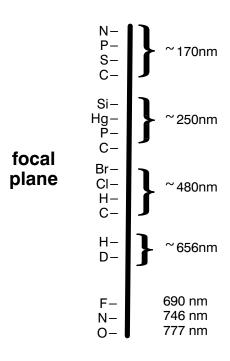
AED Focal Plane with Common Emission Regions



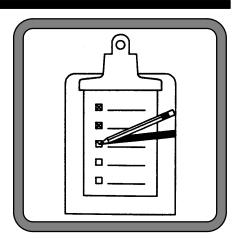
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Indicates that a procedure is also shown in the AED Maintenance video.

Using this Handbook



This handbook contains recent revisions and additions to the *Setting Up and Maintaining Your AED* manual. It is designed to **replace** the *Setting Up and Maintaining Your AED* manual shipped with your instrument. For more information about the theory of Atomic Emission Detection, see the primer *GC-AES Using Microwave Plasmas*.

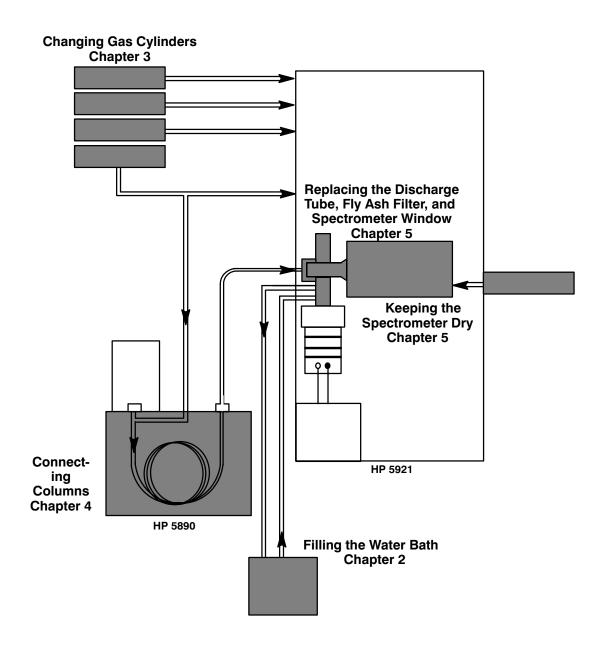
Read chapter 2 to get an overview of the entire system and to acquaint yourself with safety considerations. Most of this book covers the 3 areas of AED hardware which require maintenance:

- Water flow system—chapter 3
- Gas flow system—chapter 4
- Plasma and spectrometer hardware—chapter 6

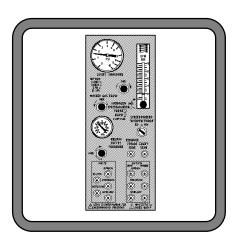
Each of these chapters begins with an overview of the particular system and then describes specific procedures. Remedies for the most common problems you may encounter with your system are described in chapter 7. The appendixes cover:

- Site requirements
- Consumables
- Table of spectral lines

Use the maintenance log printed on the inside of the front cover to keep track of maintenance procedures. A focal plane diagram is shown on the inside back cover.



Before You Use the AED



What's Involved

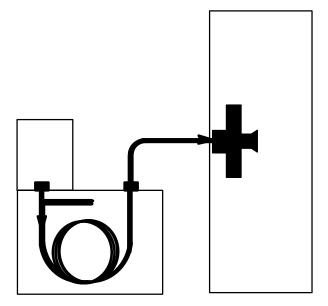
- Using your system safely
- Getting to know AED system hardware
- Configuring a gas chromatograph for the AED
- Shutting down the system
- Checking system readiness (Preflight Checklist)

Using Your System Safely

There are 6 potential safety hazards to be aware of when using the Atomic Emission Detector:

- High Temperature
- High Voltage
- Microwave Hazards
- Ultraviolet Light
- Effluent Venting
- Hydrogen Gas

High Temperature



High-temperature heated zones in the AED include the cavity assembly, the column transfer line, and the water bath. The column transfer line is surrounded by a removable insulated sleeve and should pose no burn hazard as long as the sleeve is in place. The cavity hardware under the hinged cover is easily accessible for maintenance procedures such as changing a column or replacing a discharge tube. The water bath is located behind the right panel and is exposed during filter replacement procedures. If you need to touch the cavity, column transfer line, or water bath you should turn off the instrument and wait until it has cooled to ambient temperature or wear protective gloves before touching it.

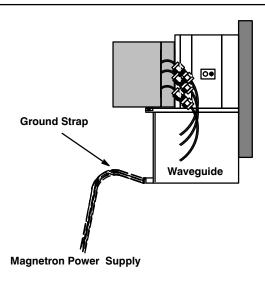
chpt 2-3

High Voltages

High-voltage hazards include the 5000 volt supply that is connected to the magnetron, the 120/220 volt supplies that are connected to the column transfer line, the emission source heater, the water bath heater, and the 115/120 volt circuits at the primary of the instrument supply.

WARNING

To reduce risk of electric shock, always disconnect the power cord when changing the fuse on the HP 5921A.



WARNING

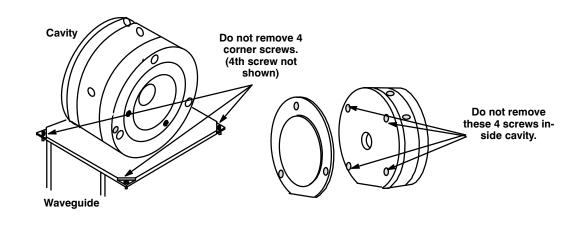
To reduce risk of electric shock, the ground strap for the magnetron power supply must always be connected to the waveguide when the HP 5921A is in operation.



If the magnetron and cavity assemblies are securely fastened to the waveguide, there is no microwave hazard.

WARNING

Never unscrew the cavity from the waveguide or disassemble it except as directed in this manual.

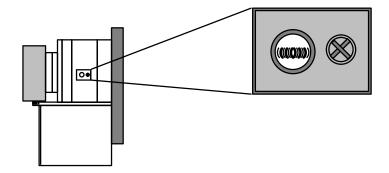


WARNING

Never operate the HP 5921A with the cavity, magnetron, or waveguide disassembled or mechanically loose.

If microwave leakage is suspected, call your HP service representative. If the instrument shows microwave leakage above 5 mW/cm² anywhere along the waveguide, shut down the instrument immediately. Measured levels of microwave leakage above 5 mW/cm² must be reported to Hewlett Packard. (5 mW/cm² at a distance of 5 cm is the amount of microwave leakage allowed from microwave ovens by the International Electrotechnical Commission and the United States Food and Drug Administration. This standard is published in IEC Publication 519-6 dated 1982 and in the Radiation Control for Health and Safety Act of 1968 (PL90-602), Section 358.)

Ultraviolet Light

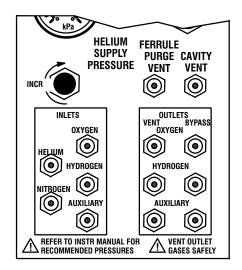


The AED plasma emits ultraviolet light which can damage the eyes if stared at for long periods of time. The view port in the cavity has a glass filter that prevents ultraviolet light from passing through to the viewer's eyes. This filter prevents light with a wavelength below 360 nanometers from leaving the cavity.

WARNING

To reduce risk of eye damage, never operate the HP 5921A without the glass filter in the view port of the cavity.





Vent AED effluents from the front panel to a fume hood, as ozone and other harmful gases may be produced by the plasma.

WARNING

To reduce risk of breathing possibly poisonous gases, vent AED effluents from the front panel to a fume hood.

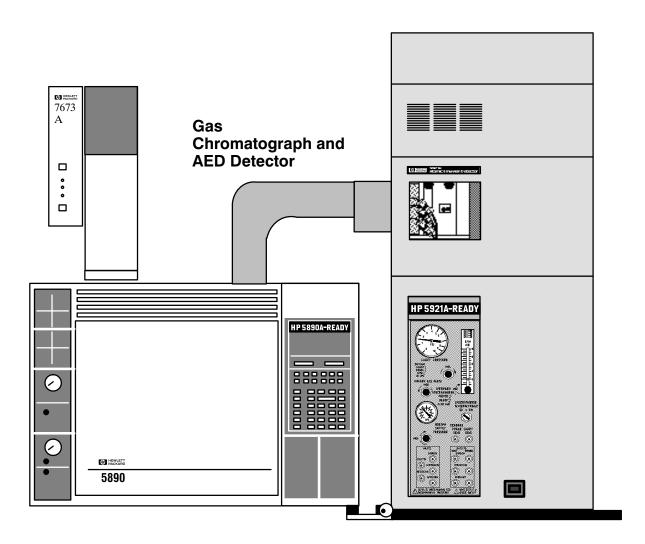
Hydrogen Gas

Ensure that hydrogen gas is plumbed to the hydrogen inlet.

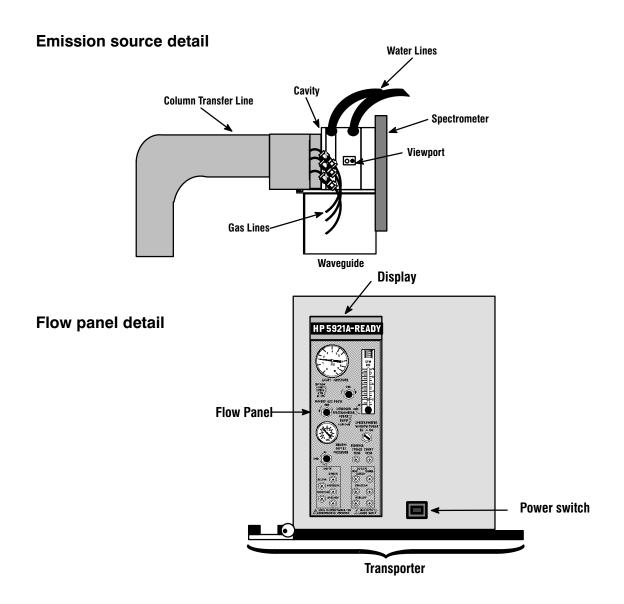
WARNING

To reduce risk of explosion, fire, and injury, ensure that hydrogen gas is connected to the inlet marked "Hydrogen".

Getting to Know AED System Hardware

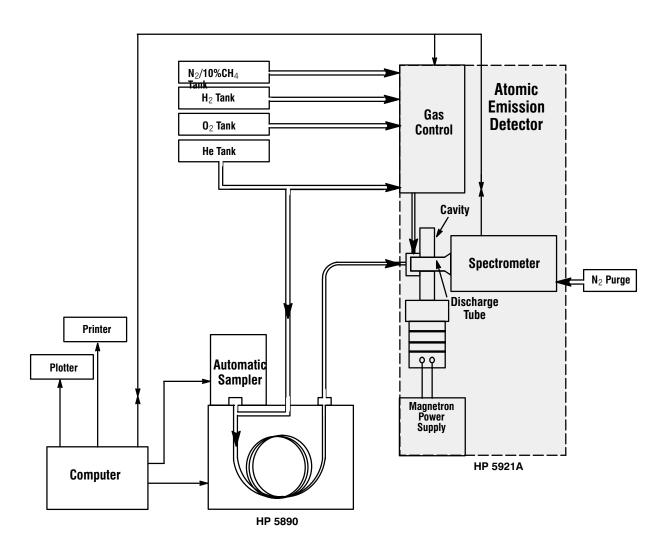


chpt 2-8



chpt 2-9

AED System Block Diagram



chpt 2-10

Configuring a Gas Chromatograph for the AED

When plumbing a GC for use with the AED use:

- Clean stainless steel or copper (never Teflon) tubing for plumbing.
- Vespel or graphite Vespel ferrules in all Swagelok unions and fittings. (Pure Vespel ferrules are used on the connections at the cavity).
- Matheson chrome-plated stainless steel diaphragm regulators.

To avoid leaks and carbon contamination in the gas system, remove all flow measuring devices. A getter may be used to purify the helium supply gas but avoid other traps in the system.

Shutting Down the System

Maintenance of the AED is easier if the instrument is kept running. However, if it becomes necessary to shut down your unit for one day or more, follow the procedures below.

Step	Reason
1. Turn HP 5921A power off.	
Turn off all gases except nitrogen. Keep ball just off the bottom of the NITROGEN SPECTROMETER PURGE flow meter (abou 200 ml/min).	A low nitrogen flow must be maintained in the spectrometer to preserve the diode array and grating.
3. Allow the water to cool and remove it from the HP 5921A.	Avoid burn hazard.
4. Follow the instructions in Chapter 3 for Replacing the Water Filter/Draining the Water Bath to purge all water lines in the	If cool water is left in the system, algae or bacteria will contaminate the cavity, water lines, and bath in a short time.
HP 5921A with helum or nitrogen gas.	



Preflight Checklist

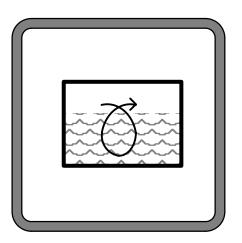
Before running an analysis with the AED system, use the checklist on the following page to ensure that the system is ready.

Note: The tank pressures listed assume little pressure drop between the tank and the detector or that pressure is being read from a single stage regulator located near the instrument. Depending on the hardware configuration, the cavity pressure can be between 2-5 psi during solvent venting.

Preflight Checklist

Water Water Bath 65°C Level OK? Gas **Pressure** He N_2 **O**₂ H_2 Aux 60-80 psi 60 psi 20 psi 25 psi 70 psi at Tanks OK? **Detector** N₂ Spect albungle2 liters/min He Supply Cavity **Panel** Pressure Pressure 30 psi Settings 1.5 psi OK? Makeup **Ferrule** Cavity **Flow Purge Vent** Vent Rate 20-50 60 ml/min ml/min OK? Split Ratio OK? Column Head Pressure OK? **Plasma** On? Solvent Vent On/Off?

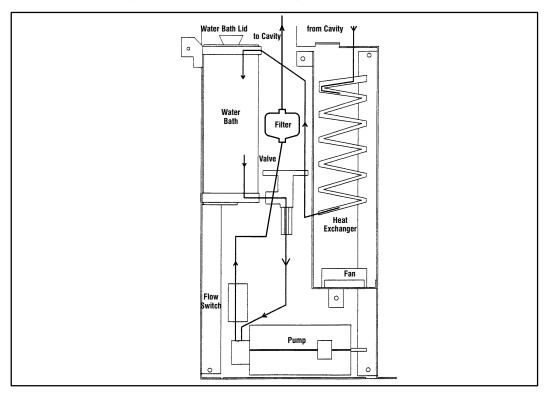
Maintaining the Water Flow System



What's Involved

- Understanding the water system
- Keeping clean water in the system

Understanding the Water Flow System



The inclusion of a water cooled discharge tube substantially increases discharge tube life and reduces background emissions. Decreased wall temperatures reduce oxygen and silicon emissions by decreasing volatilization from the tube itself. Lower discharge tube wall temperatures also decrease erosion of the interior surfaces. This allows for longer continuous use of the AED before discharge tube replacement is necessary.

Approximately 750 ml of cavity cooling water is kept at 65° C in a stainless steel bath on the right side of the instrument. Constant temperature water ensures stable detector operation and 65° C inhibits the growth of algae or bacteria. As shown on the diagram, water from the water bath passes through the valve, pump, flow switch, and

filter before reaching the cavity. The water is heated in the water bath, flows through the on/off valve, and then through the pump. From the pump, the heated water travels through the flow switch to ensure sufficient flow rate (about 400 ml/min). From the flow switch, the water passes through a 5 micron water filter before leaving the module. The water then makes its way to the cavity, where it cools the plasma. From the cavity, the water goes through a fan-cooled heat exchanger to dissipate the excess heat added by the plasma. The water then returns to the bath.

Water must be flowing through the detector at all times. The lower the water flow, the shorter the life of the discharge tube. Without sufficient water flow the flow will drop, causing the flow switch to trip. This may be caused by a

- Dry water bath
- Plugged flow system
- Clogged water filter
- Failed pump

If the flow switch trips, it deactivates the magnetron and shuts off the plasma, the pump, and all the heated zones. The instrument will then display a COVER/NO WATER message. You must then correct the water problem *and repower the system* in order to continue.

A COVER/NO WATER message could also be displayed if the hinged top door is raised. The COVER/NO WATER message also signals a fault in the water bath temperature or a low water flow rate. Low flow rates result from a clogged filter, a closed valve, or worn pump gears.

Keeping Clean Water in the System

- Check the water level at least twice a month.
- Add only distilled water to the bath to maintain the level within one inch of the top. Do not top off the water level because the lid extends within the bath. If the bath runs dry, the flow switch will deactivate and the instrument will display a COVER/NO WATER message.

Filling the Water Bath

Step	Reason
Fill the water bath to within 1 inch of the top with distilled water. Avoid spilling water into the instrument.	Distilled water will keep the system clean. Do not use tap water as it will contaminate the system. Do not use deionized water as it will corrode the system. Do not use algae inhabiters as they will affect the microwave tuning.
2. Keep the lid on the water bath.	Prevent water loss through evaporation.

- Do not put rust-prone objects in the bath, as rust will contaminate the system.
- If the system is to be shut down for more than one day, drain the water bath to prevent algae growth. Purge the water lines with helium or nitrogen. See below for instructions.

Warning

Shock Hazard—High-voltage,high—current wires are routed along the instrument base. Do not spill water into the instrument.

Replacing the Water Filter/ Draining the Bath

■ Check the water filter once a month for a clogged or darkened appearance. A COV-ER/NO WATER reading may indicate the filter needs to be changed. Below are the steps to take in replacing the water filter. You will need a 9/16" wrench, new 5 micron filter, 2 pt pozidriv screwdriver, a flathead screwdriver, and paper towels.

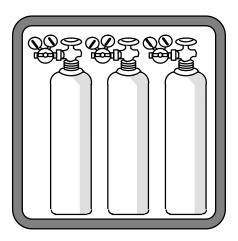
Replacing the filter

- 1. Turn off the instrument. Allow the water to cool.
- 2. Remove the right panel. Do not open the hinged door to the pc board area.
- 3. Turn on/off valve 90° clockwise when viewed from the top to prevent water bath drainage.
- 4. Disconnect the hose clamp above the water filter and pull off the hose.
- 5. Loosen the screw on the band clamp holding the filter in place.
- 6. Slide out the water filter from the metal harness.
- 7. Disconnect the bottom hose clamp and separate the filter from the hose.
- 8. Reconnect the new filter to the hose using the hose clamp.
- 9. Slide the water filter back into the metal harness.
- 10. Tighten the screw on the band clamp with the flathead screwdriver.
- 11. Reconnect the filter to the top hose and clamp.
- 12. Open the valve.
- 13. Replace the right panel with the five M4 screws.

Draining the bath

- 1. Turn off the instrument. Allow the water to cool.
- 2. Remove the right panel. Do not open the hinged door to the pc board area.
- 3. Turn valve 90° clockwise when viewed from the top to prevent water bath drainage.
- 4. Disconnect the fitting joint underneath the valve with the 9/16" wrench.
- 5. Drain the water from the bath. The bath contains approximately 750 ml of water.
- 6. Open the valve to drain the standing water.
- 7. Close the valve.
- 8. Refill the bath following the instructions above. Open the valve and check for leaks at the fitting joints. Leave the valve in the open position.
- 9. Replace the right panel with the five M4 screws.

Maintaining the Gas Flow System



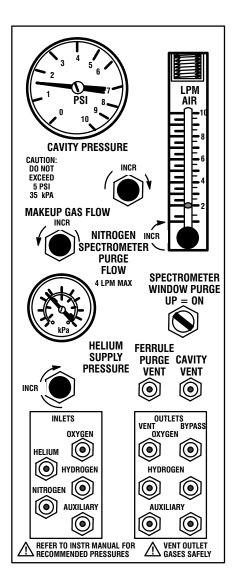
What's Involved

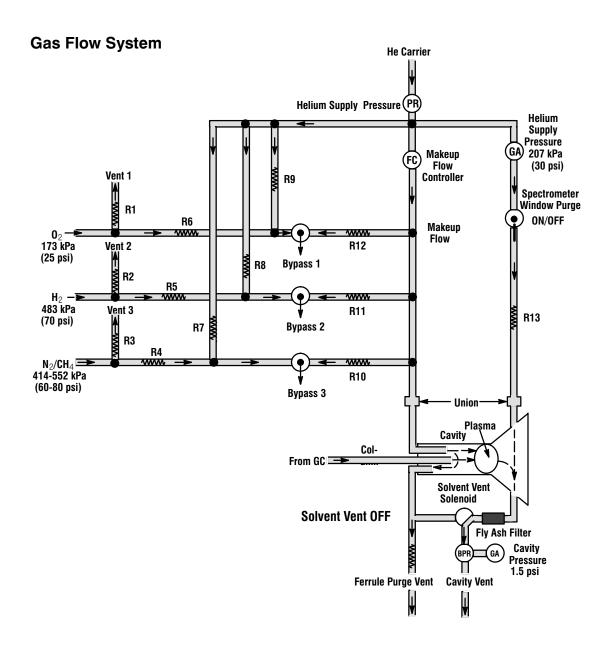
- Understanding the gas flow system
- Setting gas flows
- Changing gas cylinders

Understanding the Gas Flow System

A schematic of the AED gas flow system is shown on the opposite page. The carrier gas (helium) enters the unit through the HELIUM SUPPLY PRESSURE regulator (PR) which controls the supply pressure at 207 kPa (30 psi). Helium supplies SPECTROMETER WINDOW PURGE flow, MAKEUP GAS FLOW through the makeup flow controller (FC), and gas to the three restrictors marked R7-R9. The SPECTROMETER WINDOW PURGE flow is shown turned off in this diagram.

CAVITY PRESSURE, which is controlled by a backpressure regulator (BPR), should be maintained at 1.5 psi.





chpt 4-3

Reagent gas flow

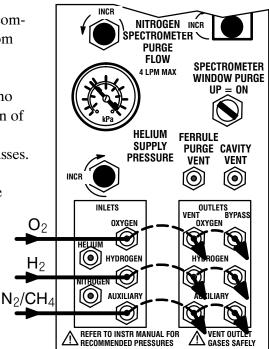
Three different reagent gases are used:

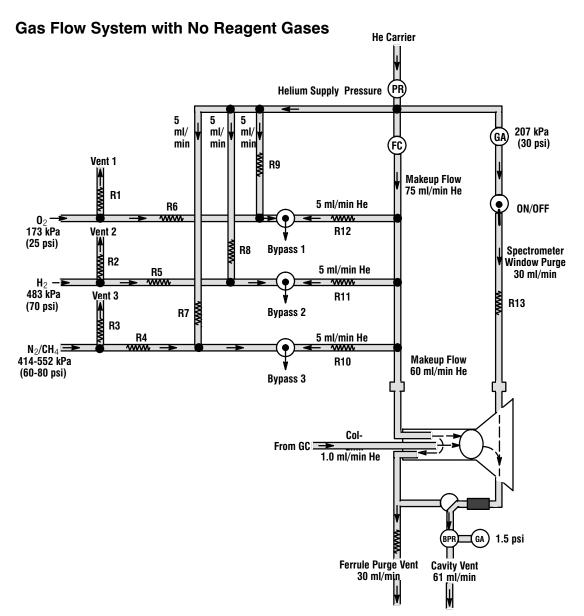
- oxygen
- hydrogen
- mixture of 10% methane in nitrogen.

The three reagent gases are turned on and off in any combination by the computer actuating the appropriate solenoid valve.

The diagram opposite shows each of the three reagent gases entering the detector at the left and about 5 ml/min is vented out through restrictors R1-R3. A small amount of each reagent gas flows through R4-R6 and then combines with 5 ml/min of helium carrier gas from the pressure regulator (PR).

The position of Bypasses 1–3 indicate that no reagent gases are turned on and the 5 ml/min of helium carrier gas and small amounts of reagent gases are vented through these bypasses. With no reagent gases turned on, 5 ml/min of helium flow is lost to each of the three reagent branches and also vented out Bypasses 1–3.





Makeup Flow + Window Purge + Column Flow = Ferrule Purge + Cavity Vent (75 - 5 - 5 - 5) ml/min + 30 ml/min + 1 ml/min = 30 ml/min + 61 ml/min 60 ml/min + 31 ml/min = 91 ml/min

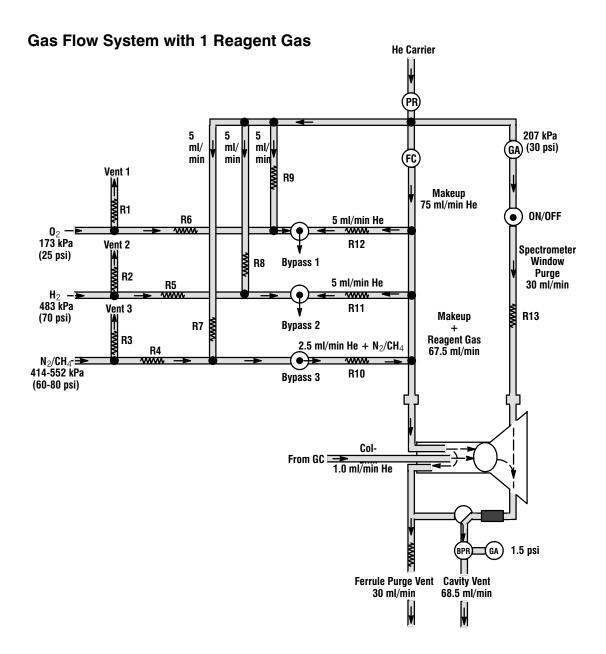
chpt 4-5

The schematic on the opposite page shows the flow pattern when nitrogen/methane (AUXILIARY gas) is turned on and oxygen and hydrogen are still bypassed. The nitrogen/methane enters the unit from the tank regulator. About 5 ml/min is vented out through restrictor R3. A low flow rate (0.02 ml/min) passes through restrictor R4. This flow of nitrogen/methane combines with 5 ml/min of helium from restrictor R7. The solenoid Bypass 3 valve is in the *closed* position and there is 5 ml/min of makeup flow opposing the flow of nitrogen/methane. This results in about 2 ml/min of the helium/nitrogen/methane mixture flowing through restrictor R10 and joining the makeup helium stream from the MAKEUP GAS FLOW controller. This combined stream of makeup and reagent gas (67.5 ml/min) then flows through the discharge tube in the cavity.

Hydrogen enters the detector and about 5 ml/min is vented out through R2. It flows through R5 at a rate of about 0.07 ml/min. Hydrogen then combines with 5 ml/min of helium from restrictor R8. In this case, however, the solenoid Bypass 2 valve is in the *vent* position. The hydrogen/helium flow goes through the solenoid valve and out Bypass 2. Since oxygen is also bypassed, it follows the same flow pattern as hydrogen and the oxygen/helium flow is vented out Bypass 1.

Makeup & Reagent +Window Purge +Column flow= Ferrule Purge +Cavity Vent (75 - 5 - 5 + 2.5) ml/min +30 ml/min + 1 ml/min = 30 ml/min +68.5 ml/min

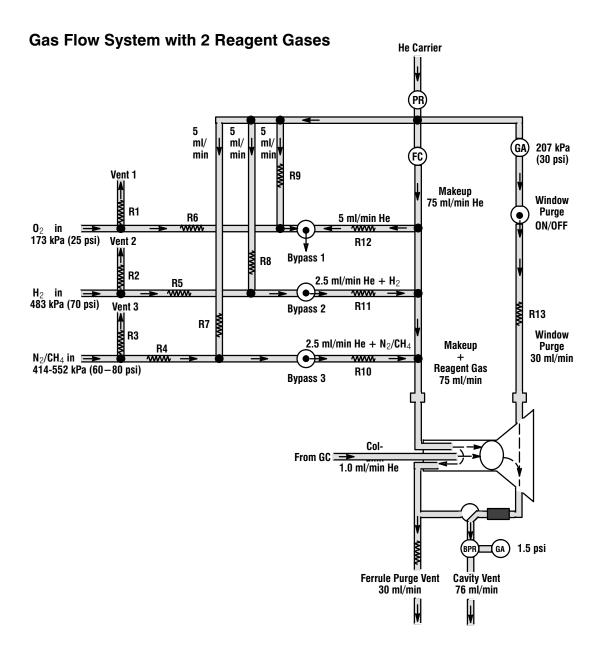
67.5 ml/min + 31 ml/min = 98.5 ml/min



When two reagents are turned on, as in the figure on the opposite page, the total makeup and reagent flow is 75 ml/min. Since in both cases 30 ml/min is used to purge the column ferrule through the **FERRULE PURGE VENT**, the actual makeup flow (not including column flow) into the plasma for one or two reagents is 37.5 or 45 ml/min respectively.

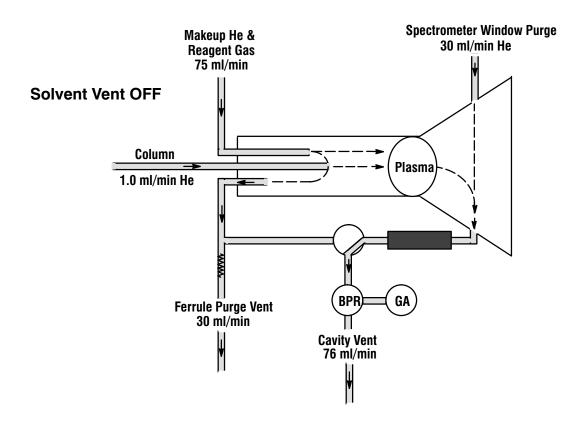
Number of reagents	Ferrule Purge Vent	Cavity Vent
no reagent 1 reagent 2 reagents	30	60 ml/min + column flow 67.5 ml/min + column flow 75 ml/min + column flow

Makeup & Reagents+Window Purge+Column flow= Ferrule Purge +Cavity Vent (75 - 5 + 2.5 + 2.5) ml/min + 30 ml/min + 1 ml/min = 30 ml/min + 76 ml/min 75 ml/min + 31 ml/min = 106 ml/min



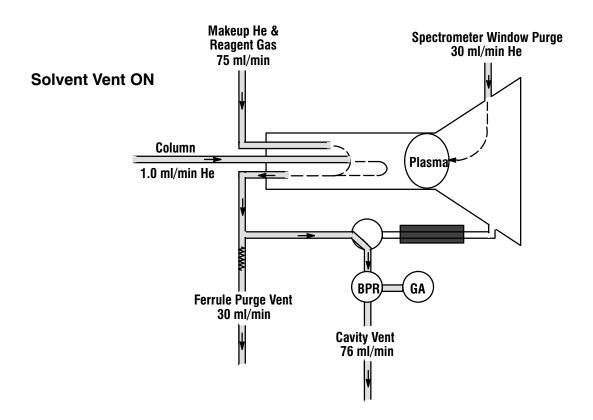
Solvent Venting

The solenoid valve that turns solvent venting on or off is controlled by the computer and is time programmable. The figure below shows the solvent vent OFF mode where the column effluent enters the plasma. The column effluent and about 37 ml/min of makeup and reagent gas flow through the plasma. The other 30 ml/min of makeup gas purges the column ferrule and exits out the **FERRULE PURGE VENT**. The makeup gas and column effluent pass through the plasma, combine with the spectrometer window purge helium, pass through the solenoid valve, and out through the **CAVITY VENT**.



chpt 4- 10

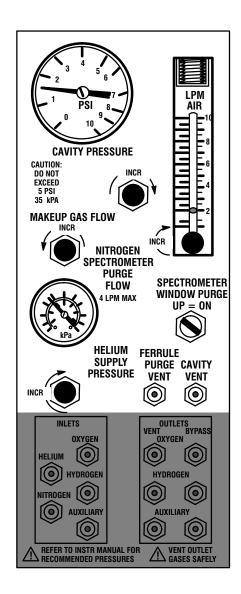
When the solvent vent is on, the solenoid valve is switched to the other position so that the exit tube on the window side of the chamber is now closed. The spectrometer window purge gas is now forced to flow backwards through the plasma. In doing so, it flows past the end of the column, and sweeps the column effluent and the combined makeup and reagent gas through the solenoid valve and out through the **CAVITY VENT.**



Setting Gas Flows

Helium flow is set using the CAVITY PRESSURE regulator and gauge, the HELIUM SUPPLY PRESSURE regulator and gauge, the SPECTROMETER WINDOW PURGE valve, and the MAKEUP GAS FLOW controller. Flows for helium are measured at the FERRULE PURGE and CAVITY VENTS.

The **NITROGEN SPECTROMETER PURGE FLOW** gauge should show a nitrogen flow of approximately 2 liters/min.



© To Set Gas Flows

Set Gas Flows	
Step	Reason
Turn off all reagent gases and the solvent vent at the computer. Ensure that a column is connected or cavity is plugged.	Simplify number of gases and flow paths in the system. When the column is connected, column flow is included in cavity vent measurements.
Use the supply pressure regulator (PR) to adjust the helium supply pressure gauge to 30 psi.	
4. Turn the WINDOW PURGE valve off. Adjust the MAKEUP GAS FLOW controller until you measure 30 ml/min at the CAVITY VENT. Adjust the CAVITY PRESSURE to 1.5 psi. CAUTION: Never allow cavity	Spectrometer window purge flow must be off to set the makeup flow.
pressure to exceed 5 psi.	
 Turn the WINDOW PURGE valve on. You should measure 60±5 ml/min at the CAVITY VENT. 	At this point, makeup and window purge gases are exiting at the cavity vent. This verifies that the flow from the Window Purge is approximately 30 ml/
6. Now measure the FERRULE PURGE VENT. The purge vent flow should measure 20-50 ml/min.	The 60 ml/min of makeup flow should be divided about equally between the cavity vent and the ferrule purge vent.
7. Turn the WINDOW PURGE valve on. You should measure 60 ml/min at the CAVITY VENT and 25-50 ml/min at the FERRULE PURGE VENT.	
<u>+</u>	

Changing Gas Cylinders

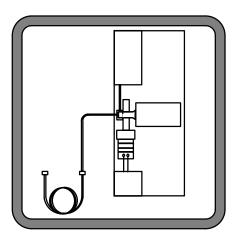
WARNING

To reduce risk of explosion, fire, and injury, ensure that hydrogen gas is connected to the inlet marked "Hydrogen".

Gas purity is most likely to be compromised when gas cylinders are being changed. Follow the procedure below to ensure gas purity.

Step	Reason				
I. If you are using a getter, cool it to room temperature.	The getter must be cooled so it won't react with the air when you open the gas line.				
2. Ensure that the inside of the valve of the new tank is clean. Remove any debris.	Dirty valves can be a source of leaks. Debris can clog restrictors in flow system.				
Disconnect the old cylinder and connect the new cylinder.					
4. Open and close the tank valve; then bleed the pressure out of the regulator. Repeat 5 times. Open the inlet fitting at the GC to bleed the gas through the line.	The regulator fills with air after it is removed from the tank. Filling it with gas a little at a time and bleeding the air out of it ensures that air won't diffuse back into the tank.				
5. Turn the gas cylinder on and use a thermal conductivity leak detector (never a water-based leak detector) to check the tank fitting and the fitting you retightened at the GC.	Dirty valves can be a source of leaks. Debris can clog restrictors in flow system. If a leak exists, a water-based leak detector will create high hydrogen backgrounds.				

Connecting Columns to the AED



What's Involved

- Installing a low-bleed column
- Installing a high-bleed column

© Changing Columns

When you change columns frequently *or* use columns with high bleed rates (thick-film or noncross-linked columns), you will want to splice analytical columns to a transfer column. The transfer column is a 1.5 meter length of cross-linked thin-film or uncoated capillary column. The analytical column is connected to the transfer column using the swept fitting in the GC oven. This fitting allows access to both ends of an analytical column in the GC oven. The transfer column prevents silicon dioxide "snowballs" from forming in the discharge tube.

When you change columns infrequently, or use low-bleed (thin-film or cross-linked) columns, you can route them from the oven through the transfer line, and connect them directly to the AED cavity.

Changing columns is demonstrated on the videotape which accompanies this manual.

Installing a Low-Bleed Column

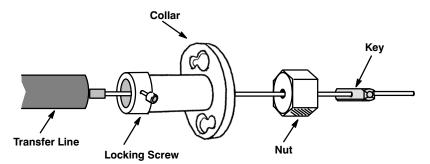
After the column is installed in the inlet, the other end is fed directly through the column transfer line and gas union into the discharge tube. The column is sealed at the gas union with a Graphpack ferrule, a key, and a ferrule nut. High-bleed columns are spliced in the oven to a section of low-bleed column (the transfer column) before they are connected to the cavity. This procedure applies to all low-bleed capillary columns.

You will need:

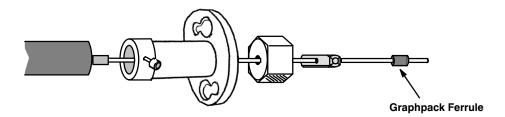
- \blacksquare 1/4", 3/8", and 5/16" open-end wrenches
- 2.5 mm Allen wrench
- typewriter correction fluid
- Exacto knife with new blades
- Graphpack ferrule to fit your new column
- ferrule reamer
- column cleaving tool
- metric ruler
- methanol

WARNING Wear safety glasses while performing this procedure.

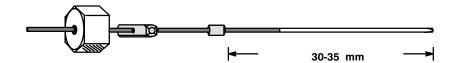
- 1. Shut down AED power. Remove the cavity block insulation from the column transfer line. Allow the transfer line, oven, and cavity to cool.
- **2.** Slide lab jack under transfer line for support. Unscrew locking screw on side of collar. Unscrew the collar from the heater block. Twist it and slide it back over the end of the transfer line securing it under the insulation.
- **3.** Disconnect transfer line by loosening the ferrule nut with the 3/8" wrench, using the 5/16" wrench to hold the body of the gas union. Move the detector away from the gas chromatograph using the transporter.
- **4.** Remove the old column and hardware from the transfer line and oven. If you suspect broken pieces of column remain inside the cavity, unscrew the three screws that hold the cavity to the receiver (refer to chapter 6 for details) and use an air line to blow out any broken pieces of glass from the cavity.
- **5.** Install the inlet side of the new column with the 1/4" wrench. Feed the other end of the column up through the transfer line until 50–60 mm of it extends past the end of the transfer line.
- **7.** Slide the ferrule nut and the key onto the column near the transfer line as shown below.



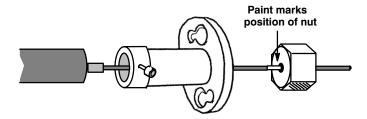
8. Use the appropriate ferrule reamer to size the internal diameter of the Graph-pack ferrule snugly to the column. Slide the ferrule onto the column with the metal end facing the key and nut. (Attempting to force a column through an undersized ferrule will damage the graphite packing inside the ferrule.)



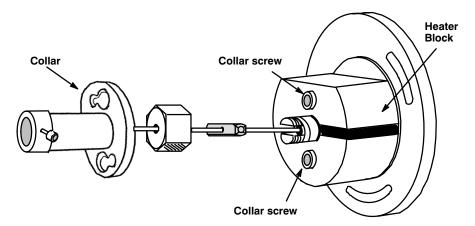
- **9.** Cut off the column end to ensure no graphite remains in the column.
- **10.** Carefully feed the column into the gas union body, until the Graphpack ferrule is seated on gas union body.
- 11. Slide the ferrule key forward and position it in slot on the gas union. Slide the ferrule nut forward and hand tighten it onto the gas union. Use the 3/8" and 5/16" wrenches to tighten the nut down on the ferrule about 1/8 of a turn. This step seats the ferrule firmly onto the column and keeps the nut and key from falling off the end of the column. Unscrew the column nut and slide the nut, key, and ferrule back near the transfer line.
- **12.** OPTIONAL: Scrape the polyimide coating off the last 25 mm of column with an exacto knife. (Use new blades.) Polyimide may also be burned off a column with helium flowing through it using a butane lighter. Wipe the end of the column with methanol to remove any polyimide residue and fingerprints.



- **13.** Move the ferrule on the column until it is 30-35 mm from the end of the column. Slide the ferrule nut and key forward on the column until they are up against the ferrule.
- **14.** Mark the column with correction fluid behind the nut.

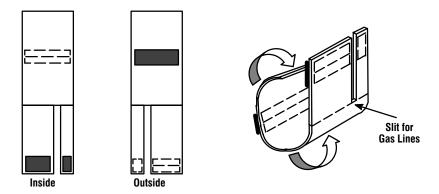


- **15.** Using the transporter and a lab jack, move the detector toward the gas chromatograph, carefully feeding the column into the gas union body, until the Graphpack ferrule is seated on gas union body.
- **16.** Slide the ferrule key forward and position it in slot on the gas union. Slide the ferrule nut forward and hand tighten it onto the gas union.



- **17.** Reposition column if necessary to get leading edge of correction fluid flush with the back of the nut.
- **18.** Tighten the ferrule nut with the 3/8" wrench on the nut and the 5/16" wrench on the gas union. Tighten 1/4 to 1/2 turn.
- **19.** While pulling gently on the column in the oven, use the transporter to guide the column into the gas union until the gap between the transfer line and the back of the ferrule nut is reduced to 5 mm or until the detector is as close as it will get to the GC.
- **20.** Slide the collar over the transfer line to the screws on the heater block. Turn the collar clockwise to capture the screws and tighten the collar against the heater block with the 2.5 mm Allen wrench. Lab jack may be removed at this point.
- **21.** Adjust transfer line until the back edge of the docking cap (at end of transfer line) is flush with the end of the collar. Use 2.5 mm Allen wrench to tighten the locking screw hand tight.

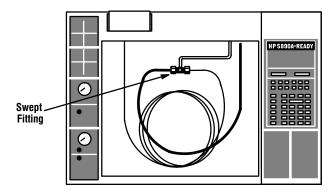
22. Reinstall insulation over the heater block.



Replacing the Cavity Insulation

23. Turn system back on.

Installing a High-Bleed Column



The high-bleed analytical column must be spliced to a short (1.5 meter) length of transfer column. The transfer column is a thin-film or uncoated piece of capillary column. It is spliced to the analytical column in the oven using a swept fitting, and runs the length of the heated transfer line to the cavity.

The transfer column may slide *into* or *over* the analytical column to create the lapped joint. Use the table below to select the diameter of transfer column you will need.

Analytical .	Transfer column					
column 1 00jum	530 or 320 μm					
200 μm	530 μm					
320 μm	530 μm					
530 μm	320, 200, or 1 00µm					

The analytical column is always looped counterclockwise in the oven and connected to the right-hand side of the fitting. The transfer column is always looped 3/4 of a turn clockwise inside the oven and connected to the left-hand side of the fitting. The column with the smaller diameter is always connected to the swept fitting first.

You will need:

- \blacksquare 1/4", 3/8", and two 5/16" open-end wrenches
- typewriter correction fluid
- column cleaving tool
- 1.5 meters of thin-film or uncoated capillary column
- metric ruler

To Install a High-Bleed Column

WARNING

Wear safety glasses while performing this procedure.

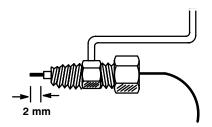
- 1. Shut down AED power. Remove the cavity block insulation from the column transfer line. Allow the transfer line, oven, and cavity to cool.
- **2.** Slide lab jack under transfer line for support. Unscrew locking screw on side of collar. Unscrew the collar from the heater block. Twist it and slide it back over the end of the transfer line securing it under the insulation.
- **3.** Disconnect transfer line by loosening the ferrule nut with the 3/8" wrench using the 5/16" wrench to hold the body of the gas union. Move the detector away from the gas chromatograph using the transporter.
- **4.** Remove the old column and hardware from the transfer line and oven. If you suspect broken pieces of column remain inside the cavity, unscrew the three screws that hold the cavity to the receiver (refer to chapter 6 for details) and use an air line to blow out any broken pieces of glass from the cavity.
- **5.** Install the inlet side of the new column with the 1/4" wrench.
- **6.** Determine the diameter of the transfer column using the table on the previous page. From the oven, route the transfer column up through the transfer line until it extends 100–200 mm past the end of the transfer line.

chpt 5-10

- **7.** Loop the oven end of the transfer line as large as possible without touching the inside of the oven and cut the end of the column flush with the right-hand side of the fitting.
- **8.** Loop the last coil of the analytical column as large as possible without touching the inside of the oven and cut the end of the column flush with the left-hand side of the fitting.

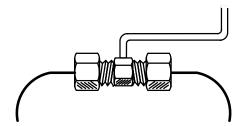
To install the column with the smaller internal diameter

- **9.** Slide 1/16 Swagelok nut and Vespel reducing ferrule onto the smaller diameter column. Loop the column as indicated above and insert the column into the appropriate side of the fitting. If the smaller column is the analytical column, insert it into the right side of the fitting. If the smaller column is the transfer column, insert it into the left side of the fitting.
- **10.** Hand-tighten the nut and ferrule. Cut off end of column so that 2 mm extend past the end of the fitting. Tighten down on ferrule 1/2 turn past hand tight with the two 5/16" wrenches.



To install the column with the larger internal diameter

- **11.** Install second Swagelok nut and Vespel reducing ferrule on larger diameter column.
- **12.** Loop the larger diameter column as described above and carefully thread the larger column over the 2 mm exposed end of the smaller diameter column already installed in the fitting. Push the larger column all the way onto the smaller column until it bottoms on the ferrule. Make sure columns are looping freely. Hand-tighten nut and ferrule.
- **13.** Mark the column at the back of the nut with correction fluid.
- **14.** Retract larger diameter column until you measure 10 mm between the end of the fitting and the mark on the column. Maintaining the 10 mm gap, tighten down on the ferrule with the two 5/16" wrenches. There should be 8 mm of overlap inside the splice.
- **15.** Ensure that columns are looping freely and not touching the oven wall.



16. Connect the transfer column to the cavity using the instructions above for installing a low-bleed column.

Maintaining the Plasma and Spectrometer Hardware



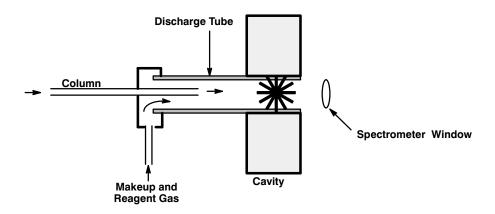
What's Involved?

- Replacing the discharge tube
- Cleaning and replacing the spectrometer window
- Replacing the fly ash filter
- Keeping the spectrometer dry

Replacing the Discharge Tube

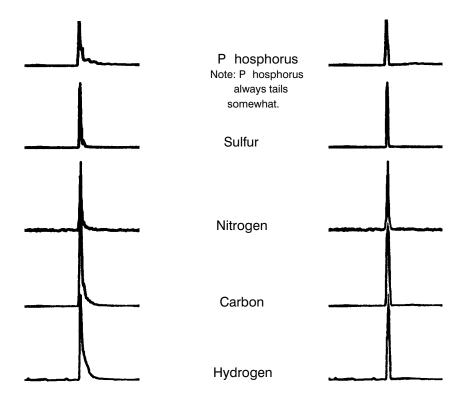
The discharge tube is a 42-mm-long tube of polyimide coated fused silica. This tube contains the plasma and bridges the gap between the column and the spectrometer. It is held in place by a Viton O-ring on the spectrometer side and by a polyimide ferrule on the GC side of the cavity. The tapered polyimide ferrule keeps the water out of the heated zone and seals the gas union to the discharge tube.

The types of analyses done will determine how dirty the discharge tube gets and how often it needs to be changed.



When Does the Discharge Tube Need Replacing?

Discharge tube life varies with the types of columns and samples used, the oven temperature, amount of instrument use, etc. Inspect your chromatograms. If a number of elements tail consistently, you may need to replace the discharge tube. See common problem symptoms in chapter 7 for more information about how to determine when the discharge tube needs replacing.

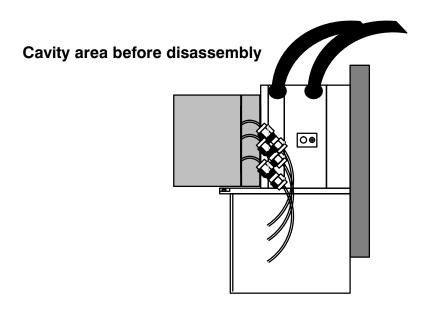


Before Changing the Discharge Tube

After Changing the Discharge Tube

You will need

- 3/8" and two 5/16" open-end wrenches
- Needlenose pliers
- 3 mm and 2.5 mm Allen wrenches
- discharge tube
- discharge tube ferrule
- discharge tube O-ring

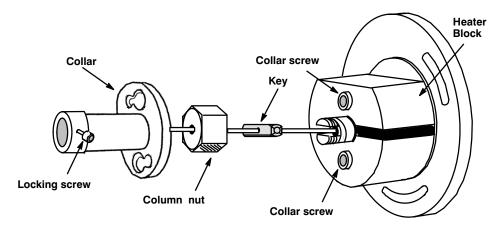


To Replace the Discharge Tube

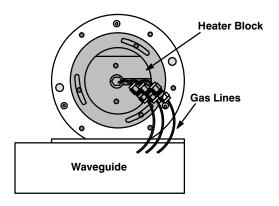
WARNING

Wear safety glasses while performing this procedure.

1. Shut down AED power. Remove the insulated sleeve from the cavity. Allow the transfer line and cavity to cool.



- **2.** Unscrew the locking screw on the transfer line collar using the 2.5 mm Allen wrench. Then loosen the two screws holding the collar to the heater block with the same wrench. Twist the collar and then slide it back over the end of the transfer line.
- **3.** Disconnect the transfer line by loosening the column nut with the 3/8" wrench while holding the body of the gas union with the 5/16" wrench. Support the transfer line with lab jack and swing out of the way. Use transporter to move detector away from gas chromatograph.



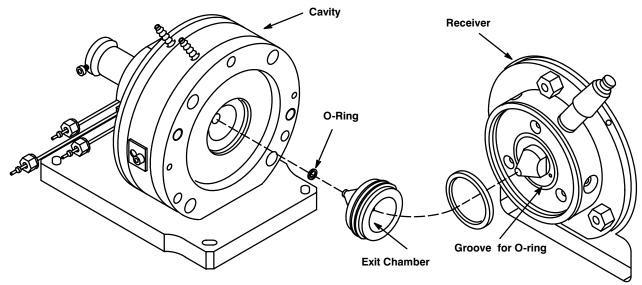
- **4.** Disconnect the 3 gas lines from the gas union with two 5/16" wrenches.
- **5.** Unscrew the heater block with 2.5 mm Allen wrench and place it on waveguide.
- **6.** Loosen the three screws holding the cavity to the receiver with the 3 mm Allen wrench. Swing cavity away from the receiver.

WARNING

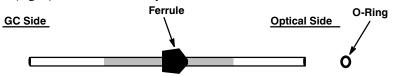
Never operate the HP 5921A with the cavity assembly disconnected from the waveguide.

- **7.** Remove brown O-ring from receiver liner and set aside.
- **8.** Unscrew exit chamber with fingers from right side of cavity.
- **9.** Disconnect the gas union holding the 5/16" wrench on the end of the union while rotating the nut counterclockwise with the 7/16" wrench. Support the gas fittings as you unscrew the nut.

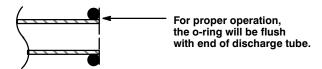
chpt 6-6



10. Remove the gas union from GC side of cavity. The discharge tube will usually come with it. If you suspect pieces of discharge tube remain inside the cavity, see *Cleaning the Cavity* later in this chapter. Remove the discharge tube O-ring from the optical (right) side of the cavity.



11. Slide the ferrule onto the new discharge tube. The end with the short taper must be toward the optics. Place discharge tube O-ring into optical side of cavity and hold there with your right hand. Push discharge tube into GC side of cavity with your left hand until you can feel that the end of the tube is flush with the O-ring.



- **12.** Keeping your right forefinger on the O-ring, carefully slip gas union back over discharge tube and hand-tighten the screw. Loosely install exit chamber.
- **13.** Connect gas union with two wrenches turning 1/4 to 1/2 turn past hand tight. Keep the tubes approximately horizontal for heater block installation.
- **14.** Finish tightening the exit chamber.
- **15.** Reinstall heater block with 3 Allen screws.
- **16.** Turn on the instrument with the cover up. The cover must be up to protect the user from hazardous electric shock. Check for water leaks. Within a few minutes, the instrument will detect that the cover is up and display a COVER/NO WATER fault message. This is normal. Turn off the instrument before proceeding.
- **17.** Reinstall O-ring on receiver.
- **18.** Slide cavity up over receiver and tighten the screws on cavity with 3.0 mm Allen wrench.
- **19.** Reconnect gas lines: 1 supply line and 2 purge lines marked "S" and "P".
- **20.** Check for gas leaks. Cap off gas union with ferrule, key, and column nut. Put finger over end of column nut and check that cavity pressure will increase to 1.5 psi.

Caution Do not increase the CAVITY PRESSURE to greater than 5 psi.	
--------------------------------------------------------------------	--

21. Reinstall transfer line and column (see instructions in chapter 5). Turn on system.

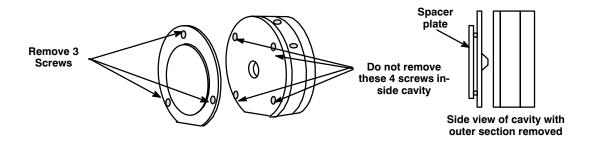
chpt 6-8

Cleaning the Cavity

Breaking a discharge tube during installation may require that the two outer sections of the cavity be removed and cleaned. This procedure assumes that the cavity has already been disconnected from the receiver.

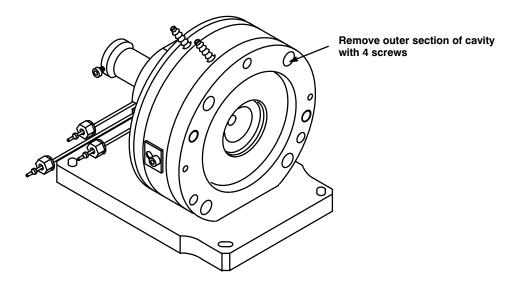
You will need

- 2.5 mm allen wrench
- cavity brushes
- union with barbed fittings
- lab tissue
- 1. Remove three screws holding the outer section of the cavity facing the heater block. Do not attempt to remove spacer plate on the outside of this section. If outer section is difficult to remove, screw screws into the extra holes provided in the section to loosen it. Once inside, remove any pieces of glass. Wipe dry.



WARNING

Do not remove the 4 screws that hold together the two inner sections of the cavity. Failure to reassemble these sections of the cavity correctly may result in microwave leakage.



- **2.** Remove the 4 screws holding the outer section of the cavity facing the receiver with the 2.5 mm allen wrench. Remove any pieces of glass. Wipe dry.
- **3.** Remove the two water lines from the cavity by pulling them off the fittings. You do not have to remove the hose clamps to do this.
- **4.** Run the larger brush through the cavity water fittings to remove debris.
- **5.** Run the smaller brush through the center hole in the cavity to remove debris.
- **6.** Connect the two water lines with the union. Turn on the the instrument with the cover up. The cover must be up to protect the user from hazardous electric shock. Within a few minutes, the instrument will display a COVER/NO WATER message. This is normal. Turn off the instrument before proceeding.
- **7.** Replace the two water lines on the cavity water fittings.
- **8.** Reassemble the two outer sections onto the center of the cavity. Continue with the steps to replace the discharge tube.

Replacing the Spectrometer Window

The spectrometer window can be cleaned by removing it and wiping it with methanol. Do not attempt to clean the window when it is inside the receiver since the O-rings holding it in place will absorb the methanol. It is most convenient to clean or replace the window when the discharge tube is being replaced.

When Does the Window Need to be Cleaned or Replaced?

Generally the window will need cleaning or replacement less often than the discharge tube replacement. If carbon 193 line is greater than 10 times the area of the carbon 165 line, the window may be dirty. For more information about what might cause a window to be replaced, see chapter 7.

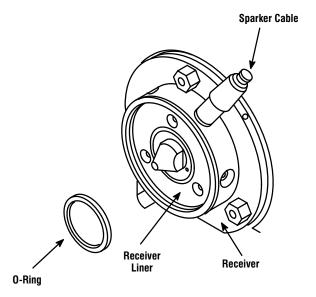
When cleaning the window, use clean methanol and a clean lab tissue. Afterwards, hold it up to the light and make sure there are no fingerprints, or dirt, or discolored areas. If the center is a different color, discard, and replace window. Always clean new windows before installing.

You will need

- 3/8" and two 5/16" open-end wrenches
- 3 mm and 2.5 mm Allen wrenches
- methanol

To Remove the Window

Follow steps 1-7 for replacing the discharge tube.



- **8.** Pull sparker cable off top of receiver. Remove sparker cable fitting and gas line fittings from top of receiver.
- **9.** Unscrew the three screws holding the white receiver liner using the 2.5 mm allen wrench. Then remove the liner and attached cone.
- **10.** Remove window and O-rings from receiver liner. Clean with methanol and lab tissue or replace the window. (The window will become solarized after some period of use.) Clean new windows with methanol before replacing. (Hold clean windows by the edges only.)
- **11.**Remove window cap and sliding seal from inside of receiver. If it won't come out easily by hand, screw the Allen head screws into it and use them to pull it out of the receiver.

chpt 6-12

12. Clean	window	with	methanol.	If any	cloudiness	remains	after	cleaning,	replace	the
windo	w.									

- **13.** Insert brown O-ring in bottom of receiver liner; insert window without getting fingerprints on it; insert second O-ring. Reassemble window cap and sliding seal on receiver liner and cone with allen head screws. Hole of window cap is beveled so larger opening is on the outside.
- **14.** Ensure that O-rings are in place in the 3 holes in the edge of the receiver liner. Press reassembled receiver liner back into receiver, lining up the holes on the edge of the liner with the holes in the receiver. Push it into place with your thumbs. The spring-loaded seal will offer resistance. The assembly should snap into place.
- **15.** Replace sparker fitting and cable into middle hole on side of receiver.
- **16.** Replace helium return and supply lines into receiver. The supply line goes to the far fitting. The return line goes to the fitting in front. (The return line is the one attached to the filter.)
- **17.** Close cavity and reinstall transfer line (Follow steps 16–20 for *Replacing the Discharge Tube.*)

Replacing the Fly Ash Filter

The fly ash filter is the greenish blue plastic part located near the waveguide inside the instrument hood. This filter keeps dirt from getting into the back pressure valve.

When Does the Filter Need to be Changed?

Change the fly ash filter every six months. This filter will need to be changed more often if oxygen analysis is done frequently. This filter *must* be changed any time that water leaks out of the water flow system. A soaked filter can, however, be reused after drying for a day.

You will need

- filter
- tweezers
- 9/16" and 1/2" open end wrenches
- 1. Locate filter inside the instrument hood to the right of the waveguide.

- **2**. Loosen the fittings on both sides of the filter.
- **3**. Replace the filter. Check that the arrow on the filter is in the direction of flow.
- **4**. Retighten the fittings.

chpt 6-14

Keeping the Spectrometer Dry

Always keep at least 2 l/min (ball just off bottom of gauge) of nitrogen flowing in the spectrometer at all times. The flow of nitrogen during use will keep moisture from damaging the diode array and grating inside the spectrometer.

Note—If the instrument will be idle for a length of time, less nitrogen is required to maintain it (100-500 ml/min).

In Case of Difficulty



What's Involved?

- How to observe a spectral line
- Checking tank pressures and gas flows
- How to find gas leaks in the system
- Diagnostic tools
- Common AED problems and remedies
- Error messages

How to Observe a Spectral Line

Many of the procedures in this chapter ask you to observe various elemental lines. This is how to do it.

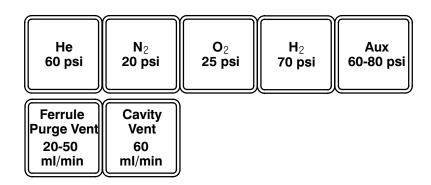
1. From **Data Acquis**, press **AED & DECTECTRS**, then **SIGNAL MONITOR**, and then **MONITOR AED SIG**. The following sets of softkeys will appear:

Carbon & Nitrogen	Carbon & Silicon	Carbon & Hydrogen	Hydrogen 656.279	Oxygen 777.340	Select Line(s)	MORE Lines	EXIT
						*	
		Helium 587.565	Helium 706.520	Fluorine 685.602	Select Line(s)	MORE LINES	EXIT

2. Use the softkeys to select the line you wish to observe.

Checking Tank Pressures and Gas Flows

The procedure for setting gas flows is found in chapter 4. Chapter 2 contains a preflight checklist of items to check before making a run. Tank pressures and measurable flows are reprinted here for your convenience.



How to Find Gas Leaks in the System

Finding gas leaks is a common troubleshooting technique referred to in many of the procedures in this chapter. Follow this procedure when you suspect a gas leak in the system.

- To determine where to look for a leak:
 - 1. Turn off column flow by removing the septum. Watch the nitrogen line. If the nitrogen peak diminishes, the leak is in the GC. If there is no change, the leak is not in the GC or is common to both systems.

 Note: Column temperature must be less than 50°C.
- If the leak is in the GC:
 - **1.** Check the liner and verify that the liner seal is *not* graphite. Graphite liner seals can leak. Make sure the septum is not leaking.
 - **2.** Inspect the flow controller tubing; play with M8 fittings to determine if they change the nitrogen line. Spray an aerosol duster at the fittings while inspecting the carbon line. If there is a leak, the carbon in the Freon will increase the carbon line.
- If the leak is *not* in the GC:
 - 1. Do a gross pressure thumb test: Put thumbs over the **FERRULE PURGE** and **CAVITY VENTS**. This should cause the **CAVITY PRESSURE** gauge to rapidly rise in value. If the value rises slowly and doesn't reach 5 psi, then there is a leak in the AED.
 - **2.** Monitor the background as you turn reagent gases on or off. If it moves up and down, leak check the reagent gases.
 - **3.** Increase the MAKEUP GAS FLOW and monitor the nitrogen 174 line. If it decreases, there is a leak in the AED.

- **4.** Leak check fittings at tanks, gas union, etc. Use a leak hunter or watch the carbon 193 line while squirting butane from a lighter or an aerosol duster at the fitting in question to see if the carbon line increases. (Butane may take 2 or 3 minutes to show up on carbon line.)
- **5.** Leak check around any gas getter in the system.
- **6.** Use the butane test (see step 4) to see if the column is cracked inside the Gerstal ferrule.

Diagnostic Tool for Air Leaks and Contamination

This procedure allows you to use the spectrometer itself to troubleshoot air leaks, find contamination, and check the level of oxygen reagent gas. The next procedure allows you to check for water leaks.

1. From **Data Acquis**, press **AED & DECTECTRS**, then **SIGNAL MONITOR**, and then **MONITOR AED SIG**. The following set of softkeys will appear:

Carbon &	Carbon &	Carbon &	Hydrogen	Oxygen	Select	MORE	EXIT
Nitrogen	Silicon	Hydrogen	656.279	777.340	Line(s)	LINES	EVII

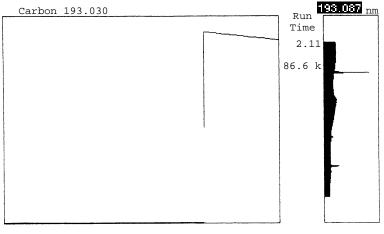
2. At [MONITOR] prompt, type UVTEST and press Return.

The ratio of the carbon 193 line to the carbon 165 line will be printed. If the number is larger than 10.0, there may be a UV transmission problem.

At [MONITOR] prompt, type LEAKS and press Return.

The areas of the nitrogen and hydrogen peaks will be printed. IF the hydrogen line is higher than 500K area counts or the nitrogen line is higher than 25K area counts, there may be a leak. The peak area of nitrogen will be higher if you have not scraped the polyimide off the end of a recently installed column.

3. Press Carbon & Nitrogen



Enter Abbreviation, press a Function Key, or TAB to go to first field.



- **4.** Find area of carbon 193 with the cursor. This peak should be less than 100K area counts and less than one-third the height of the oxygen bands. Use this line when using a butane lighter to leak test fittings. This peak area will be higher with a high stationary phase bleed column or if there are fresh fingerprints on the end of the column or contamination in the tubing.
- **5.** Find area of nitrogen 174 with the cursor. This peak should also be less than 25K area counts and less than one-third the height of the oxygen bands. This peak area will be higher if you have not scraped the polyimide from off the end of the column or if there is an air leak.

Diagnostic Tool for Water Leaks

1. From Data Acquis , press AED & DECTECTRS , then SIGNAL MONITOR , and then MONITOR AED SIG . The following set of softkeys will appear:

Carbon & Carbon & Carbon & Hydrogen Nitrogen Silicon Hydrogen 656.279					
-----------------------------------------------------------------------	--	--	--	--	--

2. At [MONITOR] prompt, type_LEAKS and press Return. The areas of the nitrogen and hydrogen peaks will be printed.

The hydrogen line should be 500K area counts.

3. If this peak is jumping around, look in the plasma window. If the plasma is flashing red, type solv on to turn **on** the solvent vent. If the plasma turns the normal pink color and the peak stops jumping around, then the discharge tube ferrule is leaking. If the plasma is flashing red only when the solvent vent is on, the discharge tube Oring is leaking.

How to Check the Photo Diode Array (PDA)

- 1. From Data Acquis , press AED & DECTECTRS , and then SIGNAL MONITOR .
- **2.** At [MONITOR] prompt, type REALIGN 706,DIODES,REPORT In about 15 minutes the terminal will print a graph showing the response of all 211 diodes. The plot should be clustered near the 100% line as shown below. The overall level of the line will vary. Individual diodes will be good as long as they don't vary more than 5% from their immediate neighbors.

3. If the height of the helium peak reported is near saturation (8300 k), then the 706 line may be too bright to run this test on your instrument. If this is the case, run the report again using the 728 line.

Relative response vs. diode number.

Common AED Problems and Remedies

This chapter contains a list of potential problem symptoms.

- **1**. Locate the symptom you are currently encountering.
- **2**. Follow each suggested action in order.
- **3**. If you encounter a symptom that is not listed, obtain HP service.

Peak tailing on all channels

Probable Causes:

- Makeup or reagent flows improperly set.
- GC inlet.
- Silicon dioxide buildup in discharge tube.

- **1**. Check makeup and reagent gas pressures and flows. If flows are too low, peaks will tail. See chapter 4 for flow setting procedures.
- **2.** Check the GC inlet. Use prepunched septa for the cool on-column injection port. Check the split flow, septum, column, and particularly the liner for proper operation.
- 3. Replace the discharge tube. Look for "white snow" (silicon dioxide) at the end of column or discharge tube. Buildup of silicon dioxide is often caused by column bleed from film thicknesses of $>0.5~\mu m$. Connect thick-film analytical columns to a length of thin-film or no-film transfer column using the union provided in the GC oven. See instructions for this procedure in chapter 4.
- **4**. Obtain HP service.

Peak tailing on certain elements (not carbon)

Probable Causes:

■ Makeup or reagent flows improperly set.

- **1**. Check makeup and reagent gas pressures and flows. If flows are too low, peaks will tail. See chapter 4 for flow setting procedures.
- **2.** Flows for certain elements need to be higher than usual. Set makeup flow to 125–175 ml/min manually or by turbo valve when analyzing for phosphorus, tin, or silicon.
- **3**. Obtain HP service.

Negative peaks on one or more elements poor selectivity

Probable Causes:

- Not enough or improper reagent gas flow.
- Chemical sample too large; detector electronics overloaded.
- Improper background subtraction (SUPAMT) amounts.

- 1. Check that appropriate reagent gases are in use and that flows are correct. See *Optimizing Your AED Technique* to choose reagent gases. If N₂/CH₄ flow is too high, negative peaks will appear. See chapter 4 for flow setting instructions.
- **2**. Check discharge tube for carbonization. If you see black rings in the plasma, the discharge tube is carbonized. If this happens, make sure all flows are set correctly, and then turn O₂ to 80 psi for several minutes. Check the plasma to see if there is carbonization. If no black rings are visible, leave the pressure at this level. If carbonization occurs, turn down the pressure 10 psi at a time until the black rings disappear.
- **3**. To check for an overloaded detector, go to Data Editor screen and enter the name of the appropriate file. Type BACK 0.0000 on the chromatogram that has negative peaks. If the peaks are still negative, then the problem is most likely electronic overload.
- **4.** Run the checkout sample to verify proper operation of the detector. Follow the instructions in *Running an AED Analysis* and use 50–75 ng of sample. Compare your chromatogram with the one in the tutorial.

Negative peaks on one or more elements poor selectivity (cont.)

- **5.** Run the @&CHECK.R sequence several times. Wide variations in SUPAMTs may indicate intermittent leaks or contamination in the system. Use diagnostic tool to check for air leaks and contamination.
- **6**. Run @&UPBACK.R to update SUPAMTs.
- **7**. Obtain HP service.

Poor sensitivity

Probable Causes:

- Chromatographic settings don't match sample.
- Makeup flow too high.
- GC inlet problem.
- Dirty spectrometer window.

- 1. If sensitivity is only poor in the UV region, skip to step 4. If all elements have poor sensitivity, check chromatographic factors: split ratio, injection amount, and makeup flow. If makeup flow is too high, peaks may be lost. See chapter 4 for flow setting procedures.
- **2.** If retention times are long and peaks are tailing, injection port septum may need to be replaced.
- **3**. Run checkout sample to verify proper operation of the detector. Follow the instructions in the tutorial manual. Observe He line intensity.
- **4.** Check that NITROGEN SPECTROMETER PURGE FLOW on front panel is 2 ml/min. If flow is OK, replace the spectrometer window. See chapter 6 for instructions for replacing the window.

Plasma won't light Probable Causes:

- Solvent vent or split ratio settings incorrect.
- Water leak.
- Helium plumbed incorrectly.
- Power fail without battery backup.

Suggested Actions:

- **1**. Check that solvent vent and split ratio are set appropriately for sample. See *Optimizing Your AED Technique*.
- **2.** Make sure helium and nitrogen are plumbed to the correct inlets.
- **3.** Check for water leak around cavity. If nothing is visible, turn off reagent gases and check the color of the plasma. If the plasma is red, there is a water leak in the cavity. Check the discharge tube and its ferrule and O-ring. See chapter 6 for instruction on how to gain access to and replace these parts.
- **4.** If system has been powered off and battery backup has failed to restore system files, plasma will need to be lit from the computer terminal.

To turn on the plasma, from the TOP LEVEL press Data Acquis, then AED & DETECTRS, then SIGNAL MONITOR, and finally Ignite Plasma.

When the plasma is lit, press **EXIT** to return to the main Data Acquisition screen.

5. Obtain HP service.

Signal is too small Probable Causes:

- Makeup flow too high.
- Proper reagent gases not present.
- GC inlet problem.
- Microwave or PDA problem.

- **1**. Check MAKEUP GAS FLOW. Because AED is a concentration detector, too much makeup gas will reduce the peak size.
- **2.** Make sure proper reagent gases are turned on. See *Optimizing Your AED Technique*.
- **3**. Check the split flow, septum, column, and liner for proper operation. Use prepunched septa for the cool on-column injection port.
- **4.** Check helium 706 intensity with the reagents off and solvent vent off. If intensity is much dimmer than before, there could be a microwave problem. Obtain HP service.
- **5**. If there are no peaks, check the PDA. If diodes vary more than 5%, obtain HP service.
- **6**. Obtain HP service.

Signals in the ultraviolet region too small

Probable Causes:

- Oxygen or hydrocarbons present in spectrometer purge gas.
- Dirty spectrometer window.

- **1**. Check for oxygen or hydrocarbons in the spectrometer purge gas (nitrogen) by changing tanks. Oxygen and hydrocarbons will keep the ultraviolet light from being detected by the PDA.
- **2.** Use diagnostic tool to determine if the size of C 193 line is 10 times the size of the C 165 line.
- **3**. Clean or replace the spectrometer window. A dirty window will pass the larger wavelength lights but will severely attenuate the ultraviolet light. See chapter 6 for instructions for this procedure.
- 4. Obtain HP service.

Molecular lines (like oxygen 185) appear to have sections missing

Probable Causes:

■ PDA diodes burned out.

- **1**. Check the PDA. If diodes vary more than 5%, obtain HP service.
- **2**. Obtain HP service.

Excess noise on all channels

Probable Causes:

- Water leak.
- PDA problem.

- 1. Check for water leak around cavity. If nothing is visible, turn off reagent gases and check the color of the plasma. If the plasma is red, there is a water leak. Check the discharge tube ferrule and O-ring. See chapter 6 for instructions on how to gain access and replace these parts.
- 2. Check dark noise. From Data Acquis press

 AED & DECTECTRS and then SIGNAL MONITOR

 At the [MONITOR] prompt, type REALIGN 706.5,NOISE

 The return variable DN0 should be less than 15. If greater than 15, obtain HP service.
- **3**. Check the PDA. If diodes vary more than 5%, obtain HP service.
- **4** Obtain HP service.

Excess noise on some channels

Probable Causes:

- Air leak.
- Contamination in the system.
- Water leak.

- **1**. Use diagnostic tool to check for air leaks and contamination in the system.
- 2. Check for water leak around cavity. If nothing is visible, turn off reagent gases and check the color of the plasma. If the plasma is red, there is a water leak. Check the discharge tube and its ferrule and O-ring. See chapter 6 for instruction on how to gain access and replace these parts.
- **3**. Obtain HP service.

Large nitrogen and/or oxygen peaks

Probable Causes:

■ Air leak.

- **1**. Use diagnostic tool to check for air leaks in the system.
- **2**. Obtain HP service.

COVER/NO WATER Probable Causes: Message

- Top cover is swung open.
- Empty water bath.
- Dirty water filter.
- Blockage in the cavity water path.
- Faulty pump performance.

- **1**. Close top cover.
- **2**. Check water bath level. Fill if necessary. See Chapter 3.
- **3.** Check water filter. Replace element if necessary. See the instructions in chapter 3.
- **4**. Clean the cavity flow path. See Chapter 6.
- **5**. Obtain HP service.

Plasma glows red Probable Causes:

- Hydrogen reagent gas turned on.
- Water leak in system.
- Cracked discharge tube.
- Discharge tube O-ring missing.

- **1**. Ensure that hydrogen reagent gas is turned off.
- **2**. Visually check water lines where they enter in the instrument and at the cavity and fly ash filter for evidence of any leaks. Use diagnostic tool to check for internal leaks.
- **3**. Replace discharge tube. See chapter 6 for instructions.
- 4. Obtain HP service.

Discharge tube shatters in cavity

Probable Causes during Installation:

- Ferrule was overtightened.
- There are glass particles in the cavity from an earlier broken tube.

Probable Causes during Use:

- Weakening of the tube over time by the plasma (a typical tube lasts 3-4 weeks).
- Weakening of the tube due to sample effects.
- Helium supply ran out during a run, extinguishing the plasma. System attempts to relight the plasma and damages the tube.

Suggested Actions:

1. Clean cavity and replace discharge tube. See instructions in chapter 6.

Retention times longer (GC settings unchanged)

Probable Causes:

- Injection port septum.
- Dirty back pressure regulator.

Suggested Actions:

- 1. Check injection port septum. Replace if necessary.
- 2. Check fly ash filter. If filter is all black, ash may have broken through and contaminated back pressure regulator. Check pressure between cavity and fly ash filter. If pressure is above 1.6 psi, replace the back pressure regulator.
- **3**. Obtain HP service.

Note—Users of short, large internal diameter columns require less head pressure to establish normal flow. If the pressure in the cavity is lower than the inlet, you will notice a reverse flow.

- 1. Add a short length of small bore column as an additional restrictor.
- 2. Increase the psi to account for addition restriction.

AED Error Messages

All messages preceded by an asterisk indicate a hardware failure and may require HP service.

Message	Explanation
COVER/NO WATER	Check cover position, water bath level, filter, water temperature. See chapter 3.
High Voltage Off	High voltage is turned off. Check position of hood.
Cavity Temp	Cavity temperature not at set point.
Xfer Line Temp	Transfer line temperature not at set point.
H ₂ O Zone Temp	Water temperature not at set point.
Microwave Delay	Filament heating delay after water system becomes stable.

Message Most Likely Assembly at Fault

*A/D RAM Failed main board

*Bad DRAM Buffer main board

*A/D CPU Failed main board

*A/D Fault main board

*A/D SW Mismatch main board

*No Cooling Fan AC board, or magnetron fan

*Bad Servo CPU main board

*Servo Read Err main board

*Servo Loop Open spectrometer

*No Slit Motor connectors, wiring harness, motor

*No Array Motor connectors, wiring harness, motor

*No Slit Limit spectrometer

No Array Top spectrometer

No Array Bottom spectrometer

Message Most Likely Assembly at Fault

No Cavity Sensor cable not attached, sensor open

No Transfer Line cable not attached, sensor open

*Bad Cavity Rdg main board

*Bad Xfer Ln Rdg main board

*Bad H₂O Rdg main board, connector

*Cavity Overheat AC board

*Xfer Ln Overheat AC board

*H₂O Overheat AC board

*Cav Not Heating AC board

*Xfer Not Heating AC board

*H₂O Not Heating AC board

*Triac Interrupt main board, AC board

Specific Environmental Specs and Site Requirements

Space and Weight Requirements

- AED Dimensions L x W x H: 2.8 m x 0.78 m x 1.4 m (9' x 2.5' x 4.5')
- AED Weight: 95 kg (210 lbs)
- Water pump requires 0.63 m x 0.63 m x 0.63 m (2' x 2' x 2') of floor space

Power Requirements

- GC-AED System: 120 VAC at 30 amps/220 VAC at 20 amps (8 outlets)
- AED: 120 VAC at 8 amps/220 VAC at 5 amps
- Gas Chromatograph: 120 VAC at 20 amps/220 VAC at 10 amps (dedicated circuit)
- Water pump and auxiliary pump: 100 V at 50 or 60 Hz/120 V, 15 amps at 60 Hz 220-240 V at 50 or 60 Hz (dedicated circuit)

Ventilation Requirements

■ Fume hood nearby to vent detector effluents from front panel.

Temperature and Humidity Requirements

■ Room temperature: 10°C to 55°C

■ Humidity: 0-95% noncondensing

Recommended Consumables

Gas Requirements

- Helium 99.9999% pure
- Hydrogen 99.997% pure
- Oxygen 99.997% pure
- Nitrogen 99.99% pure
- Nitrogen/Methane 99.999% pure

Water

■ Distilled

Parts

- discharge tube kit
- GC chamber O-ring
- optical chamber O-ring
- window O-ring
- ferrules
- fly ash filters
- spectrometer window
- water filter element

See the HP Analytical Direct Chromatography Users Catalog (HP publication number (43) 5091-2653EUS) for part numbers and prices of AED consumables.

Table of Spectral Lines

В	F	N	S
249.773	685.602	174.272	180.734
182.587 (same as S)	690.246	746.831	182.036
		744.230	182.626
Br(II)	Н		
481.670	656.285	0	S(II)
470.485	486.133	777.539	545.388
478.550	434.047	777.194	
	410.174	777.417	Se
С			
193.090	He	P	203.985
247.856	471.338	253.561	196.090
495.724 (2nd)	706.519	255.325	٥.
400.724 (2114)	700.010	253.561	Si
Cd	Hg	253.399	251.611
	•	255.490	
228.802	253.652	178.287	Sn
326.105	184.950	178.768	
	i	255.490	283.999
CI(II)	-	213.547	286.332
479.454	206.238	213.618	
481.006	206.163	214.914	
481.946	Lab		
542.325	I(II)	Pb	
725.665	466.648	217.558	
837.597	516.120	283.307	
	533.822	405.782	
	562.569		

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