

**Elemental Scientific Inc.**



**apex**

**apex Quick Installation Guide**

# apex Quick Installation Guide

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# 1

## Introduction

The Apex is a fully-integrated inlet system for ICP that connects directly to the torch injector and incorporates ESI's industry standard MicroFlow PFA nebulizer technology. This allows the Apex system to be configured for self-aspiration at rates from 20  $\mu\text{L}/\text{min}$  to 400  $\mu\text{L}/\text{min}$ , or the sample can be pumped at flow rates up to 0.7 mL/min for ICP-AES applications. The standard Apex configuration is a MicroFlow ST nebulizer operating at 300  $\mu\text{L}/\text{min}$ . The uniform aerosol produced by the MicroFlow ST nebulizer is conditioned by the Apex to deliver analytes to the plasma in optimum usable form by minimizing interferences and stabilizing the signal. The Apex's patent pending geometry and reduced memory effects produce faster rinse-out times and also allows the inlet system to be cleaned quickly and easily between applications.

The Apex system allows analysis of corrosive samples, organic solvents and high-TDS samples while delivering a substantial increase in raw sensitivity. With its O-ring-free PFA Teflon or quartz sample path, the Apex generates lower BECs, resulting in low signal-to-noise ratio with nearly 100% sample transport efficiency.

# 2

## Preparing for Installation

### Power Requirements

The Apex is powered by a desktop power supply delivering 12VDC @ 10A. This power supply is an international unit which can be plugged into any mains supplying from 100 to 240VAC  $\pm 10\%$ , with any line frequency from 47 to 63Hz @ 3.2A.

### Power Cord Set Requirements

The power cord supplied with the Apex is 2m long and meets the requirements of the country where you purchased the system. Always use approved power cords to prevent possible injury or electric shock.

### Space Requirements

The Apex can be connected directly to the injector of the ICP or by means of a length of transfer tubing. Whichever method is used there must be a 5" area surrounding the Apex footprint that is free of any obstacles. This is needed to facilitate adequate air movement around the Apex. The total area needed is 10"W x 20"D (25cmW x 51cmD).

### Gas Requirements

A nitrogen gas supply of at least 45 to 55 psi (~3.5 to 4 bar) @ 10 ml/min is needed.

### ICP Requirements

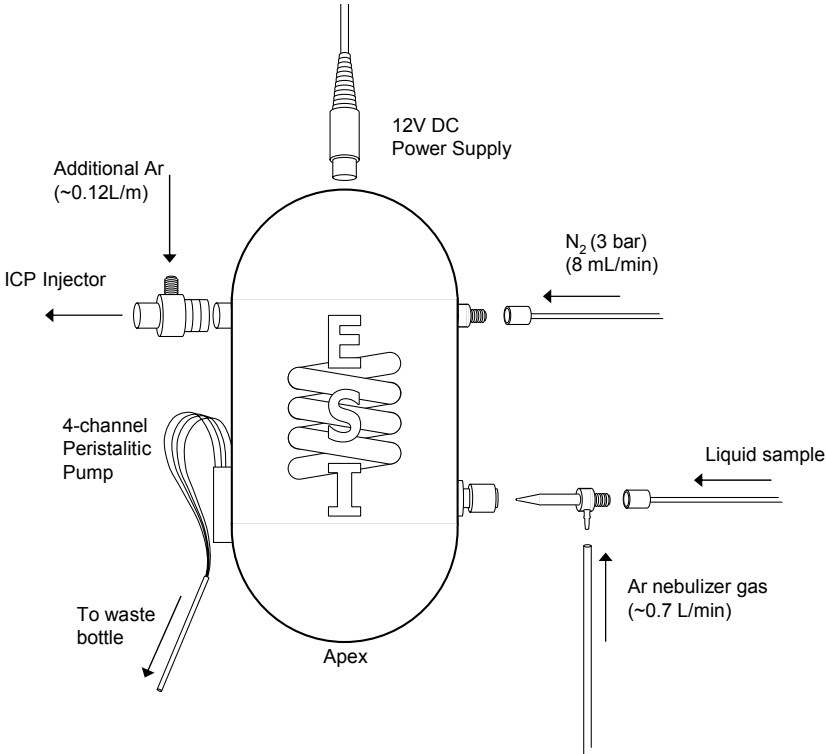
Ensure the operating performance of the ICP has been observed prior to using the Apex for the first time.

# 3

## Installing the Apex

### Port Connection Diagram

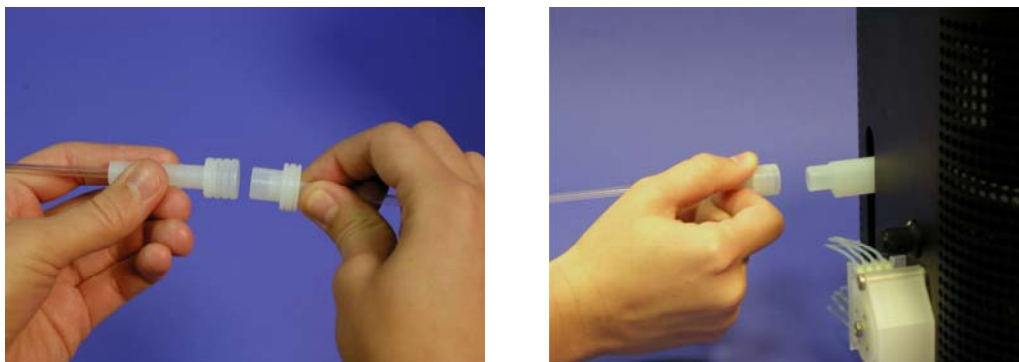
The Apex is very simple to install and can be completed in just a few minutes. Figure 1 shows a quick reference connection diagram.



**Figure 1. Apex Port Connection Diagram**

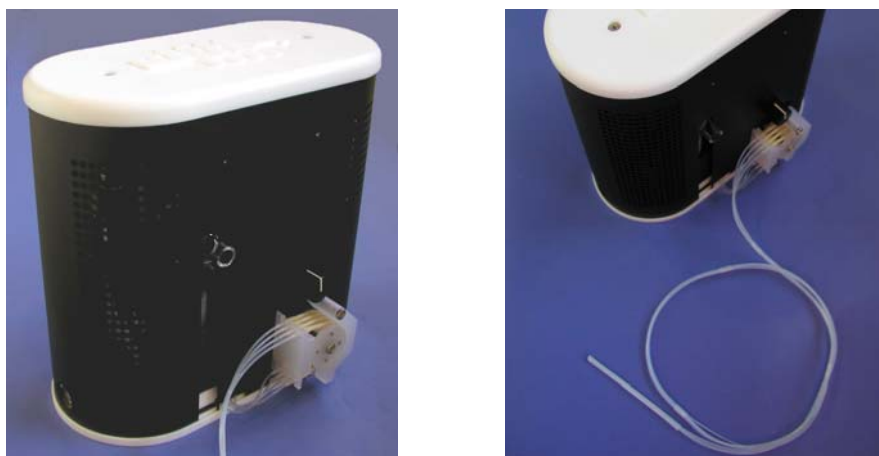
## Interfacing to the ICP

The Apex can either be connected directly to the T-connector on the torch injector or via a length of transfer tubing as shown below in Figure 2. Attach an auxiliary nebulizer gas line from the ICP instrument to the T-connector.



**Figure 2. Connection to Injector and Apex**

## Connecting the Drainage System



**Figure 3. Waste Pump and Waste Lines**

The Apex system has four drain lines located on the back of the unit that connect to the pump. These lines are pressed into the barbed fittings on the peristaltic pump and can easily be removed, if needed, by pulling. During operation observe the pump fittings to make sure there are no leaks. The tubing needs to be pushed through the barbed fittings and into the pump tubing to make a reliable connection. Place the other ends of the waste lines into an approved waste container.

## Controlling the Pump Speed

The speed of the peristaltic pump can be controlled by turning the knob located just above the pump on the back of the unit. Use a slower speed during normal operation (knob should be near the 12 o'clock position). Then use a higher speed to quickly drain the system after a flushing rinse.

## Pressure Shoe Adjustment

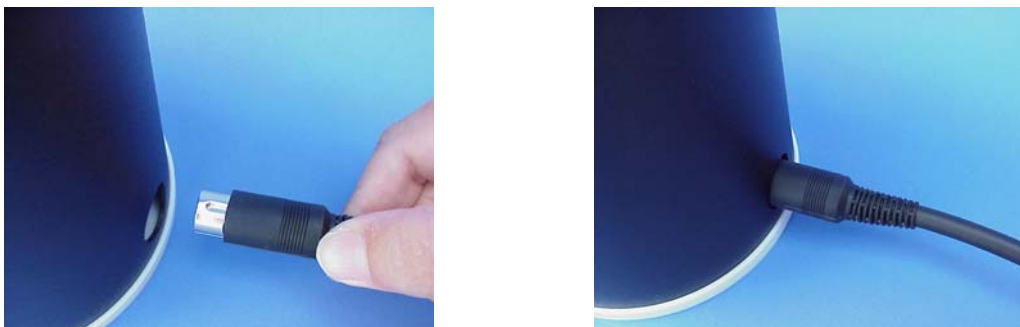
The pressure shoe on the pump squeezes the pump tubing together which allows the peristaltic pumping action to occur. Too little pressure on the tubing will lead to poor analytical results, while too much pressure will result in undue strain on the pump tubing causing premature wear and the need to replace pump tubing more often.

Adjust the amount of pressure on the shoe by turning the adjustment screw/locking mechanism located on the lower side of the pump. Proper adjustment is achieved by tightening the screw one more turn after all channels have started to pump.

## Power Supply Connections

### Apex Power Connector

Plug the 12VDC power supply into the Apex using the 8-pin round DIN connector from the power supply. There is a locating indentation on the 8-pin connector that should be oriented upwards when trying to press the plug into the socket on the Apex E. Plug the AC power cord into the power supply and the other end into the mains power supply.



**Figure 4. Connecting 12 VDC Power to the Apex**

## Connecting the Nitrogen Gas Line

Locate the nitrogen gas line, shown in **Figure 5**.



**Figure 5. Nitrogen Gas Line**

Attach the gas line **28mm** PFA ferrule nut to the Swagelok fitting on N<sub>2</sub> regulator as shown in **Figure 6a**.

**NOTE:** remove threaded cap from the Apex N<sub>2</sub> port (if present).

Attach the **6mm** gas line fitting to the Apex N<sub>2</sub> port as shown in **Figure 6b**.

For proper operation, the incoming N<sub>2</sub> pressure should be at least 45-55 pounds/square inch (~3.5 to 4 bar).



**Figure 6a**



**Figure 6b**

## Installing the Nebulizer

Insert the nebulizer into the inlet port nut and securely tighten. **BEWARE: Over tightening can damage the nebulizer.**

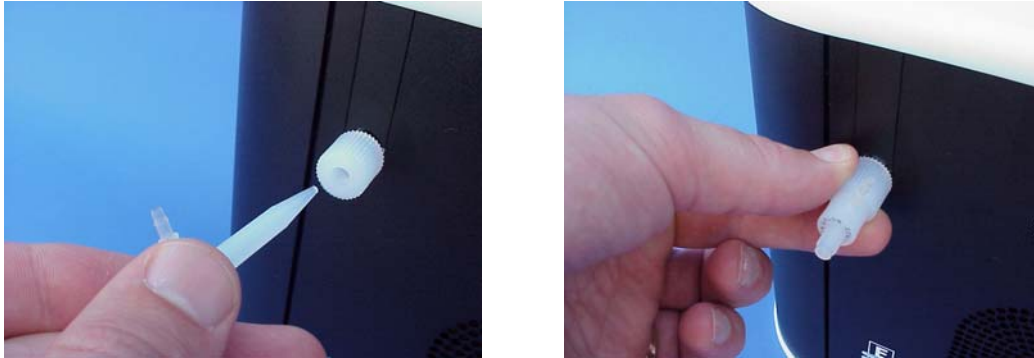


Figure 7. Attaching Nebulizer to Apex

Attach one end of the gas line to nebulizer fitting as shown in Figure 8. Attach the other end of the nebulizer gas line to the instrument supply. Attach the ST uptake capillary to the nebulizer by firmly tightening the capillary nut onto the threaded end of the nebulizer to ensure a leak-free connection.

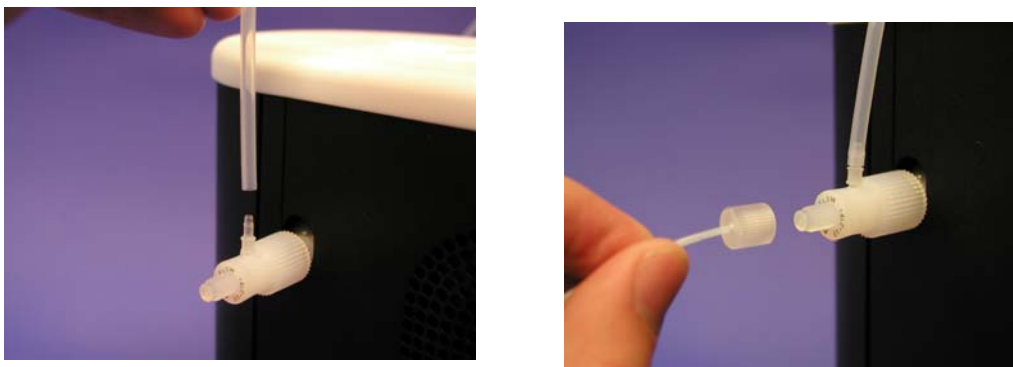


Figure 8. Attaching Nebulizer Gas Line and Capillary to ST Nebulizer

# Using the Apex

## About the Switches and Lights

There are two rocker switches located on the front panel which control the heating and cooling functions of the Apex. Each switch has three positions with the middle position “O”, being “Off” and the other two positions controlling temperature set points. There are also two LED’s positioned one on top of the other on each switch. The top LED illuminates when power is applied to the heater or cooler. The bottom LED illuminates when the temperature has reached the selected set point.



**Figure 9. Front Control Panel of Apex**

Position “1” on the red heater switch sets the heater temperature to 100°C.

Position “2” sets the heater temperature to 140°C.

Position “1” on the blue chiller switch sets the chiller temperature to –3°C.

Position “2” controls the chiller temperature to +2°C.

## Temperature Settings and Sample Types

The heater should be set at 100°C for the lower flow nebulizer and 140°C for the higher flow ST nebulizer. The chiller should be set at 2°C for aqueous samples and -5°C for organic samples.

## Nitrogen Gas Valve and Regulator

A restriction-based flow controller is included with the Apex to allow a second gas flow to be mixed with the sample aerosol stream in the last loop of the Peltier-cooled condenser. Typically, nitrogen is added through this port to increase signal intensity, improve stability, and reduce the formation of oxide interferences. Small changes in N<sub>2</sub> flow can dramatically affect the signal intensity. A length of 0.15mm i.d. fluoropolymer tubing combined with a combination flow adjustment and On/Off valve allows a controlled and stable flow of N<sub>2</sub> to enter the sample aerosol stream. The exact amount of N<sub>2</sub> will vary depending upon the experimental conditions (e.g. ICP instrument, injector diameter, analytes measured) but the optimum amount typically will be in the range 10 mL/min ±5 mL/min (inlet N<sub>2</sub> pressure required is approximately 3.5 Bar (50 PSI)). The flow controller may also be used to add non-corrosive gases other than N<sub>2</sub> to the aerosol stream.

The valve consists of an outer black knob for On/Off control and the screw that is located in the center of the knob is used to make fine adjustments of the flow rate. Turning the black knob completely clockwise shuts the flow of gas off, which is necessary during plasma ignition. To turn the flow on, the knob must be turned completely counter-clockwise. Rough adjustment of the N<sub>2</sub> flow can be accomplished by changing the inlet pressure to the N<sub>2</sub> valve.



Figure 10. Nitrogen On/Off Control and Fine Adjustment

The N<sub>2</sub> flow rate can be adjusted once for an experimental condition, then the On/Off control valve can be used for day to day operation. Turning the screw clockwise reduces the flow and turning the screw counter-clockwise increases the flow. Adjust the screw until the desired results are achieved.

In normal use, a relatively small amount of N<sub>2</sub> will be diluted by the Ar nebulizer gas flow comprising the bulk of the sample aerosol stream. However, during ICP ignition the sample aerosol stream will be turned off by the ICP instrument. The N<sub>2</sub> valve or N<sub>2</sub> supply must be shut off during ICP ignition to prevent the Apex from filling with N<sub>2</sub> during ICP start-up. If not the ICP may extinguish when the ICP instrument turns the Ar nebulizer gas supply on and introduces the N<sub>2</sub> into the plasma causing it to blow out as the Ar nebulizer gas flow is turned up.

## Start-up Procedure

1. Switch on the power supply to the Apex.
2. Set the red heater temperature switch to “1” (100°C) for sample flow rate < 100µL/min or “2” (140°C) for sample flow rate > 100µL/min.
3. Set the blue chillier temperature switch to “1” (-3°C) for organic solvents or “2” (+2°C) for aqueous solutions.
4. Ensure nitrogen controller is closed by turning fully clockwise.
5. Start plasma ignition sequence.
6. When the plasma reaches analysis mode open the nitrogen controller fully anti-clockwise.
7. Wait approximately 20 minutes for LED's on the heater and chillier to light up.

## Optimization of System

1. Place sample probe into the ICP-MS instrument Tune solution and ensure liquid flow is observed flowing through the capillary tubing.
2. Recommended initial gas settings are:
  - (a) Nebulizer gas flow = 0.7 L/min
  - (b) Auxiliary gas flow = 0.2 L/min
  - (c) Nitrogen gas flow = 0 L/min

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**NOTE:** The gas flow through the central channel of the ICP is made up of (a.) + (b.) + (c.); adjusting one gas flow will change the total flow and so will require the inverse adjustment of the other gas flows to re-optimize the signal.

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3. Optimize for sensitivity by adjusting the Argon gas flows only ((a.) & (b.)).
  4. Using a small flathead screwdriver, adjust the inner screw on the N<sub>2</sub> flow controller half a turn. Adjust the nebulizer and auxiliary gas flow rates to optimize the signal, while noting the sensitivity, stability and oxide level.
  5. Repeat step 3 until a maximum in sensitivity and stability along with a minimum oxide level is observed.
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**NOTE:** The optimization of the nitrogen is very broad, setting the N<sub>2</sub> controller 1<sup>1</sup>/<sub>4</sub> turns will produce low oxide levels and allow optimization of the sensitivity via the sample gas flow.

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6. Fine tune torch position and lens settings appropriately.

## Shut Down Procedure

1. Change the sample type to deionized water and nebulize for three minutes to rinse the system.
2. Initiate the ICP-MS Shut down procedure.
3. Turn the main N<sub>2</sub> controller fully clockwise to stop the nitrogen flow.
4. Switch off the Apex power supply.

# 5

## Maintenance

### Cleaning the Apex Flow Path

Everything needed to clean the Apex is included with your unit in the Apex cleaning kit. The kit includes (1) Apex / ACM rinse bottle, (2) solvent rinse line, (3) outlet drain tube and (4) luer adapter nut.



Figure 11. Apex cleaning kit

***Important: The ACM or Spiro membrane module, if installed, must be removed prior to performing this cleaning step. Failure to do this will cause permanent damage to the ACM or Spiro.***

1. Switch off the Apex and disconnect the power cord from the unit
2. Remove the MicroFlow nebulizer and close the nitrogen valve.
3. Fill the 500mL rinse bottle (1) with the desired cleaning solution.

4. Attach the drain tube (2) to the outlet port on the back of the Apex and direct the other end of the drain tube to an appropriate drain or waste container.
5. Replace the nebulizer nut with the luer adapter nut and tighten securely.



**Figure 12. Preparing the Apex for cleaning the flow path**

6. Attach one end of the solvent rinse line (3) to the luer adapter nut and attach the other end of the solvent rinse line to the luer fitting on the rinse bottle.



**Figure 13. Attaching Rinsing Bottle to Apex**

7. Turn the Apex upside down, this ensures there will be no air gaps when the unit is filled with cleaning solution.
8. Hold the rinse bottle upside down and squeeze the cleaning solution into the Apex until liquid starts to exit through the outlet port. Let the cleaning solution stand in the flow path for a brief period of time.



**Figure 14. Rinsing the Apex**

9. Turn the Apex back to its correct position, remove the rinse inlet line & allow unit to drain out by gravity. Drain or dispose of the cleaning solution accordingly.
10. Flush the Apex with deionized water in the same way described in steps 3 –9.
11. Restore power to the Apex, turn on the heater and turn the peristaltic pump to maximum speed to remove the remaining water through the drain lines.

## Optional Acid Vapor Cleaning

From time to time it may be necessary to perform a more aggressive cleaning procedure to obtain low blank levels. This is described here.

1. Place the Apex upside down and attach the solvent drain tube to the outlet port of the Apex, direct the other end of the solvent drain tube to an appropriate drain or waste container. Attach the rinse line to the inlet.
2. Inject 10mL strong acid into the spray chamber inlet port.

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**CAUTION:** Ensure unit is placed in a fume hood to prevent acid vapors entering the laboratory atmosphere

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3. Cap (4) the inlet port to prevent leaking and turn the heater on (Setting 1) with the peristaltic pump at the lowest setting.



**Figure 15. Inlet port cap**

4. Allow the Apex to heat overnight.
5. Taking care, return the Apex to its correct position, remove the inlet cap, and allow the unit to drain out via the peristaltic drain pump.
6. Rinse the Apex with deionized water following steps 3 - 11 in Cleaning Apex Flow Path described earlier.

# APPENDIX 1

## Areo Accessory

The Areo micro-porous Nafion membrane dryer accessory for the Apex performs on-line removal of residual water vapor from the sample aerosol stream exiting the Apex E inlet system. The Areo may also be useful for removing certain hydrocarbon solvent vapors, such as methanol, from the sample aerosol stream. Solvent vapors pass into the Nafion membrane, are transported through the membrane wall, and are removed by a counter current sweep flow of a dry gas such as nitrogen or argon. The sweep gas does not pass through the membrane or enter the sample aerosol stream, it simply serves to receive solvent vapor molecules that pass through the membrane. A wide range of sweep gas flows may be used, but typically a flow of 2 to 3 L/min will be adequate.

## Attaching Areo Dryer to Apex

Attach the Areo membrane dryer unit to the Apex by pressing the inlet connector of the Areo onto the outlet tube of the Apex.



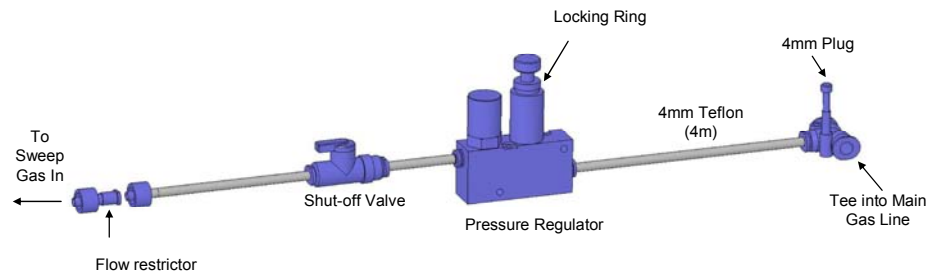
**Figure 16. Attaching Areo Dryer to Apex**

## Areo Completion Kit

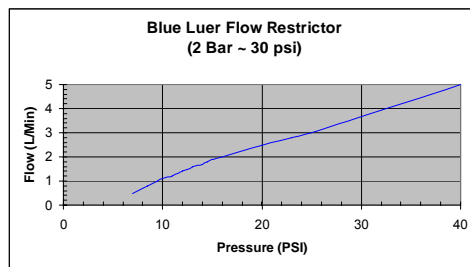
The Areo completion kit includes everything needed to tie into the existing gas line. Included is a pressure regulator, shut-off valve, tee, and custom flow restrictor that when connected as shown will deliver the proper flow of sweep gas. Adjust the pressure regulator to approximately 2 Bar to achieve a flow of about 3 L/min. See figure 14 for flow rates vs. pressure to adjust to other flow rates. Once set, you can tighten the locking ring to prevent changes in flow rates and simply use the shut-off valve to turn the flow off when not in use.

Connect the “Sweep Gas In” line from the Areo to the pressure regulator that is supplied with the unit. There are a couple of ways to connect the other end of the pressure regulator: one is to connect directly to a gas bottle; and the other is to tee into an existing gas line.

Next route the “Sweep Gas Out” from the Areo to an appropriate vent.



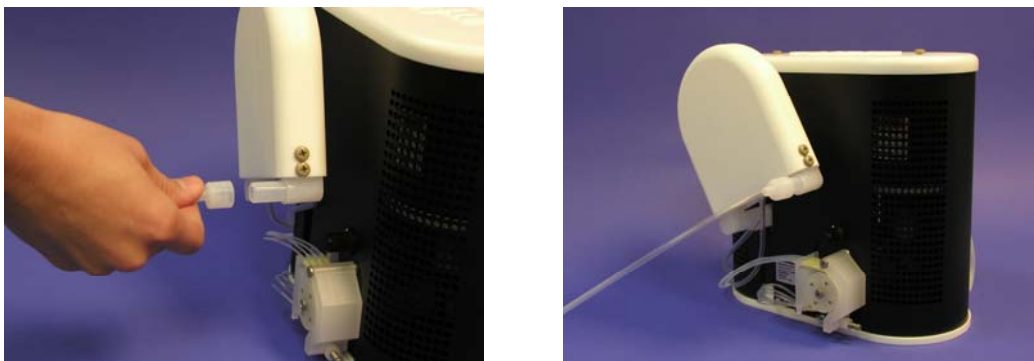
**Figure 17 Areo Completion Kit**



**Figure 18. Flow Rate vs. Pressure**

## Interfacing to the ICP

The Areo can be interfaced to the injector by a length of transfer tubing or directly connected to the ICP injector depending on the configuration of the ICP.



**Figure 19. Connecting the Areo**

## Cleaning the Areo Membrane

Should the Areo membrane require a more thorough cleaning, perform the steps listed below:

1. Disconnect power from the Apex unit.
2. Remove Areo from Apex.
3. Attach the Areo rinse connector to the rinse bottle.
4. Fill the 500mL rinse bottle with a cleaning solution, i.e. 10% HNO<sub>3</sub>.
5. Connect the rinse bottle to the Areo membrane unit by pressing the Areo rinse connector into the Areo membrane unit.
6. Place the exit of the Areo over a sink and transfer the cleaning solution into the Areo membrane by squeezing the bottle.
7. Drain the cleaning solution.
8. Attach the Areo membrane unit back onto the Apex and connect all tubing and drain lines.
9. Power on the Apex and turn on the sweep gas to allow the membrane to dry.
10. The unit will be ready for use in about 10 minutes.

## APPENDIX 2

### ACM Accessory

The Actively Cooled Membrane module (ACM) uses a Nafion membrane, which is Peltier cooled, to increase the ability to further remove water vapor from the sample aerosol stream exiting the Apex system. With Peltier cooling, the ACM performs better than the Areo, providing oxide ratios of .05% or less.

### Power Requirements

The ACM is powered by a desktop power supply delivering 12VDC, 6.6A. This power supply, in turn, needs to be plugged into the mains supply with a requirement of 100-240VAC +/- 10%, 47-63Hz, 1.9A.

### Power Cord Set Requirements

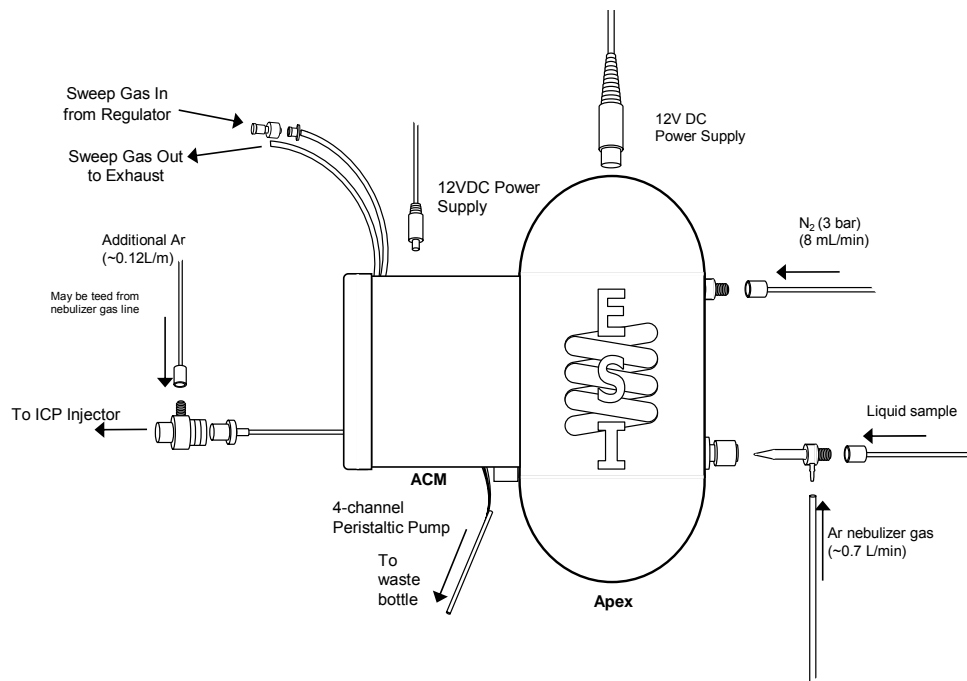
The power cord supplied with the ACM is 2m long and meets the requirements of the country where you purchased the system. Always use approved power cords to prevent possible injury or electric shock.

### Space Requirements

The addition of the ACM requires the system to be connected to the injector by a length of transfer tubing. The total area needed for the addition of the ACM is 15"Wx20"D (38cm x 51cmD).

## Connection Diagram

Figure 16 below shows a quick reference connection diagram.



**Figure 20. Apex with ACM Connection Diagram**

## Attaching ACM Module to the Apex

The ACM attaches to the Apex by mating the Teflon fitting on the back of the ACM to the chiller inside the Apex, aligning the mounting pins with the keyhole openings on the ACM, and pushing the ACM downward onto the Apex. The completed assembly is shown below in Figure 17.



**Figure 21. ACM Attached to the Apex**

## How to Install ACM

To attach the ACM to the Apex, first insert the right angle Teflon fitting, located on the back of the ACM, into the slot on the back of the Apex. At the same time align the two keyhole openings on the back of the ACM with the two mounting pins on the back of the Apex.

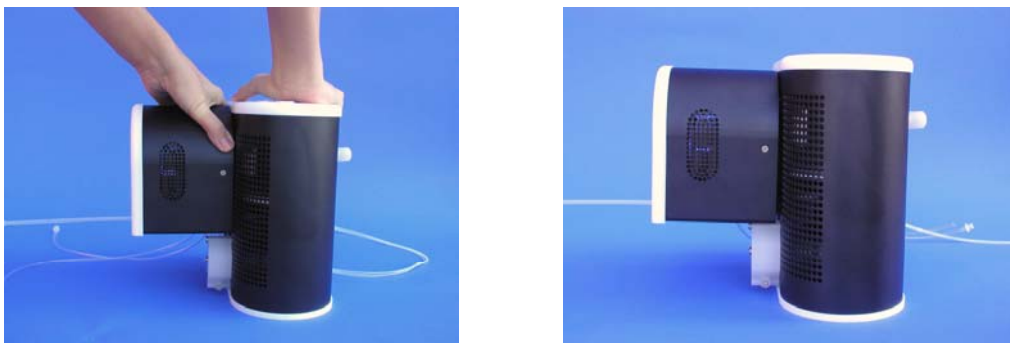


**Figure 22. Aligning ACM with Apex and Pushing Downward**



**Figure 23. Keyhole Slot on the Back of ACM and the Mating Connector on the Apex**

Hold the Apex steady with one hand and use the other to push the ACM downward approximately .4" (10mm)



**Figure 24. Pushing Downward to Attach ACM to Apex**

## ACM Completion Kit

The ACM completion kit includes everything needed to tie into the existing gas line. Included is a pressure regulator, shut-off valve, tee, and custom flow restrictor that when connected as shown below will deliver the proper flow of sweep gas. Adjust the pressure regulator to approximately 2 Bar to achieve a flow of about 3 L/min. See Figure 22 for flow rates vs. pressure to adjust to other flow rates. Once set you can tighten the locking ring to prevent changes in flow rates and simply use the shut-off valve to turn the flow off when not in use.

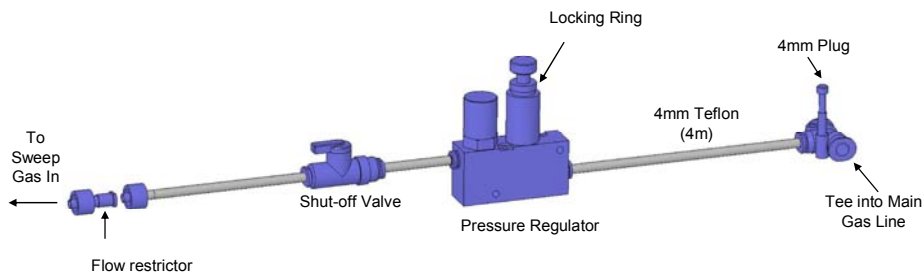


Figure 25 ACM Completion Kit

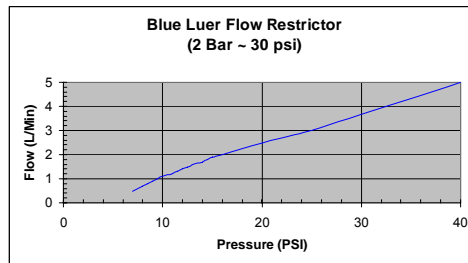


Figure 26. Flow Rate vs. Pressure

Connect the "Sweep Gas In" line from the ACM to the custom flow restrictor provided with the unit. Next, route the "Sweep Gas Out" from the ACM to an appropriate exhaust.

## Interfacing to the ICP

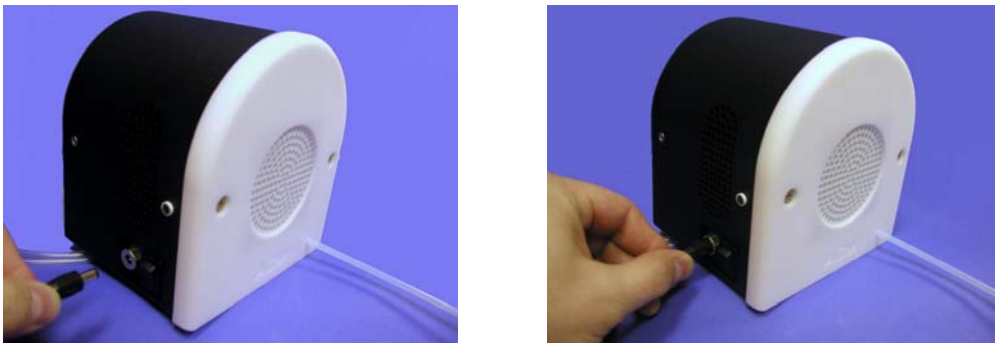
The ACM can be interfaced to the injector by a length of transfer tubing and connector that is supplied with the instrument.



**Figure 27. ACM with Injector Connector**

## ACM Power Connector

Plug the 12VDC power supply into the ACM using the 2.5mm plug from the power supply. Plug the AC power cord into the power supply and the other end into the main power supply.



**Figure 28. Connecting 12 VDC Power to the ACM**

## Cleaning the ACM Membrane

Should the ACM membrane require a more thorough cleaning, perform the steps listed below:

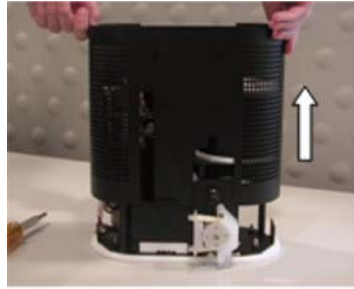
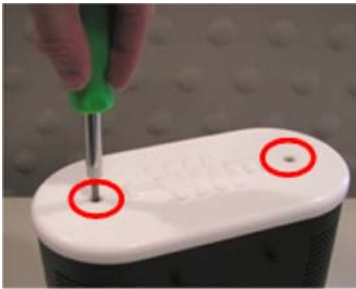
1. Disconnect power from the Apex unit and ACM module.
2. Remove ACM from Apex.
3. Attach the ACM rinse connector to the rinse bottle.
4. Fill the 500mL rinse bottle with a cleaning solution, i.e. 10% HNO<sub>3</sub>.
5. Connect the rinse bottle to the ACM module by pressing the ACM rinse connector into the ACM membrane unit.
6. Place the exit of the ACM over a sink and transfer the cleaning solution into the ACM membrane by squeezing the bottle.
7. Drain the cleaning solution.
8. Attach the ACM module back onto the Apex and connect all tubing and drain lines.
9. Power on the Apex and ACM and turn on the sweep gas to allow the membrane to dry.
10. The unit will be ready for use in approximately 10 minutes

# APPENDIX 3

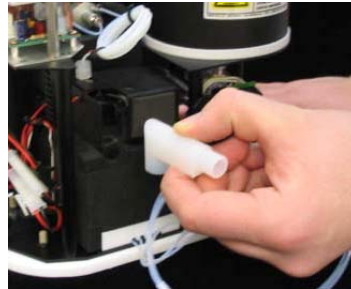
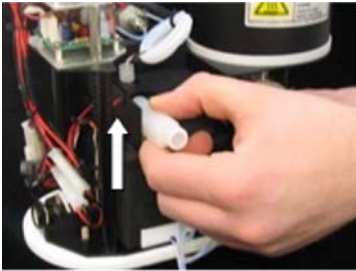
## Installing the Apex IR Mixing Chamber

Before beginning, make sure that the Apex is unplugged. If the Apex has been running, some internal components may be hot. Please use caution.

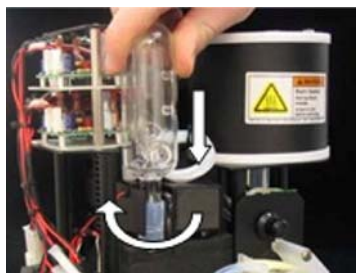
1. Remove the two PEEK screws from the top and remove top cover.
2. Remove the "C" cover plate by sliding it upward.



3. Remove the PFA elbow outlet adapter



4. Install the Teflon collar on the mixing chamber in place of the PFA elbow using a turning motion.



Note: The PFA collar may need to be heated to facilitate installation.

5. Replace the “C” cover and top plate and reinsert the PEEK screws.



Note: When replacing the “C” cover, make sure the edges are aligned with the groove in the bottom cover plate.

