APCI Reaction Flow Diagram

Samples which are introduced through the Heated Nebulizer are sprayed with the aid of a nebulizing gas into a heated probe. Within the probe 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.

1. Huertas, M.L. and Fontan, J. (1975) Evolution Times of Tropospheric Positive Ions, *Atmospheric Environ.* 9, 1018.

The gaseous sample and solvent molecules are swept from the probe via a second gas flow (Auxiliary gas) into the ion source where the ionization by APCI is induced by a corona discharge needle. The sample molecules are ionized by collision with the reagent ions created by the ionization of mobile phase solvent molecules. 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 which through collision with the sample molecules produce stable sample ions.

The sample molecules are ionized by 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 “high” pressure of the API Source.



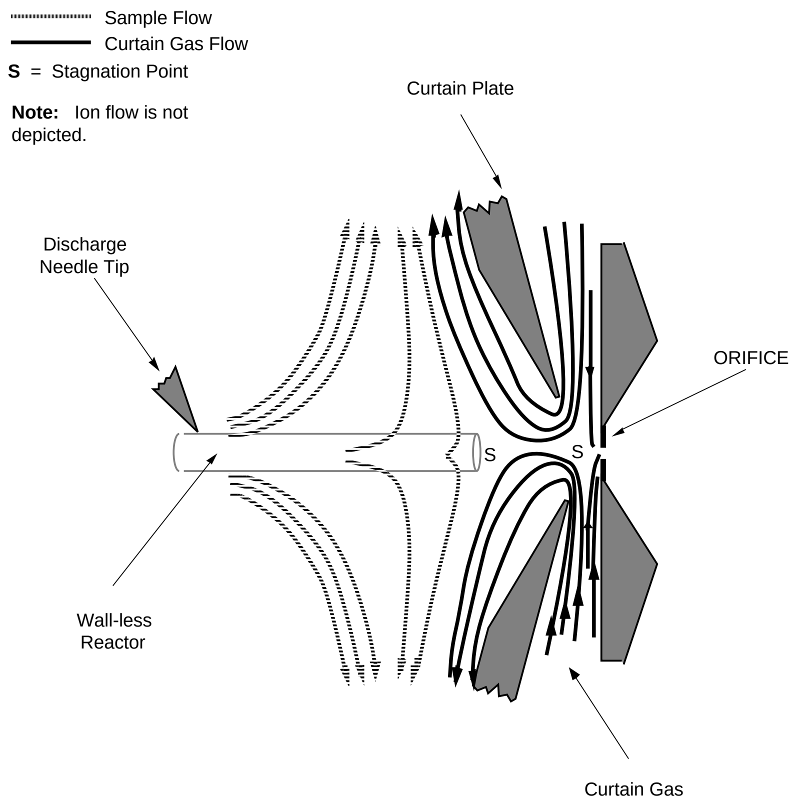
Atmospheric pressure Chemical ionization (APCI)

NOTE: 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, $(CH_3COO)^-$, the primary reagent in the negative mode. Ammonium modifiers may make protonated ammonia, $(NH_4)^+$, the primary reagent in the positive mode.

Through collisions, an equilibrium distribution of certain ions (e.g. protonated water cluster ions) is maintained. The likelihood of premature fragmentation of the sample ions in the ions source is reduced given 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.

Ionization Region

The general location of the ion-molecule reactor of the API Source is indicated by the dotted cylinder in the previous figure which constitutes 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, e.g., N_2^+ and O_2^+, are created by the loss of electrons which originate in the plasma in the immediate vicinity of the 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.



Ion Source APCI - Source Flow Streamlines

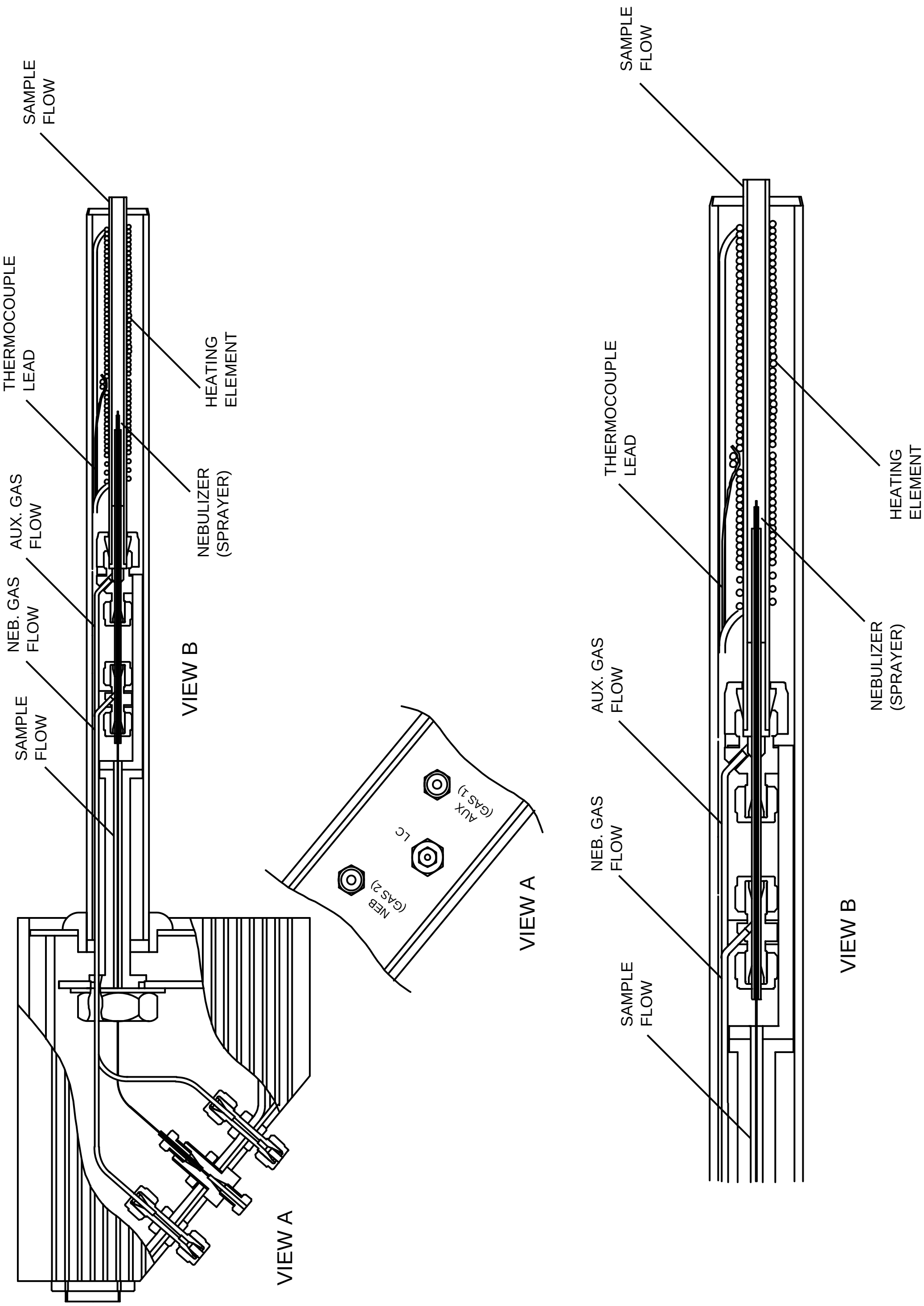
The primary ions, in turn generate intermediate ions which finally lead to the formation of sample ions. Ions of the chosen polarity drift under the influence of the electric field in the direction of the Curtain Plate and through the gas curtain into the mass analyzer. The whole ion formation process is collision dominated because of the “high” pressure of the API Source. Except in the immediate vicinity of the needle tip, where the electric field strength is greatest, the energy imparted to an ion by the electric field is small in comparison with its thermal energy.

Through collisions, an equilibrium distribution of certain ions (e.g. protonated water cluster ions) is maintained. Any excess energy which an ion may acquire in the ion-molecule reaction process is thermalized. Through the process known as collisional stabilization, many of the product ions are fixed even though many subsequent collisions occur. Both product ion and reactant ion formation are governed by equilibrium conditions at 760 Torr operating pressure.

NOTE: The API Source functions as a wall-less reactor since the ions which 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 API 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 Heated Nebulizer operation. In essence, 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. To preserve the molecular identity the temperature of the probe must be set to ensure rapid evaporation. Operating the Heated Nebulizer at temperatures above the optimal temperature may cause thermal decomposition of the sample.

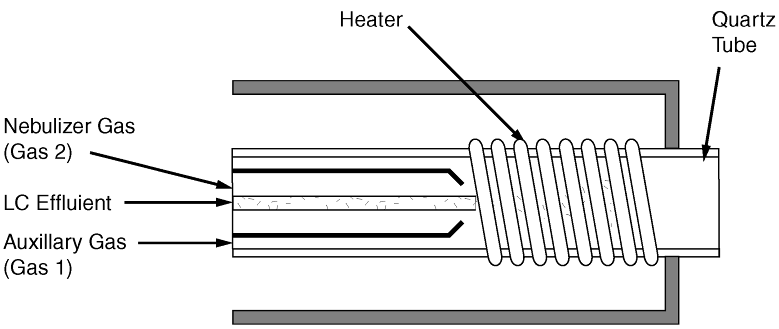
Inlet Description



Heated Nebulizer Probe Cross Section

The sprayer probe consists of 0.010" (120 µm) ID stainless steel tubing surrounded by a flow of Nebulizer Gas. The liquid sample flow is pumped through the sprayer where it is nebulized into a quartz tube surrounded by a heater. The inner wall of the quartz tube is maintained at a temperature of about 100 to 150°C. When the liquid sample is pumped into the quartz tube, the sample and solvent are vaporized. A flow of auxiliary gas (Gas 1) surrounds the sprayer carrying the vaporized sample through the quartz tube into the ionization region in the Ion Source.

The liquid sample is introduced through a Zero Volume LC fitting on the probe handle, from where it flows by stainless steel tubing to the tip of the sprayer. A high velocity jet of nebulizer gas flows coxially over the sprayer to disperse the sample as a mist of fine particles. The nebulizer gas is supplied through a 1/8” Swagelok fitting on the Heated Nebulizer handle. A flow of Auxiliary gas (Gas 1) sweeps the sample mist through the quartz vaporization tube into the reaction region of the ion source past the corona discharge needle where the sample molecules are ionized.



Heated Nebulizer Schematic

The probe temperature is maintained by a heater coil wrapped around the outside of the quartz tube. The power to the heater, and as a direct result the heater temperature, is controlled by the Temperature Control Board (TCB) mounted inside the instrument. The TCB adjusts the flow of power to the heater element as a function of the difference between the actual heater temperature and the temperature setting at the Applications Computer. The probe temperature is monitored by a thermocouple connected directly to the heater element.

NOTE: The temperature is controlled by monitoring the output of a thermocouple connected to the heater surrounding the quartz tube. At the temperature control board the thermocouple output is compared with the temperature setting, the difference determines the power flow to the heater.

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

Corona Discharge Characteristics

The corona discharge in the Heated Nebulizer Ion Source is formed by three major electric fields and fluid flow elements:

1. Corona Discharge Needle
2. Curtain Plate
3. Orifice Lens

The purpose of the corona discharge is to produce ionization of the trace species or sample gas. Primary ions, which are formed as a result of the discharge, are converted by collisional processes to final ion-molecule reaction products.

The operator has the ability to set the Corona Discharge setting at the Application Computer by adjusting the value of NC (normally set to 2) in the Analyst application.

Installation

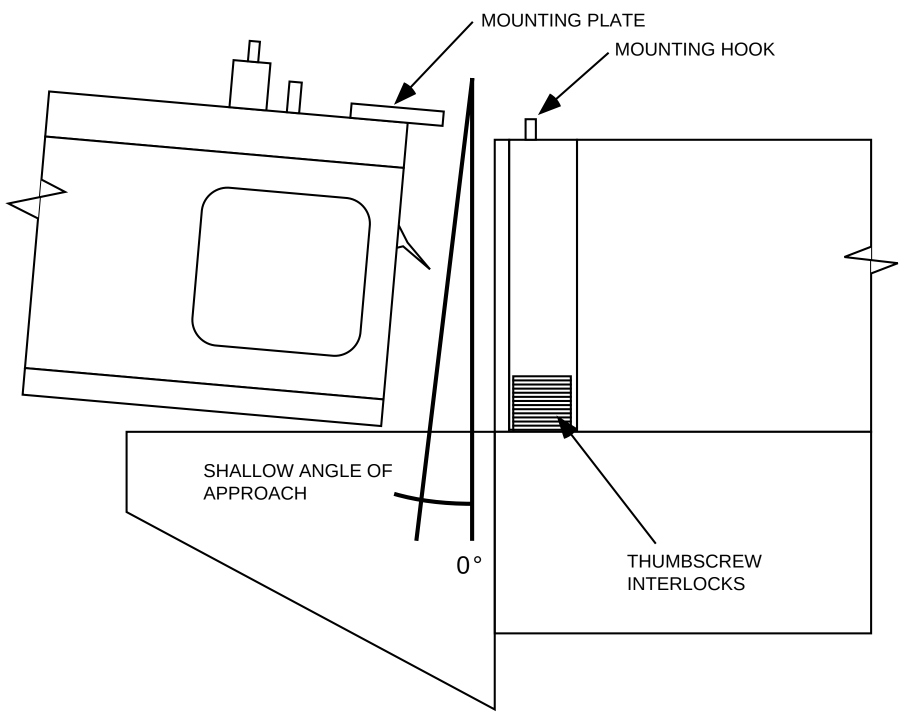
The Heated Nebulizer ion source, like the standard (not Turbo) IonSpray source, hooks to the top of the Vacuum Interface housing. Two thumbscrews mounted in the Vacuum Interface housing screw into the Heated Nebulizer to secure it in position against the vacuum interface to create an air-tight seal.

WARNING! Some surfaces on the Heated Nebulizer source will become hot during operation. Use caution when installing or removing the source or the heated probe.

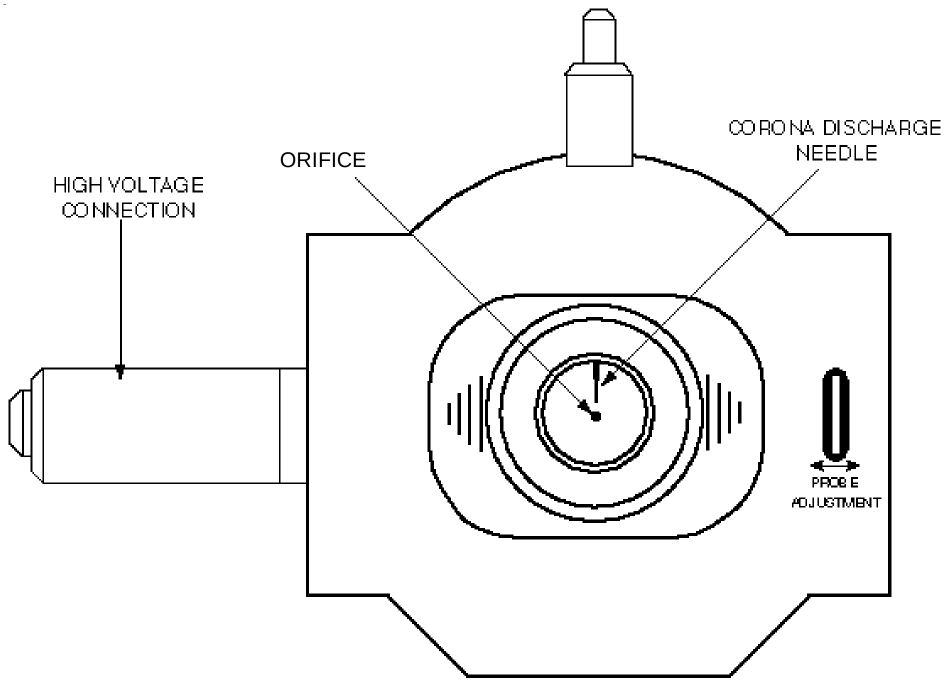
To install the Heated Nebulizer on your API instrument:

1. Install the Corona Discharge Needle into the needle chuck (friction fit).
2. Place the mounting plate on the Atmospheric Pressure Chemical Ionization (APCI) source housing over the hook on the top of the vacuum interface.
3. Align the Corona Discharge Needle as shown in the figure *Ion Source Mounting - Angle of Approach*.

CAUTION! To ensure that the Corona Discharge Needle does not become damaged as the source is attached to the instrument, use a very shallow angle of approach to the mounting pin (top of the vacuum chamber). The needle is fragile and very close to the open end of the source. Make certain that you do not bump the needle when installing the source housing.

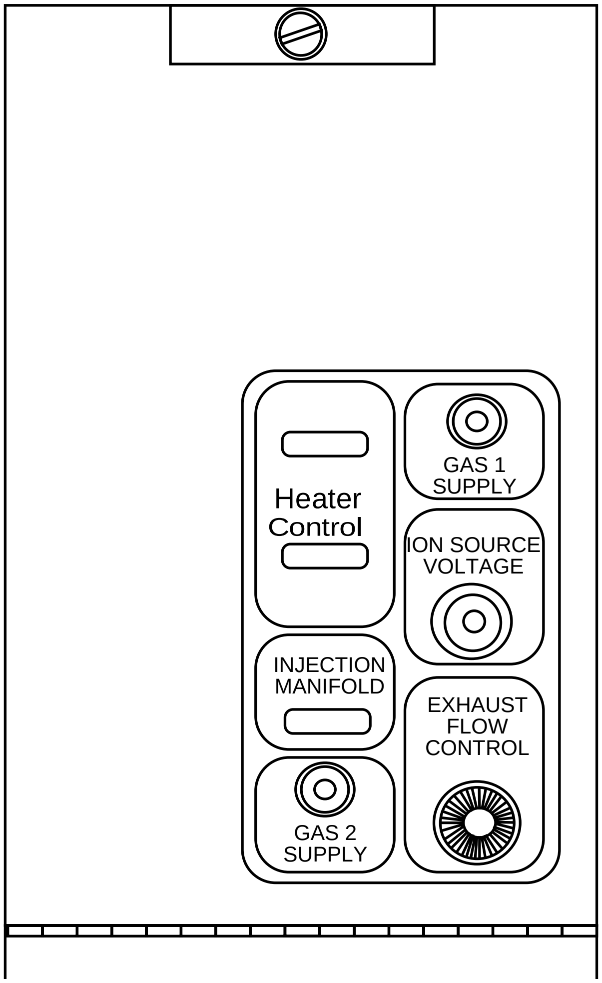


Ion Source Mounting - Angle of Approach

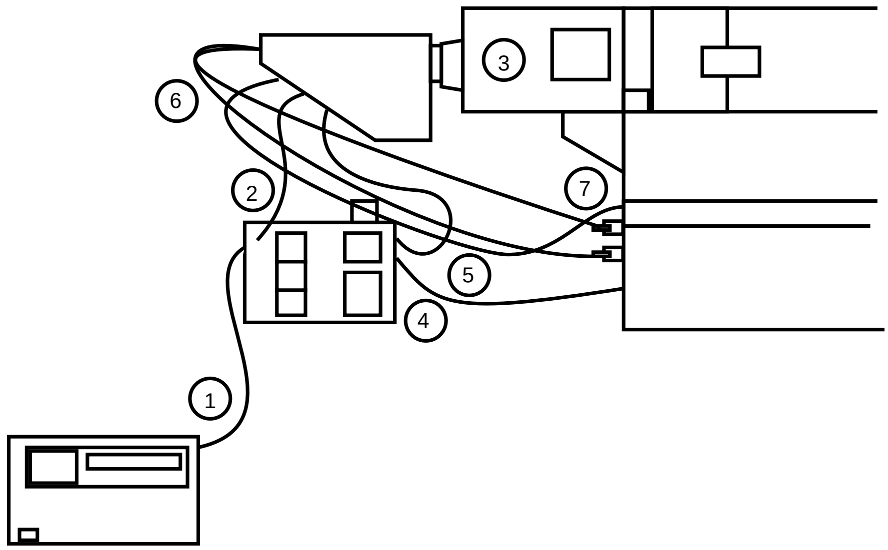


Initial Alignment of Corona Discharge Needle

4. Install the gas lines (1/8" OD Teflon tubing) from the Nebulizer Gas (Gas 2) via the gas regulator mounted on the Options Bracket, and the Auxiliary Gas (Gas 1) to the appropriate ports on the probe handle. Finger-tight connection of the fittings should be sufficient.
5. Connect low-volume (0.010" ID or less) tubing (from the exit of a column or an injector port for flow injection analysis) to the Zero Volume fitting marked LC on the handle of the probe. Ensure that all fittings are properly seated in order to minimize dead volumes.
6. Connect the HV cable to the connector on the side of the Ion Source housing, and to the Ion Source Voltage connection on the Ion Source panel.
7. Connect the Heated Nebulizer heater cable (RJ-15 connector) to the connector labeled Heater Control on the Ion Source panel.
8. Plug the three-prong thermocouple plug into the plug on the Ion Source panel, directly below the Heated Nebulizer connector.
9. Ensure that the exhaust tube is connected to the Source Exhaust port.



IonSource Panel



Ion Source Connection - Overview

10. Connect the LC pump to the Options Bracket Injector Manifold.
11. Connect the Injector Manifold to the Heated Nebulizer LC inlet.

WARNING! High Voltage Risk. Remove the high voltage connector from the instrument prior to removing the high voltage connector from from the Heated Nebulizer Ion Source housing.

12. Connect the Nebulizer Gas (Gas 2) to the regulator on Options Manifold.
13. Connect the Regulator to the probe NEB (Gas 2) connection.
14. Connect the Nebulizer Probe cables to the Heater Control on the Ion Source panel.
15. Connect the Auxiliary Gas, supplied from the Gas 1 connection on the Ion Source panel to the AUX connector on the Nebulizer probe.

You have now installed the Heated Nebulizer ion source on your API instrument. Complete the following procedure to remove the Heated Nebulizer ion source from your instrument.

To remove the Heated Nebulizer from your API instrument:

WARNING! The Heated Nebulizer Ion source may be hot for several minutes after it is removed from the instrument.

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

NOTE: The instrument **must** be in **Standby** or **Overnight Quit** mode as indicated in the first step.

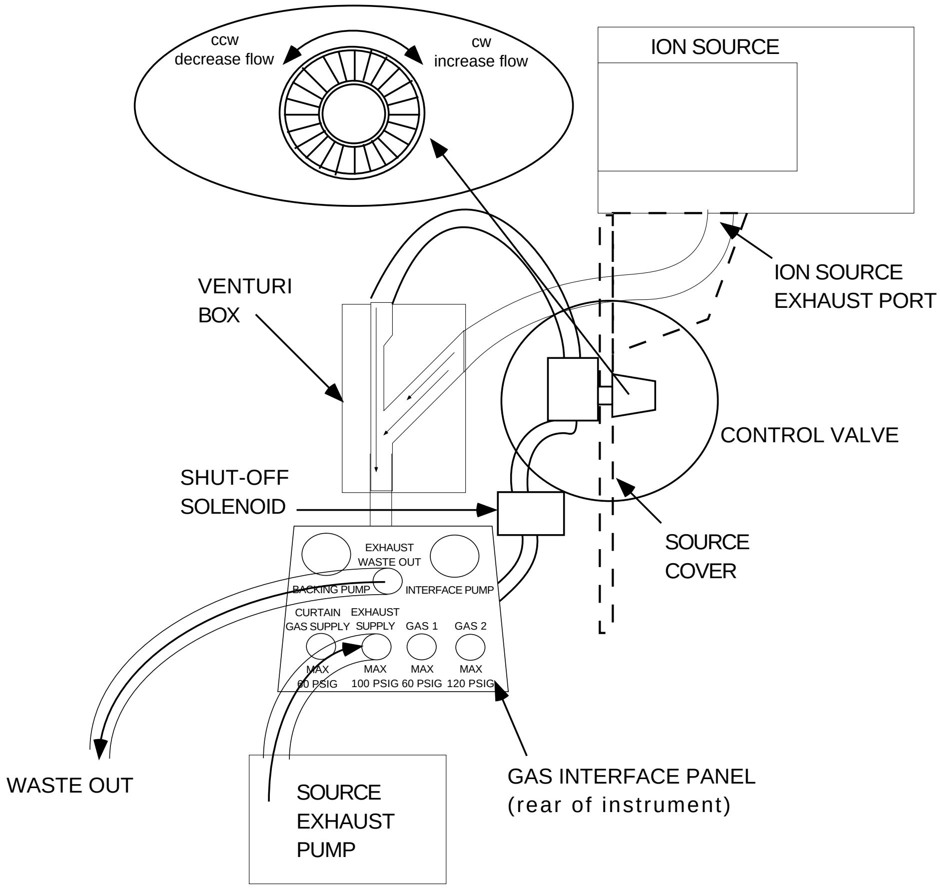
WARNING! High Voltage Risk. Remove the high voltage connector from the instrument prior to removing the high voltage connector from from the Heated Nebulizer Ion Source housing.

2. Disconnect the Auxiliary Gas, supplied from the Gas 1 on the Ion Source panel, from the AUX connector on the Nebulizer probe.
3. Disconnect the Nebulizer Probe cables from the Heater Control on the Ion Source panel.
4. Shut off the Gas 2 supply.
5. Disconnect the regulator from the NEB (Gas 2) connection.
6. Disconnect the Nebulizer Gas (Gas 2) from the regulator on the Options Manifold.
7. Disconnect the Injector Manifold from the Heated Nebulizer LC inlet.
8. Disconnect the LC pump from the Options Bracket Injector Manifold.
9. Disconnect the three-prong thermocouple plug from the Heated Nebulizer connector.
10. Disconnect the Heated Nebulizer heater cable (RJ-15) from the Heater Control connector on the Ion source panel.
11. Disconnect the low volume (0.010" ID or less) tubing (located at the exit of a column or an injector port for flow injection analysis) from the Zero Volume fitting marked LC on the handle of the probe. Ensure that all fittings are properly seated in order to minimize dead volumes.
12. Disconnect the gas lines (1/8" OD Teflon tubing) from the Nebulizer Gas (Gas 2) via the gas regulator mounted on the Options Bracket, and the Auxiliary Gas (Gas 1) from the appropriate ports on the probe handle.
13. Lift the mounting plate on the APCI Source housing off the hook on the top of the Vacuum Interface.
14. Once the source is cool, remove the Corona Discharge Needle from the needle chuck.

WARNING! To avoid exposure to chemical contamination, use gloves to remove the Corona Discharge Needle.

You have now removed the Heated Nebulizer ion source from your API instrument.

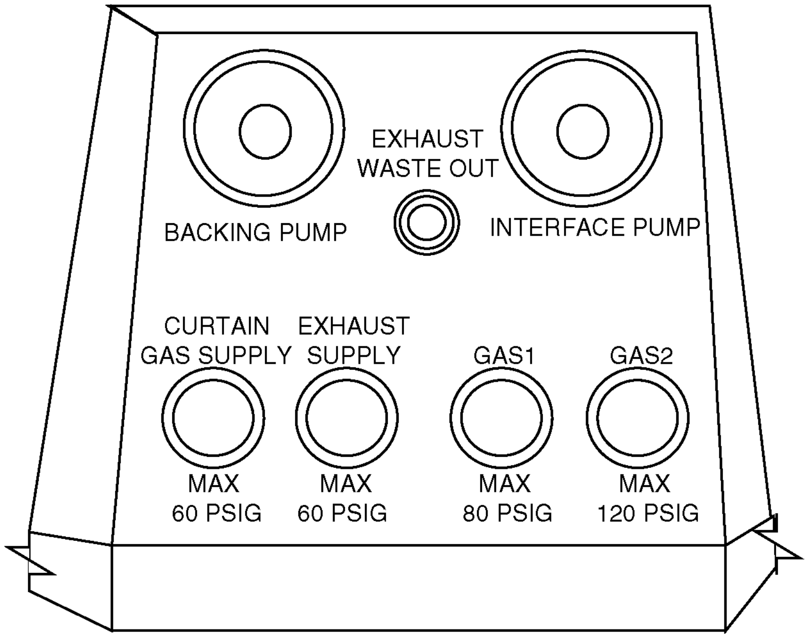
Ventilation



Source Exhaust System

The Heated Nebulizer Source requires that the Source Exhaust system is properly connected and functioning. A filtered nitrogen, or air gas supply (free from pump oil) is delivered to the Source Exhaust Pump at 60 psig pressure at a flow of at least 4 to 8 L per minute. The Exhaust Supply connection point are shown above. The Source Exhaust pump is used to vent solvent vapors which develop in the Ion Source plenum. It is highly recommended that these vapors be passed through a trap, and then vented to a fume hood, or outside port.

WARNING! Failure to provide proper ventilation of the ion source can result in hazardous vapors being released into the laboratory environment.



Gas Connection Panel

Set-Up

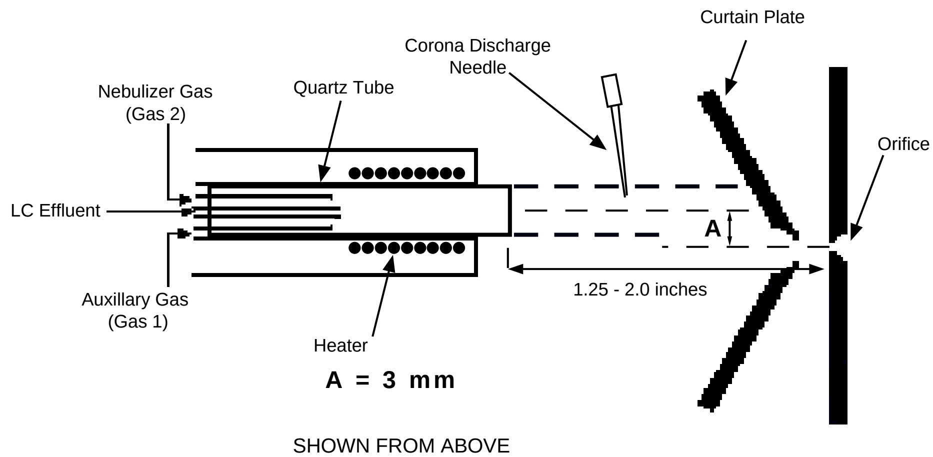
CAUTION! IF UNATTENDED USER OPERATION IS INTENDED THROUGH THE USE OF SAMPLE CONTROL, ENSURE THAT LC SHUT-OFF IS IN USE TO PREVENT FLOODING OF THE PLENUM CHAMBER.

Probe Position

The position of the probe relative to the Orifice and the corona discharge is an important factor in optimizing the Heated Nebulizer performance. The probe should be 3 mm off axis with respect to the center of the orifice. The distance of the probe from the orifice plane is not as critical, it can typically vary over the range from 3 cm [6.25 inches] to 5 cm [2 inches] from the orifice. The corona discharge needle should be on the same plane as the quartz tube, such that if the quartz tube were projected to the interface, the tip of the needle should touch the top of the virtual quartz tube.

WARNING! Do not remove the Heated Nebulizer probe from the source when the probe is hot. Allow sufficient time for cooling.

NOTE: Needle position has been set previously.



Probe and Corona Discharge Needle Position

Set-up of the Heated Nebulizer should begin with a warm-up stage, to allow the probe to heat prior to initiating the liquid sample flow. The 5 minute warm-up eliminates the possibility that solvent vapors may condense in a cold probe.

To warm up the Heated Nebulizer:

1. In the Analyst application, set the value for the curtain gas to 9

   NOTE: It is suggested that you operate the heated nebulizer with curtain gas settings adjusted to the highest flow rate possible without signal loss.

2. Turn on the Nebulizer Gas (Gas 2) to 80 psi.
3. From the Analyst application, set the Auxiliary Gas (AUX) to 6.
4. In the same State File set the heater temperature (TEM) to 400^{o}C.
5. Let the Heated Nebulizer warm-up for 5 minutes.
6. Connect the LC solvent line from the injector or autosampler to the LC connection on the probe. Turn on the solvent.
7. Adjust the exhaust pump flow until the Analyst alert status window displays an alarm. At this point turn the flow control valve about a 1/2 a turn clockwise or until the alarm is extinguished.

It takes approximately 10 minutes before the Heated Nebulizer probe reaches a temperature where the solvent mist is cleared from the plenum chamber.

Optimizing the Heated Nebulizer Set-up

The following section outlines the practical considerations which must be considered when optimizing the Heated Nebulizer performance. It is intended to provide the qualitative information necessary to aid you in quantifying the separate operating parameters.

Several parameters impact the performance of the Heated Nebulizer. To optimize the performance inject by flow injection a known compound (reserpine is recommended) and monitor the signal of the known ion. Adjust the following parameters to maximize the signal to noise ratio as shown in the following table.

Parameter Optimization for Heated Nebulizer Table

Parameter	Nominal Value	Normal Range
LC Flow (mL/min)	1	0.2 to 2
NC (µA)	2	1 to 5
Gas 1	6	3 to 15
Gas 2 (psi)	75	60 to 100
Temperature (ºC)	425	300 to 500
DP (V)	30	5 to 80
FP (V)	300	200 to 380
Sample Pump	1/2 CW turn after alert	
Curtain gas	9	6 to 12
Probe Lateral pos.	Scale 2 (3 mm)	Scale -6 to +6

Temperature:

The quantity and type of sample affects the optimal Heated Nebulizer temperature. At higher flow rates the optimal temperature increases. A more significant factor is the composition of the solvent. As the organic content of the solvent increases the optimal probe temperature should decrease. With solvents consisting of 100 percent methanol or acetonitrile the probe performance may optimize as low as 300°C. Aqueous solvents consisting of 100 per cent water at flows approximately 1mL/min require a minimum probe temperature of 425°C. Normal optimization is usually performed in increments of 25°C.

The Heated Nebulizer is normally used with sample flow rates of 1mL/min but has been used with flows from 200 µL/min to 2.0 mL/min. The heat is used to vaporize the sample and solvent sprayed into the ion source chamber. If the temperature is set too low the vaporization is incomplete and visible large droplets are expelled into the plenum. However setting the temperature too high induces thermal degradation of the sample. The optimal temperature is the lowest setting which ensures the complete vaporization of the sample.

CAUTION! Do not operate the heated Nebulizer with probe temperatures greater than 500ºC.

WARNING! Do not remove the heated nebulizer probe from the source when the probe is hot. Allow sufficient time for cooling.

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

Optimal Declustering Potential and Focusing Potential voltages should be set high enough to reduce the chemical noise but low enough to avoid fragmentation. Start with the Declustering Potential (DP) at 300V and the Focusing Potential (FP) at 30 V.

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

In general terms, the higher the Declustering Potential and Focusing Potential voltages the greater the energy imparted to the ions entering the analyzing region of the mass spectrometer. The energy helps to decluster the ions and to reduce the chemical noise in the spectrum resulting in an increase in signal to noise, or sensitivity. Increasing the voltages beyond optimal conditions can induce fragmentation before the ions enter the mass filters resulting in a decrease in sensitivity. In some instances this fragmentation can prove a valuable tool providing additional structural information.

Curtain Gas Flow

The Curtain Gas ensures a stable clean environment for the sample ions entering the mass spectrometer. The gas curtain prevents air or solvent from entering the analyzer region of the instrument while permitting the sample ions to be directed into the vacuum chamber by the electrical fields generated between the Vacuum Interface and the corona discharge needle. The presence of the solvent vapor or moisture in the analyzer region of the mass spectrometer contaminates the QØ Rod Set causing a reduction in resolution, stability, sensitivity, and an increase in chemical background noise.

In order to prevent instrument contamination the Curtain Gas flow **should be optimized at the highest possible setting** that does not result in a significant reduction in signal intensity. Refer to the System Reference Manual for further details of Vacuum Interface.

Solvent Composition

Commonly used solvents and modifiers are acetonitrile, methanol, propanol, water, acetic acid, formic acid, ammonium formate and ammonium acetate. The modifiers such as TEA, sodium phosphate, TFA and dodecyl sodium sulfate are not commonly used because they complicate the spectrum with their ion mixtures and cluster combinations. They may also suppress the strength of the target compound ion signal. The standard concentration of ammonium formate or ammonium acetate is from 2 to 10 millimole per liter for positive ions and 2 to 100 millimole per liter for negative ions. The concentration of the organic acids is 0.1% to 6.0% by volume.

Source Exhaust Pump

The Source Exhaust System is required for Heated Nebulizer operation. The exhaust pump draws the solvent vapors from the enclosed source chamber and delivers them to a trap at the rear of the instrument chassis where they can be collected. The Source Exhaust System is interlocked to the system electronics, such that if the source exhaust pump is not operating to specification the instrument electronics are disabled.

The exhaust system lowers the pressure in the source slightly below atmospheric. If the pressure in the source rises beyond a pressure sensor trip point the instrument High Voltage Power Supply is disabled. For more details on the Source Exhaust System refer to the System Reference Manual.

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

The adjustment of the source exhaust can affect the Heated Nebulizer operation. If the Source Exhaust is set too high, the pressure in the source is reduced and the signal of the target compound can be reduced.

Appendix A - Temperature Controller

Introduction

The Temperature Controller monitors and maintains the temperature of the heater probe in both the Heated Nebulizer and TurboIonSpray inlets. The Temperature Controller consists of a 420 W (8.5 Ω) heating element in the Heated Nebulizer and the TurboIonSpray probes, the Temperature Control Board (TCB) and a transformer which provides the power for both the heater and the TCB. A thermocouple attached to the heating element probe returns the actual heater temperature to the TCB.

A switch on the TCB cycles power to the heater element to maintain the probe temperature within ± 5 degrees of the temperature set by the operator at the Applications computer. The operating temperature range for the probe is 250° to 550°C.

CAUTION! When using the Heated Nebulizer, do not set the Temperature Control Board beyond 500°C

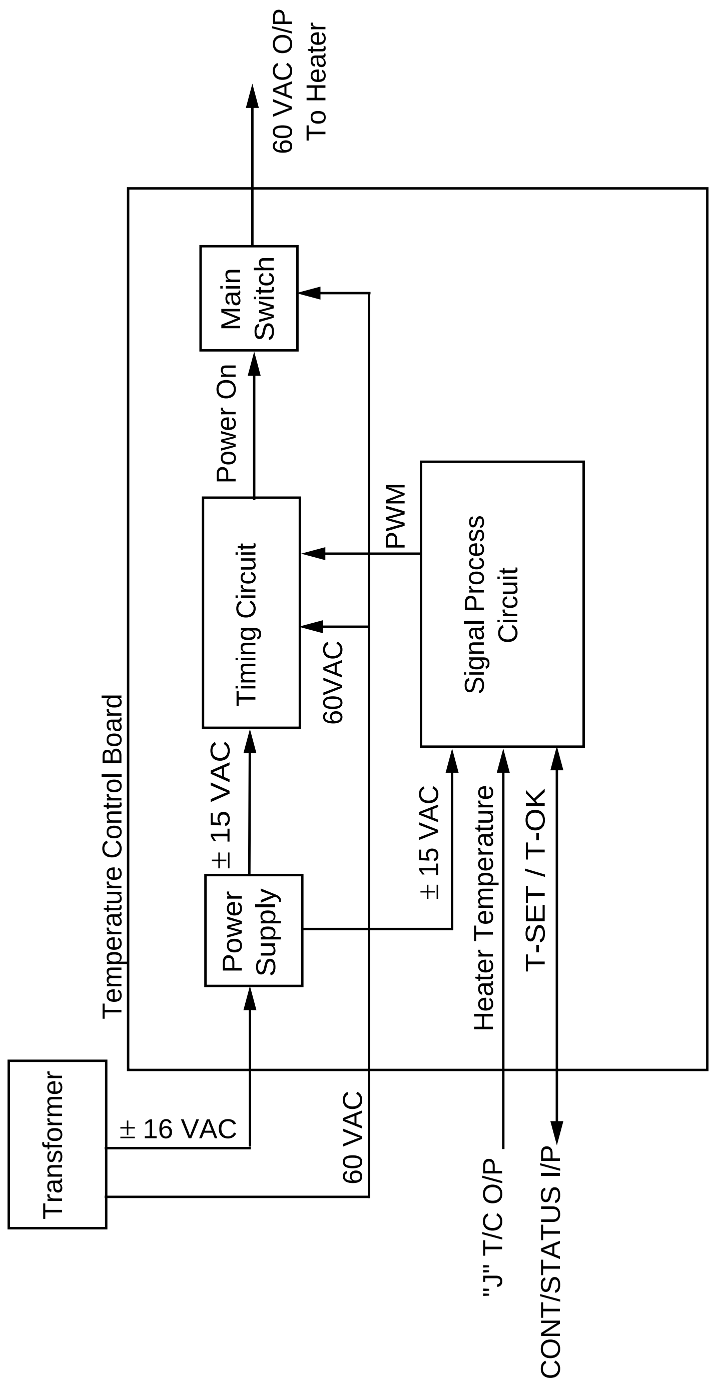
The line reduction transformer mounted on the instrument chassis behind the Q1 coil box reduces the 220 VAC power from the AC Distribution Board to the 60 VAC required by the heater and the ±16 VAC for the TCB circuitry.

The transformer and the Temperature Controller Board are standard features included with the system.

Temperature Control Board Design

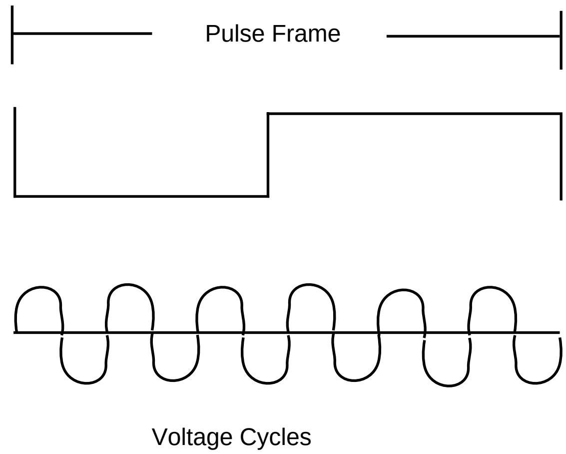
The Temperature Controller maintains the heater temperature by varying the flow of power to the heater element within a fixed period, termed the pulse frame. The TCB circuit schematic outlines the TCB control of the heater temperature.

The Timing Circuit on the TCB provides the timing mechanism for the controller, generates the pulse frame and produces the Power On signal which switches power to the heater.



Temperature Controller Board Circuit Schematic

The pulse frame and the voltage cycle frequency are a function of the 207 to 242 VAC main line frequency. For a 60 Hz supply the fixed pulse frame is 100ms (120ms for 50 Hz). Each pulse frame contains six complete voltage cycles which can be switched to the heater element as shown in the following figure. The number of cycles switched to the element per pulse is a function of the difference between the TMP voltage, which represents the actual probe temperature and the T-SET voltage, which represents the temperature setting. The larger the difference, the larger the number of voltage cycles per pulse frame switched to the heater element



TCB Pulse Frame - Voltage Cycles

The Signal Processing Circuit compares the TMP voltage to the T-SET voltage and sends the Pulse Width Modulator (PWM) signal, which determines the power flow to the heater element, to the Timing Circuit (see Figure). The PWM output is a voltage signal which can vary between 0 and 10V depending on the difference between TMP and T-SET. If PWM is 10V, all six voltage cycles of each pulse frame are switched to the heater element. If PWM is 0V, then none of the voltage cycles are switched to the heater element.

The T-OK signal is relayed to the System Controller 25 seconds after the heater temperature reaches the set temperature; T-SET ± 5. The delay allows the heater temperature to stabilize within the specified temperature range.

There are three LED indicators on the Temperature Control Board:

Temperature Controller Board LED Indicators Table

TMP LED (D23)	Indicates the heater temperature status. Red indicates the heater temperature is outside the T-SET ± 5 setting. Green indicates that TMP is within T-SET ±5.
T/C OC LED (D11)	Indicates that the thermocouple is an open circuit i.e. the thermocouple connections are faulty. The fault will cause the main switch to be shutdown.
OVER TMP SHUTDOWN LED (D7)	Indicates that main switch bypass was activated to divert power from the heater. This happens when full power is directed to the heater element for more than 3.5 minutes. It will cause a current surge which will blow the fuse F6. It indicates a likely short across the thermocouple or a short in the main TRIAC switch.

Test Points

The TMP and T-SET voltages can be checked at test points on the TCB. The voltages are scaled such that 10mV is equivalent to 1ºC. A voltmeter reading between ground and the test point TP1 reads the heater temperature. Likewise, a voltmeter reading between ground and TP2 reads the temperature setting T-SET.

Temperature Controller Board Test Points Table

Test Point 1 (TP1)	Voltage across TP1 and Ground measures Heater Temperature. (10mV= 1ºC)
Test Point 2 (TP2)	Voltage across TP2 and ground measures the temperature setting T-SET. (10mV= 1ºC)

The T-SET temperature can also be set manually at the Temperature Controller Board. To set the target heater temperature locally, toggle switch SW1 from System to Local, and adjust potentiometer R68 to the desired setting. Monitor the temperature setting, by measuring the voltage at TP2.

Temperature Controller Board Connections

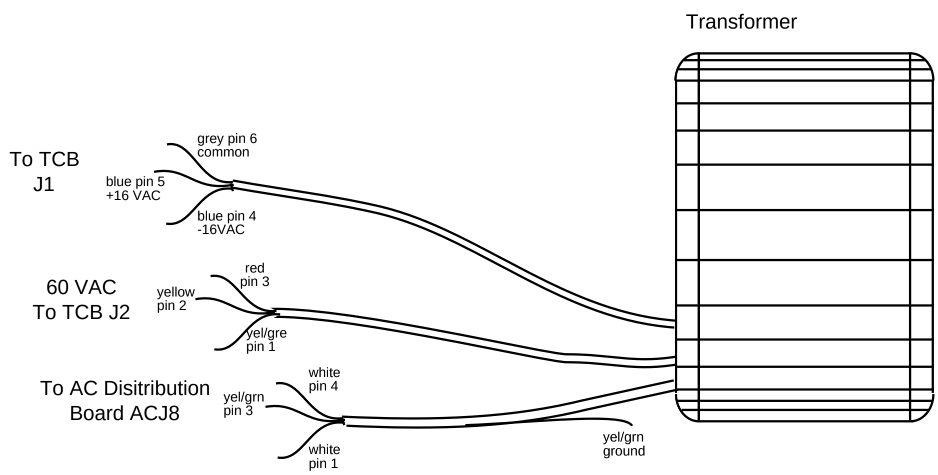
Connector	TO/FROM	Pin number
J1	± 16 VAC input from transformer	4 -16 VAC (blue) 5- +16 VAC (blue) 6- Common (grey)
J2 -	60 VAC input from transformer	1 Ground (yellow/green) 2 60 VAC return (yellow) 3 60 VAC (red)
J3	60 VAC switched to heater element.	1 60 VAC to heater 2 60 VAC return 3 Shield 4 Chassis ground 5 Ground key
J4	Thermocouple leads	1 +T/C (white) 2 -T/C (red 3 Shield (bare)
J5	Control Status to/From Motherboard	1 Signal ground 2 T-SET from Motherboard T-OK to Motherboard

Transformer

The line reduction transformer mounted on the chassis provides power exclusively for the Temperature Control Board and heater. The transformer is connected to the AC Distribution Board via the AC Cord Heated Nebulizer Cable. It converts the 207 to 242 VAC input supply to the 60 VAC output for the heating element in the Heated Nebulizer and TurboIonSpray inlet probes. A secondary winding on the transformer supplies the ± 16 VAC for the Temperature Controller Board circuitry.

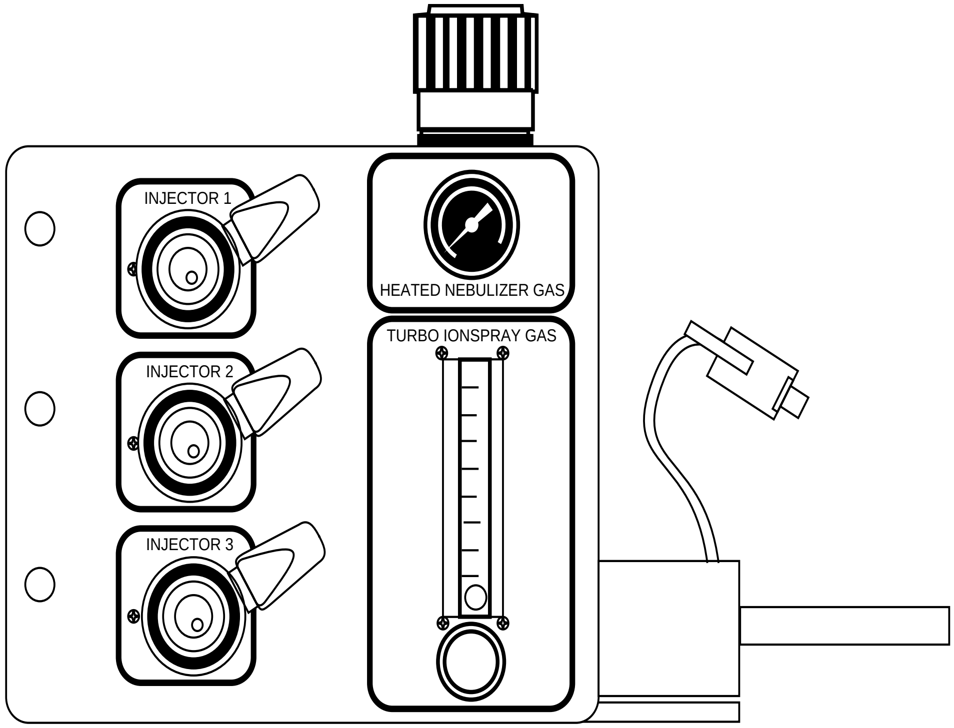
The transformer has a temperature cut off switch which shuts off the transformer if its temperature exceeds 110°C. The switch will reset automatically when the temperature falls 50°C below the trip point.

WARNING! ELECTRICAL SHOCK HAZARD. Severe electrical shock can result if you attempt to remove the API instrument panels. Turn off the power supplies, detach the power cord and wait at least one minute before removing the outside panels.



Transformer Wiring Schematic

Options Bracket



Options Bracket (WC021218)

The Options Manifold is a plate which is configured to contain the Flow Controller, Regulator Valve, and up to 3 injectors. This manifold is highly recommended for the operator to easily arrange, and organize components which are part of the Heated Nebulizer, and TurboIonSpray Ion Sources. Each component to be mounted on the manifold should come with its own mounting hardware.

The Manifold also has a connection cable which is connected to the Ion Source Panel Injector Manifold connection (RJ 9 connection) to allow for automatic injection control through Sample Control.

Appendix B- Troubleshooting

Heater Failure