Particle Instruments

Model 3480
Electrospray Aerosol Generator

Instruction Manual

P/N 1933793, Revision C
October 2002

TSI®

Model 3480 Electrospray Aerosol Generator

Instruction Manual

Manual History

The following is a manual history of the Model 3480 Electrospray Aerosol Generator (Part Number 1933793).

Revision	Date
Preliminary Version	March 1999
Final	May 1999
A	July 2000
B	February 2002
C	October 2002

In Revision A, TSI's Limitation of Warranty and Liability was updated.

In Revision B, Table 2-1 was updated, Appendix C was removed, and a few minor corrections were made throughout manual.

In Revision C, TSI's phone numbers and address were updated. Front and back manual covers were updated.

Warranty

Part Number 1933793 / Revision C / October 2002

Address TSI Incorporated / 500 Cardigan Road / Shoreview, MN 55126 / USA

Fax No. 651-490-3824

E-mail Address particle@tsi.com

Limitation of Warranty and Liability (effective July 2000)

Seller warrants the goods sold hereunder, under normal use and service as described in the operator's manual, shall be free from defects in workmanship and material for (12) months, or the length of time specified in the operator's manual, from the date of shipment to the customer. This warranty period is inclusive of any statutory warranty. This limited warranty is subject to the following exclusions:

a. Hot-wire or hot-film sensors used with research anemometers, and certain other components when indicated in specifications, are warranted for 90 days from the date of shipment.

b. Parts repaired or replaced as a result of repair services are warranted to be free from defects in workmanship and material, under normal use, for 90 days from the date of shipment.

c. Seller does not provide any warranty on finished goods manufactured by others or on any fuses, batteries or other consumable materials. Only the original manufacturer's warranty applies.

d. Unless specifically authorized in a separate writing by Seller, Seller makes no warranty with respect to, and shall have no liability in connection with, goods which are incorporated into other products or equipment, or which are modified by any person other than Seller.

The foregoing is IN LIEU OF all other warranties and is subject to the LIMITATIONS stated herein. **NO OTHER EXPRESS OR IMPLIED WARRANTY OF FITNESS FOR PARTICULAR PURPOSE OR MERCHANTABILITY IS MADE**.

TO THE EXTENT PERMITTED BY LAW, THE EXCLUSIVE REMEDY OF THE USER OR BUYER, AND THE LIMIT OF SELLER'S LIABILITY FOR ANY AND ALL LOSSES, INJURIES, OR DAMAGES CONCERNING THE GOODS (INCLUDING CLAIMS BASED ON CONTRACT, NEGLIGENCE, TORT, STRICT LIABILITY OR OTHERWISE) SHALL BE THE RETURN OF GOODS TO SELLER AND THE REFUND OF THE PURCHASE PRICE, OR, AT THE OPTION OF SELLER, THE REPAIR OR REPLACEMENT OF THE GOODS. IN NO EVENT SHALL SELLER BE LIABLE FOR ANY SPECIAL, CONSEQUENTIAL OR INCIDENTAL DAMAGES. SELLER SHALL NOT BE RESPONSIBLE FOR INSTALLATION, DISMANTLING OR REINSTALLATION COSTS OR CHARGES. No Action, regardless of form, may be brought against Seller more than 12 months after a cause of action has accrued. The goods returned under warranty to Seller's factory shall be at Buyer's risk of loss, and will be returned, if at all, at Seller's risk of loss.

Buyer and all users are deemed to have accepted this LIMITATION OF WARRANTY AND LIABILITY, which contains the complete and exclusive limited warranty of Seller. This LIMITATION OF WARRANTY AND LIABILITY may not be amended, modified or its terms waived, except by writing signed by an Officer of Seller.

Service Policy

Knowing that inoperative or defective instruments are as detrimental to TSI as they are to our customers, our service policy is designed to give prompt attention to any problems. If any malfunction is discovered, please contact your nearest sales office or representative, or call TSI’s Customer Service department at 1-800-874-2811 (USA) or (651) 490-2811.

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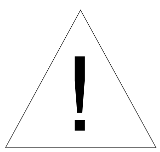
Safety

This section gives instructions to promote safe and proper handling of the Model 3480 Electrospray Aerosol Generator.

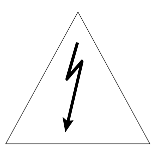
There are no user serviceable parts inside the instrument. Refer all repair and maintenance to a qualified technician. All maintenance and repair information in this manual is included for use by a qualified technician.

To prevent problems, take these precautions:

- Do **not** remove any parts from the instrument unless you are specifically told to do so in this manual.
- Do **not** remove the instrument housing or covers while power is supplied to the instrument.



Caution
If the Model 3480 is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.



WARNING
High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

Labels

The Electrospray has the eight labels shown in Figure S-1. They are described below starting from the bottom-right and moving clockwise.

1. High-Voltage Symbol Label (interior, on high-voltage power supply shield)
2. High-Voltage Symbol Label (interior, on power supply)
3. High-Voltage Symbol Label (interior, on power entry module)
4. Ground Symbol Label (interior, bottom, next to ground stud)
5. Caution, No User Serviceable Parts… Label (back of cabinet)
6. Customer Service Label (back of cabinet)

7. Serial Number Label (back of cabinet)
8. High-Voltage Symbol Label (interior, on high-voltage fitting shield)

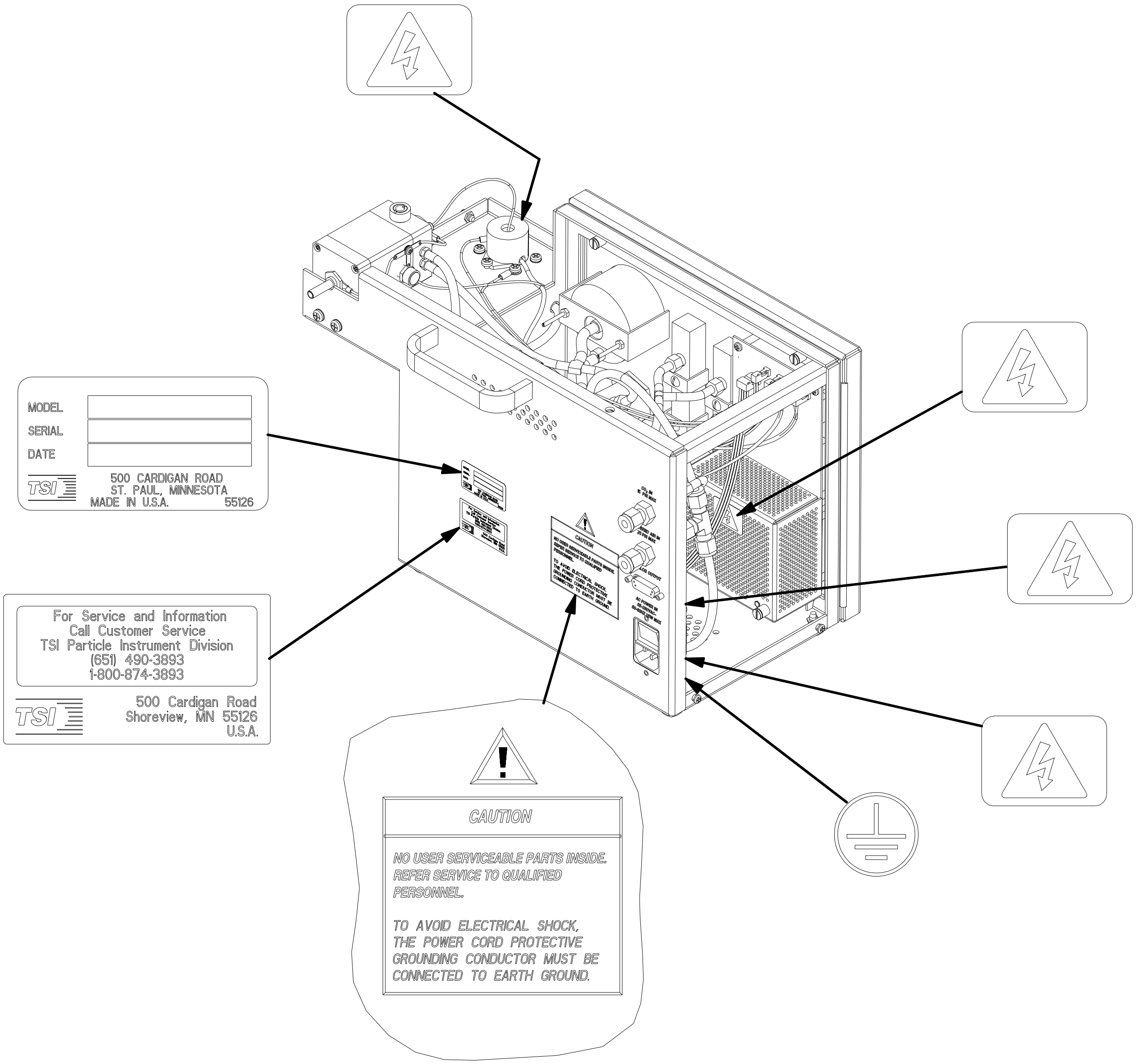
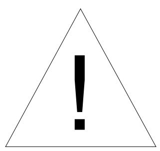


Figure S-1
Location of Warning and Information Labels

Description of Caution/Warning Symbols

The following symbol and an appropriate caution statement are used throughout the manual and on the Model 3480 to draw attention to any steps that require you to take cautionary measures when working with the Model 3480:

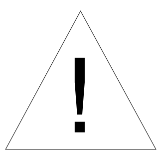
Caution



C a u t i o n

Caution means ***be careful***. It means if you do not follow the procedures prescribed in this manual you may do something that might result in equipment damage, or you might have to take something apart and start over again. It also indicates that important information about the operation and maintenance of this instrument is included.

Warning

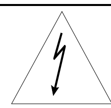
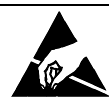
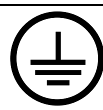
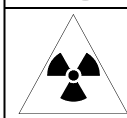


W A R N I N G

Warning means that unsafe use of the instrument could result in serious injury to you or cause irrevocable damage to the instrument. Follow the procedures prescribed in this manual to use the instrument safely.

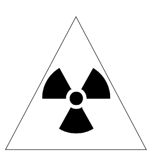
Caution or Warning Symbols

The following symbols may accompany cautions and warnings to indicate the nature and consequences of hazards:

	Warns you that uninsulated voltage within the instrument may have sufficient magnitude to cause electric shock. Therefore, it is dangerous to make any contact with any part inside the instrument.
	Warns you that the instrument is susceptible to electro-static dissipation (ESD) and ESD protection procedures should be followed to avoid damage.
	Indicates the connector is connected to earth ground and cabinet ground.
	Warns you that the Model P-2042 Nuclespot Local Air Ionizer, which is installed inside the Model 3480 Electrospray Aerosol Generator, contains Polonium-210, a radioactive material that is subject to the regulations of the U.S. Nuclear Regulatory Commission and local regulations. Carefully read the Model P-2042 Literature provided with the ionizer to determine your legal responsibilities regarding the ionizer.

Radiation Safety

The Electrospray Aerosol Generator contains a Model P-2042 Nuclespot Local Air Ionizer with a Polonium-210 source. Under normal circumstances, you will not come into contact with hazardous radiation. However, take these precautions when operating the Electrospray:



The Model P-2042 Nuclespot Local Air Ionizer with a Polonium-210 source must be installed in the Electrospray for it to operate properly. Po-210 is subject to the regulations of the U.S. Nuclear Regulatory Commission and local regulations. Carefully read the safety information provided with the ionizer to determine your legal responsibilities regarding the ionizer.

WARNING
The use of controls, adjustments, or procedures other than those specified in this manual may result in exposure to hazardous radiation.

- Corrosive materials can degrade the protective coating of gold on the ionizer inside the Electrospray. Do **not** operate the Aerosol Generator with chemicals that corrode gold.
- Do **not** operate the Aerosol Generator at temperatures above 50°C. Excess temperatures may damage the ionizer, resulting in radioactive contamination.
- The ionizer must be returned after 12 months. Contact TSI for information and instructions on returning the ionizer.
- Do **not** remove any parts from the Electrospray unless you are specifically told to do so in this manual.

Chemical Safety

Observe these warnings when operating the Electrospray Aerosol Generator:

WARNING
Any material or procedure mentioned in this manual is intended for use by qualified professionals familiar with potential chemical hazards and trained in safe laboratory procedures.

WARNING
Corrosive materials can degrade the stainless steel and aluminum chamber inside the Electrospray Aerosol Generator. Do **not** operate the Electrospray with chemicals that corrode stainless steel or aluminum.

WARNING
High concentrations of aerosols can be hazardous. Use a filter at the Electrospray exit if the generated aerosol is not being used by another instrument or in another application.

Electrical Safety

The Electrospray Aerosol Generator contains a high-voltage source on the inside of the instrument. Under normal circumstances, you will not come into contact with hazardous voltage. However, always remove power from the Electrospray before removing the cover from the instrument.

WARNING
High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

About This Manual

Purpose

This is an instruction manual for the operation and handling of the Model 3480 Electrospray Aerosol Generator.

Related Product Literature

- *Model 3080 Electrostatic Classifier Manual* (part number 1933792 TSI Incorporated)
- *Model 3010 Condensation Particle Counter* Manual (part number 1933010 TSI Incorporated)
- *Model 3022A Condensation Particle Counter* Manual (part number 1933763 TSI Incorporated)
- *Model 3025A Ultrafine Condensation Particle Counter* Manual (part number 1933762 TSI Incorporated)
- *Model 3936 SMPS (Scanning Mobility Particle Sizer)* Manual (part number 1933796 TSI Incorporated)
- *Model 3068 Aerosol Electrometer Manual* (part number 1933068 TSI Incorporated)

Reusing and Recycling

As part of TSI Incorporated's effort to have a minimal negative impact on the communities in which its products are manufactured and used:

- This manual uses recyclable paper.
- This manual has been shipped, along with the instrument, in a reusable carton.

Getting Help

To obtain assistance with this product or to submit suggestions, please contact TSI:

TSI Incorporated
500 Cardigan Road
Shoreview, MN 55126 U.S.A.
Fax: (651) 490-3824
Telephone: 1-800-874-2811 (USA) or (651) 490-2811
E-mail Address: particle@tsi.com

Submitting Comments

TSI values your comments and suggestions on this manual. Please use the comment sheet, on the last page of this manual, to send us your opinion on the manual's usability, to suggest specific improvements, or to report any technical errors.

If the comment sheet has already been used, send your comments to:

Particle Instruments
TSI Incorporated
500 Cardigan Road
Shoreview, MN 55126 U.S.A.
Fax: (651) 490-3824
E-mail Address: particle@tsi.com

CHAPTER 1

Product Overview

This chapter describes the Model 3480 Electrospray Aerosol Generator and gives an overview of how it works.

Product Description

The Model 3480 Electrospray Aerosol Generator, shown in Figure 1-1, generates monodisperse aerosols in the size range of 2 to 100 nanometers. Beginning with a liquid and adding a solute to form a solution or suspension, the Electrospray converts the sample to an aerosol by charging the liquid, pushing it through a capillary, and exerting an electric field at the capillary tip. The liquid evaporates from the droplets formed at the capillary tip while the charge on each droplet is reduced by an ionizer.

In addition to the vast combination of solutes that could be used in the Electrospray to generate aerosols, the user has the ability to control the liquid flowrate through the capillary, the intensity of the electric field at the capillary tip, and the amount of time the aerosol is exposed to the ionizer.

Figure 1-1
Model 3480 Electrospray Aerosol Generator

Applications

The successful use of the electrospray method to generate monodisperse aerosol has been documented in many publications. Although the basic principles are well understood, many of the details explaining how different operating parameters affect the electrospray method remain to be discovered. Known applications for this instrument include:

- Studies of nano-aerosols
- Instrument calibration
- Dispersion of nanometer-sized powders for nano-powder sizing
- Aerosol analysis in the macromolecular and submicrometer range
- Research involving parameters that influence the electrospray method

How the Electrospray Operates

A sample solution is stored in a cone-shaped vial, enclosed in a cylindrical pressure chamber. The chamber accommodates a capillary and a high-voltage, platinum wire, both of which are immersed in the solution. A differential pressure causes the solution to be pushed through the capillary.

An electrical field pulls the charged solution out of the capillary exit, forming droplets that are mixed with clean air and CO_2. This produces a sheath flow, which transports the droplets to the Electrospray chamber. The highly charged droplets are neutralized by a radioactive ionizer (Polonium-210), and the liquid is evaporated before the aerosol exits the instrument.

CHAPTER 2

Unpacking and Setting Up the System

Use this information in this chapter to unpack and set up the Model 3480 Electrospray Aerosol Generator.

Packing List

As you unpack the shipping container, make certain the shipment is complete. Table 2-1 gives a packing list for the Electrospray.

Table 2-1
Electrospray Packing List

Qty	Description	Part No.
1	Model 3480 Electrospray Aerosol Generator	348000
1	Electrospray Accessory Kit Including:	
1	4 Capillary Assemblies with Case	1309132
1	Ionizer Retainer Tool	3012062
1	L-Key Hex Wrench (.050 in)	3305003
1	Cable, Power Supply	1303053*
1	Instruction Manual	1933793
2	Ferrules, Nylon Front ($^1/_{16}$-in TUBE)	1611252
2	Ferrules, Nylon Back ($^1/_{16}$-in TUBE)	1611253
2	O-rings, 1-009 (FSI)	2501899
6	O-rings, 1-010 (EPDM)	2501520
2	O-rings, 1-013 (FSI)	2501567
2	O-rings, 1-014 (EPDM)	2501528
2	O-rings, 1-016 (EPDM)	2501602
2	O-rings, 1-020 (EPDM)	2501073
2	O-rings, 1-024 (BUNA)	2501024
2	O-rings, 1-027 (EPDM)	2501603
2	O-rings, 1-029 (EPDM)	2501072
1	Vacuum Grease	1101063
5 ft [1.5 m]	Tubing, Silicone Conductive $^3/_8$-in OD	3001903
10 ft [3 m]	Tubing, Polyethylene ¼-in OD	3929436

*Power cable listed is for U.S.A. use only.

Note: *A Polonium-210 ionizer is shipped separately from the Electrospray instrument and accessory kit. The ionizer must be installed for the Electrospray to operate properly. See the instructions later in this chapter for installing the ionizer.*

Mounting the Instrument

The Electrospray has no special mounting requirements other than providing good ventilation (see below). The cabinet has four non-marking rubber feet that give the instrument a good grip on clean, level surfaces. The rubber feet (Figure 2-1) are installed in the cabinet using integrated #8-32 UNC threaded fasteners and can be removed (by unscrewing) to allow other mounting fasteners to be used.

***Note**: If the cabinet is mounted to a plate, drill holes in the plate to match the ventilation holes in the bottom of the cabinet or use standoffs to raise the bottom of the cabinet at least ½ inch (1.2 cm) above the mounting plate.*

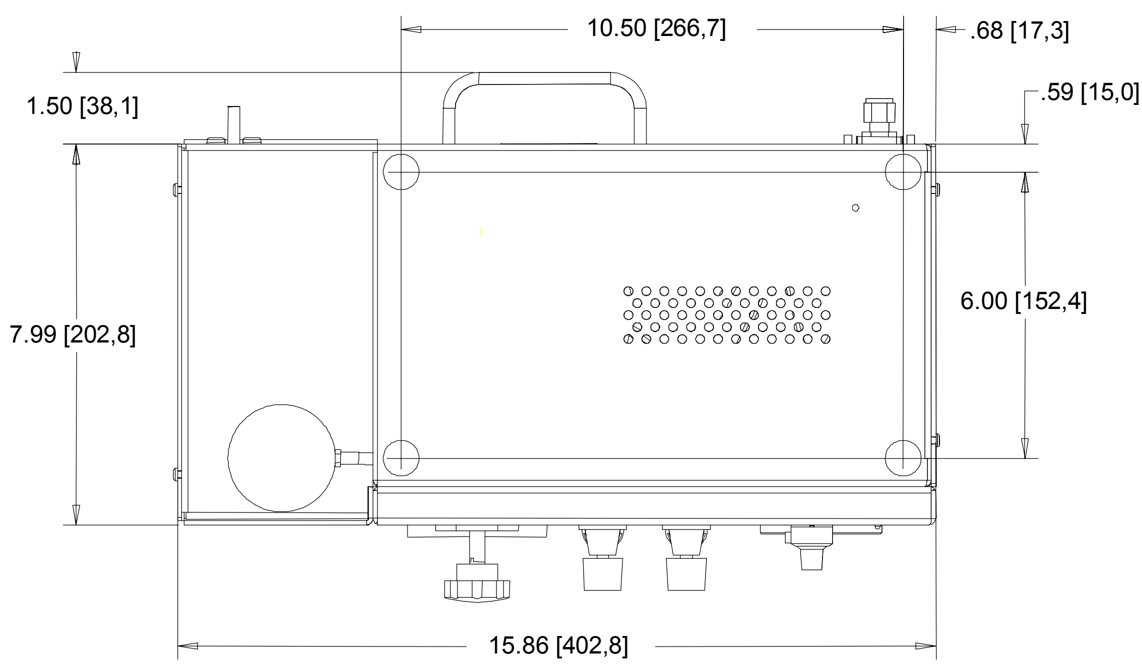


Figure 2-1
Bottom View of Electrospray Aerosol Generator Showing Location of Rubber Feet. Feet can be removed to provide mounting points on cabinet. Dimensions are in inches [mm].

Ventilation Requirements

The Electrospray cabinet is designed to be cooled by natural convection of room air drawn through the bottom of the cabinet and exhausted through the back of the cabinet.

The cabinet should be installed with at least 2-inch (50 mm) clearance between the back panel and any other surface. Also, the cabinet should be set on a clean, hard surface so that the intake air can move freely under the base of the cabinet.

Checking the Zero of the Pressure Gauge

The pressure gauge is adjusted to the zero position at the factory; however, if the needle is not lined up with the left side of the zero box marked on the gauge, you will need to re-zero the pressure gauge.

If zeroing is required, follow the steps below to reset the gauge:

1. You will need a palm-sized square of soft, clean rubber to grip the front surface of the gauge and a .050 inch hex wrench (included in the accessory kit).
2. Hold the cabinet for support and grip the front surface of the gauge with the square of rubber in the palm of your hand.
3. Unscrew the front cover of the gauge in a counter-clockwise direction.
4. A sockethead setscrew is located under the bottom of the gauge panel where it is marked “zero”.
5. Adjust the setscrew until the needle lines up with the left side of the zero box marked on the gauge.
6. Reassemble the front cover of the gauge.

Power Connection

Connect the AC power cord (supplied) to the AC POWER IN connection on the back of the Electrospray and then into an available power outlet. It is not necessary to select the correct voltage, the instrument accepts line voltages of 85 to 260 VAC, 50-60 Hz, 25 W max., single phase. The connector has a built-in on/off switch.

***Note**: Make certain the line cord is plugged into a grounded (earth grounded) power outlet. Position the Electrospray so the power cord connector is not blocked and is easily accessible.*

Note: *The Electrospray power supply contains no user-serviceable parts. If the power supply is not operating correctly, use the information in Chapter 7 to contact TSI. This instrument should not be used in a manner not specified by the manufacturer.*

Toggle the on/off switch at the AC POWER IN connection to the on position to verify the instrument has power.

Analog Output

The Electrospray has a single 15-pin, D-subminiature connector port on the back panel labeled ANALOG OUTPUT that can be used to measure signals that represent the voltage and current output on the front panel meters of the instrument. See Chapter 3 for a detailed list of the signal connections.

Filtered Air Input

The back panel of the Electrospray has an input marked FILTERED AIR IN, 25 PSI MAX that is a ¼" Swagelok® tube fitting with nylon ferrules. Insert the supplied polyethylene tubing or a similar tubing and tighten the Swagelok® nut finger-tight plus ¼-turn with a wrench. This will provide a secure connection and allow the tubing to be removed without damaging the ferrules. A regulated, compressed air supply that furnishes HEPA-filtered, clean, dry air must be used to supply filtered air to the Electrospray. TSI Model 3074B Filtered Air Supply is recommended and may be purchased separately. The recommended pressure of the filtered air supply used with the Electrospray is 15 psi.

WARNING
Supplying more than 25 PSI air pressure could result in damage to the internal components of the Electrospray and decreased sensitivity of the pressure regulator and air rotameter.

®Swagelok is a registered trademark of Swagelok Company.

CO_2 Input

Above the filtered air input, is an identical input marked CO_2 IN, 15 PSI MAX. Connect the supplied polyethylene tubing in the manner described for the Filtered Air Input, using a commercial CO_2 compressed gas tank to supply CO_2 to the Electrospray. Commercial CO_2 tanks and accessories are available from most compressed gas suppliers, in denominations ranging from 2.5 to 50 lb. The recommended pressure of the CO_2 supply used with the Electrospray is 5 psi.

WARNING
Supplying more than 15 PSI CO_2 pressure will cause the internal silicone tubing to disconnect from the CO_2 rotameter fitting. In addition, a higher CO_2 inlet pressure makes it more difficult to adjust the CO_2 flowrate.

Installing the Ionizer

A Model P-2042 Nuclespot Local Air Ionizer with a Polonium-210 source is shipped separately because of regulations that govern packaging and shipping requirements for radioactive materials. The ionizer must be installed for the Electrospray to operate properly. Before installing the ionizer, read the safety information at the beginning of this manual and the safety information provided with the ionizer.

WARNING
The use of controls, adjustments, or procedures other than those specified in this manual may result in exposure to hazardous radiation.

Use this procedure to install the ionizer:

1. Remove the ionizer retainer shown in Figure 2-2 using the ionizer retainer tool supplied in the accessory kit, and set the dummy source aside.

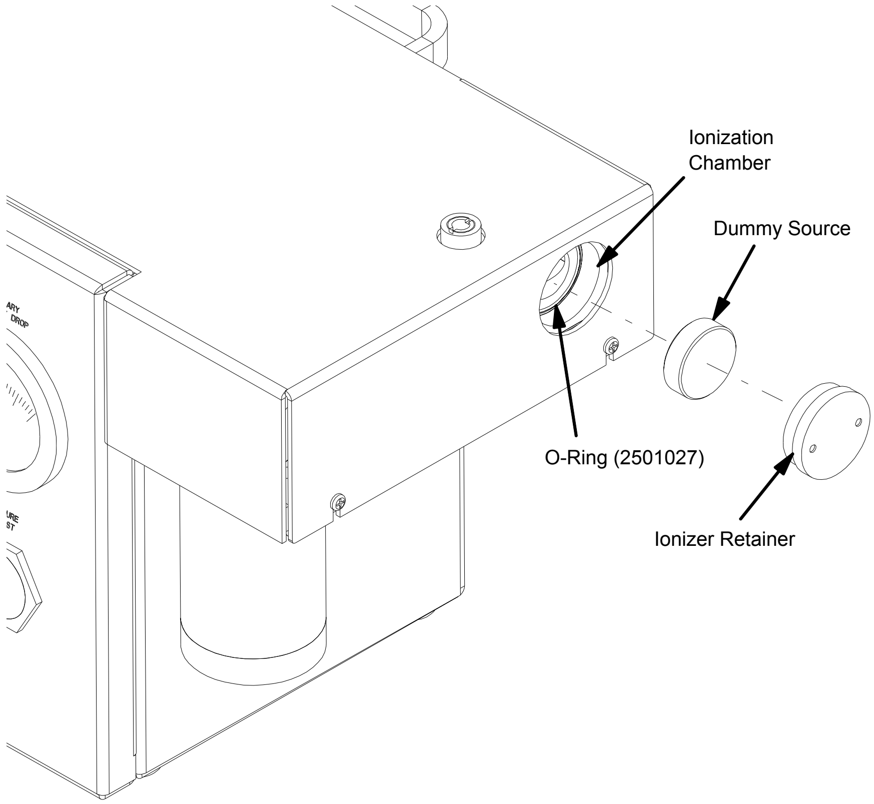


Figure 2-2
Installing the Ionizer

2. Place the ionizer in the ionizer retainer in place of the dummy source. The ionizer mesh-side should be facing the open end of the ionizer retainer.

3. Install the ionizer retainer, making sure the O-ring is between the ionization chamber and the ionizer retainer, and tighten with the ionizer retainer tool.

Note: *The ionizer must be replaced 12 months from the date on the ionizer label. Contact TSI to order a new ionizer four weeks before the ionizer expiration date. When returning an ionizer, contact TSI for handling and shipping instructions.*

CHAPTER 3

Controls, Indicators, and Connectors

Use the information in this chapter to familiarize yourself with the location and function of controls, indicators, and connectors on the Model 3480 Electrospray Aerosol Generator.

This chapter is organized into three sections describing aspects of the instrument: Front Panel, Back Panel, and Internal Components.

Front Panel

The main components of the front panel are shown in Figure 3-1. They include: the voltage and current LED displays, the voltage adjustment knob, the power and high-voltage LED indicators, the CO_2 and air rotameters, the pressure regulator and gauge, and the pressure chamber. In addition, the viewing window and ionizer retainer are integrated with the cover and are included in this section.

LED Displays

There are two LED displays on the Model 3480: Voltage (kV) and Current (nA).

- The voltage LED display is a measure of the high voltage applied to the platinum wire, which charges the liquid in the vial and capillary. A positive high voltage is standard, but a negative high-voltage module is available in place of or in addition to the positive high-voltage module.
- The current LED display is a measure of the status and stability of the Electrospray Aerosol Generator. See Chapter 4, “Operating the Electrospray” for more information on how the current reading aids in determining the status of the Electrospray, and Appendix B, “Theory of Operation” for more detail on how the current is measured.

Voltage Adjustment Knob

This knob is used to change the high voltage applied to the liquid used in the electrospray process. It can be locked by pushing the black tab clockwise on the base of the knob.

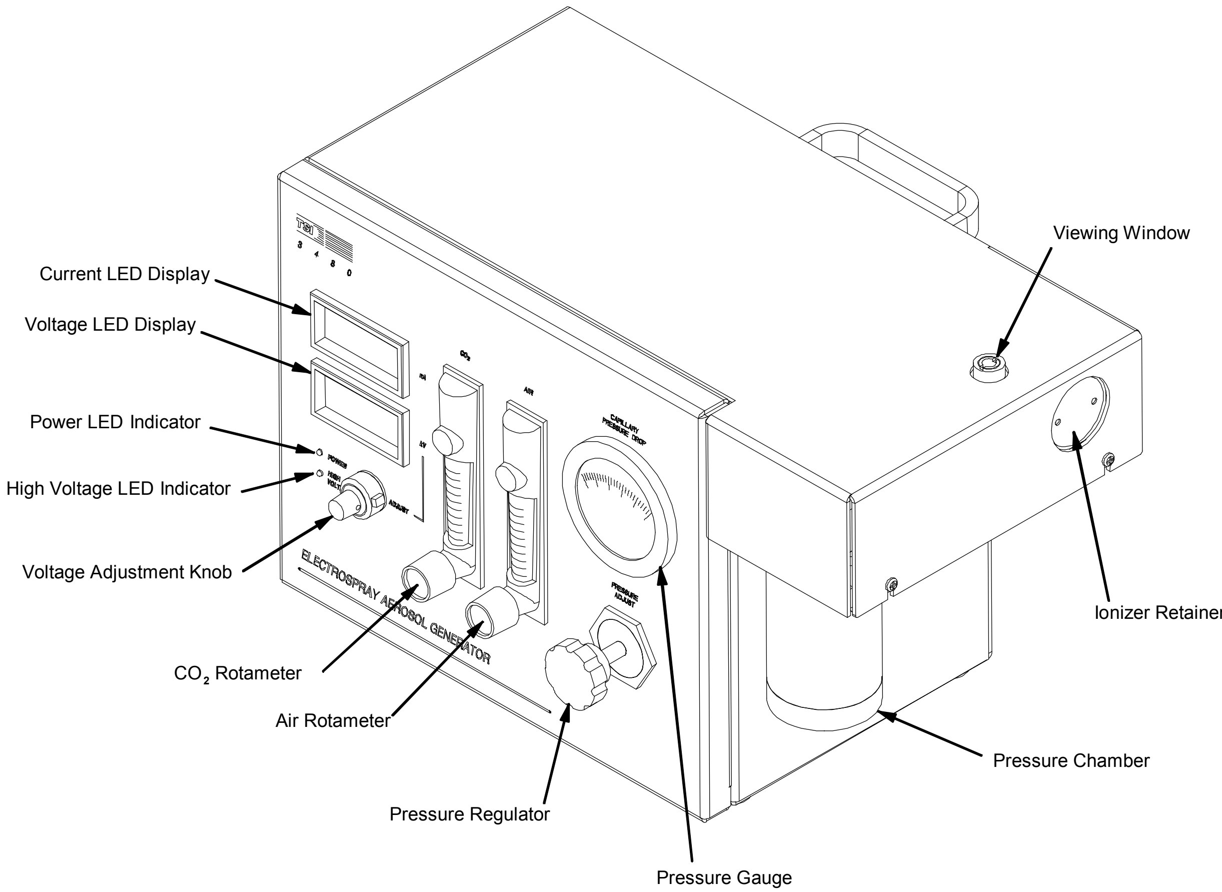


Figure 3-1
Front Panel of the Model 3480 Electrospray Aerosol Generator

Indicators

There are two status LED's on the Electrospray: power and high voltage.

- The green power LED indicates that power is supplied to the instrument.
- The high-voltage LED indicates that high voltage is being applied to the platinum wire. The high voltage is turned off if the cover is off the instrument or the bottom half of the pressure chamber is not installed.

Rotameters

The rotameters regulate the flowrates of air and CO_2 to the Electrospray chamber. The air and CO_2 flowrates are typically 1.0 and 0.1 lpm, respectively.

Pressure Regulator and Gauge

The pressure drop across the capillary is regulated and measured with these two components. See Appendix B, "Theory of Operation" for details on how the pressure drop is measured and how it can be used to approximate the flowrate of the liquid through the capillary.

Pressure Chamber

The pressure chamber holds a sample vial, which contains the liquid solution to be electrosprayed. A platinum wire and capillary extend from the top half of the pressure chamber into the liquid solution to charge and transport the liquid solution, respectively. To change the sample vial, the bottom half of the pressure chamber can be removed by twisting ⅛-turn counterclockwise. High voltage is *not* supplied to the platinum wire if the bottom half of the pressure chamber is removed. In addition, an in-line orifice provides a pressure drop equal to the differential pressure set point, eliminating the need to reduce the pressure when changing samples. See Chapter 4, "Operating the Electrospray" for detailed instructions for handling or changing the sample vial.

Viewing Window

The viewing window provides a visual inspection of the capillary tip. This is useful when observing the different operating modes of the Electrospray and when installing a capillary. It is also a good tool for troubleshooting the Electrospray. The viewing window is backlit by a green LED and the lens can be adjusted to focus on the capillary tip by rotating the lens mount.

Ionizer Retainer

The ionizer retainer allows a Model P-2042 Polonium-210 radioactive ionizer to be installed in the Electrospray chamber to meet all radiation safety requirements. The ionizer reduces the charge on the droplets produced by the Electrospray to increase the exit aerosol concentration.

Back Panel

As shown in Figure 3-2, the back panel has power and data connections, as well as air and CO_2 inlets and the aerosol exit.

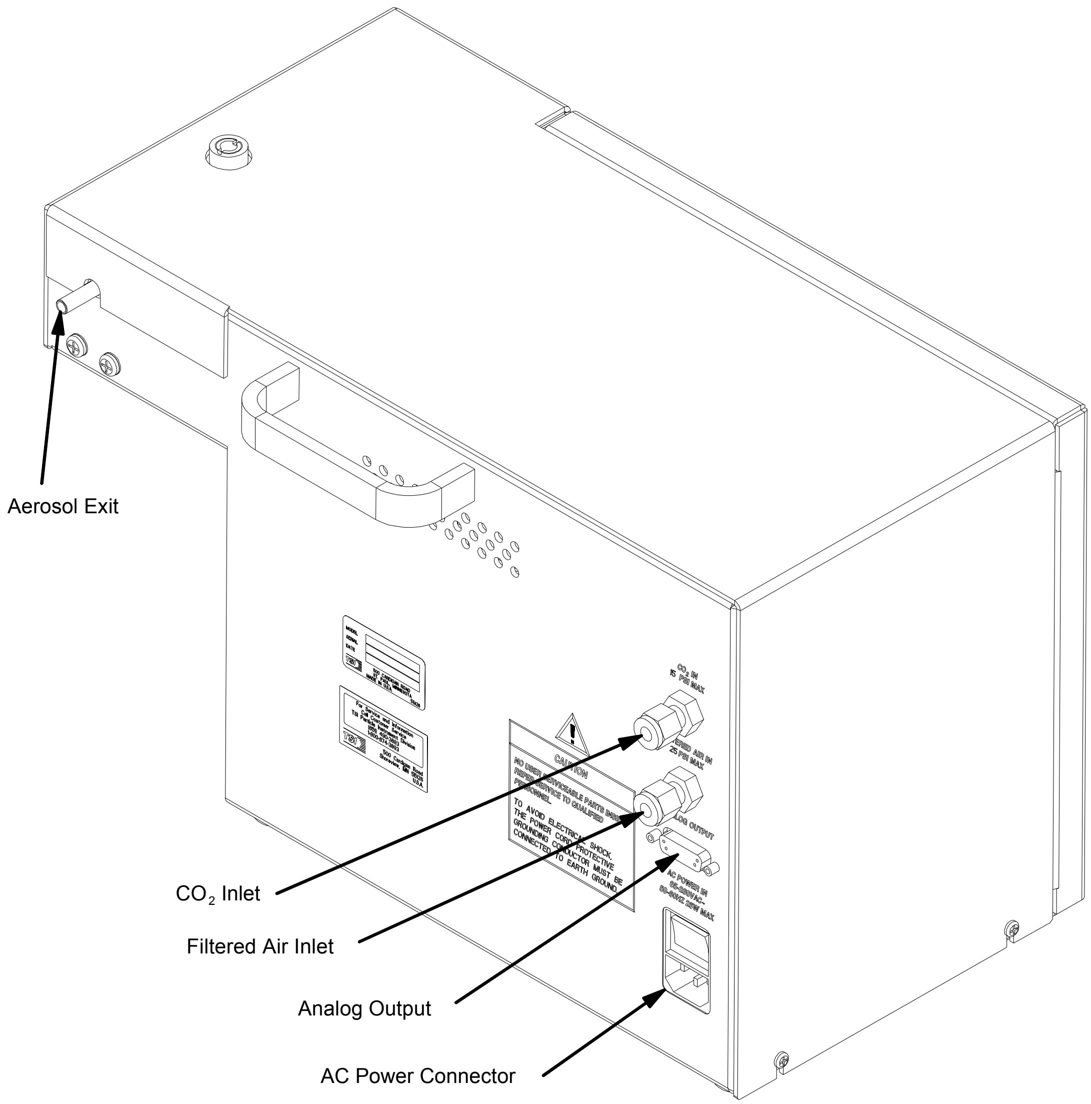


Figure 3-2
Back Panel of the Model 3480 Electrospray Aerosol Generator

Aerosol Exit

Flexible tubing with a diameter slightly less than ¼" or any ¼" Swagelok®-type fitting can be attached to the aerosol exit. Conductive tubing is supplied for use at the aerosol exit to minimize particle loss due to electrostatic charge.

Analog Output

The analog output is a standard 15-pin D-sub connection that allows analog signals to be read by an external instrument to collect Electrospray voltage and current information. The pin designations and signal connections are shown in Figure 3-3 and Table 3-1, respectively. Note the conversion factors in Table 3-1.

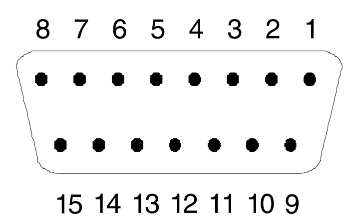


Figure 3-3
ANALOG OUTPUT Pin Designations

Table 3-1
Signal Connections for Analog Output Configurations

Pin Number(s)	Signal
1	Voltage Output (1V = 1.00 kV)
2	Voltage GND
3	Current Output (1V = 100 nA)
4	Current GND
5-15	—

AC Power Connector

The AC power connector accepts the line cord (supplied) to provide AC power to the instrument. Line voltage can be 85 to 260 VAC, 50/60 Hz, single phase, 3A max. The connector has a built-in on/off switch. Power consumption is 25 Watts, max.

Note: *Make certain the line cord is plugged into a grounded power outlet. Position the Electrospray so the power cord connector is easily accessible.*

Filtered Air Inlet

The filtered air inlet connection is described in Chapter 2, "Unpacking and Setting Up the System."

CO_2 Inlet

The CO_2 inlet connection is described in Chapter 2, "Unpacking and Setting Up the System."

Internal Components

The main internal components are shown in Figure 3-4. A brief description of each follows.

Capillary

The capillary transports the liquid to be electrosprayed from the sample vial to the Electrospray chamber. More information on the liquid flow through a capillary can be found in Appendix B, "Theory of Operation." Information on the maintenance of the capillary can be found in Chapter 5, "Maintenance."

Electrospray Chamber

The aerosol is generated inside the Electrospray chamber. See Chapter 5, "Maintenance" for instructions on how to clean and maintain the Electrospray chamber. Appendix B, "Theory of Operation" contains information on the theory of operation of the Electrospray chamber.

High-Voltage Fitting and Shield

The high-voltage fitting is attached to the top half of the pressure vessel. High voltage is supplied to the fitting, which is connected to a platinum high-voltage wire that is immersed in the liquid contained in the sample vial, hence charging the liquid. The high-voltage shield covers the high-voltage fitting to protect the user from accidental electrical shock.

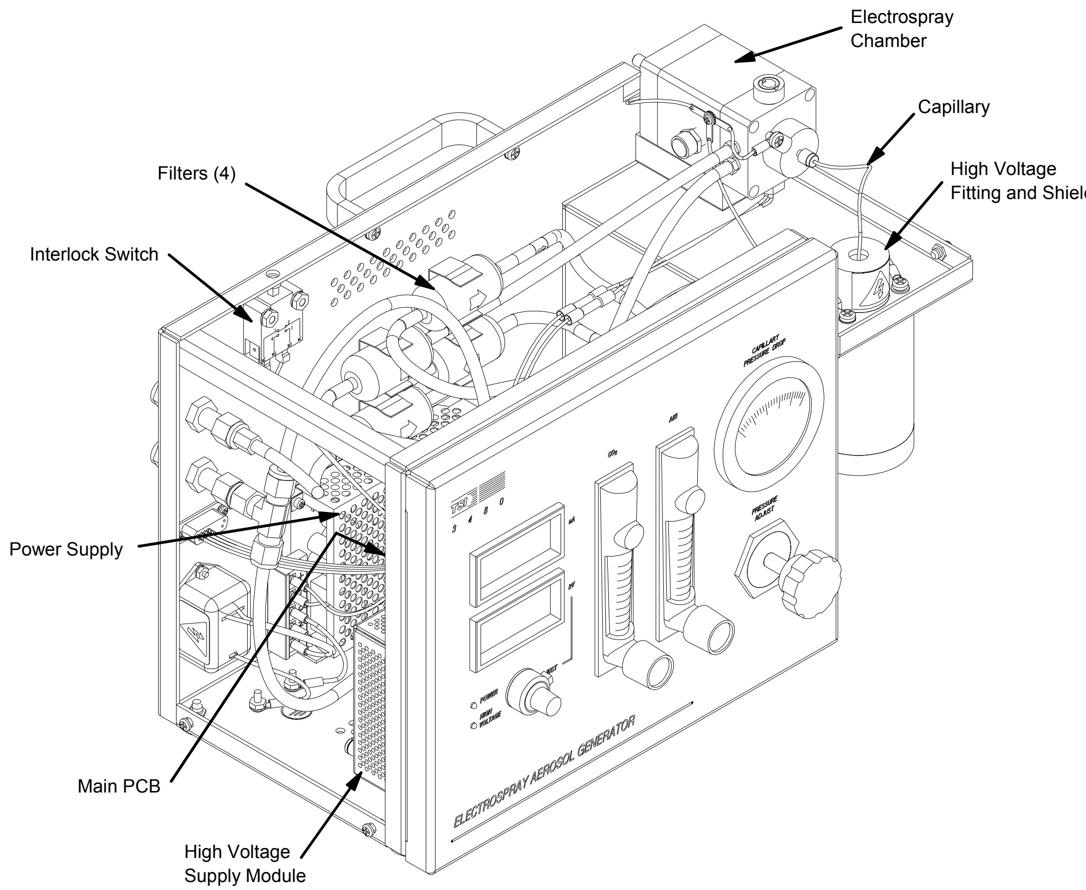


Figure 3-4
Main Internal Components

Power Supply

15V is supplied to the main PCB by the 25W power supply. The power supply contains no user serviceable parts.

Main PCB

Controls all the electronics in the Electrospray. This PCB is calibrated at the factory and there should be no need to make adjustments to its components under normal usage.

High-Voltage Supply Module

The high voltage supplied to the Electrospray chamber is generated by this component. Two modules are available: positive high voltage (standard) and negative high voltage. See Chapter 5 for instructions on servicing the high-voltage supply module.

Interlock Switch

The interlock switch is a safety measure to ensure that high voltage will not be supplied to any part of the instrument unless the instrument cover is in its proper position.

Filters

These filters are used to clean the air and CO_2 in various flow paths in the instrument. See Appendix B, "Theory of Operation" for a flow schematic of the Electrospray and see Chapter 5, "Maintenance" for information related to the maintenance of these filters.

CHAPTER 4

Operating the Electrospray

Use the information in this chapter to become familiar with how to prepare liquid solutions and operate the Model 3480 Electrospray Aerosol Generator.

Preparing Samples

Samples can be prepared and stored using commercially available materials, handling procedures, and storage equipment. Typically, a buffer solution is made, the conductivity and pH are adjusted, and a solid or nonvolatile liquid is dissolved in the buffer solution. You may find it most convenient to prepare a large (500 mL) buffer solution adjusted to the desired conductivity and pH, and then dilute sample material with the buffer solution in a standard sample vial (1.7 mL centrifuge tube).

WARNING

Any material or procedure mentioned in this manual is intended for use by qualified professionals familiar with potential chemical hazards and trained in safe laboratory procedures.

Caution

To slow the growth of contaminants in a buffer solution or sample solution, refrigerate them when not in use. In addition, use caution when handling sample vials to avoid transferring contaminants to the vial.

Preparing a Buffer Solution

A buffer solution is intended for use in the Electrospray to clean the capillary either before, after, or between samples. It is also useful to use a buffer solution as the solvent to dissolve solids or nonvolatile liquids when preparing samples. Key guidelines for choosing a buffer solution include the following:

- **volatile solution**—the solution must evaporate in the Electrospray chamber
- **chemical buffer**—the pH of the solution should not change significantly when a sample is made

- **adjustable pH**—when making certain samples, such as proteins, the pH value of the solution must be greater than the isoelectric point (pI value) for the protein or the sample will stick to the capillary walls
- **adjustable conductivity**—the primary droplet size produced by the Electrospray is inversely proportional to the cube root of the conductivity, therefore, there is a finite range of conductivities over which the Electrospray will operate

A buffer solution used extensively in the Electrospray is 20 mM ammonium acetate created by dissolving ammonium acetate in ultrapure water. De-ionized water can be used in place of ultrapure water if necessary; however, ultrapure water is recommended to reduce the chances of buffer or sample contamination growth. 20 mM ammonium acetate is volatile, a weak buffer, and has an adjustable pH and conductivity.

As an example to get started using the Electrospray, a 20 mM ammonium acetate buffer solution with a conductivity of 0.2 S/m adjusted to pH 8 is useful for dissolving solutes such as sucrose, PSL, and proteins, to name a few. The procedure for making this buffer solution (subsequently referred to as the standard buffer solution) is outlined here:

1. Dissolve 0.77 grams of ammonium acetate in 500 milliliters of ultrapure or de-ionized water.
2. Assuming the solution has an initial pH of 6.7, add 0.75 milliliters of 1M ammonium hydroxide.
3. See the following sections for measurement and adjustment of the buffer solution conductivity and pH.

Measuring and Adjusting Conductivity

A conductivity meter is useful for measuring the conductivity of a buffer solution. The conductivity can be increased or decreased by adding ammonium acetate or ultrapure water, respectively, to the buffer solution. Although the Electrospray will operate at a wide range of conductivities, a 0.2 S/m buffer solution is typically used. An experimental analysis of the useful range of liquid conductivity as a function of liquid flowrate using an electrospray method is discussed in the following journal article:

Chen, Da-Ren, David Y.H. Pui, and Stanley L. Kaufman [1995] "Electrospraying of Conducting Liquids for Monodisperse Aerosol Generation in the 4 nm to 1.8 µm Diameter Range." *J. Aerosol Sci.*, 26:963-977.

Measuring and Adjusting pH

A pH meter is useful for measuring the pH of a buffer solution. Ammonium hydroxide or acetic acid can be added to the buffer solution to increase or decrease the pH value, respectively. Since the pH value should be greater than the pI value for proteins, a useful reference for pI values of selected proteins is:

Righetti, P.G., G. Tudor, and K. Ek [1981] "Isoelectric Points and Molecular Weights of Proteins: A New Table." Chromatographic Reviews 149, *J. Chromatography*, 220:115-194.

Typical Solutes

Any solid or nonvolatile liquid that is soluble in a buffer solution can be used to make a sample for use with the Electrospray. With a standard buffer solution, useful solutes include sucrose, PSL, and proteins. The properties of each solute and methods of preparing each sample are discussed below.

Sucrose

The sucrose concentration of a sample can be varied to create continuously variable particle sizes. For a given conductivity and flowrate, the diameter of the primary droplets produced by the Electrospray will remain the same. Once the liquid evaporates from the primary droplet, the sucrose contained within the volume of the primary droplet will determine the resulting particle diameter. For example, if the concentration of sucrose is 10% volume sucrose per unit volume buffer (10% V/V), the resulting particle diameter will be $\sqrt[3]{10/100} = 46\%$ of the primary droplet diameter. Similarly, for a 0.1% V/V solution, the particle diameter will be 10% of the primary droplet diameter. Therefore, the primary droplet diameter generated by the Electrospray can be determined if the particle diameter and sucrose concentration are known.

Sucrose solutions can be made using the following method:

1. Dissolve 1.58 grams sucrose per 10 milliliters buffer to make a 10% V/V solution.
2. For lower concentrations of sucrose, dilute the 10% V/V solution with buffer solution accordingly.

Note: *Using a standard buffer solution, sucrose concentrations greater than 10% V/V are less desirable to use with the Electrospray because the viscosity of the sucrose solution increases significantly.*

PSL (Polystyrene-Latex)

PSL is used to generate test aerosols in a wide size range, from 20 nm to 160 µm—the Electrospray is useful on the low end of this range, from 20 to 100 nm. PSL is commercially available as aqueous suspensions in 15 mL dropper bottles, and samples can be easily made by adding one drop (50µL) of PSL to 1 mL of buffer solution in a standard sample vial. Unlike sucrose, PSL size is independent of concentration. Higher aerosol concentrations can be obtained by adding more PSL, which may become necessary with larger PSL sizes.

Proteins

Most proteins that have been used with the Electrospray range from three to 12 nm in diameter. Most are commercially available from many chemical companies. The diameters of particles generated from proteins are independent of sample concentration. Protein samples can be prepared using the following method:

1. Dissolve 1 mg protein per 1 milliliter buffer solution.
2. Dilute the 1mg/mL solution to obtain a 10 µg/mL solution. This concentration is sufficient for most proteins when analyzing the size distribution of the protein aerosol.

Selected proteins and their diameters are listed in Table 4-1. This is a very limited sample of the numerous proteins available.

Caution
Some proteins may be dangerous to emit into the environment. Filter dangerous aerosols before emitting them into the environment.

Table 4-1
Selected Proteins and Their Diameters

Protein	Diameter (nm)
Ubiquitin	3
Bovine Serum Albumin	6.5
Ferritin	12

Starting Up the Electrospray

After the Electrospray system has been set up (see Chapter 2, "Unpacking and Setting Up the System") and samples have been made, follow this procedure to begin generating aerosols:

1. Turn on the power switch on the back panel. Make sure the power and high-voltage LED's are on and check the viewing window to make sure the capillary tip is near the center of the viewing window.
2. Remove the bottom half of the pressure chamber by twisting it $\frac{1}{8}$-turn counterclockwise. The bottom half can then be removed by pulling downward.

   Note: *High voltage is turned off if the bottom half of the pressure chamber is removed.*
3. Insert and open the sample vial as shown in Figure 4-1. Use caution not to contaminate the sample.
4. Replace the bottom half of the pressure chamber by aligning the grooves to the pins in the top half and pushing the bottom half upwards, making sure the capillary and platinum wire are aligned with the sample vial. As the pins enter the grooves, continue pushing upwards while turning the bottom half clockwise until the bottom half locks in place.

WARNING

The capillary and platinum wire are adjusted for use with a 1.7 mL centrifuge tube, if it is necessary to use a different sized vial, see Chapter 5, "Maintenance" for instructions on adjusting the capillary and platinum wire positions to avoid breaking the capillary or damaging the platinum wire.

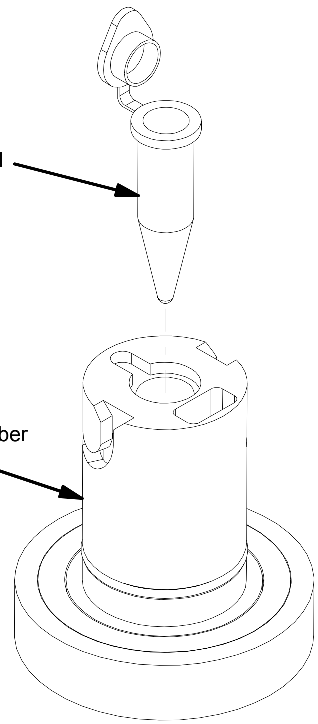


Figure 4-1
Inserting a Sample Vial into the Pressure Chamber

5. Turn the pressure regulator knob clockwise until the pressure gauge reads 3.7 psi.
6. Turn the air rotameter knob counterclockwise to reach 1.0 lpm.
7. Turn the CO_2 rotameter knob counterclockwise to reach 0.1 lpm.
8. Turn the voltage adjustment knob clockwise to reach 2.30 kV.
9. Within two minutes the liquid should be exiting the capillary. See Chapter 6, "Troubleshooting" if the liquid has not made it through the capillary after two minutes.

Generating Aerosols

The Electrospray operating parameters set in the "Starting Up the Electrospray" section should result in a stable generation of aerosol in the Cone-Jet mode of the Electrospray (see below for an explanation of the "Cone-Jet" mode). Once the liquid is flowing from the capillary tip, the high voltage can be adjusted to achieve different Electrospray operating modes. Four distinct modes (see Figure 4-2) can be observed through the viewing window or by the current LED display as the electric field is increased by increasing the high voltage: Dripping mode, Pulsating mode, Cone-Jet mode, and Corona Discharge mode. Figure 4-1 shows three photographs of the capillary tip viewed through the viewing window.

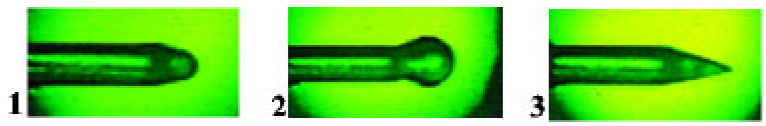


Figure 4-2

Three Views of Capillary Tip Through the Viewing Window: (1) No liquid flow, (2) Liquid flow but no electric field (Dripping mode), (3) Liquid flow and an electric field (Cone-Jet mode). The third view illustrates stable electrospray operation.

- **Dripping mode** (view 2 in Figure 4-2) occurs when a primary droplet builds up on the end of the capillary until it becomes large enough to be pulled away by the weak electric field. The current display is usually near zero in this mode. Inspection through the viewing window shows the primary droplet increasing in size until it is pulled away at a very low frequency. As the voltage is increased in this mode, the primary droplet size decreases.
- In **Pulsating mode**, the liquid at the tip of the capillary continues to drip, but at a higher frequency than in dripping mode. In addition, a visual inspection shows a more cylindrical liquid profile with a cone-shaped tip. During operation, the cylindrical liquid profile increases in length and the current display increases until the primary droplet is pulled away (and the current display rapidly decreases). As in the dripping mode, as the voltage is increased, the primary droplet size decreases while the frequency increases.
- The **Cone-Jet mode** (view 3 in Figure 4-2) occurs when the electric field strength is balanced with the liquid properties. A visual inspection shows the conical shape of the profile (Taylor Cone), but the jet of primary droplets cannot be seen since the primary droplets are very small. As voltage is increased, the profile does not change significantly while in this mode; in addition, the total current reading changes very little. The cone-jet mode is the most stable and useful mode for generating aerosols. The voltage setting in this mode is typically 2.00 to 2.50 kV using a 20 mM ammonium acetate buffer solution.

- As the voltage is increased further, the electric field becomes too strong for the liquid and surrounding gas and the **Corona Discharge mode** occurs. The strong electric field causes the tip of the liquid to become rounded. Also, the current reading is high because of the current travelling through the CO_2/air sheath flow, and erratic because of the instability of this mode.

Changing Samples

Caution must be used when changing samples to avoid contaminating either of the samples. A buffer solution should be run in the Electrospray between samples to clear the capillary of possible contaminants. Follow the procedure below (see “Starting Up the Electrospray” earlier in this chapter for details) to change samples.

Caution

To avoid contaminating samples, a buffer solution should be run between samples to clear the capillary of possible contaminants.

1. Remove the bottom half of the pressure chamber.
2. Close the lid of the vial and remove it.
3. Insert a vial containing buffer solution.
4. Replace the bottom half of the pressure chamber.
5. Run buffer solution for one minute.

***Note**: The residence time in a capillary using a standard buffer solution at 3.7 psi is approximately 112 seconds. For most applications, running a buffer solution for 60 seconds will sufficiently prevent sample contamination; however, using a highly viscous fluid or a low capillary pressure drop will increase the residence time. See Appendix B, “Theory of Operation” for an analysis of liquid flow in the capillary.*

6. Repeat steps 1–4, using a sample vial rather than a buffer solution.

7. Aerosol should be produced after a time equal to the residence time of the particular sample and operating conditions.

Shutting Down the Electrospray

The capillary should be cleaned and cleared of liquid before shutting down the Electrospray to avoid deposits on the inside of the capillary. Follow the procedure below (see "Starting Up the Electrospray" for details) to shut down the Electrospray.

Caution

Buffer should be run before shutting down the Electrospray to clean the capillary. Subsequently, the Electrospray should be run without a sample to allow the air to clear the capillary of liquid. Follow the procedure in this section to prolong capillary life and to avoid deposits on the inside of the capillary.

1. Purge the capillary (see section, "Purging the Capillary" in Chapter 5, "Maintenance").
2. Remove the bottom half of the pressure chamber.
3. Close the lid of the vial and remove it.
4. Replace the bottom half of the pressure chamber *without* inserting a vial to clear the liquid from the capillary and run air through it.
5. View the capillary through the viewing window to determine when the liquid has been cleared from the capillary. This process takes approximately 5 minutes with a standard buffer solution.
6. Turn the pressure regulator knob until the pressure gauge reads zero psi.
7. Turn the air and CO_2 rotameters to read zero lpm.
8. Turn the voltage knob counterclockwise until it reaches its stopped position.
9. Turn the power switch off.

CHAPTER 5
Maintenance

This chapter gives maintenance and service procedures for the Electrospray.

Periodic Maintenance

Periodic cleaning of several parts of the Electrospray is necessary to ensure proper performance. The capillary should be cleaned after each day's use to prevent buildup of solutes. If aerosol deposits accumulate on the orifice plate, the orifice could be reduced in size or eventually plugged. In addition, aerosol deposits will buildup on the Electrospray chamber and viewing window optics, although at a lesser frequency than on the orifice plate. The service intervals depend on the type and concentration of aerosols generated with the Electrospray. The following maintenance schedule is suggested for the Electrospray:

Table 5-1
Maintenance Schedule

Maintenance Task	How Often
Purging the capillary	Daily
Cleaning the orifice plate	As needed
Cleaning the aerosol exit port	As needed
Cleaning the viewing window lens	As needed
Replacing the ionizer	12 months
Cleaning the Electrospray chamber	As needed
Performing an air leak test	If you clean the Electrospray chamber or the viewing window lens.
Cleaning the pressure chamber	As needed

Purging the Capillary

The capillary should be purged after each day's use to keep solutes from building up on the inside of the capillary. It may need to be purged more often depending on the chemicals you are using or if you suspect the capillary has become partially or fully plugged.

WARNING

Any material or procedure mentioned in this manual is intended for use by qualified professionals familiar with potential chemical hazards and trained in safe laboratory procedures.

To purge the capillary, place one of the following solutions in a sample vial and use it as if you were generating an aerosol (see the section in Chapter 4, "Operating the Electrospray"). When shutting down the instrument, always rinse the capillary with a buffer solution and flush the capillary with air after purging the capillary to prevent deposits from forming on the capillary. Four different solutions have been used with the Electrospray to purge the capillary. They include:

- 20 mM ammonium acetate buffer solution (see the section in Chapter 4, "Preparing Samples"). This solution is convenient to use because it is the same solution recommended for use between samples to prevent sample contamination. However, this solution rinses the capillary but does not contain cleaning agents. If you suspect the capillary walls are dirty, use one of the three solutions listed below to clean the capillary and then a buffer solution to rinse the capillary.

Caution

20 mM ammonium acetate buffer solution or a similar buffer solution should be run to rinse the capillary after using any detergent such as Tween 80 or any strong alkali such as KOH to purge the capillary. After the capillary is rinsed with the buffer solution and before turning off the instrument, run air through the capillary to prevent deposits from forming if the buffer solution were left in the capillary to evaporate.

- 0.5% Tween® 80 solution (a detergent). 10% Tween 80 solution is available from Fluka and is useful if diluted with ultrapure or distilled water to 0.5%.

  Fluka
  1001 West St. Paul Avenue
  Milwaukee, WI 53233 U.S.A.
  Phone: (414) 273-5013
  Fax: (414) 273-4979
  Order: 800-358-5287; Fax 800-441-8841
  E-Mail: flukausa@sial.com

- 20 mM KOH solution. A 20 mM potassium hydroxide solution in ultrapure or distilled water is a low enough concentration to clean the capillary with little etching of the capillary walls. This solution should not be run for more than 10 minutes because capillary etching may become significant.
- M KOH solution. For heavy buildup, a 0.1 M potassium hydroxide solution in ultrapure or distilled water can be used; however, this solution should not be run for more than 2 minutes because capillary etching may become significant more quickly than for the 20 mM KOH solution.

Cleaning the Orifice Plate

WARNING

High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

The orifice plate is permanently attached to the inlet fitting of the Electrospray chamber to ensure electrical contact. Since the aerosol is generated inside the inlet fitting and then passes through the orifice plate, the inlet fitting will need to be cleaned more frequently than other components through which the aerosol passes. Figure 5-1 shows a view of the inlet fitting inside the cabinet. Use the following steps to clean the inlet fitting and orifice plate:

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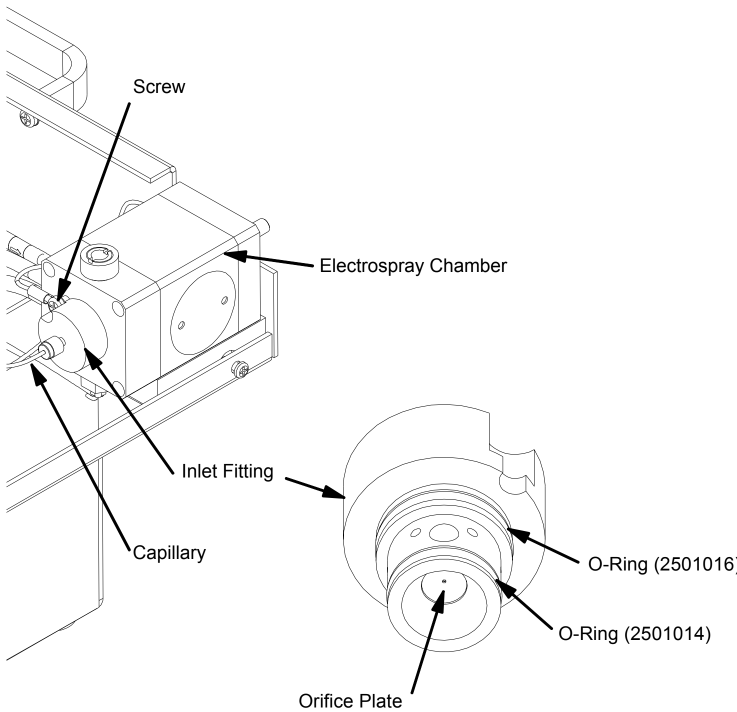


Figure 5-1
Inlet Fitting

1. Remove power from the instrument and turn off all flows.
2. Remove the cover by loosening the four screws securing the cover (they do not have to be removed) and pulling the cover upward while pushing down on the top of the front panel of the instrument or on the viewing window mount.
3. Remove the screw that holds the inlet fitting in place.
4. Pull the inlet fitting out of the Electrospray chamber. Use a twisting motion if necessary.
5. Detach the capillary from the inlet fitting (see "Removing the Capillary" later in this chapter).
6. Remove the O-rings using a tweezers or similar tool. Inspect the O-rings for damage and replace if necessary.
7. Use a soft cloth soaked in alcohol or a mild solvent to clean the outside of the inlet fitting and flush the inside of the inlet fitting with alcohol or a mild solvent to clear any deposits that may have formed.
8. Rinse the inlet fitting with clean water.

9. Blow clean, dry air through and on the inlet fitting to evaporate the water.
10. Replace the O-rings and lightly apply the vacuum grease supplied with the instrument. Remove any excess grease.
11. Replace the inlet fitting and reinstall the capillary (see "Installing the Capillary" later in this chapter).
12. Replace the cover on the instrument and tighten the four screws that secure the cover.

Cleaning the Aerosol Exit Port

The aerosol exit port can be cleaned as needed without disassembling the instrument. Simply remove power and turn off all flows to the instrument and use a cotton swab moistened with alcohol or a mild detergent to clean the inside of the aerosol exit port.

Cleaning the Viewing Window Lens

If the viewing window lens becomes dirty, it can be cleaned without removing the cover from the instrument. First try cleaning the lens without disassembling the instrument by using a cotton swab soaked in alcohol, a mild detergent, or a lens cleaner. If the lens is still dirty, use the following procedure to clean the viewing window lens:

1. Remove power from the instrument and turn off all flows.
2. Using Figure 5-2 as a guide, unscrew the viewing window mount by hand and remove the lens ring retainer using a coin.
3. Turn the viewing window mount upside down to remove the lens.
4. Clean the lens using a soft cloth soaked in alcohol, a mild detergent, or a lens cleaner.
5. If necessary, the O-rings can be removed from the viewing window mount and the viewing window mount can be cleaned using a soft cloth soaked in alcohol or a mild detergent.
6. Replace the lens in the viewing window mount, making sure the O-rings are installed properly. The flat side of the lens should be facing the O-ring.

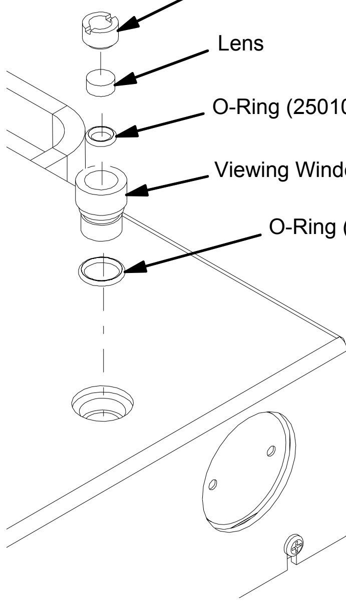
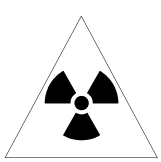


Figure 5-2
Viewing Window Assembly

7. Secure the lens by tightening the lens ring retainer using a coin. Do **not** overtighten as it could damage the lens.
8. Lightly grease the external O-ring and wipe off any excess grease before replacing the viewing window mount in the Electrospray chamber.

If the view of the capillary tip is still unsatisfactory after focusing the lens, see the section later in this chapter, "Cleaning the Electrospray Chamber."

Replacing the Ionizer



W A R N I N G
The use of controls, adjustments, or procedures other than those specified in this manual may result in exposure to hazardous radiation.

The ionizer does **not** need to be removed or cleaned under normal operating conditions. However, the ionizer should be removed if the Electrospray chamber needs to be cleaned or if the ionizer is more than twelve months old. Use the following procedure to replace the ionizer. Refer to Figure 2-2 if necessary.

1. Remove the ionizer retainer using the ionizer retainer tool supplied in the accessory kit. Contact TSI for instructions on returning the ionizer.
2. Place the new ionizer in the ionizer retainer. The ionizer mesh-side should be facing the open end of the ionizer retainer.
3. Install the ionizer retainer, making sure the O-ring is between the ionization chamber and the ionizer retainer, and tighten with the ionizer retainer tool.

Cleaning the Electrospray Chamber

W A R N I N G
High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

The Electrospray chamber should not need cleaning as often as the orifice plate under most operating conditions. However, the Electrospray chamber should be checked periodically for corrosion or aerosol deposits depending on the nature of the chemicals used in the Electrospray. It is easiest to clean the Electrospray chamber while cleaning the orifice plate. Refer to Figure 5-3 and use the following procedure to clean the Electrospray chamber:

1. Follow steps 1 through 10 in the section “Cleaning the Orifice Plate” earlier in this chapter. In addition, you may want to remove the capillary to avoid damage while cleaning the Electrospray chamber. See the section “Removing the Capillary” later in this chapter for details.
2. Remove the ionizer using the ionizer retainer tool supplied in the accessory kit and place the ionizer in a safe place. Use Figure 2-2 as a guide if necessary.

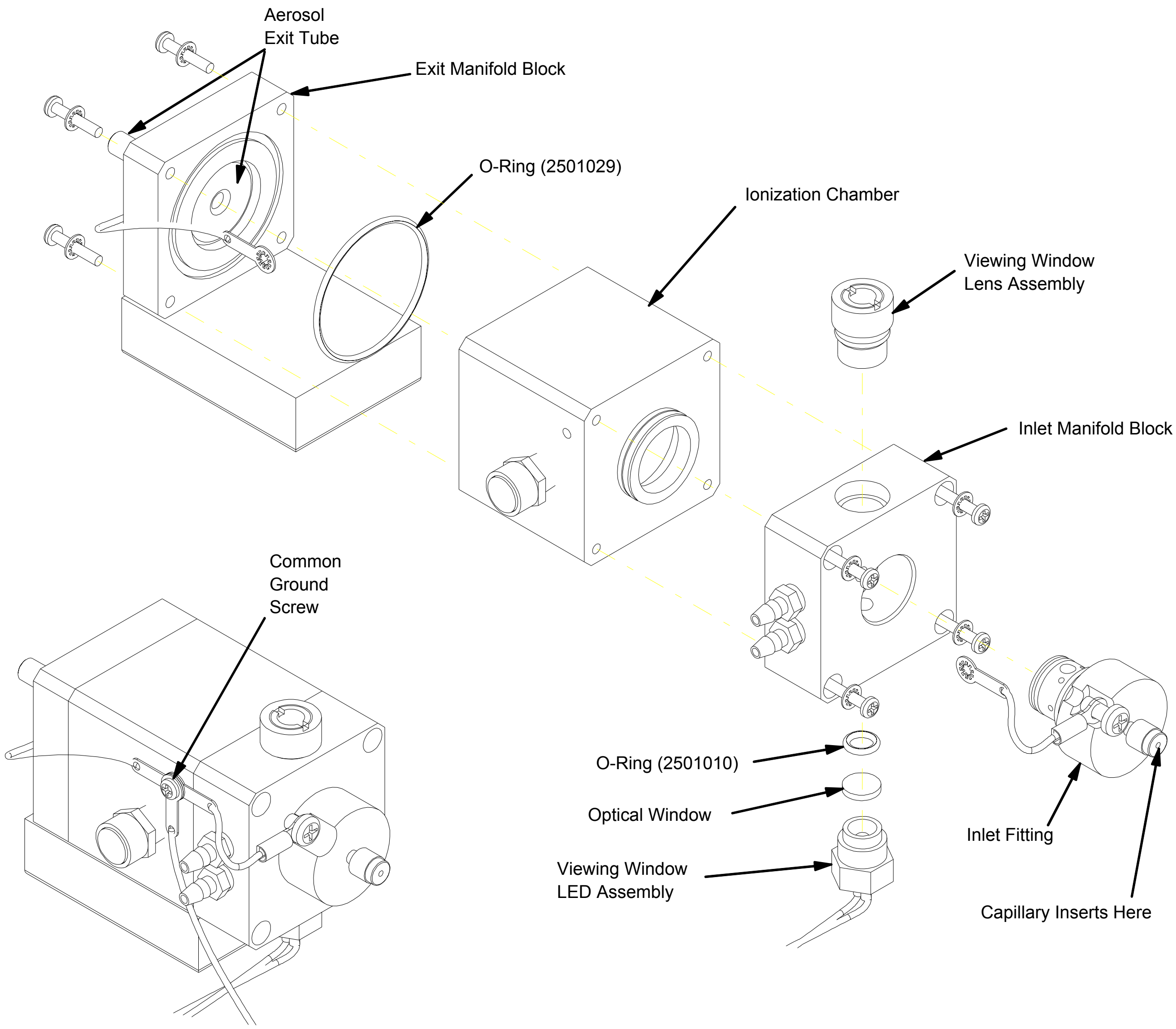


Figure 5-3
Cleaning the Electrospray Chamber

3. Remove the common ground screw on the side of the Electrospray chamber. Also detach the viewing window LED connector from the main PCB.
4. Detach the Electrospray chamber from the cabinet by removing the two screws on the back panel of the instrument behind the Electrospray chamber (not shown in Figure 5-3).
5. Remove the eight screws that fasten the two white manifold blocks to the inlet and exit of the ionization chamber. The

viewing window lens can be cleaned if necessary by following the instructions in the section “Cleaning the Viewing Window Lens” earlier in this chapter. The viewing window LED mount can also be removed to clean the optical window it holds in place by using the following procedure:

a) Unscrew the viewing window LED mount. The optical window and an O-ring will fall out of the inlet manifold.

b) Clean the optical window using a soft cloth or cotton swab moistened with alcohol, a mild detergent, or a lens cleaner.

Caution
Before replacing the viewing window LED mount, make sure the optical window is flush with the O-ring. Failure to do so could result in breakage of the optical window as the viewing window LED mount is screwed into the inlet manifold.

c) Place the O-ring and optical window in the inlet manifold, making sure the optical window is flush with the O-ring surface, then replace the viewing window LED mount.

6. Use a soft cloth or cotton swab moistened with alcohol or a mild detergent to clean the inside of the aerosol exit tube and the stainless steel surface of the aerosol exit tube that faces the inside of the Electrospray chamber.

7. Clean the inside of the ionization chamber using a soft cloth or cotton swab moistened with alcohol or a mild detergent.

8. Reassemble the inlet and exit manifolds to the ionization chamber and connect the Electrospray chamber to the cabinet.

9. Reattach the viewing window LED connector to the main PCB and reconnect the three ground wires to the ionization chamber.

10. Place the ionizer in the ionizer retainer. The ionizer mesh-side should be facing the open end of the ionizer retainer.

11. Install the ionizer retainer, making sure the O-ring is between the ionization chamber and the ionizer retainer, and tighten with the ionizer retainer tool.

12. Replace the inlet fitting. If needed, refer to step 11 in the section “Cleaning the Orifice Plate” earlier in this chapter.

13. Install the capillary tube using the instructions later in this chapter titled “Installing the Capillary.”

14. Perform and air leak test using the instructions below.

Performing an Air Leak Test

Whenever an air leak is suspected or a significant part of the Electrospray chamber has been reassembled, perform a leak test to assure proper operation. Use these steps to check the entire instrument for leaks. Skip to the next section if you want to isolate the Electrospray chamber and check it for leaks.

W A R N I N G
High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

Leak Checking the Entire Electrospray Instrument

1. Remove power from the instrument and turn off all flows.
2. Remove the cover by loosening the four screws securing the cover (they do not have to be removed) and pulling the cover upward while pushing down on the top of the front panel of the instrument or on the viewing window mount.
3. Make sure a capillary is installed and all internal tubing is connected properly (see the flow schematic in Appendix B).
4. Turn off the external air and CO_2 supplies or disconnect the external air and CO_2 connections and plug each port.
5. Plug the exhaust filter inside the instrument.
6. Connect a pressure gauge or manometer between the aerosol exit port and a leak-tight valve. The valve should be closed.
7. Connect the valve to a vacuum source or a vacuum pump capable of drawing 18 inHg vacuum.
8. Turn on the vacuum pump or vacuum system and slowly open the valve. Allow the vacuum gauge reading to become stable and then close the valve.
9. Observe the gauge reading. The reading should not change by more the 0.5 inHg in 1 minute.

10. If the instrument does leak, follow the steps below under "Isolating Leaks," or continue with the section, "Leak Checking the Electrospray Chamber and Pressure Chamber."

Leak Checking the Electrospray Chamber and Pressure Chamber

1. Remove power from the instrument.

2. Remove the cover by loosening the four screws securing the cover (they do not have to be removed) and pulling the cover upward while pushing down on the top of the front panel of the instrument or on the viewing window mount.

3. Make sure a capillary is installed.

4. Disconnect the tubing from the barbed fitting on the pressure chamber and the two barbed fittings on the Electrospray chamber.

5. Plug all three barbed fittings.

6. Connect a pressure gauge or manometer between the aerosol exit port and a leak-tight valve. The valve should be closed.

7. Connect the valve to a vacuum source or a vacuum pump capable of drawing 18 inHg vacuum.

8. Turn on the vacuum pump or vacuum system and slowly open the valve. Allow the vacuum gauge reading to become stable and then close the valve.

9. Observe the gauge reading. The reading should not change by more the 0.5 inHg in 1 minute.

10. If the instrument does leak, follow the steps below under "Isolating Leaks."

Isolating Leaks

1. Isolate the leak by wetting suspected joints with clean isopropyl alcohol while the system is under vacuum. The alcohol will be drawn into a leaky joint and evaporate.

2. After isolating the leak, repair it (usually by greasing or replacing an O-ring, or by sealing a fitting).

3. Blow clean, dry air through the repaired section to evaporate and remove any leftover alcohol.

4. If you cannot find the leak, or cannot repair the leak, please contact TSI for assistance.

Cleaning the Pressure Chamber

The pressure chamber can be cleaned if the surfaces appear dirty or the sample vial is difficult to see clearly. Refer to Figure 5-4 and use the following procedure to clean the pressure chamber:

W A R N I N G

High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

1. Remove power from the instrument and turn off all flows.
2. Remove the capillary using the instructions in the next section titled "Removing the Capillary."
3. Remove the screw attaching the high-voltage cable to the fitting on top of the pressure chamber. The fitting can then be removed by twisting counter-clockwise, using caution not to bend or damage the platinum high-voltage wire.

C a u t i o n

When removing the fitting on top of the pressure chamber, use caution not to bend or damage the platinum high-voltage wire.

4. Clean the top and bottom of the pressure chamber using a soft cloth or cotton swab moistened with alcohol or a mild detergent.
5. Generously grease the O-ring on the bottom half of the pressure chamber and the pin grooves using the vacuum grease supplied in the accessory kit. Remove excess grease and insert the bottom half into the top half of the pressure chamber. Reapply vacuum grease to the O-ring and pin grooves if necessary.
6. Reinstall the high-voltage fitting, using caution not to bend or damage the platinum high-voltage wire.

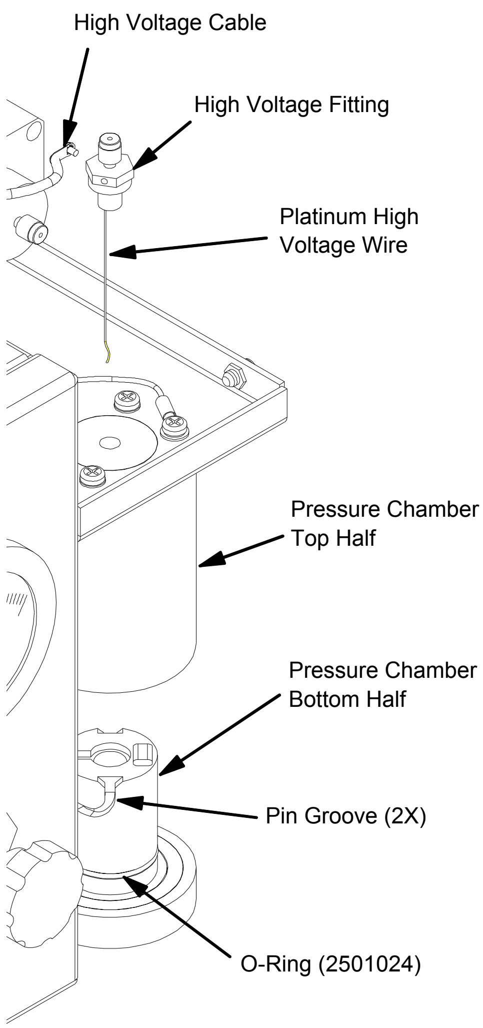


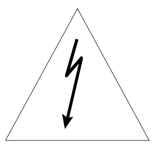
Figure 5-4
Cleaning the Pressure Chamber

7. Reattach the high-voltage cable to the inlet fitting and install the capillary using the instructions in the next section titled "Installing the Capillary."

Special Maintenance

Removing the Capillary

The capillary needs to be removed when cleaning any part of the Electrospray chamber or if the capillary becomes damaged or the capillary tip becomes dirty. Use the following steps and Figure 5-5 to remove the capillary:



W A R N I N G
High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

1. Remove power from the instrument and turn off all flows.
2. Remove the cover by loosening the four screws securing the cover (they do not have to be removed) and pulling the cover upward while pushing down on the top of the front panel of the instrument or on the viewing window mount.

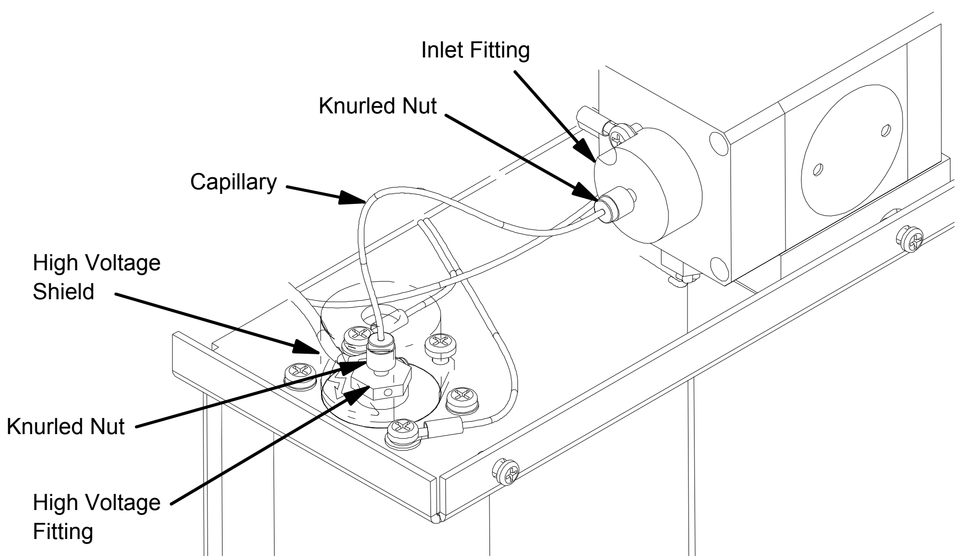


Figure 5-5
Removing or Installing the Capillary

3. Loosen the knurled nut on the inlet fitting but do not remove it.
4. Carefully pull the capillary from the inlet fitting.

Caution
When removing the capillary from the inlet fitting, the capillary can become damaged if not removed carefully. Read the note below for hints on removing the capillary without damage.

***Note**: When removing the capillary slowly, the capillary tip will have a tendency to hit the edge of the knurled nut on the inlet fitting if it is not pulled straight out from the inlet fitting. To prevent damage to the capillary, use a steady hand and pull the capillary straight out from the inlet fitting or pull the capillary out slowly until it is almost removed and then pull quickly to completely remove the capillary. One alternative solution is to remove the inlet fitting before removing the capillary. Another is to perform steps 5–7 before steps 3 and 4.*

5. Remove the high-voltage shield by loosening the mounting screw and pulling the high-voltage shield upward until it clears the capillary.
6. Loosen the knurled nut on the high-voltage fitting but do not remove it.
7. Pull the capillary straight up until it clears the high-voltage fitting.
8. Store the capillary in the case provided to prevent breakage.

Cleaning the Capillary Tip

If the capillary tip appears dirty, remove the capillary using the instructions above, and rinse or soak the capillary tip in alcohol or a mild detergent until the contaminants are removed. Then install the capillary using the instructions in the next section.

Installing the Capillary

WARNING
High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

To install the capillary, refer back to Figure 5-5 and use the following instructions:

1. Remove power from the instrument and turn off all flows.
2. Remove the cover by loosening the four screws securing the cover (they do not have to be removed) and pulling the cover upward while pushing down on the top of the front panel of the instrument or on the viewing window mount.
3. Loosen the knurled nut on the inlet fitting but do **not** remove it.
4. Remove the high-voltage shield by loosening the mounting screw and pulling the high-voltage shield upward.
5. Loosen the knurled nut on the high-voltage fitting but do not remove it.
6. Insert the long end of the capillary into the high-voltage fitting until the end of the capillary reaches the end of the platinum wire. Twist the capillary until it becomes retained by the corkscrew of the platinum wire, then tighten the knurled nut on the high-voltage fitting.
7. Attach the high-voltage shield to the cabinet by sliding the capillary through the high-voltage shield, routing the high-voltage wire through the groove in the bottom edge of the high-voltage shield, and tightening the mounting screw.
8. Turn on the power to the instrument. If the cover is removed, high voltage will not be supplied to the instrument, but the capillary viewing window LED will turn on.
9. Carefully insert the capillary tip into the inlet fitting. While looking through the viewing window, continue pushing the capillary into the inlet fitting until the tip of the capillary first appears, then tighten the knurled nut on the inlet fitting.
10. If the capillary is centered in the viewing window, installation is complete. If not, loosen the knurled nut on the inlet fitting, adjust the position of the capillary, and retighten the knurled nut.

Note: *Due to variations in the dimensions of the viewing window components, the capillary may appear slightly off center in the direction transverse to the capillary axis. This is not a cause for concern however, because the center of the orifice plate is aligned to the inlet fitting axis at the factory.*

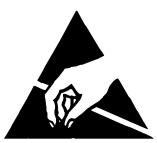
11. Replace and secure the cabinet cover. The Electrospray is now ready for use (See Chapter 4, "Operating the Electrospray").

Adjusting the Position of the Capillary Tip

If the capillary does not appear in the center of the viewing window or you wish to move the capillary closer or further from the orifice plate, refer to the instruction preceding this subsection titled, “Installing the Capillary.” Step 10 lists instructions for adjusting the position of the capillary tip. Make sure to remove power from the instrument and turn off all flows before removing the cover from the instrument.

Adjusting the Position of the Capillary and the Platinum Wire in the Pressure Chamber

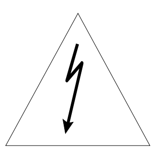
In the pressure chamber, the capillary and the platinum wire are positioned at the factory to extend near the bottom of a standard sample vial (1.7mL centrifuge tube). In most cases, the sample to be electrosprayed can be put into a standard sample vial; however, if a smaller vial must be used, the following things must be done:



C a u t i o n

The electronic circuits within this instrument are susceptible to electrostatic discharge (ESD) damage. Use ESD precautions to avoid damage.

- ❑ Use only a table top with a grounded conducting surface.
- ❑ Wear a grounded, static-discharging wrist strap.



W A R N I N G

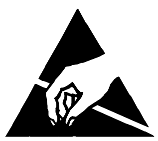
High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

- ❑ Obtain a short piece of a non-conductive piece of hollow tubing or a similar object to place in the bottom half of the pressure chamber to allow the smaller vial to be placed securely.
- ❑ Cut the platinum high-voltage wire to length or replace the platinum high-voltage wire with a new platinum wire of desired shape and size. To modify or replace the platinum high-voltage wire, use the instructions earlier in this chapter titled “Cleaning the Pressure Chamber” to remove the capillary and the high-voltage fitting. The platinum high-voltage wire is soldered to the inside of the brass pin on the bottom of the high-voltage fitting

and can be removed by heating with a soldering iron. Finally, solder the new platinum wire to the inside of the brass pin and replace the high-voltage fitting.

- ❑ Reinstall the capillary using the instructions earlier in this chapter titled “Installing the Capillary.”

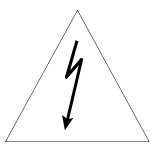
Reversing the High-Voltage Polarity



C a u t i o n

The electronic circuits within this instrument are susceptible to electrostatic discharge (ESD) damage. Use ESD precautions to avoid damage.

- ❑ Use only a table top with a grounded conducting surface.
- ❑ Wear a grounded, static-discharging wrist strap.



W A R N I N G

High voltage is accessible in several locations within this instrument. Make sure you unplug the power source before removing the cover or performing maintenance procedures.

Note: *Refer to Figure 5-6 and note the location of the high-voltage power supply to determine whether a high-voltage module has a positive or negative polarity. In a positive module (shown), the high-voltage power supply is located on the exposed side of the high-voltage PCB. In a negative module, the high-voltage power supply is located on the opposite side of the high-voltage PCB.*

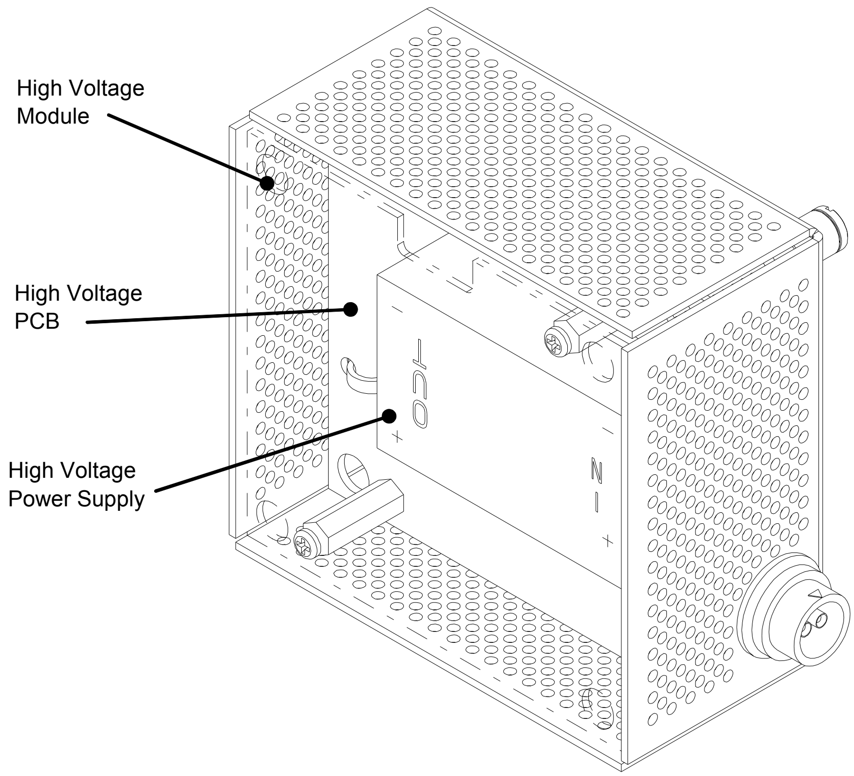


Figure 5-6
Determination of High-Voltage Polarity

The standard Electrospray is equipped with a positive high-voltage module, which causes the generated droplets to initially have a high-positive charge. However, if initially negatively charged droplets are desired, a negative high-voltage module is available from TSI. Reversing the high-voltage polarity is a procedure that should only be performed by a qualified electronics technician observing ESD precautions. To replace one high-voltage module with another, refer to Figure 5-7 and proceed as follows:

1. Remove power from the instrument and turn off all flows.
2. Remove the cover by loosening the four screws securing the cover (they do not have to be removed) and pulling the cover upward while pushing down on the top of the front panel of the instrument or on the viewing window mount.

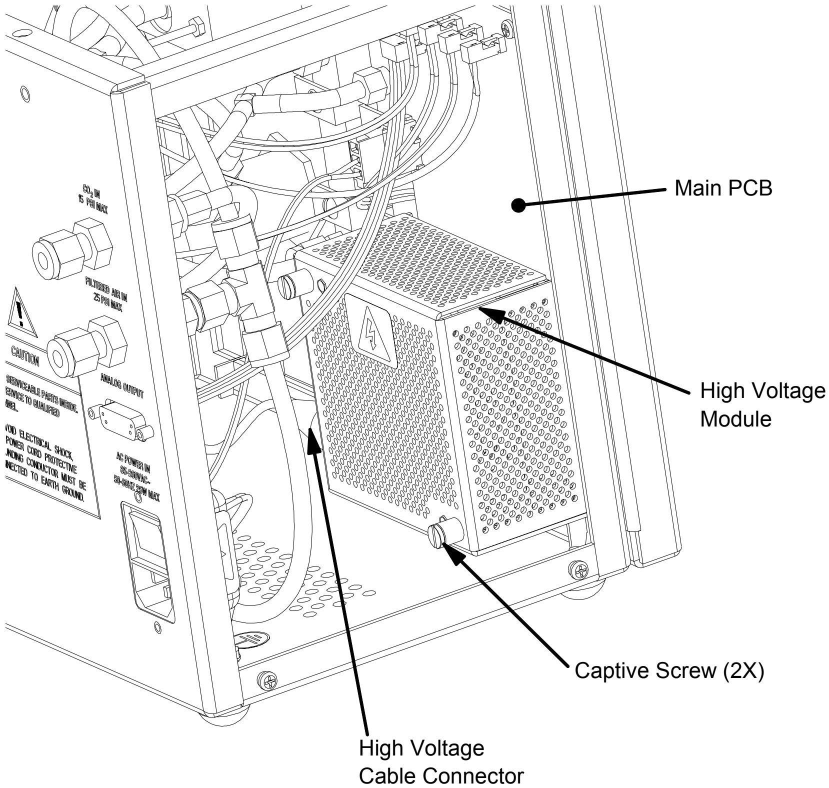


Figure 5-7
Reversing the High-Voltage Polarity

3. Disconnect the high-voltage cable from the high-voltage module by gripping the connector and twisting the collar ⅛-turn clockwise. The connector should then separate easily from the socket.
4. Loosen the two captive screws attached to the high-voltage module until the screws pop out.
5. Pull the high-voltage module straight out until it clears the spacers used to secure the main PCB.
6. Install the high-voltage module with the desired polarity in the same orientation as the high-voltage module that was removed, making sure the six connector pins on the main PCB are aligned with the six sockets on the high-voltage PCB. Tighten the two captive screws finger tight.
7. Reconnect the high-voltage cable by pushing the connector into the socket. There is a ⅛-turn spring locking connect that secures the connector when it is seated properly.

8. Replace the cover on the instrument and tighten the four screws that secure the cover.

Cleaning the Pressure Regulator

The air supplied to the pressure regulator should always be from a clean, dry air supply, eliminating the need to clean the pressure regulator. However, if you suspect the regulator has become dirty, refer to Appendix A for the manufacturer's specifications and instructions for maintaining the pressure regulator.

Cleaning the Rotameters

The air supplied to the air rotameter should always be from a clean, dry air supply, eliminating the need to clean the air rotameter. However, if you suspect the air rotameter has become dirty, refer to Appendix A for the manufacturer's specifications and instructions for maintaining the air rotameter.

The CO_2 supplied to the CO_2 rotameter should be supplied from a compressed gas cylinder containing dry CO_2 gas. After the CO_2 enters the Electrospray instrument from the back panel, it flows through a filter before entering the CO_2 rotameter; therefore, the CO_2 rotameter should not need to be cleaned under normal operating conditions. However, if you suspect the CO_2 rotameter has become dirty, refer to Appendix A for the manufacturer's specifications and instructions for maintaining the CO_2 rotameter.

Replacing the Filters

Because the level of contaminants entering the Electrospray in the air and CO_2 is low, the four internal filters in the Model 3480 do not need to be replaced under normal operating conditions.

Replacement Parts

This subsection contains information on replacement parts available from TSI and their part numbers. See also the section in Chapter 2 for information on parts included in the accessory kit, which are also available as replacement parts.

Table 5-2
Replacement Parts

Part Description	Part Number
Shield, High-Voltage Fitting	2904157
Wire, High-Voltage Platinum	3301754
Assembly, Viewing Window LED	1035928
Optical Window, Viewing Window LED	2502623
Lens, Viewing Window	2502622
Filter, Balston BQ In-Line	1602060
Tubing, Silicone ⅛" Barb	3001257
High-Voltage Module (please specify positive or negative)	1035907

CHAPTER 6
Troubleshooting

This chapter lists potential problems and their solutions.

Electrospray Operation is Unstable

If the Electrospray is not operating in the Cone-Jet mode (see Chapter 4, “Operating the Electrospray”) or the current display is not stable, first try decreasing the voltage setting to its minimum value and then increase the voltage setting until the Cone-Jet mode appears in the viewing window. If the Cone-Jet mode appears, the current display should be stable. If this procedure does not resolve your problem, use Table 6-1 to pinpoint possible solutions.

Table 6-1
Troubleshooting if the Electrospray Operation is Unstable

Problem	Solutions
Electrospray is operating in the Dripping mode (see Chapter 4, "Operating the Electrospray") *or* Current display is much lower and more unstable than normal	Increase the voltage setting Increase the capillary pressure drop Increase the air flowrate Purge the capillary (see Chapter 5) Clean the capillary tip (see Chapter 5) Adjust the position of the capillary (see Chapter 5, "Installing the Capillary") Clean the inlet fitting (see Chapter 5) Increase the conductivity of the sample (see Chapter 4)
Electrospray is operating in the Pulsating mode (see Chapter 4, "Operating the Electrospray") *or* Current display is lower and more unstable than normal	Increase the voltage setting Increase the capillary pressure drop Purge the capillary (see Chapter 5) Clean the capillary tip (see Chapter 5) Adjust the position of the capillary (see Chapter 5, "Installing the Capillary") Clean the inlet fitting (see Chapter 5) Increase the conductivity of the sample (see Chapter 4)
Electrospray is operating in the Corona Discharge mode (see Chapter 4, "Operating the Electrospray") *or* Current display is higher and more unstable than normal	Decrease the voltage setting Decrease the capillary pressure drop Increase the CO_2 flowrate Purge the capillary (see Chapter 5) Clean the capillary tip (see Chapter 5) Adjust the position of the capillary (see Chapter 5, "Installing the Capillary") Clean the inlet fitting (see Chapter 5) Decrease the conductivity of the sample (see Chapter 4)
No liquid is flowing through the capillary	Make sure the voltage, pressure, and flowrates are adjusted properly Make sure the capillary is immersed in the sample vial solution See the section below, "Unplugging the Capillary"
There is no current reading	Make sure the voltage, pressure, and flowrates are adjusted properly Make sure the capillary is immersed in the sample vial solution Make sure the platinum wire is immersed in the sample vial solution

Unplugging the Capillary

Each capillary is checked before leaving the factory to ensure proper operation. However, a capillary can become fully or partially plugged due to several reasons, including those listed in Table 6-2.

As a general rule, if the capillary becomes plugged and the capillary tip is not broken, first try applying back pressure to the capillary (see the procedure listed below), then try applying a 5 psi forward pressure drop across the capillary, and finally try cleaning the capillary tip or purging the capillary (see Chapter 5). If the capillary is still plugged, it should be discarded and replaced (see Chapter 5).

Table 6-2
Troubleshooting if the Capillary has Become Plugged

Reason	Solution
A contaminant or large particle in the sample vial has plugged the capillary inlet	Back pressure the capillary using the procedure listed below
Residue has built up on the inner walls of the capillary	Purge the capillary (see Chapter 5)
Residue has built up on the capillary tip	Clean the capillary tip (see Chapter 5)
The capillary has not been cleaned properly when shutting down the Electrospray	Purge the capillary (see Chapter 5)
The capillary tip has become broken during maintenance or handling procedures	Replace the capillary (see Chapter 5, "Removing the capillary" and "Installing the capillary")

Applying Back Pressure to the Capillary

If the capillary becomes plugged, the first attempt to unplug the capillary should be to apply back pressure to the capillary. The following procedure should unplug the capillary if a contaminant or large particle in the sample vial has plugged the capillary inlet:

1. Remove power from the instrument and turn off all flows.

2. Connect a compressed air source capable of generating 5 psi pressure or a large syringe (>50 cc) to the aerosol exit. Do **not** exceed 5 psi since a relief valve is attached to the Electrospray chamber. If the relief valve opens, remove all pressure from the

Electrospray chamber and the relief valve will reset automatically.

Caution
Applying more than 5 PSI pressure to the Electrospray chamber will cause a relief valve to open. If the relief valve opens, remove all pressure from the Electrospray chamber and the relief valve will reset automatically.

3. With a sample solution installed in the bottom half of the pressure chamber, apply up to 5 psi pressure to the aerosol exit for 15 seconds.
4. Remove pressure from the aerosol exit and run the Electrospray under normal operating conditions to check if the capillary is unplugged. If not, repeat steps 1–4 except apply back pressure for several minutes until bubbles can be seen exiting the capillary into the sample vial. If bubbles are not seen after 5 minutes at 5 psi back pressure, refer to the alternative solutions mentioned earlier in this chapter to unplug the capillary.

CHAPTER 7

Contacting Customer Service

This chapter gives directions for contacting people at TSI Incorporated for technical information and directions for returning the Model 3480 Electrospray for service.

Technical Contacts

- If you have any difficulty setting up or operating the Electrospray, or if you have technical or application questions about this system, contact an applications engineer at TSI Incorporated, 1-800-874-2811 (USA) or (651) 490-2811.
- If the Electrospray does not operate properly, or if you are returning the instrument for service, contact TSI Particle Instruments at 1-800-874-2811 (USA) or (651) 490-2811.

Returning the Electrospray for Service

Call TSI Particle Instruments at 1-800-874-2811 (USA) or (651) 490-2811 for specific return instructions. Customer Service will need this information when you call:

- The instrument model number
- The instrument serial number
- A purchase order number (unless under warranty)
- A billing address
- A shipping address

WARNING
The Model P-2042 Ionizer must be removed from the Electrospray prior to shipping the instrument. See the section titled "Replacing the Ionizer" in Chapter 5 for instructions on how to remove the ionizer. Do **not** ship the ionizer to TSI if the instrument must be returned, rather keep the ionizer and ship the instrument without the ionizer.

***Note**: The ionizer must be replaced 12 months from the date on the ionizer label. Contact TSI to order a new ionizer four weeks before the ionizer expiration date. When returning an ionizer, contact TSI for handling and shipping instructions.*

APPENDIX A
Model 3480 Specifications

The following specifications—which are subject to change—list the most important features of the Model 3480 Electrospray Aerosol Generator.

Table A-1
Specifications of the Model 3480 Electrospray Aerosol Generator

Mode of operation	Generation of aerosols from liquid solutions or suspensions using an electrospray method.
Particle type	Solids or nonvolatile liquids soluble in 20 mM ammonium acetate solution in ultrapure water or 0.05% trifluoroacetic acid in ultrapure water. Suspensions in some solvents up to a particle size of 100 nm.
Initial droplet diameter	150 nm nominal
Particle generation rate	>10^7 particles/cm^3
Particle size range	2 to 100 nm
Liquid conductivity	0.2 S/m nominal
Liquid flowrate	66 nL/min nominal
Differential pressure	0 to 5 psi (3.7 psi nominal)
Flowrates	
Air	0.2 to 2.5 L/min (1 L/min nominal)
CO_2	0.05 to 0.5 L/min (0.1 L/min nominal)
Aerosol	0.25 to 3 L/min, determined by the sum of air and CO_2 flowrates
Ionizer*	Bipolar, Po-210, 5 millicurie, half-life of 138 days, shipped separately
Front panel displays	LED, 3.5 digit voltage and current displays
Ports	
Air inlet	¼ in. OD Swagelok connection
CO_2 inlet	¼ in. OD Swagelok connection
Aerosol outlet	¼ in. OD Stainless Steel tube
Voltage range	+0.5 to +3.5 kV (2.0 to 2.5 kV nominal, negative high-voltage module available)
Current range	0 to 2000 nA (280 to 320 nA nominal)
Dimensions (LWH)	20.3 cm × 40.4 cm × 25.7 cm (8.0 in. × 15.9 in. × 10.1 in.)
Weight	6.8 kg (15 lb)
Power requirements	85 to 260 VAC, 50/60 Hz, 25 W maximum
Fuse (not replaceable by user)	2.5A, 250 V, type 5x20 mm (not replaceable by operator)
Environmental Conditions	Indoor use Altitude up to 2000 m (6500 ft) Ambient temperature 10-50 °C Ambient humidity 0-90% RH non-condensing Over-voltage category II Pollution degree II

*TSI Incorporated is authorized by the United States Nuclear Regulatory Commission to distribute these Ionizers. If your location is within the United States, no other federal license is required. Check local regulations for your own protection. Ionizers are shipped separately. End-user name and address is required. Contact TSI to return used ionizers or obtain replacements.

Pressure Regulator Maintenance

The air supplied to the pressure regulator should always be from a clean, dry air supply, eliminating the need to clean the pressure regulator. However, if you suspect the regulator has become dirty, use Figure A-1 and the instructions provided by the manufacturer below to disassemble and clean the regulator.

Caution
Remove air supply pressure and bleed off output pressure prior to performing maintenance.

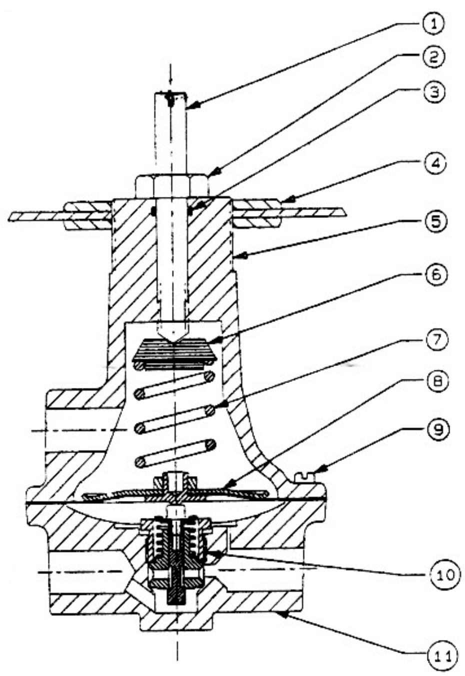


Figure A-1
Pressure Regulator Assembly

To disassemble the pressure regulator, loosen adjusting handwheel (1) until spring tension is relieved. Remove six screws (9) and lift off bonnet (5), spring plate (6), spring (7) and diaphragm assembly (8). To remove nozzle assembly (10), use $\frac{5}{8}$” socket wrench to remove nozzle assembly to avoid damage to the nozzle. All parts may be cleaned by immersion in a suitable solvent and blowing dry with air stream.

To reassemble, replace nozzle assembly (10) and place diaphragm assembly (8) over body (11), with diaphragm plate up. Place spring (7) and spring plate (6) on diaphragm assembly (8), re-install bonnet (5) and tighten six screws (9). The six screws should be tightened alternately.

Rotameter Maintenance

If you suspect either rotameter has become dirty, refer to Figure A-2 and the instructions provided by the manufacturer below to clean either rotameter.

Caution
Remove air supply pressure and bleed off output pressure prior to performing maintenance.

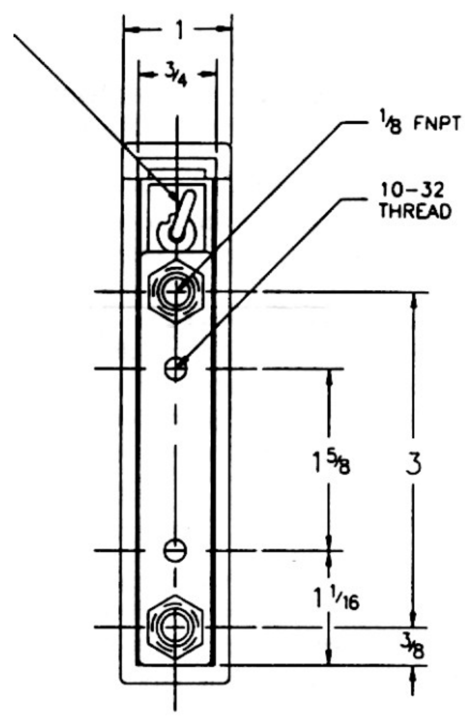


Figure A-2
Rotameter Back View

Occasional cleaning may become necessary if dirt appears in the flowmeter or if float movement is restricted. To clean, rotate "Retaining Clip" (Located in the back of flowmeter) counter-clockwise to align clip with slot and then pull out. Then remove top plug, ring (if required), outlet fitting and float only. Tube does not need to be removed. Wash tapered hole, float, outlet fitting and top plug with clean water and a soft brush. Rinse all parts with clean water and dry thoroughly. Avoid use of solvents and strong bases for cleaning. Reassemble by reversing above.

APPENDIX B
Theory of Operation

This appendix describes theories of operation for the Electrospray Aerosol Generator.

System Description

The Electrospray consists of three main subsystems that are used to generate a monodisperse aerosol.

- Pressure chamber—capillary—Electrospray chamber
- Air and CO_2 flow control
- High-voltage control

The pressure chamber—capillary—Electrospray chamber schematic is shown in Figure B-1. A description of how the Electrospray generates aerosol follows.

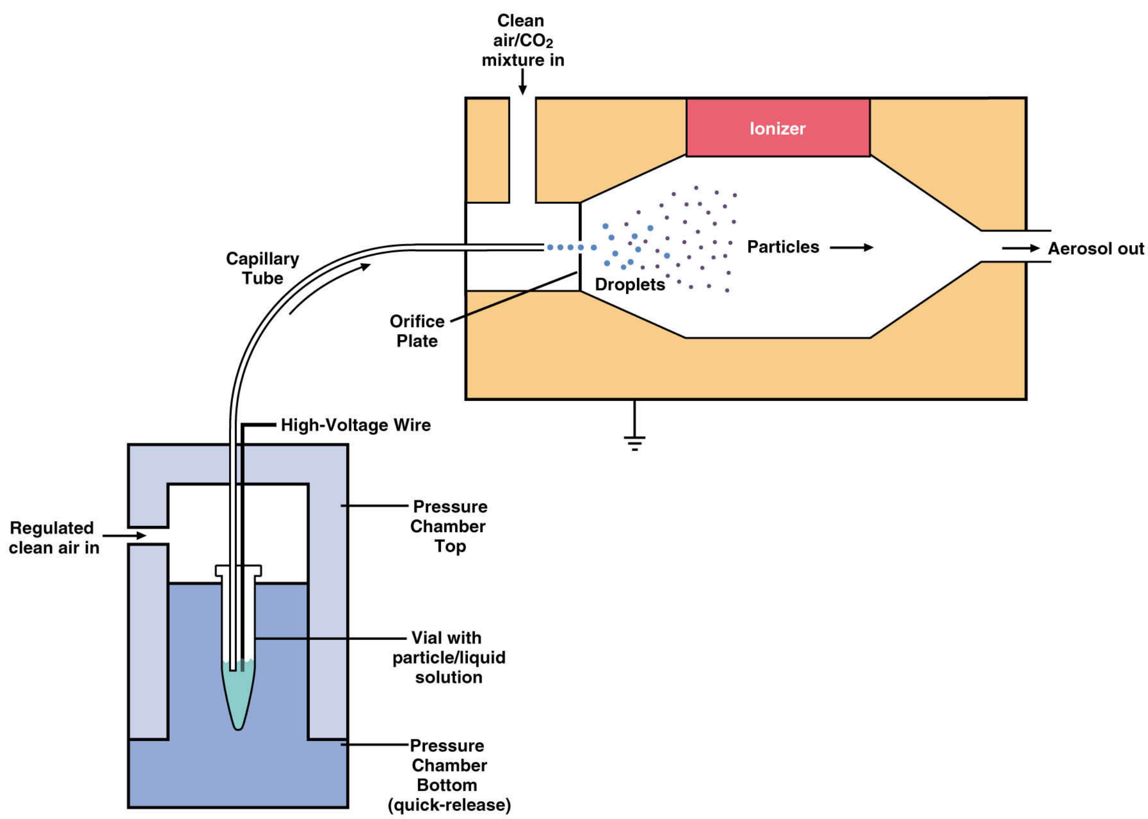


Figure B-1
Schematic Diagram of the Electrospray

A sample solution is stored in a cone-shaped vial, enclosed in a cylindrical pressure chamber. The chamber accommodates a capillary and a platinum high-voltage wire, both of which are immersed in the solution. Maintaining a differential pressure causes the solution to be pushed through the capillary.

The high-voltage control regulates an electrical field exerted at the capillary exit, which pulls the charged solution out of the capillary, forming droplets that are mixed with clean air and CO_2. This produces a sheath flow, which transports the droplets to the Electrospray chamber. The highly charged droplets are neutralized by a Polonium-210 source, and the liquid is evaporated before the aerosol exits the instrument.

The remainder of this section discusses liquid flow in the capillary, the Electrospray chamber design, aerosol neutralization, the flow control system, and the high-voltage control and current measurement methods used in the Electrospray.

Capillary Flow Characteristics

The liquid flowrate Q (nL/min) through the capillary is governed by the following equation for Poiseuille flow in a circular tube:

$$Q = c \cdot \pi \cdot \left(\frac{D_C}{2}\right)^4 \cdot \left(\frac{1}{8 \cdot \mu}\right) \cdot \left(\frac{\Delta P}{L}\right) \qquad \text{Equation B-1}$$

where:

D_c is the capillary inner diameter (µm)
µ is the liquid viscosity (poise)
ΔP is the pressure across the capillary (psi)
L is the length of the capillary (cm)
c is a constant to account for all unit conversions (4.14E4)

The capillary in the Electrospray has a diameter of 25 ±2 µm and a length of 25 cm, a typical pressure across the capillary is 3.7 psi (¼ atm), and the viscosity of water at 20°C is 0.89E-2 poise. Given these conditions, the nominal liquid flowrate is 66 nL/min. Table B-1 lists values of the liquid flowrate as a function of pressure and capillary diameter. Also included in the table is the volume of the capillary, residence time of the liquid in the capillary, and the length of time for 1 mL to flow through the capillary.

Table B-1
Capillary Flow Characteristics for Water at 20°C

Capillary Pressure Drop ΔP (psi)	Capillary Inner Diameter D_c (µm)	Liquid Flowrate Q (nL/min)	Capillary Volume V (nL)	Liquid Residence Time t_r (sec)	Sample Time t_s (days/mL)
2.00	25	35.7	122.7	206	19.5
2.20	25	39.2	122.7	188	17.7
2.40	25	42.8	122.7	172	16.2
2.60	25	46.4	122.7	159	15.0
2.80	25	49.9	122.7	147	13.9
3.00	25	53.5	122.7	138	13.0
3.20	25	57.1	122.7	129	12.2
3.40	25	60.7	122.7	121	11.4
3.60	25	64.2	122.7	115	10.8
3.70	**25**	**66.0**	**122.7**	**112**	**10.5**
3.80	25	67.8	122.7	109	10.2
4.00	25	71.4	122.7	103	9.7
4.20	25	74.9	122.7	98	9.3
4.40	25	78.5	122.7	94	8.8
4.60	25	82.1	122.7	90	8.5
4.80	25	85.6	122.7	86	8.1
5.00	25	89.2	122.7	83	7.8
2.00	23	25.6	103.9	244	27.2
2.00	25	35.7	122.7	206	19.5
2.00	27	48.5	143.1	177	14.3
3.00	23	38.3	103.9	163	18.1
3.00	25	53.5	122.7	138	13.0
3.00	27	72.8	143.1	118	9.5
3.70	23	47.3	103.9	132	14.7
3.70	25	66.0	122.7	112	10.5
3.70	27	89.8	143.1	96	7.7
4.00	23	51.1	103.9	122	13.6
4.00	25	71.4	122.7	103	9.7
4.00	27	97.1	143.1	88	7.2
5.00	23	63.9	103.9	98	10.9
5.00	25	89.9	122.7	83	7.8
5.00	27	121.3	143.1	71	5.7

Calculating Primary Droplet Diameter

As the liquid reaches the end of the capillary, the electric field induces a surface charge on the liquid. The electrostatic charge of the liquid produces forces that cause the liquid to disperse into a fine spray of charged droplets. The diameter of the charged droplet as it first leaves the tip of the capillary is referred to as the primary droplet diameter. After the primary droplet is generated, the liquid from the primary droplet evaporates in the ionization chamber and an aerosol particle remains. If a sucrose solution is used to generate an aerosol, the sucrose concentration can be varied to generate different particle diameters. For any sucrose particle diameter D_p, the primary droplet diameter D_d can be calculated if the sucrose concentration C (expressed as a decimal) is known by using the following expression:

$$D_d = \frac{1}{C^{1/3}} \cdot D_p \qquad \text{Equation B-2}$$

Empirical formulas expressing the particle diameter (from which the primary droplet diameter can be calculated) of a 0.1% V/V sucrose solution as a function of $(Q/K)^{1/3}$, where Q is the liquid flowrate and K is the electrical conductivity of the liquid, have been reported in publications such as:

Chen, Da-Ren, David Y.H. Pui, and Stanley L. Kaufman [1995] "Electrospraying of Conducting Liquids for Monodisperse Aerosol Generation in the 4 nm to 1.8 µm Diameter Range." *J. Aerosol Sci.*, 26:963-977.

Electrospray Chamber

Immediately after the highly charged primary droplets are generated at the capillary tip, they are transported by an air/ CO_2 sheath flow mixture through an orifice plate to the ionization chamber. This must be done quickly because after the primary droplet is generated, the liquid begins to evaporate, which decreases the surface area of the droplet, thereby increasing the surface charge density. If the surface charge density becomes too large, the Coulombic repulsive forces on the droplet will cause Rayleigh disintegration, resulting in a less monodisperse aerosol.

To reduce the charge on each primary droplet, the Electrospray chamber houses a Polonium-210 radioactive alpha-emitter ionizer. The nature of the flow through the orifice plate into the ionization chamber and the placement of the ionizer cause the droplets to encounter the ions nearly immediately upon their formation.

Equilibrium Charging Theory

The equilibrium charge distribution generated by the ionizer of the Electrospray can be represented by a theoretical model developed by Wiedensohler [1986], which is an approximation of the Fuchs [1963] diffusion theory for particle sizes in the submicrometer regime.

Figure B-2 shows the measured data of Wiedensohler [1986] and theoretical curves based on the theory of Fuchs [1963] and calculated by Wiedensohler [1988]. The theoretically determined charge distribution agrees well with experimental data. It can be seen from the figure that the fraction of positively charged particles is different from the fraction of negatively charged particles. Table B-2 lists the fractions of particles in air that carry +1, +2, +3, and +4 charge units.

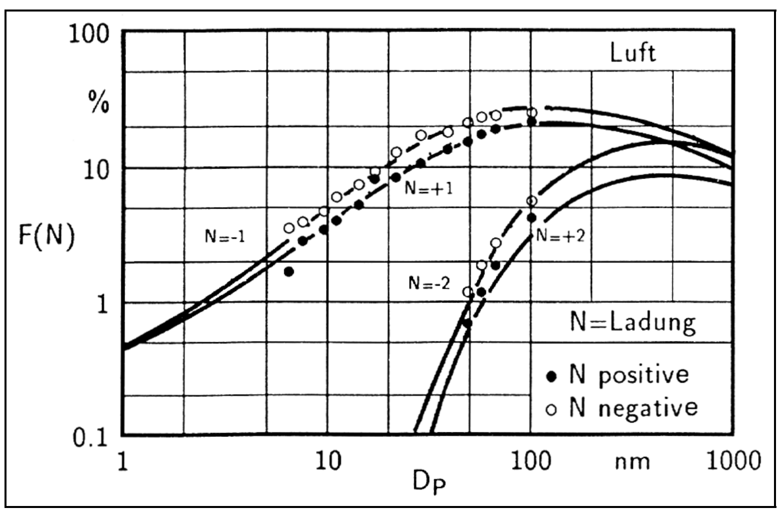


Figure B-2
Bipolar Particle Charge Distribution in Air [Wiedensohler and Fissan, 1988]

Table B-2
Midpoint Particle Diameters and Fraction of Total Particle Concentration that Carries +1, +2, +3, and +4 Elementary Charges

Particle Diameter Midpoint, nm	**Fraction of Total Particle Concentration That Carries This Number (1-4) of Positive Charges** +1	+2	+3	+4
10	0.0411	0	0	0
11	0.0460	0	0	0
12	0.0514	0	0	0
14	0.0573	0	0	0
15	0.0638	0	0	0
17	0.0709	0	0	0
18	0.0784	0	0	0
21	0.0866	0.00012	0	0
23	0.0953	0.00023	0	0
25	0.1041	0.00041	0	0
28	0.1145	0.0007	0	0
31	0.1241	0.0011	0	0
35	0.1343	0.0018	0	0
38	0.1446	0.0027	0	0
43	0.1550	0.0040	0	0
48	0.1650	0.0056	0	0
53	0.1748	0.0078	0	0
59	0.1841	0.0105	0	0
66	0.1925	0.0138	0	0
74	0.2000	0.0180	0.00045	0
83	0.2064	0.0225	0.00089	0
93	0.2113	0.0279	0.0016	0
104	0.2148	0.0339	0.0028	0
117	0.2167	0.0406	0.0046	0.00014
132	0.2167	0.0477	0.0072	0.00031
150	0.2149	0.0552	0.0105	0.00064
170	0.2113	0.0627	0.0149	0.0012

The formulas used to calculate Table B-2 are shown below. They are taken from Wiedensohler [1988]. To calculate the fraction of particles carrying zero, one or two charges, use Equation B-4 which is an approximation of the Fuchs model. Equation B-4 is valid for size ranges: 1 nm ≤ D_p ≤1000 nm for N = -1, 0, 1; for 20 nm ≤ D_p ≤ 1000 nm for N = -2, 2; and for $D_p \leq 20$ nm for $N \leq 1$.

$$f(N) = 10^{\left[\sum_{i=0}^{5} a_i(N)\left(\log\frac{D_p}{nm}\right)^i\right]} \qquad \text{Equation B-4}$$

Table B-3
Coefficients for Equation B-4

$a_i(N)$	N=-2	N=-1	N=0	N=1	N=2
a_0	-26.3328	-2.3197	-0.0003	-2.3484	-44.4756
a_1	35.9044	0.6175	-0.1014	0.6044	79.3772
a_2	-21.4608	0.6201	0.3073	0.4800	-62.8900
a_3	7.0867	-0.1105	0.3372	0.0013	26.4492
a_4	-1.3088	-0.1260	0.1023	-0.1553	-5.7480
a_5	0.1051	0.0297	-0.0105	0.0320	0.5049

For the fraction of particles carrying three or more charges, use Equation B-5 which is based on an derivation by Gunn from 1956.

$$f(N) = \frac{e}{\sqrt{4\pi^2 \varepsilon_0 D_p kT}} \exp \frac{-\left[N - \frac{2\pi\varepsilon_0 D_p kT}{e^2} \ln\left(\frac{Z_{i+}}{Z_{i-}}\right)\right]^2}{2\frac{2\pi\varepsilon_0 D_p kT}{e^2}} \qquad \text{Equation B-5}$$

where:

- e = elementary charge
  = 1.60217733E-19 coulomb
- ε_0 = dielectric constant
  = 8.854187817E-12 farad/m (for air)
- D_p = particle diameter [m]
- k = Boltzmann's constant
  = 1.380658E-23 joule/K (for air)
- T = Temperature [K]
- N = number of elementary charge units
- Z_{i+}/Z_{i-} = ion mobility ratio
  = 0.875 (Wiedensohler)

Air and CO_2 Flow Control

This section describes the flow of air and CO_2 through the Electrospray. A flow schematic is shown in Figure B-3.

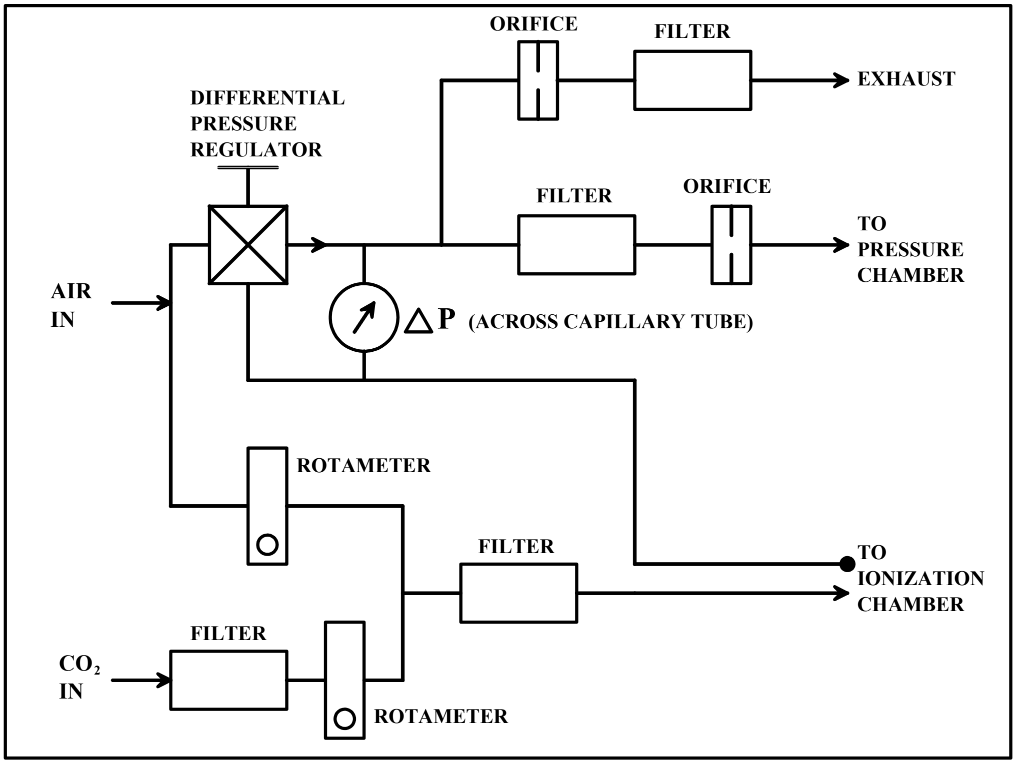


Figure B-3
Electrospray Flow Schematic

The differential pressure regulator is used to maintain the pressure drop across the Electrospray capillary; therefore, if the flowrates of the air and CO_2 into the Electrospray chamber are changed, the capillary pressure drop will not change. The high pressure tap for the pressure gauge is connected to the output of the pressure regulator, which is at the same pressure as the pressure chamber. The low pressure tap is connected in the same orientation as the air/CO_2 mixture inlet to the ionization chamber, which is immediately upstream of the capillary tip.

The orifice leading to the pressure chamber is used to allow the quick-release pressure chamber bottom to be removed without changing the position of the differential pressure regulator knob.

The exhaust orifice is used to provide “bleed” and “relief” features to the differential pressure regulator. This increases the regulator stability and responsiveness by keeping the regulator in a dynamic state. It also provides a quicker exhaust when the regulator output pressure exceeds the set point pressure.

The air and CO_2 rotameters provide separate controls for each flow before the flows are mixed together immediately upon exit of the rotameter. The filter provides a final clean of the sheath flow as well as a means to mix the air and CO_2 thoroughly before the entrance to the ionization chamber.

Voltage and Current Measurement

The voltage and current of the Electrospray circuit are displayed on the digital front-panel meters. This section describes how the voltage and current displays are measured.

Voltage Measurement

The voltage at the output of the high-voltage supply is divided down through series resistors. Two 10 MΩ resistors are used to reduce the maximum current of the high-voltage supply. If a person comes into contact with the high-voltage fitting of the Electrospray the maximum current is 0.25 mA. A further divider of two 50 MΩ and one 100 kΩ resistor divides the voltage of the Electrospray down by a factor of 1000. The sense voltage is picked up at the 100 kΩ resistor and buffered through an OP-27 follower. Two IN-457, low leakage diodes are used as clamping diodes across the op-amp inputs. In order to reduce the voltage for the ±2 V voltmeter display, the voltage is further divided down by a factor of 10. This also provides the opportunity to balance any divider tolerance on the high-voltage divider. A capacitor of 0.1 µF on the input of a second op-amp follower with the resistors provides a filter for any noise.

Current Measurement

To measure the current of the Electrospray, an op-amp operation as a current-to-voltage converter is used. The current path measured goes from the positive high-voltage supply side into the current-to-voltage converter, and then into the analog ground. From the analog ground, the current path continues through the grounded electrospray side, through the Electrospray (in the form of charged droplets), through the liquid in the capillary, through a 20 MΩ protection resistor, and into the negative high-voltage supply side.

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