Agilent 5975T LTM GC/MSD

Operation Manual

Agilent Technologies

Notices

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Safety Notices

CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

About This Manual

This manual contains information for operating the Agilent 5975T LTM GC/MSD system.

1 "Introduction"

Chapter 1 describes general information about the 5975T LTM GC/MSD, including a hardware description, general safety warnings, and hydrogen safety information.

2 "Installing LTM Columns"

Chapter 2 shows you how to prepare a LTM column module for use with the MSD, install it in the instrument, and connect it to the inlet and the GC/MSD interface.

3 "Operating in Electron Impact (EI) Mode"

Chapter 3 describes basic tasks such as setting temperatures, monitoring pressures, tuning, venting, and pumpdown.

4 "General Maintenance"

Chapter 4 describes routine maintenance tasks performed during normal operation of the 5975T, such as baking out the column and changing the source.

Where to Find Information

In addition to this document, Agilent provides several learning products that document how to install, operate, maintain, and troubleshoot the Agilent 5975T LTM GC/MSD system.

Before operating your instrument, be sure to read the safety and regulatory information included on the Agilent GC and GC/MS Hardware User Information & Utilities DVD. The most common safety hazards when working on the instrument are:

- Burns caused by touching heated areas on or in the instrument
- Release of pressurized gas containing hazardous chemical compounds caused by opening inlets
- Glass cuts or puncture wounds caused by sharp capillary column ends
- Use of hydrogen as a GC carrier gas

Online User Documentation

Now your Agilent instrument documentation is in one place, at your fingertips.

The Agilent GC and GC/MS Hardware User Information & Utilities DVD that ships with your instrument provides an extensive collection of online help, videos, and books for current Agilent gas chromatographs, mass selective detectors, and GC samplers. Included are localized versions of the information you need most, such as:

- Getting Familiar documentation
- Safety and Regulatory guide
- Site Preparation information
- Installation information
- Operating guides
- Maintenance information
- Troubleshooting details

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Contents

1 Introduction

This chapter provides an overview of the instrument hardware, hydrogen safety and other general safety precautions for the Agilent Technologies 5975T LTM GC/MSD.

Abbreviations Used

The abbreviations in Table 1 are used in discussing this product. They are collected here for convenience.

Table 1 Abbreviations

Abbreviation	Definition
AC	Alternating current
ALS	Automatic liquid sampler
BFB	Bromofluorobenzene (calibrant)
DC	Direct current
DFTPP	Decafluorotriphenylphosphine (calibrant)
EI	Electron impact ionization
EM	Electron multiplier (detector)
EMV	Electron multiplier voltage
EPC	Electronic pneumatic control
eV	Electron volt
GC	Gas chromatograph
HED	High-energy dynode (refers to detector and its power supply)
id	Inside diameter
LAN	Local Area Network
LCP	Local control panel
LTM	Low thermal mass
m/z	Mass to charge ratio
MSD	Mass Selective Detector
OFN	Octafluoronaphthalene (calibrant)
PFHT	2,4,6-tris(perfluoroheptyl)-1,3,5-triazine (calibrant)
PFTBA	Perfluorotributylamine (calibrant)

Table 1 Abbreviations (continued)

Abbreviation	Definition
Quad	Quadrupole mass filter
RF	Radio frequency
RFPA	Radio frequency power amplifier
Torr	Unit of pressure, 1 mm Hg
Turbo	Turbomolecular (pump)

The GC/MSD

The GC/MSD features:

- LTM GC column with rapid heating and cooling capabilities
- Capillary guard column to minimize contamination to the LTM
- Local Control Panel (LCP) for local monitoring
- Choice of foreline pump - rotary vane (wet), scroll (dry), or diaphragm (dry)
- Heated electron-ionization ion source
- Heated hyperbolic quadrupole mass filter
- High-energy dynode (HED) electron multiplier detector
- Heated GC/MSD interface
- ChemStation control for operating the GC/MSD

Physical description

The 5975T LTM GC/MSD is a rectangular box, approximately 41 cm high, 60 cm wide, and 54 cm deep. The weight is 46.5 kg for the mainframe. The attached foreline (roughing) pump weighs an additional 15 kg (standard pump) for wet, and 4.5 kg for dry.

The basic components of the instrument are: the frame/cover assemblies, the local control panel, the vacuum system, the EPC, the GC/MSD interface, the inlet, the LTM column module, the electronics, and the analyzer.

Local control panel

The local control panel displays the status of the instrument, displays error messages, and allows setting and display of some instrument parameters for the Agilent 5975T LTM GC/MSD. These parameters are normally controlled using the Agilent ChemStation.

Vacuum gauge

The MSD may be equipped with an optional external vacuum gauge. Operation of the gauge controller is described in this manual.

Table 2 5975T LTM GC/MSD models and features

Feature	
High vacuum pump	Standard turbo
Optimal He column flow mL/min	1.2
Maximum recommended column gas flow mL/min	2
Maximum gas flow, mL/min*	2.4

* Expect degradation of spectral performance and sensitivity.

GC/MSD Hardware Description

Figure 1 is an overview of a 5975T LTM GC/MSD system.

Figure 1 5975T LTM GC/MSD system

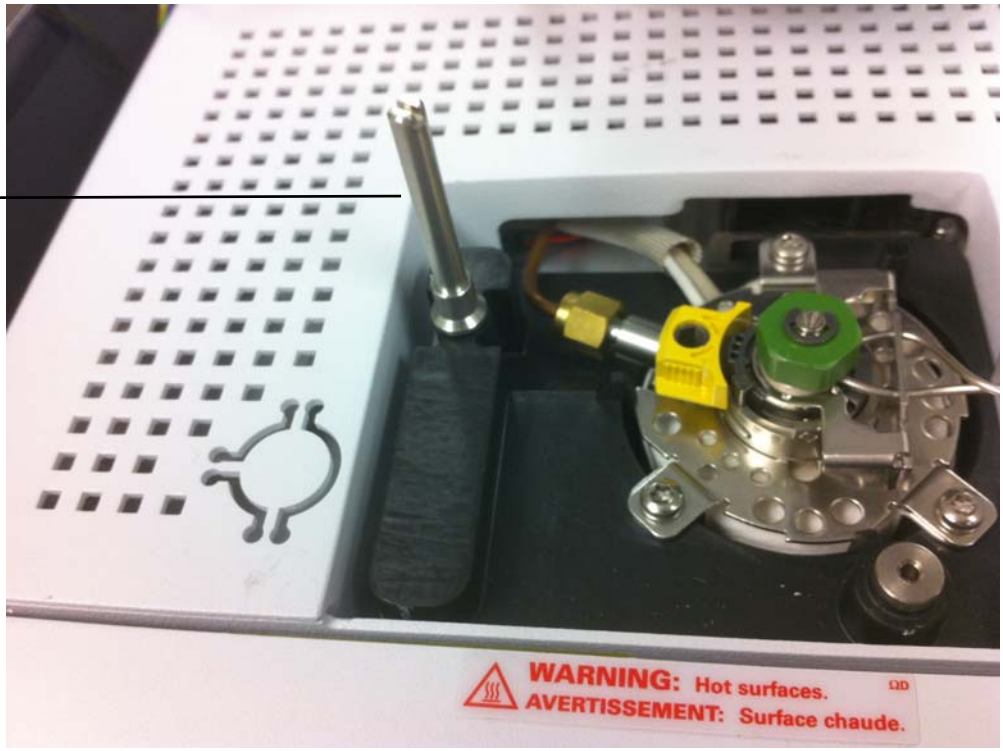


Figure 2 Top View of 5975T with ALS parking post

Important Safety Warnings

There are several important safety notices to always keep in mind when using the GC/MSD.

Many internal parts of the GC/MSD carry dangerous voltages

If the GC/MSD is connected to a power source, even if the power switch is off, potentially dangerous voltages exist on:

- The wiring between the GC/MSD power cord and the AC power supply, the AC power supply itself, and the wiring from the AC power supply to the power switch.

With the power switch on, potentially dangerous voltages also exist on:

- All electronics boards in the instrument.
- The internal wires and cables connected to these boards.
- The wires for any heater (oven, detector, inlet, or valve box).

WARNING **All these parts are shielded by covers. With the covers in place, it should be difficult to accidentally make contact with dangerous voltages. Unless specifically instructed to, never remove a cover unless the detector, inlet, or oven are turned off.**

WARNING **If the power cord insulation is frayed or worn, the cord must be replaced. Contact your Agilent service representative.**

Electrostatic discharge is a threat to GC/MSD electronics

The printed circuit boards in the GC/MSD can be damaged by electrostatic discharge. Do not touch any of the boards unless it is absolutely necessary. If you must handle them, wear a grounded wrist strap and take other antistatic precautions. Wear a grounded wrist strap any time you must remove the MSD right side cover.

Many parts are dangerously hot

Many parts of the GC/MSD operate at temperatures high enough to cause serious burns. These parts include but are not limited to:

- The inlet
- The guard column enclosure and its contents
- The guard column nuts attaching the guard columns to the inlet, LTM column, and MSD.
- The analyzer
- The foreline pump

Always cool these areas of the system to room temperature before working on them. If you must perform maintenance on hot parts, use a wrench and wear gloves. Whenever possible, cool the part of the instrument that you will be maintaining before you begin working on it.

WARNING **The insulation around the inlet is made of refractory ceramic fibers. To avoid inhaling fiber particles, we recommend the following safety procedures: ventilate your work area; wear long sleeves, gloves, safety glasses, and a disposable dust/mist respirator; dispose of insulation in a sealed plastic bag; wash your hands with mild soap and cold water after handling the insulation.**

The oil pan under the standard foreline pump can be a fire hazard

Oily rags, paper towels, and similar absorbents in the oil pan could ignite and damage the pump and other parts of the GC/MSD.

WARNING **Combustible materials (or flammable/non-flammable wicking material) placed under, over, or around the foreline (roughing) pump constitutes a fire hazard. Keep the pan clean, but do not leave absorbent material such as paper towels in it.**

Hydrogen Safety

WARNING **The use of hydrogen (H_2) as a carrier gas is potentially dangerous.**

WARNING **When using H_2 as the carrier gas or fuel gas, be aware that hydrogen gas can flow into the guard column enclosure and create an explosion hazard. Therefore, be sure that the supply is turned off until all connections are made and ensure that the inlet, MSD, and column fittings are either connected to a column or capped at all times when hydrogen gas is supplied to the instrument.**

Hydrogen is flammable. Leaks, when confined in an enclosed space, may create a fire or explosion hazard. In any application using hydrogen, leak test all connections, lines, and valves before operating the instrument. Always turn off the hydrogen supply at its source before working on the instrument.

Hydrogen is a commonly used GC carrier gas. Hydrogen is potentially explosive and has other dangerous characteristics.

- Hydrogen is combustible over a wide range of concentrations. At atmospheric pressure, hydrogen is combustible at concentrations from 4% to 74.2% by volume.
- Hydrogen has the highest burning velocity of any gas.
- Hydrogen has a very low ignition energy.
- Hydrogen that is allowed to expand rapidly from high pressure can self-ignite.
- Hydrogen burns with a nonluminous flame which can be invisible under bright light.

Dangers unique to GC/MSD operation

Hydrogen presents a number of dangers. Some are general, others are unique to GC or GC/MSD operation. Dangers include, but are not limited to:

- Combustion of leaking hydrogen.
- Combustion due to rapid expansion of hydrogen from a high-pressure cylinder.
- Accumulation of hydrogen in the guard column enclosure and subsequent combustion.
- Accumulation of hydrogen in the MSD and subsequent combustion.

Hydrogen accumulation in a GC/MSD

WARNING **The GC/MSD cannot detect leaks in inlet and/or detector gas streams. For this reason, it is vital that column fittings should always be either connected to a column or have a cap or plug installed.**

All users should be aware of the mechanisms by which hydrogen can accumulate (Table 3) and know what precautions to take if they know or suspect that hydrogen has accumulated. Note that these mechanisms apply to *all* mass spectrometers, including the GC/MSD.

Table 3 Hydrogen accumulation mechanisms

Mechanism	Results
Mass spectrometer turned off	A mass spectrometer can be shut down deliberately. It can also be shut down accidentally by an internal or external failure. In an external failure, the mass spectrometer shutdown does not shut off the flow of carrier gas. As a result, hydrogen may slowly accumulate in the mass spectrometer. However, in an external power failure, the EPC will be turned off and the flow of gas stopped.

Table 3 Hydrogen accumulation mechanisms (continued)

Mechanism	Results
Mass spectrometer manual shutoff valves closed	Some mass spectrometers are equipped with manual foreline pump shutoff valves. In these instruments, the operator can close the shutoff valves. Closing the shutoff valves does not shut off the flow of carrier gas. As a result, hydrogen may slowly accumulate in the mass spectrometer.

WARNING **Once hydrogen has accumulated in a mass spectrometer, extreme caution must be used when removing it. Incorrect startup of a mass spectrometer filled with hydrogen can cause an explosion.**

WARNING **After a power failure, the mass spectrometer may start up and begin the pumpdown process by itself. This does not guarantee that all hydrogen has been removed from the system or that the explosion hazard has been removed.**

Precautions

Take the following precautions when operating a GC/MSD system with hydrogen carrier gas.

Equipment precaution

You MUST make sure the front side-plate thumbscrew is fastened finger-tight. Do not overtighten the thumbscrew; it can cause air leaks.

WARNING **Failure to secure your MSD as described above greatly increases the chance of personal injury in the event of an explosion.**

General laboratory precautions

- Avoid leaks in the carrier gas lines. Use leak-checking equipment to periodically check for hydrogen leaks.
- Eliminate from your laboratory as many ignition sources as possible (open flames, devices that can spark, sources of static electricity, etc.).
- Do not allow hydrogen from a high pressure cylinder to vent directly to atmosphere (danger of self-ignition).
- Use a hydrogen generator instead of bottled hydrogen.

Operating precautions

- Turn off the hydrogen at its source every time you shut down the instrument.
- Turn off the hydrogen at its source every time you vent the MSD (do not heat the capillary column without carrier gas flow).
- Turn off the hydrogen at its source every time shutoff valves in an MSD are closed (do not heat the capillary column without carrier gas flow).
- Turn off the hydrogen at its source if a power failure occurs.
- If a power failure occurs while the GC/MSD system is unattended, even if the system has restarted by itself:
  1. Immediately turn off the hydrogen at its source.
  2. Turn off the instrument and allow it to cool for 1 hour.
  3. Eliminate **all** potential sources of ignition in the room.
  4. Open the vacuum manifold of the MSD to atmosphere.
  5. Wait at least 10 minutes to allow any hydrogen to dissipate.
  6. Start up the instrument as normal.

When using hydrogen gas, check the system for leaks to prevent possible fire and explosion hazards based on local Environmental Health and Safety (EHS) requirements. Always check for leaks after changing a tank or servicing the gas lines. Always make sure the vent line is vented into a fume hood.

Safety and Regulatory Certifications

The 5975T LTM GC/MSD conforms to the following safety standards:

- Canadian Standards Association (CSA): CAN/CSA-C222 No. 61010-1
- International Electrotechnical Commission (IEC): 61010–1
- EuroNorm (EN): 61010–1

The 5975T LTM GC/MSD conforms to the following regulations on Electromagnetic Compatibility (EMC) and Radio Frequency Interference (RFI):

- CISPR 11/EN 55011: Group 1, Class A
- IEC/EN 61326
- AUS/NZ

This ISM device complies with Canadian ICES-001. Cet appareil ISM est conforme a la norme NMB–001 du Canada.

The 5975T LTM GC/MSD is designed and manufactured under a quality system registered to ISO 9001.

Information

The Agilent Technologies 5975T LTM GC/MSD meets the following IEC (International Electro-technical Commission) classifications: Equipment Class I, Laboratory Equipment, Installation Category II, Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and is designed for use in stationary or mobile labs. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired. Whenever the safety protection of the MSD has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

Refer servicing to qualified service personnel. Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard.

Symbols

Warnings in the manual or on the instrument must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions violates safety standards of design and the intended use of the instrument. Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

See accompanying instructions for more information.

Indicates a hot surface.

Indicates hazardous voltages.

Indicates earth (ground) terminal.

Indicates potential explosion hazard.

Indicates radioactivity hazard.

Indicates electrostatic discharge hazard.

Indicates that you must not discard this electrical/electronic product in domestic household waste.

Electromagnetic compatibility

This device complies with the requirements of CISPR 11. Operation is subject to the following two conditions:

- This device may not cause harmful interference.
- This device must accept any interference received, including interference that may cause undesired operation.

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

1. Relocate the radio or antenna.
2. Move the device away from the radio or television.
3. Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
4. Make sure that all peripheral devices are also certified.
5. Make sure that appropriate cables are used to connect the device to peripheral equipment.
6. Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.
7. Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

Sound emission declaration

Sound pressure

Sound pressure Lp <70 dB according to EN 27779:1991.

Schalldruckpegel

Schalldruckpegel LP <70 dB am nach EN 27779:1991.

Cleaning/Recycling the Product

To clean the unit, disconnect the power and wipe down with a damp, lint-free cloth. For recycling, contact your local Agilent sales office.

Liquid Spillage

Do not spill liquids on the instrument.

Moving or Storing the GC/MSD

The best way to keep your GC/MSD functioning properly is to keep it pumped down and hot, with carrier gas flow. If you plan to move or store your GC/MSD, a few additional precautions are required. The GC/MSD must remain upright at all times; this requires special caution when moving. The GC/MSD should not be left vented to atmosphere for long periods. Consult the Hardware Installation Manual for Field Transportable Units for information on how to secure, pack, transport and routinely set up the unit.

1 Introduction

2
Installing LTM Columns

Before you can operate your GC/MSD system, you must select, install, and condition a GC column. This chapter will show you how to install and condition a column.

Overview

The LTM section of the 5975T LTM GC/MSD consists of a heated LTM column module and two guard columns that connect the LTM to the split/splitless inlet and MSD transfer line. These guard columns help limit contamination in the LTM, and the MSD.

The sample flows through the inlet, into the first guard column (inlet guard column) into the LTM, then through the second guard column (MSD guard column) into the MSD transfer line to the MSD. See Figure 3.

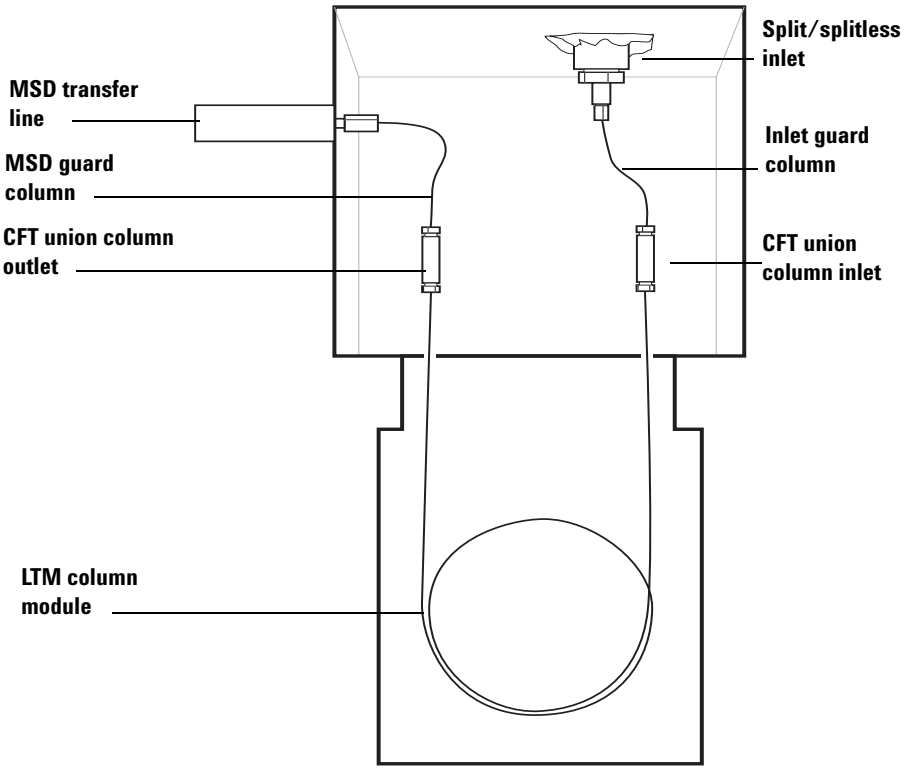


Figure 3 5975T LTM GC/MSD column diagram

Columns

Many types of LTM columns can be used with the MSD but there are some restrictions.

During tuning or data acquisition the rate of column flow into the MSD should not exceed the maximum recommended flow. Therefore, there are limits to column flow. Exceeding recommended flow will result in degradation of mass spectral and sensitivity performance.

Remember that column flows vary greatly with temperature. See "To Measure Column Flow Linear Velocity" for instructions on how to measure actual flow in your column. Use the Flow Calculation software and Table 4 to determine whether a given column will give acceptable flow with realistic head pressure.

Table 4 Gas flows

Feature	
High vacuum pump	Standard turbo
Optimal gas flow, mL/min*	1.2
Maximum recommended gas flow, mL/min	2
Maximum gas flow, mL/min†	2.4
Maximum column id	0.32 mm (30 m)

* Total gas flow into the MSD = column flow

† Expect degradation of spectral performance and sensitivity.

Conditioning columns

Conditioning a column before it is connected to the GC/MSD interface is essential.

A small portion of the capillary column stationary phase is often carried away by the carrier gas. This is called column bleed. Column bleed deposits traces of the stationary phase in the MSD ion source. This decreases MSD sensitivity and makes cleaning the ion source necessary.

Column bleed is most common in new or poorly crosslinked columns. It is much worse if there are traces of oxygen in the carrier gas when the column is heated. To minimize column bleed, all capillary columns should be conditioned ***before*** they are installed in the GC/MSD interface.

Conditioning ferrules

Heating ferrules to their maximum expected operating temperature a few times before they are installed can reduce chemical bleed from the ferrules.

Tips and hints

- The column installation procedures for the 5975T LTM GC/MSDs is different from that for previous MSDs. Using the procedure from another instrument may ***not*** work and may damage the column or the MSD.
- You can remove old ferrules from column nuts with an ordinary push pin.
- Always use carrier gas that is at least 99.9995% pure.
- Because of thermal expansion, new ferrules may loosen after heating and cooling a few times. Check for tightness after two or three heating cycles.
- Always wear clean gloves when handling columns, especially the end that will be inserted into the GC/MSD interface.

WARNING **If you are using hydrogen as a carrier gas, do not start carrier gas flow until the column is installed in the MSD and the MSD has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the MSD and an explosion may occur.** **See "Hydrogen Safety".**

WARNING **Always wear safety glasses when handling capillary columns. Use care to avoid puncturing your skin with the end of the column.**

To Install an Inlet Guard Column in the Split/Splitless Inlet

This procedure is for attaching the inlet guard column to the inlet. See Figure 3.

Materials needed

- Capillary guard column
- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Ferrules (Vespel)
  - 0.40-mm id, for 0.25-mm id columns (5181-3323)
  - 0.5-mm id, for 0.32-mm id columns (5062-3514)
- Ferrules (SilTite)
  - 0.3-mm id, for < 0.25 mm id column (5188-5361)
  - 0.4-mm id, for < 0.32 mm id column (5188-5362)
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Inlet column nut (5020-8292)
- Magnifying loupe
- Septum (may be old, used inlet septum)
- Metric ruler
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)

Procedure

1 Load your maintenance method for cooling down the column enclosure. See "Column maintenance method" on page 120.

WARNING **Be careful! The guard column heated enclosure or internal accessories may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands or allow the parts to cool before beginning the work.**

WARNING **Always wear safety glasses when handling capillary columns. Use care to avoid puncturing your skin with the end of the column.**

CAUTION Wear clean, lint-free gloves to prevent contamination of the parts.

2 Slide a septum, column nut, and conditioned ferrule onto the free end of the column (Figure 4). The tapered end of the ferrule should point away from the column nut.

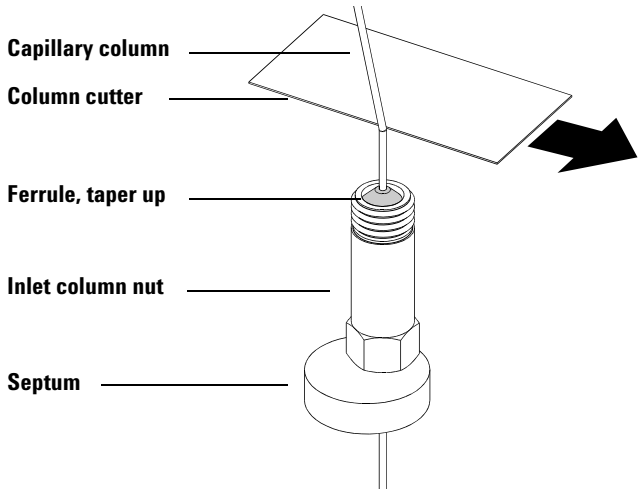


Figure 4 Preparing a capillary column for installation

3 Use the sharp edge of the column cutter to score the column 2 cm from the end. See Figure 5.

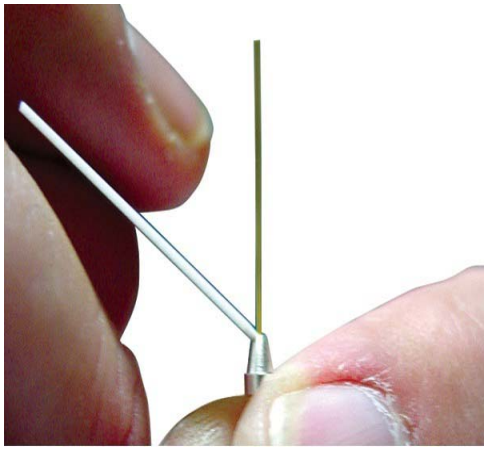


Figure 5 Column cutter edge

4 Break off the end of the column. Hold the column against the column cutter with your thumb. Break the column against the edge of the column cutter.

5 Inspect the end for jagged edges or burrs. If the break is not clean and even, repeat steps 3 and 4.

6 Wipe the outside of the free end of the column with a lint-free cloth moistened with methanol.

7 Position the column so it extends 4 to 6 mm past the end of the ferrule (Figure 6).

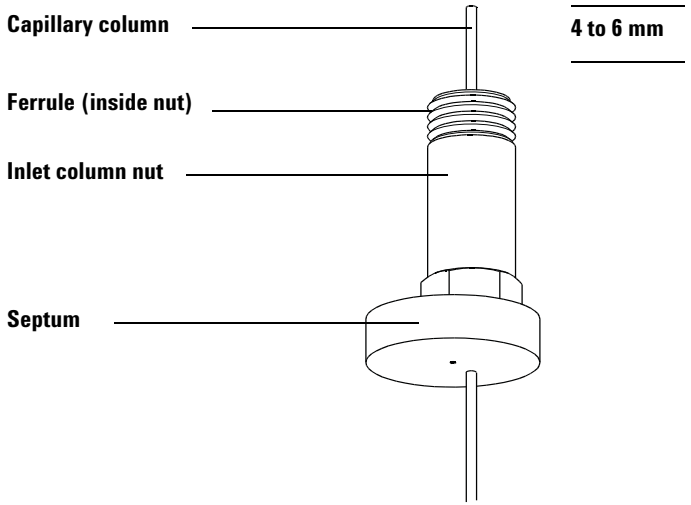


Figure 6 Installing a capillary column for a split/splitless inlet

8 Slide the septum to place the nut and ferrule in the correct position.

9 Insert the column in the inlet, and finger-tighten the nut.

10 Tighten the column nut an additional 1/4 to 1/2 turn. The column should not slide with a gentle tug.

11 Carefully wind the guard column clockwise around the three column brackets. See Figure 7.

12 Check the Guard Column Enclosure to be sure that the column does not touch the heated walls.

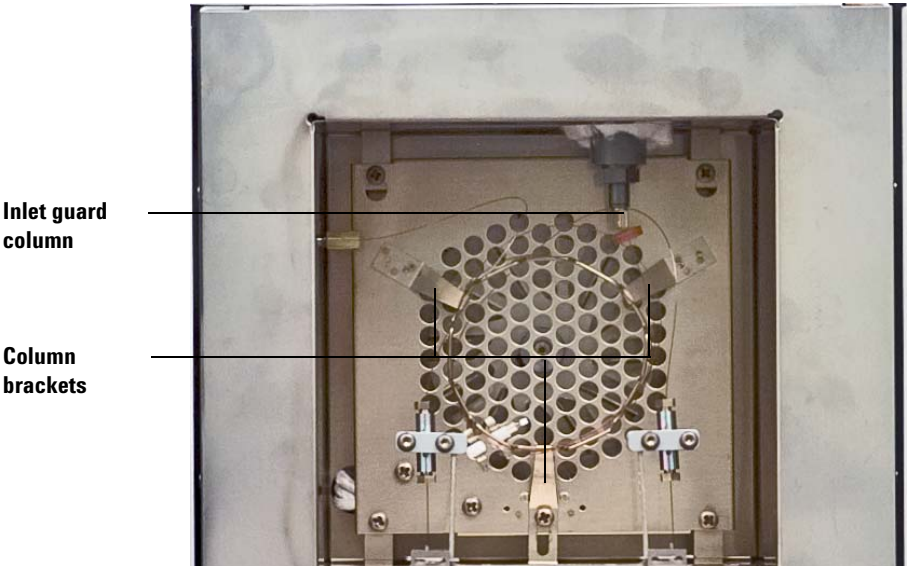


Figure 7 Column brackets

To Install an MSD Guard Column in the GC/MSD Interface

This procedure is for installing an MSD guard column from the LTM to the GC/MSD interface. See Figure 3.

Materials needed

- Guard column
- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Ferrules (Vespel)
  - 0.40-mm id, for 0.25-mm id columns (5181-3323)
  - 0.5-mm id, for 0.32-mm id columns (5062-3514)
- Ferrules (SilTite)
  - 0.3-mm id, for < 0.25 mm id column (5188-5361)
  - 0.4-mm id, for < 0.32 mm id column (5188-5362)
- Flashlight
- Hand lens (magnifying loupe)
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Interface column nut (SilTite: G2855-20555, Vespel: 05988-20066)
- Safety glasses
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)

CAUTION Note that the column installation procedure for the 5975T LTM GC/MSDs is different from that for most previous MSDs. Using the procedure from another instrument may result in poor sensitivity and possible damage to the MSD.

Procedure

1 Load your maintenance method for cooling down the column enclosure. See "Column maintenance method" on page 120.

WARNING **Be careful! The guard column heated enclosure or internal accessories may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands or allow the parts to cool before beginning the work.**

WARNING **Always wear safety glasses when handling capillary columns. Use care to avoid puncturing your skin with the end of the column.**

CAUTION Wear clean, lint-free gloves to prevent contamination of the parts.

2 Vent the MSD (page 97) and open the analyzer chamber (page 99). Be sure you can see the end of the GC/MSD interface.

3 Slide an interface nut and conditioned ferrule onto the free end of the guard column. The tapered end of the ferrule must point towards the nut.

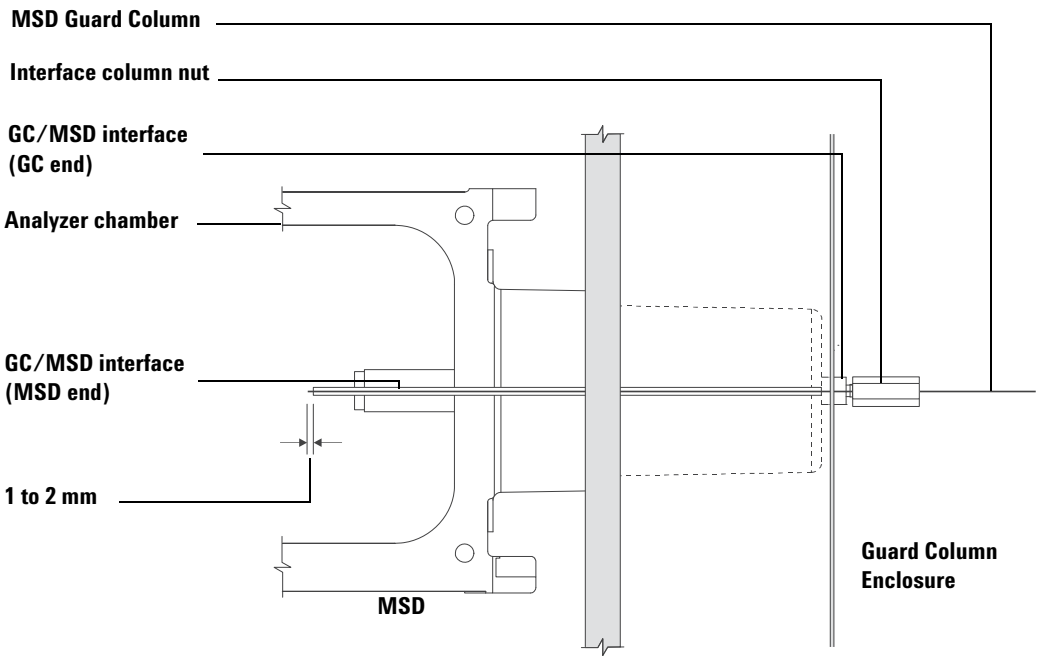


Figure 8 Installing a guard column in the GC/MSD interface

4 Slide the column into the GC/MSD interface (Figure 8) until you can pull it out through the analyzer chamber.

5 Break 1 cm off the end of the column. Do not let any column fragments fall into the analyzer chamber. They could damage the high vacuum pump.

6 Clean the outside of the free end of the column with a lint-free cloth moistened with methanol.

7 Adjust the column so it projects 1 to 2 mm past the end of the interface.

Use the flashlight and hand lens if necessary to see the end of the column inside the analyzer chamber. Do ***not*** use your finger to feel for the column end.

8 Hand-tighten the nut. Make sure the position of the column does not change as you tighten the nut.

9 Tighten the nut 1/4 to 1/2 turn. Check the tightness after one or two heat cycles.

10 Carefully wind the guard column clockwise around the three column brackets. See Figure 7.

11 Check the Guard Column Enclosure to be sure that the column does not touch the enclosure walls.

To Install the LTM Module Assembly in the Instrument

The LTM module comes attached to the fan and preswaged with CFT unions for guard column connections. After you remove the module from the packaging, it is ready for installation.

WARNING **Be careful! The guard column heated enclosure or internal accessories may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands or allow the parts to cool before beginning the work.**

WARNING **Disconnect the power cord to the instrument from the building power supply. Never power the instrument when changing the column module. Dangerous voltages exist inside the instrument when connected to the building power supply.**

1. Open the LTM door on the right side of the GC/MSD.
2. Route the transfer line and column module cables (total of three cables and connections per module) into the column enclosure and through the rectangular slot on the top left of the electronics housing. See Figure 9.
3. Push the cable bundle into the bushing at the top of the slot to anchor the bundle. See Figure 9.

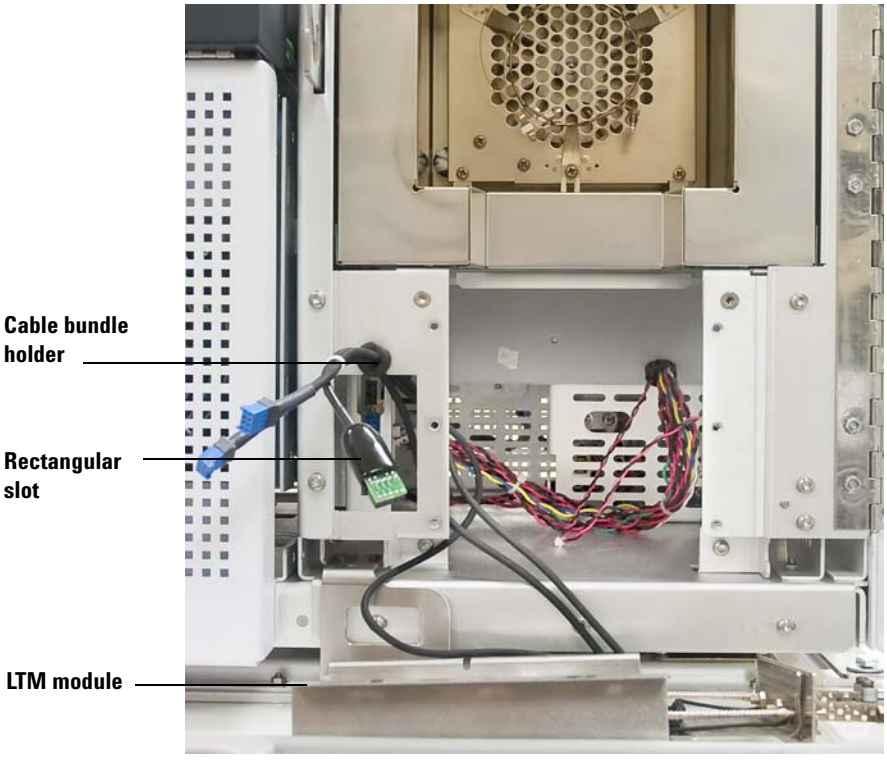


Figure 9 Cable Routing

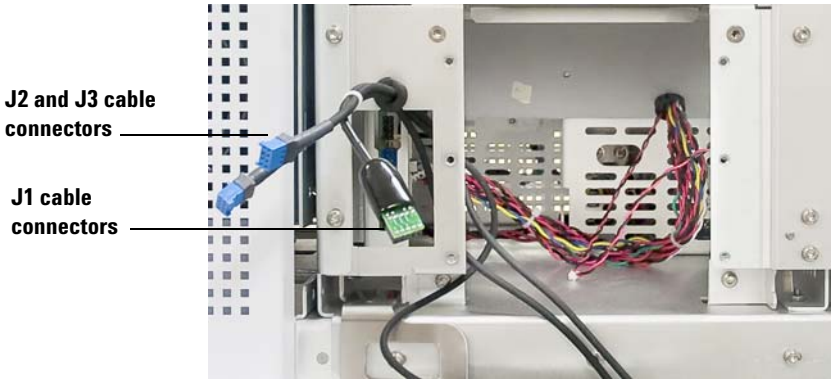


Figure 10 LTM cable connections

4 Hold the LTM module with the fan facing toward you, and align the unit against the opening in the front of the instrument.

5 Slide the LTM module into position and seat it securely in the instrument.

6 Secure the module with the four captive screws around the outside of the fan plate. See Figure 11.

   If there is resistance in engaging the captive screws with the module assembly, do not force the screw. Instead, loosen and back the screw out of its fixture, and examine it for insulation debris. The screw should be wiped clean with a paper towel, and lubricated with either graphite powder or an anti-seize compound such as Loctite Heavy Duty Antiseize, P/N 51609 (Loctite Corporation, Rocky Hill, CT) or Sprayon Dry Graphite Lube, P/N S00204 (Sherwin Williams, Solon, OH).

7 Connect the green column cable connector to the small circuit board in the uppermost connector, J1 (a 10-pin connector), on the left side of the card. The circuit board side of the connector faces outward and this is the only way the connector will join with the connector on the board.

8 Connect the two blue transfer line cable connectors, which are interchangeable, to the lower 8-pin connector, J2 and J3. These also are keyed and will only go onto the board one way. When removing the transfer line connectors from the board, remove them by gripping the connector and not by pulling on the cable because there are several fine wires in the connector. See Figure 10.

9 Connect the fan cable to the bottom connector.

CAUTION When securing the module do not over tighten!

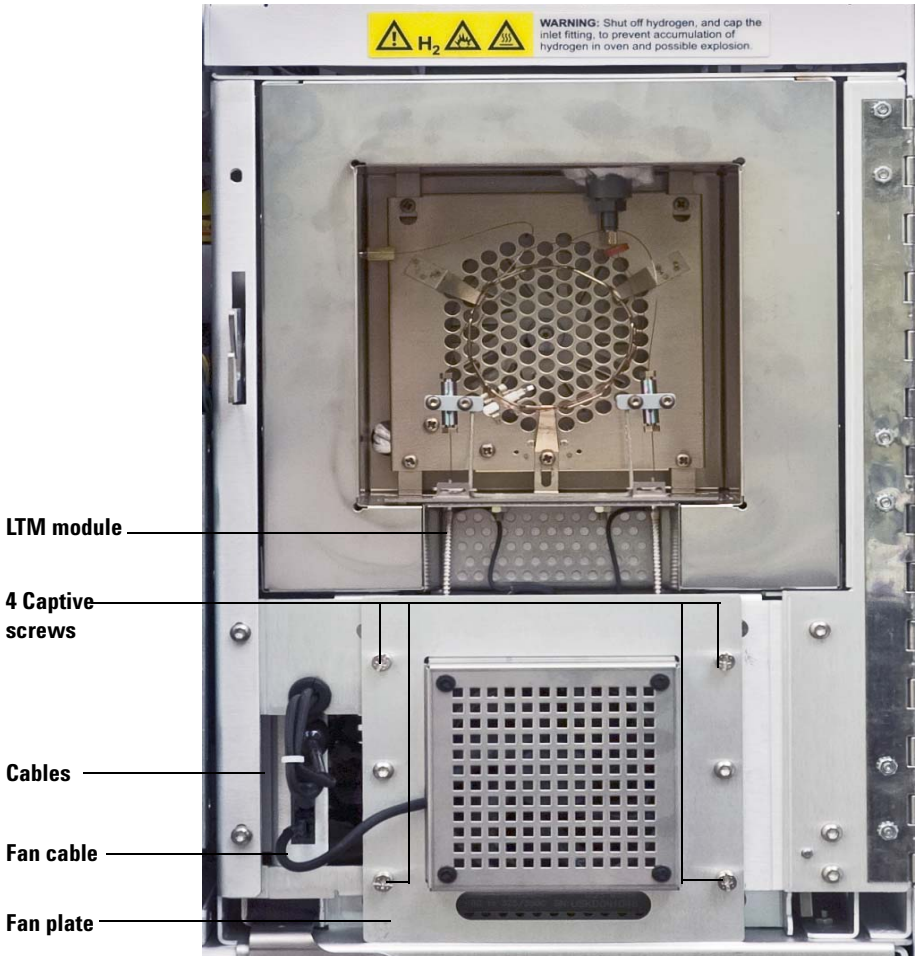


Figure 11 LTM module installed on the 5975T

10 From the ChemStation, edit the column configuration for the LTM module and change the carrier gas type configured if switching to a different carrier gas supply.

Now you are ready to condition the LTM column module, if necessary. See "To Condition the LTM Column Module" on page 56.

To attach the guard columns from the MSD and inlet to the module's CFT union connections see "To Install an Inlet Guard Column in the Split/Splitless Inlet" on page 33, "To Install an MSD Guard Column in the GC/MSD Interface" on page 38, and "To Attach the Guard Columns to the LTM CFT Unions" on page 52.

To Remove the LTM Module Assembly from the Instrument

This procedure is used to remove an LTM column module for replacement or for instrument storage.

Materials needed

- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Internal column nut (G2855-20530)
- Ferrules (SilTite)
  - 0.3-mm id, for < 0.25 mm id column (5188-5361)
  - 0.4-mm id, for < 0.32 mm id column (5188-5362)
- Wire for plug (G2855-60593)
- Union (G3182-60580)
- Safety glasses
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)

Procedure

1 Load your maintenance method for cooling down the column enclosure. See "Column maintenance method" on page 120.

2 When the instrument state changes to **Ready**, turn off the inlet and the LTM column from the ChemStation.

Keep the analyzer under vacuum unless you are performing analyzer maintenance.

WARNING **Be careful! The guard column heated enclosure or internal accessories may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands or allow the parts to cool before beginning the work.**

3 Open the LTM door on the right side of the GC/MSD.

CAUTION Wear clean, lint-free gloves to prevent contamination of the parts.

4 Remove the MSD guard column from the LTM column outlet union and immediately cap the CFT fitting on the guard column to maintain vacuum if required. Use an interface column nut and blanking ferrule attached to a union at one end with the guard column attached at the other end.

5 Remove the inlet guard column from the LTM column inlet union.

6 Turn off the instrument and disconnect the power cord from the building power supply.

WARNING **Disconnect the power cord to the instrument from the building power supply. Never power the instrument when changing the column module. Dangerous voltages exist inside the instrument when connected to the building power supply.**

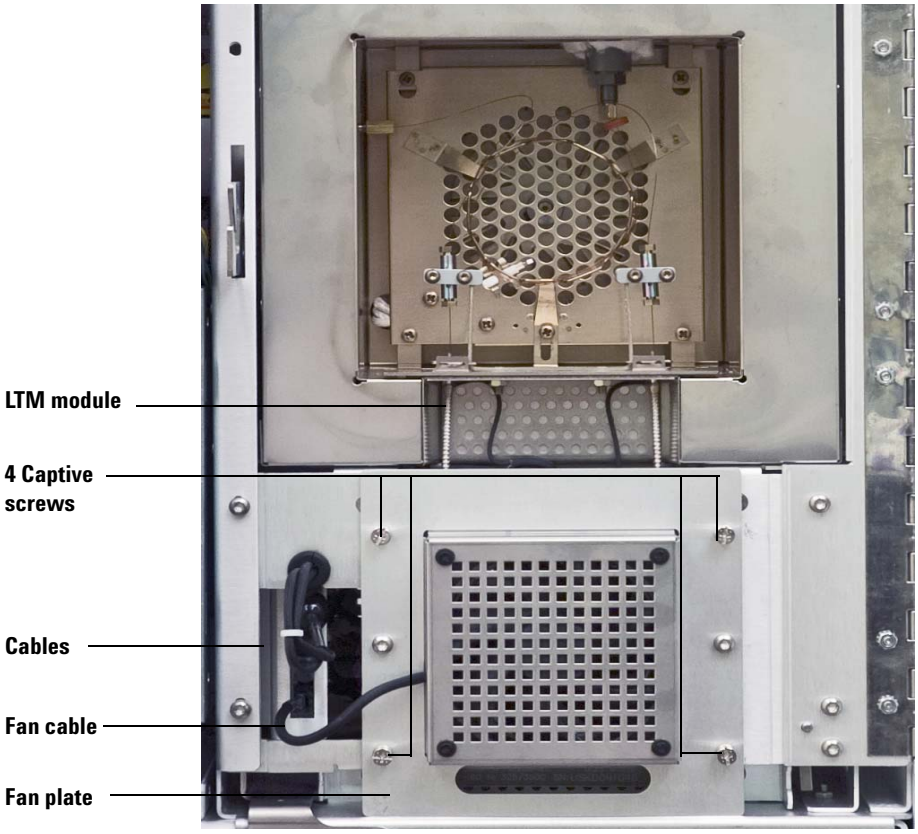


Figure 12 LTM module installed on the 5975T

7 Loosen the four captive screws around the outside of the fan plate. See Figure 12.

8 Remove the LTM column module by pulling it toward you out of the instrument.

9 Place the LTM column module on the bench space in the front of the instrument while you disconnect the cables. See Figure 13.

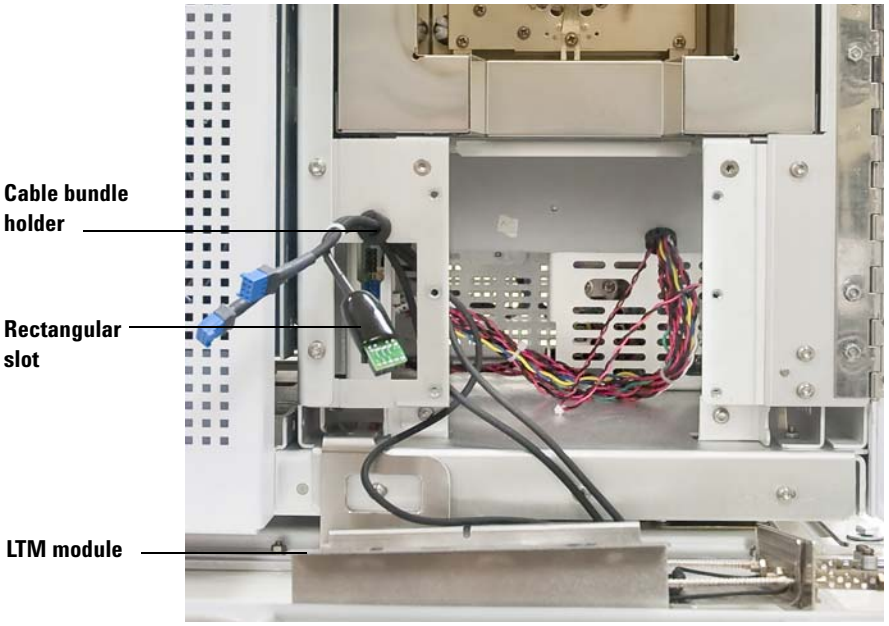


Figure 13 Disconnecting the cables

10 Remove the green column cable connector from the small circuit board connector, J1.

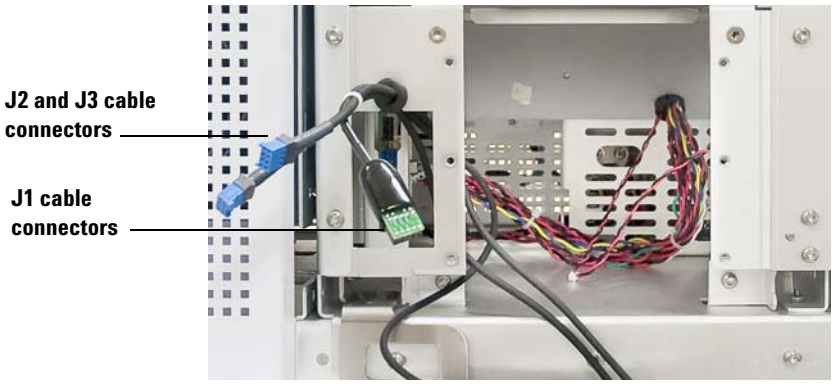


Figure 14 LTM cable connections

11 Remove the two blue transfer line cable connectors from the connectors, J2 and J3 them by gripping the connector. Do not pull on the cable because there are several fine wires in the connector that can be broken. See Figure 14.

12 Remove the fan cable from the bottom connector.

13 Slide the bushing securing the cable bundle out of the attachment point on the frame to finish removing the column module assembly. See Figure 13.

14 Cap the column ends and place the column module assembly in its storage container.

To Attach the Guard Columns to the LTM CFT Unions

This procedure is used to attach the inlet and MSD guard columns to the CFT Ultimate Unions on the LTM. See Figure 3.

Materials needed

- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Ferrules (SilTite)
  - 0.3-mm id, for < 0.25 mm id column (5188-5361)
  - 0.4-mm id, for < 0.32 mm id column (5188-5362)
- Hand lens (magnifying loupe)
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Internal column nut (G2855-20530)
- Safety glasses
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)

Procedure

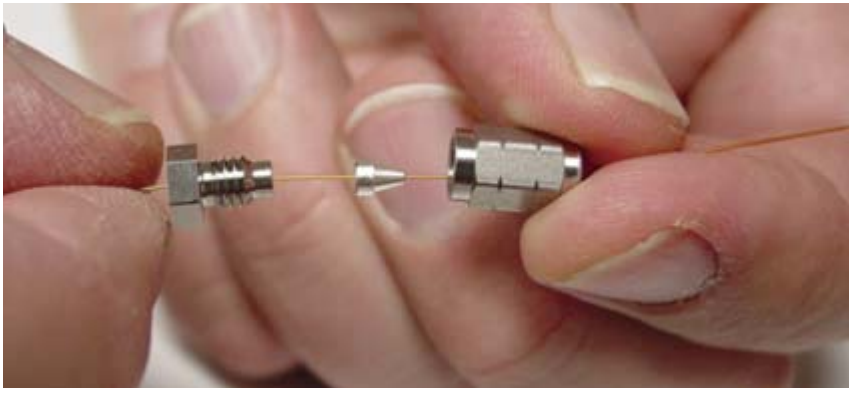
1 Load your maintenance method for cooling down the column enclosure. See "Column maintenance method" on page 120.

WARNING **Be careful! The guard column heated enclosure or internal accessories may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands or allow the parts to cool before beginning the work.**

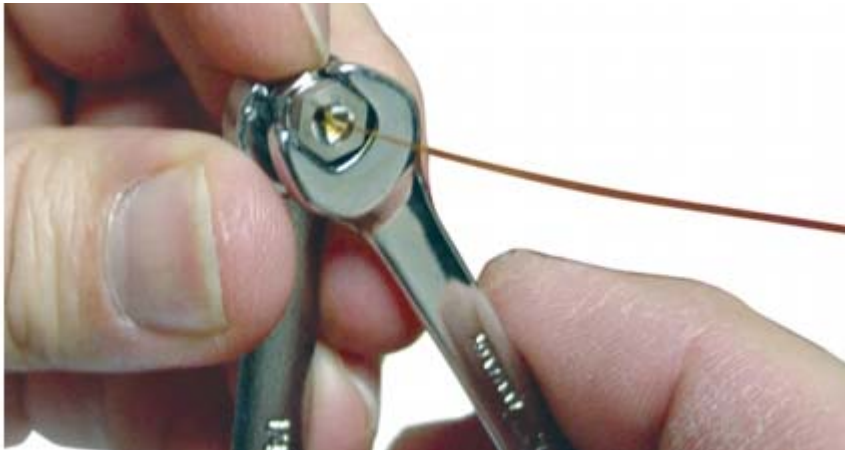
CAUTION Wear clean, lint-free gloves to prevent contamination of the parts.

2 Pass the column end through the internal nut and SilTite ferrule leaving approximately 1 cm of fused silica column protruding beyond the ferrule.

Thread the swaging nut or swaging tool onto the internal nut with the column protruding.



3 Using two wrenches against each other, tighten the two nuts together a little at a time, occasionally checking to see if the ferrule is gripping the column. When the ferrule starts to grip, tighten one of the nuts an additional 45 to 60 degrees (one flat).



4 Remove the swaging nut or swaging tool.

5 Use the sharp edge of the wafer column cutter to trim the column at the small end of the ferrule. See Figure 5. Leave approximately 0.3 mm of column extending beyond the ferrule. The column cannot extend more than 0.5 mm from the end of the ferrule. Check the end of the column with a magnifier. The end of the column does not need to be perfectly square, but cracks should not extend under the ferrule.

6 Insert the assembled ferrule and nut into the CFT Union attached to the LTM column assembly. Tighten with a wrench by 15 to 20 degrees of rotation.

7 Tighten the two screws on the two LTM brackets to secure the CFT fittings.

8 If needed, install the free end of the guard column into the inlet or GC/MSD transfer line. See "To Install an Inlet Guard Column in the Split/Splitless Inlet" on page 33 or "To Install an MSD Guard Column in the GC/MSD Interface" on page 38.

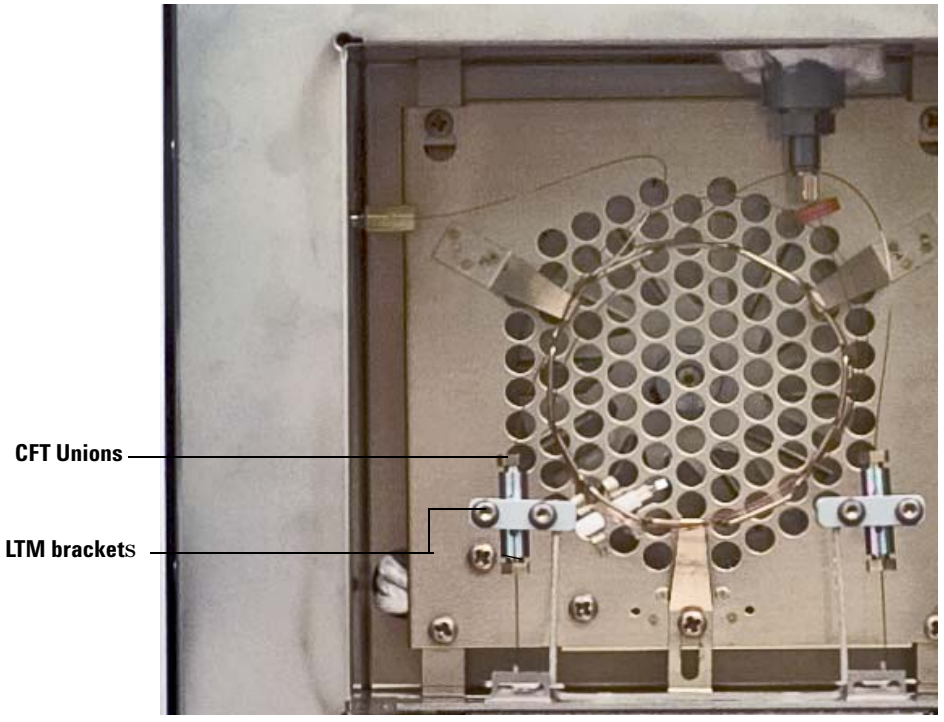


Figure 15 Guard columns attached

To Condition the LTM Column Module

This procedure is used to prepare a new LTM column module for use on the 5975T GC/MSD.

Materials needed

- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Ferrules (SilTite)
  - 0.3-mm id, for < 0.25 mm id column (5188-5361)
  - 0.4-mm id, for < 0.32 mm id column (5188-5362)
- Hand lens (magnifying loupe)
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Internal column nut (G2855-20530)
- Unions (G3182-60580)
- Safety glasses
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)

WARNING **Do not condition your LTM column module with hydrogen. Hydrogen accumulation in the guard column enclosure can result in an explosion. If you plan to use hydrogen as your carrier gas, first condition the column with ultrapure (99.999% or better) inert gas such as helium, nitrogen, or argon.**

CAUTION Wear clean, lint-free gloves to prevent contamination of the parts.

Procedure

1 If necessary, remove the existing LTM column module assembly. See "To Remove the LTM Module Assembly from the Instrument" on page 47. This leaves the instrument in a powered off state.

2 Install the new LTM column module that requires conditioning. See "To Install the LTM Module Assembly in the Instrument" on page 42.

3 If necessary, install a new liner and septum in the inlet before powering on the instrument.

4 Power on the instrument. The MSD transfer line should be capped off. If the analyzer was vented, pump down the instrument. See "Pumping Down" on page 73.

5 Connect a new guard column to the inlet. See "To Install an Inlet Guard Column in the Split/Splitless Inlet" on page 33.

6 Connect this guard column to the LTM column module inlet union. See "To Attach the Guard Columns to the LTM CFT Unions" on page 52.

7 From the ChemStation, edit the column configuration for this LTM module and change the carrier gas type configured if switching to a different carrier gas supply.

8 Turn on the inlet, set its mode to splitless, and set the column flow to 30 cm/s.

9 Check for leaks.

CAUTION Do not heat the column without a flow of carrier gas. You will damage the column.

10 Allow the carrier gas to flow through the column for 5 minutes without heating the LTM column or guard column enclosure.

11 Close the LTM module door.

12 Set the guard column enclosure temperature to 10°C below the maximum LTM column temperature.

13 Ramp the LTM column temperature at 5 °C/minute to 10 °C above your highest analytical temperature.

14 Once the LTM column temperature exceeds 80 °C, inject 5 µL methanol into the inlet. Repeat two more times at 5-minute intervals. This helps remove any contamination from the column before it is installed into the GC/MSD interface.

CAUTION Never exceed the maximum column temperature.

15 Hold the temperature of 10 °C above your highest analytical temperature for 3 hours while allowing a flow rate of 30 cm/s through the column.

WARNING **Be careful! The guard column heated enclosure or internal accessories may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands or allow the parts to cool before beginning the work.**

16 Cool down the enclosure and attach the MSD guard column to the LTM column module outlet union.

17 Check for leaks.

18 If your method requires hydrogen carrier gas, disconnect the current carrier gas supply from the instrument and attach the hydrogen supply. Verify that the analyzer is under vacuum and the vacuum pumps are working before opening the hydrogen supply line. From the ChemStation, change the configured carrier gas to hydrogen.

19 With a column flow of 30 cm/s, wait for 5 to 10 minutes before increasing the LTM column temperature to a low standby temperature.

See also

For more information about installing a capillary column, refer to the application note *Optimizing Splitless Injections on Your GC for High Performance MS Analysis*, publication number 5988-9944EN.

3
Operating in Electron Impact (EI) Mode

CAUTION The software and firmware are revised periodically. If the steps in these procedures do not match your MSD ChemStation software, refer to the manuals and online help supplied with the software for more information.

Operating the GC/MSD from the Data System

The software performs tasks such as pumping down, monitoring pressures, setting temperatures, tuning, and preparing to vent. These tasks are described in this chapter. Data acquisition and data analysis are described in the manuals and online help supplied with the MSD ChemStation software.

Operating the GC/MSD from the LCP

The local control panel (LCP) shows the status of the GC/MSD or initiates a task on the MSD without accessing the Agilent GC/MSD ChemStation.

The GC/MSD ChemStation may be located anywhere on the site local area network (LAN), so the GC/MSD ChemStation might not be near the instrument itself. And because the LCP communicates with the GC/MSD ChemStation via the LAN, you can access GC/MSD ChemStation software functions, such as tuning and starting a run, right from the MSD.

NOTE Only certain functions are available from the LCP; the GC/MSD ChemStation is the full-featured controller for most instrument control operations.

Modes of operation

The LCP has two modes of operation: Status and Menu.

Status mode requires no interaction and simply displays the current status of the MSD instrument or its various communication connections. If you select [**Menu**], then [**No/Cancel**], you will be returned to the Status mode.

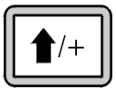
Menu mode allows you to query various aspects of the GC/MSD and to initiate some actions like running a method or sequence or preparing to vent the system.

To access a particular menu option:

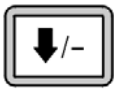
Press [**Menu**] until the desired menu appears.

Press [**Item**] until the desired menu item appears.

Use one or more of the following keys as appropriate to respond to prompts or select options:



Use [**Up**] to increase the displayed value or to scroll up (such as in a message list).



Use [**Down**] to decrease the displayed value or to scroll down (such as in a message list).

Use [**Yes/Select**] to accept the current value.

Use [**No/Cancel**] to return to the Status mode.

After you make your selection, or if you cycle through all available menus, the display automatically returns to Status mode.

Pressing [**Menu**], then [**No/Cancel**], will always display the Status mode.

Pressing [**No/Cancel**] twice will always return to the Status mode.

LCP Status Messages

The following messages may be displayed on the LCP to inform you of the status of the system. If the LCP is currently in Menu mode, cycle through the menus to return to Status mode.

NOTE No messages will be displayed if an online instrument session is not currently running on the GC/MSD ChemStation.

ChemStation Loading <timestamp>

The Agilent MSD Productivity ChemStation software is starting up.

Executing <type>tune

A tuning procedure is in progress (type = QuickTune or Autotune).

Instrument Available <timestamp>

The Agilent MSD Productivity ChemStation software is not running.

Loading Method <method name>

Method parameters are being sent to the system.

Loading MSD Firmware

The MSD's firmware is being initialized.

The following messages alternately appear on the LCP if the MSD does *NOT* complete its bootup sequence properly:

Server not Found
Check LAN Connection

Seeking Server
Bootp Query xxx

These messages indicate that the MSD has not received its unique IP address. If the messages persist after you have logged onto your account on the GC/MSD ChemStation, consult the Troubleshooting section of the Software Installation manual.

Loading OS

The operating system of the instrument controller is being initialized.

<method> Complete <timestamp>

The run and subsequent data processing are done. The same message appears even if the run was terminated prematurely.

Method Loaded <method name>

Method parameters were sent to the system.

MS locked by <computer name>

MS parameters can only be changed from the GC/MSD ChemStation.

Press Sideplate

A reminder during startup to press the MSD sideplate to ensure an adequate vacuum seal.

Run: <method> Acquiring <datafile>

A run is in progress; data is being acquired to the designated data file.

System status messages displayed during startup

The following messages are displayed on the LCP display during startup:

- **Loading OS**
- **Loading MSD Firmware**

Continue to press the sideplate of the MSD until the **MSD Ready** message appears. This helps the instrument to pump down more quickly.

LCP Menus

To access a particular menu option, press [**Menu**] until the desired menu appears, then press [**Item**] until the desired menu item appears. Table 5 through Table 10 list the menus and selections.

NOTE Many menu items, especially on the ChemStation, MS Parameters, and Maintenance menus, have no effect when the instrument is acquiring data.

Table 5 ChemStation menu

Action	Description
Run Method	Displays the current method name and starts an analysis.
Run Sequence	Displays the current sequence and starts a sequence.
Run Current Tune	Displays the current tune file and starts an autotune.
# of Messages	Displays the number of messages and the text of the most recent message. Use the arrow keys to scroll through previous messages (up to 20).
Release ChemStation	Disassociates the GC/MSD ChemStation from the MSD.
Connection Status	Displays the LAN connection status for the MSD. Remote = connected to GC/MSD ChemStation online session Local = not connected to GC/MSD ChemStation online session
Name of Instrument	Displays the name of the instrument if connected to GC/MSD ChemStation online session. The name of the instrument is the name assigned by the GC/MSD ChemStation Configuration dialogue.

Table 6 Maintenance menu

Action	Description
Prepare to vent	Reminds you to shut down the GC then prepares the instrument for venting when **[Yes/Select]** is pressed.
Pumpdown	Initiates a pumpdown sequence.

Table 7 MS Parameters menu

Action	Description
Turbo Pump Speed	Displays the turbo pump speed.
MSD Fault Status	Reports a summary fault status code (number) in 'dec' (decimal) and 'hex' (hexadecimal) format covering all possible fault combinations.
Ion Source Temp, °C	Displays and sets the ion source temperature.
Quadrupole Temp, °C	Displays and sets the quadrupole temperature.

NOTE MS parameters cannot be set from the LCP while an online GC/MSD ChemStation session is connected to the MSD.

Table 8 Network menu

Action	Description
MSD IP via keyboard	Displays the IP address for the MSD.
Gateway IP Address	Displays the gateway IP address for the MSD.
Subnet Mask	Displays the subnet mask for the MSD.
ChemStation IP	Displays the IP address for the GC/MSD ChemStation.
GC IP Address	Displays the IP address for the GC.
Ping gateway	Checks communication with the gateway.
Ping ChemStation	Checks communication with the GC/MSD ChemStation.
Ping GC	Checks communication with the GC.
MS Controller MAC	Displays the MAC address of the SmartCard in the MSD.

Table 9 Version menu

Action	Description
Control firmware	Displays the MSD firmware version.
Operating system	Displays the GC/MSD ChemStation operating system version.
Front panel	Displays the version of the LCP.
Log amplifier	Displays version information.
Sideboard	Displays the sideboard type.
Mainboard	Displays the mainboard type.
Serial number	Is assigned to the MSD by GC/MSD ChemStation Configuration dialogue.

Table 10 Controller menu

Action	Description
Reboot controller	Starts the LAN/MS control card.
Test LCP?	Initiates a diagnostic test of the two-line display.
Test HTTP link to GC/MSD ChemStation?	Checks the status of the HTTP server.

To access a particular menu item in the parameters listed in Tables 11 and 12, press [**Menu**] until the desired menu appears, then press [**Item**] once. The LCP automatically scrolls through the parameters one at a time. Press [**Item**] again to stop at the desired parameter.

Table 11 GC Device Status menu

Action	Description
GC Inlet Heater	Displays the status of the GC inlet heater (Ready/Not Ready)
GC-MSD Interface	Displays the status of the GC-MSD interface temperature.
GC Conn. Zone Heater	Displays the status of the guard column enclosure heater.
GC Column Heater	Displays the status of the LTM heater.

Table 11 GC Device Status menu (continued)

Action	Description
GC Inlet Flow	Displays the status of the inlet gas flow.
GC Inlet Pressure	Displays the status of the inlet pressure controller.
GC Inlet Septum Purge	Displays the status of the inlet septum purge.
GC-ChemStation Conn	Displays the status of the ChemStation software.
GC-APG Remote	Checks the status of the HTTP server.

Table 12 GC Parameter Values menu

Action	Description
GC Inlet T	Displays the inlet temperature.
GC Column T	Displays the LTM temperature.
GC Conn. Zone Temp	Displays the temperature of the guard column enclosure.
GC-MSD Interface T	Displays the temperature of the GC-MSD interface.
GC Inlet Flow	Displays the inlet gas flowrate.

The following GC network parameters are accessed by hitting the [**Menu**] button until you reach the desired parameter.

Table 13 GC Network Parameters

Action	Description
GC IP Address	Displays the IP address for the GC.
GC Gateway IP Address	Displays the Gateway IP address for the GC.
GC Subnet Mask	Displays the subnet mask for the GC.
Reboot LTM GC Now?	Allows you to reboot the LTM GC.
GC MAC Address	Displays the MAC address for the GC.
GC ChemStation IP	Displays the IP address for the GC ChemStation.

The GC/MSD Interface

The GC/MSD interface (Figure 16) is a heated conduit into the MSD for the capillary column. It is bolted onto the right side of the analyzer chamber, with an O-ring seal. It has a protective cover which should be left in place.

One end of the GC/MSD interface passes through the side of the gas chromatograph and extends into the guard column enclosure. This end is threaded to allow connection of the column with a nut and ferrule. The other end of the interface fits into the ion source. The last 1 to 2 millimeters of the capillary column extend past the end of the guide tube and into the ionization chamber.

The GC/MSD interface is heated by an electric cartridge heater. The interface temperature can be set from the MSD ChemStation. A sensor (thermocouple) in the interface monitors the temperature.

The GC/MSD interface should be operated in the 250 ° to 350 °C range. Subject to that restriction, the interface temperature should be slightly higher than the maximum guard column enclosure temperature, but ***never*** higher than the maximum guard column temperature.

See Also

"To Install an MSD Guard Column in the GC/MSD Interface" .

WARNING **The GC/MSD interface operates at high temperatures. If you touch it when it is hot, it will burn you.**

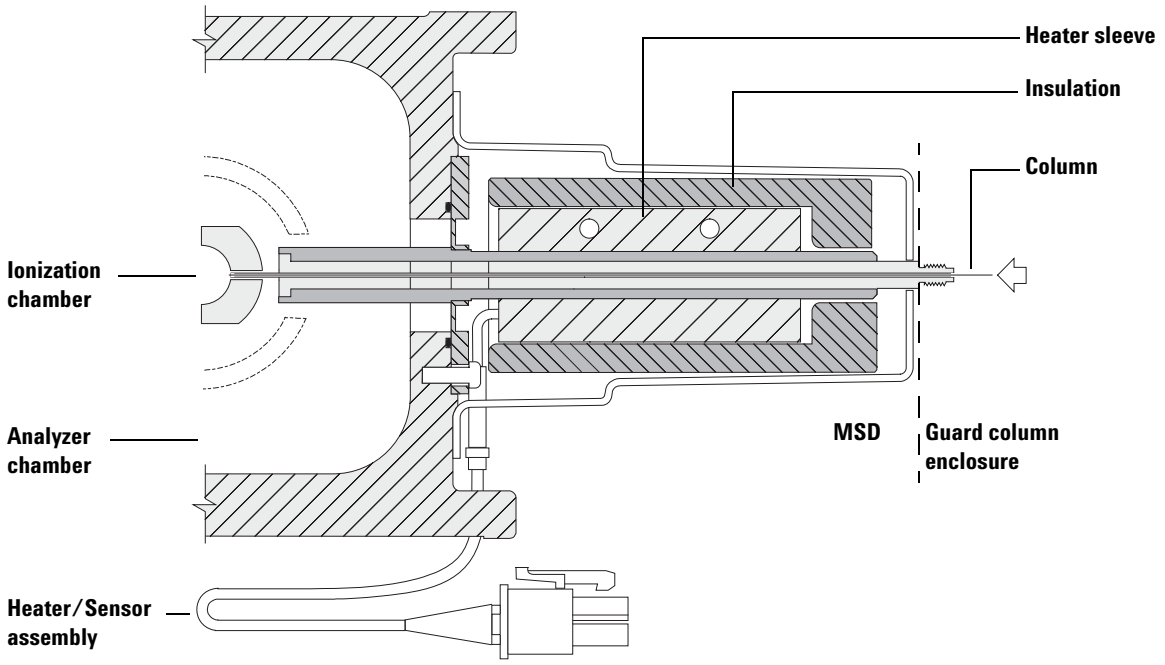


Figure 16 The GC/MSD interface

Before You Turn On the GC/MSD

Verify the following ***before*** you turn on or attempt to operate the GC/MSD.

- The vent valve must be closed (the knob turned all the way clockwise).
- All other vacuum seals and fittings must be in place and fastened correctly. (The the front side plate screw should not be tightened, unless hazardous carrier or reagent gasses are being used.
- The instrument is connected to a grounded power source.
- A conditioned LTM column module and a guard column are installed in the GC inlet and in the GC/MSD interface.
- The instrument is on, but the heated zones for the GC/MSD interface, the GC inlet, LTM column module, and guard column enclosure are off.
- Carrier gas of at least 99.9995% purity is plumbed to the inlet with the recommended traps.
- If hydrogen is used as carrier gas, carrier gas flow must be off and the front sideplate thumbscrew must be loosely fastened.
- If hydrogen is being used as a carrier gas the optional micro-ion vacuum gauge must be turned off.
- The foreline pump exhaust is properly vented.

WARNING **The exhaust from the foreline pump contains solvents and the chemicals you are analyzing. If using the standard foreline pump, it also contains traces of pump oil. If you are using toxic solvents or analyzing toxic chemicals, remove the oil trap (standard pump) and install a hose (10-mm id) to take the foreline pump exhaust outside or to a fume (exhaust) hood. If using toxic chemicals you should also attach the split vent and purge vent outlets to this exhaust system. Be sure to comply with local regulations. The oil trap supplied with the standard pump stops only pump oil. It does not trap or filter out toxic chemicals.**

WARNING **If you are using hydrogen as a carrier gas, do not start carrier gas flow until the MSD has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the instrument and an explosion may occur. Read "Hydrogen Safety" before operating the instrument with hydrogen carrier gas.**

WARNING **If you are using hydrogen as a carrier gas, do not turn on the Micro-Ion vacuum gauge if there is any possibility that hydrogen has accumulated in the analyzer chamber. Read "Hydrogen Safety" before operating the MSD with hydrogen carrier gas. Pump down for 25 minutes before you turn on the vacuum gauge.**

Pumping Down

The data system or local control panel helps you pump down the MSD. The process is mostly automated. Once you close the vent valve and turn on the main power switch (while pressing on the sideplate), the MSD pumps down by itself. The data system software monitors and displays system status during pumpdown. When the pressure is low enough, the program turns on the ion source and mass filter heaters and prompts you to turn on the GC/MSD interface heater. The MSD will shut down if it cannot pump down correctly.

Using the menus or MS monitors, the data system can display the motor speed for turbo pump MSDs (percent spin speed).

The LCP can also display this data.

WARNING **If you are using hydrogen as a carrier gas, do not turn on the Micro-Ion vacuum gauge if there is any possibility that hydrogen has accumulated in the analyzer chamber. Read "Hydrogen Safety" before operating the MSD with hydrogen carrier gas. Pump down for 25 minutes before you turn on the vacuumgauge.**

Controlling Temperatures

MSD temperatures are controlled through the data system. The MSD has independent heaters and temperature sensors for the ion source and quadrupole mass filter. You can adjust the setpoints and view these temperatures from the data system or from the local control panel.

The GC/MSD interface temperature can be set and monitored from the data system.

Controlling Column Flow

Carrier gas flow is controlled by head pressure in the GC inlet. For a given head pressure, column flow will decrease as the LTM column temperature increases. With electronic pneumatic control (EPC) and the column mode set to **Constant Flow**, the same column flow is maintained regardless of temperature.

The MSD can be used to measure actual column flow. You inject a ***small*** amount of air or other unretained chemical and time how long it takes to reach the MSD. With this time measurement, you can calculate the column flow. See page 87.

Venting the MSD

A program in the data system guides you through the venting process. It turns off the heaters or the turbo pump at the correct time. It also lets you monitor temperatures in the MSD and indicates when to vent the MSD.

The MSD ***will*** be damaged by incorrect venting. A turbo pump will be damaged if it is vented while spinning at more than 50% of its normal operating speed.

WARNING **Make sure the GC/MSD interface and the analyzer zones are cool (below 100 °C) before you vent the MSD. A temperature of 100 °C is hot enough to burn skin; always wear cloth gloves when handling analyzer parts.**

WARNING **If you are using hydrogen as a carrier gas, the carrier gas flow must be off before turning off the power. If the foreline pump is off, hydrogen will accumulate in the MSD and an explosion may occur. Read "Hydrogen Safety" before operating the MSD with hydrogen carrier gas.**

CAUTION Never vent the MSD by allowing air in through either end of the foreline hose. Use the vent valve or remove the column nut and column.

Do not vent while the turbo pump is still spinning at more than 50%.

Do not exceed the maximum recommended total gas flow. See Table 2, "5975T LTM GC/MSD models and features," on page 15.

To View MSD Analyzer Temperature and Vacuum Status

You can also use the Local Control Panel to perform this task. See the G1701EA GC/MSD *ChemStation Getting Started* manual for more information.

Procedure

1 In Instrument Control view, select **Edit Tune Parameters** from the Instrument menu (Figure 17).

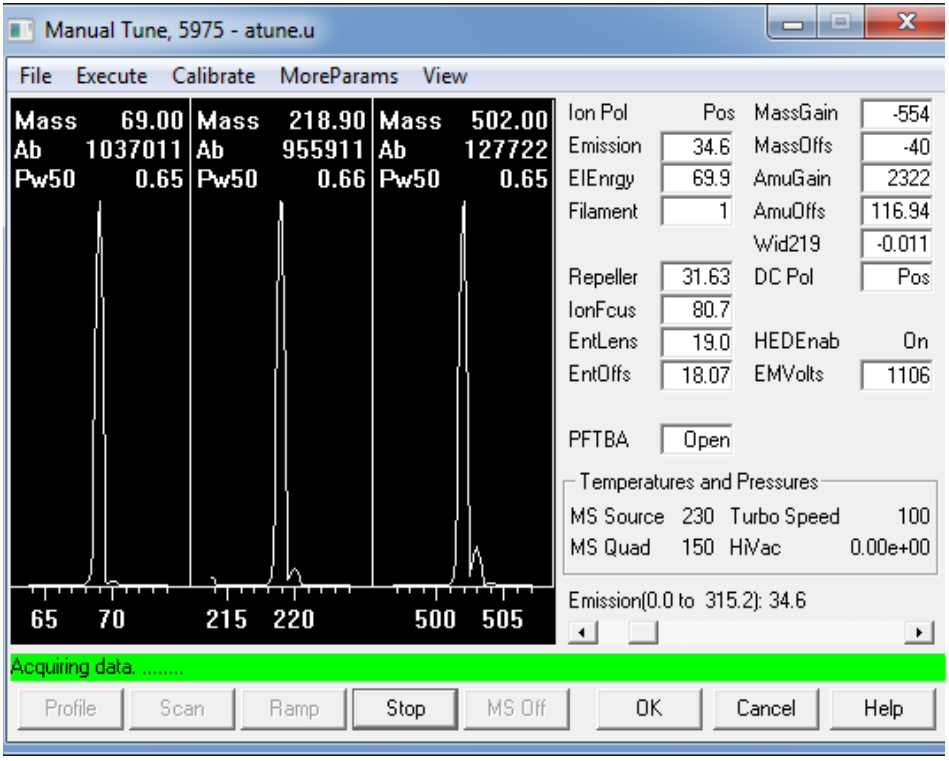


Figure 17 Tune parameters

2 Select the tune file you plan to use with your method from the **Load MS Tune File** dialog box.

3 Analyzer temperatures and vacuum status are displayed in the **Zones** field.

Unless you have just begun the pumpdown process, the turbo pump should be running at least 80% speed. MSD heaters remain off as long as the turbo pump is operating at less than 80%. Normally, the turbo pump speed will be at 100%.

The MSD heaters turn on at the end of the pumpdown cycle and turn off at the beginning of the vent cycle. The reported setpoints will not change during venting or pumpdown, even though both the MSD zones are turned off.

To Set Monitors for MSD Temperature and Vacuum Status

A monitor displays the current value of a single instrument parameter. They can be added to the standard instrument control window. Monitors can be set to change color if the actual parameter varies beyond a user-determined limit from its setpoint.

Procedure

1. Select **MS Monitors** from the Instrument menu.
2. In the **Edit MS Monitors** box, under **Type**, select **Zone**.
3. Under **Parameter**, select **MS Source** and click **Add**.
4. Under **Parameter**, select **MS Quad** and click **Add**.
5. Under **Parameter**, select **TurboSpd** and click **Add**.
6. Select any other monitors you want and **Add** them.
7. Click **OK**. The new monitors will be stacked on top of each other in the lower right corner of the Instrument Control window. They must be moved for you to see them all.
8. Click and drag each monitor to the desired position. See Figure 18 for one way of arranging the monitors.

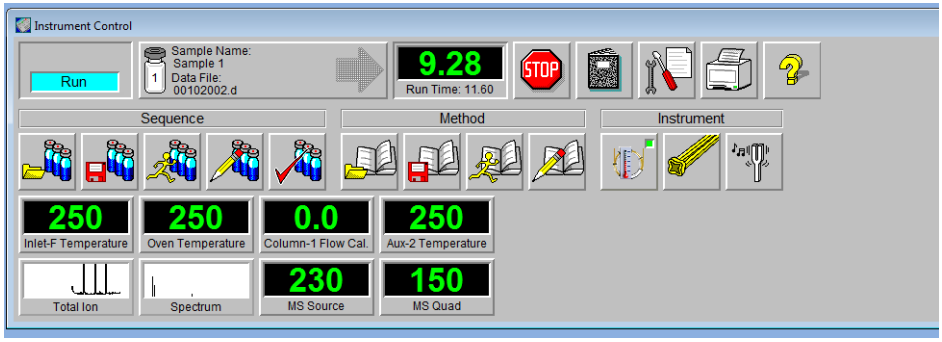


Figure 18 Arranging monitors

9. To make the new settings part of the method, select **Save** from the Method menu.

To Set the MSD Analyzer Temperatures

Setpoints for the MSD ion source and mass filter (quad) temperatures are stored in the current tune (*.u) file. When a method is loaded, the setpoints in the tune file associated with that method are downloaded automatically.

Procedure

1 In Instrument Control view, select **Edit Tune Parameters** from the Instrument menu.

2 Select **Temperatures** from the **MoreParams** menu (Figure 19).

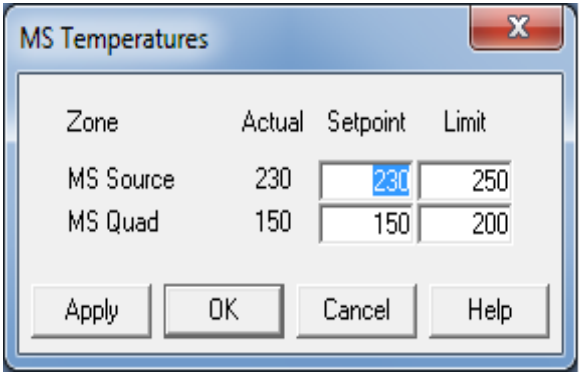


Figure 19 Setting temperatures

3 Type the desired Source and Quad (mass filter) temperatures in the setpoint fields. See Table 14 for recommended setpoints.

The GC/MSD interface, ion source, and quadrupole heated zones interact. The analyzer heaters may not be able to accurately control temperatures if the setpoint for one zone is much different from that of an adjacent zone.

WARNING **Do not exceed 200 °C for the quadrupole or 350 °C for the source.**

4 To close the screen, click:

- **Apply** to send the new temperature setpoints to the MSD.
- **OK** to change the currently loaded tune file but not download anything to the MSD.
- **Cancel** to exit the panel without changing the currently loaded tune file or downloading anything to the MSD.

5 When the **Save MS Tune File** dialog box appears, either click **OK** to save your changes to the same file or type a new file name and click **OK**.

Table 14 Recommended temperature settings

	EI operation
MS Source	230
MS Quad	150

To Set the GC/MSD Interface Temperature from the ChemStation

Procedure

1 Select **View>Instrument Control**.

2 Select **Instrument>GC Edit Parameters.**

3 Click the **Aux** icon to edit the interface temperature (Figure 20).

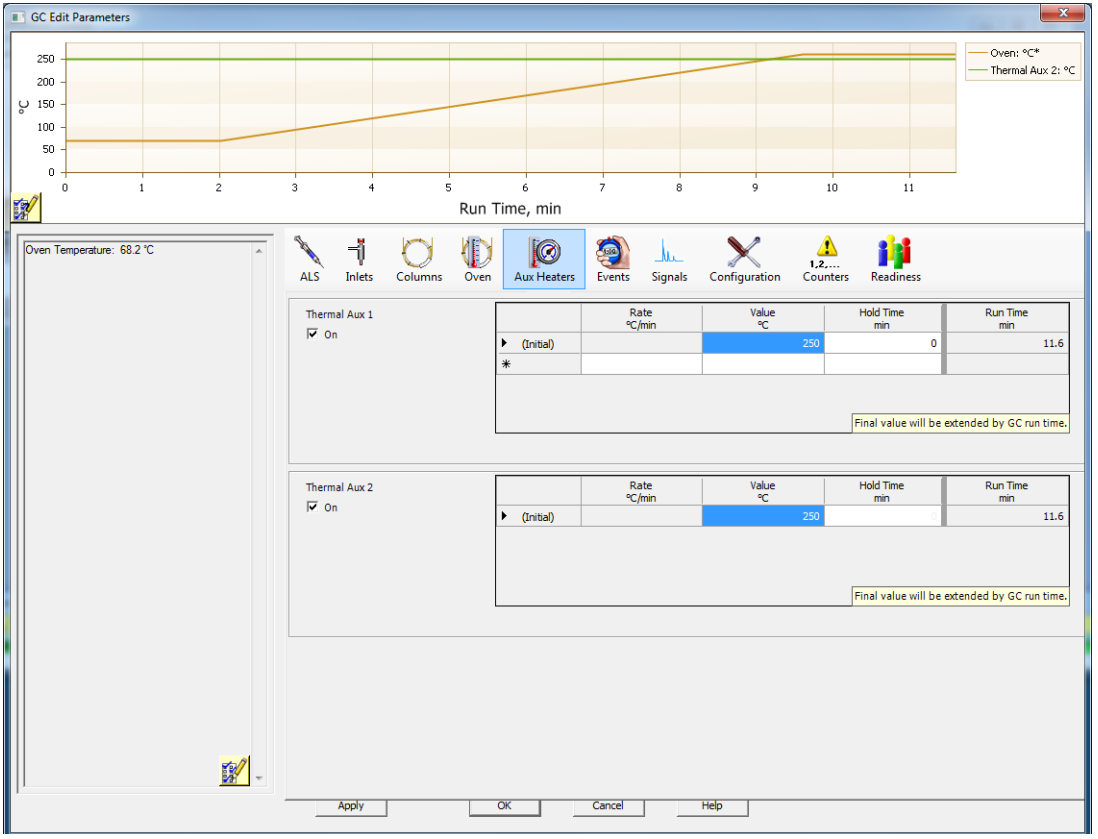


Figure 20 Setting the interface temperature

4 Check the heater **On** and type the setpoint in the **Value °C** column.

The typical setpoint is 280 °C. The limits are 0 °C and 350 °C. A setpoint below ambient temperature turns off the interface heater.

5 Click **Apply** to download setpoints or click **OK** to download setpoints and close the window.

6 To make the new settings part of the method, select **Save** from the Method menu.

CAUTION Make sure that the carrier gas is turned on and the column has been purged of air before heating the GC/MSD interface or the LTM column.

To Monitor High Vacuum Pressure

Pressure monitoring requires an optional external G4363A Ion gauge and controller. The readout for this gauge is mounted on top of the instrument.

Materials needed

- Ion gauge and controller (G4363A)

WARNING **If you are using hydrogen as a carrier gas, do not turn on the Ion gauge and controller if there is any possibility that hydrogen has accumulated in the analyzer chamber. Read "Hydrogen Safety" before operating the MSD with hydrogen carrier gas. Pump down for 25 minutes before you turn on the vacuum gauge.**

Procedure

1. Start up and pump down the MSD (page 106).
2. Turn on the gauge controller mounted on top of the analyzer cover.
3. The vacuum readout is displayed on the front of the optional vacuum controller.

The largest influence on operating pressure in EI mode is the carrier gas (column) flow. Table 15 lists typical pressures for various helium carrier gas flows. These pressures are approximate and will vary from instrument to instrument by as much as 30%.

Table 15 Ion Gauge and Controller Reading

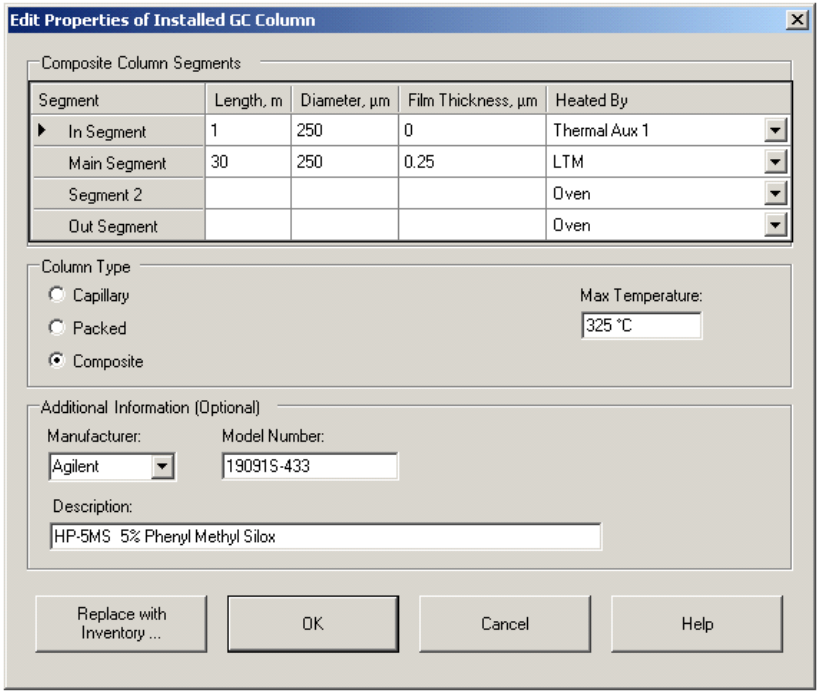
He Column flow rate, mL/min	Gauge reading, Torr Standard turbo pump
0.5	1.3E–05
0.7	1.83E–05
1	2.61E–05
1.2	3.11E–05
2	5.25E–05
3	8.01E–05

If the pressure is consistently higher than those listed, refer to the online help in the MSD ChemStation software for information on troubleshooting air leaks and other vacuum problems.

To Configure the LTM Column

Procedure

1. From the ChemStation Instrument Control window select **Instrument>GC Parameters** to display the **Edit GC Parameters** window.
2. Select the **Configuration** icon and then select the **Column** tab to display the screen where the current configured column is displayed.
3. To select a different LTM column previously assigned to inventory, click on the **Inventory** button to display the **Local Column Inventory** table.
4. Select the LTM column from this table that you are installing on the instrument. Click the **Install Selected Column** button to configure the instrument to use this column. The **Configuration** screen is displayed with the new column selection.
5. Click the first column labeled "1" (tooltip = Add/Modify this column) to display the **Edit Properties of Installed GC Column** dialog. The previously selected column is specified in the **Additional Information (Optional)** area.



6 In the **Column Type** area select **Composite** and set the manufacturer's recommended maximum temperature in the **Max Temperature** field.

7 In the **Composite Column Segments** area, **Segment** column, **Main Segment** item select **LTM** from the **Heated By** column dropdown.

8 In the **Composite Column Segments** area, **Segment** column, **In Segment** item select **Thermal Aux 1** from the **Heated By** column dropdown. Enter the Length, Diameter, and Film Thickness for the inlet guard column.

9 In the **Composite Column Segments** area, **Segment** column, **Out Segment** item select **Thermal Aux 1** from the **Heated By** column dropdown. Enter the Length, Diameter, and Film Thickness for the MSD guard column.

10 Click the **OK** button to complete the column configuration for the instrument.

11 From the **Configuration** screen, select the **Modules** tab to display the screen where the carried gas for the inlet is displayed.

12 For the **Front Inlet**, select the carrier gas used from the dropdown menu.

13 Click the **OK** button to save this configuration.

To Measure Column Flow Linear Velocity

With capillary columns, such as those used with the MSD, linear velocity is often measured rather than volumetric flow rate.

Procedure

1. Set Data Acquisition for splitless manual injection and selected ion monitoring (SIM) of *m/z* 28.
2. Press **Prep Run** on the LCP keypad.
3. Inject 1 µL of air into the GC inlet and press **Start Run**.
4. Wait until a peak elutes at *m/z* 28. Note the retention time.
5. Calculate the average linear velocity.

   $$\textit{Average linear velocity (cm/s)} = \frac{100\ L}{t}$$

   where:

   L = Length of the column in meters

   t = Retention time in seconds

   Be sure to account for any pieces of column broken off. A 1-meter section missing from a 25-meter column can yield a 4% error.
6. Use this velocity to verify the MSD ChemStation flow calculations (page 88).

   If the numbers disagree, click **Change** to calibrate the column dimensions.
7. To calculate the volumetric flow rate.

   $$\textit{Volumetric flow rate (mL/min)} = \frac{0.785\ D^2\ L}{t}$$

   where:

   D = Internal column diameter in millimeters

   L = Column length in meters

   t = Retention time in minutes

To Confirm Column Flow

Volumetric flow can be calculated from the column head pressure if the column dimensions are known.

Procedure

1 In the Instrument Control view, select **Instrument>GC Edit Parameters.**

2 Click the **Columns** icon (Figure 21 shows an example).

3 Select the appropriate column.

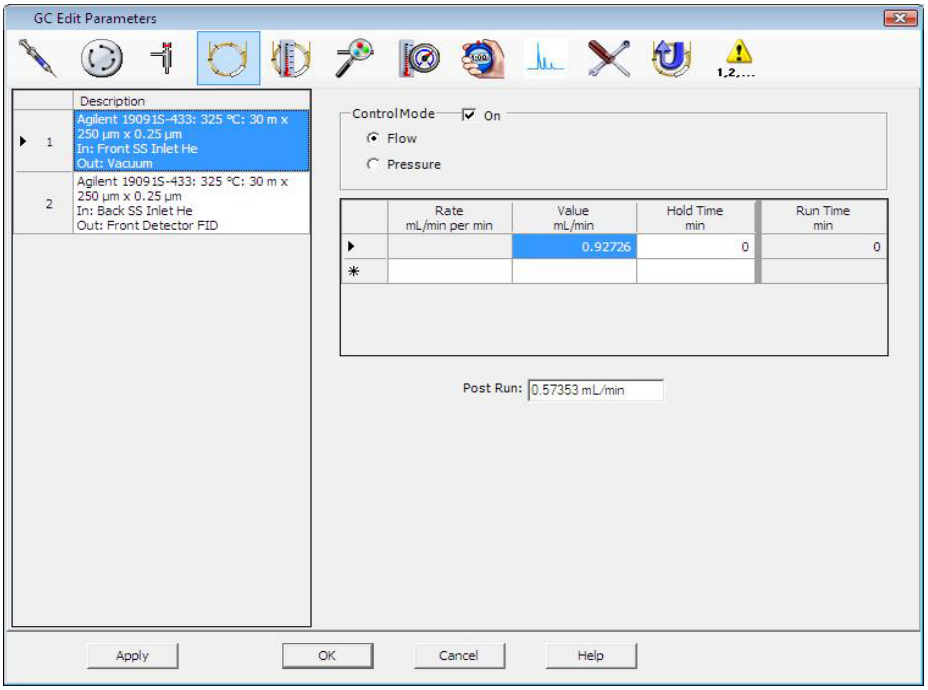


Figure 21 Calculating column flow

To Tune the MSD

You can also use the Local Control Panel to run the autotune that is currently loaded in the MSD ChemStation. See “Operating the GC/MSD from the LCP” .

Procedure

1. In the Instrument Control View, verify the correct tune file is loaded. For most applications, ATUNE.U (**Autotune)** gives good results. STUNE.U (**Standard Tune)** is not recommended as it may reduce sensitivity.
2. Set the system to the same conditions (LTM column temperature and column flow, and MSD analyzer temperatures) that will be used for data acquisition.
3. Select **Tune MSD** to perform a complete tune, or select **Quick Tune** to adjust peak width, mass assignment, and abundance, without changing ion ratios. The tune will start immediately.
4. Wait for the tune to complete and to generate the report.

   Save your tune reports. To view history of tune results, select **Checkout>View Previous Tunes...**.

To manually tune your MSD or to perform special autotunes, go to the Tune and Vacuum Control View.

From this Tune menu, in addition to the tunes available from Instrument Control, you can select special autotunes for specific spectral results, such as **DFTPP Tune** or **BFB Tune**.

See the manuals or online help provided with your MSD ChemStation software for additional information about tuning.

To Verify System Performance

Materials needed

- 1 pg/µL (0.001 ppm) OFN sample (5188-5348)

Verify the tune performance

1. Verify that the system has been pumping down for at least 60 minutes.
2. Set the LTM column temperature to 150 °C and the column flow to 1.0 mL/min.
3. In the Instrument Control view, select **Checkout Tune** from the Checkout menu. The software will perform an autotune and print the report.
4. When the autotune has completed, save the method and then select **Evaluate Tune** from the Checkout menu.

   The software will evaluate the last autotune and print a System Verification – Tune report.

Verify the sensitivity performance

1. Set up to inject 1 µL of OFN, either with the ALS or manually.
2. In the Instrument Control view, select **Sensitivity Check** from the Checkout menu.
3. Click the appropriate icons in the Instrument | Edit window to edit the method for the type of injection.
4. Click **OK** to run the method.

   When the method is completed, an evaluation report will be printed.

   Verify that rms signal-to-noise ratio meets the published specification. Please see the Agilent Web site at www.agilent.com/chem for specifications.

High-Mass Testing (5975T LTM GC/MSDs)

Setup conditions

1. Obtain a sample of PFHT (5188-5357).
2. Load tune file ATUNE.U then auto tune the MSD.
3. Resolve the PFHT.M method under x\5975\PFHT.M where x is instrument number being used.
4. Update and save the method.

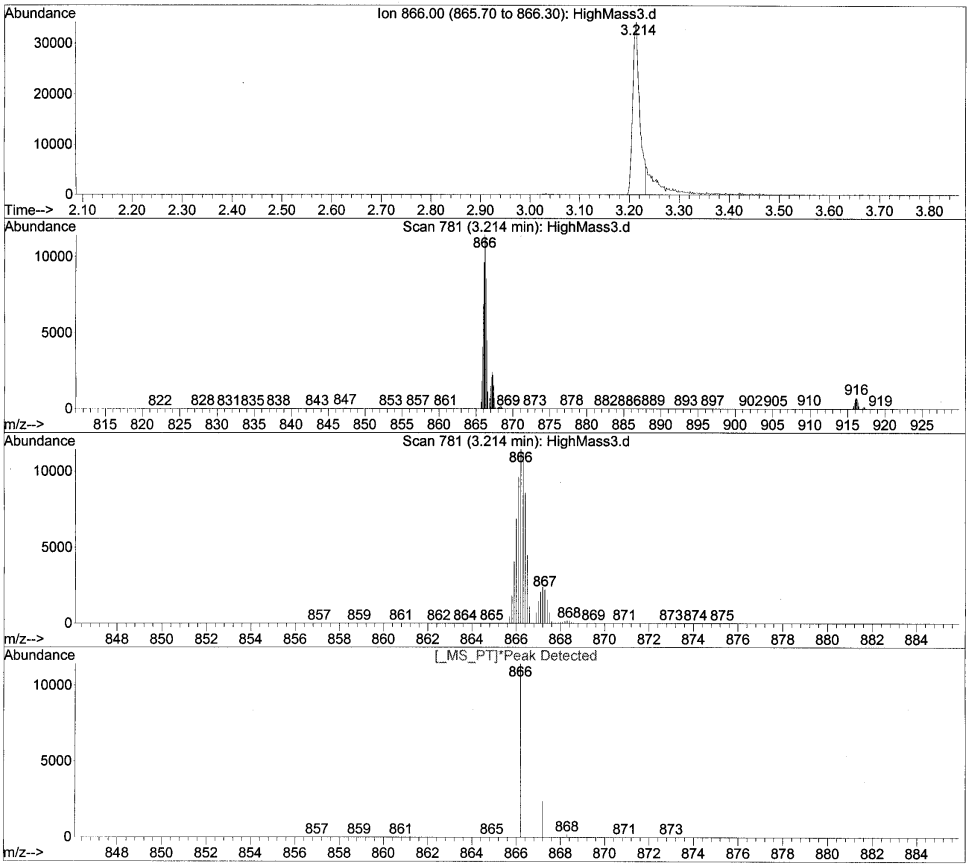
High-mass checkout

1. Load sample into a vial and place in position 2.
2. Select **High Mass Check** from the **Checkout** menu.
3. Follow the instructions on screen.
4. The Run is completed and results are printed within 5 minutes.

Results

```
                        *PFHT HIGH MASS REPORT

Data File : C:\msdchem\1\5975\HighMass3.d                Vial: 2
Acq On    : 28 Apr 2005  15:07                        Operator:
Sample    : *HIGH MASS TEST                           Inst    : Instrument #1
Misc      : _[]                                       Multiplr: 1.00
Barcode   : *EXPECTED=*  <NONE>  ACTUAL=*  <NONE>  Sample Amount:0.00
MS Integration Params: NA
```



*	MASS	ACTUAL	ISOTOPE	ABUND	ISOTOPE	RATIO	RELATIVE	WIDTH
	866.00	866.20	867.20	11439	2402	21.00	100.00	0.512
	867.00	867.20	868.30	2402	171	7.12	21.00	0.512
	916.00	916.20	917.20	742	155	20.89	6.49	0.553

Figure 22 PFHT high mass report

Results will indicate the recommended amount to adjust AMU offset for high-mass. If your results are within 5 units of the targeted amount, there is no need to make adjustments.

Adjustments

1 Verify ATUNE.U has been loaded.

2 Select **Edit Tune Parameters** from the Instrument menu via Instrument Control.

3 Click on **MoreParams** and select **DynamicRamping Params...**

   a Select AMU offset from the drop down box.

   b If the values on the right side are greyed out then select the **Enable Dynamic Ramping For This Lens** checkbox.

   c Enter in the recommend offset and click **OK.**

4 Click **OK** on the Edit Parameters box. The Save MS Tune File dialog box appears.

   You can overwrite the existing ATUNE.U to include high-mass adjustment or save this file to a new name, for example, ATUNEHIGH.U.

NOTE Anytime an ATUNE.U is performed it will overwrite the AMU offset which was entered. This is the reason for renaming the tune.

5 Load the PFHT.M and the saved tune file, then save the method.

6 Rerun test mixture (repeat high-mass checkout). If the correction is within 5 units, no further adjustments are required.

To Open the LTM/Guard Column Enclosure Door

Push on the latch under the bottom left corner of the door to the LTM/guard column enclosure and open the door.

Figure 23 Removing covers

To Open the LCP/Analyzer Window

1 Open the two latches at the top of the LCP by lifting them up and away from the LCP window.

2 Pull the LCP forward from the top to allow the panel to swing down.

To Remove the Analyzer Top Cover

1 Open the LCP/Analyzer window. See Figure 24.

2 Loosen the two captive screws on the front bottom left and top right corners of the cover, inside the LCP opening.

3 Slide the cover forward until it is free of the mainframe, and lift it off the instrument.

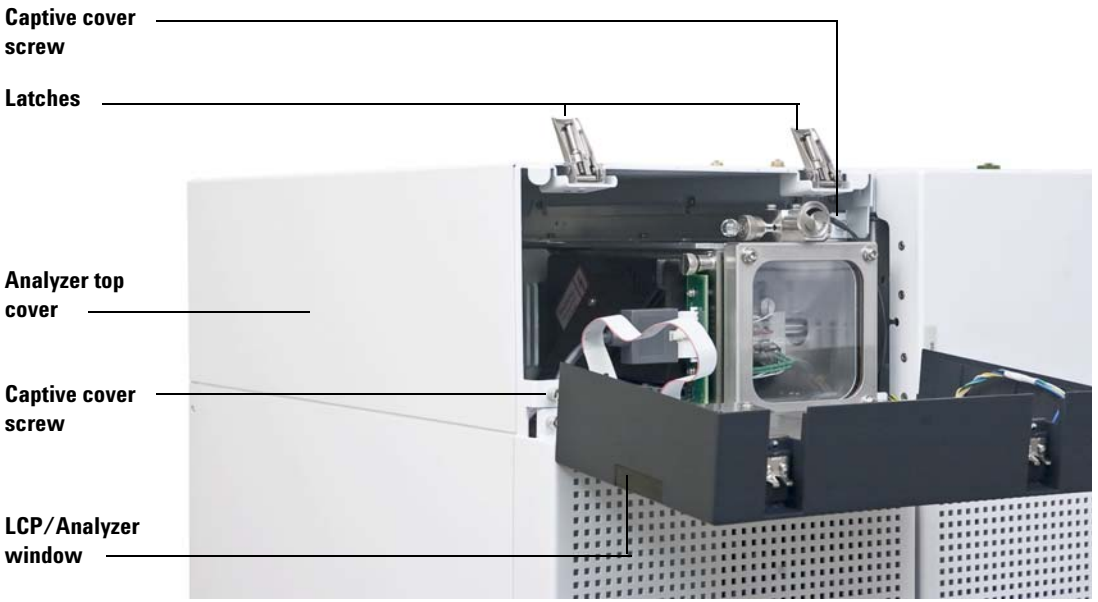


Figure 24 Removing analyzer top cover

WARNING **Do not remove any other covers. Dangerous voltages are present under other covers.**

To Vent the MSD

Procedure

1 Select **Vent** from the Vacuum menu in the software. Follow the instructions presented.

2 Set the GC/MSD interface heater and the LTM column temperatures to ambient (room temperature).

WARNING **If you are using hydrogen as a carrier gas, the carrier gas flow must be off before turning off the MSD power. If the turbo pump is off, hydrogen will accumulate in the MSD and an explosion may occur. Read "Hydrogen Safety" before operating the MSD with hydrogen carrier gas.**

CAUTION Be sure the LTM column and the GC/MSD interface are cool before turning off carrier gas flow.

3 When prompted, turn off the power switch.

4 Unplug the power cord.

WARNING **When the MSD is vented, do not put the ChemStation into Instrument Control view. Doing so will turn on the interface heater.**

5 Open the LCP window. See "To Open the LCP/Analyzer Window" on page 95

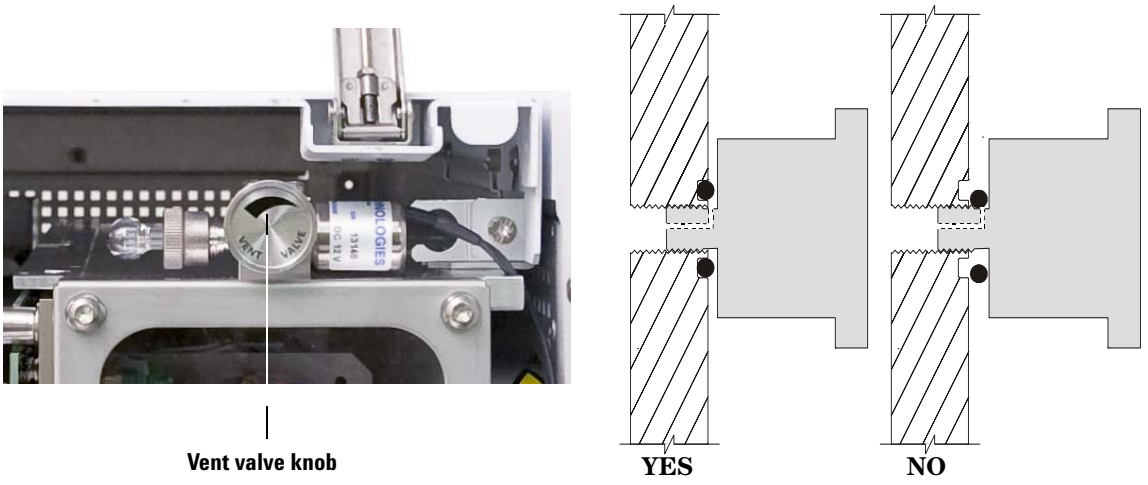


Figure 25 Venting the MSD

6 Turn the vent valve knob (Figure 25) counterclockwise *only* 3/4 turns or until you hear the hissing sound of air flowing into the analyzer chamber.

Do ***not*** turn the knob too far or the O-ring may fall out of its groove. Be sure to retighten the knob before pumping down.

WARNING **Allow the analyzer to cool to near room temperature before touching it.**

CAUTION Always wear clean gloves while handling any parts that go inside the analyzer chamber.

WARNING **When the MSD is vented, do not put the ChemStation into Instrument Control view. Doing so will turn on the interface heater.**

To Open the Analyzer Chamber

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Wrist strap, antistatic
  - Small (9300-0969)
  - Medium (9300-1257)
  - Large (9300-0970)

CAUTION Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded antistatic wrist strap and take other antistatic precautions (see page 75) before you open the analyzer chamber.

Procedure

1 Vent the MSD (page 94).

2 Remove the analyzer top cover. (See "To Remove the Analyzer Top Cover" on page 95)

3 Disconnect the side board control cable and the source power cable from the side board.

4 Loosen the side plate thumbscrews (Figure 26) if they are fastened.

The rear side plate thumbscrew should be unfastened during normal use. It is only fastened during shipping. The front side plate thumbscrew should only be fastened if hydrogen or other flammable or toxic substances are used for carrier gas.

CAUTION In the next step, if you feel resistance, **stop**. Do not try to force the side plate open. Verify that MSD is vented. Verify that both the front and rear side plate screws are completely loose.

5 *Gently* swing the side plate out.

WARNING **The analyzer, GC/MSD interface, and other components in the analyzer chamber operate at very high temperatures. Do not touch any part until you are sure it is cool.**

CAUTION Always wear clean gloves to prevent contamination when working in the analyzer chamber.

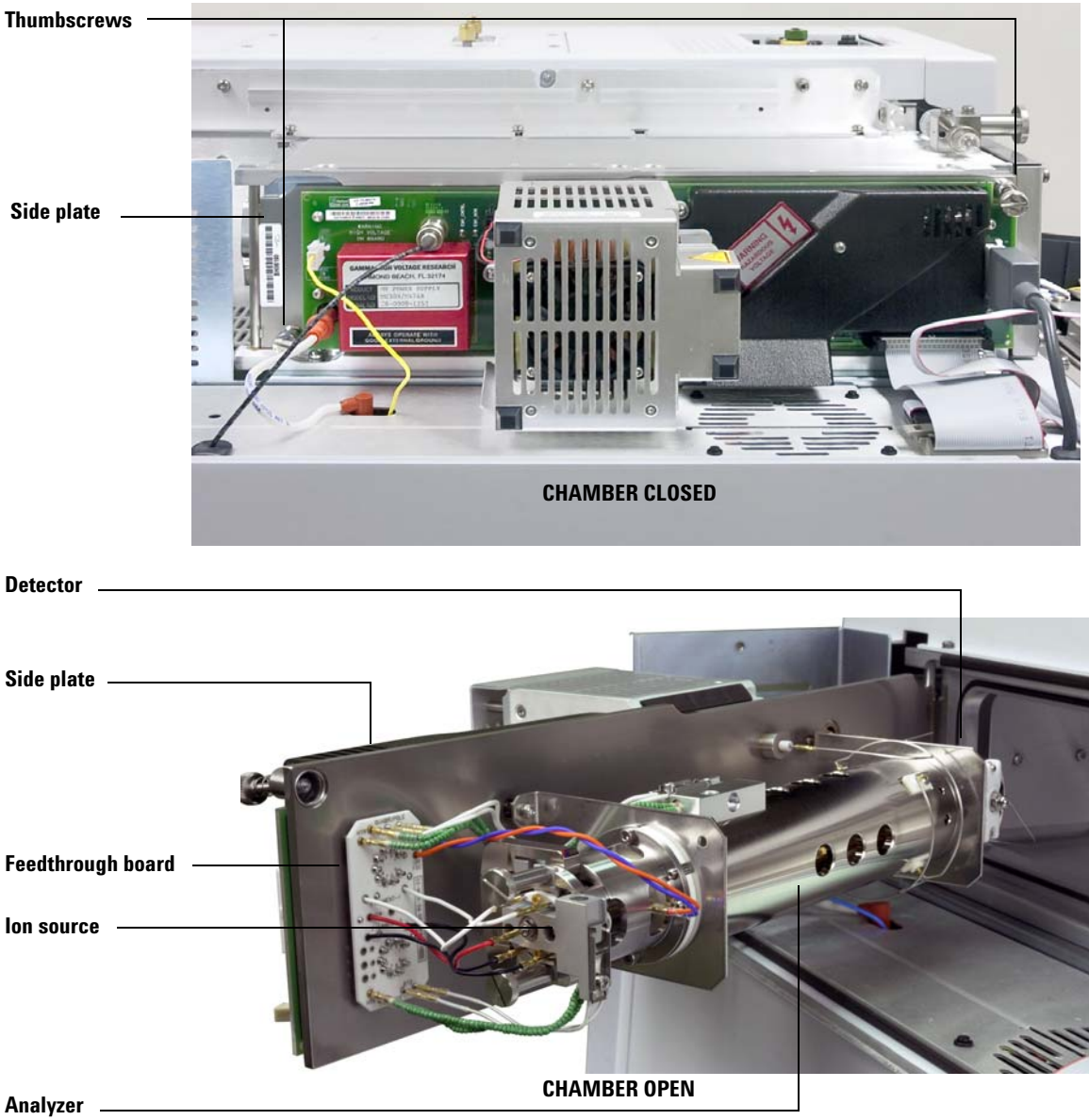


Figure 26 The analyzer chamber

To Close the Analyzer Chamber

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)

Procedure

1 Make sure all the internal analyzer electrical leads are correctly attached.

The wiring is described in Table 16 and illustrated in Figure 27 and Figure 28. The term “Board” in the table refers to the feedthrough board located next to the ion source.

Table 16 Analyzer wiring

Wire description	Attached to	Connects to
Green beaded (2)	Quad heater	Board, top left (HTR)
White with braided cover (2)	Quad sensor	Board, top (RTD)
White (2)	Board, center (FILAMENT-1)	Filament 1 (top)
Red (1)	Board, center left (REP)	Repeller
Black (2)	Board, center (FILAMENT-2)	Filament 2 (bottom)
Orange (1)	Board, top right (ION FOC)	Ion focus lens
Blue (1)	Board, top right (ENT LENS)	Entrance lens
Green beaded (2)	Ion source heater	Board, bottom left (HTR)
White (2)	Ion source sensor	Board, bottom (RTD)

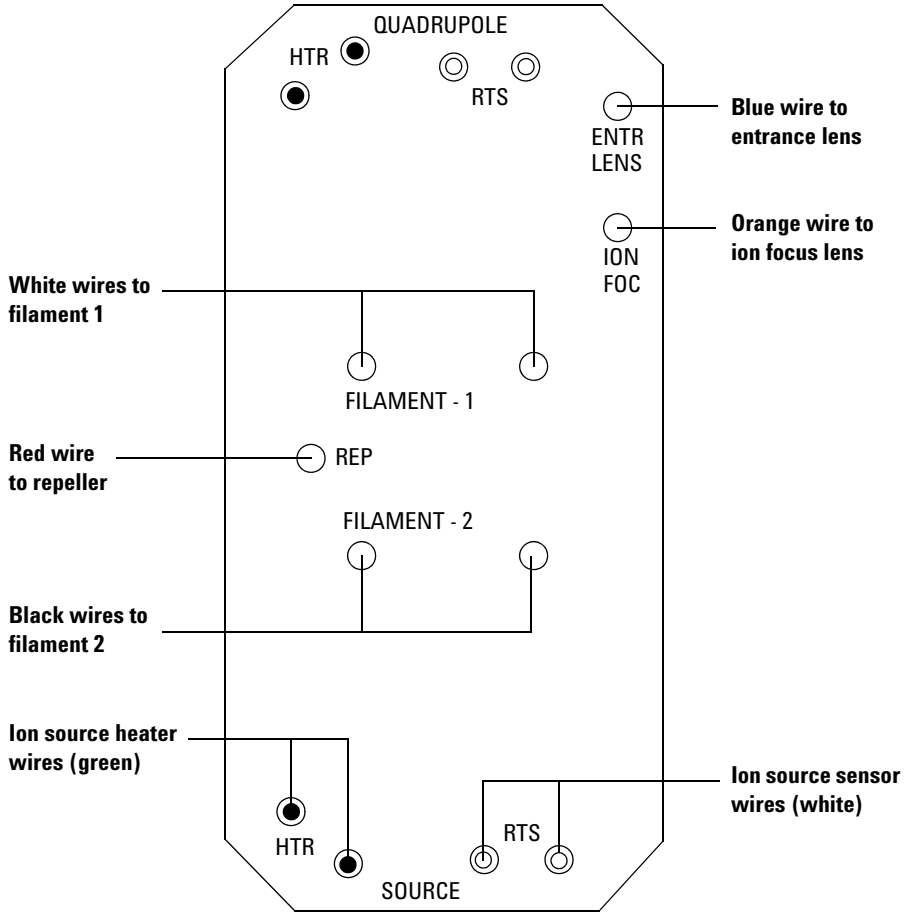


Figure 27 Feedthrough board wiring

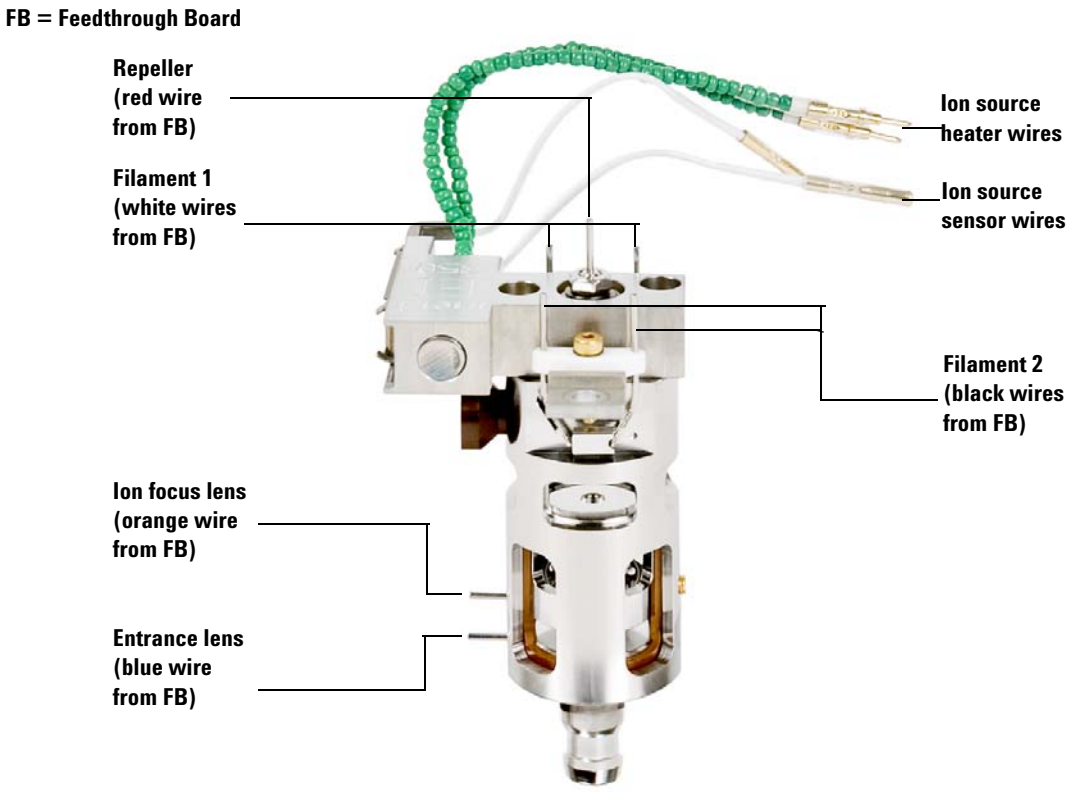


Figure 28 Ion source wiring

2 Check the side plate O-ring.

Make sure the O-ring has a ***very*** light coat of Apiezon L high vacuum grease. If the O-ring is very dry, it may not seal well. If the O-ring looks shiny, it has too much grease on it. (Refer to the 5975T LTM GC/MSD Troubleshooting and Maintenance Manual for lubricating instructions.)

3 Close the side plate.

4 Reconnect the side board control cable and source power cable to the side board.

5 Make sure the vent valve is closed.

6 Pump down the MSD (See "To Pump Down the MSD" on page 106.).

7 If hydrogen or other flammable or toxic substance is used for carrier gas, *gently* hand tighten the front side plate thumbscrew.

WARNING **The front thumbscrew must be fastened if hydrogen (or other hazardous gas) is being used as the GC carrier gas. In the unlikely event of an explosion, it may prevent the side plate from opening.**

CAUTION Do not overtighten the thumbscrew; it can cause air leaks or prevent successful pumpdown. Do not use a screwdriver to tighten the thumbscrew.

8 Once the MSD has pumped down, replace the analyzer cover and close the LCP/Analyzer window.

To Pump Down the MSD

You can also use the Local Control Panel to perform this task. See "Operating the GC/MSD from the LCP" .

WARNING **Make sure your MSD meets all the conditions listed in the introduction to this chapter (page 69) before starting up and pumping down the MSD. Failure to do so can result in personal injury.**

WARNING **If you are using hydrogen as a carrier gas, do not start carrier gas flow until the MSD has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the MSD and an explosion may occur. Read "Hydrogen Safety" before operating the MSD with hydrogen carrier gas.**

Procedure

1 Open the LCP/Analyzer window and close the vent valve.

2 Plug the power cord into the building supply.

3 Select **Tune and Vacuum Control** from the **View** menu in the MSD ChemStation.

Select **Pump Down** from the **Vacuum** menu.

4 When prompted, press the start button on the front of the instrument.

5 Reaching through the LCP/Analyzer window opening, press lightly on the side board to ensure a correct seal. Press on the metal box on the side board.

The foreline pump will make a gurgling noise. This noise should stop within a minute. If the noise continues, there is a ***large*** air leak in your system, probably at the side plate seal, the interface column nut, or the vent valve.

6 Close the LCP/Analyzer window.

7 Once communication with the PC has been established, click **OK**.

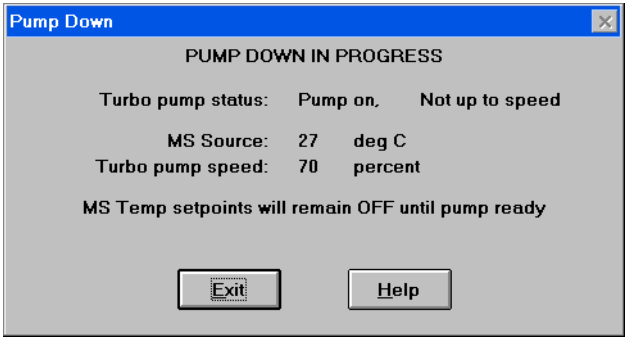


Figure 29 Pumping down

CAUTION Within 10 to 15 minutes the turbo pump speed should be up to 80% (Figure 29). The pump speed should eventually reach 95%. If these conditions are not met, the MSD electronics will shut off the foreline pump. In order to recover from this condition, you must power cycle the MSD. If the MSD does not pump down correctly, see the manual or online help for information on troubleshooting air leaks and other vacuum problems.

8 When prompted, turn on the GC/MSD interface heater, guard column enclosure, and LTM column. Click **OK** when you have done so.

The software will turn on the ion source and mass filter (quad) heaters. The temperature setpoints are stored in the current autotune (*.u) file.

CAUTION Do not turn on any GC heated zones until carrier gas flow is on. Heating a column with no carrier gas flow will damage the column.

9 After the message **Okay to run** appears, wait 2 hours for the MSD to reach thermal equilibrium. Data acquired before the MSD has reached thermal equilibrium may not be reproducible.

To Move or Store the Instrument

This procedure is for moving or storing the instrument around the same location. If the instrument needs to be shipped for use in a different location, please refer to the Agilent 5975T LTM GC/MSD Hardware Installation Manual for Field Transportable Units for the appropriate instructions.

Materials needed

- Ferrule, blank (5181-3308)
- Interface column nut (05988-20066)
- Wrench, open-end, 1/4-inch × 5/16-inch (8710-0510)

Procedure

1. Vent the instrument. (page 97).
2. Remove the guard column from the GC/MSD interface and install a blank ferrule and interface nut.
3. Tighten the vent valve.
4. Remove the analyzer top cover (page 95).
5. Finger-tighten the side plate thumbscrews (Figure 30).

CAUTION Do not overtighten the side plate thumbscrews. Overtightening will strip the threads in the analyzer chamber. It will also warp the side plate and cause leaks.

6. Plug in the instrument power cord.
7. Turn on the instrument to establish a rough vacuum. Verify that the turbo pump speed is greater than 50%.
8. Turn off the instrument.
9. Replace the analyzer cover.
10. Disconnect the LAN, remote, and power cables.
11. Disconnect the carrier gas supply.

Figure 30 Side plate thumbscrews

The GC/MSD can now be stored or moved. The foreline pump cannot be disconnected; it must be moved with the MSD. Make sure the GC/MSD remains upright and is never tipped on its side or inverted.

CAUTION The GC/MSD must remain upright at all times. If you need to ship your GC/MSD to another location, contact your Agilent Technologies service representative for advice about packing and shipping.

4
General Maintenance

Before Starting

For your safety, read all of the information in this introduction before performing any maintenance tasks.

Scheduled maintenance

Common maintenance tasks are listed in Table 17. Performing these tasks when scheduled can reduce operating problems, prolong system life, and reduce overall operating costs.

Keep a record of system performance (tune reports) and maintenance operations performed. This makes it easier to identify variations from normal operation and to take corrective action.

Table 17 Maintenance schedule

Task	Every week	Every 6 months	Every year	As needed
Vacuum System				
Check the foreline pump oil level	X			
Replace the foreline pump oil		X		
Check the dry foreline pump diaphragms				X
Change scroll pump seals			X	
Lubricate sideplate or vent valve O-rings*				X
MSD				
Tune the MSD				X
Clean the ion source				X
Check the calibration vial(s)	X			
GC				
Check the carrier gas trap(s) on the GC				X
Trim or replace the guard columns				X
Replace inlet septum, liner, and o-ring				X

Table 17 Maintenance schedule (continued)

Task	Every week	Every 6 months	Every year	As needed
Change inlet gold seal				X
Replace GC carrier gas supplies				X

* Vacuum seals other than the side plate O-ring and vent valve O-ring do not need to be lubricated. Lubricating other seals can interfere with their correct function.

Tools, spare parts, and supplies

Some of the required tools, spare parts, and supplies are included in the shipping kit. You must supply others yourself. Each maintenance procedure includes a list of the materials required for that procedure.

High voltage precautions

Whenever the MSD is plugged in, even if the power switch is off, potentially dangerous voltage (120 VAC or 200/240 VAC) exists on:

- The wiring and fuses between where the power cord enters the instrument and the power switch

When the power switch is on, potentially dangerous voltages exist on:

- Electronic circuit boards
- Toroidal transformer
- Wires and cables between these boards
- Wires and cables between these boards and the connectors on the back panel of the MSD
- Some connectors on the back panel (for example, the foreline power receptacle)

Normally, all of these parts are shielded by safety covers. As long as the safety covers are in place, it should be difficult to accidentally make contact with dangerous voltages.

WARNING **Perform no maintenance with the instrument turned on or plugged into its power source unless you are instructed to by one of the procedures in this chapter.**

Some procedures in this chapter require access to the inside of the GC/MSD while the power switch is on. Do not remove any of the electronics safety covers in any of these procedures. To reduce the risk of electric shock, follow the procedures carefully.

Dangerous temperatures

Many parts in the instrument operate at, or reach, temperatures high enough to cause serious burns. These parts include, but are not limited to:

- GC/MSD interface
- Analyzer parts
- Vacuum pumps
- Inlet
- LTM column
- Guard column heated enclosure

WARNING **Never touch these parts while your instrument is on. After the instrument is turned off, give these parts enough time to cool before handling them.**

WARNING **The foreline pump can cause burns if touched when operating.**

The inlet, guard column heated enclosure, and LTM column also operate at very high temperatures. Use the same caution around these parts.

Chemical residue

The split vent outlet and purge vent outlet contain high concentrations of the chemicals you are analyzing along with the carrier gas. If you are processing hazardous chemicals, these outlets should be vented to a safe location outside the laboratory.

Only a small portion of your sample is ionized by the ion source. The majority of any sample passes through the ion source without being ionized. It is pumped away by the vacuum system. As a result, the exhaust from the foreline pump will contain traces of the carrier gas and your samples. Exhaust from the standard foreline pump also contains tiny droplets of foreline pump oil.

An oil trap is supplied with the standard foreline pump. This trap stops ***only*** pump oil droplets. It ***does not*** trap any other chemicals. If you are using toxic solvents or analyzing toxic chemicals, do not use this oil trap. For all foreline pumps, install a hose to take the exhaust from the foreline pump outdoors or into a fume hood vented to the outdoors. For the standard foreline pump, this requires removing the oil trap. Be sure to comply with your local air quality regulations.

WARNING **The oil trap supplied with the standard foreline pump stops only foreline pump oil. It does not trap or filter out toxic chemicals. If you are using toxic solvents or analyzing toxic chemicals, remove the oil trap and install a hose to take the foreline pump exhaust outside or to a fume hood.**

The oil in the standard foreline pump also collects traces of the samples being analyzed. All used pump fluid should be considered hazardous and handled accordingly. Dispose of used fluid correctly, as specified by your local regulations.

WARNING **When replacing pump fluid, use appropriate chemical-resistant gloves and safety glasses. Avoid all contact with the fluid.**

Electrostatic discharge

All of the printed circuit boards in the MSD contain components that can be damaged by electrostatic discharge (ESD). Do not handle or touch these boards unless absolutely necessary. In addition, wires, contacts, and cables can conduct ESD to the electronics boards to which they are connected. This is especially true of the mass filter (quadrupole) contact wires which can carry ESD to sensitive components on the side board. ESD damage may not cause immediate failure but it will gradually degrade the performance and stability of your MSD.

When you work on or near printed circuit boards or when you work on components with wires, contacts, or cables connected to printed circuit boards, always use a grounded antistatic wrist strap and take other antistatic precautions. The wrist strap should be connected to a known good earth ground. If that is not possible, it should be connected to a conductive (metal) part of the assembly being worked on, but ***not*** to electronic components, exposed wires or traces, or pins on connectors.

Take extra precautions, such as a grounded antistatic mat, if you must work on components or assemblies that have been removed from the MSD. This includes the analyzer.

CAUTION

To be effective, an antistatic wrist strap must fit snugly (not tight). A loose strap provides little or no protection.

Antistatic precautions are not 100% effective. Handle electronic circuit boards as little as possible and then only by the edges. Never touch components, exposed traces, or pins on connectors and cables.

Maintaining the Vacuum System

Periodic maintenance

As listed earlier in Table 17, some maintenance tasks for the vacuum system must be performed periodically. These include:

- Checking the foreline pump fluid (every week)
- Checking the calibration vial (every 6 months)
- Ballasting the foreline pump
- Replacing the foreline pump oil every 6 months
- Tightening the foreline pump oil box screws (first oil change after installation, standard foreline pump)
- Checking the dry foreline pump diaphragms (typically every 3 years)
- Changing scroll pump tip seals (yearly)

Failure to perform these tasks as scheduled can result in decreased instrument performance. It can also result in damage to your instrument.

Other procedures

Tasks such as replacing a Micro-Ion vacuum gauge should be performed only when needed. See the *Agilent 5975T LTM GC/MSD Troubleshooting and Maintenance Manual* or the online help in the MSD ChemStation software for symptoms that indicate this type of maintenance is required.

More information is available

If you need more information about the locations or functions of vacuum system components, see the *Agilent 5975T LTM GC/MSD Troubleshooting and Maintenance Manual*.

Most of the procedures in this chapter are illustrated with video clips on this 5975T LTM GC/MSD Instrument Utilities DVD.

Maintaining the Analyzer

Scheduling

None of the analyzer components require periodic maintenance. Some tasks, however, must be performed when GC/MSD behavior indicates they are necessary. The *Agilent 5975T LTM GC/MSD Troubleshooting and Maintenance Manual* provides information about symptoms that indicate the need for analyzer maintenance. The troubleshooting material in the online help in the ChemStation software provides more extensive information.

Precautions

Cleanliness

Keep components clean during analyzer maintenance. Analyzer maintenance involves opening the analyzer chamber and removing parts from the analyzer. During analyzer maintenance procedures, take care to avoid contaminating the analyzer or interior of the analyzer chamber. Wear clean gloves during all analyzer maintenance procedures. After cleaning, parts must be thoroughly baked out before they are reinstalled. After cleaning, analyzer parts should be placed only on clean, lint-free cloths. It is recommended that a spare, clean EI source be ready to install when the source in use needs cleaning. For information on how to clean an EI source, refer to the *Agilent 5975T LTM GC/MSD Troubleshooting and Maintenance Manual*.

CAUTION If not done correctly, analyzer maintenance can introduce contaminants into the MSD.

WARNING **The analyzer operates at high temperatures. Do not touch any part until you are sure it is cool.**

Some parts can be damaged by electrostatic discharge

The wires, contacts, and cables connected to the analyzer components can carry electrostatic discharges (ESD) to the electronics boards to which they are connected. This is especially true of the mass filter (quadrupole) contact

wires which can conduct ESD to sensitive components on the side board. ESD damage may not cause immediate failure but will gradually degrade performance and stability. See page 115 for more information.

CAUTION Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded antistatic wrist strap (see page 115) and take other antistatic precautions ***before*** you open the analyzer chamber.

Some analyzer parts should not be disturbed

The mass filter (quadrupole) requires no periodic maintenance. In general, the mass filter should never be disturbed. In the event of extreme contamination, it can be cleaned, but such cleaning should only be done by a trained Agilent Technologies service representative. The HED ceramic insulator must never be touched.

CAUTION Incorrect handling or cleaning of the mass filter can damage it and have a serious, negative effect on instrument performance. Do not touch the HED ceramic insulator.

More information is available

If you need more information about the locations or functions of analyzer components, refer to the *Agilent 5975T LTM GC/MSD Troubleshooting and Maintenance Manual*. Many procedures in this manual are illustrated with video clips.

Maintenance Methods

Before performing maintenance procedures on the inlet, LTM column module, guard column enclosure, and GC/MS transfer line, the instrument must be made safe. Agilent recommends that you create and store the following maintenance methods. The methods below will:

- Prevent damage to the instrument (electronics, columns, etc.)
- Avoid injury to the user (burns, shocks, etc.)
- Allow you to perform maintenance on specific areas while leaving the rest of the components at operating temperature

NOTE From operating temperature the inlet may require 12 hours or longer to cool down to the maintenance method setpoint.

Column maintenance method

Create a method for cooling down the instrument prior to performing maintenance tasks on the capillary column system between the inlet and MSD.

- Set the LTM column temperature to **35 °C**. This allows the LTM column module fan to quickly cool the column.
- Set the heater to **Off** for the guard column enclosure.
- Set the MSD transfer line heater **Off**.
- Set the inlet temperature to **35 °C** and set the inlet gas flow to **30 cm/s**.
  - Wait for the LTM column to cool down before turning off column carrier gas flow at the source. Cap both ends of the column to keep air out if you are removing it.
  - When possible, keep inert carrier gas flowing to protect the column.
- Turn off the analyzer filament and EM.

Once the method setpoints are reached, the instrument becomes **Ready**. Next, a procedure normally instructs you to turn heaters off and shut down gas flows allowing maintenance to be performed.

WARNING **Be careful! The LTM column, guard column enclosure, GC/MSD transfer line, analyzer and inlet may be hot enough to cause burns. If hot, wear heat-resistant gloves to protect your hands.**

To Change the Septum on the Split/Splitless Inlet

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Replacement septum
  - Septum retainer nut for headspace (18740-60830)
  - Septum retainer nut (18740-60835)
  - 11-mm septum, high-temperature, low-bleed, 50/pk (5183-4757)
  - 11-mm septum, prepierced, long life, 50/pk (5183-4761)
  - Merlin Microseal septum (high-pressure) (5182-3444)
  - Merlin Microseal septum (30 psi) (5181-8815)
- Wrench, hex for changing septum
- 0- or 00-grade steel wool (optional)
- Tweezers
- Wrench, capillary inlet (optional)

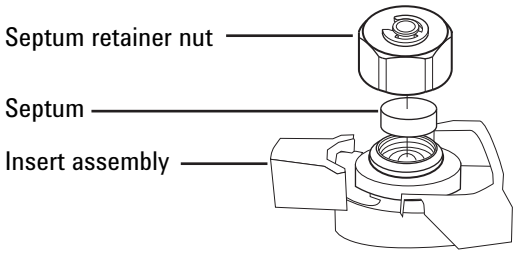
Procedure

1 Set the inlet pressure to **0** and wait for the GC to become ready.

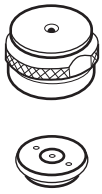
WARNING **Be careful! The oven and/or inlet may be hot enough to cause burns. If the inlet is hot, wear heat-resistant gloves to protect your hands.**

2 Remove the septum retainer nut or Merlin cap.

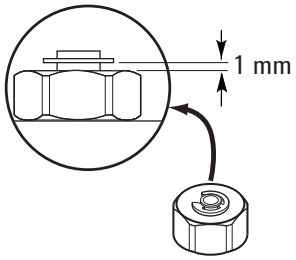
3 Use tweezers to remove the septum or Merlin Microseal from the retainer nut. Do not gouge or scratch the interior of the septum head.



4 Firmly press the new septum or Merlin Microseal into the fitting. The metal parts side of the Merlin Microseal should face down (toward the oven).



5 Install the septum retainer nut or Merlin cap and finger-tighten. Tighten the septum retainer nut until the C-ring is about 1 mm above the nut.



6 Restore the analytical method.

7 Reset the septum counter.

To Change the Liner and O-Ring on the Split/Splitless Inlet

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Replacement O-ring
  - Nonstick fluorocarbon liner O-ring (for temperatures up to 350 °C), 10/pk (5188-5365)
  - Graphite O-ring for split liner (for temperatures above 350 °C), 10/pk (5180-4168)
  - Graphite O-ring for splitless liner (for temperatures above 350 °C), 10/pk (5180-4173)
- Replacement liner
  - Split Low-pressure drop, glass wool, single taper, 870 µL (5183-4647)
  - Split Glass wool, 990 µL (19251-60540)
  - Split–Manual only Empty pin and cup, 800 µL (18740-80190)
  - Split–Manual only Packed pin and cup, 800 µL (18740-60840)
  - Splitless Single taper, glass wool, 900 µL (5062-3587)
  - Splitless Single taper, no glass wool, 900 µL (5181-3316)
  - Splitless Dual taper, no glass wool, 800 µL (5181-3315)
  - Splitless–Direct inject 2-mm id, quartz, 250 µL (18740-80220)
  - Splitless–Direct inject 2-mm id, 250 µL (5181-8818)
  - Direct inject –Headspace or purge and trap 1.5-mm id, 140 µL (18740-80200)
  - Direct column connect Single taper, splitless 4-mm id (G1544-80730)
  - Direct column connect Dual taper, splitless 4-mm id (G1544-80700)
- Tweezers
- Wrench, hex for changing septum
- Wrench, capillary inlet (optional)

Procedure

1 Turn off the inlet heater and flow.

WARNING **Be careful! The oven and/or inlet may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands.**

2 Slide the locking tab forward (counterclockwise). Lift the septum assembly straight up and away from the inlet to avoid chipping or breaking the liner.

3 Loosen the O-ring from the sealing surface with tweezers.

4 Grasp the liner with tweezers and pull it out.

5 Inspect the surface of the gold seal for graphite or rubber septum contamination. If required, replace the gold seal. (See the *Agilent 5975T LTM GC/MSD Troubleshooting and Maintenance Manual*.)

Clean the inlet if there is visible or suspected contamination. (See the *Agilent 5975T LTM GC/MSD Troubleshooting and Maintenance Manual.*)

Clean O-ring residue from sealing surface.

CAUTION Wear clean, lint-free gloves to prevent contamination of parts with dirt and skin oils.

6 Slide a new O-ring onto the replacement liner.

7 Return the liner to the inlet, pushing it all the way in until the liner contacts the gold seal.

8 Line up the tab on the bottom of the septum assembly with the slot on the insert assembly and push down to connect. Slide the locking tab to the back.

9 Turn on the inlet. Allow the inlet and column to purge with carrier gas for 15 minutes before heating the inlet or the LTM column.

10 Restore the analytical method.

11 Reset the liner counter.

12 Check for leaks.

To Bakeout the Column Flow Path

This procedure is used to bakeout contaminants in the inlet, guard column, and LTM column module.

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Pliers, long-nose (8710-1094)
- Guard column
- Column nut (5020-8292)
- Ferrules (Vespel)
  - 0.40-mm id, for 0.25-mm id columns (5181-3323)
  - 0.5-mm id, for 0.32-mm id columns (5062-3514)

Procedure

1 Load your maintenance method for cooling down the column enclosure. See "Column maintenance method" on page 120.

WARNING **Be careful! The guard column heated enclosure or internal accessories may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands or allow the parts to cool before beginning the work.**

WARNING **Always wear safety glasses when handling capillary columns. Use care to avoid puncturing your skin with the end of the column.**

CAUTION Wear clean, lint-free gloves to prevent contamination of the parts.

2 Vent the instrument. See "Venting the MSD" on page 75.

3 Remove the MSD guard column from the GC/MSD transfer line and cap the GC/MSD transfer line.

4 If necessary, install a new liner and septum in the inlet before powering on the instrument.

5 Remove the inlet guard column and discard or trim for subsequent reuse.

6 Trim the MSD transfer line end of the MSD guard column at the ferrule. Attach this end of the guard column to the inlet. See "To Install an Inlet Guard Column in the Split/Splitless Inlet" on page 33. This configuration moves the original MSD guard column to the inlet and reverses the carrier flow through the LTM column module.

7 From the ChemStation, change the carrier gas type configured if switching to a different carrier gas supply.

8 If the column was previously used in analyses of toxic chemicals, attach the open end of the LTM column to an exhaust system and vent to a safe location. This also applies to the split vent and purge vent outlets on the inlet.

9 Turn on the inlet, set the mode to split, and the column flow to 30 cm/s.

10 Check for leaks.

CAUTION Do not heat the column without a flow of carrier gas. You will damage the column.

11 Allow the carrier gas to flow through the column for 5 minutes without heating the LTM column or guard column enclosure.

12 Close the LTM module door.

13 Set the guard column enclosure temperature to 10°C below the maximum LTM column temperature.

14 Ramp the LTM column temperature 5 °C/minute to 10 °C above your highest analytical temperature.

15 Once the LTM column temperature exceeds 80 °C, inject 5 µL methanol into the inlet. Repeat two more times at 5-minute intervals. This helps remove any contamination from the column before it is installed into the GC/MSD interface.

CAUTION Never exceed the maximum column temperature.

16 Hold the temperature of 10 °C above your highest analytical temperature for 1 hour while allowing a flow rate of 30 cm/s through the column.

WARNING **Be careful! The guard column heated enclosure or internal accessories may be hot enough to cause burns. If either is hot, wear heat-resistant gloves to protect your hands or allow the parts to cool before beginning the work.**

17 Load your maintenance method for cooling down the column enclosure. See "Column maintenance method" on page 120.

18 Attach the installed inlet guard column to the LTM column module inlet union.

19 Install a new MSD guard column between the MSD transfer line and LTM column outlet union. See "To Install an MSD Guard Column in the GC/MSD Interface" on page 38 and "To Attach the Guard Columns to the LTM CFT Unions" on page 52.

20 Pump down the instrument. See "Pumping Down" on page 73.

21 Turn on the inlet, set the mode to splitless, and the column flow to 30 cm/s.

22 Check for leaks.

23 If your method requires hydrogen carrier gas, disconnect the current carrier gas supply from the instrument and attach the hydrogen supply. Verify that the analyzer is under vacuum and the vacuum pumps are working before opening the hydrogen supply line. From the ChemStation, change the configured carrier gas to hydrogen.

24 With a column flow of 30 cm/s, wait for 5 to 10 minutes before increasing the LTM column temperature to a low standby temperature.

25 Restore the analytical method.

To Bakeout Contaminants from the Split/Splitless Inlet

This procedure is used to bakeout inlet contaminants mostly through the split vent line.

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Pliers, long-nose (8710-1094)
- Guard column
- Column nut (5020-8292)
- Ferrules (Vespel)
  - 0.40-mm id, for 0.25-mm id columns (5181-3323)
  - 0.5-mm id, for 0.32-mm id columns (5062-3514)

Procedure

1. Put the inlet into split mode.
2. Set the column flow to the normal operating value, or set the capillary column gas velocity to 30 cm/s.
3. Set the inlet split vent flow to 200 mL/min.
4. Purge the column with carrier flow for at least 10 minutes before heating the LTM column.
5. Set the inlet temperature to 300 °C or 25 °C above the normal operating temperature.
6. Set the guard column enclosure temperature to the column manufacturer's maximum column temperature.
7. Set the LTM column to 25 °C above the method final column temperature to bake contaminants from the column. Do not exceed the column manufacturer's maximum temperature limit.
8. Hold these system temperatures and flows for 30 minutes.
9. Restore the analytical method.

To Remove the EI ion source

WARNING **Be careful! The LTM column, guard column enclosure, GC/MSD transfer line, and inlet may be hot enough to cause burns. If hot, wear heat-resistant gloves to protect your hands.**

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Pliers, long-nose (8710-1094)

Procedure

1 Vent the MSD. See page 97.

2 Open the analyzer chamber. See page 99.

Make sure you use an antistatic wrist strap and take other antistatic precautions before touching analyzer components.

3 Disconnect the seven wires from the ion source. Do not bend the wires any more than necessary (Figure 31 and Table 18).

Table 18 Ion source wires

Wire color	Connects to	Number of leads
Blue	Entrance lens	1
Orange	Ion focus	1
White	Filament 1 (top filament)	2
Red	Repeller	1
Black	Filament 2 (bottom filament)	2

CAUTION Pull on the connectors, not on the wires.

4 Trace the wires for the ion source heater and temperature sensor to the feedthrough board. Disconnect them there.

5 Remove the thumbscrews that hold the ion source in place.

6 Pull the ion source out of the source radiator.

WARNING **The analyzer operates at high temperatures. Do not touch any part until you are sure it is cool.**

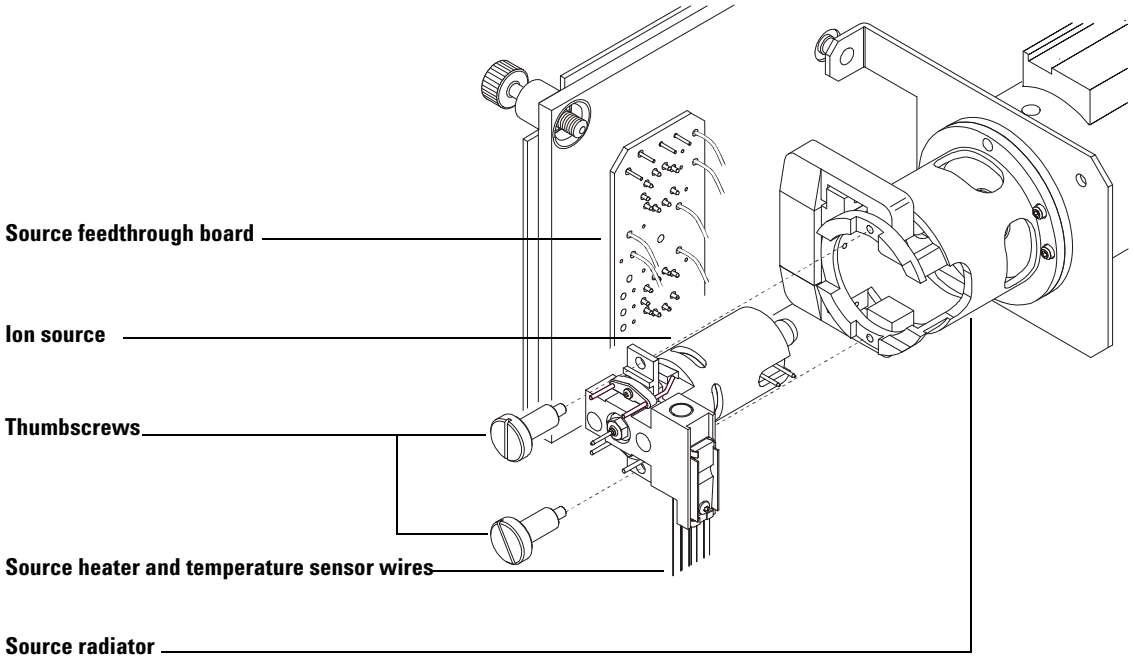


Figure 31 Removing the ion source

To Reinstall the EI Ion Source

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Pliers, long-nose (8710-1094)

Procedure

1. Slide the ion source into the source radiator (Figure 32).
2. Install and hand tighten the source thumbscrews. Do not overtighten the thumbscrews.
3. Connect the ion source wires as shown in "To Close the Analyzer Chamber" . Close the analyzer chamber.

4 Pump down the MSD. See page 106.

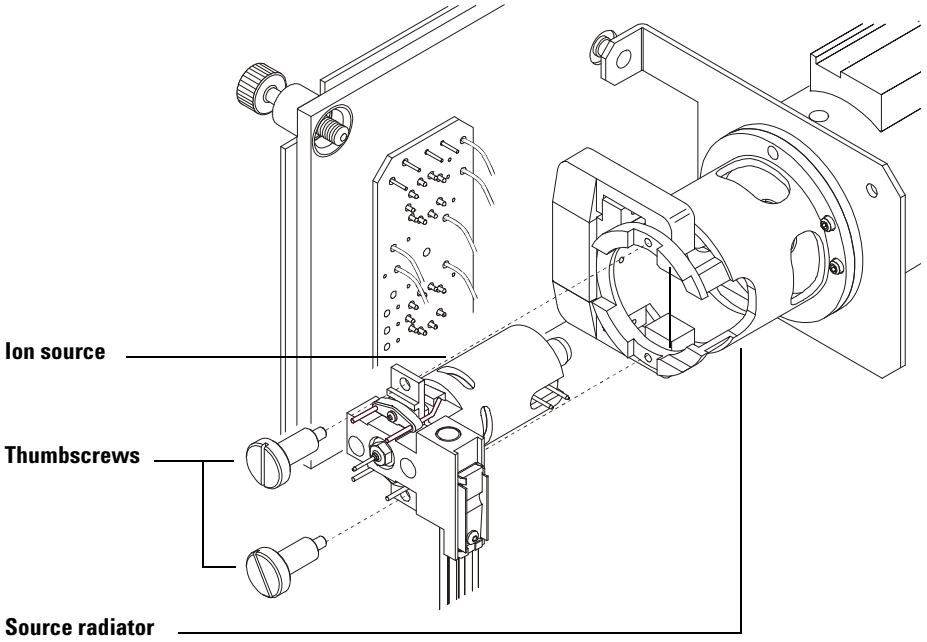


Figure 32 Installing the EI ion source

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