5973 inert
Mass Selective Detector

Hardware Manual

Agilent Technologies

5973 inert
Mass Selective Detector

Hardware Manual

Notices

Manual Part Number

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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.

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Introduction

This manual describes the operation, troubleshooting, and maintenance of the Agilent Technologies 5973 inert Mass Selective Detector (MSD)

5973 inert MSD Version

5973 inert MSDs are equipped with or one of two turbomolecular (turbo) pumps. Chemical Ionization is available for the turbo pump MSDs only. The serial number label displays a product number that tells what kind of MSD you have. In this manual, the term “CI MSD” applies to the EI/PCI/NCI MSD.

Model number	Description
G2578A	Standard turbo EI MSD
G2579A	Performance turbo EI MSD
G2589A	Performance turbo EI/PCI/NCI MSD

About this manual

- The introduction describes general information about the 5973 inert MSD.
- Chapter 1 shows you how to prepare and install a capillary column.
- Chapter 2 describes basic tasks such as setting temperatures, monitoring pressures, tuning, and venting, and pumpdown.
- Chapter 3 describes basic tasks necessary to operate a CI MSD in CI mode.
- Chapter 4 provides a quick reference for identifying causes of poor instrument performance or malfunctions.
- Chapter 5 provides a quick reference for identifying problems unique to CI MSDs.
- Chapter 6 features maintenance procedures.
- Chapter 7 features maintenance procedures unique to CI MSDs.
- Chapter 8 describes operation of the components of the vacuum system.
- Chapter 9 describes the GC/MSD interface, and the CI flow module.
- Chapter 10 describes operation of the analyzer (ion source, mass filter, and detector).
- Chapter 11 describes the electronics that control the MSD.
- Chapter 12 contains illustrated parts identification and part numbers.
- Appendix A is an overview of chemical ionization theory.

For updated information, check the Agilent Technologies Life Sciences/ Chemical Analysis web site at **http://www.agilent.com/chem**.

Other User Information

Additional information is contained in the following documentation:

- *5973N and 5973 inert Mass Selective Detector Hardware Installation Manual*[1]
- *5973N and 5973 inert Mass Selective Detector Site Preparation Guide*[1]
- *6890 Series GC manuals*
- GC accessories (autosampler, etc.) manuals
- G1701DA MSD Productivity ChemStations software manuals and online help
- *5973N and 5973 inert Mass Selective Detector Specifications* (5988-9991EN)
- *Hydrogen Carrier Gas Safety Guide* (5955-5398)[1]
- *5973N and 5973 inert Mass Selective Detector Local Control Panel (LCP) Quick Reference*[1]
- For updated information, see the Agilent Technologies web site at http://www.agilent.com/chem

1. Located on this CD-ROM [5973 and 5973 inert Mass Selective Detector User Information]

The 5973 inert MSD

The 5973 inert MSD is a stand-alone capillary GC detector

The 5973 inert Mass Selective Detector (MSD) is designed for use with the 6890 Series Gas Chromatograph. The MSD features:

- Control panel for locally monitoring and operating the MSD
- One of two different high vacuum pumps
- Rotary vane foreline pump
- Independently heated electron-ionization ion source
- Independently heated hyperbolic quadrupole mass filter
- High-energy dynode (HED) electron multiplier detector
- Independently heated GC/MSD interface
- Chemical ionization (EI/PCI/NCI) model available

Physical description

The 5973 inert MSD is a rectangular box, approximately 42 cm high, 26 cm wide, 65 cm deep. The weight is 26 kg for the standard turbo pump mainframe and 29 kg for the performance turbo pump mainframe. The attached rough pump weighs an additional
11 kg.

The basic components of the instrument are: the frame/cover assemblies, the control panel, the vacuum system, the GC interface, the electronics, and the analyzer.

The control panel allows local monitoring and operation of the MSD

The control panel acts as a local user interface to the MSD. You can perform some basic tasks such as running a tune, a method, or a sequence; and monitor MSD status from the control panel.

An optional gauge controller is available for measuring vacuum

The 5973 inert MSD is equipped with a triode ionization gauge tube. With an 59864B Gauge Controller, the tube can be used to measure pressure (high vacuum) in the vacuum manifold. Installation and operation of the gauge controller is described in this manual.

The gauge controller is ***required*** for chemical ionization (CI) operation.

	5973 inert MSD models and features		
Feature	**G2578A**	**G2579A**	**G2589A**
High vac pump	Standard turbo	Performance turbo	Performance turbo
Optimal He column flow mL/min	1	1 to 2	1 to 2
Maximum recommended gas flow, mL/min[a]	2.0	4	4
Maximum gas flow, mL/min[b]	2.4	6.5	6.5
Max column id	0.32 mm (30m)	0.53 mm (30 m)	0.53 mm (30 m)
CI capability	no	no	PCI/NCI
DIP capability (3rd Party)	yes	yes	no

a. Total gas flow into the MSD: column flow plus reagent gas flow (if applicable).
b. Expect degradation of spectral performance and sensitivity.

CI MSD hardware description

In this manual, the term "CI MSD" applies to the EI/PCI/NCI MSD. The CI hardware allows the 5973 inert MSD to produce high- quality, classical CI spectra, which include molecular adduct ions. A variety of reagent gases can be used.

The 5973 inert CI system adds to the 5973 inert MSD:

- Redesigned EI/CI GC/MSD interface
- CI ion source and interface tip seal
- Reagent gas flow control module
- Bipolar HED power supply (for PCI/NCI MSD ***only***)
- A methane/isobutane gas purifier is provided, and is required. It removes oxygen, water, hydrocarbons, and sulfur compounds.

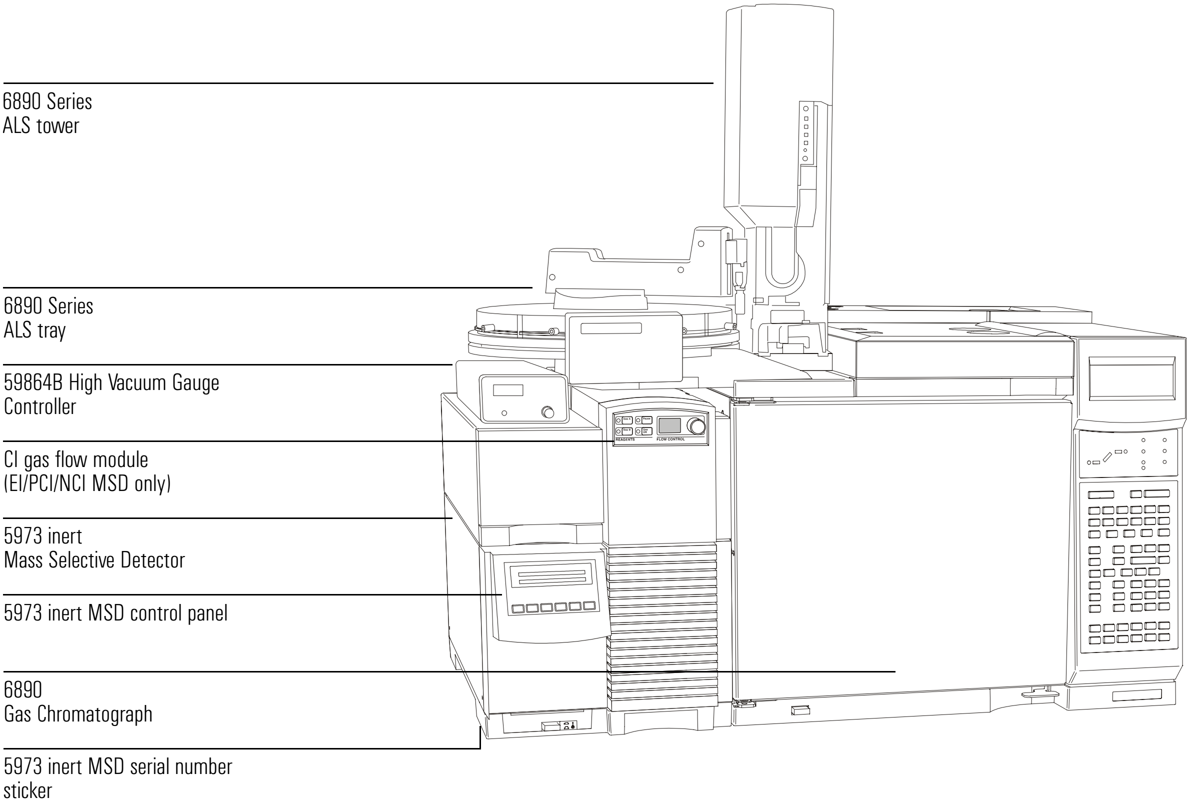
A high vacuum gauge controller (59864B) is ***required*** for the CI MSD.

To achieve the relatively high source pressure required for CI while still maintaining high vacuum in the quadrupole and detector, the MSD CI system has been carefully optimized. Special seals along the flow path of the reagent gas and very small openings in the ion source keep the source gases in the ionization volume long enough for the appropriate reactions to occur.

The EI/CI interface has special plumbing for reagent gas. A spring-loaded insulating seal fits onto the tip of the interface.

Switching back and forth between CI and EI takes less than an hour, although a 1– to 2–hour wait is ***required*** in order to purge the reagent gas lines and bake out water and other contaminants. Switching from PCI to NCI requires about 2 hours for the ion source to cool.

CI MSD hardware description



Important Safety Warnings

Before moving on, there are several important safety notices that you should always keep in mind when using the 5973 inert Mass Selective Detector.

Many internal parts of the MSD carry dangerous voltages

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

- The wiring between the 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 MSD electronics

The printed circuit (PC) boards in the 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 MSD operate at temperatures high enough to cause serious burns. These parts include but are not limited to:

- The inlets
- The oven and its contents
- The detectors
- The column nuts attaching the column to an inlet or detector
- The valve box

You should always cool these areas of the MSD to room temperature before working on them. They will cool faster if you first set the temperature of the heated zone to room temperature. Turn the zone off after it has reached the setpoint. 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 **Be careful when working behind the instrument. During cool-down cycles, the MSD emits hot exhaust which can cause burns.**

WARNING **The insulation around the inlets, detectors, valve box, and the insulation cups 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.**

Hydrogen

Hydrogen gas may be used as carrier gas, and/or as fuel for the FID. When mixed with air, hydrogen can form explosive mixtures.

WARNING

When using hydrogen (H_2) as the carrier gas or fuel gas, be aware that hydrogen gas can flow into the oven and create an explosion hazard. Therefore, be sure that the supply is off until all connections are made, and ensure that the inlet and detector 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.

WARNING

The 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.

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 5973 inert Mass Selective Detector conforms to the following safety standards:

- Canadian Standards Association (CSA): C22.2 No. 1010.1
- CSA/Nationally Recognized Test Laboratory (NRTL): UL 61010A–1
- International Electrotechnical Commission (IEC): 61010–1
- EuroNorm (EN): 61010–1

The 5973 inert Mass Selective Detector 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.

CE

The 5973 inert Mass Selective Detector is designed and manufactured under a quality system registered to ISO 9001.

Information

The Agilent Technologies 5973 inert Mass Selective Detector meets the following IEC (International Electro-technical Commission) classifications: Safety Class I, Transient Overvoltage Category II, Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and is designed for use indoors. 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 5973 Mass Selective Detector 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.

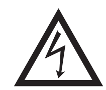
Symbols

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.

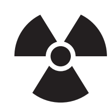
Indicates hazardous voltages.



Indicates earth (ground) terminal.

Indicates explosion hazard.

Indicates radioactivity hazard.



Indicates electrostatic discharge hazard.

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 Certification for Federal Republic of Germany

Sound pressure

Sound pressure Lp < 70 dB am according to ISO 7779:1988.

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.

Cleaning/Recycling the Product

1

Installing GC Columns

How to connect GC columns to the 5973 inert MSD

Installing GC columns

Before you can operate your GC/MSD system, you must select, condition, and install a GC column. This chapter will show you how to install and condition a column. For correct column and flow selection, you must know what type of vacuum system your MSD has. The serial number tag on the lower front of the left side panel shows the model number.

Many types of GC 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 length and flow. Exceeding recommended flow will result in degradation of mass spectral and sensitivity performance.

Remember that column flows vary greatly with oven temperature (unless the GC is set for constant flow). See *To measure column flow linear velocity* (page 58) for instructions on how to measure actual flow in your column. Use the Flow Calculation software to determine whether a given column will give acceptable flow with realistic head pressure.

Feature	**G2578A**	**G2579A**	**G2589A**
High vac pump	Standard turbo	Performance turbo	Performance turbo, EI/ PCI/NCI
Optimal gas flow, mL/min[a]	1	1 to 2	1 to 2
Maximum recommended gas flow, mL/min	2.0	4	4
Maximum gas flow, mL/min[b]	2.4	6.5	4
Max column id	0.32mm (30m)	0.53 mm (30m)	0.53mm (30m)

a. Total gas flow into the MSD: column flow *plus* reagent gas flow (if applicable).
b. Expect degradation of spectral performance and sensitivity.

Conditioning a column before it is installed into 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 cross-linked 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 is also beneficial

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

- Note that the column installation procedure for the 5973 MSDs is different from that for ***all*** previous MSDs. Using the procedure from another instrument will ***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 99.999% 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. Read the *Hydrogen Carrier Gas Safety Guide* (5955-5398) before operating the MSD with hydrogen carrier gas.**

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

To prepare a capillary column for installation

Materials needed:

Capillary column
Column cutter (5181-8836)
Ferrules
- 0.27-mm id, for 0.10-mm id columns (5062-3518)
- 0.37-mm id, for 0.20-mm id columns (5062-3516)
- 0.40-mm id, for 0.25-mm id columns (5181-3323)
- 0.47-mm id, for 0.32-mm id columns (5062-3514)
- 0.74-mm id, for 0.53-mm id columns (5062-3512)

Gloves, clean
- large (8650-0030)
- small (8650-0029)

Inlet column nut (5181-8830)
Magnifying glass
Septum (may be old, used inlet septum)

1 **Slide a septum, column nut, and conditioned ferrule onto the free end of the column.**

The tapered end of the ferrule should point away from the column nut.

2 **Use the column cutter to score the column 2 cm from the end.**

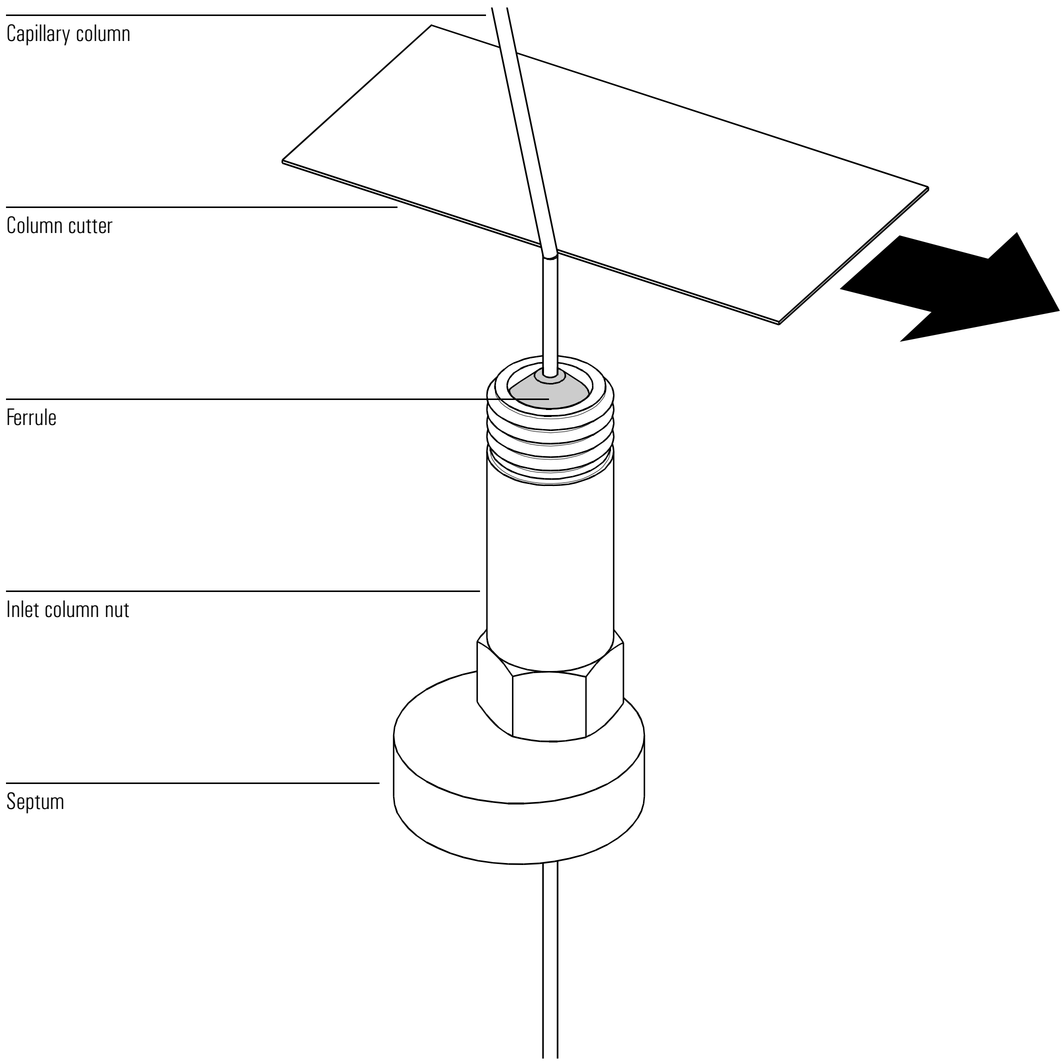
3 **Break off the end of the column.**

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

4 **Inspect the end for jagged edges or burrs.**

If the break is not clean and even, repeat steps 2 and 3.

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


Capillary column
Column cutter
Ferrule
Inlet column nut
Septum

To install a capillary column in a split/splitless inlet

Materials needed:

Gloves, clean
  large (8650-0030)
  small (8650-0029)
Metric ruler
Wrench, open-end, 1/4-inch × 5/16-inch (8710-0510)

To install columns in other types of inlets, refer to your 6890 Series Gas Chromatograph Operating Manual.

1 **Prepare the column for installation (page 32).**

2 **Position the column so it extends 4 to 6 mm past the end of the ferrule.**

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

4 **Insert the column in the inlet.**

5 **Slide the nut up the column to the inlet base and finger tighten the nut.**

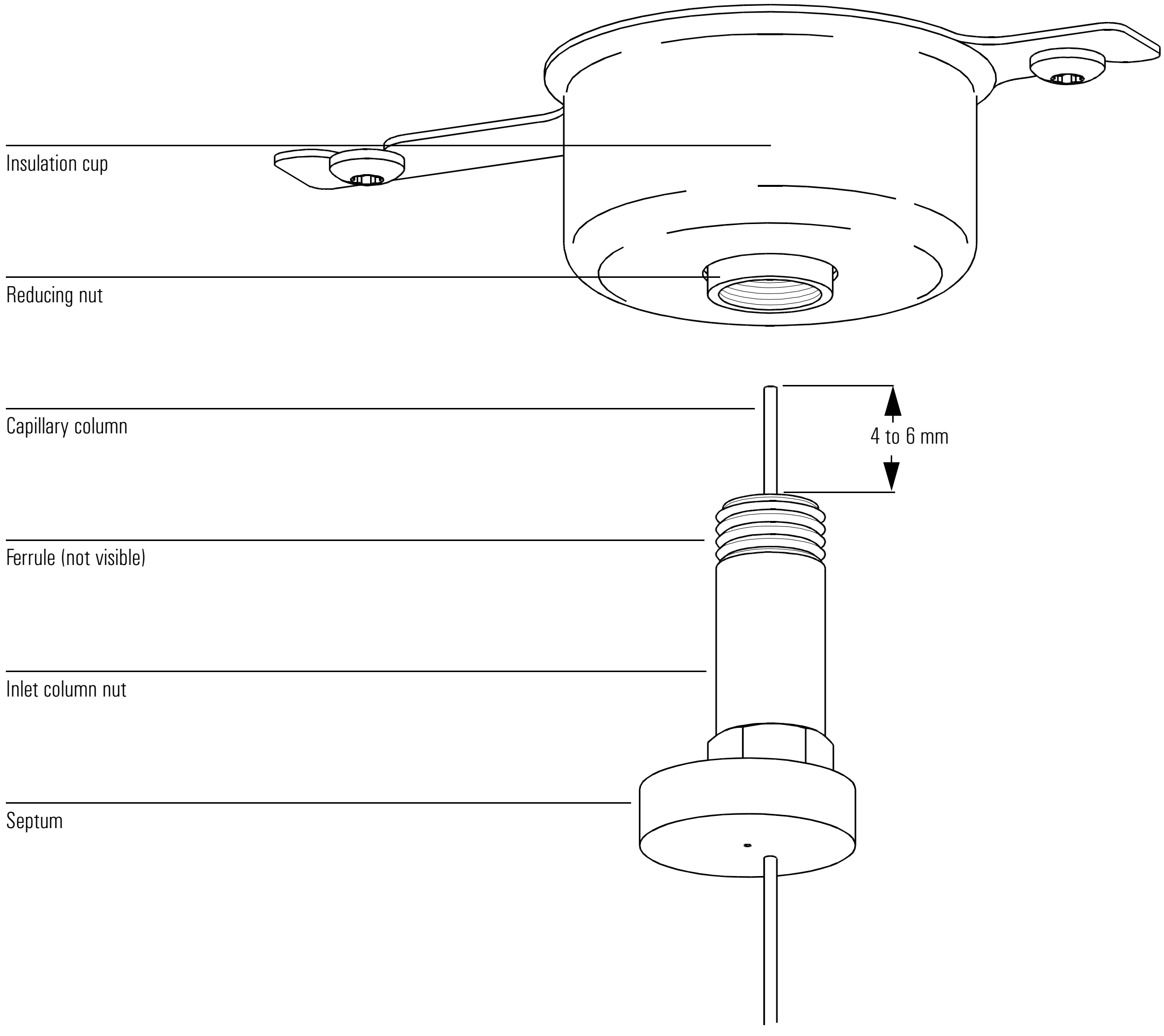
6 **Adjust the column position so the septum is even with the bottom of the column nut.**

7 **Tighten the column nut an additional 1/4 to 1/2 turn.**

The column should not slide with a gentle tug.

8 **Start carrier gas flow.**

9 **Verify flow by submerging the free end of the column in isopropanol. Look for bubbles.**


Insulation cup
Reducing nut
Capillary column
4 to 6 mm
Ferrule (not visible)
Inlet column nut
Septum

To condition a capillary column

Materials needed: Carrier gas, (99.999% pure or better)
Wrench, open-end, 1/4-inch × 5/16-inch (8710-0510)

WARNING **Do not condition your capillary column with hydrogen. Hydrogen accumulation in the GC oven 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.**

1 **Install the column in the GC inlet, page 34.**

2 **Allow the carrier gas to flow through the column for 5 minutes without heating GC oven.**

3 **Ramp the oven temperature at 5°C/minute to 10°C above your highest analytical temperature.**

4 **Once the oven temperature exceeds 80°C, inject 5 µL methanol into GC; repeat two more times at 5-minute intervals.**

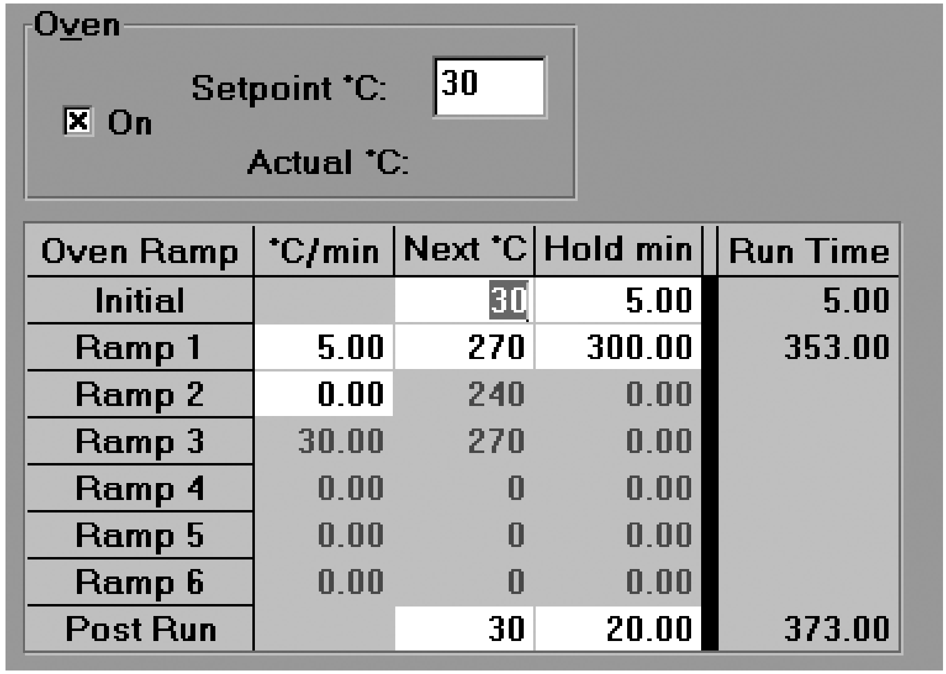
   This will help remove any contamination from the column before it is installed into the GC/MSD interface.

CAUTION Do not exceed the maximum temperature rating of the column.

5 **Hold this temperature. Allow the carrier gas to flow for several hours.**

6 **Return the GC oven 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.


Oven
Setpoint °C: 30
On
Actual °C:

Oven Ramp	°C/min	Next °C	Hold min	Run Time
Initial		30	5.00	5.00
Ramp 1	5.00	270	300.00	353.00
Ramp 2	0.00	240	0.00	
Ramp 3	30.00	270	0.00	
Ramp 4	0.00	0	0.00	
Ramp 5	0.00	0	0.00	
Ramp 6	0.00	0	0.00	
Post Run		30	20.00	373.00

To install a capillary column in the GC/MSD interface

Materials needed:

Column cutter (5181-8836)
Ferrules
- 0.3-mm id, for 0.10-mm id columns (5062-3507)
- 0.4-mm id, for 0.20- and 0.25-mm id columns (5062-3508)
- 0.5-mm id, for 0.32-mm id columns (5062-3506)
- 0.8-mm id, for 0.53-mm id columns (5062-3538)

Flashlight
Hand lens (magnifying glass)
Gloves, clean
- large (8650-0030)
- small (8650-0029)

Interface column nut (05988-20066)
Safety glasses
Wrench, open-end, 1/4-inch × 5/16-inch (8710-0510)

CAUTION Note that the column installation procedure for the 5973 MSDs is different from that for all previous MSDs. Using the procedure from another instrument may result in poor sensitivity and possible damage to the MSD.

1 **Condition the column (page 36).**

2 **Vent the MSD (page 64) and open the analyzer chamber (page 66).**

Be sure you can see the end of the GC/MSD interface.

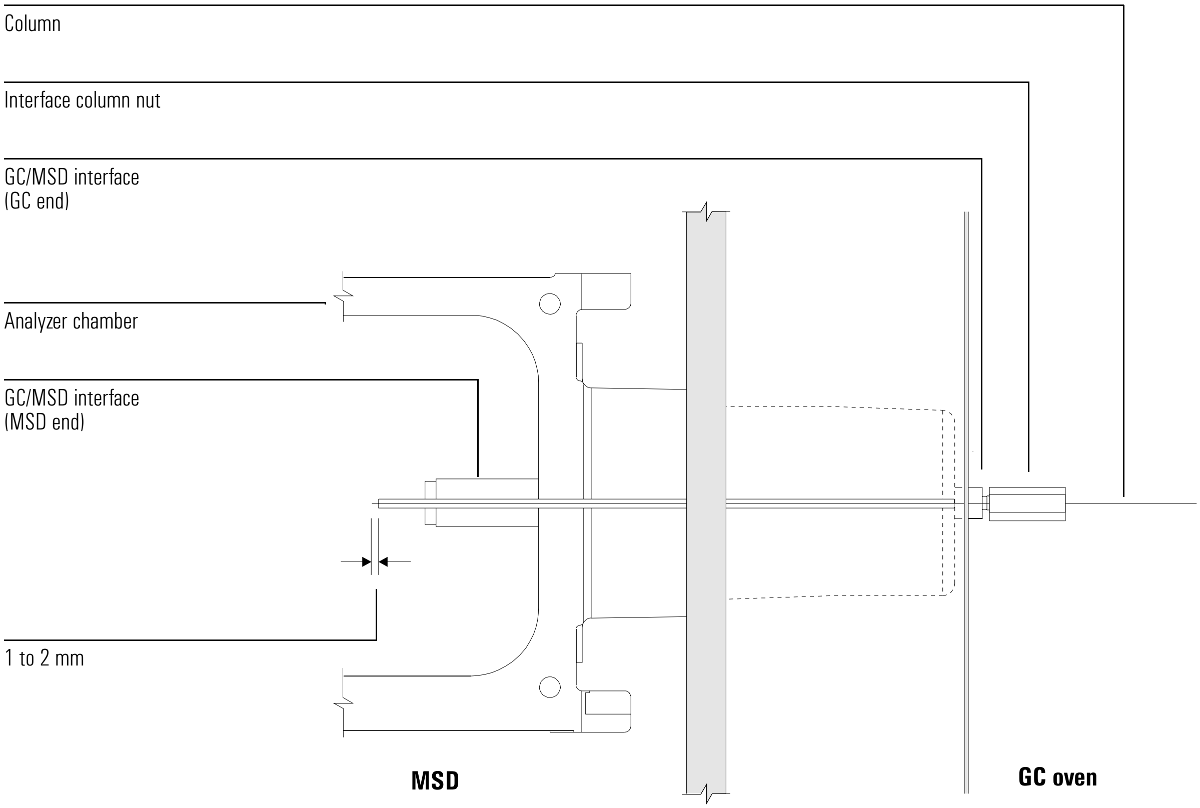
3 **Slide an interface nut and conditioned ferrule onto the free end of the GC column.**

The tapered end of the ferrule must point towards the nut.

4 **Slide the column into the GC/MSD interface until you can pull it out through the analyzer chamber.**

5 **Break 1 cm off the end of the column (page 32).**

Do not let any column fragments fall into the analyzer chamber. They could damage the turbo 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 GC/MSD 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.

To install a capillary column using the installation tool

Materials needed:

Column cutter (5181-8836)
Column installation tool (*not supplied with the MSD)* (G1099-20030)
Ferrules
  0.3-mm id, for 0.10-mm id columns (5062-3507)
  0.4-mm id, for 0.20- and 0.25-mm id columns (5062-3508)
  0.5-mm id, for 0.32-mm id columns (5062-3506)
  0.8-mm id, for 0.53-mm id columns (5062-3538)
Gloves, clean
  large (8650-0030)
  small (8650-0029)
Interface column nut (05988-20066)
Septum (may be old, used inlet septum)
Wrenches, open-end, 1/4-inch × 5/16-inch (8710-0510) – 2 required

Note: The column installation tool is not recommended for applications requiring optimal sensitivity performance. See “To install a capillary column without the installation tool”, page 38.

1 **Vent the MSD. See page 64.**

2 **Slide a septum, interface column nut, and conditioned ferrule onto the free end of the column.**

The tapered end of the ferrule should point toward the nut.

3 **Insert the column into the column installation tool.**

Slide the column through until the end extends past the end of the tool.

4 **Cut 1 cm off the end of the column (page 32).**

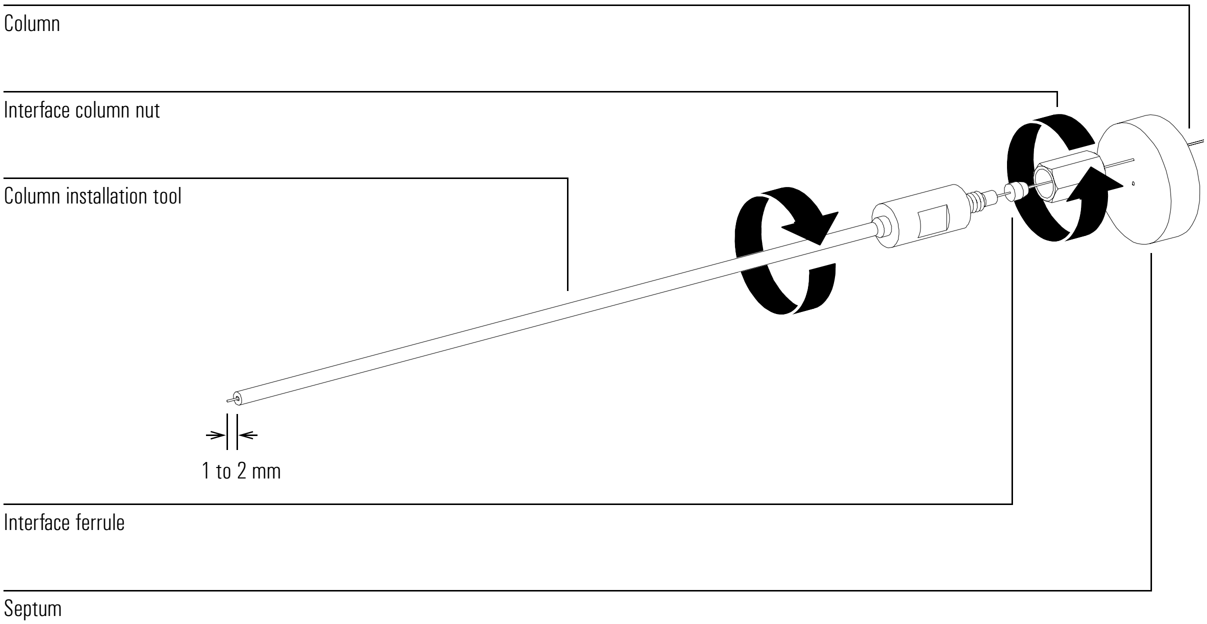
5 **Position the column so that 1 to 2 mm extends past the end of the tool. Hand tighten the nut.**

6 **Slide the septum to touch the end of the nut.**

The septum will help assure that the position is correct.

7 **Use two wrenches to tighten the nut 1/4 to 1/2 turn.**

The column should not slide when tugged ***gently***.



8 **Remove the column and nut from the installation tool.**

The total length from the septum to the end of the column is 176 mm.

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

10 **Insert the column into the GC/MSD interface.**

11 **Tighten the nut 1/4 to 1/2 turn.**

Check tightness after one or two heat cycles.

12 **Pump down the MSD.**

CAUTION The column installation tool must be kept ***clean*** to prevent contaminating the column and the ion source. Keep it in its storage tube, and clean it by flushing with methanol after each use.

2

Operating the MSD

How to perform some basic operating procedures for the MSD

Operating the MSD

Operation of the MSD from the data system

The software performs tasks such as pumpdown, 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.

Operation of the MSD from the control panel

You can use the 5973 inert MSD control panel to perform many of the same tasks that the ChemStation can perform. See the *5973N and 5973 inert Mass Selective Detector Local Control Panel (LCP) Quick Reference (*G2589-90072) for more information.

Some conditions must be met before you turn on the MSD

Verify the following ***before*** you turn on or attempt to operate the 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 MSD is connected to a grounded power source.
- The GC/MSD interface extends into the GC oven.
- A conditioned capillary column is installed in the GC inlet and in the GC/MSD interface.
- The GC is on, but the heated zones for the GC/MSD interface, the injection port, and the oven are off.
- Carrier gas of at least 99.999% purity is plumbed to the GC 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.
- The foreline pump exhaust is properly vented.

WARNING **The exhaust from the foreline pump contains solvents and the chemicals you are analyzing. It also contains traces of pump oil. The supplied oil trap stops only 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. Install a hose (11 mm id) to take the foreline pump exhaust outside or to a fume (exhaust) hood.**

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 the *Hydrogen Carrier Gas Safety Guide* (5955-5398) before operating the MSD with hydrogen carrier gas.**

The data system or control panel help you pump down the MSD

Pumpdown 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 contains a program that monitors and displays system status during pumpdown. When the pressure is low enough, the program turns on the ion source and mass filter heaters. It also prompts you to turn on the GC/MSD interface heater. The 5973 inert MSD will shutdown if it cannot pump down correctly.

Monitoring the pressure in the MSD

The data system displays the turbo pump motor speed for the turbo pump MSDs.

Each MSD is equipped with a triode ionization gauge tube. If your MSD is also equipped with an 59864B Gauge Controller, the triode gauge can measure the pressure in the analyzer chamber. The high vacuum pressure measured by the triode gauge cannot be monitored through the data system. It is displayed on the gauge controller.

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 control panel.

The GC/MSD interface heater is powered and controlled by the Thermal Aux #2 heated zone of the 6890 Series GC. The GC/MSD interface temperature can be set and monitored from the data system or from the GC keypad.

Column flow is controlled through the data system

Carrier gas flow through the GC column is controlled by head pressure in the GC. For a given head pressure, the column flow will decrease as the GC oven temperature increases. With electronic pneumatic control (EPC) set to **Const Flow** (constant flow), the same column flow is be maintained regardless of oven 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 58..

The data system aids in venting

A program in the data system guides you through the venting process. It switches off the GC and MSD heaters and the diffusion pump heater or 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 diffusion pump will backstream vaporized pump fluid onto the analyzer if the MSD is vented before the diffusion pump has fully cooled. 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. 100°C is still 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 MSD power. If the foreline pump is off, hydrogen will accumulate in the MSD and an explosion may occur. Read the *Hydrogen Carrier Gas Safety Guide* (5955-5398) 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.

CAUTION

Do not vent or shut off the power on a diffusion pump MSD while the pump is hot.
Do not vent while the turbo pump is still spinning at more than 50%.

CAUTION

Do not exceed the maximum recommended total gas flow. See "5973 inert MSD models and features" on page 18.

Moving or storing the MSD requires special care

The best way to keep your MSD functioning properly is to keep it pumped down and hot, with carrier gas flow. If you plan to move or store your MSD, a few additional precautions are required. The MSD must remain upright at all times; this requires special caution when moving. The MSD should not be left vented to atmosphere for long periods.

To view MSD analyzer temperature and vacuum status

Software changes

The software is revised periodically. If the steps in this procedure do not match your MSD ChemStation software, refer to the manuals and online help supplied with the software for more information.

See also

You can also use the Control Panel to perform this task. See the *5973N and 5973 inert Mass Selective Detector Local Control Panel (LCP) Quick Reference Guide* (G2589-90072) for more information.

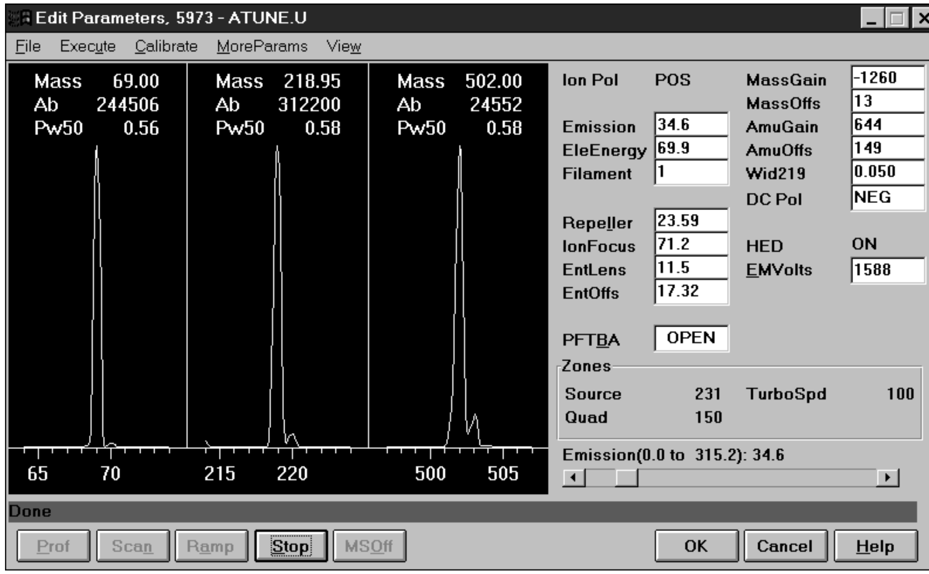
1 **In Instrument Control view, select Edit MS Tune Parameters from the Instrument menu.**

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


Edit Parameters, 5973 - ATUNE.U
File Execute Calibrate MoreParams View
Mass 69.00
Ab 244506
Pw50 0.56
Mass 218.95
Ab 312200
Pw50 0.58
Mass 502.00
Ab 24552
Pw50 0.58
65 70 215 220 500 505
Ion Pol POS
Emission 34.6
EleEnergy 69.9
Filament 1
Repeller 23.59
IonFocus 71.2
EntLens 11.5
EntOffs 17.32
PFTBA OPEN
MassGain -1260
MassOffs 13
AmuGain 644
AmuOffs 149
Wid219 0.050
DC Pol NEG
HED ON
EMVolts 1588
Zones
Source 231
Quad 150
TurboSpd 100
Emission(0.0 to 315.2): 34.6
Done
Prof Scan Ramp Stop MSOff
OK Cancel Help

To set monitors for MSD temperature and vacuum status

Monitors display 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 value varies beyond a user-determined limit from the parameter setpoint. This procedure describes how to add monitors to your instrument control view.

Software changes

The software is revised periodically. If the steps in this procedure do not match your MSD ChemStation software, refer to the manuals and online help supplied with the software for more information.

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.**

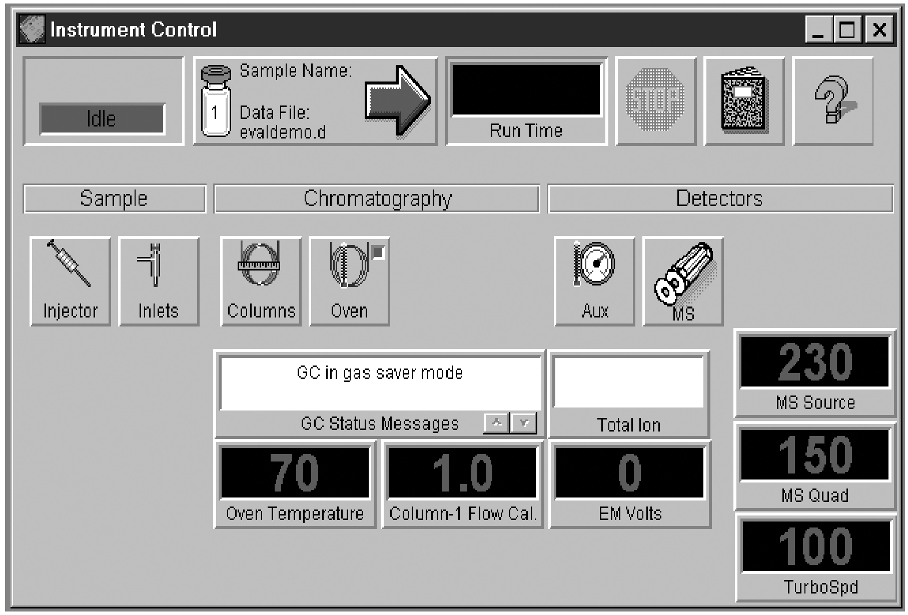
6 **Click OK.**

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.

7 **Click and drag each monitor to the desired position.**

See the accompanying illustration for an example of arranging the monitors.

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


Instrument Control
Idle
Sample Name:
Data File:
evaldemo.d
1
Run Time
Sample
Chromatography
Detectors
Injector
Inlets
Columns
Oven
Aux
MS
GC in gas saver mode
GC Status Messages
Total Ion
230
MS Source
150
MS Quad
100
TurboSpd
70
Oven Temperature
1.0
Column-1 Flow Cal.
0
EM Volts

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.

Software changes

The software is revised periodically. If the steps in this procedure do not match your MSD ChemStation software, refer to the manuals and online help supplied with the software for more information.

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

2 **Select Temperatures from the MoreParams menu.**

3 **Type the desired Source and Quad (mass filter) temperatures in the setpoint fields and click OK.**

See Table 1 on page 53 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 lower than that of an adjacent zone.

CAUTION

Do not exceed 200°C for the quadrupole or 300°C for the source.

4 **Click OK in the Edit Parameters window to apply the new temperature setpoints.**

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.**

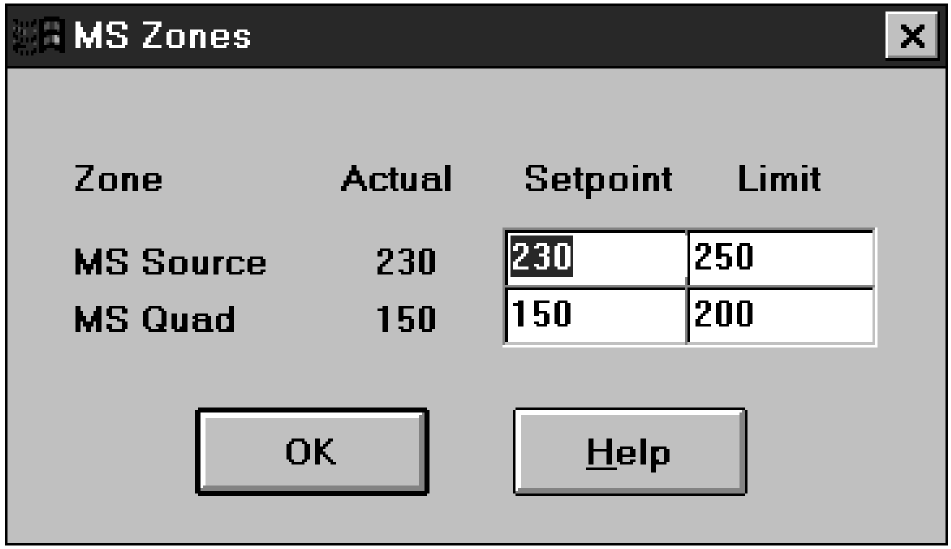


Table 1 **Recommended temperature settings**

	EI operation	PCI operation	NCI operation
MS Source	230	250	150
MS Quad	150	150	150

To set the GC/MSD interface temperature from the PC

Software changes

The software is revised periodically. If the steps in this procedure do not match your MSD ChemStation software, refer to the manuals and online help supplied with the software for more information.

See also

You can also use the Control Panel to perform this task. See the *5973N and 5973 inert Mass Spectrometer Detector Local Control Panel (LCP) Quick Reference* (G2589-90072) for more information.

1 **Select Instrument Control from the View menu.**

2 **Click the Aux button to display the Instrument | Edit | Aux: (6890) window.**

3 **Verify that MSD is selected under Type and Thermal Aux #2 is selected under Aux Channel.**

4 **Turn the heater on, and type the setpoint in the Next °C column. Do *not* set temperature ramps.**

5 **The typical setpoint is 280°C.**

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

C A U T I O N

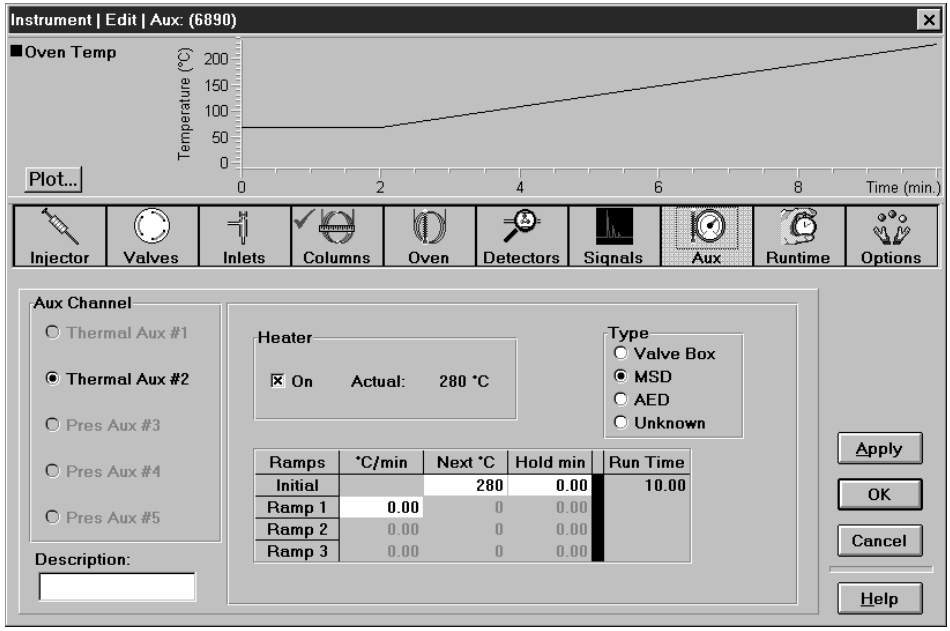
Never exceed the maximum temperature for your column.

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

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

C A U T I O N

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 GC oven.


Instrument | Edit | Aux: (6890)
Oven Temp
Temperature (°C)
Plot...
Time (min.)
Injector
Valves
Inlets
Columns
Oven
Detectors
Signals
Aux
Runtime
Options
Aux Channel
Thermal Aux #1
Thermal Aux #2
Pres Aux #3
Pres Aux #4
Pres Aux #5
Description:
Heater
On
Actual: 280 °C
Type
Valve Box
MSD
AED
Unknown
Ramps
°C/min
Next °C
Hold min
Run Time
Initial
Ramp 1
Ramp 2
Ramp 3
280
0.00
10.00
Apply
OK
Cancel
Help

To monitor high vacuum pressure

Materials needed:

Gauge controller (59864B)
Triode ionization gauge cable (8120-6573)

WARNING **Never connect or disconnect the cable from the triode gauge tube while the MSD is under vacuum. Risk of implosion and injury due to broken glass exists.**

WARNING **If you are using hydrogen as a carrier gas, do not turn on the triode gauge tube if there is any possibility that hydrogen has accumulated in the analyzer chamber. The triode gauge filament can ignite hydrogen. Read the *Hydrogen Carrier Gas Safety Guide* (5955-5398) before operating the MSD with hydrogen carrier gas.**

1 **Connect the gauge controller to the ionization gauge tube (page 73).**

2 **Start up and pump down the MSD (page 70).**

3 **Switch on the power switch on the back of the gauge controller.**

4 **Press and release the GAUGE button.**

After a few seconds, the pressure should be displayed.

Pressure is displayed in the format **X.X – X** where **– X** is the base 10 exponent. Units are Torr.

The gauge controller will not turn on if the pressure in the MSD is above approximately 8×10^{-3} Torr. The gauge controller will display all 9s and then go blank. The triode gauge tube can measure pressures between approximately 8×10^{-3} and less than 2×10^{-6} Torr. The gauge controller is calibrated for nitrogen, but all pressures listed in this manual are for helium. Refer to the manual for the 59864B for information on relative sensitivity to different gases.

The largest influence on operating pressure in EI mode is the carrier gas (column) flow. The following table 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 2 **Typical MSD pressure readings for various helium carrier gas flow rates**

Turbo pump MSDs		
Column flow (ml/min)	Triode gauge reading (Torr), *Performance* turbo pump	Triode gauge reading (Torr), *Standard* turbo pump
1.0	1.5×10^{-5}	4.0×10^{-5}
2.0	3.0×10^{-5}	8.0×10^{-5}
2.4	3.5×10^{-5}	1.0×10^{-4} (Not recommended)
3.0	4.5×10^{-5}	Not supported
4.0	5.0×10^{-5}	Not supported

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.

If the pressure rises above approximately 8×10^{-3} Torr, the gauge controller will turn off the triode gauge tube. The gauge tube ***does not*** turn back on automatically.

To measure column flow linear velocity

Materials needed: Syringe

1 **Set Data Acquisition for splitless manual injection and selected ion monitoring (SIM) of *m/z* 28.**

2 **Press the Prep Run button on the GC keypad.**

3 **Inject 1 µl of air into the injection port and press the Start Run button.**

4 **Wait until a peak elutes at *m/z* 28.**

Note the retention time.

5 **Calculate the average linear velocity.**

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

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 value to verify the MSD ChemStation flow calculations (page 59).**

If the numbers disagree, click the **Change** button 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:

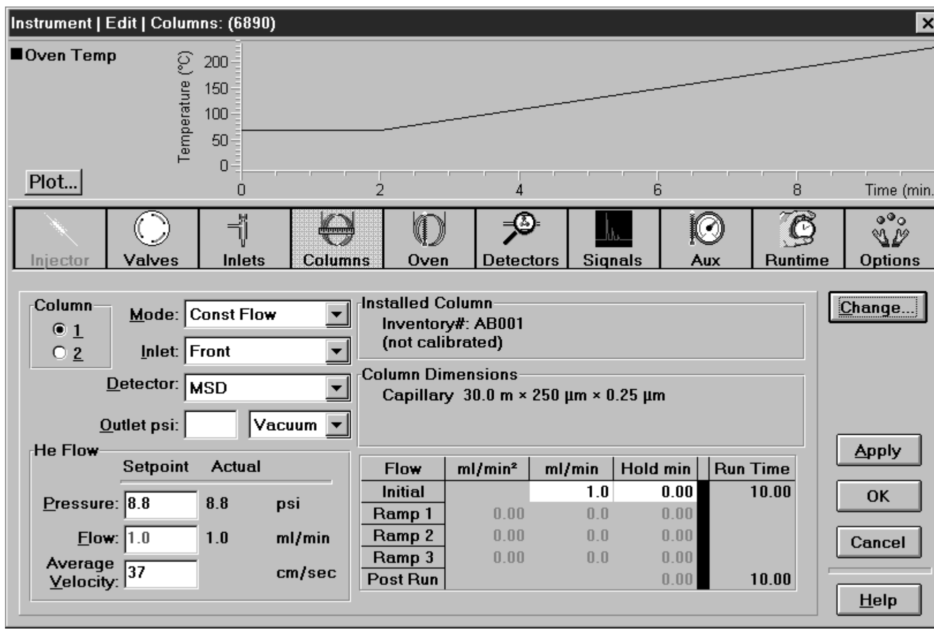
D = internal column diameter in millimeters
L = the column length in meters
t = the retention time in minutes

To calculate column flow

1 **In the Instrument Control view, click the Columns icon.**

2 **Check that the correct column dimensions are entered.**

3 **Type the desired value in the pressure field.**



4 **If the Average Velocity displayed is different from that obtained on page 58, click the Change button to calibrate the column dimensions.**

To tune the MSD

Software changes *The software is revised periodically. If the steps in this procedure do not match your MS ChemStation software, refer to the manuals and online help supplied with the software for more information.*

See also You can also use the Control Panel to run the autotune that is currently loaded in the PC memory. See the *5973N and 5973 inert Mass Selective Detector Local Control Panel (LCP) Quick Reference (*G2589-90072) for more information.

1 **In the Instrument Control View, select Perform MS Autotune from the Instrument menu.**

2 **Select the tune program you wish to use.**

The tune will start immediately. For most applications, **Autotune** gives the best results. **Standard Tune** is not recommended, as it may reduce sensitivity. **Quick Tune** is used to adjust peak width, mass assignment, and abundance, without changing ion ratios. If your system is configured for chemical ionization (CI), you will be able to access the CI Tunepanel from this box. Always tune the MSD with the same GC oven temperature and column flow, and the same analyzer temperatures that will be used for data acquisition.

3 **Wait for the tune to complete and to generate the report.**

Save your tune reports. To view history of tune results, select **View Tunes...** under the Qualify menu.

4 **To manually tune your MSD or to perform special autotunes, select Manual Tune from the View menu.**

In the Tune and Vacuum Control viewTune and Vacuum Control view, you can manually adjust most tune parameters to suit special needs.

From the Tune menu, in addition to the tunes available from Instrument Control, you can select special autotunes for specific spectral results: **DFTPP Tune**, **BFB Tune**, or **Target 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 (8500-5441)

Verify the tune performance

1 **Verify that the system has been pumping down for at least 60 minutes.**

2 **Set the GC oven temperature to 150°C, and the column flow to 1.0 ml/min.**

3 **In the Instrument Control view, select Checkout Tune from the Qualify menu.**

The software will perform an autotune and print out the report.

4 **When the autotune has completed, save the method, and then select Tune Evaluation from the Qualify 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 Qualify 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 print out.

Verify that rms signal-to-noise ratio meets the published specification. See the *5973N and 5973 inert Mass Selective Detector Local Control Panel (LCP) Quick Reference (*G2589-90072).

To remove the MSD covers

Materials needed: Screwdriver, TORX T-15 (8710-1622)

The analyzer cover is removed for venting and for many maintenance procedures. The lower MSD cover is removed to check the fluid level in the diffusion pump and for a few maintenance procedures. If you need to remove one of the MSD covers, follow these procedures:

Analyzer cover

1 **Grasp the front of the analyzer cover and lift up enough to unlatch the five front tabs.**

2 **Reach back and grasp the back edge of the analyzer cover.**

3 **Pull forward to disengage the rear spring latch.**
It may take a firm pull to disengage the latch.

To reinstall the analyzer cover, reverse these steps.

Lower MSD cover

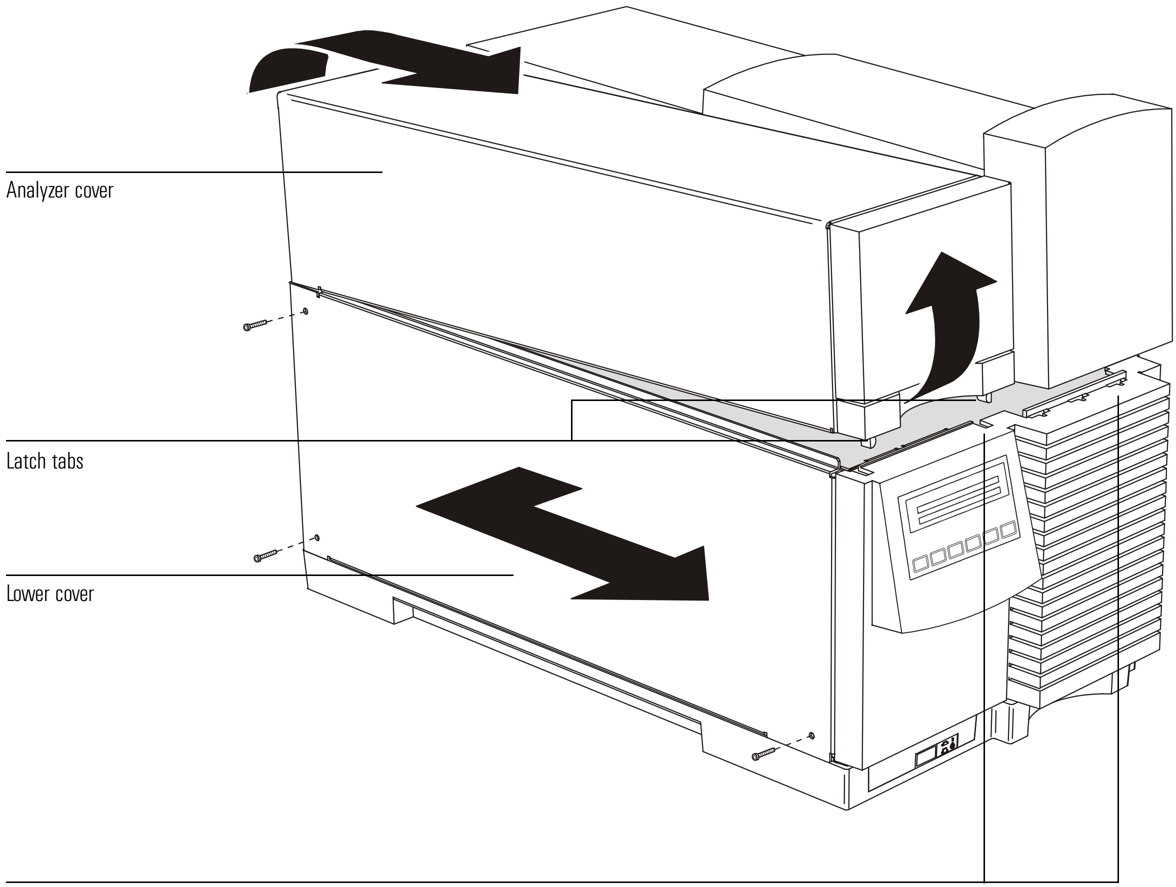
1 **Remove the analyzer cover.**

2 **Remove the 3 screws that hold the lower MSD cover in place.**

3 **Pull the cover left slightly to disengage the two right side tabs and then pull it straight forward.**

To reinstall the lower MSD cover, reverse these steps.

WARNING **Do not remove any covers other than the upper and lower MSD covers. Dangerous voltages are present under other covers.**



C A U T I O N	Do not use excessive force, or the plastic tabs that hold the cover to the mainframe will break off.

To vent the MSD

Firmware changes

The firmware is revised periodically. If the steps in this procedure do not match your MSD control panel, refer to the manuals and online help supplied with the software, or the 5973N and 5973 inert Mass Selective Detector Local Control Panel (LCP) Quick Reference (G2589-90072) *for more information.*

1. **If your system is equipped with a gauge controller, switch off the triode gauge controller.**
2. **Before venting a CI MSD, press the Gas Off button (turns off the reagent gas flow and closes the isolation valve.)**

WARNING **On a CI MSD, the Gas Off light must be on when the MSD is venting.**

3. **Select Vent from the from the Vacuum menu in the software. Follow the instructions presented.**
4. **Set the GC/MSD interface heater and the GC oven temperatures to ambient (25°C).**

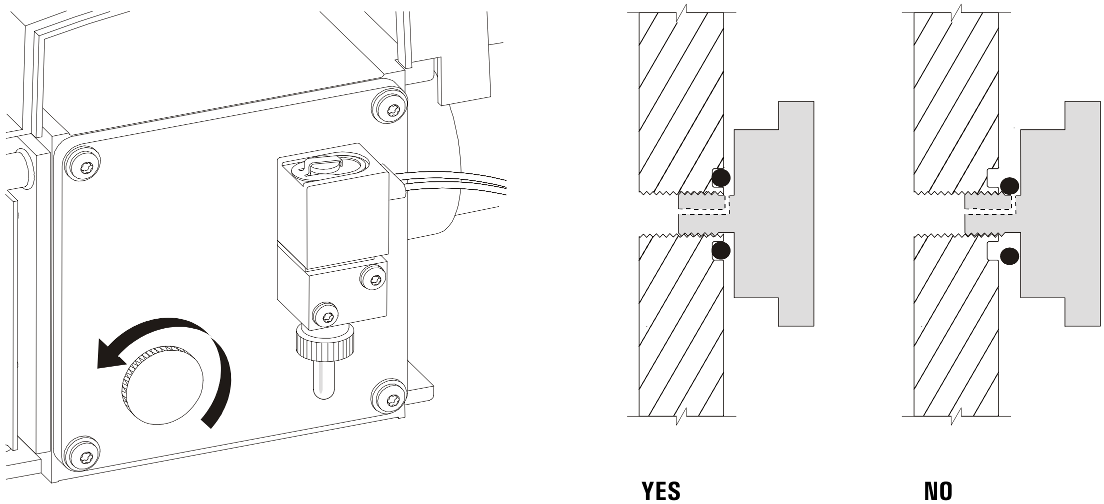
WARNING **If you are using hydrogen as a carrier gas, the carrier gas flow must be off before turning off the MSD power. If the foreline pump is off, hydrogen will accumulate in the MSD and an explosion may occur. Read the *Hydrogen Carrier Gas Safety Guide* (5955-5398) before operating the MSD with hydrogen carrier gas.**

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

5. **When prompted, turn off the MSD power switch.**
6. **Unplug the MSD power cord.**

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

7. **Remove the analyzer cover (page 62).**



8 **Turn the vent valve knob 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 Top 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, anti-static
- 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 anti-static wrist strap and take other anti-static precautions (see page 168) before you open the analyzer chamber.

1 **Vent the MSD (page 64).**

2 **Disconnect the side board control cable and the source power cable from the side board.**

3 **Loosen the side plate thumbscrews, if they are fastened.**

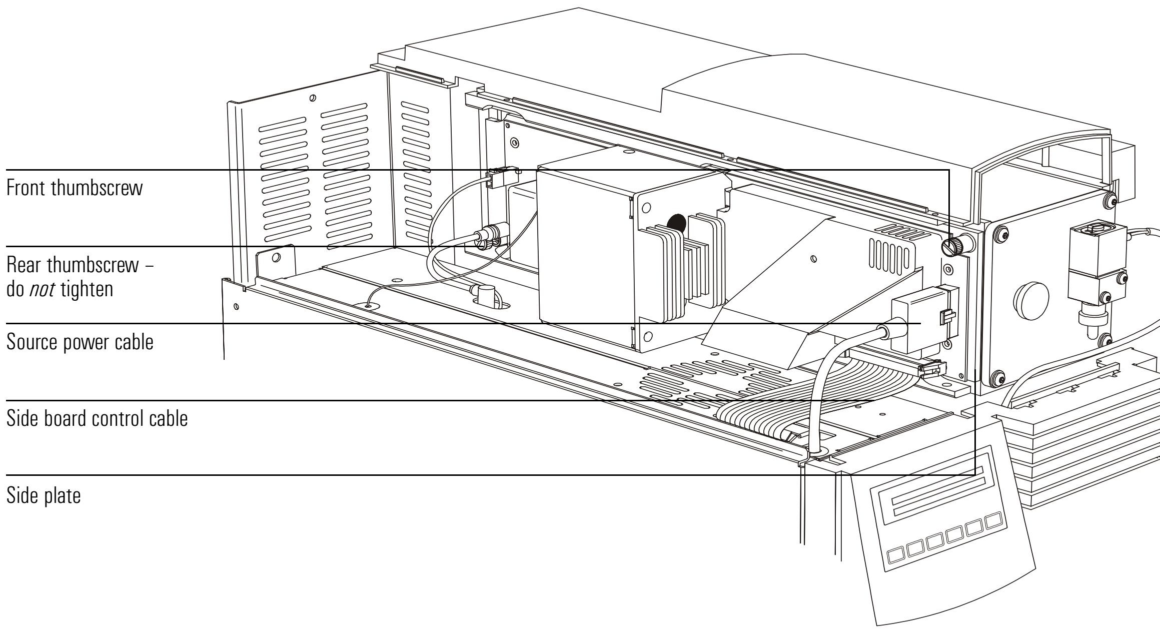
The rear side plate thumbscrew should be unfastened during normal use. It is only fastened during shipping. The front side plate thumbscrew should only be fastened for CI operation or if hydrogen or other flammable or toxic substances are used for carrier gas.

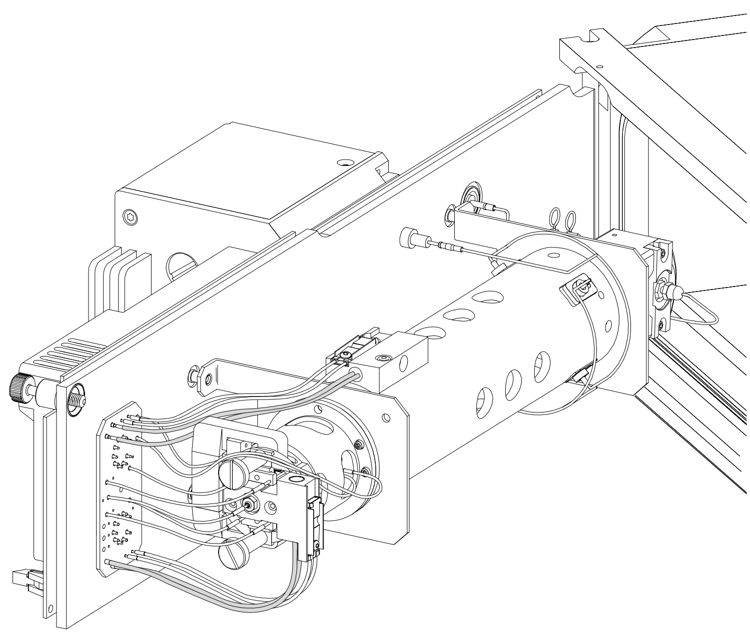
4 ***Gently* swing the side plate out.**

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.

CAUTION 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.


Front thumbscrew
Rear thumbscrew – do *not* tighten
Source power cable
Side board control cable
Side plate



To close the analyzer chamber

Materials needed: Gloves, clean, lint-free
large (8650-0030)
small (8650-0029)

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

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. See page 197 for instructions for lubricating the side plate O-ring.

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 (page 70).

7 *Gently* hand tighten the front side plate thumbscrew.

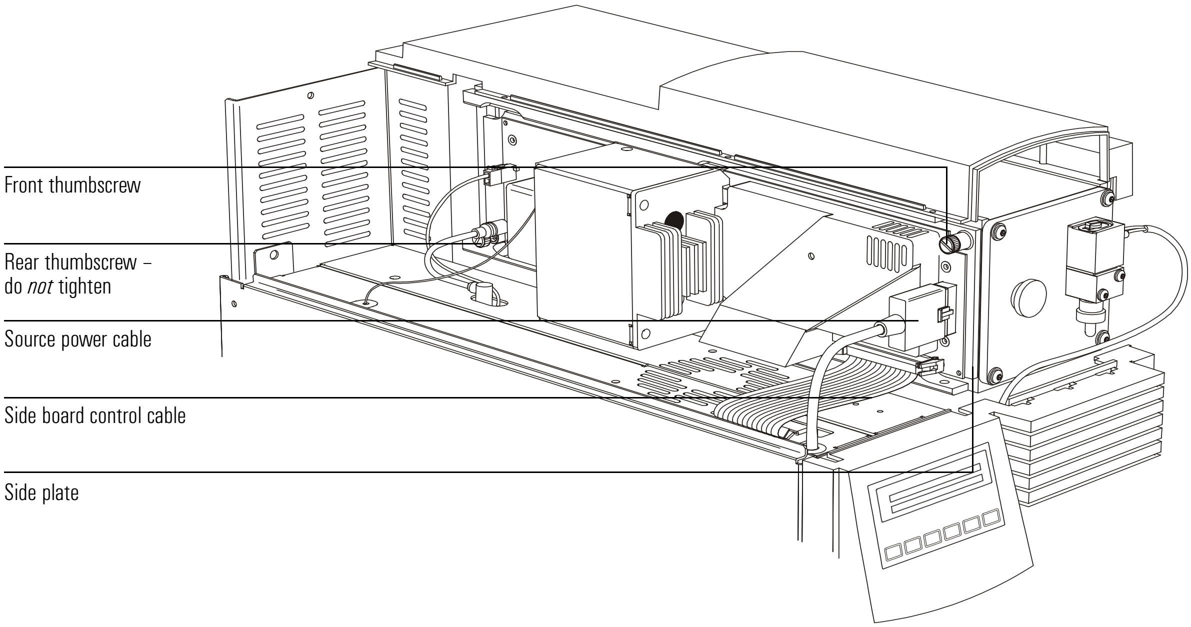
This is only necessary for CI MSDs, or if hydrogen or other flammable or toxic substance is used for carrier gas.

WARNING **This thumbscrew must be fastened for CI operation or 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, reinstall the analyzer cover.

Wait until after pumpdown to reinstall the analyzer cover.


Front thumbscrew
Rear thumbscrew – do *not* tighten
Source power cable
Side board control cable
Side plate

To pump down the MSD

Software changes

The software is revised periodically. If the steps in this procedure do not match your MSD ChemStation software, refer to the manuals and online help supplied with the software for more information.

See also

You can also use the Control Panel to perform this task. See the *5973N and 5973 inert Mass Selective Detector Local Control Panel (LCP) Quick Reference* (G2589-90072) for more information.

WARNING

Make sure your MSD meets all the conditions listed in the introduction to this chapter (page 44) 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 the *Hydrogen Carrier Gas Safety Guide* (5955-5398) before operating the MSD with hydrogen carrier gas.

1 **Plug in the MSD power cord.**

2 **Select Diagnostics/Vacuum Control from the View menu.**

Select Pump Down from the Vacuum menu.

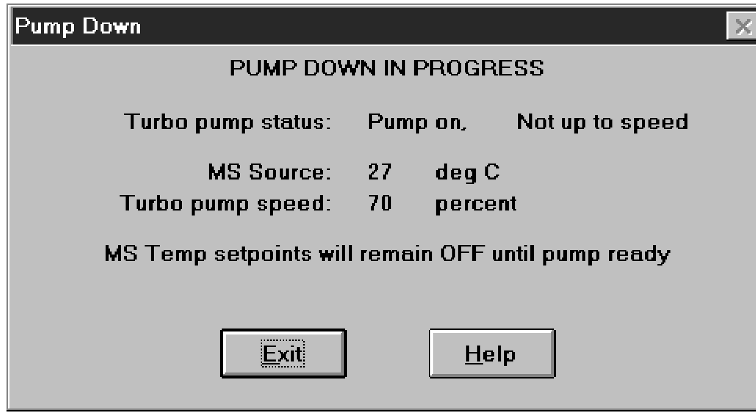
3 **When prompted, switch on the MSD**

4 **Press lightly on the side board to ensure a correct seal.**

Press on the metal box on the side board.

The rough 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.

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



CAUTION

Within 10 to 15 minutes the turbo pump speed should be up to 80%. The turbo 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.

6 **When prompted, turn on the GC/MSD interface heater and GC oven. 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.

7 **After the message Okay to run appears, wait two hours for the MSD to reach thermal equilibrium.**

Data acquired before the MSD has reached thermal equilibrium may not be reproducible.

8 **Reinstall the MSD top cover.**

The top cover was removed during the vent procedure.

To pump down the CI MSD

Software changes

The software is revised periodically. If the steps in this procedure do not match your MSD ChemStation software, refer to the manuals and online help supplied with the software for more information.

See also

You can also use the Control Panel to perform this task. See the *5973N and 5973 inert Mass Selective Detector Local Control Panel (LCP) Quick Reference (*G2589-90072) for more information.

1 **Follow the instructions in the previous module.**

See “To pump down the MSD” on page 70.

After the software prompts you to turn on the interface heater and GC oven, perform the following steps.

2 **Check vacuum gauge controller to verify that the pressure is decreasing.**

3 **Press Gas A and Purge, and verify that the Gas A and Purge lights are on.**

4 **Verify that PCICH4.U is loaded, and accept the temperature setpoints.**

Always start up, and verify system performance in PCI mode before switching to NCI.

5 **Set the GC/MSD interface to 320°C.**

6 **Purge for at least one hour.**

7 **Press the Purge button to turn off Purge.**

8 **Set Gas A to 20%.**

9 **Let system bake out and purge for at least two hours. If you will be running NCI, best sensitivity, bake the MSD out overnight.**

To connect the gauge controller

Materials needed:

Gauge controller (59864B)
Power cord
Triode gauge cable (8120-6573)
The high-vacuum gauge controller is ***required*** for operating the MSD in CI mode.

WARNING

Never connect or disconnect the cable from the triode gauge tube while the MSD is under vacuum. Risk of implosion and injury due to broken glass exists.

CAUTION

Be sure to orient the cable and the gauge tube as illustrated. Excessive force on the pins can break the tube. Do not stress the cable.

1 **Connect the triode gauge cable to the triode gauge tube.**

2 **Connect the other end of the triode gauge cable to the gauge controller.**

3 **Connect the power cord to the gauge controller.**

4 **Connect the other end of the power cord to an appropriate electrical outlet.**

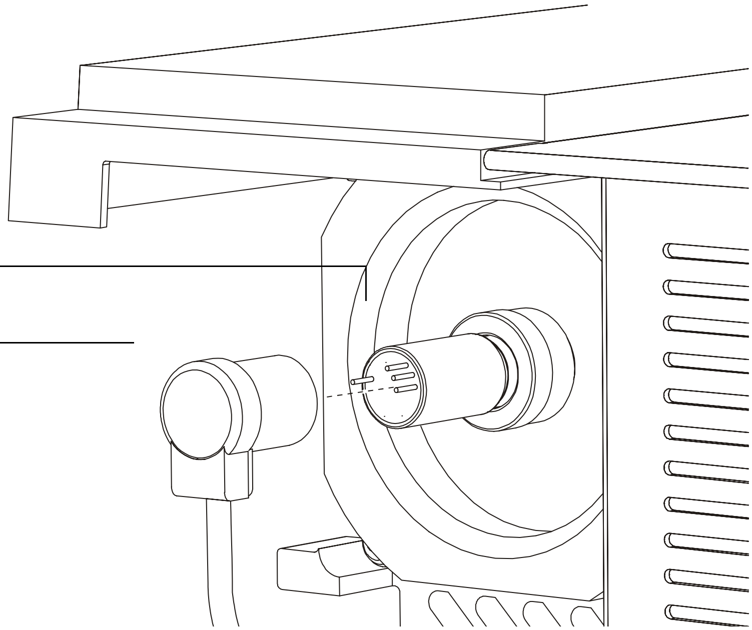
If you wish to share one controller among MSDs, obtain one cable for each instrument. Leave a cable connected to the triode gauge tube on each MSD. This will avoid having to vent the MSD before connecting the controller.

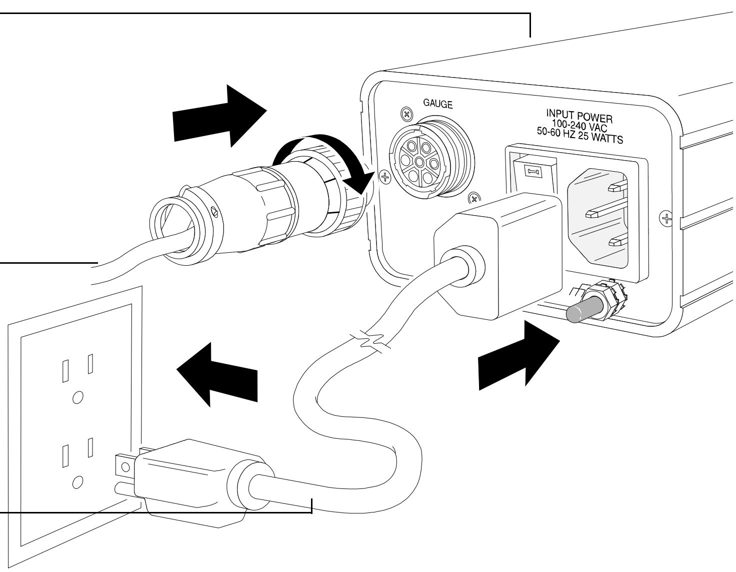
5 **Pump down the MSD.**

CAUTION

Do ***not*** use a 59864A (older model) triode gauge controller during data acquisition. This model can be used for diagnostic purposes ***only***.

To connect the gauge controller





To move or store the MSD

Materials needed:

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

1 **Vent the MSD (page 64).**

2 **Remove the column and install a blank ferrule and interface nut.**

3 **Tighten the vent valve.**

4 **If the MSD has a gauge controller, disconnect the cable from the triode gauge tube.**

5 **Move the MSD away from the GC (page 178).**

   Unplug the GC/MSD interface heater cable from the GC.

6 **Install the interface nut with the blank ferrule.**

7 **Remove the analyzer cover (page 62).**

8 **Tighten the side plate thumbscrews to "finger tight".**

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.

9 **Plug the MSD power cord in.**

10 **Switch the MSD on to establish a rough vacuum.**

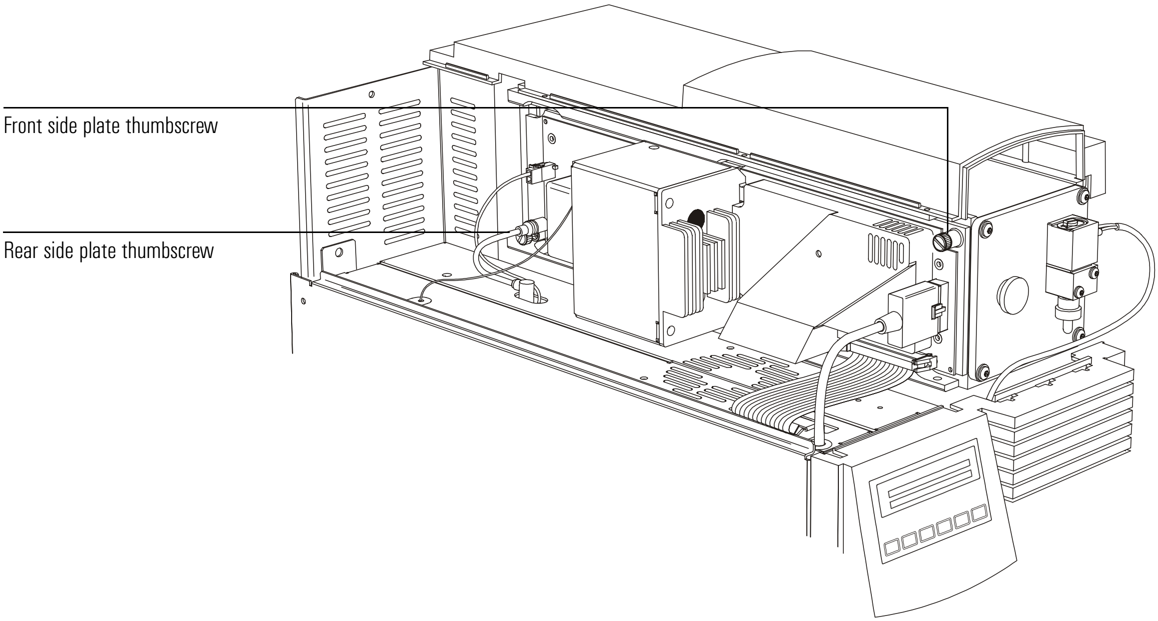
   Verify that the turbo pump speed is greater than 50%.

11 **Switch the MSD off.**

12 **Reinstall the analyzer cover.**

13 **Disconnect the LAN, remote, and power cables.**

To move or store the MSD



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

CAUTION

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

To set the interface temperature from a 6890 GC

1 **Press the Aux # key on the GC keypad.**

2 **Press 2.**

By default, the GC/MSD interface is powered by heated zone Thermal Aux #2 on the 6890 Series GC. Verify that the display shows **THERMAL AUX 2 (MSD)**.

3 **Use the number keys to type in the new temperature setpoint.**

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

CAUTION Never exceed the maximum temperature of your column.

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 GC oven.

4 **Press the Enter key to download the new setpoint.**

If you want the new setpoint to become part of the current method, click **Save** under the Method menu. Otherwise, the first time a method is loaded, all the setpoints in the method will overwrite those set from the GC keyboard.

To set the interface temperature from a 6890 GC

3

Operating the CI MSD

Operating the MSD in CI mode

This chapter provides information and instructions about operating the 5973 inert CI MSDs in CI mode. Most of the material is related to methane chemical ionization but one section discusses the use of other reagent gases.

NOTE

Sequencing is not appropriate for automating methods that use different reagent gases or gas flows, as these parameters must be set ***manually***.

The software provides instructions for setting the reagent gas flow and for performing CI autotunes. Autotunes are provided for PCI with methane reagent gas and for NCI with any reagent gas.

General guidelines

- Always use the highest purity methane (and other reagent gases, if applicable.) Methane must be at least 99.99% pure.
- Always verify that the MSD is performing well in EI mode before switching to CI. See "To verify system performance" on page 61.
- Make sure the CI ion source and GC/MSD interface tip seal are installed.
- Make sure the reagent gas plumbing has no air leaks. This is determined in PCI mode, checking for *m/z* 32 after the methane pre-tune.

To operate the CI MSD

Operating your MSD in the CI mode is slightly more complicated than operating in the EI mode. After tuning, gas flow, source temperature (Table 3 on page 80), and electron energy may need to be optimized for your specific analyte.

Table 3 **Temperatures for CI operation**

	Ion source	Quadrupole	GC/MSD interface
PCI	250°C	150°C	320°C
NCI	150°C	150°C	280°C

Start the system in PCI mode first.

By bringing the system up in PCI mode first, you will be able to do the following:

- Set up the MSD with methane first, even if you are going to use another reagent gas.
- Check the interface tip seal by looking at the *m/z* 28 to 27 ratio (in the methane flow adjust panel.).
- Tell if a gross air leak is present by monitoring the ions at *m/z* 19 (protonated water) and 32.
- Confirm if the MS is generating “real” ions and not just background noise.

It is nearly impossible to perform any diagnostics on the system in NCI. In NCI, there are no reagent gas ions to monitor for any gas. It is difficult to diagnose an air leak and difficult to tell whether a good seal is being created between the interface and the ion volume.

To switch from EI to CI operating mode

CAUTION

Always verify MSD performance in EI before switching to CI operation. See page 51. Always set up the CI MSD in PCI first, even if you are going to run NCI

1. **Vent the MSD. See page 64.**
2. **Open the analyzer.**
3. **Remove the EI ion source.**

CAUTION

Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded anti-static wrist strap
See “Electrostatic discharge is a threat to the MSD electronics during maintenance” on page 168. Take anti-static precautions ***before*** you open the analyzer chamber.

4. **Install the CI ion source. See page 246.**
5. **Install the interface tip seal. See page 248.**
6. **Close the analyzer.**
7. **Pump down the MSD. See page 70.**

To set up the software for CI operation

1 Switch to the Tune and Vacuum Control view.

2 Select Load Tune Values from the File menu.

3 Select the tune file PCICH4.U.

4 If CI autotune has never been run for this tune file, the software will prompt you through a series of dialog boxes. *Accept the default values unless you have a very good reason for changing anything.*

The tune values have a dramatic effect on MSD performance. Always start with the default values when first setting up for CI, and then make adjustments for your specific application. See the table below for default values for the Tune Control Limits box.

Default Tune Control Limits, used by CI autotune only. These limits should *not* be confused with the parameters set in Edit MS Parameters, or with those appearing on the tune report.

Reagent gas	**Methane**		**Isobutane**[a]		**Ammonia**[a]	
Ion polarity	Positive	Negative[a]	Positive[a]	Negative[a]	Positive[a]	Negative[a]
Abundance target[b],	$1x10^6$	$1x10^6$	N/A[c]	$1x10^6$	N/A[c]	$1x10^6$
Peakwidth target[d]	0.6	0.6	N/A[c]	0.6	N/A[c]	0.6
Maximum repeller	4	4	N/A[c]	4	N/A[c]	4
Maximum emission current[e], µA	240	50	N/A[c]	50	N/A[c]	50
Max electron energy, eV	240	240	N/A[c]	240	N/A[c]	240

a. Always set up in PCI with methane first, then switch to your desired ion polarity and reagent gas.
b. Adjust higher or lower to get desired signal abundance. Higher signal abundance also gives higher noise abundance. This is adjusted for data acquisition by setting the EMV in the method.
c. There are no PFDTD ions formed in PCI with any reagent gas but methane, hence, CI autotune is not available with these configurations.
d. Higher peakwidth values give better sensitivity, lower values give better resolution.
e. Optimum emission current maximum for NCI is very compound-specific, and must be selected empirically. Optimum emission current for pesticides, for example, may be about 200 µA.

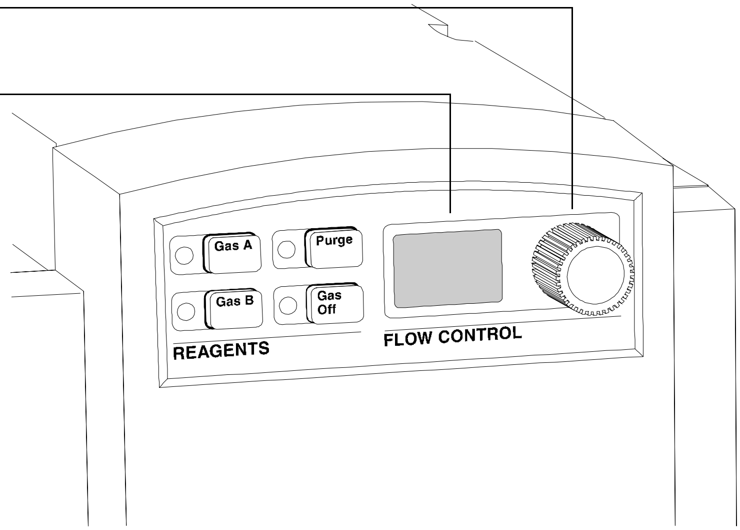
To operate the reagent gas flow control module

For a video demonstration of the gas flow control module, see the 5973 inert MSD Manual CD-ROM.

Flow control module state diagram:						
Result	Gas A flows	Gas B flows	Purge with Gas A	Purge with Gas B	Pump out flow module	Standby, vented, or EI mode
Control panel lights (LEDs)						
Gas A (green)	On	Off	On	Off	Off	Off
Gas B (amber)	Off	On	Off	On	Off	Off
Purge (red)	Off	Off	On	On	On	Off
Gas Off (red)	Off	Off	Off	Off	On	On
Valve state						
Valve A	Open	Closed	Open	Closed	Closed	Closed
Valve B	Closed	Open	Closed	Open	Closed	Closed
MFC setting	On → setpoint	On → setpoint	On → 100%	On → 100%	On → 100%	Off (→0%)
Isolation valve	Open	Open	Open	Open	Open	Closed

Flow control knob (mass flow control knob)

Flow control display



To set up methane reagent gas flow

The reagent gas flow must be adjusted for maximum stability before tuning the CI system. Do the ***initial*** setup with methane in positive ion mode (PCI). No flow adjustment procedure is available for NCI, as no negative reagent ions are formed.

Adjusting the methane reagent gas flow is a three-step process: setting the flow control, pre-tuning on the reagent gas ions, and adjusting the flow for stable reagent ion ratios, for methane, *m/z* 28/27.

Your data system will prompt you through the flow adjustment procedure.

CAUTION After the system has been switched from EI to CI mode, or vented for any other reason, the MSD must be baked out for at least 2 hours before tuning.

1 **Press the Gas A button. Verify that only the Gas A light is on.**

2 **Adjust the flow to 20% for PCI/NCI MSDs.**

3 **Check the vacuum gauge controller to verify correct pressure. See page 96.**

4 **Select Methane Pretune from the Setup menu.**

The methane pretune tunes the instrument for optimum monitoring of the ratio of methane reagent ions *m/z* 28/27.

5 **Examine the displayed profile scan of the reagent ions.**

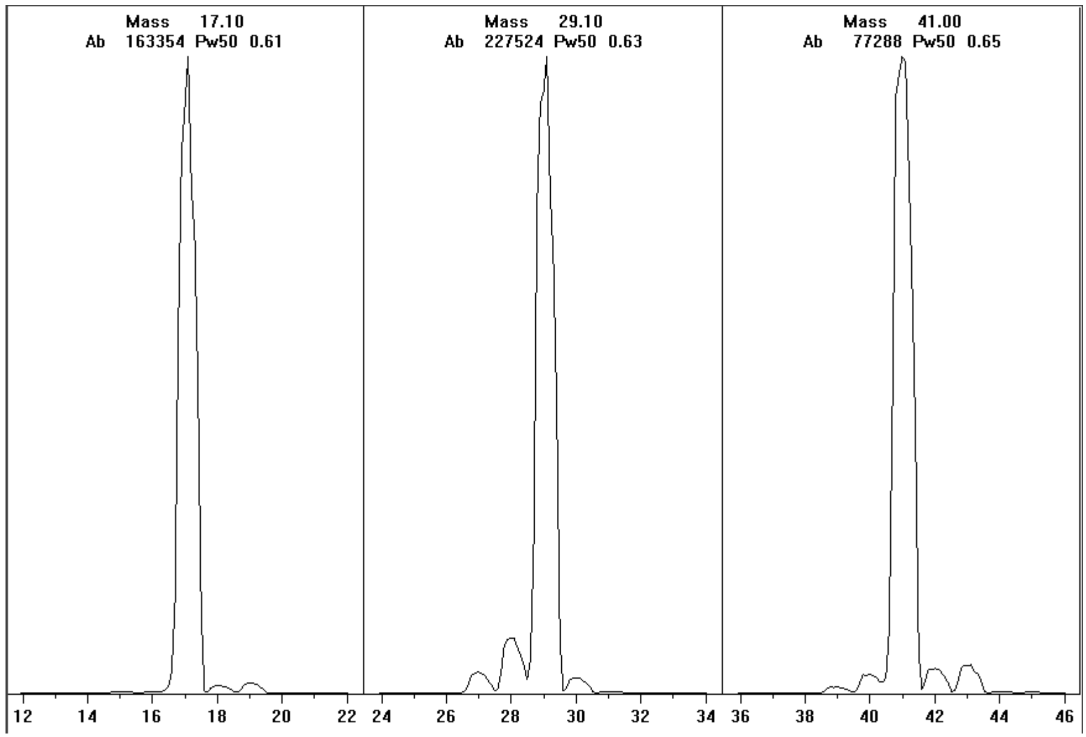
- Make sure there is no visible peak at *m/z* 32. A peak there indicates an air leak. If such a peak is present, find and repair the leak before proceeding. Operating in the CI mode with an air leak will rapidly contaminate the ion source.
- Make sure that the peak at *m/z* 19 (protonated water) is less than 50% of the peak at *m/z* 17.

6 **Perform the Methane Flow Adjust.**

Adjust the methane flow on the PCI/NCI MSD to get the ratio of *m/z* 28/27 between 1.5 and 5.0.

CAUTION

Continuing with CI autotune if the MSD has an air leak or large amounts of water will result in ***severe*** ion source contamination. If this happens, you will need to ***vent the MSD*** and ***clean the ion source***.



Methane pre-tune after more than a day of baking out. Note the low abundance of *m/z* 19 and absence of any visible peak at *m/z* 32. Your MSD will probably show more water at first, but the abundance of *m/z* 19 should still be less than 50% of *m/z* 17.

CI autotune

After the reagent gas flow is adjusted, the lenses and electronics of the MSD should be tuned. Perfluoro-5,8-dimethyl-3,6,9-trioxidodecane (PFDTD) is used as the calibrant. Instead of flooding the entire vacuum chamber, the PFDTD is introduced directly into the ionization chamber through the GC/MSD interface by means of the gas flow control module.

CAUTION

After the system has been switched from EI to CI mode, or vented for any other reason, the MSD must be purged and baked out for at least 2 hours before tuning. Longer bakeout is recommended before running samples requiring optimal sensitivity.

There is a PCI autotune for methane only, as there are no PFDTD ions produced by other gases in positive mode. PFDTD ions are visible in NCI for any reagent gas. Always tune for methane PCI first regardless of which mode or reagent gas you wish to use for your analysis.

There are no tune performance criteria. If CI autotune completes, it passes. EMVolts (electron multiplier voltage) at or above 2600 V, however, indicates a problem. If your method requires EMVolts set at +400, you may not have adequate sensitivity in your data acquisition.

CAUTION

Always verify MSD performance in EI before switching to CI operation. See page 51. Always set up the CI MSD in PCI first, even if you are going to run NCI

Reagent gas	Methane		Isobutane		Ammonia		EI
Ion polarity	Positive	Negative	Positive	Negative	Positive	Negative	N/A
Emission	150 µA	50 µA	150 µA	50 µA	150 µA	50 µA	35 µA
Electron energy	150 eV	150 eV	150 eV	150 eV	150 eV	150 eV	70 eV
Filament	1	1	1	1	1	1	1 or 2
Repeller	3 V	3 V	3 V	3 V	3 V	3 V	30 V
Ion focus	130 V	130 V	130 V	130 V	130 V	130 V	90 V
Entrance lens offset	20 V	20 V	20 V	20 V	20 V	20 V	25 V
EMVolts	1200	1200	1200	1200	1200	1200	1200
Gas Off	Off	Off	Off	Off	Off	Off	On
Gas select valve	A	A	B	B	B	B	None
Suggested flow	20%	40%	20%	40%	20%	40%	N/A
Source temp	250°C	150°C	250°C	150°C	250°C	150°C	230°C
Quad temp	150°C	150°C	150°C	150°C	150°C	150°C	150°C
Interface temp	320°C	280°C	320°C	280°C	320°C	280°C	280°C
Autotune	Yes	Yes	No	Yes	No	Yes	Yes

To perform a positive CI autotune (methane only)

CAUTION

Always verify MSD performance in EI before switching to CI operation. See page 51. Always set up the CI MSD in PCI first, even if you are going to run NCI

1 **Verify that the MSD performs correctly in EI mode first. See page 61.**

2 **Load the PCICH4.U tune file (or an existing tune file for the reagent gas you are using).**

If you use an existing tune file, be sure to save it with a new name if you don't want to over write the existing values.

3 **Accept the default settings.**

4 **Perform methane setup. See page 86.**

5 **Under the Tune menu, click CI Autotune.**

CAUTION

Avoid tuning more often than is absolutely necessary; this will minimize PFDTD background noise, and help prevent ion source contamination.

There are no tune performance criteria. If autotune completes, it passes. If the tune sets the electron multiplier voltage (EMVolts) at or above 2600 V, however, you may not be able to acquire data successfully if your method sets EMVolts to "+400" or higher.

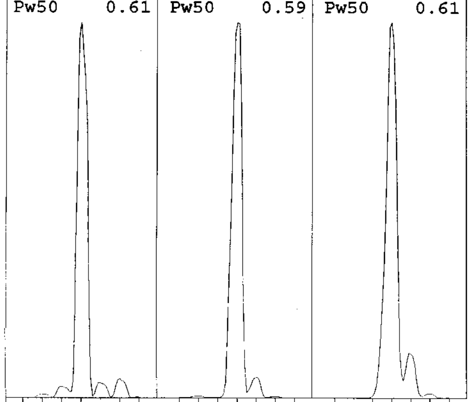
The autotune report contains information about air and water in the system.

The 19/29 ratio shows the abundance of water.

The 32/29 ratio shows the abundance of oxygen.

```
                    PCI mode Methane Tune
Instrument: GC_MS Instrument #1
Wed Aug 25 11:19:30 1999                 C:\MSDCHEM\1\5973\PCICH4.U
Flow: 20                                                     Gas: A
```

Mass 41.00	Mass 267.00	Mass 599.00
Ab 315018	Ab 725285	Ab 691110
Pw50 0.61	Pw50 0.59	Pw50 0.61

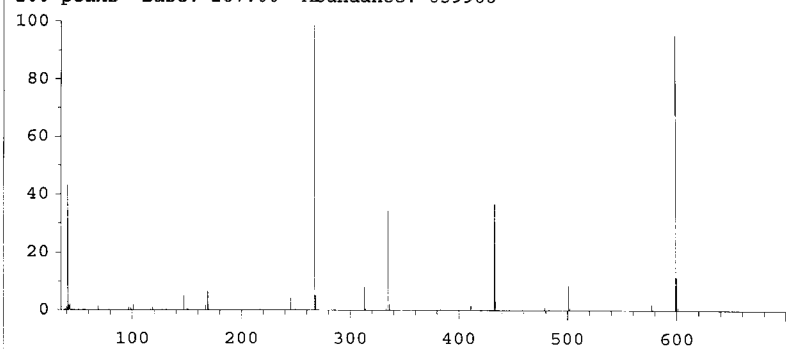



```
Ion Pol       POS  MassGain    -199
                   MassOffs     -11
Emission    242.3  AmuGain     1537
EleEnergy    54.2  AmuOffs      120
Filament        1  Wid219    -0.058
                   DC Pol       NEG
Repeller     2.82
IonFocus    120.5  HED           ON
EntLens      11.0  EMVolts      918
EntOffs     18.57

                   Samples        8
PFDTD        OPEN  Averages       3
                   StepSize    0.10

Zones:
MS Source     250  TurboSpd     100
MS Quad       150
```

Scan: 35.00 - 700.00 Samples: 8 Thresh: 100 Step: 0.10
200 peaks Base: 267.00 Abundance: 659968



Mass	Abund	Rel Abund	Iso Mass	Iso Abund	Iso Ratio
41.10	285952	100.00	42.00	11676	4.08
267.00	659968	230.80	268.00	35528	5.38
599.00	630784	220.59	600.00	75248	11.93

```
CI Reagent Ions: 17/29 Ratio: 0.48  19/29 Ratio: 0.07  32/29 Ratio: 0.00
                 28/27 Ratio:  2.9  28/29 Ratio: 0.08
                 41/29 Ratio: 0.30  29 Abundance: 948608 counts
```

To perform a negative CI autotune (any reagent gas)

CAUTION

Always verify MSD performance in EI before switching to CI operation. See page 51. Always set up the CI MSD in PCI first, with methane as the reagent gas first, even if you are going to be using a different reagent gas or going to run NCI.

1 **Load NCICH4.U (or an existing tune file for the reagent gas you are using).**

   If you use an existing tune file, be sure to save it with a new name if you don't want to over write the existing values.

2 **Accept the default temperature and other settings.**

3 **If you don't already have an NCI tune file for your reagent gas, use Select Reagent Gas under the Setup menu.**

4 **Under the Tune menu, click CI Autotune.**

CAUTION

Avoid tuning unless absolutely necessary; this will minimize PFDTD background noise, and help prevent ion source contamination.

There are no tune performance criteria. If autotune completes, it passes. If the tune sets the electron multiplier voltage (EMVolts) at or above 2600 V, however, you may not be able to acquire data successfully if your method sets EMVolts to "+400" or higher.

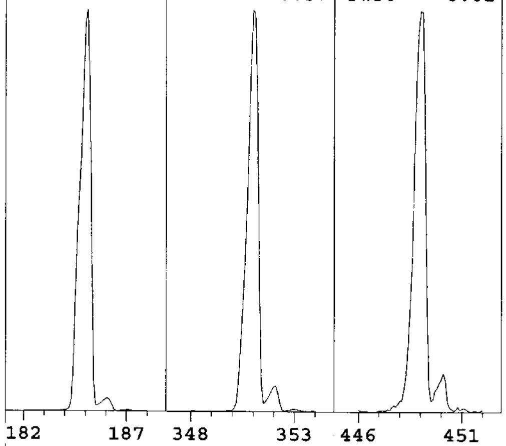
NCI Methane Tune

Instrument: GC/MS Instrument #1

Tue Sep 23 10:53:30 1997 C:\MSDCHEM\1\5973\NCITUNE.U

Flow: 40 Gas: A

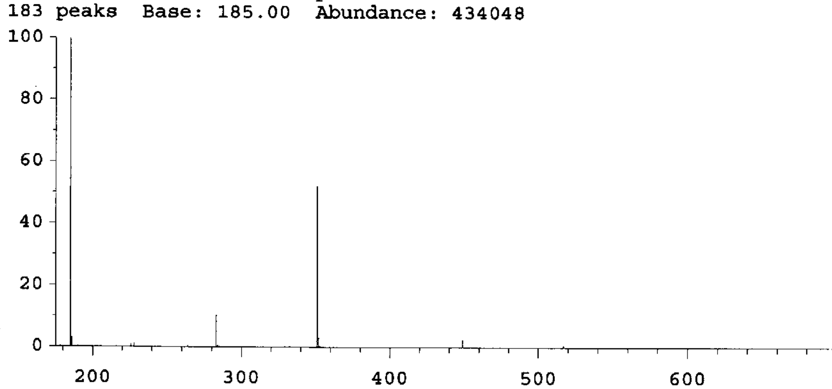
Mass	185.10	Mass	351.00	Mass	449.00
Ab	513077	Ab	260962	Ab	12776
Pw50	0.62	Pw50	0.57	Pw50	0.61



Ion Pol	NEG	MassGain	-403
		MassOffs	-10
Emission	49.4	AmuGain	1433
EleEnergy	123.7	AmuOffs	130
Filament	1	Wid219	0.032
		DC Pol	POS
Repeller	3.99		
IonFocus	149.0	HED	ON
EntLens	7.5	EMVolts	1765
EntOffs	19.58		
		Samples	8
PFDTD	OPEN	Averages	3
		StepSize	0.10
Zones:			
MS Source	150	TurboSpd	100
MS Quad	106		

Scan: 175.00 - 700.00 Samples: 8 Thresh: 100

183 peaks Base: 185.00 Abundance: 434048



Mass	Abund	Rel Abund	Iso Mass	Iso Abund	Iso Ratio
185.00	434048	100.00	186.00	14115	3.25
351.00	226560	52.20	352.00	13892	6.13
449.00	11096	2.56	450.00	1142	10.29

To verify positive CI performance

Materials needed: Benzophenone, 100 pg/µL (8500-5440)

CAUTION Always verify MSD performance in EI before switching to CI operation. See page 51. Always set up the CI MSD in PCI first, even if you are going to run NCI.

1 **Verify that the MSD performs correctly in E1 and PCI mode.**

2 **Verify that the PCICH4.U tune file is loaded.**

3 **On the flow control panel, turn Purge off. Set Gas A to 20% flow.**

4 **In Tune and Vacuum Control view, perform CI setup. See page 88.**

5 **Run CI Autotune. See page 88.**

6 **Run the PCI sensitivity method: BENZ_PCI.M, using 1 µL of 100 pg/µL Benzophenone.**

7 **Verify that the system conforms to the published sensitivity specification.**

See also *5973N and 5973 inert Mass Selective Detector Specifications* (5988-9991EN)

To verify negative CI performance

This procedure is for EI/PCI/NCI MSDs ***only***

Materials needed: OFN, 1 pg/µL (8500-5441)

CAUTION Always verify MSD performance in EI before switching to CI operation. See page 51. Always set up the CI MSD in PCI first, even if you are going to run NCI

1 **Verify that the MSD performs correctly in EI mode.**

2 **Load the NCICH4.U tune file, and accept the temperature setpoints.**

3 **Turn Purge and Gas A on and let the system stabilize for 90 minutes.**

4 **Turn Purge off, and set Gas A to 40% flow.**

5 **In Tune and Vacuum Control view, run CI Autotune. See page 95.**

Note that there are no criteria for a "passing" Autotune in CI. If the Autotune completes, it passes.

6 **Run the NCI sensitivity method: OFN_NCI.M using 1 µL of 1 pg/µL OFN.**

7 **Verify that the system conforms to the published sensitivity specification.**

See also *5973N and 5973 inert Mass Selective Detector Specifications* (5988-9991EN)

To monitor high vacuum pressure

Materials needed: Gauge controller (59864B)
Triode gauge cable (8120-6573)

WARNING **Never connect or disconnect the cable from the triode gauge tube while the MSD is under vacuum. Risk of implosion and injury due to broken glass exists.**

WARNING **If you are using hydrogen as a carrier gas, do not turn on the triode gauge tube if there is any possibility that hydrogen has accumulated in the manifold. The triode gauge filament can ignite hydrogen. Read the *Hydrogen Carrier Gas Safety Guide* (5955-5398) before operating the MSD with hydrogen carrier gas.**

1 **Connect the gauge controller to the triode gauge tube. See page 73.**

2 **Start up and pump down the MSD. See page 70.**

3 **Switch on the power switch on the back of the gauge controller.**

4 **Press and release the GAUGE button.**

After a few seconds, the pressure should be displayed.

Pressure is displayed in the format **X.X – X** where **– X** is the base 10 exponent. Units are Torr.

The gauge controller will not turn on if the pressure in the MSD is above approximately 8×10^{-3} Torr. The gauge controller will display all 9s and then go blank. The triode gauge tube can measure pressures between approximately 8×10^{-3} and 3×10^{-6} Torr. The gauge controller is calibrated for nitrogen, but all pressures listed in this manual are for helium.

The largest influence on operating pressure is the carrier gas (column) flow. The following table lists typical pressures for various helium carrier gas flows. These pressures are approximate and will vary from instrument to instrument.

Typical pressure readings

Use the 59864B high-vacuum gauge controller. Note that the mass flow controller is calibrated for methane, and the high vacuum gauge controller is calibrated for nitrogen, so these measurements are not accurate, but are intended as a guide to typical observed readings. They were taken with the following set of conditions. Note that these are typical PCI temperatures:

Source temperature	250°C
Quad temperature	150°C
Interface temperature	320°C
Helium carrier gas flow	1 mL/min

MFC (%)	Pressure (Torr)	
	Methane	**Ammonia**
	EI/PCI/NCI MSD (Performance turbo pump)	EI/PCI/NCI MSD (Performance turbo pump)
10	5.5×10^{-5}	5.0×10^{-5}
15	8.0×10^{-5}	7.0×10^{-5}
20	1.0×10^{-4}	8.5×10^{-5}
25	1.2×10^{-4}	1.0×10^{-4}
30	1.5×10^{-4}	1.2×10^{-4}
35	2.0×10^{-4}	1.5×10^{-4}
40	2.5×10^{-4}	2.0×10^{-4}

Familiarize yourself with the measurements on ***your*** system under operating conditions, and watch for ***changes*** that may indicate a vacuum or gas flow problem. Measurements will vary by as much as 30% from one MSD and gauge controller to the next.

To use other reagent gases

This section describes the use of isobutane or ammonia as the reagent gas. You should be familiar with operating the CI-equipped 5973 inert MSD with methane reagent gas before attempting to use other reagent gases.

CAUTION Do not use nitrous oxide as a reagent gas. It radically shortens the life span of the filament.

Changing the reagent gas from methane to either isobutane or ammonia changes the chemistry of the ionization process and yields different ions. The principal chemical ionization reactions encountered are described in general in Appendix A, *Chemical Ionization Theory*. If you are not experienced with chemical ionization, we suggest reviewing that material before you proceed.

CAUTION Not all setup operations can be performed in all modes with all reagent gases. See the following table for details.

Reagent gas/ mode	Reagent ion masses	PFDTD Calibrant ions	Flow adj ions: Ratio EI/PCI/NCI MSD Performance turbo pump Recommended flow: 20%
Methane/ PCI	17, 29, 41[a]	41, 267, 599	28/27: 1.5 – 5.0
Methane/ NCI	17, 35, 235[b]	185, 351, 449	N/A
Isobutane/ PCI	39, 43, 57	N/A	57/43: 5.0 – 30.0
Isobutane/ NCI	17, 35, 235	185, 351, 449	N/A
Ammonia/ PCI	18, 35, 52	N/A	35/18: 0.1 – 1.0
Ammonia/ NCI	17, 35, 235	185, 351, 517	N/A

a. There are ***no*** PFDTD ions formed with any reagent gas but methane. Tune with methane and use the same parameters for the other gas.

b. There are ***no negative*** reagent gas ions formed. To pretune in negative mode, use background ions:
17 (OH^-), 35 (Cl^-), and 235 (ReO_3^-). These ions can not be used for reagent gas flow adjustment. Set flow to 40% for NCI and adjust as necessary to get acceptable results for your application.

Isobutane CI

Isobutane (C_4H_{10}) is commonly used for chemical ionization when less fragmentation is desired in the chemical ionization spectrum. This is because the proton affinity of isobutane is higher than that of methane; hence, less energy is transferred in the ionization reaction. Addition and proton transfer are the ionization mechanisms most often associated with isobutane. The sample itself influences which mechanism dominates.

Ammonia CI

Ammonia (NH_3) is commonly used for chemical ionization when less fragmentation is desired in the chemical ionization spectrum. This is because the proton affinity of ammonia is higher than that of methane; hence, less energy is transferred in the ionization reaction. Because many compounds of interest have insufficient proton affinities, ammonia chemical-ionization spectra often result from the addition of NH_4^+ and then, in some cases, from the subsequent loss of water. Ammonia reagent ion spectra have principal ions at *m/z* 18, 35, and 52, corresponding to NH_4^+, $NH_4(NH_3)^+$, and $NH_4(NH_3)_2^+$.

To adjust your MSD for isobutane or ammonia chemical ionization, use the following procedure:

1 Perform a standard Positive CI autotune with methane and PFDTD.

2 Under the Setup menu, click Select Reagent Gas and select Isobutane or Ammonia.

This will change the menus to use the selected gas, and select appropriate default tune parameters.

3 Select a new tune file name, or load an existing PCI tune file for the specific gas.

If you use an existing tune file, be sure to save it with a new name if you don't want to over write the existing values. Accept the default temperature and other settings.

4 Turn Gas B on.

After the amber light stops flashing and the Purge light goes off, set the gas flow to 20%.

5 Click Isobutane (or Ammonia) Flow Adjust on the Setup menu.

There is no CI autotune for isobutane or ammonia in PCI.

If you wish to run NCI with isobutane or ammonia, load **NCICH4.U,** or load an existing NCI tune file for the specific gas.

NOTE Be sure to read the following application note: *Implementation of Ammonia Reagent Gas For Chemical Ionization On 5973 MSDs (5968-7844).*

CAUTION

Use of ammonia affects the maintenance requirements of the MSD. See the maintenance chapter for more information.

CAUTION

The pressure of the ammonia supply must be less than 5 psig. Higher pressures can result in ammonia condensing from a gas to a liquid.

Always keep the ammonia tank in an upright position, below the level of the flow module. Coil the ammonia supply tubing into several vertical loops by wrapping the tubing around a can or bottle. This will help keep any liquid ammonia out of the flow module.

Ammonia tends to break down vacuum pump fluids and seals. Ammonia CI makes more frequent vacuum system maintenance necessary.

See also

To minimize foreline pump damage from ammonia, 252.

CAUTION

When running ammonia for five or more hours a day, the foreline pump must be ballasted for at least one hour a day to minimize damage to pump seals. See page 252. Always purge the MSD with methane after flowing ammonia.

Frequently, a mixture of 5% ammonia and 95% helium or 5% ammonia and 95% methane is used as a CI reagent gas. This is enough ammonia to achieve good chemical ionization while minimizing its negative effects.

Carbon dioxide NCI

Carbon dioxide is often used as a buffer gas for negative CI. It has obvious advantages of availability and safety.

To switch from CI to EI operating mode

1 **Press the Gas Off button to close the isolation valve.**

2 **Vent the MSD. See page 64.**
The software will prompt you for the appropriate actions.

3 **Open the analyzer.**

4 **Remove the CI interface tip seal. See page 248.**

5 **Remove the CI ion source. See page 246.**

6 **Install the EI ion source. See page 214.**

7 **Place the CI ion source and interface tip seal in the ion source storage box.**

8 **Pump down the MSD. See page 70.**

9 **Load your EI tune file.**

CAUTION Always wear clean gloves while touching the analyzer or any other parts that go inside the analyzer chamber.

CAUTION Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded anti-static wrist strap and take other anti-static precautions ***before*** you open the analyzer chamber.
See page 168.

4

Troubleshooting the MSD

How to identify the symptoms and causes of problems in your MSD

Troubleshooting the MSD

This chapter is a quick reference to symptoms and possible causes of the most common problems experienced by the 5973 inert Mass Selective Detector (MSD). Related symptoms are grouped in these categories:

- General symptoms
- Chromatographic symptoms
- Mass spectral symptoms
- Pressure symptoms
- Temperature symptoms
- Error messages
- Contamination
- Air leaks

For each symptom, one or more possible causes are listed. The possible causes listed are not in a strict order. In general, however, the possible causes listed first are the most likely causes ***or*** the easiest to check and correct. See CI Troubleshooting, 131 for help with CI-specific problem.s

This section is only a quick reference. No corrective actions are listed for the possible causes for each symptom. For more extensive troubleshooting information, see the *Troubleshooting (5973 inert MSD)* section in the online help of the MSD ChemStation software. The online troubleshooting provides more explanation and, in many cases, corrective actions.

WARNING

This chapter does not include corrective actions for the possible causes listed. Some of the corrective actions required may be dangerous if performed incorrectly. Do not attempt any corrective actions unless you are sure you know the correct procedure and the dangers involved. See the *Troubleshooting (5973 inert MSD)* section in the online help and the other chapters in this manual for more information.

If the material in this chapter and in the online help proves insufficient to help you diagnose a problem, contact your Agilent Technologies service representative.

Troubleshooting tips and tricks

The following are general rules for troubleshooting, with specific examples.

Rule 1: "Look for what has been changed."

Many problems are introduced accidentally by human actions. Every time any system is disturbed, there is a chance of introducing a new problem.

- If the MSD was just pumped down after maintenance, suspect air leaks or incorrect assembly.
- Carrier gas or helium gas purifier were just changed, suspect leaks or contaminated or incorrect gas.
- If the GC column was just replaced, suspect air leaks or contaminated or bleeding column.

Rule 2: "If **complex** isn't working, go back to **simple**."

A complex task is not only more difficult to perform, but also more difficult to troubleshoot as well.

- If you're having trouble detecting your sample, verify that autotune is successful.

Rule 3: "Divide and conquer."

This technique is known as "half-split" troubleshooting. If you can isolate the problem to only part of the system, it is much easier to locate.

- To determine whether an air leak is in the GC or the MSD, you can vent the MSD, remove the column, and install the blank interface ferrule. If the leak goes away, it was in the GC.

General symptoms

This section describes symptoms you might observe when first turning on the GC/MSD system. All of these symptoms would prevent operation of the system.

GC does not turn on

This refers to a condition in which nothing happens when the GC is switched on. The GC fans do not turn on and the keypad display does not light.

- Disconnected GC power cord
- No voltage or incorrect voltage at the electrical outlet
- Failed fuse in the GC
- GC power supply is not working correctly

MSD does not turn on

This refers to a condition in which nothing happens when the MSD is switched on. The foreline pump does not start. The cooling fan for the high vacuum pump does not turn on. The control panel is not on.

- Disconnected MSD power cord
- No voltage or incorrect voltage at the electrical outlet
- Failed primary fuses
- MSD electronics are not working correctly

Foreline pump is not operating

This refers to a condition where the MSD is receiving power (the fan is operating and the control panel is lit) but the foreline pump is not operating.

- Large air leak (usually the analyzer door open) has caused pumpdown failure. See “Pumpdown failure shutdown” on page 259. Note that you must power cycle the MSD to recover from this state.
- Disconnected foreline pump power cord
- Malfunctioning foreline pump

MSD turns on but then the foreline pump shuts off

MSDs will shut down both the foreline pump and the high vacuum pump if the system fails to pump down correctly. This is usually because of a large air leak: either the sideplate has not sealed correctly or the vent valve is still open. This feature helps prevent the foreline pump from sucking air through the system, which can damage the analyzer and pump.

See “Pumpdown failure shutdown” on page 259. Note that you must power cycle the MSD to recover from this state.

Control panel says “No server found”

- Disconnected LAN cable between MSD and the hub, or the hub and the PC
- PC is turned off
- Holding the No/Cancel key down for five seconds will bypass error and allow the user to look at the Local Control Panel.

Chromatographic symptoms

This section describes symptoms you may observe in the chromatograms generated by data acquisition. In general, these symptoms do not prevent you from operating your GC/MSD system. They indicate, however, that the data you are acquiring may not be the best data obtainable. These symptoms can be caused by instrument malfunctions but are more likely caused by incorrect chromatographic technique.

Two of the symptoms: *If sensitivity is low* and *If repeatability is poor*, also apply to mass spectral data.

No peaks

If an analysis shows no chromatographic peaks, only a flat baseline or minor noise, run one of the automated tune programs. If the MSD passes tune, the problem is most likely related to the GC. If the MSD does not pass tune, the problem is most likely in the MSD.

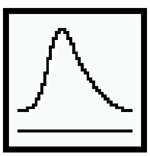
Passes tune

- Incorrect sample concentration
- No analytes present
- Syringe missing from the ALS or not installed correctly
- Injection accidentally made in split mode instead of splitless mode
- Empty or almost empty sample vial
- Dirty injection port
- Leaking injection port*
- Loose column nut at the injection port*

* These could cause a fault condition in the GC that would prevent the GC from operating.

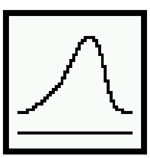
Does not pass tune

- Calibration vial is empty
- Excessive foreline or analyzer chamber pressure
- Very dirty ion source
- Calibration valve is not working correctly
- Bad signal cable connection
- Filament has failed or is not connected correctly
- Bad ion source wiring connection
- Bad detector wiring connection
- Failed electron multiplier horn



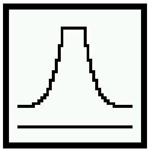
Peaks are tailing

- Active sites in the sample path
- Injection is too large
- Incorrect injection port temperature
- Insufficient column flow
- GC/MSD interface temperature is too low
- Ion source temperature is too low



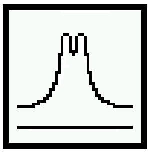
Peaks are fronting

- Column film thickness mismatched with analyte concentration (column overload)
- Initial oven temperature is too low
- Active sites in the sample path
- Injection is too large
- Injection port pressure too high
- Insufficient column flow



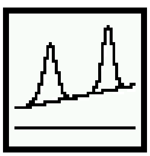
Peaks have flat tops

- Insufficient solvent delay
- Incorrect scale on the display
- Injection is too large
- Electron multiplier voltage is too high



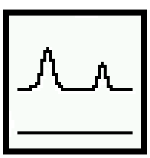
Peaks have split tops

- Bad injection technique
- Injection is too large



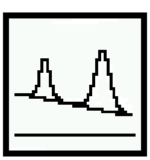
Baseline is rising

- Column bleed
- Other contamination



Baseline is high

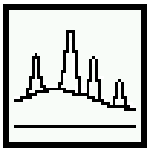
- Column bleed
- Other contamination
- Electron multiplier voltage is too high



Baseline is falling

A falling baseline indicates contamination is being swept away. Wait until the baseline reaches an acceptable level. Common causes include:

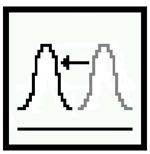
- Residual water air and water from a recent venting
- Column bleed
- Septum bleed
- Splitless injection time too long (inlet is not properly swept, resulting in excess solvent on the column and slow solvent decay)



Baseline wanders

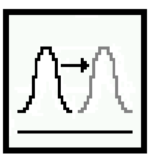
- Insufficient carrier gas supply pressure*
- Malfunctioning flow or pressure regulator*
- Intermittent leak in the injection port*

* These could cause a fault condition in the GC that would prevent the GC from operating.



Retention times for all peaks drift – shorter

- Column has been shortened
- Initial oven temperature was increased
- Column is getting old



Retention times for all peaks drift – longer

- Column flow has been reduced
- Initial oven temperature was decreased
- Active sites in the sample path
- Leaks in the injection port*

* This could cause a fault condition in the GC that would prevent the GC from operating.

Poor sensitivity

- Incorrect tuning
- Tune file that does not match the type of analysis
- Repeller voltage is too low
- Incorrect temperatures (oven, GC/MSD interface, ion source, or mass filter)
- Incorrect sample concentration
- Leaking injection port*
- Dirty injection port
- Incorrect split ratio
- Purge off time in splitless mode is too short
- Excessive pressure in the MSD
- Dirty ion source
- Air leak
- Poor filament operation
- Detector (HED electron multiplier) is not working correctly
- Incorrect mass filter polarity

* This could cause a fault condition in the GC that would prevent the GC from operating.

Poor Repeatability

- Dirty syringe needle
- Dirty injection port
- Leaking injection port*
- Injection is too large
- Loose column connections
- Variations in pressure, column flow, and temperature
- Dirty ion source
- Loose connections in the analyzer
- Ground loops

* This could cause a fault condition in the GC that would prevent the GC from operating.

Mass spectral symptoms

This section describes symptoms you might observe in mass spectra. Some of these symptoms will appear in the mass spectra of samples. Others you will observe only in a tune report. Some of these symptoms have causes that can be corrected by the operator. Others, however, require service by an Agilent Technologies service representative.

Two symptoms listed under Chromatographic symptoms: *If sensitivity is poor* and *If repeatability is poor*, also apply to mass spectra.

No peaks

- Ion source cables not connected
- Bad connections to or from the detector
- HED power supply output cable has failed (white cable)
- Other electronics failure

Isotopes are missing or isotope ratios are incorrect

- Peaks are too wide or too narrow
- Scan speed is too high (scan mode)
- Dwell time is too short (SIM mode)
- Electron multiplier voltage is too high
- Repeller voltage is too high
- High background
- Dirty ion source

High background

- Pressure in the analyzer chamber is too high
- Air leak
- Contamination

High abundances at *m/z* 18, 28, 32, and 44 or at *m/z* 14 and 16

- System was recently vented (residual air and water)
- Air leak

Large peaks at *m/z* 14 and 16 are symptomatic of especially large leaks.

Mass assignments are incorrect

Small shape changes at the top of the mass peaks can cause 0.1 amu shifts in mass assignments. Shifts greater than 0.2 amu indicate a possible malfunction.

- MSD has not had enough time to reach thermal equilibrium
- Large variations in the temperature of the laboratory
- MSD has not been tuned recently, or at the temperature at which it is operating
- Incorrect tune file (inappropriate parameters)

Peaks have precursors

The tune report lists the size of the precursors for the tune masses. Small precursors are not unusual. If the precursors are unacceptably large for your application, one of the following may be responsible:

- Repeller voltage is too high
- Peaks are too wide
- Incorrect dc polarity on the quadrupole mass filter
- Dirty quadrupole mass filter

Peak widths are inconsistent

- MSD has not had enough time to reach thermal equilibrium
- Large variations in the temperature of the laboratory
- Incorrect tuning
- Calibration vial is empty or almost empty
- Calibration valve is not working correctly
- Dirty ion source
- Electron multiplier is nearing the end of it useful lifetime
- Ground loop problems

Relative abundance of *m/z* 502 is less than 3%

Autotune should give an *m/z* 502 relative abundance greater than 3%. The relative abundance of *m/z* 502 can, however, vary a great deal depending on column flow, ion source temperature, and other variables. As long as relative abundance is above 3%, the stability of the relative abundance is more important than the absolute value. If you observe significant changes in the relative abundance of *m/z* 502 for a fixed set of operating parameters, there may be a problem. The charts in the MSD ChemStation software are useful for identifying changes. Select **View Tunes** from the Qualify menu in the Instrument Control view.

Low ***relative*** abundance of *m/z* 502 should not be confused with low ***absolute*** abundances at high masses. Sensitivity at high masses can be excellent even if the relative abundance of *m/z* 502 is near 3%. If your MSD produces low absolute abundances at high masses, refer to the symptom *High mass sensitivity is poor.*

Tune programs other than autotune have different relative abundance targets. The DFTPP and BFB target tune programs tune the 5973 inert MSD to achieve about a 0.8% ratio of *m/z* 502/69.

- Tune program/tune file has a different relative abundance target (3% only applies to Autotune)
- Not enough time for the MSD to warm up and pump down
- Analyzer chamber pressure is too high
- Ion source temperature is too high
- Column (carrier gas) flow is too high
- Poor filament operation
- Dirty ion source
- Air leak
- Incorrect dc polarity on the quadrupole mass filter

Spectra look different from those acquired with other MSDs

Ion ratios are different from those in older MSDs. This is due to the HED detector, and is normal. To get spectra similar to older MSDs, use **Standard Spectra Tune,** available in the Manual Tune view. Note that this tune takes much longer to complete than **Autotune.**

High mass sensitivity is poor

This refers to a condition where the ***absolute*** abundance at the upper end of the mass range is poor. Absolute abundance should not be confused with the ***relative*** abundance (percentage) of *m/z* 502 to *m/z* 69. Sensitivity at high masses can be excellent even if the relative abundance of *m/z* 502 is low.

- Wrong tune program
- Wrong tune file
- Repeller voltage is too low
- Not enough time for the MSD to warm up and pump down
- Analyzer chamber pressure is too high
- Column (carrier gas) flow is too high
- Poor filament operation
- Dirty ion source
- Air leak
- Incorrect dc polarity on the quadrupole mass filter

Pressure symptoms

This section describes unusual pressure readings and their possible causes. The symptoms in this section are based on typical pressures. At typical column flow rates (0.1 - 2.0 mL/minute), the foreline pressure will be approximately 20 to 100 mTorr. The analyzer chamber pressure will be approximately 1×10^{-6} to 1.4×10^{-4} Torr. These pressures can vary widely from instrument to instrument so it is very important that you are familiar with the pressures that are typical for your instrument at given carrier gas flows.

Turbomolecular pumps are controlled according to their speed and do not have foreline pressure gauges. The analyzer chamber pressures can only be measured if your system is equipped with the optional gauge controller.

Foreline pressure is too high

If the pressure you observe for a given column flow has increased over time, check the following:

- Column (carrier gas) flow is too high
- Air leak (usually the sideplate is not pushed in or vent valve is open)
- Foreline pump oil level is low or oil is contaminated
- Foreline hose is constricted
- Foreline pump is not working correctly

Analyzer chamber pressure is too high (EI operating mode)

If the pressure you observe is above 1.0×10^{-4} Torr, or if the pressure you observe for a given column flow has increased over time, check the following:

- Column (carrier gas) flow is too high
- Air leak
- Foreline pump is not working correctly (see *Foreline pressure is too high*)
- Turbomolecular pump is not working correctly

Foreline pressure is too low

If the pressures you observe are below 20 mTorr, check for the following:

- Column (carrier gas) flow is too low
- Column plugged or crushed by an overtightened nut
- Empty or insufficient carrier gas supply*
- Bent or pinched carrier gas tubing*
- Foreline gauge is not working correctly

* These could create a fault condition in the GC that would prevent the GC from operating.

Analyzer chamber pressure is too low

If the pressures you observe are below 1×10^{-6} Torr, check for the following:

- Column (carrier gas) flow is too low
- Column plugged or crushed by overtightened nut
- Empty or insufficient carrier gas supply*
- Bent or pinched carrier gas tubing*

* These could create a fault condition in the GC that would prevent the GC from operating.

Gauge controller displays 9.9+9 and then goes blank

This indicates the pressure in the analyzer chamber is above 8×10^{-3} Torr.

- Solvent peak from an on-column injection
- MSD has not had enough time to pump down
- Excessive foreline pressure
- Bad connection or bad cable between the triode gauge and gauge controller
- Triode gauge tube has failed
- Line voltage too low
- Turbomolecular pump is not working correctly

Power indicator on the gauge controller does not light

- Unplugged gauge controller power cord
- Incorrect or inadequate line voltage at the electrical outlet
- Failed gauge controller fuse

Temperature symptoms

The MSD has three heated zones:

- Ion source (**Source** in the MSD ChemStation software)
- Mass filter (**Quad** in the MSD ChemStation software)
- GC/MSD interface (**Thermal Aux #2** in the MSD ChemStation software)

Each of these heated zones has a heater cartridge and temperature sensor. The ion source and mass filter are powered and controlled by the MSD. The GC/MSD interface is powered and controlled by the GC.

Ion source will not heat up

- High vacuum pump is off or has not reached normal operating conditions*
- Incorrect temperature setpoint
- Ion source has not had enough time to reach temperature setpoint
- Ion source heater cartridge is not connected*
- Ion source temperature sensor is not connected*
- Ion source heater failed (burned out or shorted to ground)*
- Ion source temperature sensor failed*
- Source power cable is not connected to the side board*
- MSD electronics are not working correctly

* These will cause an error message.

Mass filter (quad) heater will not heat up

- High vacuum pump is off or has not reached normal operating conditions*
- Incorrect temperature setpoint
- Mass filter has not had enough time to reach temperature setpoint
- Mass filter heater cartridge is not connected*
- Mass filter temperature sensor is not connected*
- Mass filter heater failed (burned out or shorted to ground)*
- Mass filter temperature sensor failed*
- Source power cable is not connected to the sideboard*
- MSD electronics are not working correctly

* These will cause an error message.

GC/MSD interface will not heat up

- Incorrect setpoint
- Setpoint entered in wrong heated zone
- GC/MSD interface has not had enough time to reach temperature setpoint
- GC is off
- GC experienced a fault and needs to be reset*
- GC/MSD interface heater/sensor cable is not connected*
- GC/MSD heater failed (burned out)*
- GC/MSD sensor failed*
- GC electronics are not working correctly*

* These will cause a GC error message. GC error messages are described in the documentation supplied with your GC.

Error messages

Sometimes, a problem in your MSD will cause an error message to appear in the MSD ChemStation software. Some error messages appear only during tuning. Other messages will appear during tuning or data acquisition. Sometimes, instead of a message, only a number will appear. This number will represent one or more error messages. To translate a number into an error message:

1. Note the number.
2. Select **Diagnostics/Vacuum Control** from the View menu.
3. Select **MS Error Codes** from the Status menu.
4. Type in the error number and press **Enter**.

The corresponding error message(s) will appear.

Some error messages are "latched". These messages remain active in your data system even if the condition that caused the message has corrected itself. If the cause is removed, these messages can be removed by checking instrument status through the data system.

Difficulty in mass filter electronics

- Pressure in the analyzer chamber is too high
- RFPA is not adjusted correctly
- Mass filter (quad) contacts are shorted or otherwise not working correctly
- Mass filter is not working correctly
- MSD electronics are not working correctly

Difficulty with the electron multiplier supply

- Large peak such as the solvent peak eluted while the analyzer was on
- Pressure in the analyzer chamber is too high
- MSD electronics are not working correctly

Difficulty with the fan

If a cooling fan fault occurs, the vacuum control electronics automatically shut off the high vacuum pump and the ion source and mass filter heaters. Therefore, the message: *The system is in vent state* may also appear. It is important to note that even though the high vacuum pump is off, the analyzer chamber may not actually be vented. See *The system is in vent state* in this section for precautions to take.

- One of the fans is disconnected
- One of the fans has failed
- MSD electronics are not working correctly

Difficulty with the HED supply

The only time this error occurs is if the output of the supply cannot get to its destination (the HED):

- Large peak such as the solvent peak eluted while the analyzer was on
- Pressure in the analyzer chamber is too high
- Detector is not working correctly
- MSD electronics are not working correctly

Difficulty with the high vacuum pump

In an MSD equipped with a turbomolecular pump, this indicates the pump failed to reach 50% of full speed within 7 minutes or experienced a fault.

You must switch the MSD off and back on to remove this error message. Be sure the turbo pump has slowed down before switching off the MSD. The message will reappear if the underlying problem has not been corrected.

Turbomolecular pump

- Large vacuum leak is preventing the turbo pump from reaching 50% of full speed
- Foreline pump is not working correctly
- Turbo pump is not working correctly
- Turbo pump controller is not working correctly
- MSD electronics are not working correctly

High Foreline pressure

- Excessive carrier gas flow (typically > 5 ml/min)
- Excessive solvent volume injected
- Large vacuum leak
- Severely degraded foreline pump oil
- Collapsed or kinked foreline hose
- Foreline pump is not working correctly

Internal MS communication fault

- MSD electronics are not working correctly

Lens supply fault

- Electrical short in the analyzer
- MSD electronics are not working correctly

Log amplifier ADC error

- MSD electronics are not working correctly

No peaks found

- Emission current was set to 0
- Electron multiplier voltage is too low
- Amu gain or offset is too high
- Poor mass axis calibration
- Amu gain or offset is too high

- Calibration vial is empty or almost empty
- Excessive pressure in the analyzer chamber
- Air leak
- Electron multiplier voltage is too low
- Signal cable is not connected
- Electrical leads to the detector are not connected correctly
- HED power supply output cable failed
- Electrical leads to the ion source are not connected correctly
- Filament shorted to the source body

Temperature control disabled

- One of the heater fuses has failed
- MSD electronics are not working correctly

Temperature control fault

This indicates that something has gone wrong with the temperature control of either the ion source or mass filter (quad) heater. The cause can be further isolated by selecting **Status/MS Temp Ctlr Status** in the Diagnostics/Vacuum Control view. One of the following should be displayed as the cause:

- Source temperature sensor is open
- Source temperature sensor is shorted
- Mass filter (quad) temperature sensor is open
- Mass filter (quad) temperature sensor is shorted
- No heater voltage (heater fuse has probably failed)
- Heater voltage is too low
- Temperature zone has timed out (heater failed, bad heater wiring, or loose temperature sensor)
- Problem with the temperature control electronics
- Source heater is open
- Source heater is shorted
- Mass filter heater is open
- Mass filter heater is shorted

The high vacuum pump is not ready

- Turbomolecular pump is on but has not had enough time (5 minutes) to reach 80% of its normal operating speed
- Turbomolecular pump is not working correctly
- MSD electronics are not working correctly

The system is in standby

This message is triggered by a shutdown signal on the remote start cable. It is usually caused by a GC fault, an ALS fault, or a bad cable connection. Once the cause of the fault is corrected, selecting **MS ON** or checking MSD status should remove the message.

The system is in vent state

Although the message says the system is in vent state, if the fault has just occurred the MSD may actually still be under vacuum and the turbo pump may still be at high speed. Wait at least 30 minutes after seeing this message before you actually vent the MSD.

CAUTION Venting the MSD too soon after this message appears can result in damage to a turbomolecular pump.

- System was vented on purpose (no problem)
- Fan fault has turned off the high vacuum pump (power cycle the MSD to clear the fault)
- Fuse for the high vacuum pump has failed
- MSD electronics are not working correctly

There is no emission current

- Filament is not connected properly. Try the other filament
- Filament has failed. Try the other filament
- MSD electronics are not working correctly

There is not enough signal to begin tune

- Corrupted tune file
- Poor mass axis calibration
- Amu gain or offset is too high
- Calibration vial is empty or almost empty
- Excessive pressure in the analyzer chamber
- Air leak
- Electron multiplier voltage is too low
- Signal cable is not connected
- Electrical leads to the detector are not connected correctly
- Electrical leads to the ion source are not connected correctly
- Filament shorted to the source body

Air leaks

Air leaks are a problem for any instrument that requires a vacuum to operate. Leaks are generally caused by vacuum seals that are damaged or not fastened correctly. Symptoms of leaks include:

- Higher than normal analyzer chamber pressure or foreline pressure
- Higher than normal background
- Peaks characteristic of air (*m/z* 18, 28, 32, and 44 or *m/z* 14 and 16)
- Poor sensitivity
- Low relative abundance of *m/z* 502 (this varies with the tune program used)

Leaks can occur in either the GC or the MSD. The most likely point for an air leak is a seal you recently opened.

In the GC, most leaks occur in:

- Injection port septum
- Injection port column nut
- Broken or cracked capillary column

Leaks can occur in many more places in the MSD:

- GC/MSD interface column nut
- Side plate O-ring (all the way around)
- Vent valve O-ring
- Triode gauge tube (cracked at connector)
- Calibration valve
- GC/MSD interface O-ring (where the interface attaches to the analyzer chamber)
- Front and rear end plate O-rings
- Turbomolecular pump O-ring

Contamination

Contamination is usually identified by excessive background in the mass spectra. It can come from the GC or from the MSD. The source of the contamination can sometimes be determined by identifying the contaminants. Some contaminants are much more likely to originate in the GC. Others are more likely to originate in the MSD.

Contamination originating in the GC typically comes from one of these sources:

- Column or septum bleed
- Dirty injection port
- Injection port liner
- Contaminated syringe
- Poor quality carrier gas
- Dirty carrier gas tubing
- Fingerprints (improper handling of clean parts)

Contamination originating in the MSD typically comes from one of the following sources:

- Air leak
- Cleaning solvents and materials
- Foreline pump oil
- Fingerprints (improper handling of clean parts)

The following table lists some of the more common contaminants, the ions characteristic of those contaminants, and the likely sources of those contaminants.

Table 4 **Common contaminants**

Ions (*m/z*)	Compound	Possible source
18, 28, 32, 44 or 14, 16	H_2O, N_2, O_2, CO_2 or N, O	Residual air and water, air leaks, outgassing from Vespel ferrules

Table 4 Common contaminants

Ions (*m/z*)	Compound	Possible source
31, 51, 69, 100, 119, 131, 169, 181, 214, 219, 264, 376, 414, 426, 464, 502, 576, 614	PFTBA and related ions	PFTBA (tuning compound)
31	Methanol	Cleaning solvent
43, 58	Acetone	Cleaning solvent
78	Benzene	Cleaning solvent
91, 92	Toluene or xylene	Cleaning solvent
105, 106	Xylene	Cleaning solvent
151, 153	Trichloroethane	Cleaning solvent
69	Foreline pump oil or PFTBA	Foreline pump oil vapor or calibration valve leak
73, 147, 207, 221, 281, 295, 355, 429	Dimethylpolysiloxane	Septum bleed or methyl silicone column bleed
77, 94, 115, 141, 168, 170, 262, 354, 446	Diffusion pump fluid and related ions	Diffusion pump fluid
149	Plasticizer (phthalates)	Vacuum seals (O-rings) damag- ed by high temperatures, vinyl gloves
Peaks spaced 14 amu apart	Hydrocarbons	Fingerprints, foreline pump oil

5

CI Troubleshooting

Troubleshooting

This chapter outlines the troubleshooting of 5973 inert MSDs equipped with PCI/NCI. Most of the troubleshooting information in the previous chapter also applies to CI MSDs.

Common CI-specific problems

Because of the added complexity of the parts required for CI, there are many potential problems added. By far the greatest number and most serious problems with CI are associated with leaks or contamination in the reagent gas introduction system. NCI is especially sensitive to the presence of air, and air leaks small enough to cause no problems in PCI can destroy NCI sensitivity.

As with EI, if the MSD tunes well, and no air leak is present, sample sensitivity problems should be addressed by GC injection port maintenance first.

- Wrong reagent gas
- Reagent gas not hooked up or hooked up to wrong reagent gas inlet port
- Wrong ions entered in tune file
- Wrong tune file selected
- Not enough bake-out time has elapsed since vent (background is too high)
- Wrong column positioning (extending > 2 mm past tip of interface.)
- Interface tip seal not installed
- EI source installed in CI mode
- EI filament or other EI source parts in CI ion source
- Air leaks in reagent gas flow path
- CI filament has stretched and sagged:
    - High EMV
    - Linear (no inflection point) electron energy (EIEnrgy) ramp.

Troubleshooting tips and tricks

The following are general rules for troubleshooting, with specific CI examples.

Rule 1: "Look for what has been changed."

Many problems are introduced accidentally by human actions. Every time any system is disturbed, there is a chance of introducing a new problem.

- If the MSD was just pumped down after maintenance, suspect air leaks or incorrect assembly.
- If the reagent gas bottle or gas purifier were just changed, suspect leaks or contaminated or incorrect gas.
- If the GC column was just replaced, suspect air leaks or contaminated or bleeding column.
- If you have just switched ion polarity or reagent gas, suspect the tune file you have loaded in memory. Is it the appropriate file for your mode of operation?

***Rule 2: "If* complex *isn't working, go back to* simple."**

A complex task is not only more difficult to perform, but also more difficult to troubleshoot as well. For example, CI requires more parts to work correctly than EI does.

- If you're having trouble with NCI, verify that PCI still works.
- If you're having trouble with other reagent gases, verify that methane still works.
- If you're having trouble with CI, verify that EI still works.

Rule 3: "Divide and conquer."

This technique is known as "half-split" troubleshooting. If you can isolate the problem to only part of the system, it is much easier to locate.

- To isolate an air leak, start by shutting the gas select valve while leaving the isolation valve and MFC open (turn on **Purge** and **Gas Off**.) If abundance of *m/z* 32 decreases, the problem is "upstream" of the flow module.

Air leaks

How do I know if I have an air leak?

Large air leaks can be detected by vacuum symptoms: loud gurgling noise from the foreline pump, inability of the turbo pump to reach 95% speed, or, in the case of smaller leaks, high pressure readings on the high vacuum gauge controller.

Note that the mass flow controller is calibrated for methane, and the high vacuum gauge controller is calibrated for nitrogen, so these measurements are not accurate in absolute terms, but are intended as a guide to typical observed readings. They were taken with the following set of conditions:

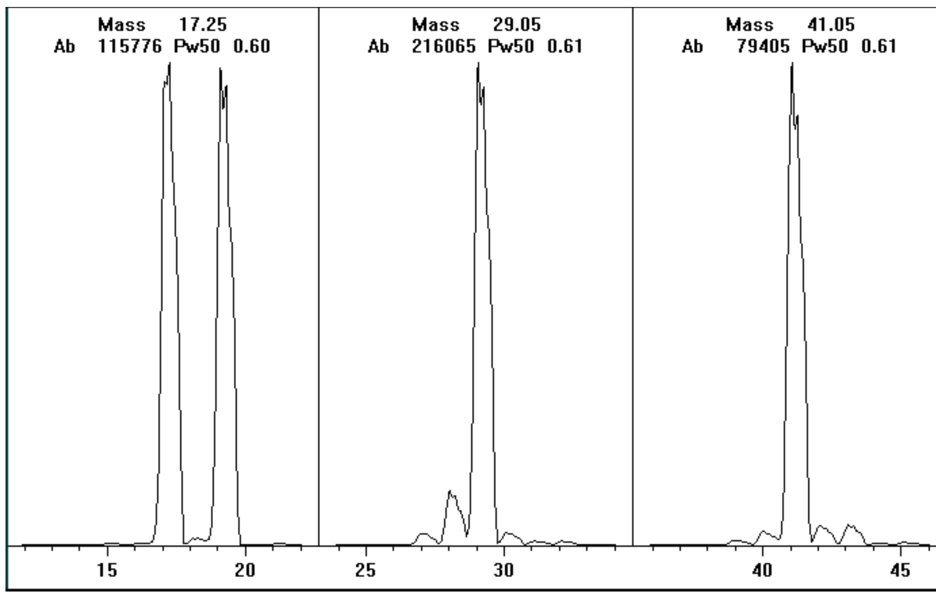
Source temperature	250°C
Quad temperature	150°C
Interface temperature	320°C
Helium column flow	1 mL/min

MFC (%)	**Pressure (Torr)**	
	Methane	**Ammonia**
	Performance turbo pump	Performance turbo pump
10	5.5×10^{-5}	5.0×10^{-5}
15	8.0×10^{-5}	7.0×10^{-5}
20	1.0×10^{-4}	8.5×10^{-5}
25	1.2×10^{-4}	1.0×10^{-4}
30	1.5×10^{-4}	1.2×10^{-4}
35	2.0×10^{-4}	1.5×10^{-4}
40	2.5×10^{-4}	2.0×10^{-4}

Familiarize yourself with the measurements on ***your*** system under operating conditions, and watch for ***changes*** that may indicate a vacuum or gas flow problem.

Always look for small air leaks when setting up methane flow. Run the ***methane*** pretune (starting with a good PCI tune file). The abundance of *m/z* 19 (protonated water) should be less 50% of *m/z* 17 for acceptable PCI performance; for NCI, the abundance of *m/z* 19 (protonated water) should be less than 25% that of *m/z* 17. If the MSD was just pumped down, look for the abundance of *m/z* 19 to be decreasing.

There should not be any peak visible at *m/z* 32 (O_2). This almost always indicates an air leak.



Special negative CI notes

Since NCI is so extremely sensitive, air leaks that are not detectable in EI or PCI can cause sensitivity problems in NCI. To check for this kind of air leak in NCI, inject OFN. The base peak should be at m/z 272. If the abundance of m/z 238 is much greater than that of m/z 272, you have an air leak.

How do I find the air leak?

1 **Look for the last seal that was disturbed.**

- If you just pumped down the MSD, press on the sideplate to check for proper seal. Poor alignment between the analyzer and the GC/MSD interface seal can prevent the sideplate from sealing.
- If you just replaced the reagent gas bottle or gas purifier, check the fittings you just opened and refastened.

2 **Check for tightness of seals at injection port and interface column nuts.**

Ferrules for capillary columns often loosen after several heat cycles. Do not over-tighten the interface nut.

3 **If any of the VCR fittings in the flow module have been loosened and then retightened, the gasket must be replaced. These gaskets are good for one use only.**

CAUTION

Do not loosen the nuts on any VCR fittings unless you intend to replace the gaskets. Otherwise, you ***will*** create an air leak.

4 **Remember that most small air leaks visible in CI mode are located in either the carrier gas or reagent gas flow paths.**

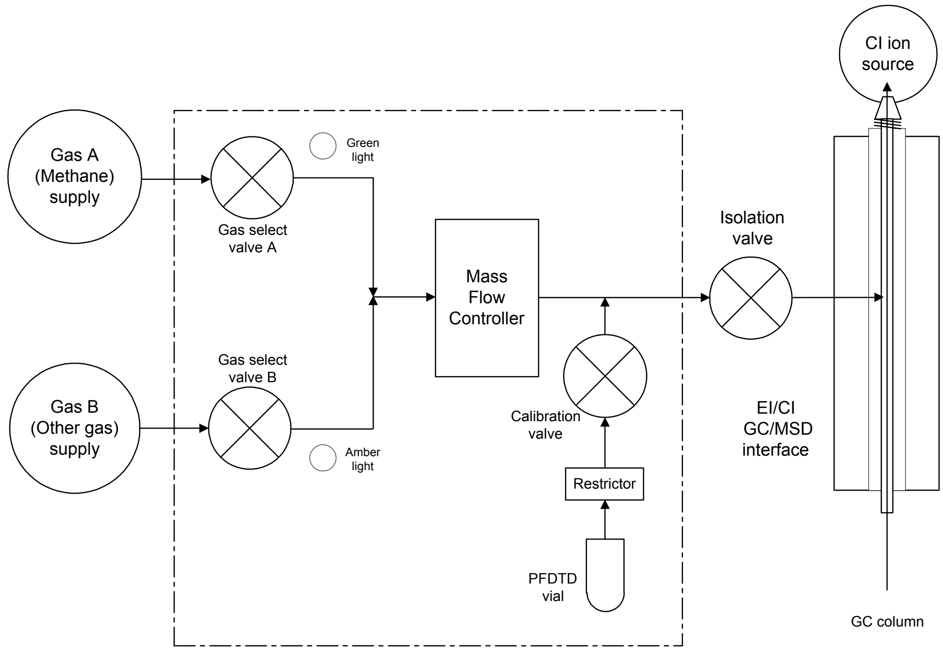
Leaks into the analyzer chamber are not likely to be seen in CI because of the higher pressure inside the ionization chamber.

5 **Half-split the system.**

- By closing valves starting at the gas select valves (**Gas Off** and **Purge** turned on), then moving farther “downstream” to the isolation valve (**Gas Off** turned on and **Purge** turned off.)
- You can cool and vent the MSD, remove the GC column, and cap off the interface.

If you are used to using argon or other introduced gas to find air leaks, note that this does not work well for the reagent gas flow system — it takes as long as 15 minutes for the peak to reach the ion source if the leak is at the inlet to the flow module.

Schematic of CI flow control module



Flow module state diagram:						
Result	Gas A flows	Gas B flows	Purge with Gas A	Purge with Gas B	Pump out flow module	Standby, vented, or EI mode
Control panel lights (LEDs)						
Gas A (green)	On	Off	On	Off	Off	Off
Gas B (amber)	Off	On	Off	On	Off	Off
Purge (red)	Off	Off	On	On	On	Off
Gas Off (red)	Off	Off	Off	Off	On	On
Valve state						
Valve A	Open	Closed	Open	Closed	Closed	Closed
Valve B	Closed	Open	Closed	Open	Closed	Closed
MFC	On → setpoint	On → setpoint	On → 100%	On → 100%	On → 100%	Off (→0%)
Isolation valve	Open	Open	Open	Open	Open	Closed

Pressure-related symptoms (overview)

The following symptoms are all related to high vacuum pressure. Each symptom is discussed in more detail in the following pages.

The mass flow controller is calibrated for methane, and the high vacuum gauge controller is calibrated for nitrogen, so these measurements are not accurate in absolute terms, They are intended as a guide to typical observed readings. They were taken with the following set of conditions:

Source temperature	250°C
Quad temperature	150°C
Interface temperature	320°C
Helium carrier gas flow	1 mL/min.

MFC (%)	**Pressure (Torr)**	
	Methane	**Ammonia**
	Performance turbo pump	Performance turbo pump
10	5.5×10^{-5}	5.0×10^{-5}
15	8.0×10^{-5}	7.0×10^{-5}
20	1.0×10^{-4}	8.5×10^{-5}
25	1.2×10^{-4}	1.0×10^{-4}
30	1.5×10^{-4}	1.2×10^{-4}
35	2.0×10^{-4}	1.5×10^{-4}
40	2.5×10^{-4}	2.0×10^{-4}

Poor vacuum without reagent gas flow

Possible Cause Excess water in the background.

Action Scan from 10 – 40 amu. A large peak at *m/z* 19 (> *m/z* 17) indicates water in the background. If water is present, allow the instrument to bake out more and flow reagent gas through the lines to purge any accumulated water.

Possible Cause Air leak.

Action Run Methane Pretune. See page 86. A visible peak at *m/z* 32 indicates air in the system. Check for and correct any leaks. See the *Leaks* section at the beginning of this chapter.

Possible Cause The foreline pump is not working properly.

Action Replace the pump oil. If that does not help, it may be necessary to replace the pump. Contact your local Agilent Technologies Customer Engineer.

Possible Cause The turbo pump is not working properly.

Action Check the pump speed. It should be at least 95%. Contact your local Agilent Technologies service representative

CAUTION Use of ammonia as reagent gas can shorten the life of the foreline pump oil and possibly of the foreline pump itself. See the Maintenance chapter in this manual.

High pressure with reagent gas flow

Possible Cause The reagent gas flow rate is too high.

Action On the flow controller, turn down reagent gas flow as appropriate. Verify that reagent ion ratios are correct. See page 86.

Possible Cause Air leak.

Action Run Methane Pretune. See page 86. Visible peak at *m/z* 32 indicates air in the system. Check for and correct any leaks. See the *Leaks* section at the beginning of this chapter.

Possible Cause Interface tip seal wasn't installed.

Action Check the source storage box. If the seal is not in the box, vent the MSD and verify that the seal is correctly installed.

Pressure does not change when reagent flow is changed

Possible Cause The reagent gas regulator is closed.

Action Check and, if necessary, open the reagent gas regulator.

Possible Cause The reagent gas regulator is set to the wrong pressure.

Action Set the reagent gas regulator to 10 psi (70 kPa) for methane or to 3 – 10 psi (20 – 70 kPa) for isobutane or ammonia.

Possible Cause The valve on the reagent gas bottle is closed.

Action Check and, if necessary, open the valve on the reagent gas bottle.

Possible Cause The reagent gas supply is empty.

Action Check, and if necessary, replace the reagent gas supply.

Possible Cause Reagent lines kinked, bent, pinched, or disconnected.

Action Inspect the reagent lines and repair any defects. Check especially to make sure the reagent line is connected to the rear of the flow module. Be sure the methane line is connected to the Gas A inlet.

Possible Cause GC/MSD interface clogged or damaged.

Action Check for flow and repair or replace components as indicated.

Signal-related symptoms (overview)

This section describes symptoms related to the signal. The symptom may be too much signal, too little signal, a noisy signal, or an incorrect signal. Signal-related symptoms are generally observed during tuning but may also be observed during data acquisition.

Error messages in autotune due to insufficient signal may vary.

The following symptoms are covered in more detail in this section:

- No peaks
- No or low reagent gas signal. See page 145.
- No or low PFDTD signal. See page 148.
- Excessive noise. See page 150.
- Low chromatographic signal abundance. See page 150.
- Low signal-to-noise ratio. See page 150.
- Large peak at *m/z* 19. See page 151.
- Peak at *m/z* 32. See page 152.

No peaks

When troubleshooting “no peaks” it is important to specify what mode of operation is being used, and what kind of peaks are not being seen. Always start with methane PCI and verify presence of reagent ions.

No reagent gas peaks in PCI

If MSD has been working well and nothing seems to have been changed

- Wrong tune file loaded, or tune file corrupted
- Wrong ion polarity (there are no reagent ions visible in NCI)
- No reagent gas flow; look for background ions and check pressure
- Wrong reagent gas selected for the tune file (looking for wrong ions)
- Large air leak
- Dirty ion source
- Poor vacuum (pump problem). See page 138.

If MSD was recently switched from EI to CI

- Interface tip seal not installed
- No reagent gas flow
- Analyzer not sealed (big air leak)
- Wrong tune file loaded or tune file corrupted
- Ion source not assembled or connected correctly
- Wrong reagent gas selected for the tune file (looking for wrong ions)

No PFDTD peaks in PCI

- Incorrect reagent gas. There ***are*** no PCI PFDTD peaks created with isobutane or ammonia. Switch to methane.
- Analyzer not sealed (big air leak)
- No calibrant in vial
- Defective calibration valve
- Air leak in carrier or reagent gas path

No reagent gas peaks in NCI

- Reagent gases do not ionize in NCI; look for background ions instead.
- Verify tune parameters
- If no background ions are visible, go back to methane PCI

No PFDTD calibrant peaks in NCI

- Look for background ions: 17 (OH-), 35 (Cl-), and 235 (ReO3-).
- Verify tune parameters
- Go back to methane PCI

No sample peaks in NCI

- Look for background ions: 17 (OH-), 35 (Cl-), and 235 (ReO3-).
- Go back to methane PCI
- Poor quality reagent gas (purity less than 99.99%)

Large peak at *m/z* 238 in NCI OFN spectrum

- Look for background ions: 17 (OH-), 35 (Cl-), and 235 (ReO3-).
- Find and fix your small air leak

No or low reagent gas signal

Possible Cause If you have just installed the CI ion source, and have an air leak or large amounts of water in the system, and have run one or more autotunes, the ion source is probably dirty now.

Action Fix the air leak.
Clean the ion source. Then bake out for two hours before tuning. See “To set up your MSD for CI operation” on page 245..

Possible Cause The wrong reagent gas is flowing.

Action Turn on the correct reagent gas for your tune file.

Possible Cause Ion polarity is set to **Negative**. No reagent gas ions are formed in NCI.

Action Switch to **Positive** ionization mode.

Possible Cause The reagent gas flow is set too low.

Action Increase the reagent gas flow.

Possible Cause Reagent gas supply tubing is blocked, kinked, pinched, or disconnected.

Action Inspect and, if necessary, repair or replace the reagent gas supply tubing.

Possible Cause Wrong filament wires are connected to filament.

Action Make sure that the filament 1 wires are connected to the CI ion source filament and that the filament 2 wires are connected to the dummy filament.

No or low reagent gas signal

Possible Cause Carbon has built up on the filament, or filament has sagged out of alignment.

Action Inspect the filament. If necessary, replace the filament.

Possible Cause Too much air or water in the system.

Action Run the methane pretune. Peaks at *m/z* 32 and 19 usually indicate air and water, respectively. Bake out and purge the instrument until there is no visible peak at *m/z* 32 and the peak at *m/z* 19 is reduced to a very low level. If the peak at *m/z* 32 does not decrease, an air leak is likely. See the *Leaks* section at the end of this chapter for more information.

Possible Cause The signal cable is not connected.

Action Check and, if necessary, reconnect the signal cable.

Possible Cause The filament or filament support is shorted to the ion source body or repeller.

Action Inspect the filament. If necessary, realign the filament support arms.

Possible Cause The electron inlet hole is blocked.

Action Inspect the electron inlet hole. If necessary, clean the hole with a clean toothpick and a slurry of aluminum oxide powder and methanol. If the electron inlet hole is that dirty, the entire ion source probably needs to be cleaned. See the *Maintenance* chapter in this manual for more information.

Possible Cause Ion source wires are not connected, or incorrectly connected.

Action Inspect the repeller. Make sure the repeller lead is firmly attached to the repeller. Inspect the wires to the ion focus and entrance lenses. If the connections are reversed, correct the problem.

Possible Cause One of the detector leads (in the analyzer chamber) is not connected.

Action Check and, if necessary, reconnect the electron multiplier leads.

Possible Cause Saturated methane / isobutane gas purifier.

Action Replace the gas purifier.

Possible Cause Poor quality methane (purity below 99.99%.)

Action Replace the methane with high-purity methane. If necessary, clean and purge the reagent gas lines and clean the ion source.

No or low PFDTD signal, but reagent ions are normal

Possible Cause You are flowing any reagent gas but methane in PCI.

Action Switch to methane.

Possible Cause Wrong or corrupted tune file loaded.

Action Check your tune file.

Possible Cause No PFDTD in the calibrant vial.

Action Inspect the calibration vial on the back of the flow controller. If necessary, fill the vial with PFDTD. Do not fill the vial completely; keep the level at least 0.5 cm from the top of the vial.

Possible Cause The pressure of the methane entering the flow controller is too high.

Action Make sure the regulator on the methane supply is set to 10 psig (70 kPa).

Possible Cause The CI ion source is dirty.

Action Clean the ion source. See the *Maintenance* chapter in this manual for more information.

Possible Cause The calibration valve was not purged after the vial was refilled.

Action Purge the calibration valve as described in the *Maintenance* chapter. Then clean the ion source.

Possible Cause The calibrant vial was overfilled. Excess PFDTD can quench the chemical ionization reactions.

Action Check the level of the PFDTD in the calibration vial as described in *Maintenance* chapter.

Possible Cause Poor quality methane (purity below 99.99%.)

Action Replace the methane with high-purity methane. If necessary, clean and purge the reagent gas lines and clean the ion source.

Excessive noise or low signal-to-noise ratio

Possible Cause The GC injection port needs maintenance.

Action Refer to the 6890 Series GC manual.

Possible Cause The CI ion source is dirty.

Action Clean the ion source. See the *Maintenance* chapter in this manual for more information.

Possible Cause Poor vacuum

Action Check the pressure on the high vacuum gauge controller.

Possible Cause Air leak.

Action Run Methane Pretune (in PCI). Large peak at *m/z* 32 indicates air in the system. Check for and correct any leaks. See the *Leaks* section at the beginning of this chapter.

Possible Cause Saturated methane / isobutane gas purifier.

Action Replace the gas purifier.

Possible Cause Poor quality methane (purity below 99.99%.)

Action Replace the methane with high-purity methane. If necessary, clean and purge the reagent gas lines and clean the ion source.

Possible Cause Reagent gas flows too high (in EI/PCI MSDs).

Action Verify that the reagent gas setup is correct.

Large peak at *m/z* 19

If the abundance of the peak at *m/z* 19 is more than half abundance of the peak at *m/z* 17, then there is probably too much water in the system.

Possible Cause The system was not baked out sufficiently after it was last vented.

Action Bake out the system as described in the *Maintenance* chapter of this manual.

Possible Cause Moisture left over in the reagent gas supply tubing and flow module.

Action Purge the reagent gas supply lines for at least 60 minutes. See the *Maintenance* chapter.

Possible Cause Contaminated reagent gas supply.

Action Replace the reagent gas supply and purge the lines and flow module.

Possible Cause Saturated methane / isobutane gas purifier.

Action Replace the gas purifier.

Peak at *m/z* 32

A visible peak at *m/z* 32 in methane pretune often indicates air in the system.

Possible Cause Residual air from recent venting — check for water indicated by a large peak at *m/z* 19.

Action Bake out the system as described in the *Maintenance* chapter of this manual.

Possible Cause New or dirty reagent gas supply tubing.

Action Purge the reagent gas supply lines and flow module *for at least 60 minutes*. See “To set up your MSD for CI operation” on page 245..

Possible Cause Air leak.

Action Check for leaks and correct any that you find. See the *Leaks* section at the end of this chapter for more information. After all leaks have been corrected, clean the ion source.

Possible Cause Contaminated reagent gas supply. Suspect this if you have recently replaced your gas tank, and you have ruled out air leaks.

Action Replace the reagent gas supply.

Possible Cause The capillary column is broken or disconnected.

Action Inspect the capillary column. Make sure it is not broken and it is installed correctly.

Possible Cause Saturated methane / isobutane gas purifier.

Action Replace the gas purifier.

Tuning-related symptoms (overview)

This section describes symptoms related to tuning. Most symptoms involve difficulties with tuning or with the results of tuning. The following symptoms are covered in this section:

- CI ion ratio is difficult to adjust or unstable
- High electron multiplier voltage
- Can not complete autotune
- Peak widths are unstable

Reagent gas ion ratio is difficult to adjust or unstable

Possible Cause The interface tip seal is incorrectly placed, damaged, or missing.

Action Inspect the interface tip seal. If necessary, remove and reinstall it to insure a good seal with the CI ion source. Replace it if it is damaged. Install it if it is missing.

Possible Cause Residual air and water in the MSD or in the reagent gas supply lines.

Action Run the methane pretune. Air will appear as a peak at *m/z* 32 and excessive water as a peak at *m/z* 19 > *m/z* 17. If either of conditions is present, purge the reagent gas supply lines and bake out the MSD. See page 254. Continued presence of a large peak at *m/z* 32 may indicate an air leak. After correcting the problems, you may need to clean the ion source.

Possible Cause Air leak.

Action Run Methane Pretune (in PCI). Large peak at *m/z* 32 indicates air in the system. Check for and correct any leaks. See the *Leaks* section at the beginning of this chapter.

Possible Cause The reagent gas supply is at the wrong pressure.

Action Check the regulator on the reagent gas supply. It should be adjusted to 20 psi (140 kPa).

Possible Cause A leak in the reagent gas delivery path. This is especially likely if you have set the methane flow much higher than normal and the ratio is still too low.

Action Check the reagent gas path. Tighten fittings.

Possible Cause The CI ion source is dirty.

Action Clean the ion source. See the *Maintenance* chapter of this manual for more information.

High electron multiplier voltage

The electron multiplier voltage can range from a few hundred volts to 3000 V. If the CI autotune program consistently sets the electron multiplier voltage at or above 2600 V, but can still find peaks and complete the tune, it may indicate a problem.

Possible Cause The filament is worn out. The CI filament may wear out without actually breaking. Check the Electron Energy ramp; the curve should have a definite maximum with an inflection point. If the curve is linear with a positive slope and no inflection point, and the EMV is high, the filament has stretched to the point where it does not line up with the hole in the ion source body, and most electrons are not getting into the source.

Action Replace the filament.

Possible Cause The analyzer is not at the proper operating temperature.

Action Verify the ion source and quadrupole temperatures. The default source temperature is 250°C for PCI and 150°C for NCI. The quadrupole temperature is 150°C for both CI modes.

Possible Cause The CI ion source is dirty.

Action Clean the ion source. See the *Maintenance* chapter in this manual for more information.

Possible Cause The electron multiplier (detector) is failing. Switch to EI mode and confirm.

Action Replace the electron multiplier.

Can not complete autotune

Possible Cause Wrong or corrupted tune file.

Action Check the tune parameters.

Possible Cause The *m/z* 28/27 ion ratio (for methane) is incorrect. The correct ratio should be between 1.5 and 5.0.

Action If the ion ratio is incorrect, adjust it. See page 98.

Possible Cause The CI ion source is dirty.

Action Clean the ion source. See the *Maintenance* chapter in this manual for more information.

Possible Cause Too much air or water in the system.

Action See the *Leaks* section of this chapter for more information. After eliminating these problems, clean the ion source.

Peak widths are unstable

Possible Cause Wrong or corrupted tune file.

Action Check the tune parameters.

Possible Cause The CI ion source is dirty.

Action Clean the ion source. See the *Maintenance* chapter of this manual for more information.

Possible Cause Air leak.

Action Run Methane Pretune (in PCI). A visible peak at *m/z* 32 indicates air in the system. Check for and correct any leaks. See the *Leaks* section at the beginning of this chapter. After eliminating all air leaks, clean the ion source.

Maintaining the MSD

How to perform common maintenance procedures for the MSD. Many of these procedures are demonstrated on the MSD Maintenance CD-ROM.

Before starting

You can perform much of the maintenance required by your MSD. For your safety, read all of the information in this introduction before performing any maintenance tasks.

Some parts of the MSD require regularly scheduled maintenance

Common maintenance tasks are listed in Table 4. 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 5 **Maintenance schedule**

Task	Every week	Every 6 months	Every year	As needed
Tune the MSD				✓
Check the foreline pump oil level	✓			
Check the calibration vial(s)		✓		
Replace the foreline pump oil[1]		✓		
Clean the ion source				✓
Check the carrier gas trap(s) on the GC				✓
Replace the worn out parts				✓
Lubricate sideplate or vent valve O-rings[2]				✓

1 Every 3 months for CI MSDs using ammonia reagent gas.

2 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.

Maintenance requires the proper tools, spare parts, and supplies

Some of the required tools, spare parts, and supplies are included in the GC shipping kit, MSD shipping kit, or MSD tool kit. You must supply others yourself. Each maintenance procedure includes a list of the materials required for that procedure. Tables 5 and 6 summarize these.

Table 6 **Tools**

Description	Part number
Ball driver	
1.5-mm	8710-1570
2.0-mm	8710-1804
2.5-mm	8710-1681
Funnel	————
Hex nut driver, 5.5-mm	8710-1220
Pliers, long-nose (1.5-inch nose)	8710-1094
Screwdriver	
flat-blade, large	8730-0002
Torx, T-10	8710-1623
Torx, T-15	8710-1622
Torx, T-20	8710-1615
Tweezers, non-magnetic	8710-0907
Wrench, open-end	
1/4-inch × 5/16-inch	8710-0510
10-mm	8710-2353
Wrist strap, anti-static	
small	9300-0969
medium	9300-1257
large	9300-0970

Table 7 Spare parts and supplies

Description	Part number
Abrasive paper	5061-5896
Alumina abrasive powder	
1 kilo	8660-0791
Aluminum foil, clean	————
Beakers, glass, 50 ml and 500 ml	————
Cloths, clean, lint-free (quantity 20)	05980-60051
Container, for catching old foreline pump oil, ≈ 500 ml	————
Cotton swabs (quantity 100)	5080-5400
Electron multiplier horn	05971-80103
Filament assembly	G2590-60053
CI filament assembly	G1099-80053
Foreline exhaust oil trap	G1099-80037
Foreline pump oil, 1 liter	6040-0834
Gloves	
chemical-resistant (resistant to oils and solvents)	————
clean, lint-free (large)	8650-0030
clean, lint-free (small)	8650-0029
Grease, Apiezon L, high vacuum	6040-0289
Heater/sensor assemblies	
GC/MSD interface	G1099-60107
ion source	G2589-60177
mass filter	G1099-60172
Octafluoronaphthalene, 1 pg/ul	8500-5441
Perfluorotributylamine (PFTBA), certified (1 gram)	8500-0656
PFTBA sample kit	05971-60571
Evaluation sample A	05970-60045
Solvents, reagent-grade — acetone, methanol, methylene chloride	————
Triode gauge tube	0960-0897
Ultrasonic bath	————

Many parts of the MSD carry high voltages that are potentially dangerous

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
- Turbomolecular pump controller
- 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 MSD turned on or plugged into its power source unless you are instructed to by one of the procedures in this chapter.

One or two procedures in this chapter require access to the inside of the 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.

If your instrument is equipped with the optional gauge controller, potentially dangerous voltage also exists where the cable from the gauge controller connects to the triode gauge tube. Turn off the gauge controller if you are going to be working near the triode gauge tube.

Many parts are hot enough to be dangerous

Many parts in the MSD 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

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

WARNING **The GC/MSD interface heater is powered by the Thermal Aux #2 heated zone on the GC. The interface heater can be on, and at a dangerously high temperature, even though the MSD is off. The GC/MSD interface is well insulated. Even after it is turned off, it cools very slowly.**

The GC injection ports and GC oven also operate at very high temperatures. Use the same caution around these parts. See the documentation supplied with your GC for more information.

Chemical residue is another potential danger

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. It will also contain tiny droplets of foreline pump oil.

An oil trap is supplied with the 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 the oil trap. Instead, install a hose to take the exhaust from the foreline pump outdoors or into a fume hood vented to the outdoors. Be sure to comply with your local air quality regulations.

WARNING

The oil trap 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. Do not use the trap if you have a CI MSD. Install a hose to take the foreline pump exhaust outside or to a fume hood.

The fluids in the foreline pump also collect 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 is a threat to the MSD electronics during maintenance

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 anti-static wrist strap and take other anti-static 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, anti-static mat, if you must work on components or assemblies that have been removed from the MSD. This includes the analyzer.

CAUTION

In order to be effective, an anti-static wrist strap must fit snugly (not tight). A loose strap provides little or no protection.

CAUTION

Anti-static 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

The vacuum system requires some periodic maintenance

As listed earlier in Table 4, 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 (daily in MSDs using ammonia reagent gas)
- Replacing the foreline pump oil (every 6 months; every 3 months for CI MSDs using ammonia reagent gas)
- Tightening the foreline pump oil box screws (first oil change after installation)

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

Other procedures should be performed as needed

Tasks such as replacing a triode gauge tube should be performed only when needed. See “Troubleshooting the MSD” on page 103 and see 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 “Vacuum System” on page 257

Most of the procedures in this chapter are illustrated with video clips in the 5973 inert MSD Manual CD-ROM.

To check and add foreline pump oil

Materials needed:

Foreline pump oil (6040-0834)
Funnel

See also

For a video demonstration of this procedure, click on the icon.

1 **Examine the oil level window.**

The oil level should be above the lower line. The foreline pump oil should be almost clear. If the oil level is near or below the lower line, follow the steps 2 - 6 to add foreline pump oil.

WARNING

Never add oil while the foreline pump is on.

If your MSD is nearing its scheduled time for replacement of the foreline pump oil, replace the oil instead of adding oil. If the oil is dark or cloudy, replace it. See “To drain the foreline pump” on page 172 for instructions about replacing the foreline pump oil.

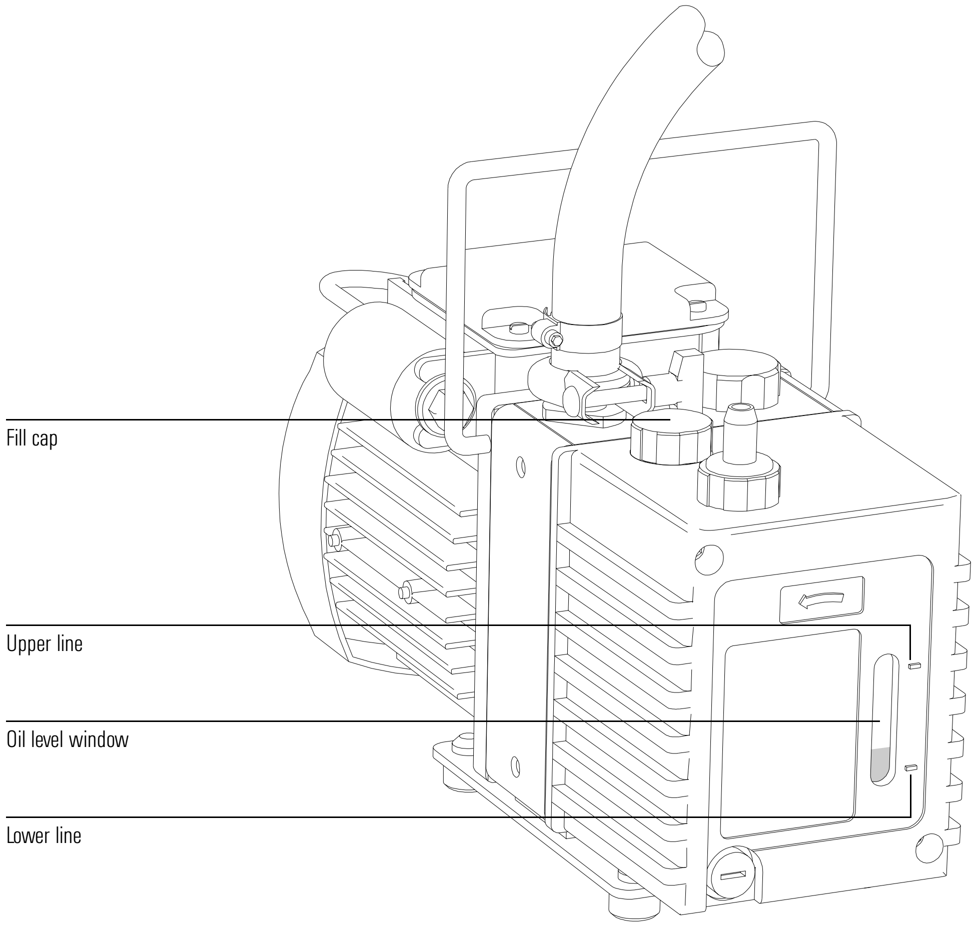
2 **Vent the MSD. See page 64.**

3 **Remove the fill cap.**

4 **Add pump fluid until the oil level int he window is near, but not above, the upper line.**

5 **Reinstall the fill cap.**

6 **Pump down the MSD. Pump down the MSD. See page 70.**


Fill cap
Upper line
Oil level window
Lower line

To drain the foreline pump

Materials needed:

Book or other solid object approximately 5 cm thick
Container for catching old pump oil, 500 ml
Gloves, oil- and solvent-resistant
Screwdriver, flat-blade, large (8730-0002)

See also

For a video demonstration of this procedure, click on the icon.

1 Vent the MSD. See page 64.

2 If necessary, slide the foreline pump out from under the analyzer chamber.

The foreline pump may be located on the floor, on the lab bench next to or behind the MSD, or under the analyzer chamber at the back of the MSD.

WARNING **The foreline pump may be hot.**

3 Place a book or other object under the pump motor to tilt it up slightly.

4 Remove the fill cap.

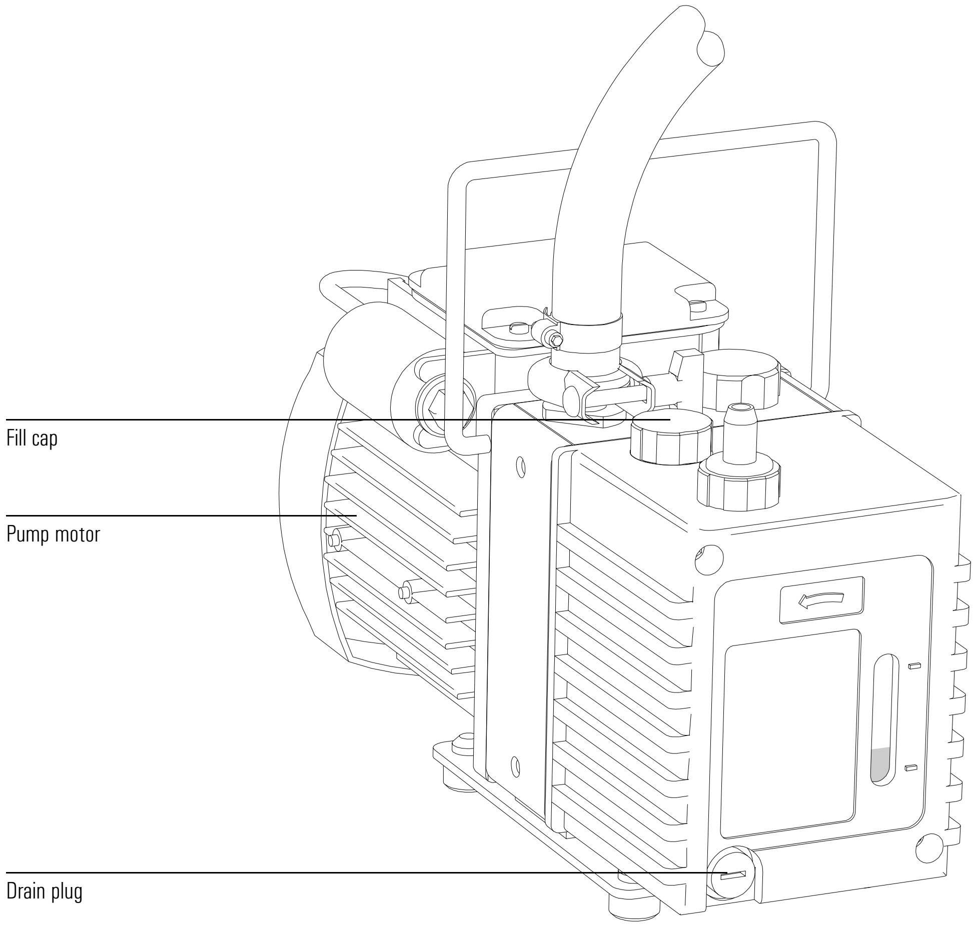
5 Place a container under the drain plug.

6 Remove the drain plug.

Allow the pump oil to drain out. The oil drains faster if it is still warm.

WARNING **The old pump oil may contain toxic chemicals. Treat it as hazardous waste.**

7 Refill the foreline pump. See page 174.


Fill cap
Pump motor
Drain plug

To refill the foreline pump

Materials needed:

Foreline pump oil (6040-0834) – approximately 0.28 liters required
Funnel
Gloves, oil- and solvent-resistant
Screwdriver, flat-blade, large (8730-0002)
Drain plug O-ring (if required) (0905-1515)

See also

For a video demonstration of this procedure, click on the icon.

1 **Drain the foreline pump. See page 172.**

2 **Reinstall the drain plug.**

If the old O-ring appears worn or damaged, replace it.

3 **Remove the propping object from under the pump motor.**

4 **Add foreline pump oil until the oil level in the window is near, but not above, the upper line.**

The foreline pump requires approximately 0.28 liters of oil.

5 **Wait a few minutes for the oil to settle.**

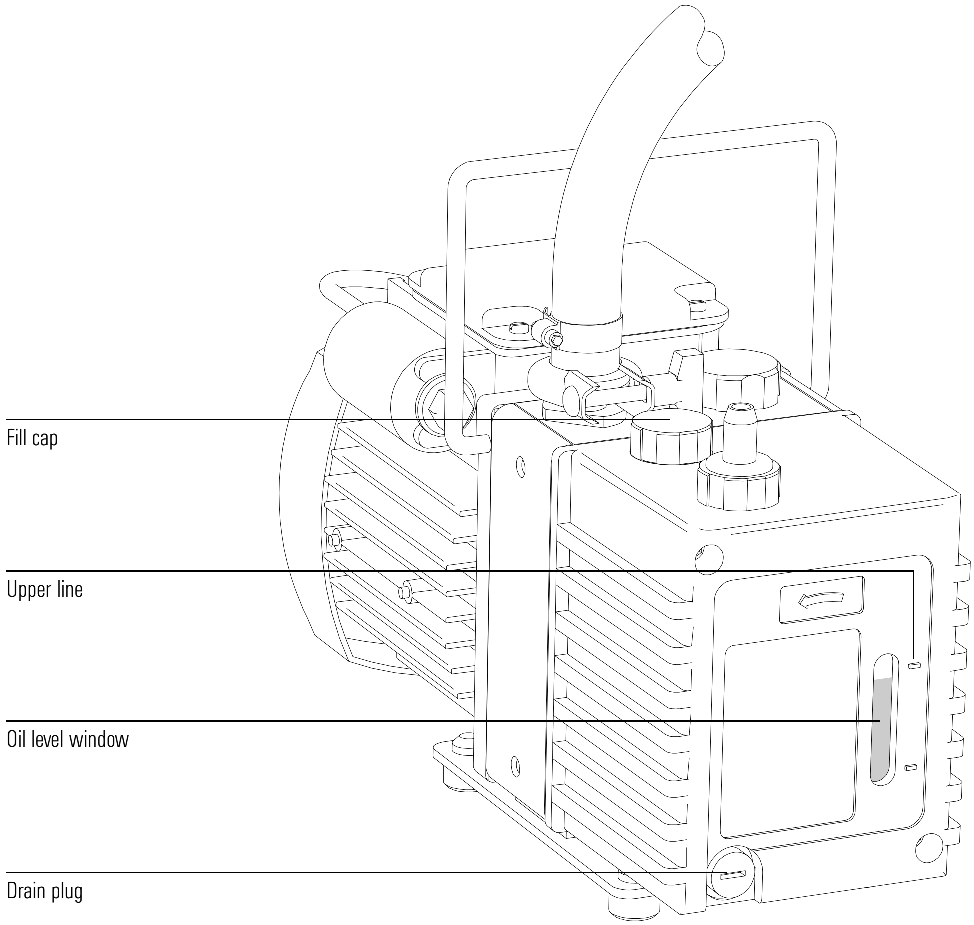
If the oil level drops, add oil to bring the oil level to near the upper line.

6 **Reinstall the fill cap.**

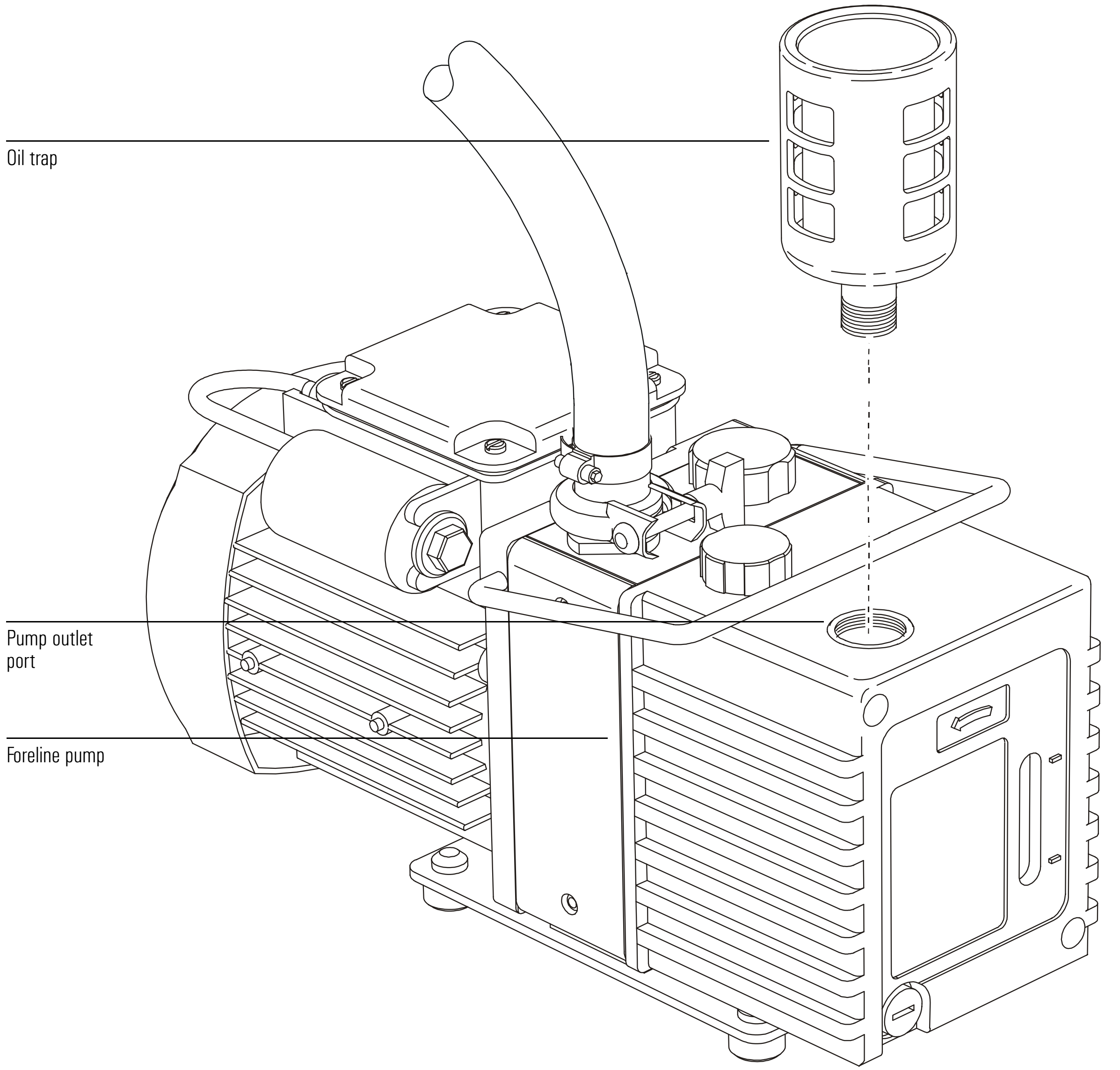
7 **If necessary, slide the foreline pump back under the analyzer chamber.**

The foreline pump may be located on the floor, on the lab bench next to or behind the MSD, or under the analyzer chamber at the back of the MSD.

8 **Pump down the MSD. See page 70.**


Fill cap
Upper line
Oil level window
Drain plug

To refill the foreline pump



To replace the turbo pump

The turbo pumps are ***not*** customer-replaceable parts. The procedure is demonstrated on the 5973 inert Manual CD-ROM, and is for use by Agilent Technologies service personnel only.

To separate the MSD from the GC

Materials needed: Wrench, open-end, 1/4-inch × 5/16-inch (8710-0510)

1 **Vent the MSD. See page 64.**

2 **Turn off the GC.**

3 **Remove the capillary column from the GC/MSD interface.**

WARNING **Make sure the GC/MSD interface and GC oven have cooled before you remove the column.**

4 **If necessary, slide the foreline pump out from under the analyzer chamber.**

The foreline pump may be located on the floor, on the lab bench next to or behind the MSD, or under the analyzer chamber at the back of the MSD.

5 **Move the MSD away from the GC until you have access to the GC/MSD interface cable.**

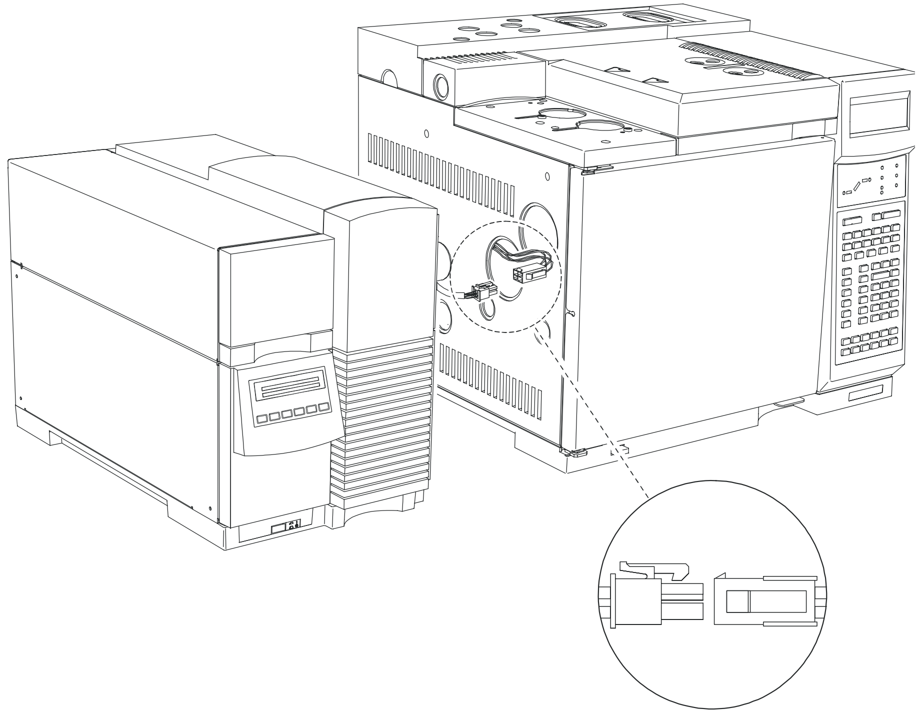
6 **Place a column nut with a blank ferrule on the end of the interface.**

This will help prevent contamination out of the MSD.

7 **Disconnect the GC/MSD interface cable.**

Disconnecting the cable with the GC on can cause a fault condition.

8 **Continue to move the MSD until you have access to the part requiring maintenance.**



To reconnect the MSD to the GC

Materials needed: Wrench, open-end, 1/4-inch × 5/16-inch (8710-0510)

1. **Position the MSD so the end of the GC/MSD interface is near the GC.**

2. **Reconnect the GC/MSD interface cable.**

3. **Slide the MSD to its regular position next to the GC.**

   Be careful not to damage the GC/MSD interface as it passes into the GC. Make sure the end of the GC/MSD interface extends into the GC oven.

4. **If necessary, slide the foreline pump back under the analyzer chamber.**

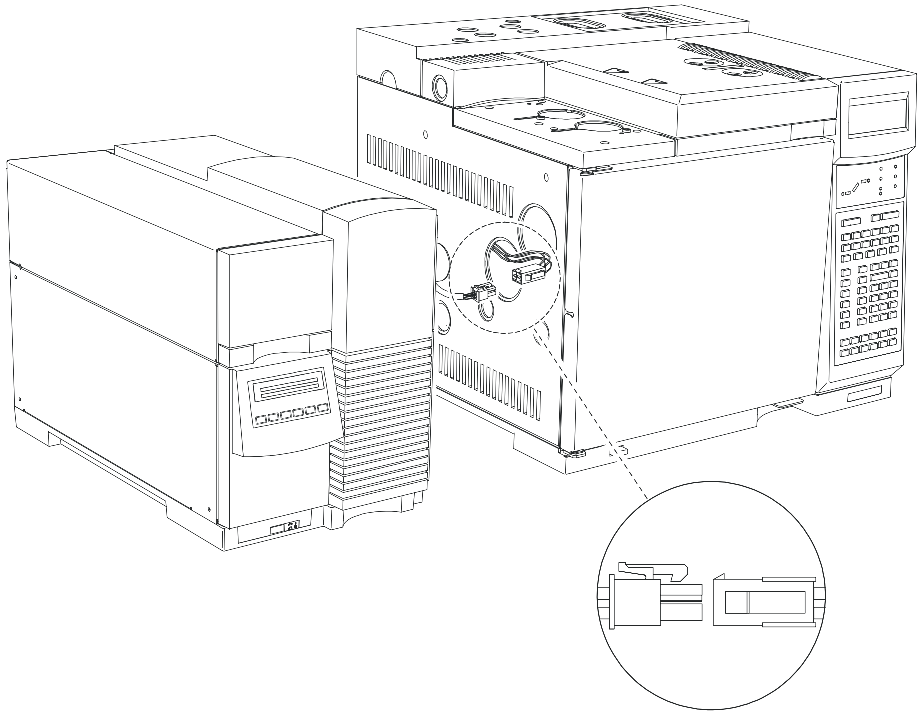
   The foreline pump may be located on the floor, on the lab bench next to or behind the MSD, or under the analyzer chamber at the back of the MSD.

5. **Reinstall the capillary column.** **See page 38.**

6. **Pump down the MSD.** **See page 70.**

7. **Turn on the GC.**

   Re-establish appropriate temperature setpoints for the GC/MSD interface and GC oven.



To remove the EI calibration vial

Materials needed: None

1 **Stop any tuning or data acquisition.**

2 **Turn off the analyzer.**

There are several ways to turn off the analyzer:

- In the Tune and Vacuum Control view, select **MS OFF** from the Execute menu.
- In the Instrument Control view in the Edit Parameters dialog box, select **MS OFF** from the Execute menu.
- In the Tune and Vacuum Control view, select **MS OFF** from the Execute menu.

3 **If your MSD is equipped with a gauge controller, switch off the triode gauge and the gauge controller.**

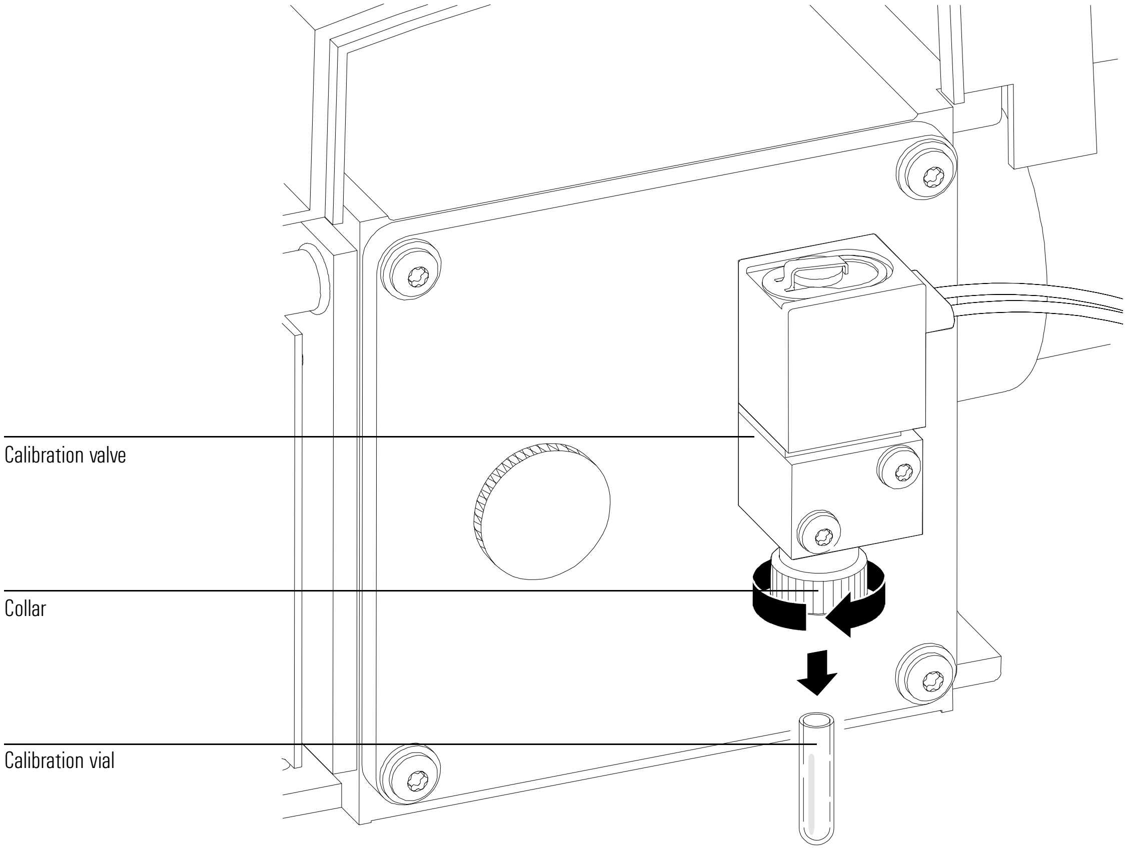
4 **Remove the analyzer cover. See page 62.**

5 **Loosen the calibration vial collar by turning it counterclockwise.**

Counterclockwise as viewed from the bottom (vial side) of the collar. Do not remove the collar.

6 **Pull the calibration vial out.**

You may feel some resistance due to residual vacuum.


Calibration valve
Collar
Calibration vial

To refill and reinstall the EI calibration vial

Materials needed: PFTBA (05971-60571) or other tuning compound

1 **Remove the calibration vial. See page 187.**

2 **Pour PFTBA into the vial, or use a pipette.**

Leave the top 6-mm of the vial unfilled.

3 **Push the calibration vial into the valve as far as possible.**

4 **Withdraw the vial 1 mm.**

This prevents damage when you tighten the collar.

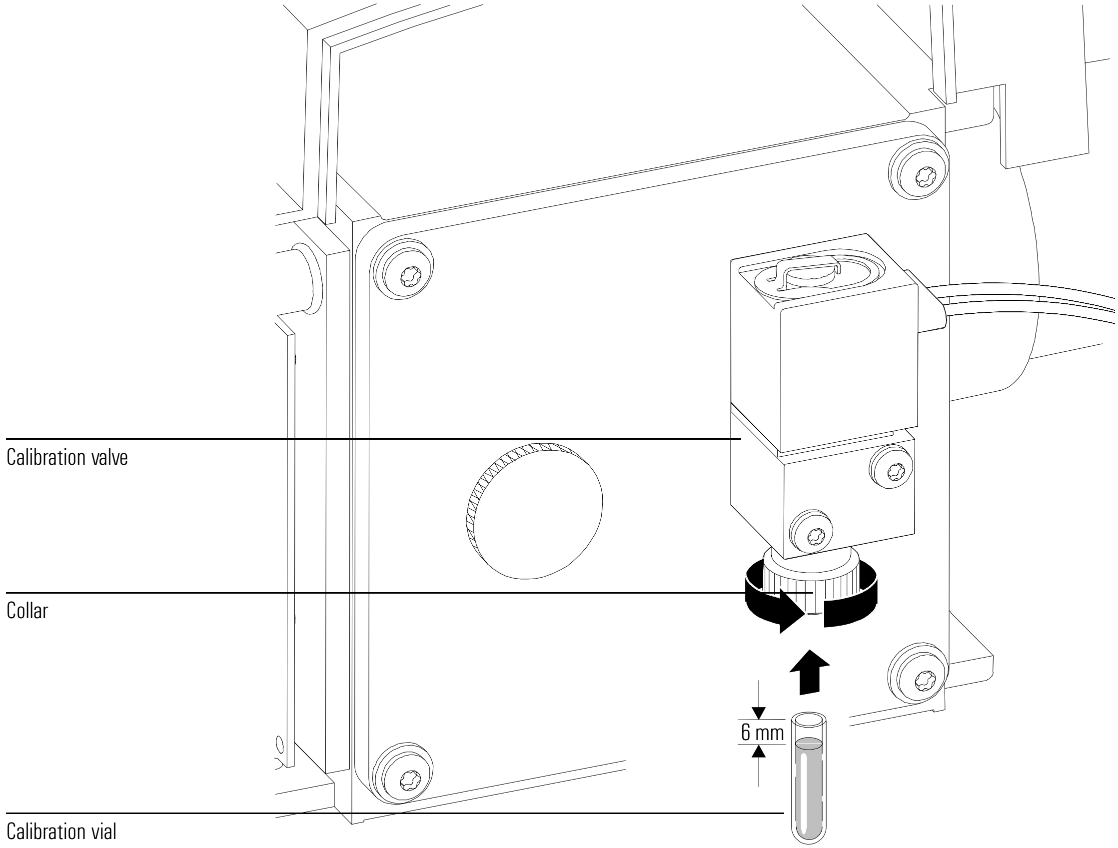
5 **Turn the collar clockwise to tighten it.**

Clockwise as viewed from the bottom (vial side) of the collar. The collar should be snug but not overly tight. Do ***not*** use a tool to tighten the collar. It does not require that much force.

6 **Reinstall the analyzer cover.**

7 **Select Purge Cal Valve from the Vacuum menu in the Tune and Vacuum Control view.**

CAUTION Failure to purge the calibration valve will result in damage to the filaments and detector.


Calibration valve
Collar
6 mm
Calibration vial

To purge the calibration valves

CAUTION

After removing a calibrant vial, you ***must*** purge the calibration valve. Failure to do so will result in damage to the filaments and the electron multiplier.

EI calibration valve

After adding new PFTBA to the calibrant vial, you must purge the air out of the vial and valve.

1 **If the vacuum gauge controller is on, turn it off.**

2 **In Diagnostics and Vacuum Control view, select Purge Calibrant Valve under the Vacuum menu.**

This will open the CI calibration valve for several minutes with all analyzer voltages turned off.

Special Procedure for CI MSD

CI calibration valve

After adding new PFDTD to the calibrant vial, you must purge the air out of the vial and valve.

3 **If the vacuum gauge controller is on, turn it off.**

4 **Turn on Gas A.**

5 **Turn on Purge.**

6 **Verify that PCICH4.U is loaded.**

7 **In Diagnostics and Vacuum Control view, select Purge Calibrant Valve under the Vacuum menu.**

This will open the CI calibration valve for several minutes with all analyzer voltages turned off.

To remove the EI calibration valve

Materials needed: Screwdriver, Torx T-15 (8710-1622)

1 **Vent the MSD. See page 64.**

2 **Disconnect the calibration valve cable from the connector next to the fan.**

3 **Loosen the collar and remove the calibration vial.**

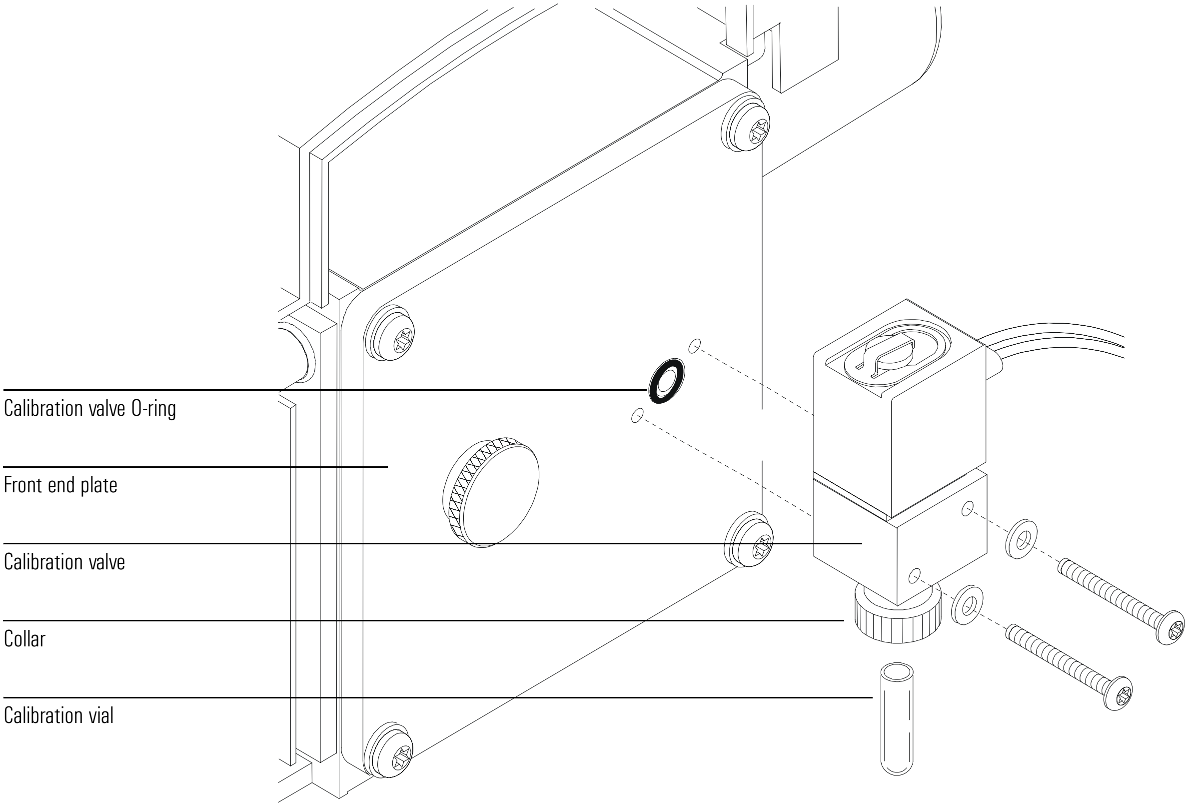
Turn the collar counterclockwise as viewed from the bottom (vial side) of the thumbscrew. Just loosen the collar, do not remove it.

CAUTION

Removing the valve with the vial installed can result in liquid calibrant getting into the restrictor of the valve. Liquid in the restrictor will prevent diffusion of PFTBA into the analyzer chamber for tuning. If this happens, the valve should be replaced.

4 **Remove the calibration valve from the front end plate.**

To remove the EI calibration valve



To reinstall the EI calibration valve

Materials needed:

Calibration valve
for standard turbo (G1099-60201)
for performance turbomolecular pump (G1099-60204)
O-ring, for calibration valve (0905-1217) – replace if the old O-ring is damaged
PFTBA (05971-60571) or other tuning compound
Screwdriver, Torx T-15 (8710-1622)
Calibration vial (05980-20018)

1 **Remove the old calibration. See page 187.**

2 **Make sure the calibration valve O-ring is in place.**

If the O-ring is worn or damaged, replace it.

3 **Install the calibration valve.**

Tighten the screws that hold it in place. Make sure you use the calibration valve that matches the high vacuum pump in your MSD. The different calibration valves have different restrictors. Using the wrong valve will interfere with tuning.

4 **Reconnect the calibration valve cable to the connector next to the fan.**

5 **Remove the vial from the new calibration valve. See page 182.**

The valve is supplied with a vial already installed.

6 **Fill and reinstall the calibration vial. See page 184.**

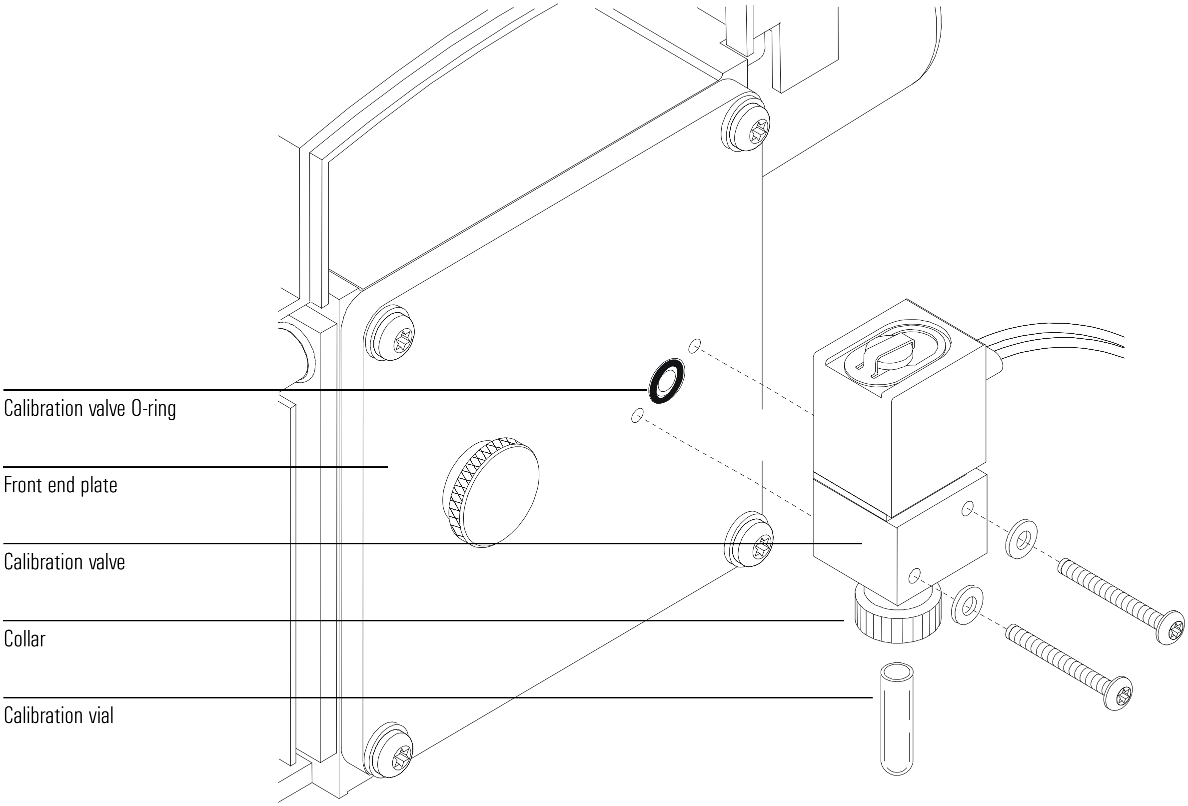
7 **Pump down the MSD. See page 70.**

8 **Select Purge Calibrant Valve from the Vacuum menu in the Tune and Vacuum Control view.**

CAUTION

Failure to purge the calibration valve will result in damage to the filaments and detector.

To reinstall the EI calibration valve



To replace the fan for the high vacuum pump

Materials needed: Fan (G1099-60564)
Screwdriver, Torx T-15 (8710-1622)

1 **Vent the MSD. See page 64.**

2 **Remove the upper and lower MSD covers. See page 62.**

3 **Disconnect the fan wiring from the connector on the MSD frame.**

4 **Remove the 4 fan screws and remove the fan.**

Keep the 4 screws.

WARNING **Do not touch the high vacuum pump. The high vacuum pumps could still be hot enough to burn you.**

5 **Disconnect the fan wiring and safety grill from the old fan.**

The fan wiring ends in a small connector on the back of the fan.

6 **Connect the fan wiring and safety grill to the new fan.**

7 **Install the new fan and reinstall the 4 screws.**

The flow arrow on the side of the fan points towards the pump.

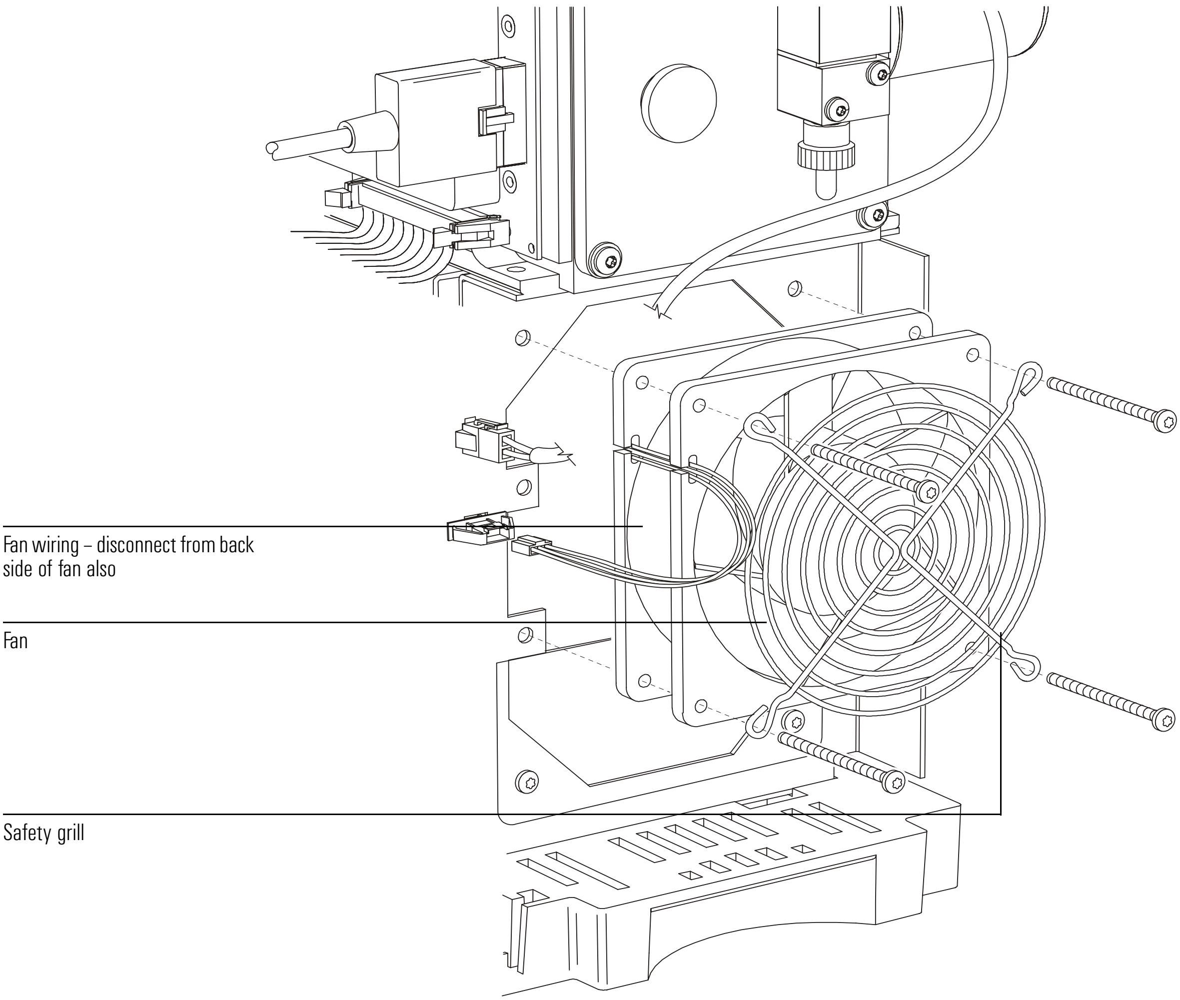
WARNING **Make sure the safety grill that shields the fan blades is in place.**

8 **Connect the fan wiring to the fan connector on the MSD frame.**

9 **Reinstall the MSD covers.**

10 **Pump down the MSD. See page 70.**

To replace the fan for the high vacuum pump



To remove the triode gauge tube

Materials needed:

Gloves, clean, lint-free
- large (8650-0030)
- small (8650-0029)

1 Vent the MSD. **See page 64.**

2 Disconnect the cable from the triode gauge tube.

WARNING

Never connect or disconnect the cable from the triode gauge tube while the MSD is under vacuum. The stress could cause the tube to implode.

3 Loosen the triode gauge collar by turning it counterclockwise.

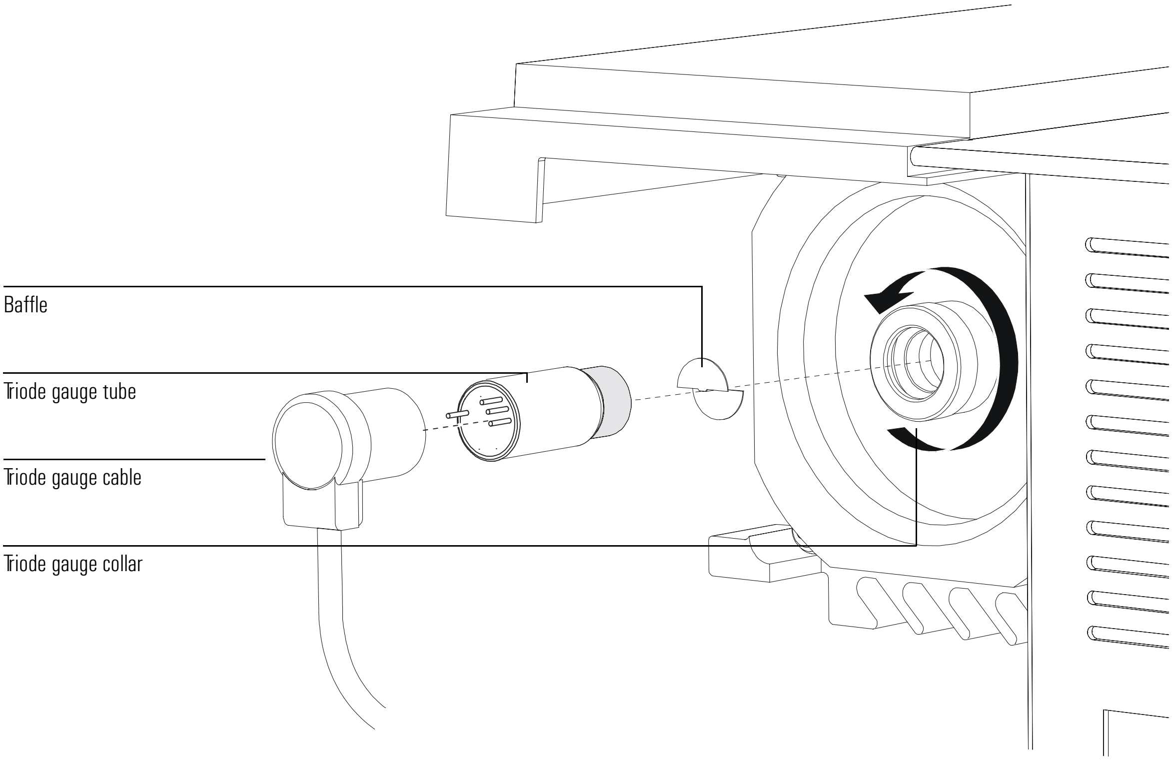
Do not remove the collar.

4 Pull the triode gauge tube out of the collar.

5 Remove the baffle from the open end of the triode gauge tube.

Wear clean gloves when handling the baffle. If you set the baffle down, make sure it is on a clean surface.

To remove the triode gauge tube



To reinstall a triode gauge tube

Materials needed:

Gloves, clean, lint-free
large (8650-0030)
small (8650-0029)
Triode gauge tube (0960-0897)

1 **Remove the old triode gauge tube. See page 193.**

2 **Slide the baffle into the open end of the new triode gauge tube.**

Wear clean gloves when handling the baffle and new triode gauge tube. If you set the baffle down, make sure it is on a clean surface.

3 **Slide the triode gauge tube into the collar.**

Leave 3 mm of the metal sleeve exposed. Be sure the pins are oriented as in the illustration.

4 **Gently hand tighten the collar by turning it clockwise.**

WARNING **Do not overtighten; you can break the tube or damage the O-ring.**

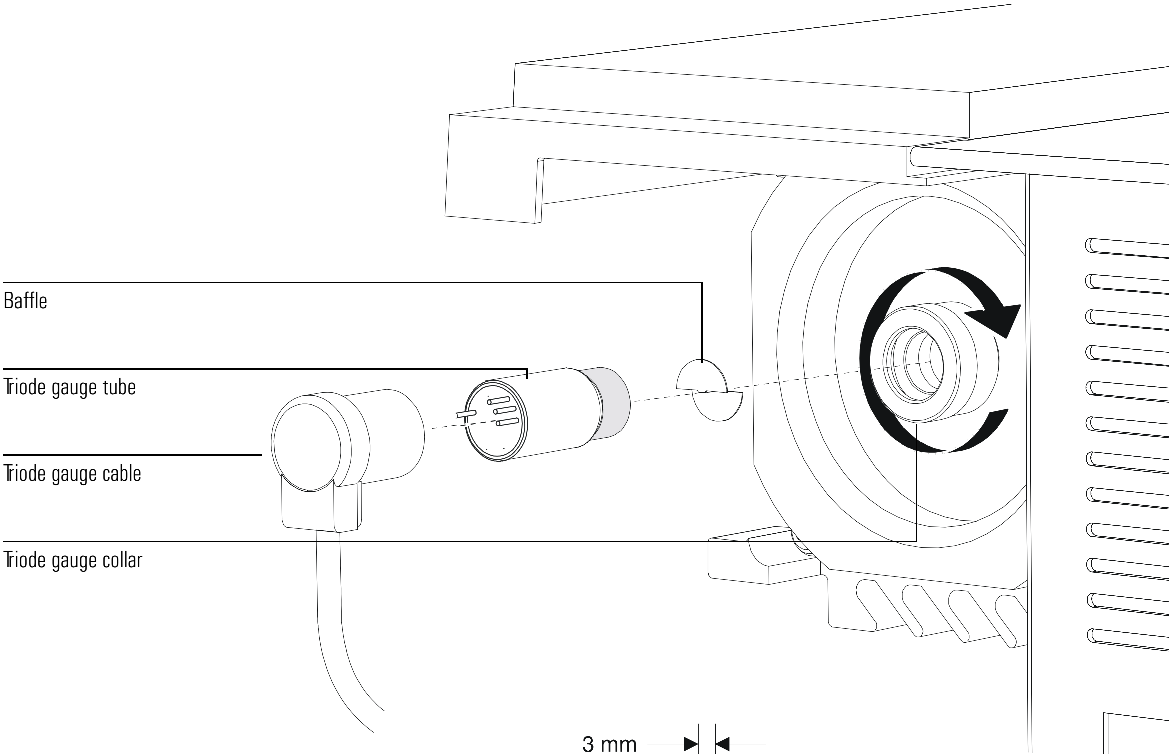
5 **Reconnect the cable from the gauge controller to the triode gauge tube.**

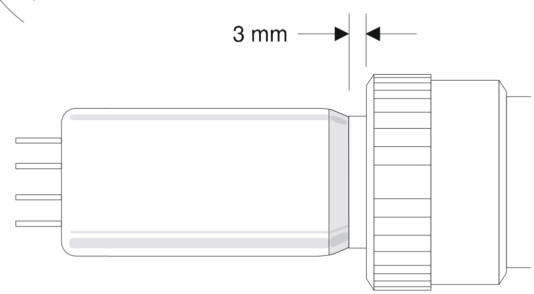
Route the cable so it does not put stress on the triode gauge tube.

CAUTION Be careful when attaching the cable. Too much force can break the tube. Do not move the controller or cable while connected to the tube.

6 **Pump down the MSD. See page 70.**

To reinstall a triode gauge tube





To lubricate the side plate O-ring

Materials needed:

Cloths, clean (05980-60051)
Gloves, clean, lint-free
  large (8650-0030)
  small (8650-0029)
Grease, Apiezon L, high vacuum (6040-0289)

The side plate O-ring needs a thin coat of grease to ensure a good vacuum seal. If the side plate O-ring appears dry, or does not seal correctly, lubricate it using this procedure. A good test is to wipe off the side plate with methanol, then close the analyzer chamber. If the O-ring has enough grease on it, it will leave a faint trace on the side plate.

CAUTION

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.

1 **Vent the MSD. See page 64.**

2 **Open the analyzer chamber. See page 66.**

3 **Use a clean, lint-free cloth or glove to spread a *thin* coat of high vacuum grease only on the exposed surface of the O-ring.**

CAUTION

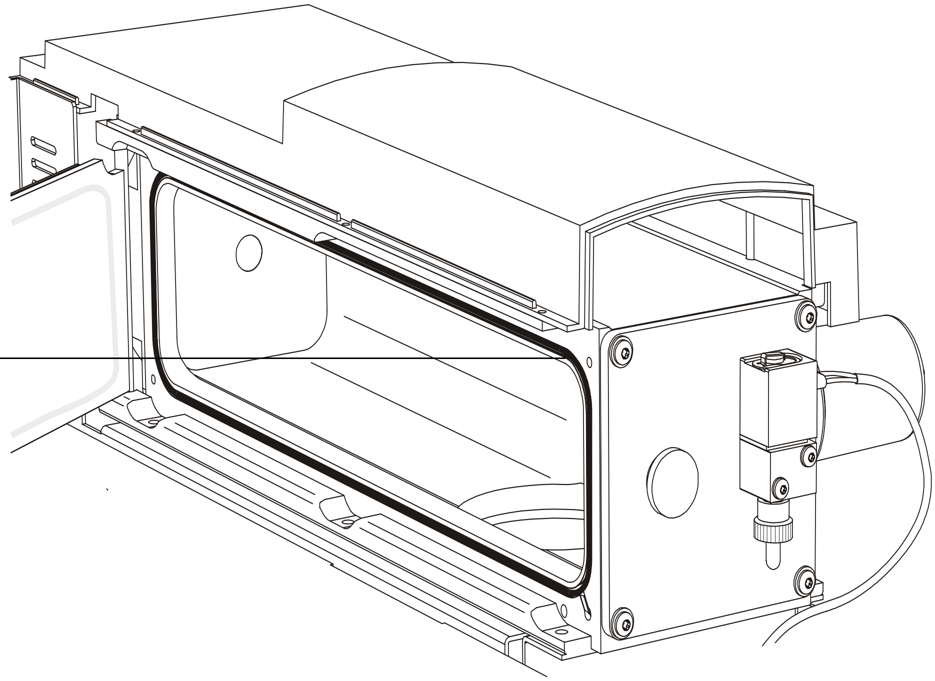
Do not use anything except the recommended vacuum grease. Excess grease can trap air and dirt. Grease on surfaces of the O-ring other than the exposed surface can trap air, resulting in air spikes during operation.

4 **Use a clean, lint-free cloth or glove to wipe away excess grease.**

If the O-ring looks shiny, there is too much grease on it.

5 **Close the analyzer chamber. See page 68.**

6 **Pump down the MSD. See page 70.**



See also See video demonstration of this procedure.

To lubricate the vent valve O-ring

Materials needed:

Cloths, clean (05980-60051)
Gloves, clean, lint-free
  large (8650-0030)
  small (8650-0029)
Grease, Apiezon L, high vacuum (6040-0289)
O-ring, vent valve (0905-1217) - replace if the old O-ring is worn or damaged

The vent valve O-ring needs a thin coat of lubrication to ensure a good vacuum seal and smooth operation. If the vent valve O-ring does not turn smoothly, or does not seal correctly, lubricate it using this procedure.

CAUTION

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.

1 **Vent the MSD. See page 64.**

2 **Completely remove the vent valve knob.**

3 **Inspect the O-ring.**

If the O-ring appears damaged, replace it.

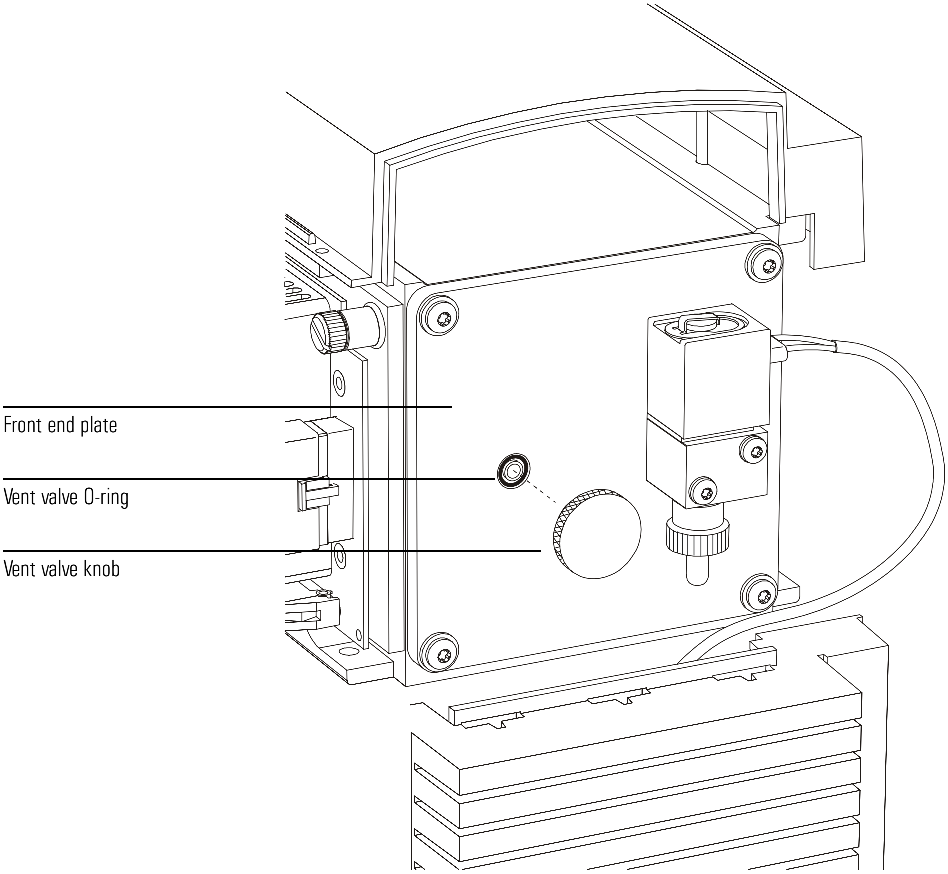
4 **Use a clean, lint-free cloth or glove to spread a *thin* coat of high vacuum grease on the exposed surface of the O-ring.**

CAUTION

Excess grease can trap air and dirt. Grease on surfaces of the O-ring other than the exposed surface can trap air, resulting in air spikes during operation.

5 **Use a clean, lint-free cloth or glove to wipe away excess grease.**

If the O-ring looks shiny, there is too much grease on it.



6 Reinstall the vent valve knob.

CAUTION

Be very careful when reinstalling the vent valve knob. It is very easy to cross thread the knob and damage the threads in the front end plate. Be sure the O-ring stays in place.

7 Pump down the MSD. See page 70.

Maintaining the analyzer

The analyzer requires no periodic maintenance

None of the analyzer components requires periodic maintenance. Some tasks, however, must be performed when MSD behavior indicates they are necessary. These tasks include:

- Cleaning the ion source
- Replacing filaments
- Replacing the electron multiplier horn

Troubleshooting the MSD, on page 103 provides information about symptoms that indicate the need for analyzer maintenance. The troubleshooting material in the online help in the MSD ChemStation software provides more extensive information.

Care must be taken during analyzer maintenance to keep components clean

Analyzer maintenance involves opening the analyzer chamber and removing parts from the analyzer. During analyzer maintenance procedures, care must be take to avoid contaminating the analyzer or interior of the analyzer chamber. Clean gloves should be worn 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.

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 168 for more information.

CAUTION

Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded anti-static wrist strap (see page 168) and take other anti-static 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 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 Chapter 10, *Analyzer*, on page 287.

Most of the procedures in this chapter are illustrated with video clips.

To remove the ion source

Materials needed:

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

1 **Vent the MSD. See page 64.**

2 **Open the analyzer chamber. See page 66.**

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

3 **Disconnect the seven wires from the ion source.**

Do not bend the wires any more than necessary.

CAUTION

Pull on the connectors, not on the wires.

4 **Disconnect the wires for the ion source heater and temperature sensor from the feedthrough board.**

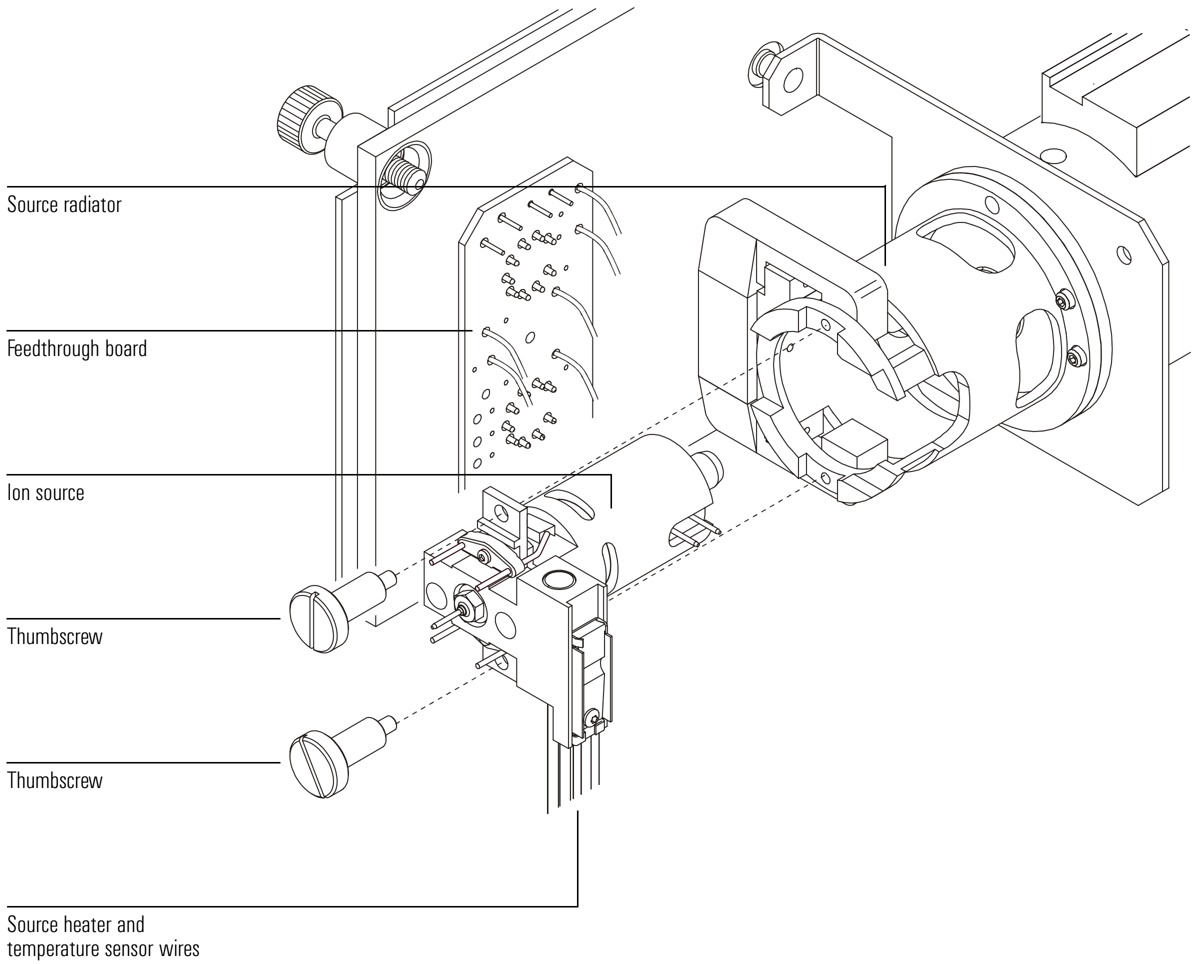
5 **Remove the thumbscrews that hold the ion source in place.**

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.

To remove the ion source



To disassemble the ion source

Materials needed:

Gloves, clean, lint-free
  large (8650-0030)
  small (8650-0029)
Hex ball driver, 1.5-mm (8710-1570)
Hex ball driver, 2.0-mm (8710-1804)
Hex nut driver, 5.5-mm (8710-1220)
Wrench, open-end, 10-mm (8710-2353)

1 **Remove the ion source. See page 203.**

2 **Remove the filaments.**

3 **Separate the repeller assembly from the source body.**

The repeller assembly includes the source heater assembly, repeller, and related parts. The source washer is between the repeller block and the ion source body; after prolonged periods of time, the source washer could bond to the repeller block. This is normal.

4 **Remove the repeller.**

5 **Unscrew the interface socket.**

A 10-mm open-end wrench fits on the flats on the interface socket.

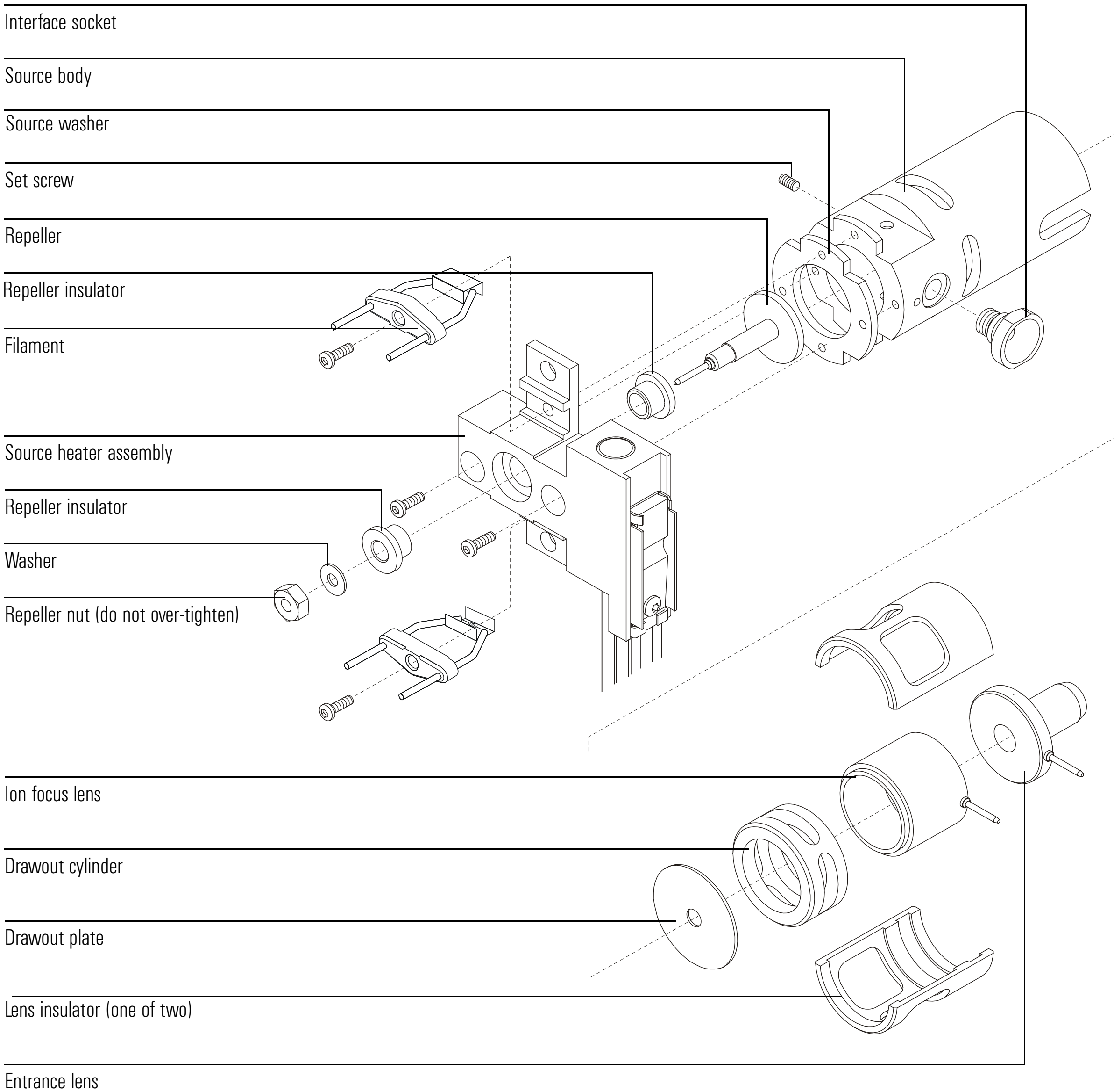
6 **Remove the setscrew for the lenses.**

7 **Push the lenses out of the source body.**

NOTE

Video will show the standard Ion Source Assembly procedures. It will not show the source washer. The source washer is used only in the inert ion source assembly.

To disassemble the ion source



To clean the ion source

Materials needed:

Abrasive paper (5061-5896)
Alumina abrasive powder (8660-0791)
Aluminum foil, clean
Cloths, clean (05980-60051)
Cotton swabs (5080-5400)
Glass beakers, 500 ml
Gloves, clean, lint-free
  large (8650-0030)
  small (8650-0029)
Solvents
  acetone, reagent-grade
  methanol, reagent-grade
  methylene chloride, reagent grade
Ultrasonic bath

1 Disassemble the ion source. See page 205.

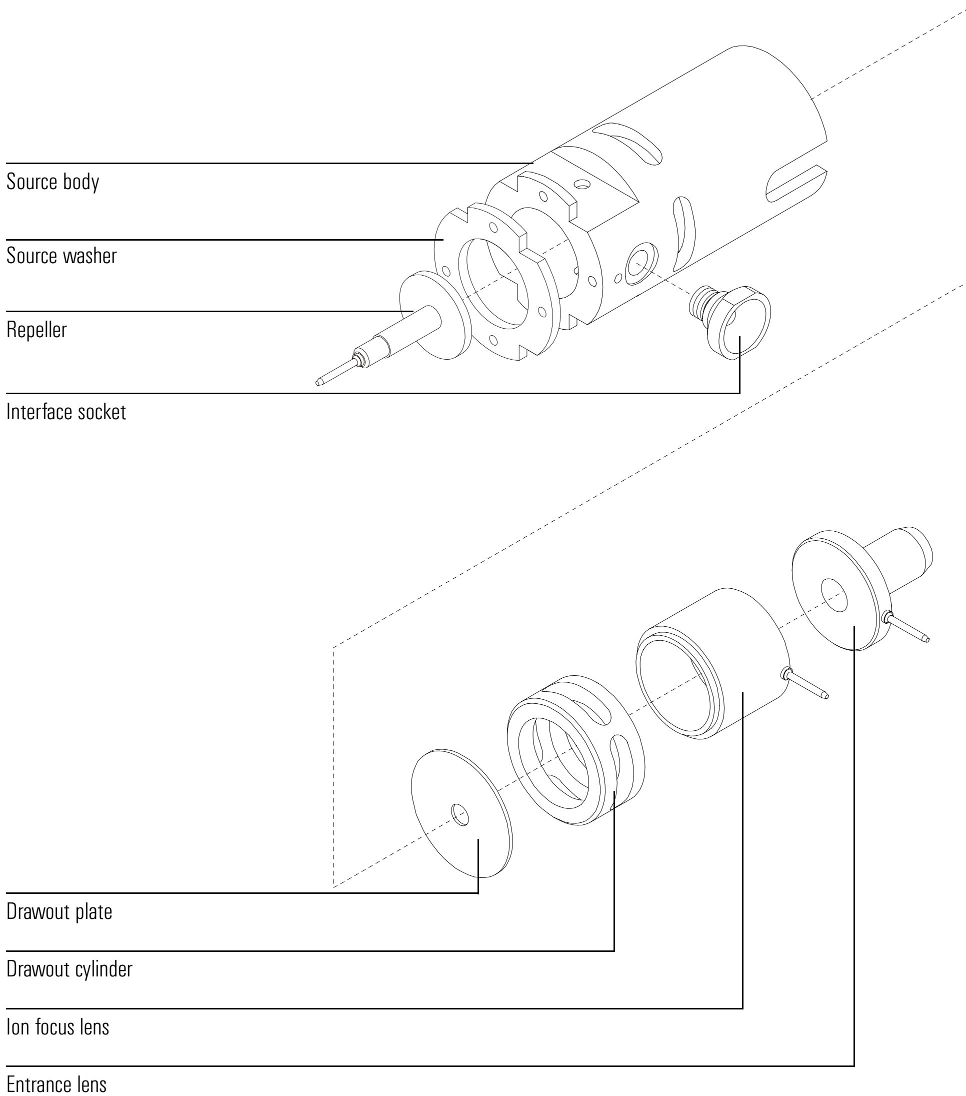
2 Collect the following parts to be cleaned:

- Repeller
- Interface socket
- Source body
- Drawout plate
- Drawout cylinder
- Ion focus lens
- Entrance lens

These are the parts that contact the sample or ion beam. The other parts normally should not require cleaning.

CAUTION

If insulators are dirty, clean them with a cotton swab dampened with reagent-grade methanol. If that does not clean the insulators, replace them. Do not abrasively or ultrasonically clean the insulators.


Source body
Source washer
Repeller
Interface socket
Drawout plate
Drawout cylinder
Ion focus lens
Entrance lens

Major contamination

In the event of a major contamination, the other source components must be cleaned (ultrasonically but not abrasively) or replaced.

CAUTION

The filaments, source heater assembly, and insulators cannot be cleaned ultrasonically. Replace these components if major contamination occurs.

3 **Abrasively clean the surfaces that contact the sample or ion beam.**

Use an abrasive slurry of alumina powder and reagent-grade methanol on a cotton swab. Use enough force to remove all discolorations. Polishing the parts is not necessary; small scratches will not harm performance. Also abrasively clean the discolorations where electrons from the filaments enter the source body.

4 **Rinse away all abrasive residue with reagent-grade methanol.**

Make sure ***all*** abrasive residue is rinsed way ***before*** ultrasonic cleaning. If the methanol becomes cloudy or contains visible particles, rinse again.

5 **Separate the parts that were abrasively cleaned from the parts that were not abrasively cleaned.**

6 **Ultrasonically clean the parts for 15 minutes in each of the following solvents:**

Ultrasonically clean each group of parts separately.

- Methylene chloride (reagent-grade)
- Acetone (reagent-grade)
- Methanol (reagent-grade)

WARNING

All of these solvents are hazardous. Work in a fume hood and take all appropriate precautions.

7 **Place the parts in a clean beaker. *Loosely* cover the beaker with clean aluminum foil (dull side down).**

8 **Dry the cleaned parts in an oven at 100 – 150 °C for 30 minutes.**

WARNING

Let these parts cool before you handle them.

NOTE

Take care to avoid recontaminating cleaned and dried parts. Put on new, clean gloves before handling the parts. Do not set the cleaned parts on a dirty surface. Set them only on clean, lint-free cloths.

To reassemble the ion source

Materials needed:

Gloves, clean, lint-free
  large (8650-0030)
  small (8650-0029)
Hex ball driver, 1.5-mm (8710-1570)
Hex ball driver, 2.0-mm (8710-1804)
Hex nut driver, 5.5-mm (8710-1220)
Wrench, open-end, 10-mm (8710-2353)

1 **Slide the drawout plate and the drawout cylinder into the source body.**

2 **Assemble the ion focus lens, entrance lens, and lens insulators.**

3 **Slide the assembled parts into the source body.**

4 **Install the setscrew that holds the lenses in place.**

5 **Reinstall the repeller, repeller insulators, washer, and repeller nut into the source heater assembly.**

The resulting assembly is called the repeller assembly.

CAUTION

Do not overtighten the repeller nut, or the ceramic repeller insulators will break when the source heats up. The nut should only be “finger-tight”.

6 **Place the source washer onto the ion source body. Parts are key; there is only one orientation that will fit onto the ion source body.**

7 **Reconnect the repeller assembly to the source body.**

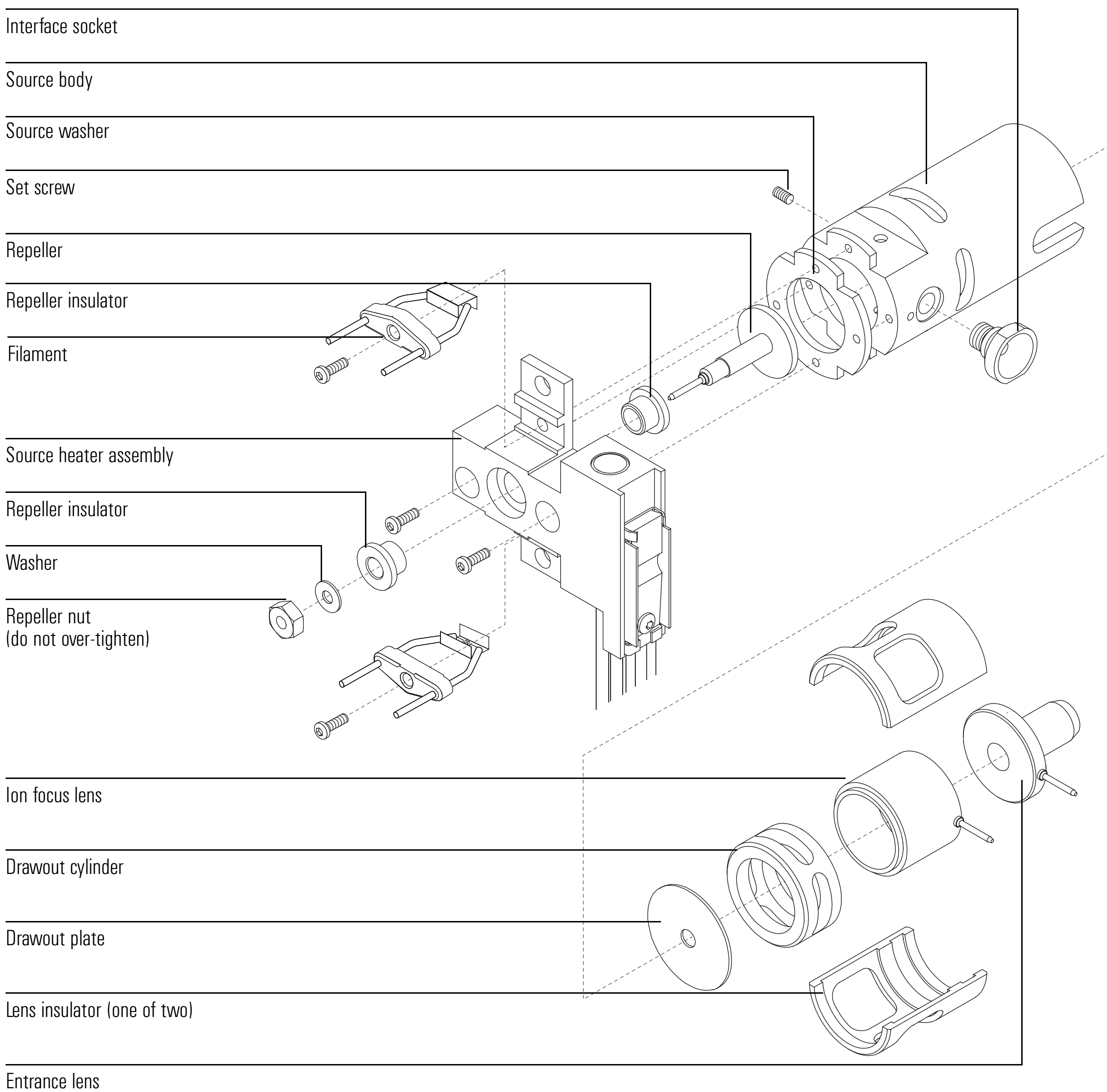
The repeller assembly includes the source heater assembly, repeller, and related parts.

8 **Reinstall the filaments.**

9 **Reinstall the interface socket.**

CAUTION

Do not overtighten the interface socket. Overtightening could strip the threads.


Interface socket
Source body
Source washer
Set screw
Repeller
Repeller insulator
Filament
Source heater assembly
Repeller insulator
Washer
Repeller nut
(do not over-tighten)
Ion focus lens
Drawout cylinder
Drawout plate
Lens insulator (one of two)
Entrance lens

To reinstall the ion source

Materials needed:

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

1 Slide the ion source into the source radiator.

2 Install and hand tighten the source thumbscrews.

Do not overtighten the thumbscrews.

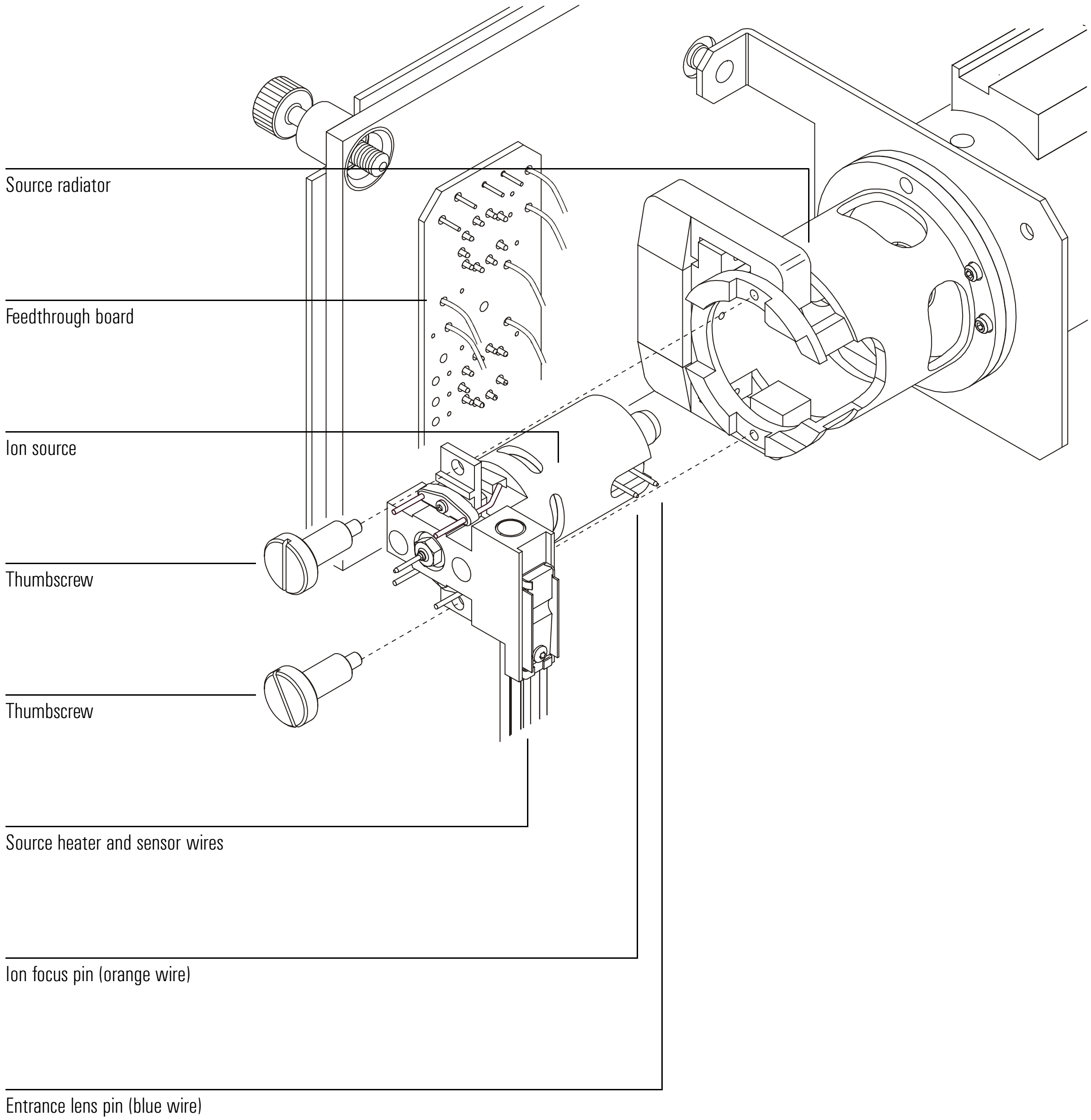
3 Reconnect the 7 wires to the appropriate pins on the ion source.

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

4 Reconnect the source heater and temperature sensor wires to the pins on the feedthrough board.

5 Close the analyzer chamber. See page 68.

6 Pump down the MSD. See page 70.


Source radiator
Feedthrough board
Ion source
Thumbscrew
Thumbscrew
Source heater and sensor wires
Ion focus pin (orange wire)
Entrance lens pin (blue wire)

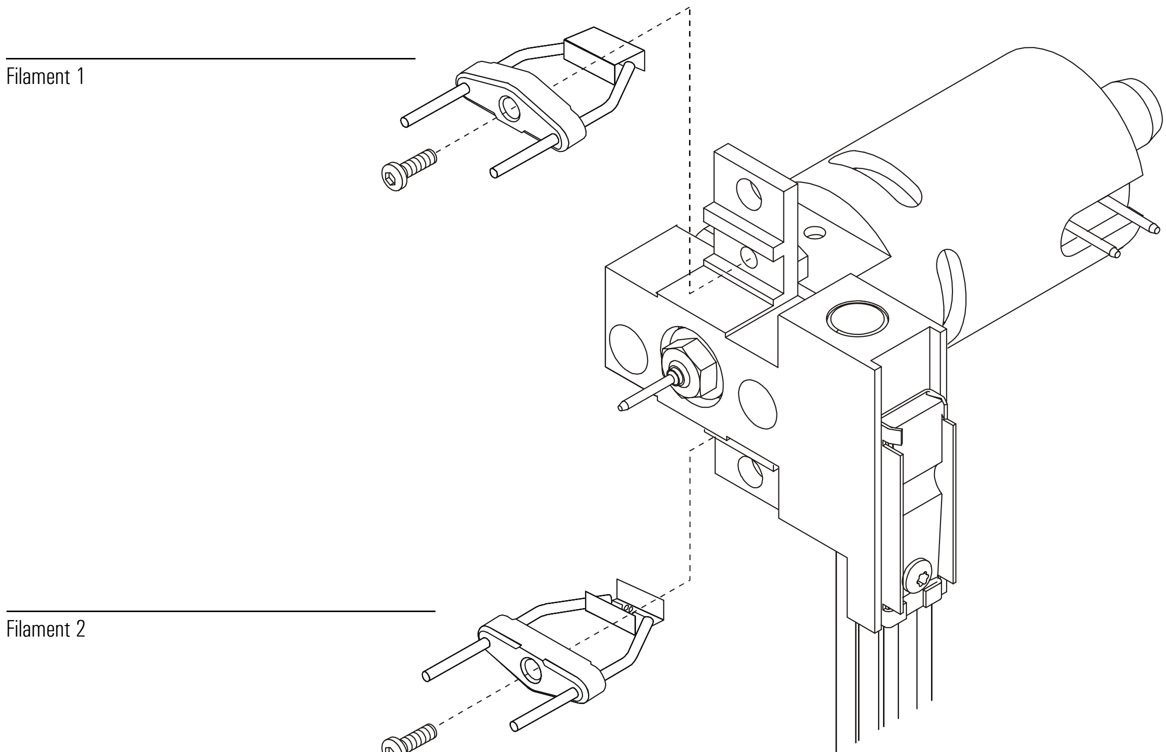
To remove a filament

Materials needed:

Gloves, clean, lint-free
  large (8650-0030)
  small (8650-0029)
Hex ball driver, 1.5-mm (8710-1570)

1. **Vent the MSD. See page 64.**
2. **Open the analyzer chamber. See page 66.**
3. **Remove the ion source. See page 203.**
4. **Remove the filament(s) to be replaced.**

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


Filament 1
Filament 2

To reinstall a filament

Materials needed:

Filament assembly (G1099-60053)
Gloves, clean, lint-free
  large (8650-0030)
  small (8650-0029)
Hex ball driver, 1.5-mm (8710-1570)

1 **Install the new filament.**

2 **Reinstall the ion source. See page 214.**

3 **Close the analyzer chamber. See page 68.**

4 **Pump down the MSD. See page 70.**

5 **Autotune the MSD. See page 60.**

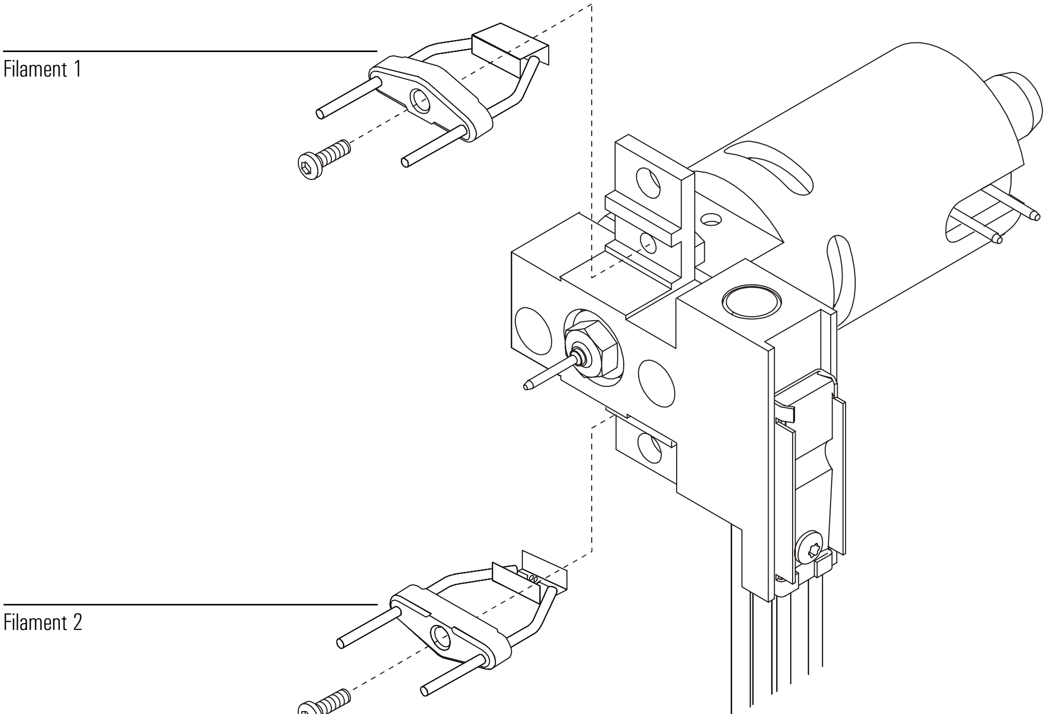
6 **In the Edit Parameters dialog box (Instrument/Edit MS Tune Parameters), select the other filament.**

7 **Autotune the MSD again.**

8 **Select and use the filament that give the best results.**

   If you decide to use the first filament, run Autotune again to make sure the tune parameters are compatible with the filament.

9 **Select Save Tune Parameters from the File menu.**


Filament 1
Filament 2

To remove the heater and sensor from the ion source

Materials needed:

Gloves, clean, lint-free
  large (8650-0030)
  small (8650-0029)
Hex ball driver, 1.5-mm (8710-1570)
Hex ball driver, 2.0-mm (8710-1804)
Hex nut driver, 5.5-mm (8710-1220)

1 **Vent the MSD. See page 64.**

2 **Open the analyzer chamber. See page 66.**

3 **Remove the ion source from the source radiator. See page 203.**

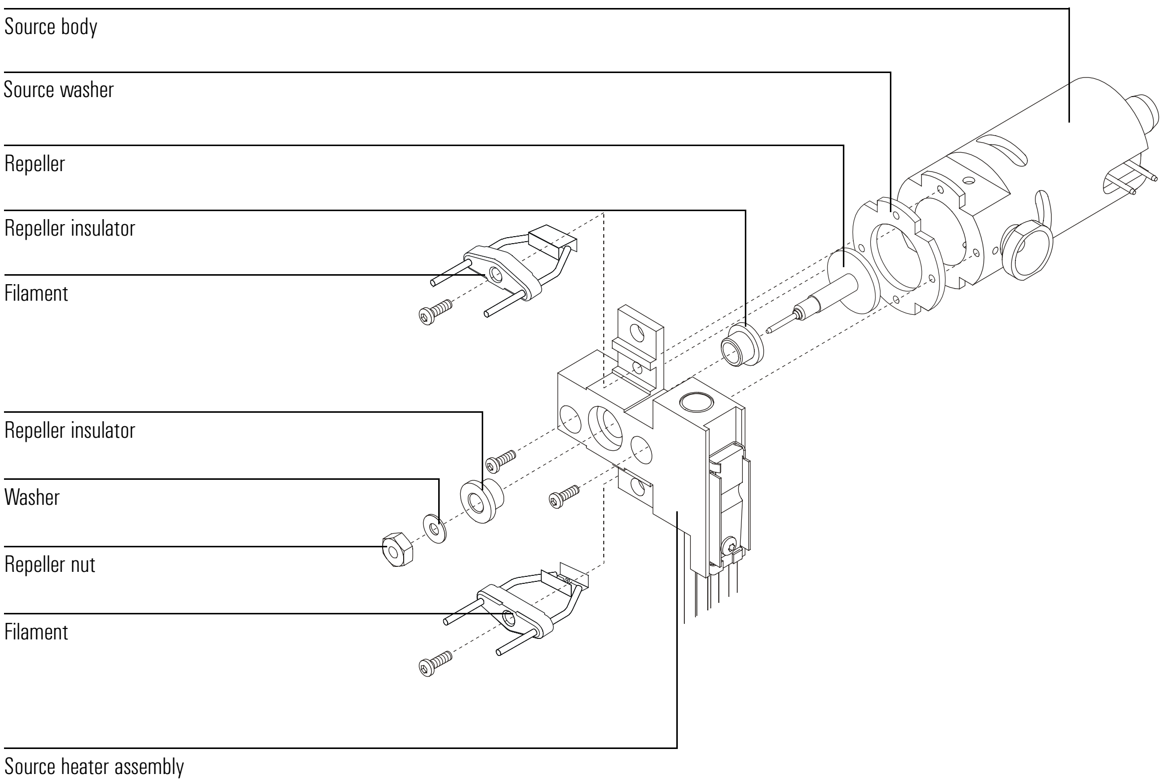
4 **Remove the filaments.**

5 **Remove the repeller assembly.**

The repeller assembly includes the source heater assembly, repeller, and related parts.

6 **Remove the repeller nut, washer, repeller insulators, and repeller.**

You do not need to remove the heater and temperature sensor from the heater block. The new source heater assembly includes all three parts already assembled.


Source body
Source washer
Repeller
Repeller insulator
Filament
Repeller insulator
Washer
Repeller nut
Filament
Source heater assembly

To reinstall the heater and sensor in the ion source

Materials needed:

Gloves, clean, lint-free
large (8650-0030)
small (8650-0029)
Hex ball driver, 1.5-mm (8710-1570)
Hex ball driver, 2.0-mm (8710-1804)
Hex nut driver, 5.5-mm (8710-1220)
Source heater assembly (G2589-60177)

1 **Unpack the new source heater assembly.**

The heater, temperature sensor, and heater block are already assembled.

2 **Reinstall the repeller, repeller insulators, washer, and repeller nut.**

The resulting assembly is called the repeller assembly.

CAUTION Do not overtighten the repeller nut, or the ceramic repeller insulators will break when the source heats up. The nut should only be “finger-tight”.

3 **Connect the repeller assembly to the source body.**

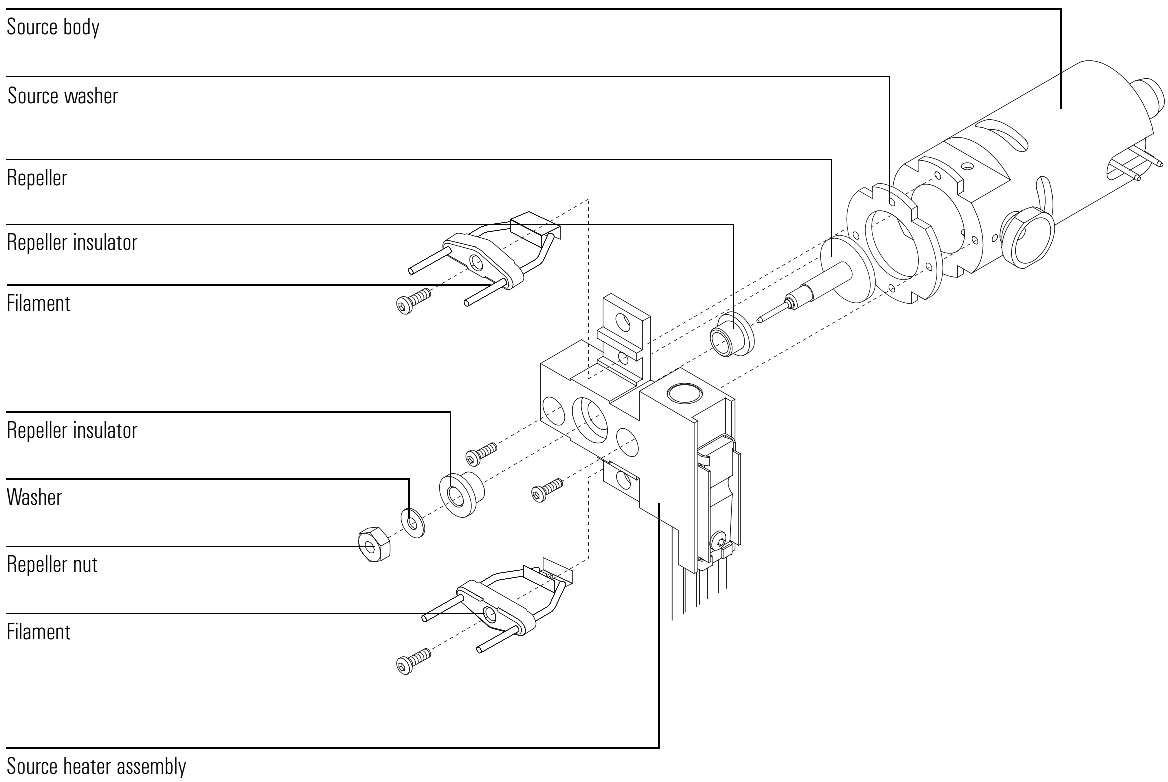
4 **Reinstall the filaments.**

5 **Reinstall the ion source in the source radiator. See page 214.**

Do not forget to reconnect the wires from the feedthrough board to the ion source. Do not forget to reconnect the heater and temperature sensor wires to the feedthrough board.

6 **Close the analyzer chamber. See page 68.**

7 **Pump down the MSD. See page 70.**


Source body
Source washer
Repeller
Repeller insulator
Filament
Repeller insulator
Washer
Repeller nut
Filament
Source heater assembly

To remove the heater and sensor from the mass filter

Materials needed:

Gloves, clean, lint-free
    large (8650-0030)
    small (8650-0029)
Hex ball driver, 1.5-mm (8710-1570)
Hex ball driver, 2.0-mm (8710-1804)

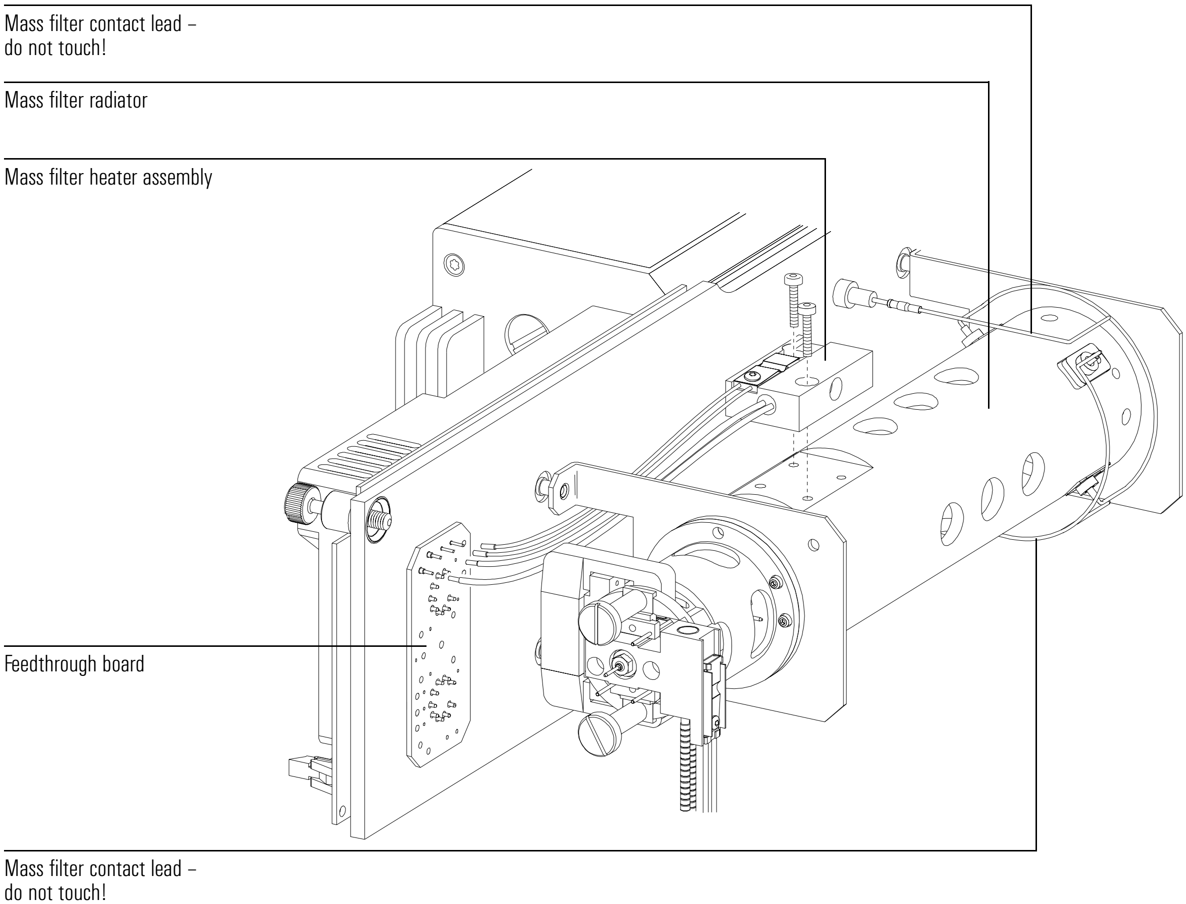
1 **Vent the MSD. See page 64.**

2 **Open the analyzer chamber. See page 66.**

3 **Disconnect the mass filter heater and temperature sensor wires from the feedthrough board.**

4 **Remove the mass filter heater assembly from the mass filter radiator.**

CAUTION Do not touch the mass filter contact leads. This could cause ESD damage to the side board.


Mass filter contact lead – do not touch!
Mass filter radiator
Mass filter heater assembly
Feedthrough board
Mass filter contact lead – do not touch!

To reinstall the heater and sensor in the mass filter

Materials needed:

Gloves, clean, lint-free
- large (8650-0030)
- small (8650-0029)

Hex ball driver, 1.5-mm (8710-1570)
Hex ball driver, 2.0-mm (8710-1804)
Mass filter heater assembly (G1099-60172)

1 **Unpack the new mass filter heater assembly.**

The heater, temperature sensor, and heater block are already assembled.

2 **Install the heater assembly on top of the mass filter radiator.**

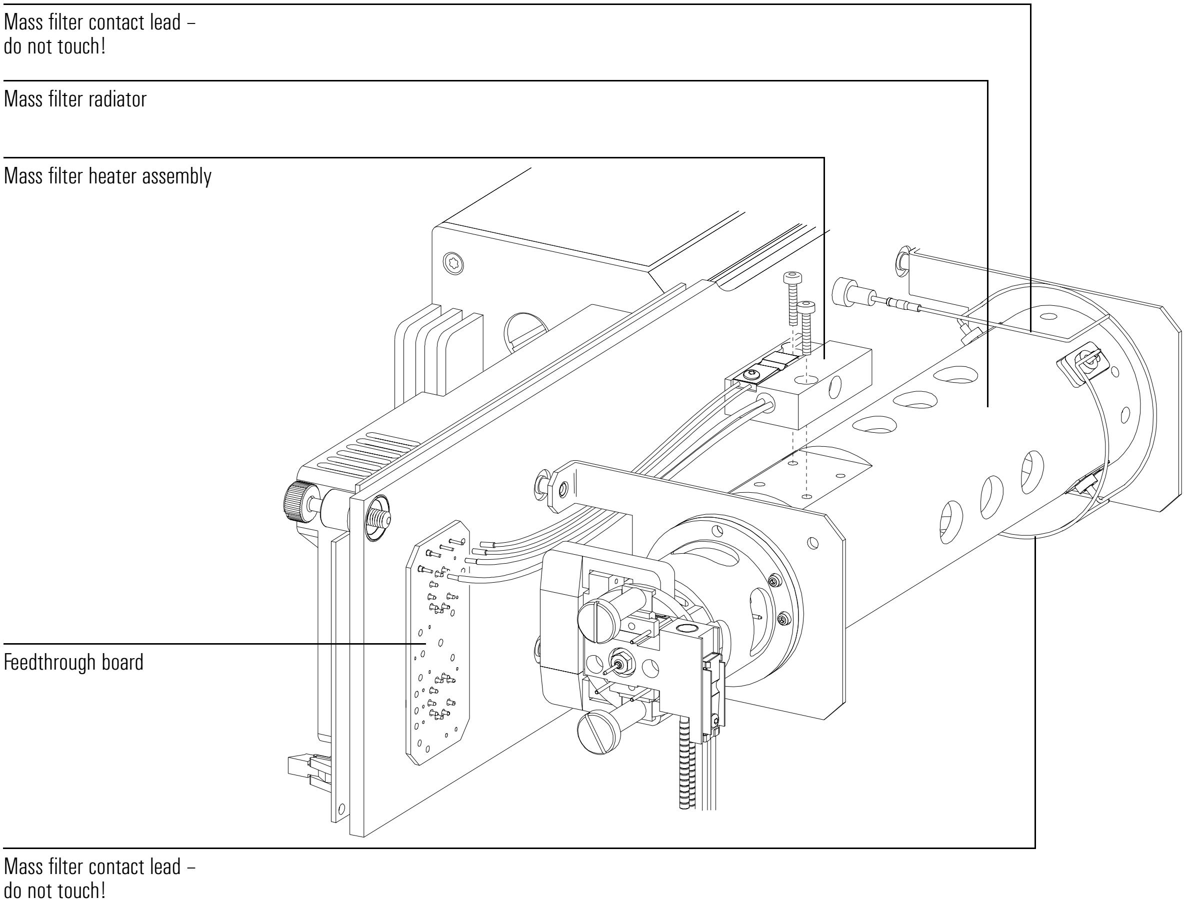
3 **Connect the heater and temperature sensor wires to the feedthrough board.**

4 **Close the analyzer chamber. See page 68.**

5 **Pump down the MSD. See page 70.**

CAUTION

Do not touch the mass filter contact leads. This could cause ESD damage to the side board.


Mass filter contact lead – do not touch!
Mass filter radiator
Mass filter heater assembly
Feedthrough board
Mass filter contact lead – do not touch!

To replace the electron multiplier horn

Materials needed:

Electron multiplier horn (05971-80103)
Gloves, clean, lint-free
large (8650-0030)
small (8650-0029)

1 **Vent the MSD. See page 64.**

2 **Open the analyzer chamber. See page 66.**

3 **Open the retaining clip.**

Pinch the two arms of the clip together and swing the clip down.

4 **Remove the electron multiplier horn.**

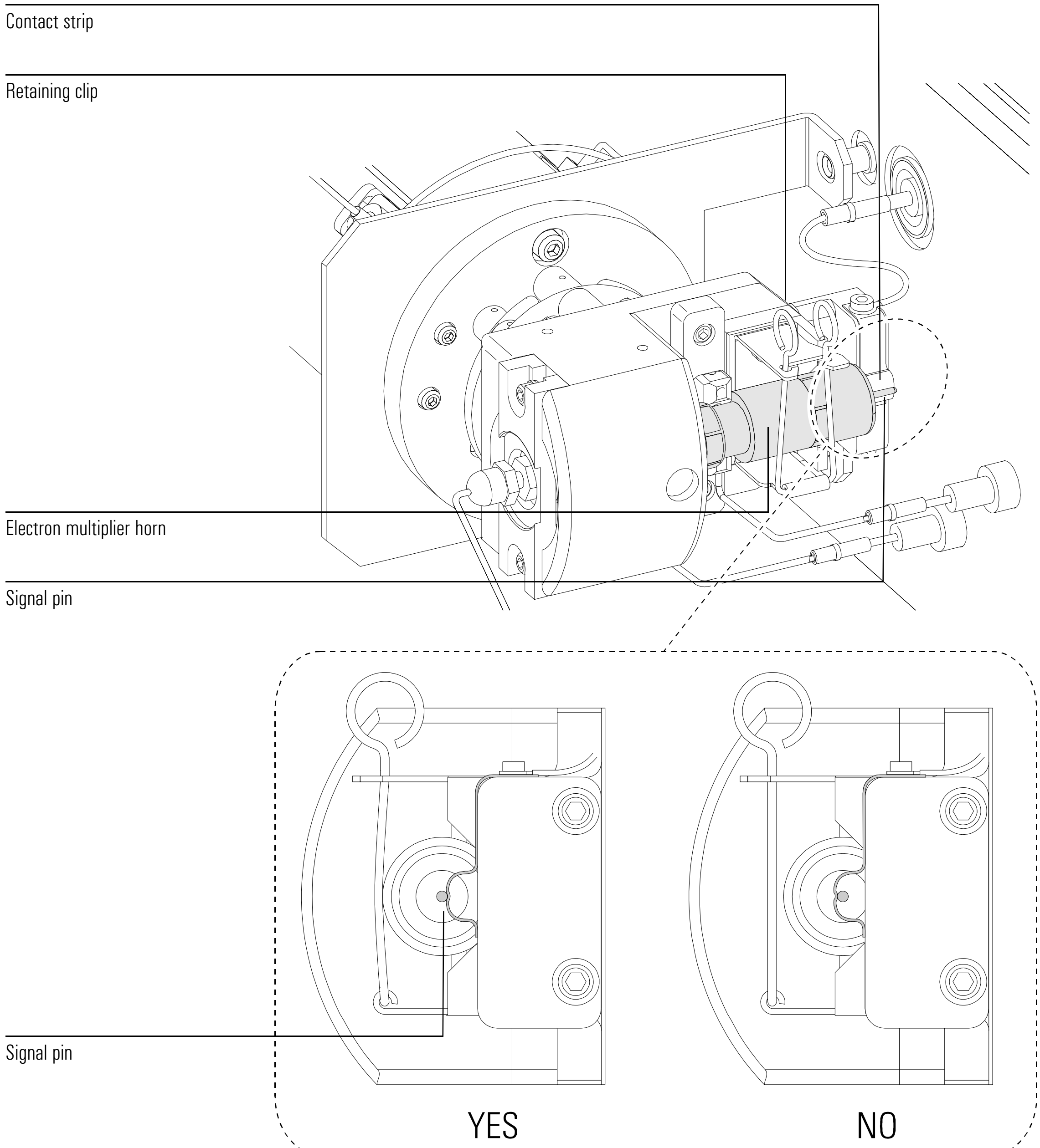
5 **Install the new electron multiplier horn.**

6 **Close the retaining clip.**

The signal pin on the horn should rest ***on the outside*** of the loop in the contact strip. ***Do not*** put the signal pin on the inside of the loop in the contact strip. Incorrect installation will result in poor sensitivity or no signal.

7 **Close the analyzer chamber. See page 68.**

8 **Pump down the MSD. See page 70.**


Contact strip
Retaining clip
Electron multiplier horn
Signal pin
Signal pin
YES
NO

Maintaining the GC/MSD interface

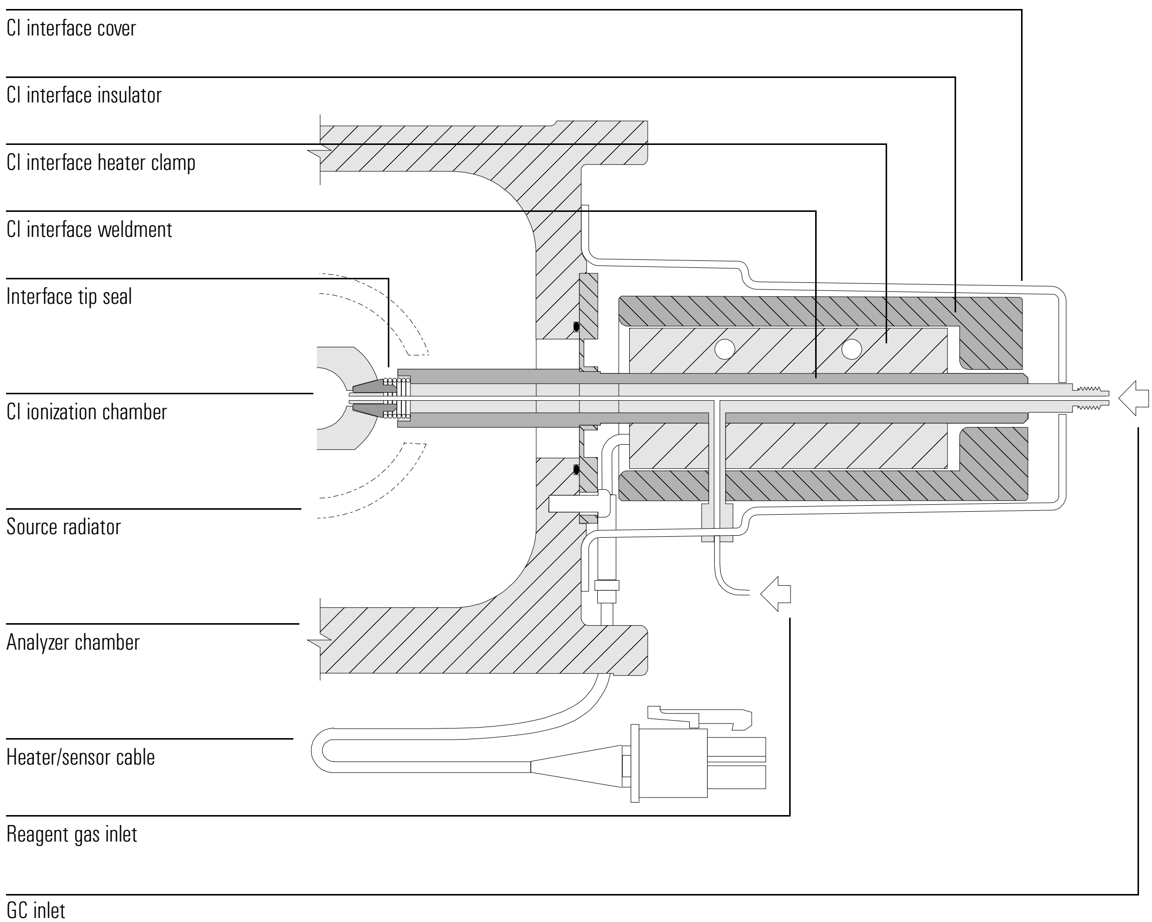
The GC/MSD interface requires no periodic maintenance

Rarely, the heater cartridge in the GC/MSD interface fails. In those cases, it is necessary to replace the heater and sensor. This section contains procedures for removing the heater and sensor and installing new ones.

More information is available

If you need more information about the locations or functions of GC/MSD interface components, refer to *GC/MSD Interface*, on page 281.

Most of the procedures in this chapter are illustrated with video clips.


CI interface cover
CI interface insulator
CI interface heater clamp
CI interface weldment
Interface tip seal
CI ionization chamber
Source radiator
Analyzer chamber
Heater/sensor cable
Reagent gas inlet
GC inlet

To remove the GC/MSD interface heater and sensor

Materials needed: Screwdriver, Torx T-15 (8710-1622)
Hex driver, 1.5 mm (8710-1570)

1 **Vent the MSD. See page 64.**

Make sure you turn off the GC/MSD interface heater. This heater is controlled and powered by the GC.

2 **Separate the MSD from the GC. See page 178.**

3 **Remove the cover from the GC/MSD interface.**

WARNING **The GC/MSD interface operates a very high temperatures. It is also well insulated. Make sure the interface is cool *before* you touch it.**

4 **Slide the insulation off of the GC/MSD interface.**

5 **Loosen the two heater sleeve screws.**

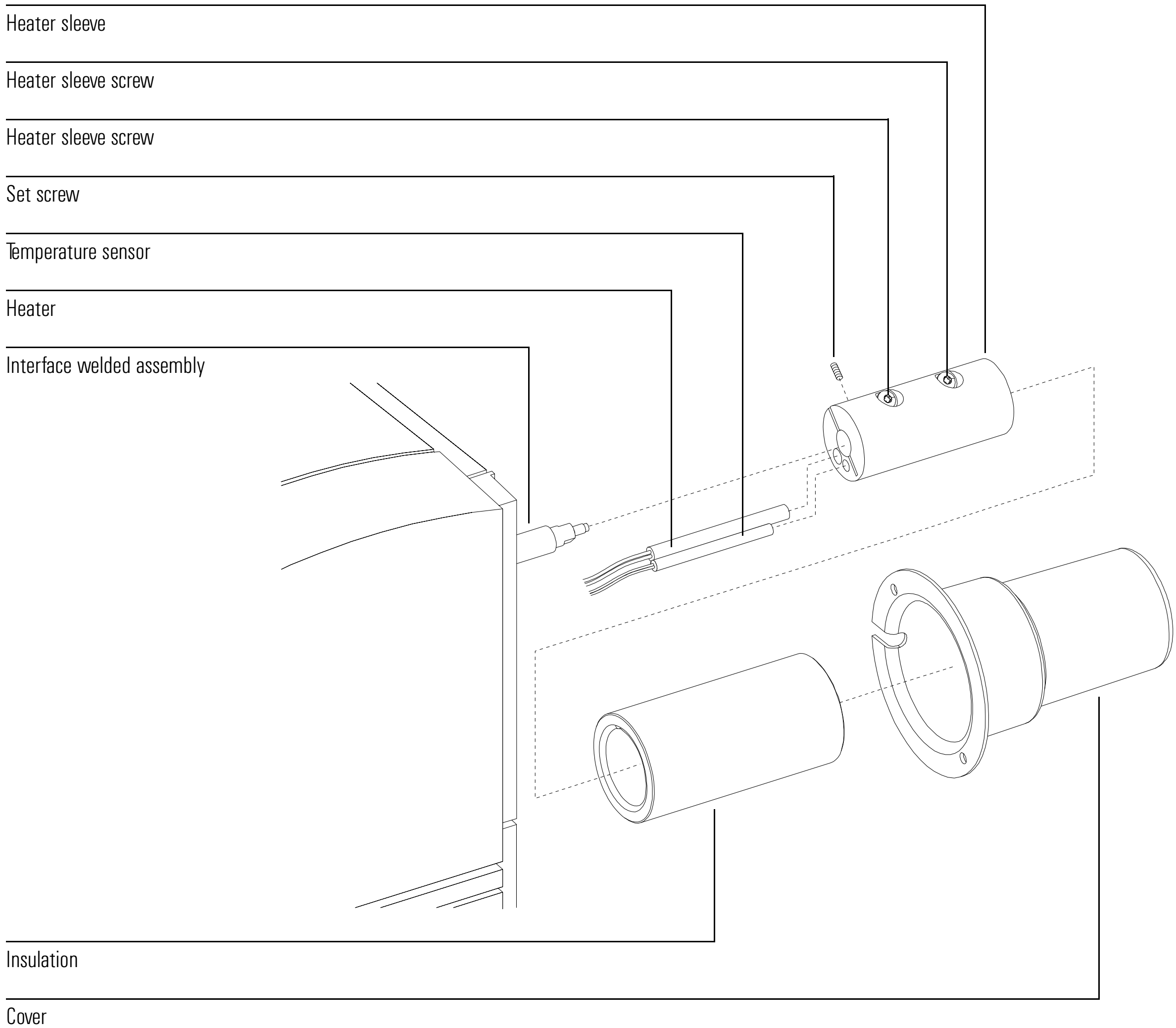
6 **Slide the heater sleeve off of the GC/MSD interface.**

It may be necessary to ***gently*** pry open the slot in the heater sleeve to loosen the heater sleeve from the interface.

7 **Loosen the setscrew and remove the heater and temperature sensor from the heater sleeve.**

Heat and oxidation often result in a heater, or less frequently a temperature sensor, being "welded" inside the heater sleeve. The holes for the heater and sensor pass all the way through the heater sleeve. A rod can be inserted to drive the stuck part out. However, to function correctly the heater and sensor must have perfect contact with their holes. If a heater or sensor is difficult to remove, the holes will probably be damaged enough that the heater sleeve should be replaced. Polishing the holes is not an acceptable solution since it will enlarge the holes.

CAUTION Installing a new heater and sensor in a damaged heater sleeve will result in poor performance of the heated zone and could reduce the lifetime of the new parts.


Heater sleeve
Heater sleeve screw
Heater sleeve screw
Set screw
Temperature sensor
Heater
Interface welded assembly
Insulation
Cover

To reinstall the GC/MSD interface heater and sensor

Materials needed:

GC/MSD interface heater assembly (G1099-60107)
Heater sleeve (G1099-20210) – replace the old sleeve if it is damaged
Screwdriver, Torx T-15 (8710-1622)
Hex driver, 1.5 mm (8710-1570)

1 **Slide the new heater and temperature sensor into the heater sleeve.**

2 **Reinstall the setscrew.**

3 **Slide the heater sleeve onto the GC/MSD interface.**

Align the heater sleeve so the screws are on top. Tighten the screws evenly.

4 **Slide the insulation onto the GC/MSD interface.**

CAUTION

There is a shallow groove along the inner surface of the insulation. This groove ***must*** line up with the heads of the screws in the heater sleeve. If it does not, you can crack or otherwise damage the insulation.

5 **Reinstall the GC/MSD interface cover.**

Make sure the wires from the heater and sensor pass through the cutout in the interface cover.

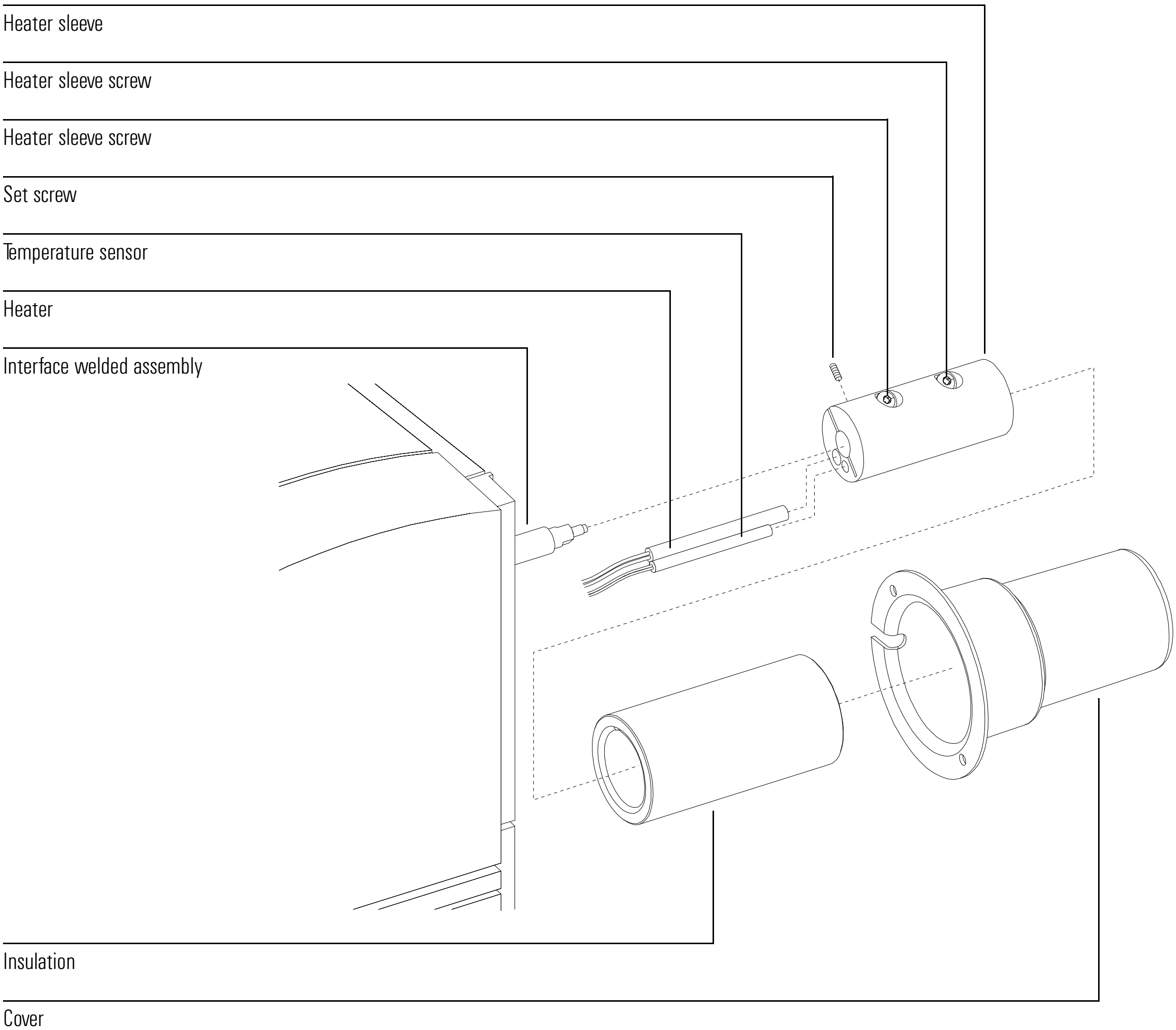
6 **Reconnect the MSD to the GC. See page 180.**

7 **Make sure you reconnect the GC/MSD interface cable to the GC. Make sure you reinstall the capillary column.**

8 **Pump down the MSD. See page 70.**

9 **Turn on the GC.**

Re-establish appropriate temperature setpoints for the GC/MSD interface and GC oven.


Heater sleeve
Heater sleeve screw
Heater sleeve screw
Set screw
Temperature sensor
Heater
Interface welded assembly
Insulation
Cover

Maintaining the electronics

The MSD electronics do not require any scheduled maintenance

None of the electronic components of the MSD need to be replaced on a regular schedule. None of the electronic components in the MSD need to be adjusted or calibrated on a regular schedule. Avoid unnecessary handling of the MSD electronics.

Very few of the electronic components are operator serviceable

The primary fuses can be replaced by the operator. The RF coils can be adjusted by the operator. All other maintenance of the electronics should be performed by your Agilent Technologies service representative.

WARNING **Improper use of these procedures could create a serious safety hazard. Improper use of these procedures could also result in serious damage to, or incorrect operation of, the MSD.**

WARNING **Vent the MSD and disconnect its power cord before performing any of these procedures *except* adjusting the RF coils.**

Electrostatic discharge is a threat to the MSD electronics during maintenance

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 printed circuit 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 anti-static wrist strap and take other anti-static 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, anti-static mat, if you must work on components or assemblies that have been removed from the MSD. This includes the analyzer.

CAUTION

In order to be effective, an anti-static wrist strap must fit snugly (not tight). A loose strap provides little or no protection.

CAUTION

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

More information is available

If you need more information about the functions of electronic components, refer to Chapter 8, *Electronics*, on page 307.

Most of the procedures in this chapter are illustrated with video clips.

To adjust the RF coils

Materials needed: Screwdriver, flat-blade, large (8730-0002)

1 **Make sure the MSD is at thermal equilibrium.**

It takes at least 2 hours ***after*** all heated zones have reached their setpoints for the MSD to reach thermal equilibrium.

2 **Remove the analyzer cover See page 62.**

WARNING **Do not remove the side board cover, the RF cover, or any other covers. Dangerous voltages are present under these covers.**

3 **Make sure the RF cover is secure and no screws are missing.**

A loose RF cover or missing screw can ***significantly*** affect coil adjustment.

4 **In the Tune and Vacuum Control view, select Optimize Quadrupole Frequency from the Execute menu.**

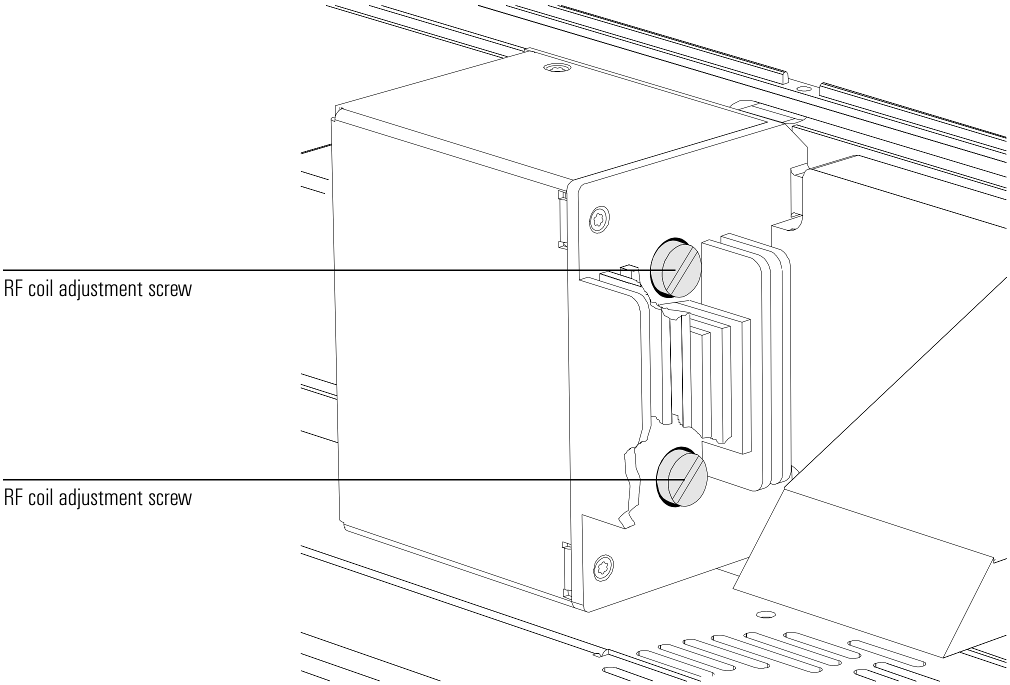
5 **Enter an amu value of 100.**

6 **Slowly turn the RF coil adjustment screws to minimize the voltage displayed.**

Turn the adjustment screws alternately. Turn each screw only a little bit at a time. Keep the screws at ***equal*** extension. The minimum voltage is typically between 70 and 100 mV.

CAUTION Do not use a coin to adjust the RF coils. If you drop it, it could fall into the electronics fan and cause significant damage.

7 **When the voltage is minimized, click the Stop button.**



8 Repeat steps 4 through 7 for 650 amu.

The minimum voltage is typically between 500 and 650 mV.

9 Exit the Set Optimize Quadrupole Frequency program.

10 Select MS OFF from the Execute menu.

11 Reinstall the analyzer cover.

12 Tune the MSD. See page 60.

To replace the primary fuses

Materials needed:

Fuse, T8 A, 250 V (2110-0969) – 2 required
Screwdriver, flat-blade (8730-0002)

The most likely cause of failure of the primary fuses is a problem with the foreline pump. If the primary fuses in your MSD fail, check the foreline pump.

1 Vent the MSD and unplug the power cord from the electrical outlet.

If one of the primary fuses has failed, the MSD will already be off, but for safety you should switch off the MSD and unplug the power cord. It is not necessary to allow air into the analyzer chamber.

WARNING

Never replace the primary fuses while the MSD is connected to a power source.

WARNING

If you are using hydrogen as a GC carrier gas, a power failure may allow it to accumulate in the analyzer chamber. In that case, further precautions are required. See the *Hydrogen Carrier Gas Safety Guide* (5995-5398).

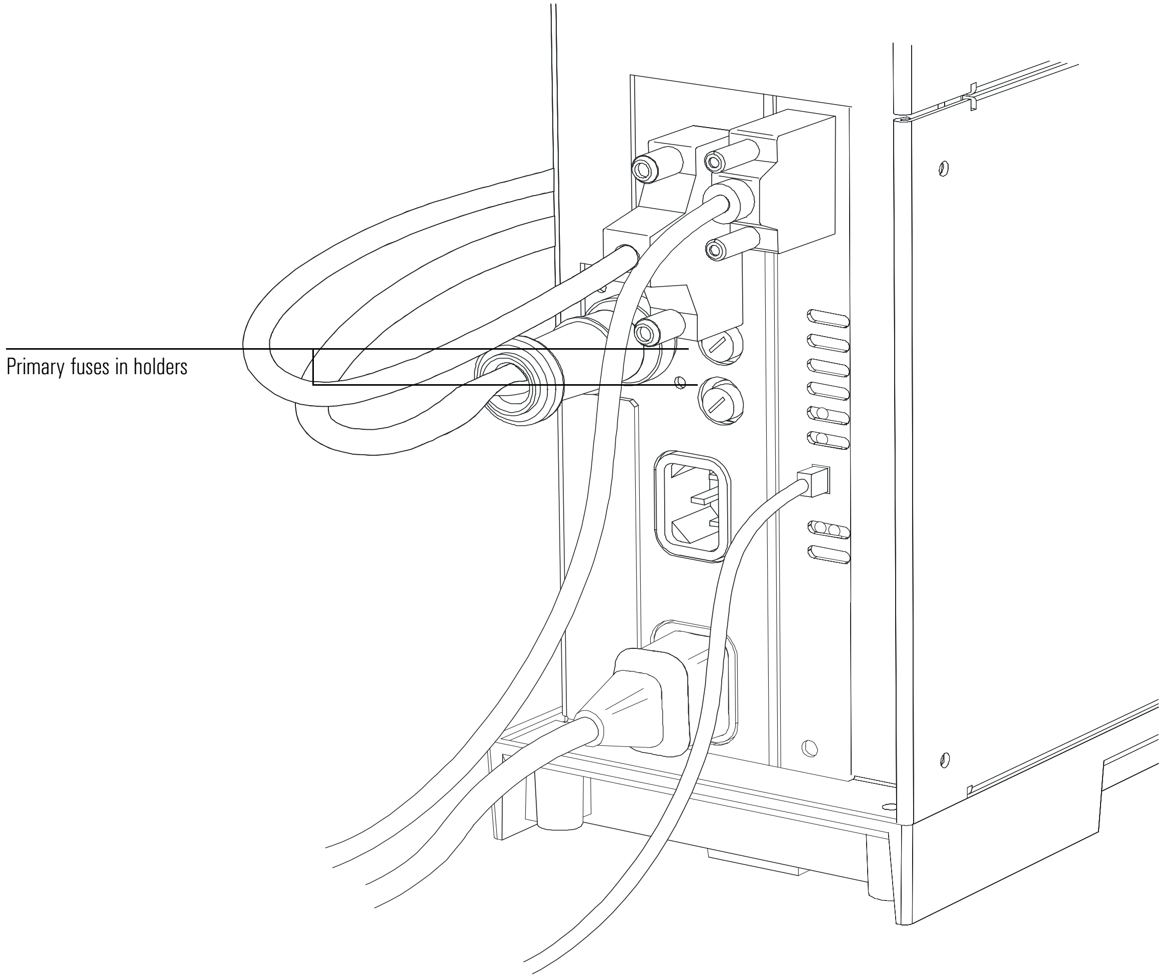
2 Turn one of the fuse holders counterclockwise until it pops out.

The fuse holders are spring loaded.

3 Remove the old fuse from the fuse holder.

4 Install a new fuse in the fuse holder.

5 Reinstall the fuse holder.



6 **Repeat steps 3 - 6 for the other fuse.**
   Always replace both fuses.

7 **Reconnect the MSD power cord to the electrical outlet.**

8 **Pump down the MSD. See page 70.**
   Always replace both fuses.

To replace the primary fuses

7

CI Maintenance

CI Maintenance

This chapter describes maintenance procedures and requirements that are unique to 5973 inert MSDs equipped with the Chemical Ionization hardware.

Maintenance videos on the multimedia manual in the 5973 inert User Preparation Kit

Most of these maintenance procedures are demonstrated on the multimedia MSD Reference Collection. Please view these videos.

CI increases the need for ion source cleaning

The primary effect of operating the MSD in CI mode is the need for more frequent ion source cleaning. In CI operation, the ion source chamber is subject to more rapid contamination than in EI operation because of the higher source pressures required for CI.

WARNING **Always perform any maintenance procedures using hazardous solvents under a fume hood. Be sure to operate the MSD in a well-vented room.**

Ammonia CI increases the need for foreline pump maintenance

Ammonia, when used as a reagent gas, it will also change the maintenance requirements slightly. Ammonia causes the foreline pump oil to break down more quickly. Therefore, the oil in the foreline vacuum pump must be checked and replaced more frequently.

Always purge the MSD with methane after flowing ammonia.

Be sure to install the ammonia so the tank is in an upright position. This will help prevent liquid ammonia from getting into the flow module.

To set up your MSD for CI operation

Setting up your CI MSD for operation in CI mode requires special care to avoid contamination and air leaks.

General guidelines

- Before venting in EI mode, verify that the GC/MSD system is performing correctly. See To verify system performance, 61.
- Make sure the reagent gas inlet line(s) are equipped with gas purifiers (not applicable for ammonia.)
- Use extra-high purity reagent gases; 99.99% or better for methane and as pure as is available for other reagent gases.

To install the CI ion source

CAUTION

Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded anti-static wrist strap and take other anti-static precautions ***before*** you open the analyzer chamber.

1 **Vent the MSD and open the analyzer. See page 64.**

2 **Remove the EI ion source. See page 203.**

3 **Remove the CI ion source from its storage box and insert the ion source into the radiator.**

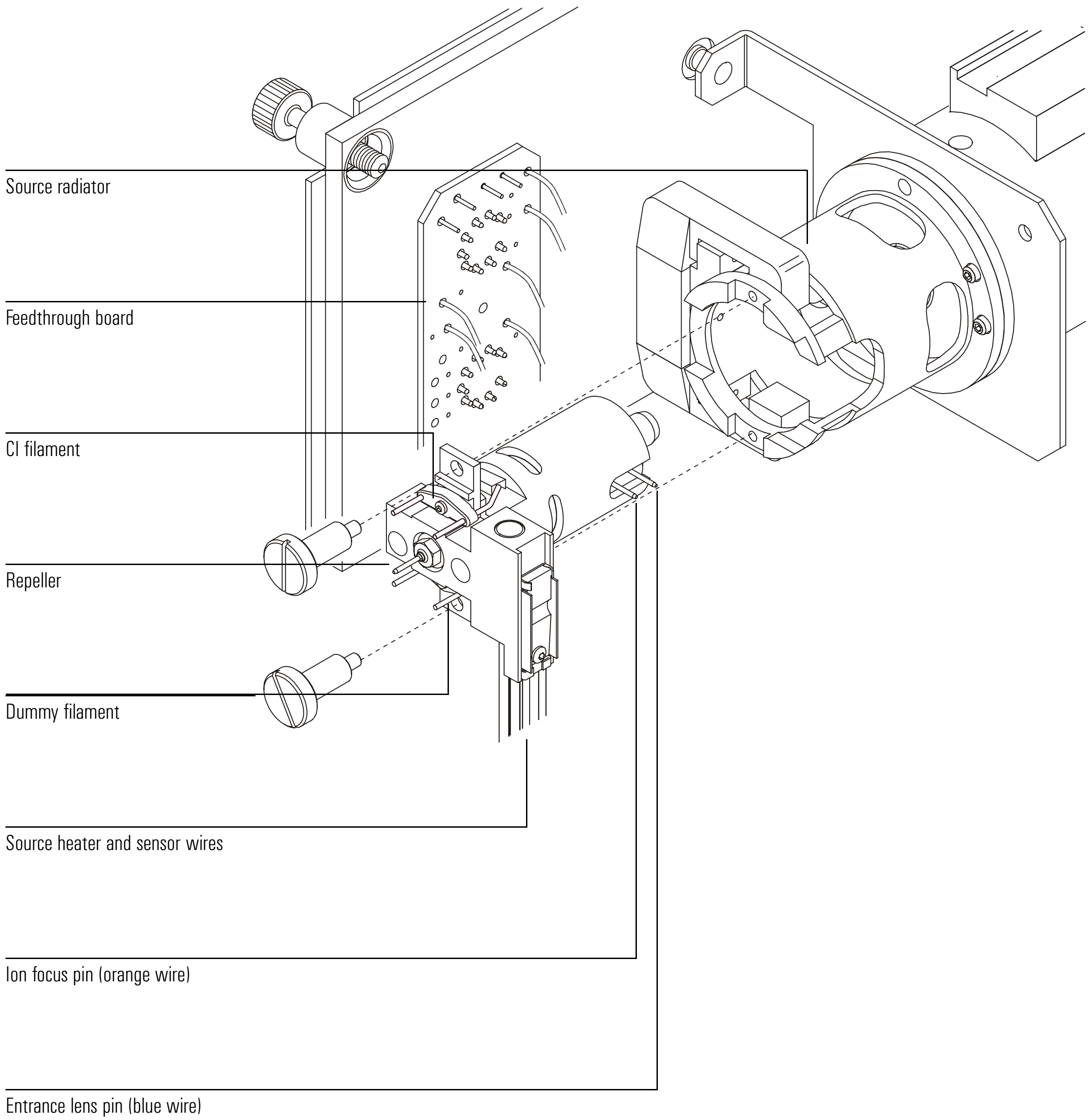
4 **Reinstall the thumbscrews.**

5 **Connect the dummy filament, repeller, and CI filament wires.**

6 **Reconnect colored wires to the appropriate pins on the ion source.**

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	Dummy filament “(Filament 2”)	2

7 **Connect the heater and sensor cables.**


Source radiator
Feedthrough board
CI filament
Repeller
Dummy filament
Source heater and sensor wires
Ion focus pin (orange wire)
Entrance lens pin (blue wire)

To install the CI interface tip seal

Materials needed: Interface tip seal (G1099-60412)

The interface tip seal must be in place for CI operation. It is necessary to achieve adequate ion source pressure for CI.

CAUTION Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded anti-static wrist strap and take other anti-static precautions ***before*** you open the analyzer chamber.

1 **Remove the seal from the ion source storage box.**

2 **Place the seal over the end of the interface. See the illustration on the previous page.**

To remove the seal, reverse the above steps.

3 **Verify that the CI ion source is installed.**

4 ***Gently*** **check the alignment of the analyzer and the interface.**

When the analyzer is aligned correctly, the analyzer can be closed all the way with no resistance except the spring tension from the interface tip seal.

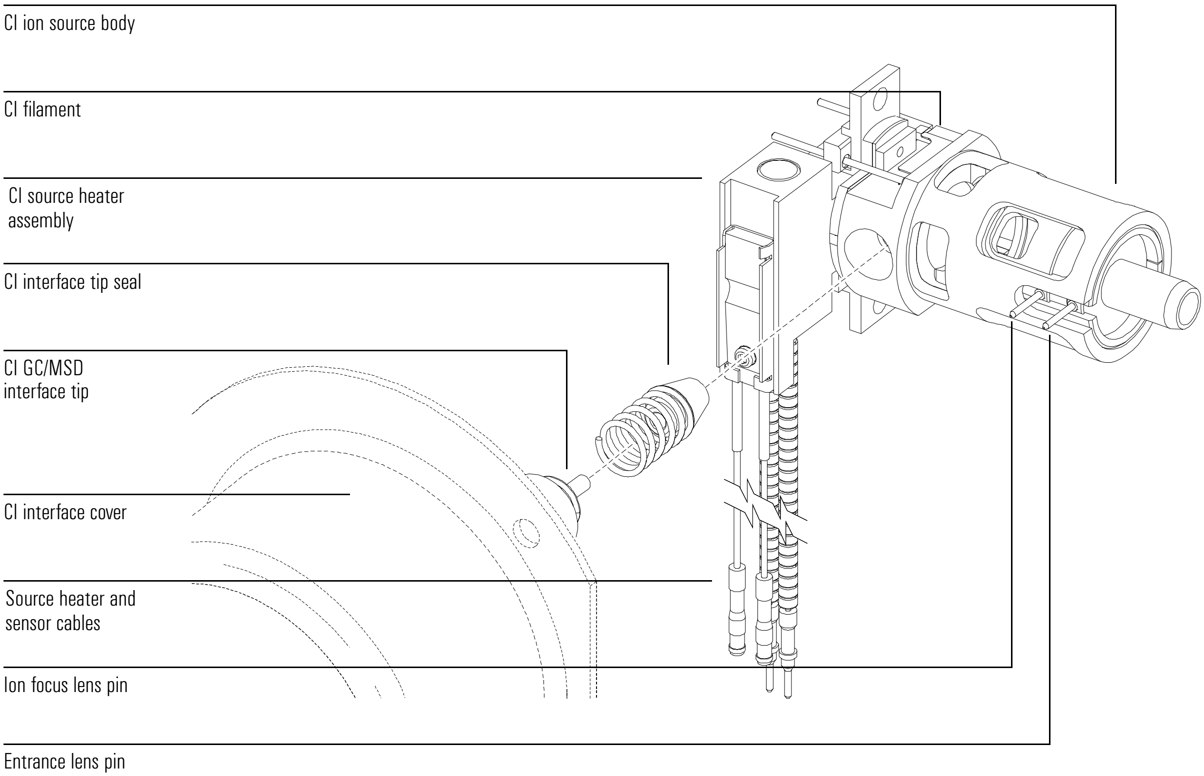
CAUTION Forcing the analyzer closed if these parts are misaligned will damage the seal or the interface or the ion source, or will keep the sideplate from sealing.

5 **You can align the analyzer and interface by wiggling the side plate on its hinge.**

If the analyzer still won't close, contact your Agilent Technologies service representative.

See video link to analyzer alignment. This procedure should be done by an Agilent Technologies Service representative. The following figure shows the alignment of the interface, tip seal, and CI ion source.

To install the CI interface tip seal



To clean the CI ion source

The CI ion source has slightly different cleaning requirements than the standard EI ion source. See the procedure in the 5973 inert MSD CD-ROM.

Frequency of cleaning

Because the CI ion source operates at much higher pressures than the EI ion source, it will probably require more frequent cleaning than the EI ion source. Cleaning of the source is not a scheduled, periodic maintenance procedure. The source should be cleaned whenever there are performance anomalies that are associated with a dirty ion source. See the *Troubleshooting* chapter for symptoms that indicate a dirty ion source. ***Visual appearance is not an accurate guide to cleanliness of the CI ion source. The CI ion source can show little or no discoloration yet still need cleaning.*** Let analytical performance be your guide.

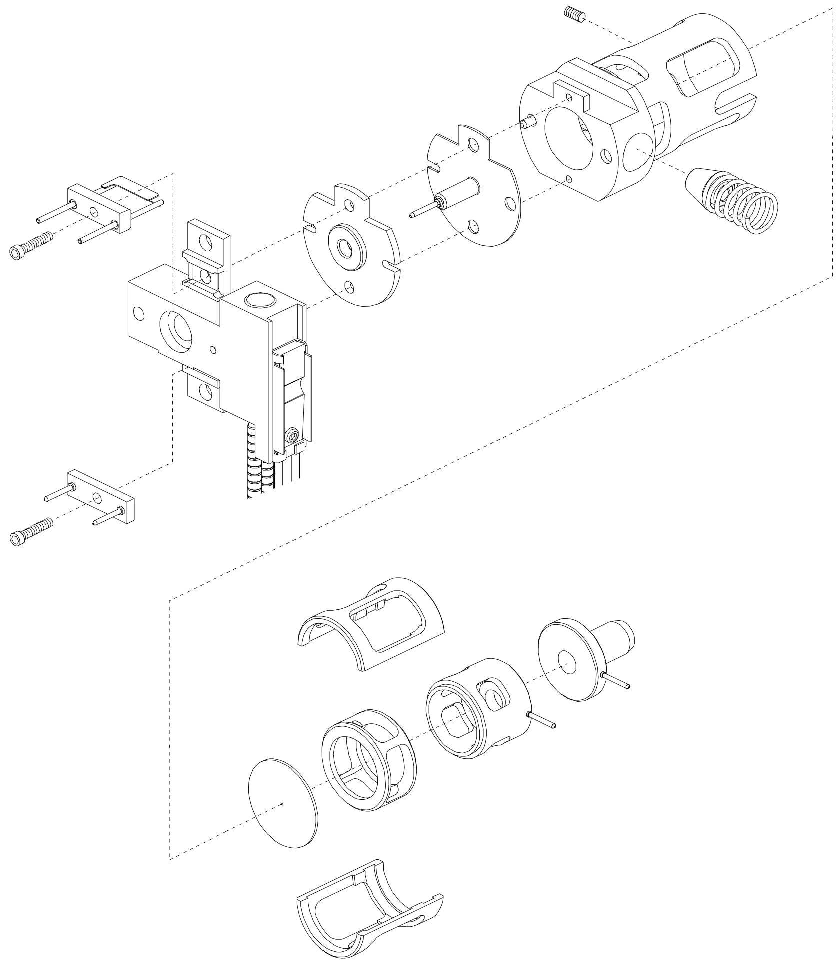
Cleaning procedure

Cleaning the CI ion source is very similar to cleaning the EI ion source. Use the cleaning procedure in *To clean the ion source, 207* with the following exceptions:

- The CI ion source may not look dirty but deposits left by chemical ionization are very difficult to remove. Clean the CI ion source thoroughly.
- Use a round wooden toothpick to gently clean out the electron entrance hole in the source body and the ion exit hole in the drawout plate.
- Do not use halogenated solvents, and use hexane for the final rinse.

See Also Refer to the MSD Reference Collection for video demonstrations of ion source cleaning and other maintenance procedures.

CAUTION Do not use any halogenated solvents to clean the CI ion source.

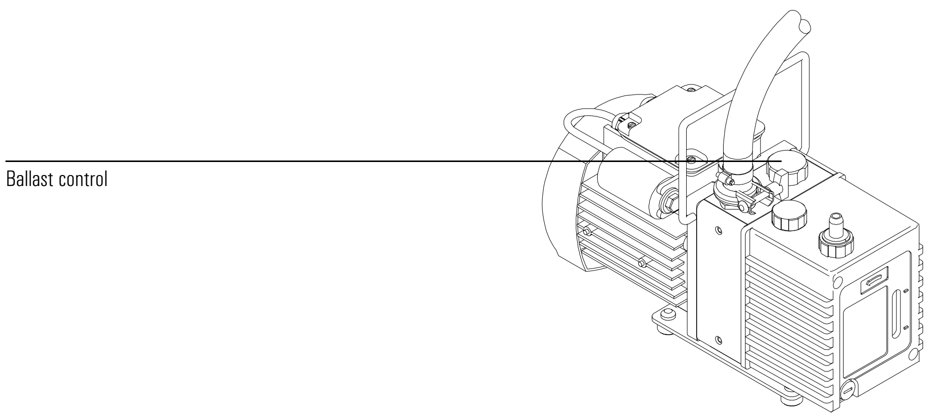


To minimize foreline pump damage from ammonia

Gas ballasting for an hour every day removes most, of the ammonia from the pump oil. This will greatly increase the life of the pump.

CAUTION

Only perform this procedure if the pump is at normal operating temperature. The water in the air can cause condensation of the ammonia at the ballast valve if the pump is cold.



1 **Open the ballast valve on the foreline pump all the way (several turns counterclockwise)**

2 **The sound of the pump will get much louder.**

3 **Leave the ballast valve open for one hour.**

You can continue to run samples while the pump is ballasting.

4 **Close the ballast valve.**

Leaving the ballast valve open all the time will result in loss of pump oil and damage to the pump.

CAUTION

Always purge the flow module with methane after flowing ammonia.
The use of ammonia reagent gas also requires that the foreline pump oil be changed every 2–3 months instead of the usual six months.

To replace the methane/isobutane gas purifier

Materials needed:

Methane/isobutane gas purifier (G1999-80410)
Front ferrule for 1/8-inch tubing (5180-4110)
Rear ferrule for 1/8-inch tubing (5180-4116)
Tubing cutter (8710-1709)

The methane/isobutane gas purifier needs to be replaced after four tanks of reagent gas. This frequency may vary depending on purity of the gas and care taken in uncapping and installing the gas purifier. A large leak upstream from the gas purifier can quickly exhaust the reduced metal of its oxygen and moisture traps.

1 **To install the methane/isobutane gas purifier, follow the instructions on the label for installation and replacement intervals.**

CAUTION

Be sure not to remove the caps until you are ready to install the gas purifier. Only remove the caps in the gas flow to prevent contamination by air.

WARNING

Methane is flammable. Extinguish all flames in the area before turning on gas flow.

2 **Disconnect the fittings on the old filter.**

3 **Remove the ferrules from the tubing at the outlet of the gas purifier.**

Using the tubing cutter, cut off the end of the tubing with the ferrules.

4 **Install the new filter.**

5 **Purge the new filter.**

6 **Cap the old filter and prepare it to be sent for regeneration.**

See the instructions on the label.

To clean the reagent gas supply lines (tubing)

Materials needed:

Clean, dry nitrogen
Heat gun
Tubing cutter (8710-1709)

If the reagent gas lines become contaminated, they can be cleaned.

1 **Disconnect the reagent gas tubing from the gas supply, the gas purifier, and the MSD.**

2 **Cap the gas purifier following the instructions on the label.**

3 **Connect one end of the tubing to a supply of clean, dry nitrogen and turn on gas flow.**

4 **Use the heat gun to warm the tubing, starting at the supply end and working your way to the free end.**

5 **Repeat for any other pieces of tubing that need to be cleaned.**

6 **Reconnect the tubing to the gas supply, gas purifier, and MSD.**

Follow the instructions on the gas purifier label.

WARNING **Do not heat the gas tubing when reagent gas is flowing.**

CAUTION Do not put liquids into the tubing. Do not heat the tubing when it is connected to the MSD.

To refill the CI calibrant vial

Materials needed: PFDTD calibrant (8500-8130)

1 **Set the reagent gas flow to Gas Off.**

2 **Vent the MSD.**

3 **Remove the capillary column from the GC/MSD interface.**

4 **Pull the MSD away from the GC. See page 178.**

5 **Loosen the nut holding the vial in place.**

6 **Remove the calibrant vial.**

CAUTION

Do ***not*** rinse the vial with any solvents. ***Never*** expose the inside of the vial to chlorinated solvents or isopropyl alcohol or water — this will result in severe loss of CI sensitivity.

7 **Fill the vial to no closer than 6 mm of the top with fresh PFDTD calibrant (8500-8130).**

8 **Replace the vial and tighten the nut.**

9 **Reposition the MSD next to the GC. See page 180.**

10 **Reinstall the capillary column.**

11 **Pump down the MSD. See page 70.**

12 **Purge the calibration valve. See page 186.**

CAUTION

After removing the calibrant vial, you ***must*** purge the calibration valve. Failure to do so will result in severe contamination of the ion source and damage to the filament and electron multiplier.

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8

Vacuum System

This chapter describes components of the vacuum system in the MSD

Vacuum System

The vacuum system is essential to MSD operation

The vacuum system creates the high vacuum (low pressure) required for the MSD to operate. Without the vacuum, the molecular mean free path would be too short, and ions would collide with air molecules before they could reach the detector. Operation at high pressures also would damage analyzer components.

The 5973 inert MSD has one of two kinds of vacuum system: a standard turbo molecular pump, and a performance turbomolecular (turbo) pump; this determines the maximum column flow that the MSD will support.

Model number	Description	Maximum recommended column flow
G2578A	Standard Turbo Pump, EI	2.0 mL/min
G2579A	Performance Turbo Pump, EI	4.0 mL/min
G2589A	Performance Turbo Pump, EI, PCI, NCI	4.0 mL/min

Many parts of the vacuum system are common to both, but some parts are specific to the high vacuum pump.

Most vacuum system operation is automated. Operator interaction is through the data system or control panel. Monitoring of the vacuum system is done through the data system and or control panel, and through the optional gauge controller.

Common vacuum system problems

The most common problems associated with any vacuum system are air leaks. Symptoms of air leaks include:

- Loud gurgling noise from the foreline pump (very large leak.)
- Inability of the turbo pump to reach 95% speed
- Higher than normal high vacuum gauge controller readings

The 5973 inert MSD will *not* pump down successfully unless you press on the side plate (analyzer door) when you turn on the MSD power. Continue to press until the sound from the foreline pump becomes quieter.

Pumpdown failure shutdown

The system will shut down both the high vacuum and the foreline pump if the system fails to pump down correctly. One condition that triggers the shutdown on turbo pump MSDs is a turbo pump speed below 80% after 7 minutes.

- Turbo pump MSDs: turbo pump speed below 80% after 7 minutes

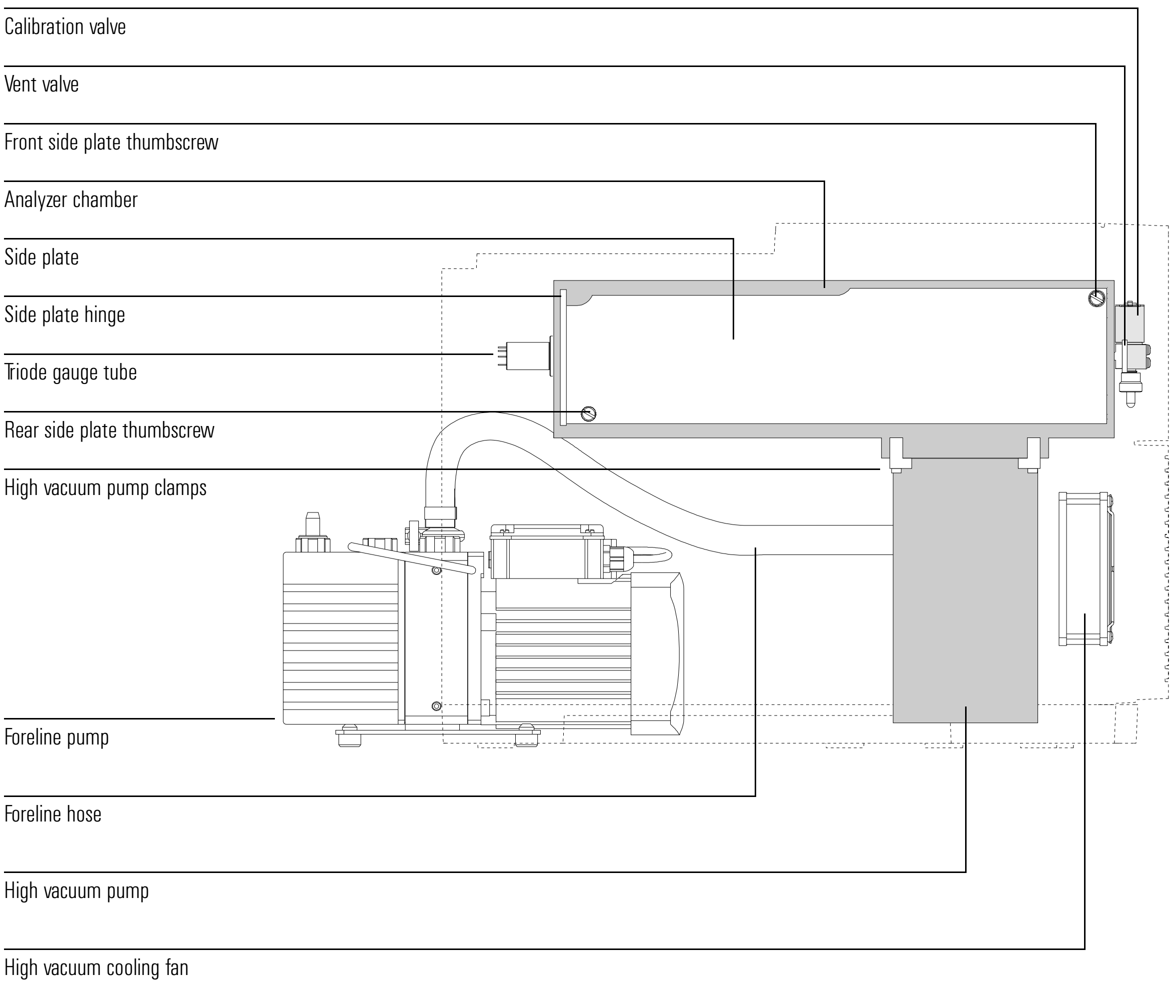
This is usually because of a ***large*** air leak: either the sideplate has not sealed correctly or the vent valve is still open. This feature helps prevent the foreline pump from sucking air through the system, which can damage the analyzer and pump.

To restart the MSD, find and correct the air leak, then switch the power off and on. Be sure to press on the sideplate when turning on the MSD power to ensure a good seal.

Many components make up the vacuum system

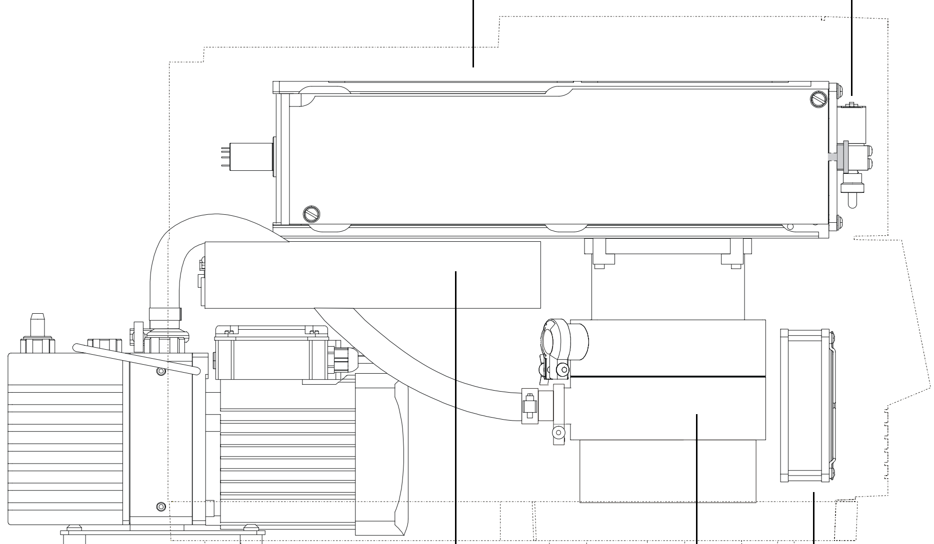
- Analyzer chamber
- Side plate (analyzer door), and front and rear end plates
- Vacuum seals
- Foreline (rough) pump
- High vacuum pump (turbomolecular pump)
- Calibration valve(s) and vent valve
- Vacuum control electronics
- Vacuum gauges and gauge control electronics

Each of these is discussed in more detail in the following material.


Calibration valve
Vent valve
Front side plate thumbscrew
Analyzer chamber
Side plate
Side plate hinge
Triode gauge tube
Rear side plate thumbscrew
High vacuum pump clamps
Foreline pump
Foreline hose
High vacuum pump
High vacuum cooling fan

Turbo pump MSD vacuum system

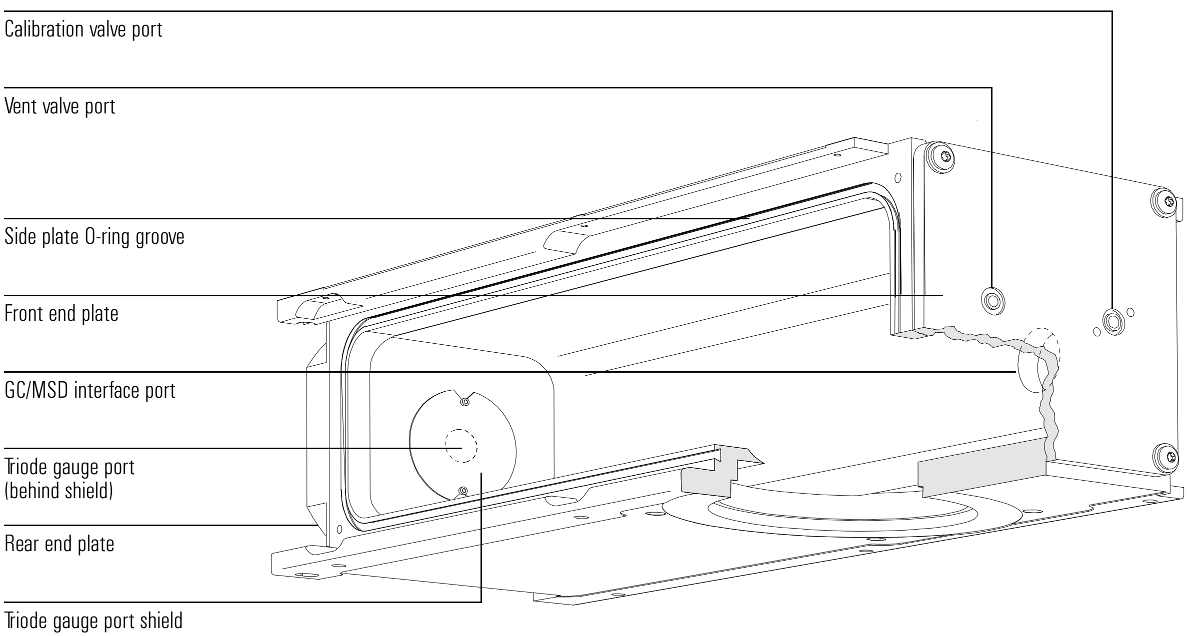
The 5973 inert MSD can have one of two turbo pumps. The performance turbo pump can accept up to 4 mL/min carrier gas flow, while the standard turbo pump can accept up to 2.4 mL/min carrier gas flow. The turbo pump has a screen to keep debris out of the pump, but no baffle is necessary. Pump speed is controlled by the turbo controller; there is no foreline gauge.



Turbo pump analyzer chamber

The analyzer chamber is the chamber in which the analyzer operates. The manifold is extruded and machined from an aluminum alloy. Large openings in the side, front, and rear of the analyzer chamber are closed by plates. O-rings provide the seals between the plates and the manifold. Ports in the manifold and the plates provide attachment points for the triode gauge tube, calibration valve, vent valve, GC/MSD interface, and high vacuum pump.

The turbo pump and the mounting bracket for the turbo controller are clamped directly to the manifold.



Side plate

The side plate is a flat stainless steel plate that covers the large opening in the side of the analyzer chamber. The side plate is attached to the manifold with a hinge. The analyzer assembly is attached to the side plate inside the analyzer chamber. The hinge allows the side plate to swing away from the manifold for easy access to the analyzer.

Several electrical feedthroughs are built into the side plate. Wires connect the feedthroughs to analyzer components. The electronic side board is mounted on the atmospheric side of the side plate.

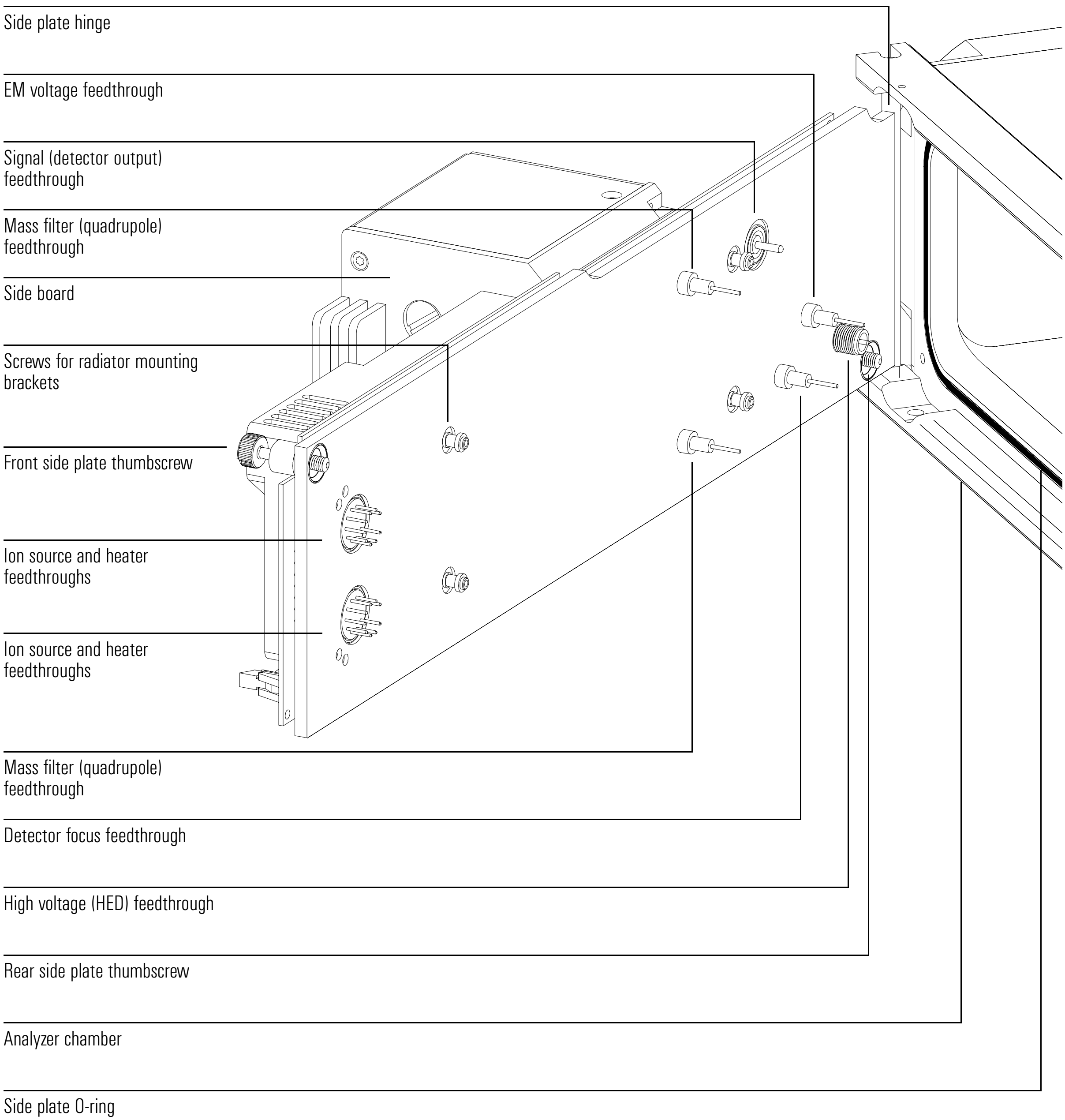
Thumbscrews are located at each end of the side plate.

CAUTION

Fasten both side plate thumbscrews for shipping or storage ***only***. For normal operation the both thumbscrews should be loose. For operation with hydrogen carrier gas, or with flammable or explosive CI reagent gases, the front thumbscrew should be fastened just finger tight. Overtightening will warp the side plate and cause air leaks. ***Do not*** use a tool to tighten the side plate thumbscrews.

CAUTION

When you turn on the power to pump down the MSD, be sure to press on the side board to ensure a good seal.


Side plate hinge
EM voltage feedthrough
Signal (detector output) feedthrough
Mass filter (quadrupole) feedthrough
Side board
Screws for radiator mounting brackets
Front side plate thumbscrew
Ion source and heater feedthroughs
Ion source and heater feedthroughs
Mass filter (quadrupole) feedthrough
Detector focus feedthrough
High voltage (HED) feedthrough
Rear side plate thumbscrew
Analyzer chamber
Side plate O-ring

Vacuum seals

Several types of Viton elastomer O-ring seals are used to prevent air leaks into the analyzer chamber. All these O-rings, and the surfaces to which they must seal, must be kept clean and protected from nicks and scratches. A single hair, piece of lint, or scratch can produce a serious vacuum leak. Two of the O-rings are ***lightly*** lubricated with Apiezon-L vacuum grease: the side plate O-ring and the vent valve O-ring.

Face seals

A face seal is an O-ring that fits in a shallow groove. The sealing surface is usually a flat plate. The manifold side plate and end plate O-rings fit into grooves around the large openings in the analyzer chamber. The side plate swings into place against the side plate O-ring, and must be held in place when the MSD is turned on for pumpdown in order to assure a good seal.

The front and rear end plates are screwed onto the manifold, and should not need to be removed. The GC/MSD interface fastens to the manifold with three screws.

The calibration valve assembly is fastened onto the front end plate by two screws. The vent valve knob threads into the front end plate. Small O-rings in grooves in the front end plate provide vacuum seals.

The diffusion pump baffle adapter has a groove for its O-ring. The baffle adapter is clamped to the manifold with 4 claw grips.

KF (NW) seals

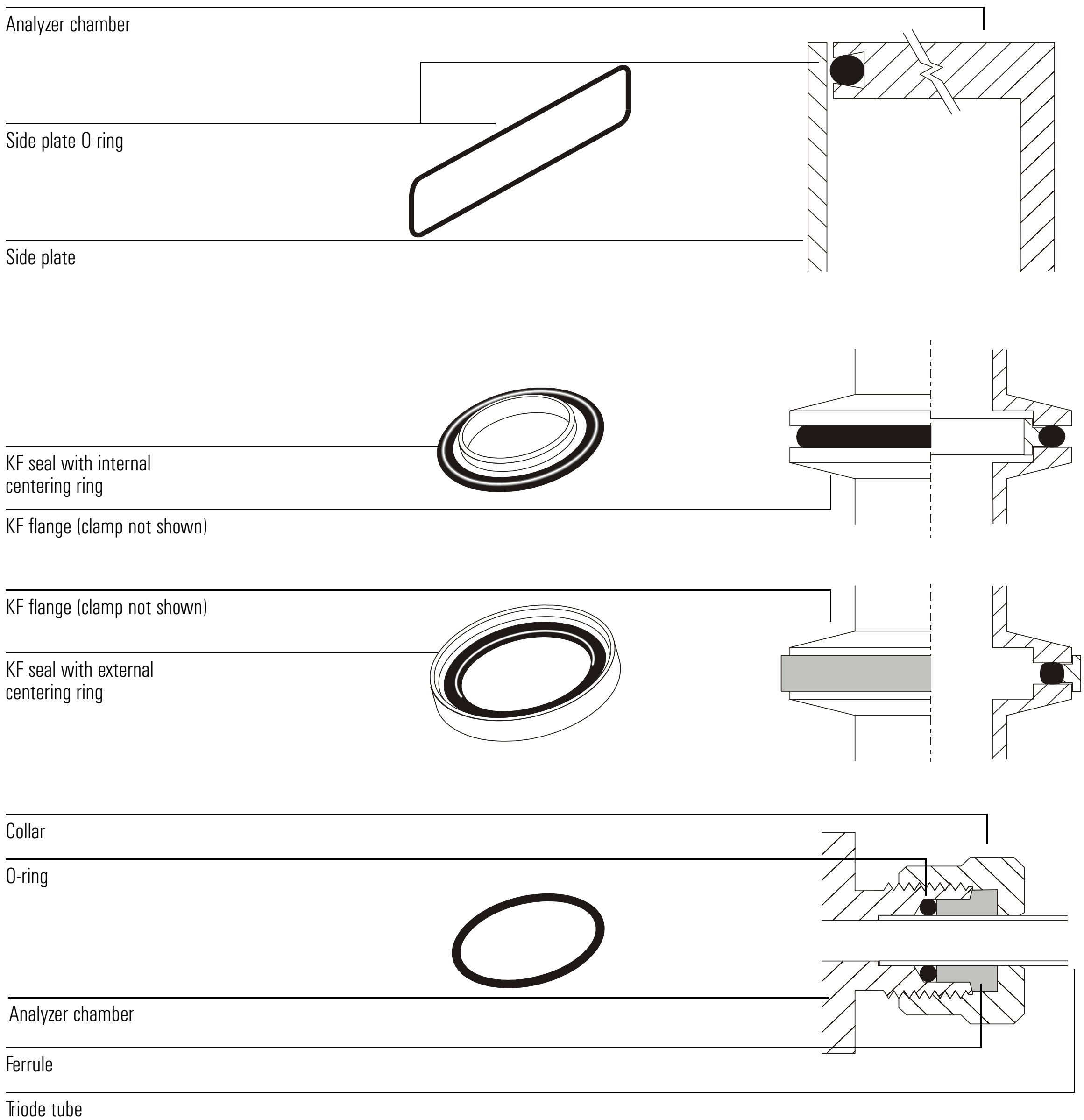
Most of the seals for the high vacuum pumps, foreline gauge, and foreline pump are KF seals. KF seals have an O-ring supported by a centering ring. The centering ring can be either on the inside or the outside of the O-ring. The clamp presses two flanges against the O-ring, making a seal. KF clamps must not be overtightened.

Compression seals

A compression fitting consists of a threaded fitting on the analyzer chamber and a threaded collar with a ferrule and O-ring. A cylindrical part fits inside the collar. Tightening the collar presses the ferrule, compressing the O-ring around the part. The triode gauge tube and calibration vial use compression seals.

High voltage feedthrough seal

The high voltage (HED) feedthrough seal is an O-ring that is compressed against the side plate by a threaded collar.



Foreline pump

The foreline pump reduces the pressure in the analyzer chamber so the high vacuum pump can operate. It also pumps away the gas load from the high vacuum pump. The foreline pump is connected to the high vacuum pump by a 130-cm hose called the foreline hose.

The foreline pump is a two-stage rotary-vane pump. The foreline pump turns on when the MSD power is turned on. The foreline pump has a built-in anti-suckback valve to help prevent backstreaming in the event of a power failure.

The foreline pump can be placed under the analyzer chamber at the rear of the MSD (with the exhaust outlet to the rear), or on the floor below the MSD.

An oil trap (not shown) is available that can be used to filter pump oil out of the foreline pump exhaust. This trap stops ***only*** pump oil. Do not use the trap if you are analyzing toxic chemicals or using toxic solvents, or if you have a CI MSD. Instead, install an 11-mm id hose to remove the exhaust from your lab.

WARNING

The foreline pump exhaust contains traces of solvents, analytes, and foreline pump oil. The supplied oil trap stops only 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.

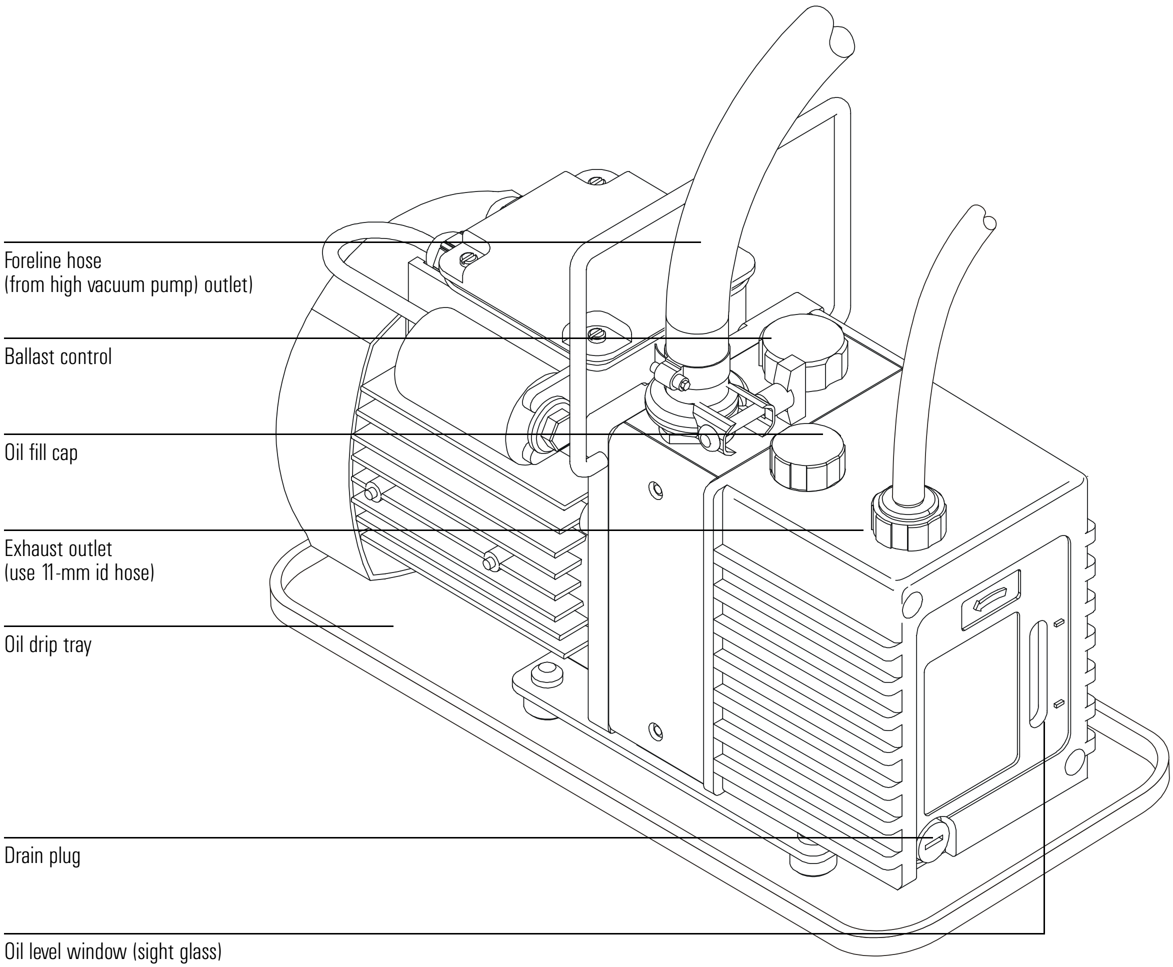
CAUTION

Do not place the foreline pump near any equipment that is sensitive to vibration.

CAUTION

The ballast control knob controls the amount of air allowed into the pump. Keep the ballast control closed (fully clockwise) at all times, except when ballasting the pump.

A window (sight glass) in the front of the foreline pump shows the level of the foreline pump oil. There are two marks next to the window. The level of the pump oil should never be above the upper mark or below the lower mark. If the level of pump oil is near the lower mark, add foreline pump oil.


Foreline hose
(from high vacuum pump) outlet)
Ballast control
Oil fill cap
Exhaust outlet
(use 11-mm id hose)
Oil drip tray
Drain plug
Oil level window (sight glass)

Turbomolecular pump and fan

The turbo pump in the MSD is clamped directly to the bottom of the analyzer chamber.

The turbo pump has a cylindrical body with its inlet open to the interior of the analyzer chamber. Inside the pump body is a central shaft or cylinder. Sets of small blades (airfoils) radiate from the central shaft. The shaft spins at up to 60,000 revolutions per minute in the performance turbo pump, and 90,000 rpm in the standard turbo pump.

The turbo pump transports gas by momentum transfer. The turbine blades are angled so that when they strike a gas molecule it is deflected downward. Each set of blades pushes the gas molecules further down toward the pump outlet. The foreline pump is connected by a hose to the outlet of the turbo pump. It removes the gas molecules that reach the outlet.

A controller regulates current to the pump and monitors pump motor speed and temperature. A cooling fan is located between the turbo pump and the front panel of the MSD. The fan draws air from outside the MSD and blows it over the pump.

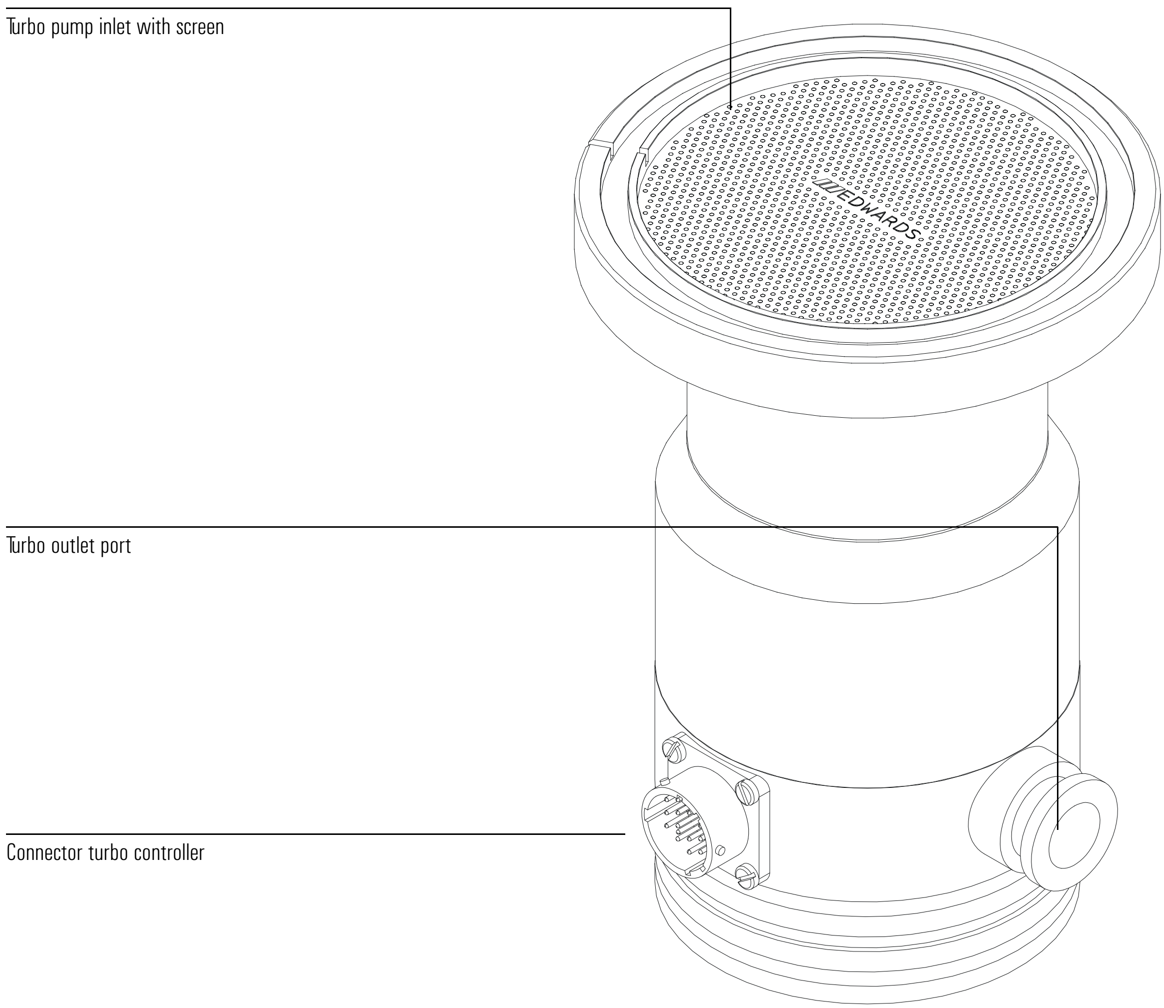
The turbo pump turns on automatically as soon as the MSD power is switched on. The system will allow the analyzer to be turned on when the turbo pump is greater than 80% speed, but the pump normally operates at 100% speed. Turbo pump MSDs typically maintain an indicated pressure below 8×10^{-5} Torr for helium column flows up to 4 ml/minute for the performance turbo pump, and up to 2 mL/minute for the standard turbo pump. Pressure (vacuum) can only be measured if your MSD is equipped with the optional gauge controller.

The turbo pump spins up (starts) and spins down (stops) quickly. This simplifies pumpdown and venting. From initial power-on, the system can pump down to operating pressure in 5 to 10 minutes.

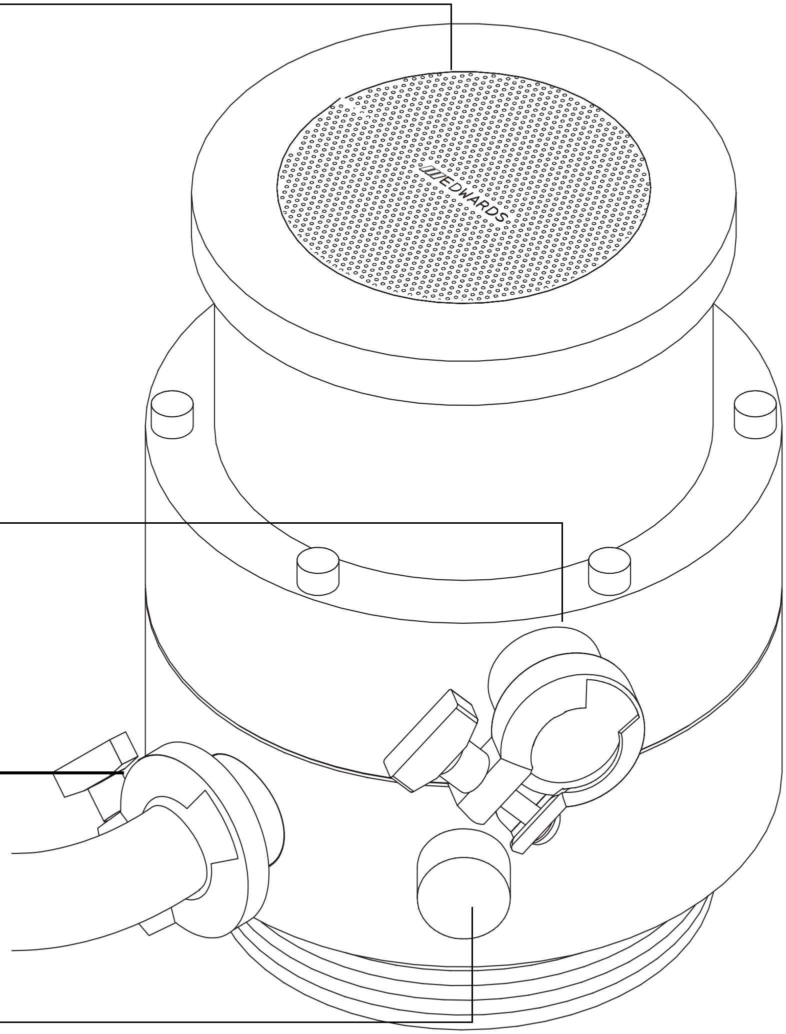
See Also

Gauge controller, page 277
To pump down the MSD, page 70
To vent the MSD, page 64
Turbo pump control, see 316
Table 2. Typical MSD pressure readings for various carrier gas flow rates, page 57

Standard turbo pump



Performance turbo pump



Calibration valves and vent valve

Calibration valves

A calibration valve is an electromechanical valve with a vial to hold the tuning compound. When a calibration valve is opened, tuning compound in the vial diffuses into the ion source. EI MSDs have only one calibration valve, while CI MSDs have another calibration valve for the CI tuning compound. The valves are controlled by the MSD ChemStation.

EI calibration valve

The EI calibration valve is held onto the front end plate of the analyzer chamber by two screws. A small O-ring provides a face seal.

The standard turbo pump MSD has a calibration valve with restrictor with less restriction than that in the performance turbo MSD; this is to allow the correct diffusion of calibrant for each vacuum system.

Perfluorotributylamine (PFTBA) is the most commonly used tuning compound for EI operation. PFTBA is required for automatic tuning of the MSD. Other compounds can be used for manual tuning.

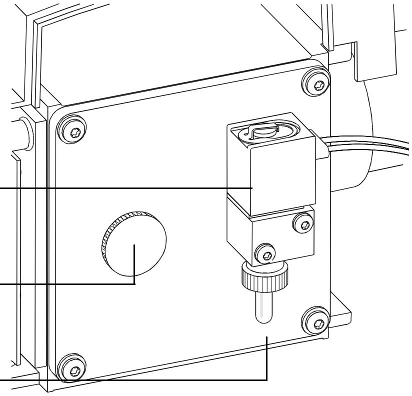
CI calibration valve

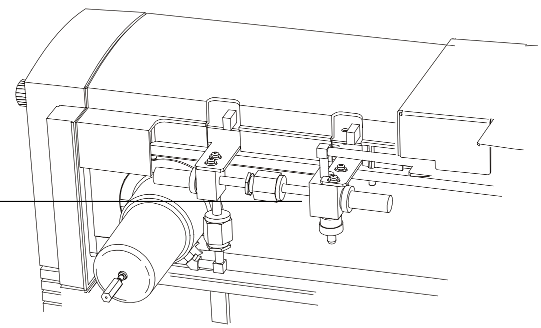
The CI tuning compound is perfluoro-5,8-dimethyl-3,6,9-trioxidodecane (PFDTD). The CI calibration valve is part of the reagent gas flow control module. It is controlled by the ChemStation software, and opens automatically during CI autotune or manual tuning, allowing PFDTD to diffuse through the GC/MSD interface and into the ion source.

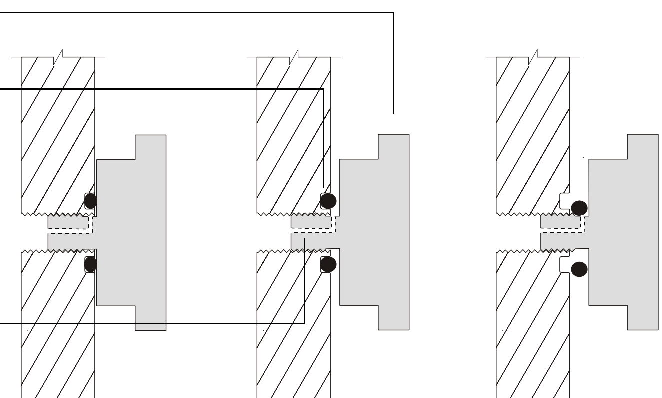
Vent valve

The vent valve knob screws into a threaded port in the front end plate. An O-ring is compressed between the knob and the end plate to form a seal. The threaded end of the knob has an air passage inside it, allowing air to flow into the manifold when the knob is partially unscrewed. If you turn the knob too far, the O-ring can come out of its slot.

Calibration valves and vent valve







Valve closed **Valve open** **Valve open too far**

Triode gauge tube

The MSD is equipped with a triode gauge tube connected to the analyzer chamber. With the optional 59864B Gauge Controller, the triode gauge can be used to measure the pressure (high vacuum) in the analyzer chamber. The triode gauge will not operate at pressures above 8×10^{-3} Torr. The triode gauge cannot be used without the gauge controller.

WARNING **Parts of the triode gauge tube operate at approximately 150 VDC. Turn off the triode gauge before working near it.**

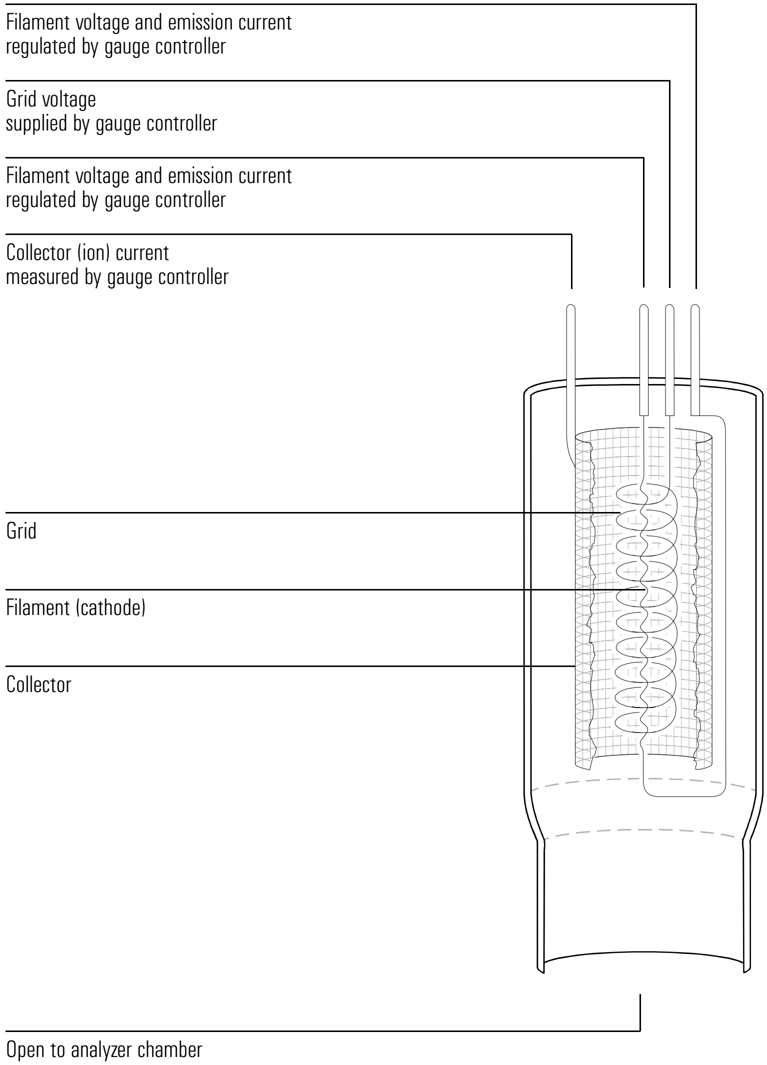
WARNING **The triode filament can ignite hydrogen. Never turn on the tube if there is a possibility that hydrogen has accumulated in the manifold.**

The triode gauge relies on the ionization of gas molecules to establish a pressure-dependent current flow. In the triode gauge, a regulated electrical current is passed through a filament called the cathode, causing it to emit electrons. The electrons accelerate from the filament toward a surrounding grid which is held at a higher potential (+150 VDC).

The emitted electrons ionize gas molecules in the tube. Positive ions are driven to a grounded wire mesh collector. At the collector, the positive ions regain missing electrons. This generates current in the collector. The number of ions formed is a function of the number of gas molecules present, that is, the gas pressure. Therefore, pressure can be calculated based on the current applied to the filament (cathode) and the current measured in the collector.

Since one end of the triode gauge tube is open to the analyzer chamber, the pressure in the triode gauge is essentially the same as the pressure in the analyzer chamber. To prevent electronic noise from the triode gauge tube from interfering with the detector, a small z-fold baffle is inserted into the stem of the triode gauge tube; and a shield is installed in the analyzer chamber between the tube port and the ion source.

Unlike some other pressure gauges that work by ionization, the triode gauge does not require degassing to remove accumulated ions from the surfaces in the gauge. In some cases, however, new gauge tubes will not display pressures accurately until they have been turned on for several hours.



The glass around the connector pins is easily cracked if the pins are moved too much. Be very careful when connecting and disconnecting the cable to avoid damage to the tube and creating air leaks.

Gauge controller

The optional 59864B Gauge Controller allows you to use the triode gauge tube to monitor the pressure in the MSD analyzer chamber. This can aid in everyday operation and in troubleshooting.

The 59864B Gauge Controller includes the controller and a cable for connecting the controller to the triode gauge. A power cord is supplied with a plug appropriate for the country from which the order was placed. The gauge controller can operate on all voltages between 100 and 240 VAC (nominal) and at ac frequencies of 50 to 60 hertz. The fuse in the gauge controller is appropriate for all allowed voltages.

The gauge controller regulates emission current to the filament of the triode gauge tube. It also measures the ion current in the collector. From these data, the gauge controller calculates and displays the pressure present in the analyzer chamber. The analyzer chamber pressure (in Torr) is displayed on the front panel of the controller.

The gauge controller is calibrated for nitrogen (N_2). The carrier gas is usually helium, which has does not ionize as readily as nitrogen. Therefore, the ***indicated*** pressure for helium is approximately 6 times lower than the absolute pressure. For example, a reading of 2.0×10^{-5} Torr versus an absolute pressure of 1.2×10^{-4} Torr. In a CI MSD, the indicated pressure reflects the contribution of both the carrier gas and the reagent gas. The distinction between indicated and absolute pressure is not important for normal operation of the MSD. Of greater concern are changes in pressure from hour to hour or day to day. These changes can indicate air leaks or other problems with the vacuum system. All the pressures listed in this manual are indicated pressures for helium carrier gas. The gauge controller setpoints are also indicated pressures.

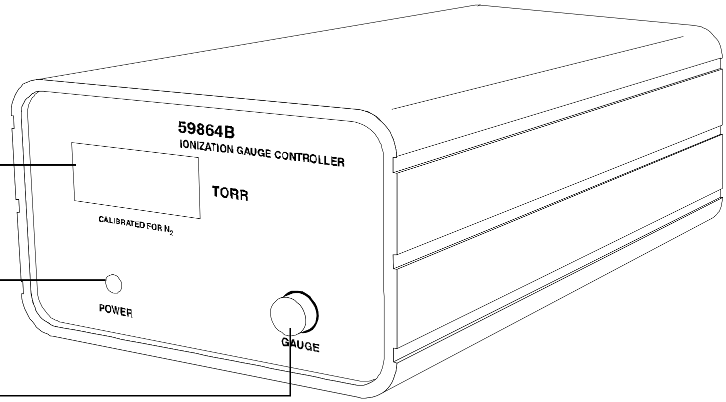
See Also

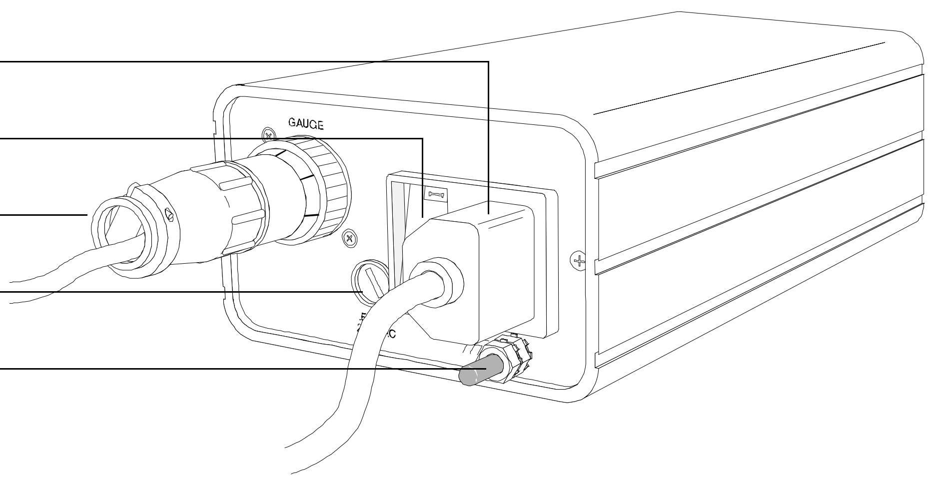
To monitor high vacuum pressure, page 56

WARNING

The filament in the triode gauge tube can ignite hydrogen. Never turn on the tube if there is a possibility that hydrogen has accumulated in the manifold.

Gauge controller





GC/MSD Interfaces and CI Flow Control

This chapter describes the function of the GC/MSD interfaces and the CI reagent gas flow control module

GC/MSD interface

The GC/MSD interface 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. A channel machined into the flange for the seal provides thermal isolation between the heated interface and the O-ring and manifold. The GC/MSD interface is covered by 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 GC oven. This end is threaded (thread size is 10 × 32), allowing connection of the column with a nut and ferrule. The other end of the GC/MSD interface fits into the ion source. The last two 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 heater is powered and controlled by Thermal Aux #2 heated zone of the 6890 Series GC. The GC/MSD interface temperature can be set from the MSD ChemStation or from the keypad of the gas chromatograph. A sensor (thermocouple) in the GC/MSD interface monitors the temperature.

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

See Also

To install a capillary column in the GC/MSD interface, 38

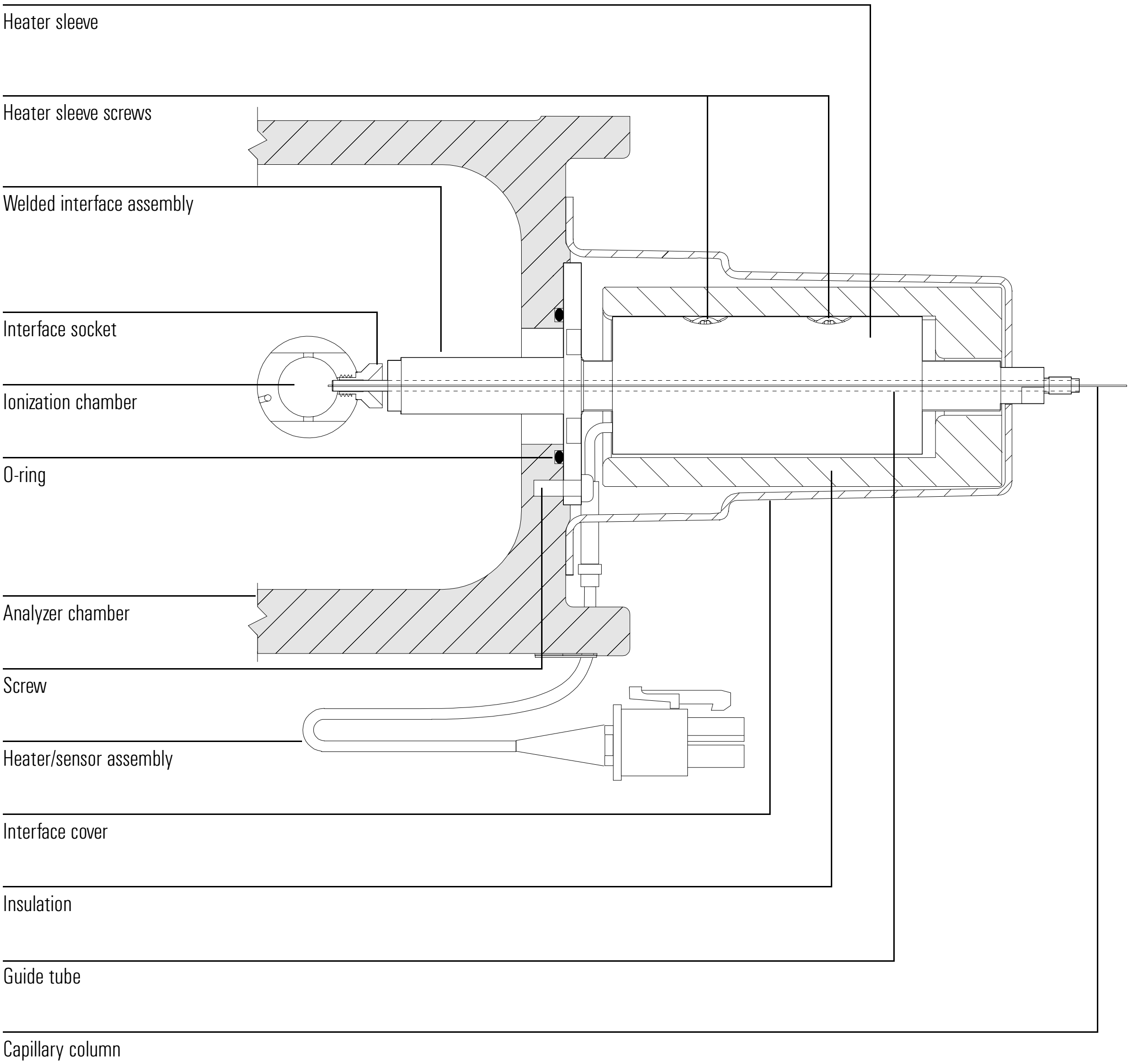
CAUTION

Never exceed the maximum column temperature, either in the interface or the GC oven.

WARNING

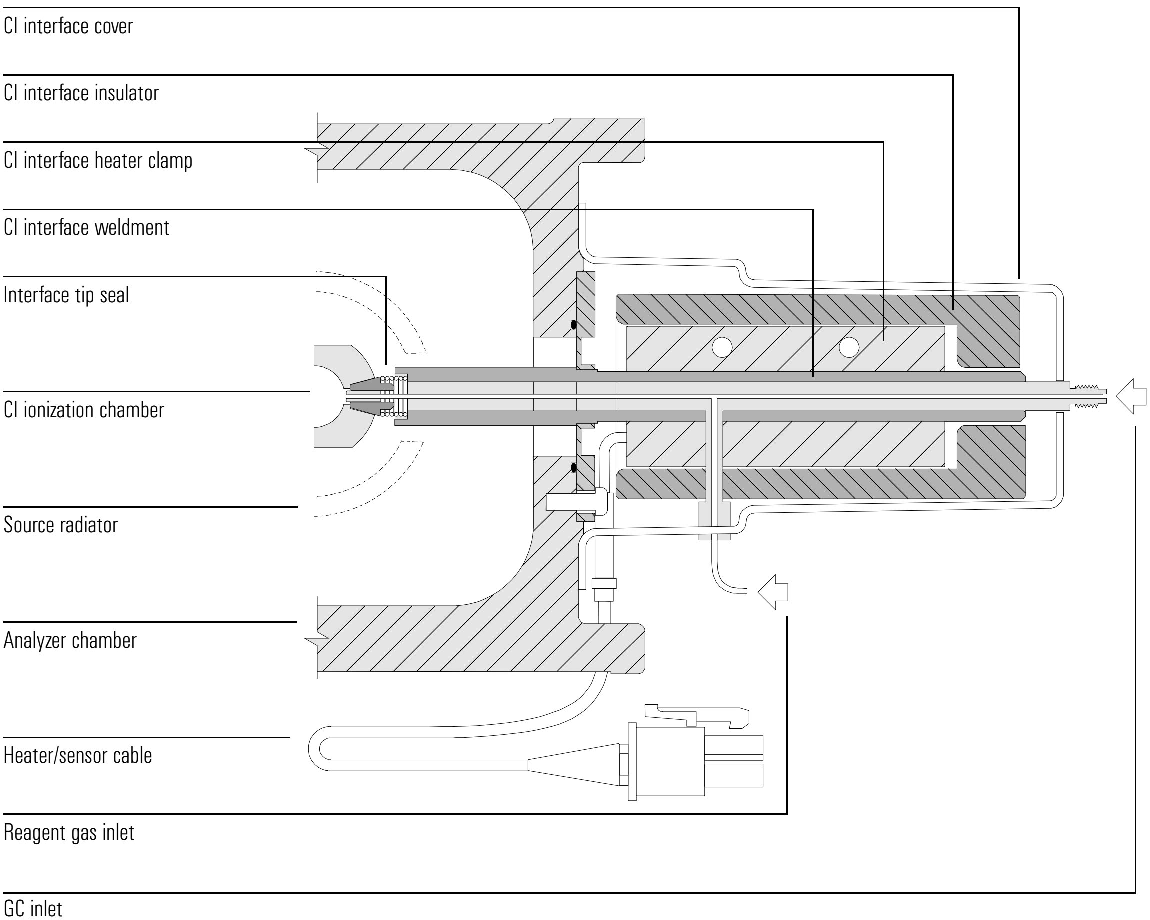
The GC/MSD interface operates at high temperatures. If you touch it when it is hot, it will burn you.

EI GC/MSD interface



EI/CI GC/MSD interface (CI interface)

The CI interface mounts onto the side of the analyzer chamber, with one end in the GC oven and the other in the MSD. Reagent gas is plumbed into the interface. The tip of the interface assembly extends into the ionization chamber. A spring-loaded seal keeps reagent gases from leaking out around the tip. The reagent gas is plumbed into the interface body, and mixes with carrier gas and sample in the ion source. This interface is also used for EI operation in CI MSDs.

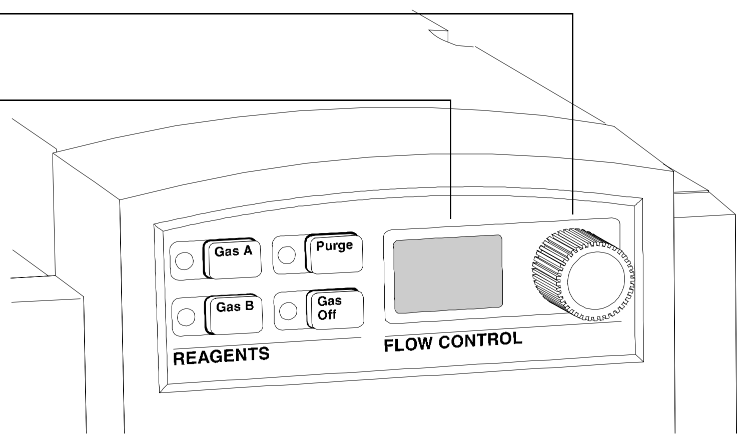


Reagent gas flow control module

The CI reagent gas flow control module regulates the flow of reagent gas into the EI/CI GC/MSD interface. The flow control module consists of a mass flow controller (MFC), gas select valves, CI calibration valve, isolation valve, control panel, control and display electronics, and plumbing. The back panel provides Swagelok inlet fittings for methane and one other reagent gas. The other fittings in the flow module are VCR fittings; VCR fittings have a disposable gasket that must be replaced every time the seal is opened. Operation of the flow module is through the control panel on the front. **Gas A** must be plumbed with Methane. **Gas B** can be plumbed with any other reagent gas.
Operation of the flow module is through the control panel on the front.

Flow control knob

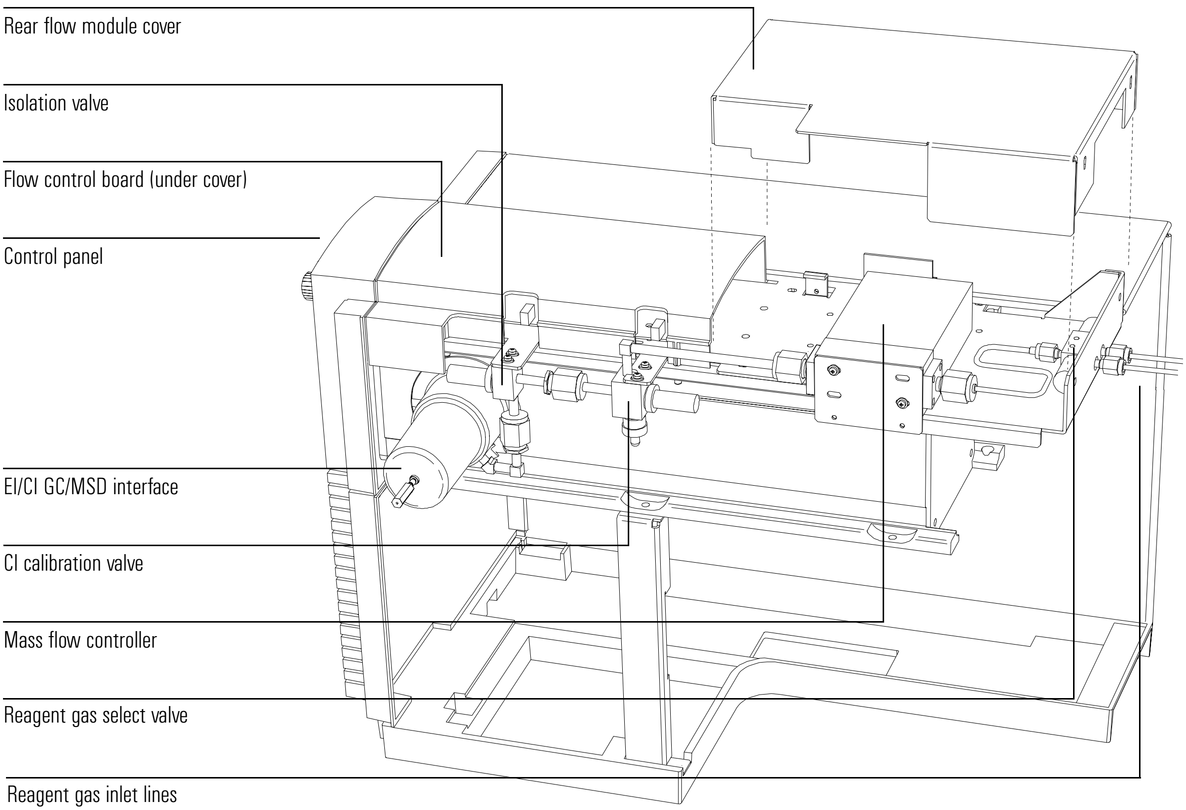
Flow control display



- Each button on the control pane has an LED (light) next to it. When that button is active, the light is on.
- **Gas A** or **Gas B** opens the chosen gas select valve. Only one can be open at a time.
- **Gas Off** closes the gas select and isolation valves. **Gas Off** also sets the MFC to 0% flow, unless **Purge** is on. The **Gas Off** LED must be off to turn on **Gas A** or **Gas B**
- The flow control knob is used to adjust the flow. When no gases are turned on the Flow Control display will show dashes: ––.
- **Purge** sets the MFC to 100% of total flow (fully open), regardless of the position of the flow control knob or the state of the select valves.
- The flow control display shows the gas flow as a percentage of the total possible flow (5 ml/min for ***methane***). If the display is flashing, the controller cannot maintain the selected flow. This usually means that the reagent gas supply does not have high enough pressure.

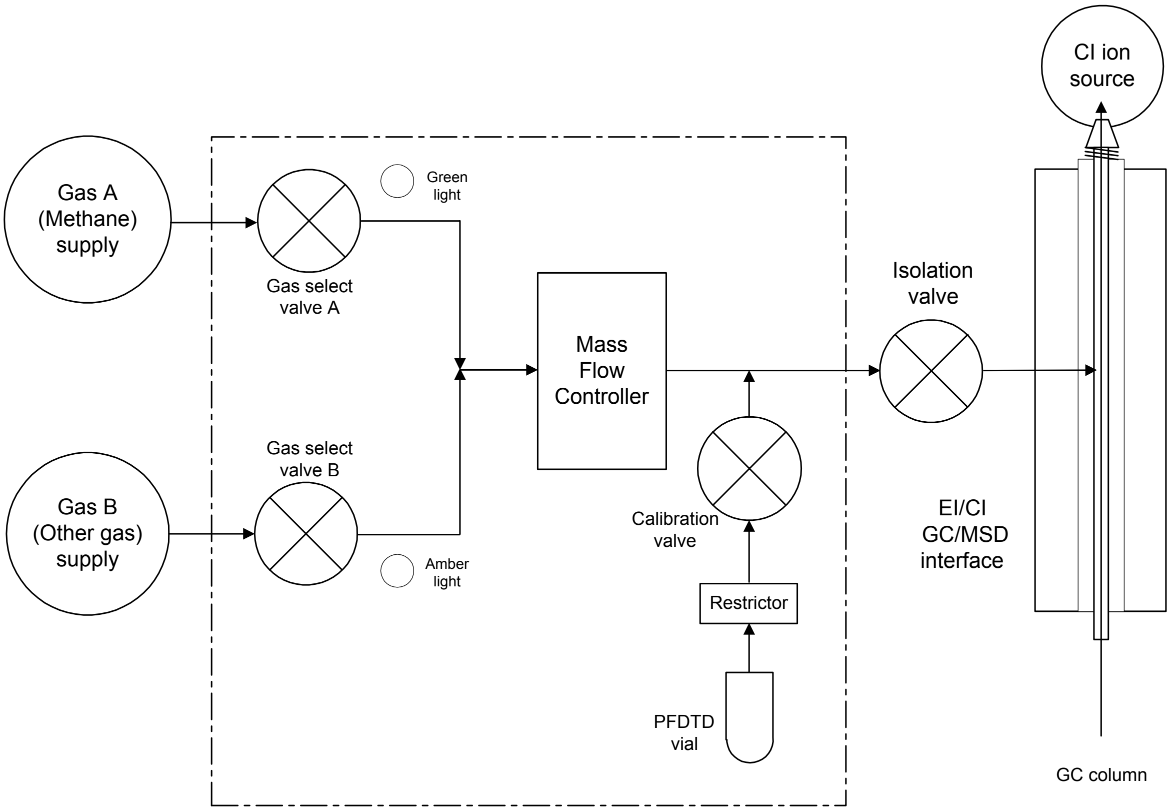
Reagent gas flow control module

- The flow control knob adjusts the gas flow. If the selected flow rate can not be achieved or maintained, the numbers in the flow control display will flash.
- The CI calibration valve is controlled by the ChemStation software, and opens automatically during CI autotune or manual tuning, allowing PFDTD to diffuse into the ion source.
- The isolation valve prevents contamination of the flow control module by atmosphere while the MSD is vented or by PFTBA during EI operation.



C A U T I O N Try to avoid tuning any more often than absolutely necessary to minimize PFDTD background and ion source contamination.

Reagent gas flow control module schematic



When you turn off one gas and turn on the other, the system sets a 6-minute delay with **Gas Off** and **Purge** both on to pump out the flow control module. The light for the selected reagent gas will flash, indicating the delay timer is active. Once the delay is finished, the **Purge** and **Gas Off** lights will turn off, and the light for the selected gas will stop flashing and stay on.

When the MSD is turned off, all valves are closed, and all lights are off. At startup, all valves are closed and all lights are off, except **Gas Off**.

The flow control board remembers the flow setting for each gas. When either gas is selected, the control board automatically sets the same flow that was used for that gas last time.

Flow control module state diagram:

Result	Gas A flows	Gas B flows	Purge with Gas A	Purge with Gas B	Pump out flow module	Standby, vented, or EI mode
Control panel lights (LEDs)						
Gas A (green)	On	Off	On	Off	Off	Off
Gas B (amber)	Off	On	Off	On	Off	Off
Purge (red)	Off	Off	On	On	On	Off
Gas Off (red)	Off	Off	Off	Off	On	On
Valve state						
Valve A	Open	Closed	Open	Closed	Closed	Closed
Valve B	Closed	Open	Closed	Open	Closed	Closed
MFC	On → setpoint	On → setpoint	On → 100%	On → 100%	On → 100%	Off (→0%)
Isolation valve	Open	Open	Open	Open	Open	Closed

10

Analyzer

This chapter describes the parts of the analyzer

Analyzer

The analyzer is the heart of the MSD

The analyzer ionizes the sample, filters the ions, and detects them. The sample components exiting the GC column flow into the ion source. In the ion source, the sample molecules are ionized and fragmented. The resulting positive ions are repelled from the ion source into the quadrupole mass filter. The mass filter allows selected ions to pass through the filter and strike the detector. The detector generates a signal current proportional to the number of ions striking it.

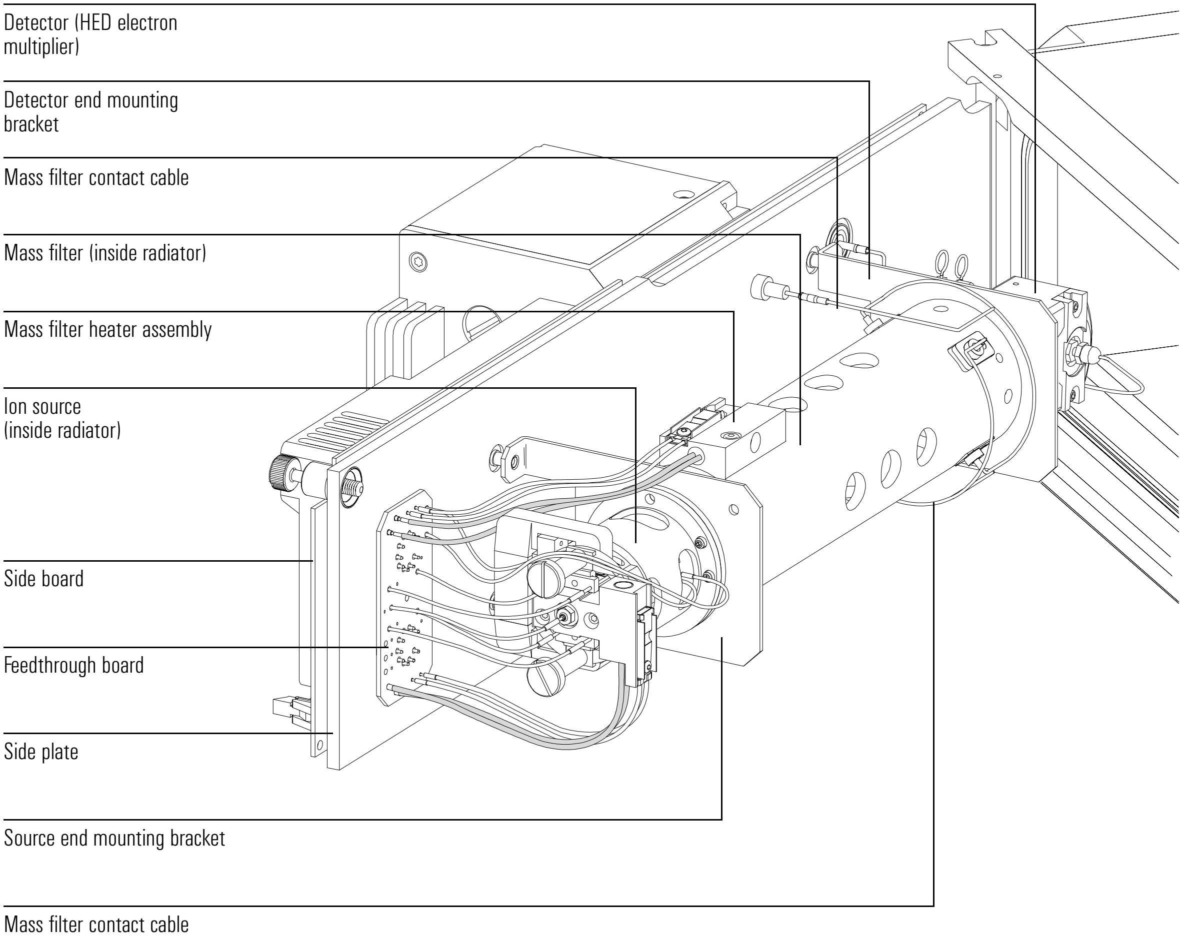
The analyzer is attached to the vacuum side of the side plate. The side plate is hinged to allow easy access to the analyzer. Both the ion source and the mass filter are independently heated. Each is mounted inside a radiator for correct heat distribution.

Each of the parts of the analyzer is discussed in the following material.

The analyzer has four basic components

The analyzer consists of the following components:

- Ion source
- Mass filter
- Detector
- Heaters and radiators



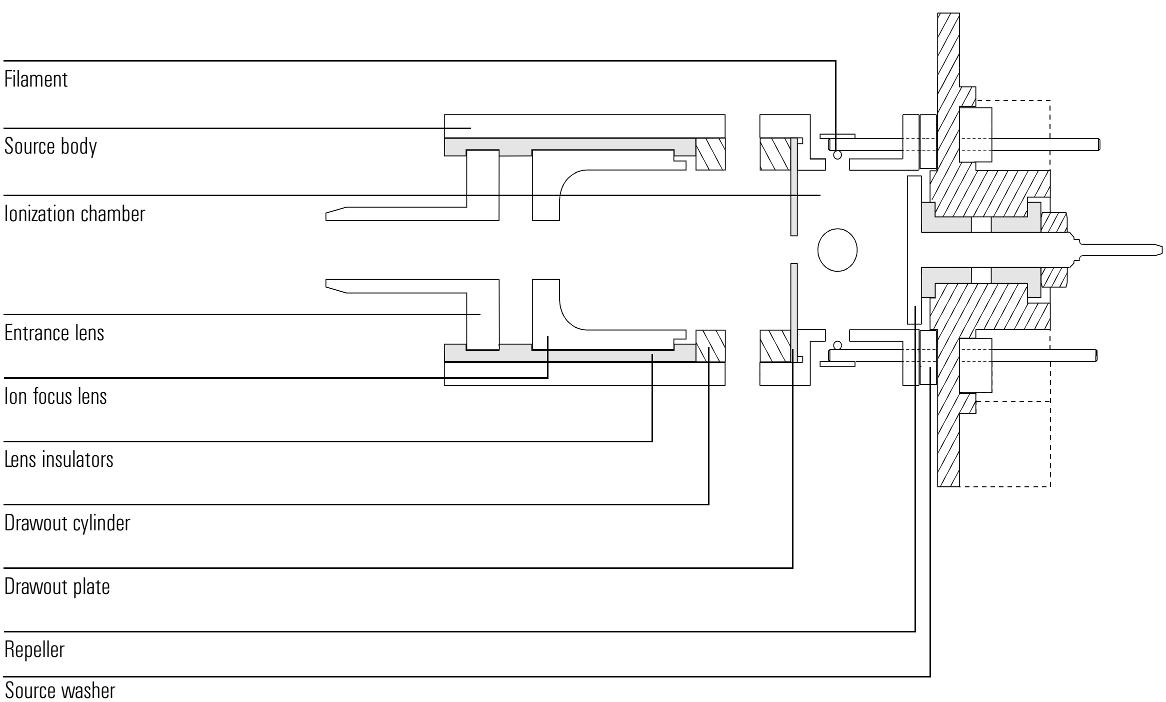
Detector (HED electron multiplier)
Detector end mounting bracket
Mass filter contact cable
Mass filter (inside radiator)
Mass filter heater assembly
Ion source (inside radiator)
Side board
Feedthrough board
Side plate
Source end mounting bracket
Mass filter contact cable

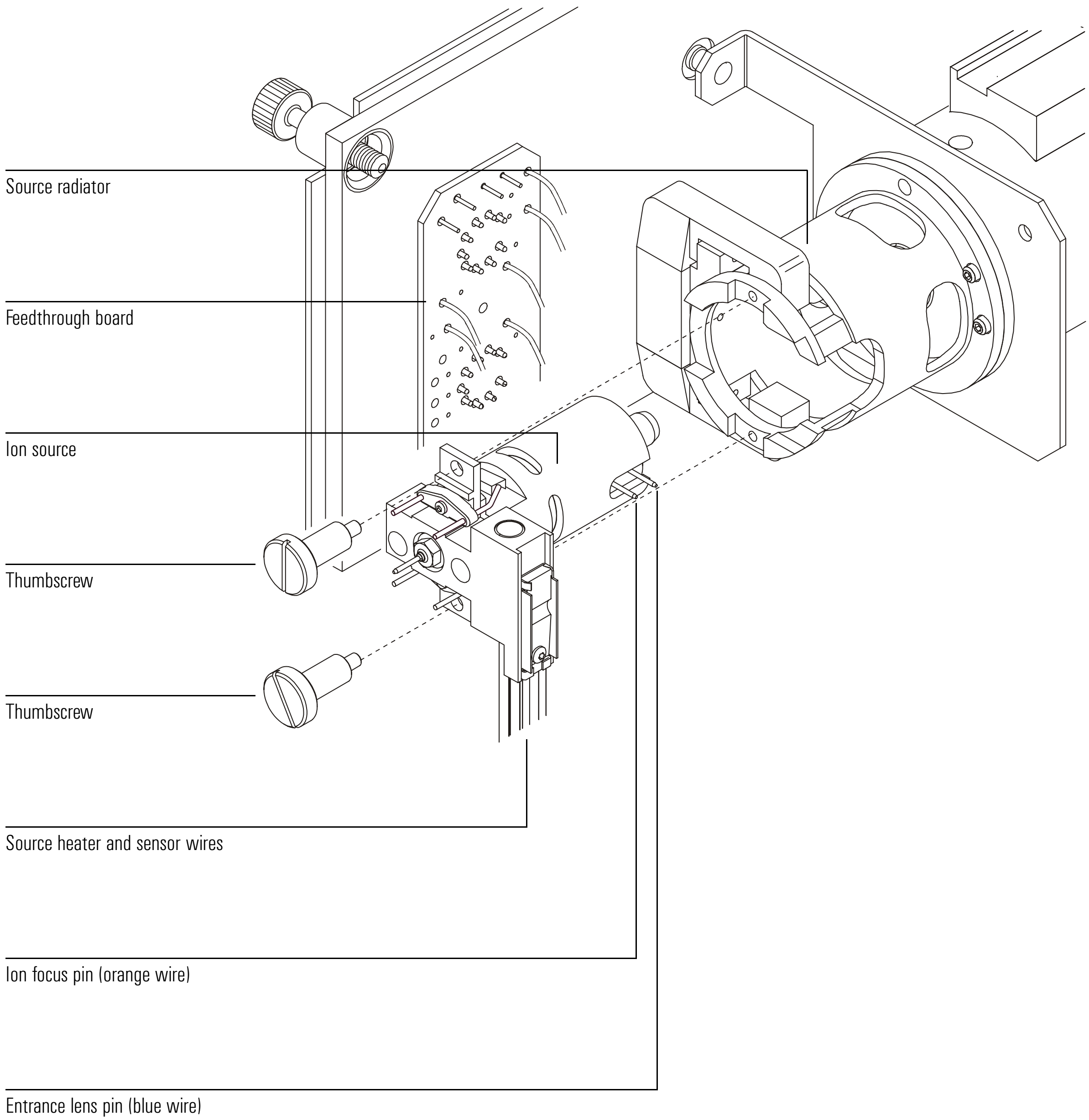
Ion source

The ion source operates by electron ionization (EI). The sample enters the ion source from the GC/MSD interface. Electrons emitted by a filament enter the ionization chamber, guided by a magnetic field. The high-energy electrons interact with the sample molecules, ionizing and fragmenting them. The positive voltage on the repeller pushes the positive ions into the lens stack, where they pass through several electrostatic lenses. These lenses concentrate the ions into a tight beam, which is directed into the mass filter.

Ion source body

The ion source body is a cylinder. Its cylindrical geometry ensures proper alignment of the lens stack. It holds the other parts of the ion source. With the repeller and the drawout plate, it forms the ionization chamber. The ionization chamber is the space where the ions are formed. Slots in the source body help the vacuum system to pump away carrier gas and un-ionized sample molecules or fragments.




Source radiator
Feedthrough board
Ion source
Thumbscrew
Thumbscrew
Source heater and sensor wires
Ion focus pin (orange wire)
Entrance lens pin (blue wire)

Filaments

Two filaments are located on opposite sides of the outside of the ion source. The ***active*** filament carries an adjustable ac emission current. The emission current heats the filament, causing it to emit electrons; these electrons ionize the sample molecules. In addition, ***both*** filaments have an adjustable dc bias voltage. The bias voltage determines the energy on the electrons, usually -70 eV.

The CI ion source has only one filament of a different design from the EI filaments. A“dummy” filament provides connections for the Filament 2 wires.

The filament is shut off automatically if there is a general instrument shutdown. There are three parameters that affect the filaments: filament selection (**Filament**), filament emission (**Emission**) current, and electron energy (**EIEnrgy**).

Filament selection

The filament selection parameter (**Filament**) allows you to select which filament in the ion source is active.

Sometimes, one filament will give better performance than the other. To select the better of the two filaments, run two autotunes, one with each filament. Use the filament that gives the best results.

Emission current

The filament emission current (**Emission)** is variable between 0 and -315 µA, but should be set to the software default for normal operation.

Electron energy

The electron energy (**EIEnrgy**) is the amount of energy on the ionizing electrons. The electron energy is determined by the bias voltage; -70 VDC bias on the filament causes emitted electrons to possess -70 eV (electron volts). This value is adjustable between -5 to -241 VDC, but for normal operation, set this parameter to 70.

Filament care

Like the filaments in incandescent light bulbs, the ion source filaments will eventually burn out. Certain practices will reduce the chance of early failure:

- If you have an optional 59864B Gauge Controller, use it to verify that the system has an adequate vacuum before turning on the analyzer, especially after any maintenance has been performed.
- If you are controlling your MSD from the Edit Parameters screen, always select **MSOff** before changing any of the filament parameters.
- When setting up data acquisition parameters, set the solvent delay so that the analyzer will ***not*** turn on while the solvent peak is eluting.
- When the software prompts **Override solvent delay?** at the beginning of a run, ***always*** select **NO**.
- Higher emission current will reduce filament life.
- Higher electron energy will reduce filament life.
- Leaving the filament on for short times (≤ 1 minute) during data acquisition will reduce filament life.

Magnet

The field created by the magnet directs the electrons emitted by the filament into and across the ionization chamber. The magnet assembly is a permanent magnet with a charge of 350 gauss in the center of the field.

Repeller

The Repeller forms one wall of the ionization chamber. A positive charge on the Repeller pushes positively-charged ions out of the source through a series of lenses. The Repeller voltage is also known as the ion energy, although the ions only receive about 20% of the Repeller energy. The Repeller voltage can be varied from 0 to +42.8 VDC. Some tune programs use a fixed Repeller voltage. Others ramp the repeller voltage to find the optimum setting.

- Setting Repeller voltage too low results in poor sensitivity and poor high mass response.
- Setting Repeller voltage too high results in precursors (poor mass filtering) and poor low mass resolution.

Drawout plate and cylinder

The drawout plate forms another wall of the ionization chamber. The ion beam passes through the hole in the drawout plate and into the drawout cylinder. The drawout cylinder is slotted. The slots correspond to slots in the source body. These slots allow carrier gas and un-ionized sample molecules or fragments to be pulled away by the vacuum system. The drawout plate and drawout cylinder are both at ground potential.

Ion focus

The voltage on the ion focus lens can be varied from 0 to -127 VDC. A typical voltage is between -70 and -90 VDC. In general:

- Increasing the ion focus voltage improves sensitivity at lower masses.
- Decreasing the ion focus voltage improves sensitivity at higher masses.
- Incorrect ion focus adjustment results in poor high mass response.

Entrance lens

The entrance lens is located at the entrance to the quadrupole mass filter. This lens minimizes the fringing fields of the quadrupole which discriminate against high-mass ions. There is a permanent +4.4 volt voltage added to the entrance lens. The total voltage applied to the entrance lens is the sum of the entrance lens offset and entrance lens gain and the +4.4 volt permanent offset.

$$\text{Entrance lens voltage} = +4.4\ \text{VDC} + \text{offset} + (\text{gain} \times \text{mass})$$

Entrance lens offset

The entrance lens offset (**EntOff**) controls the fixed voltage applied to the entrance lens. It can be varied from 0 to -64 VDC (-20 V is typical). Increasing the entrance lens offset generally increases the abundance of ions at low masses without substantially decreasing the abundance of high mass ions.

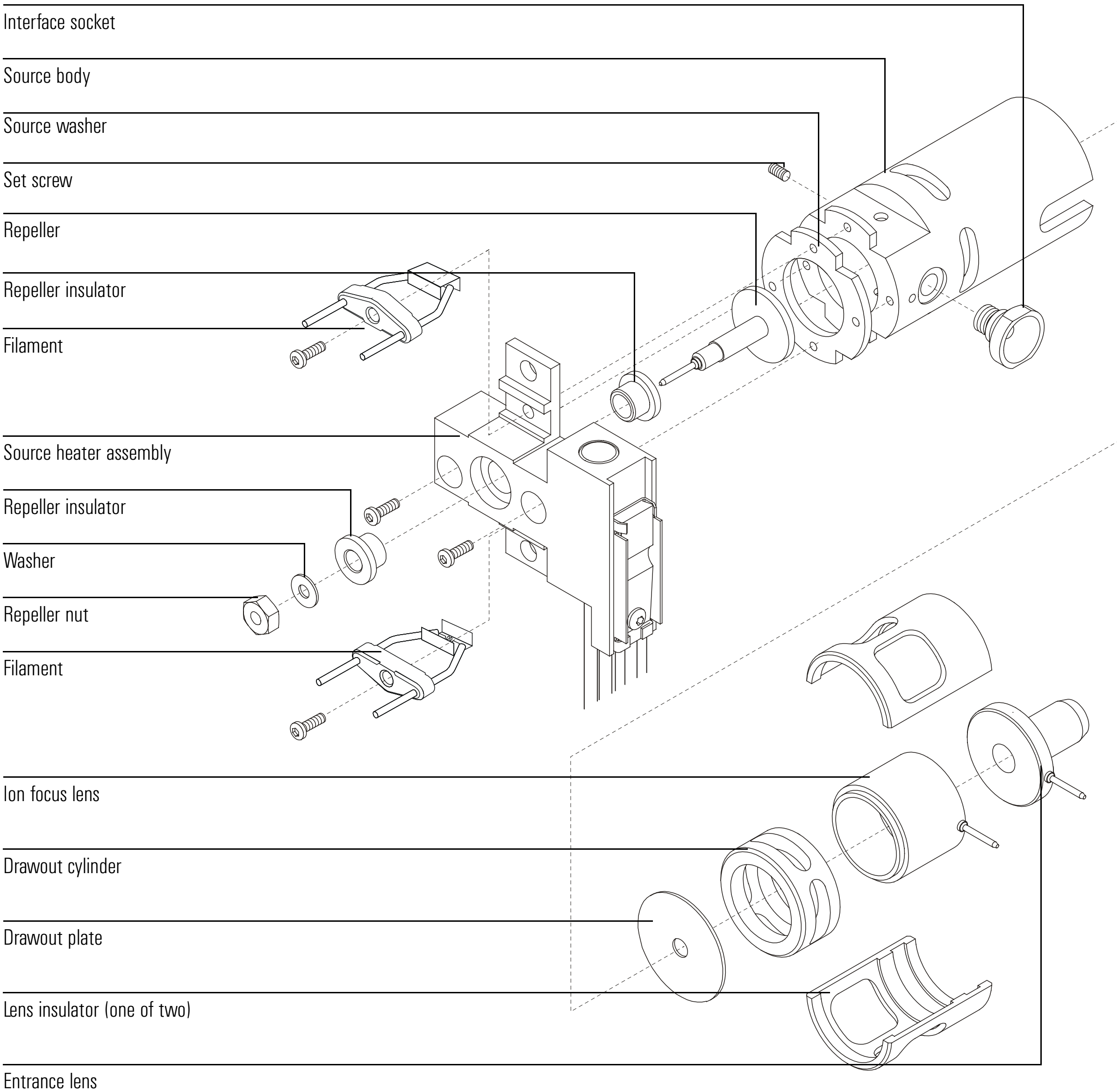
Entrance lens gain

Entrance lens gain (**EntLens**) controls the variable voltage applied to the entrance lens. It determines how many volts are applied for each amu. It can be varied from 0 to -128 mV/amu. A typical range is 0 to -40 mV/amu.

Source Washer

The source washer is a bonding barrier between the repeller block and the ion source body. Without the source washer, it is possible for the repeller block to bond to the source body (at high temperatures over time). The source washer, however, will bond to the repeller block over time; this is normal. When running the source at nominal temperatures (230°C), users may not see this bonding effect (between repeller block and source washer). Due to the risk of bending or damaging the source washer, use care when handling or cleaning (if necessary) this part.

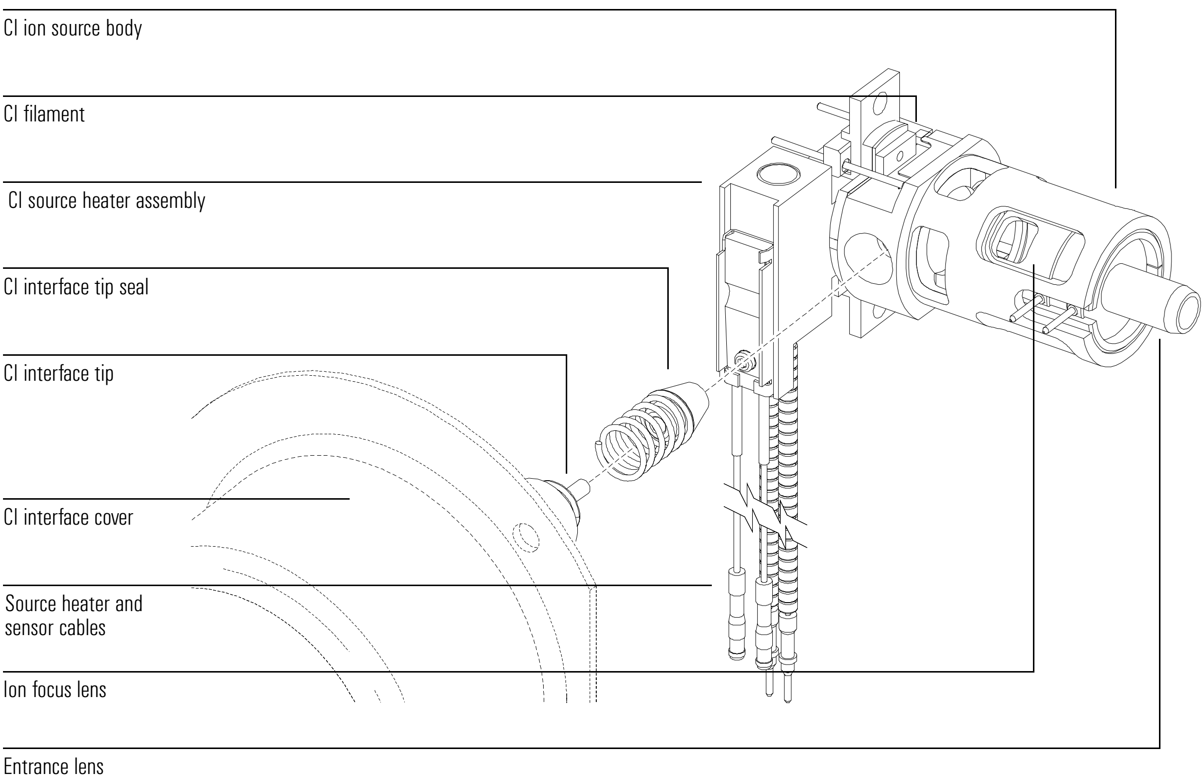
Ion source


Interface socket
Source body
Source washer
Set screw
Repeller
Repeller insulator
Filament
Source heater assembly
Repeller insulator
Washer
Repeller nut
Filament
Ion focus lens
Drawout cylinder
Drawout plate
Lens insulator (one of two)
Entrance lens

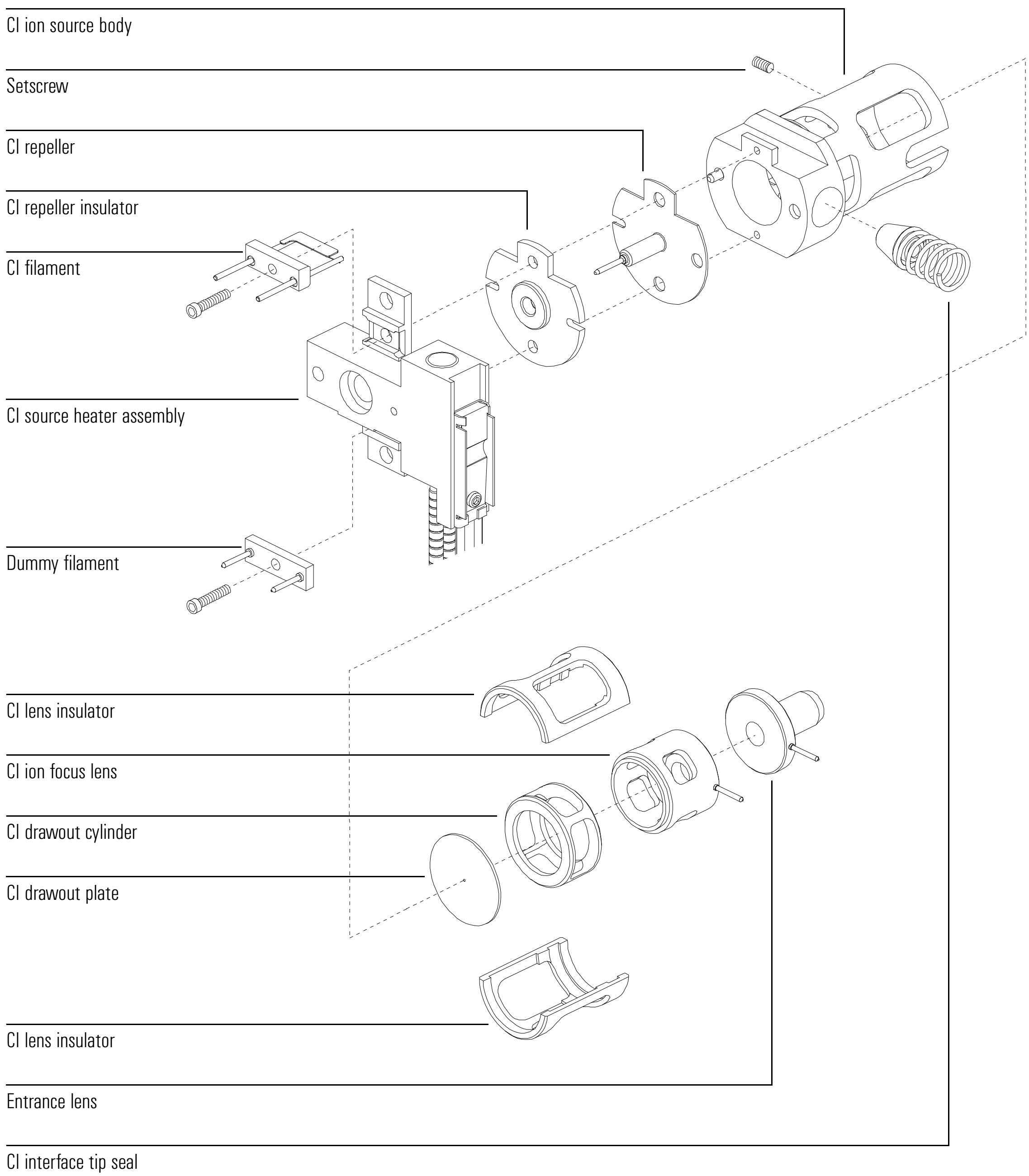
CI ion source

The CI ion source is similar to the EI source, but only has one part in common with the EI source — the entrance lens. The single CI filament has a straight wire and a reflector. There is a “dummy” filament to provide connections for the other wires.

The holes in the ion source (electron-entrance and ion-exit) are very small (0.5 mm), making it possible to pressurize the ionization chamber. Both the source body and the plate are at repeller potential, electrically isolated from the radiator and the CI interface tip. The seal for the interface tip ensures a leak-tight seal and electrical isolation between the CI interface and ion source.



CI ion source



Quadrupole mass filter

The mass filter separates ions according to their mass-to-charge ratio (*m/z*). At a given time, only ions of a selected mass-to-charge ratio can pass through the filter to the detector. The mass filter in the MSD is a quadrupole.

The quadrupole is a fused-silica (quartz) tube coated with a thin layer of gold. The four hyperbolic surfaces create the complex electric fields necessary for mass selection. Opposing segments are connected; adjacent segments are electrically isolated. One pair has positive voltages applied, the other negative.

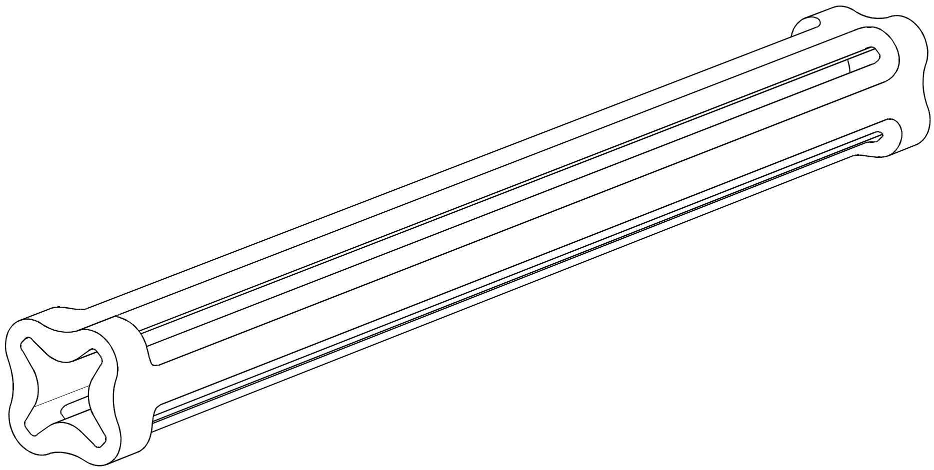
A combined direct current (dc) and radio frequency (RF) signal is applied to the two pairs of segments. The magnitude of the RF voltage determines the mass-to-charge ratio of the ions that pass through the mass filter and reach the detector. The ratio of dc-to-RF voltage determines the resolution (widths of the mass peaks). There are several parameters that control the dc and RF voltages. All these parameters are set by Autotune, but also can be manually adjusted in the Edit Parameters window:

- AMU gain (**AmuGain**)
- AMU offset (**AmuOffs**)
- 219 width (**Wid219**)
- DC polarity (**DC Pol**)
- Mass (axis) gain (**MassGain**)
- Mass (axis) offset (**MassOffs**)

AMU gain

AMU gain (**AmuGain**) affects the ratio of dc voltage to RF frequency on the mass filter. This controls the widths of the mass peaks.

- Higher gain yields narrower peaks.
- AMU gain affects peaks at high masses more than peaks at low masses.



AMU offset

AMU offset (**AmuOffs**) also affects the ratio of dc voltage to RF frequency on the mass filter.

- Higher offset yields narrower peaks.
- AMU offset generally affects peak widths equally at all masses.

219 width

m/z 219 is a prominent ion near the middle of the mass range of PFTBA. The width parameter (**Wid219**) makes small corrections to the *m/z* 219 peak width. Amu gain and amu offset must be readjusted after the 219 width is changed. If you are tuning with a compound other than PFTBA, there may not be an ion at *m/z* 219. In that case, set the 219 width to the last value found for it by Autotune or set it to 0.

DC polarity

The dc polarity (**DC Pol**) parameter selects the orientation of the direct current applied to the quadrupole mass filter. The dc polarity that works best for your MSD is determined at the factory. It is listed on the final test sheet accompanying your MSD. It is also listed on a label on the cover over the RF coils. This cover can be viewed by removing the upper MSD cover.

CAUTION

Using the non-preferred dc polarity may result in very poor performance. Always use the factory-specified polarity.

Mass (axis) gain

Mass gain (**MassGain**) controls the mass assignment, that is, assignment of a particular peak to the correct *m/z* value.

- A higher gain yields higher mass assignment.
- Mass gain affects peaks at high masses more than peaks at low masses.

Mass (axis) offset

Mass offset (**MassOffs**) also controls the mass assignment.

- A higher offset yields higher mass assignment.
- Mass offset generally affects peaks equally at all masses.

Quadrupole maintenance

The mass filter requires no periodic maintenance. It should not be removed from the radiator. If ***absolutely*** necessary (that is, if the only alternative is replacement), the quadrupole can be cleaned. Cleaning must be performed by Agilent Technologies service personnel.

CAUTION

Never put the quadrupole in an ultrasonic cleaner.

CAUTION

Never change the physical orientation of the quadrupole mass filter.

CAUTION

The fused-quartz quadrupole is fragile and will break if dropped or handled roughly.

CAUTION

The material in the cusps of the quadrupole is very hygroscopic. If exposed to water, the quadrupole must be dried ***very*** slowly to prevent damage.

Detector

The detector in the MSD analyzer is a high energy conversion dynode (HED) coupled to an electron multiplier (EM). The detector is located at the exit end of the quadrupole mass filter. It receives the ions that have passed through the mass filter. The detector generates an electronic signal proportional to the number of ions striking it. The detector has three main components: the detector focus lens, the high energy dynode, and the electron multiplier horn.

Detector focus lens

The detector focus lens directs the ion beam into the HED, which is located off axis. The voltage on the detector focus lens is fixed at -350 V.

High energy dynode

The high energy dynode (HED) operates at -10,000 volts for EI and PCI, and +10,000 volts for NCI. The HED is located off-axis from the center of the quadrupole mass filter to minimize signals due to photons, hot neutrals, and electrons coming from the ion source. When the ion beam hits the HED, electrons are emitted. These electrons are attracted to the more positive electron multiplier horn. Do not touch the ceramic insulator.

Electron multiplier horn

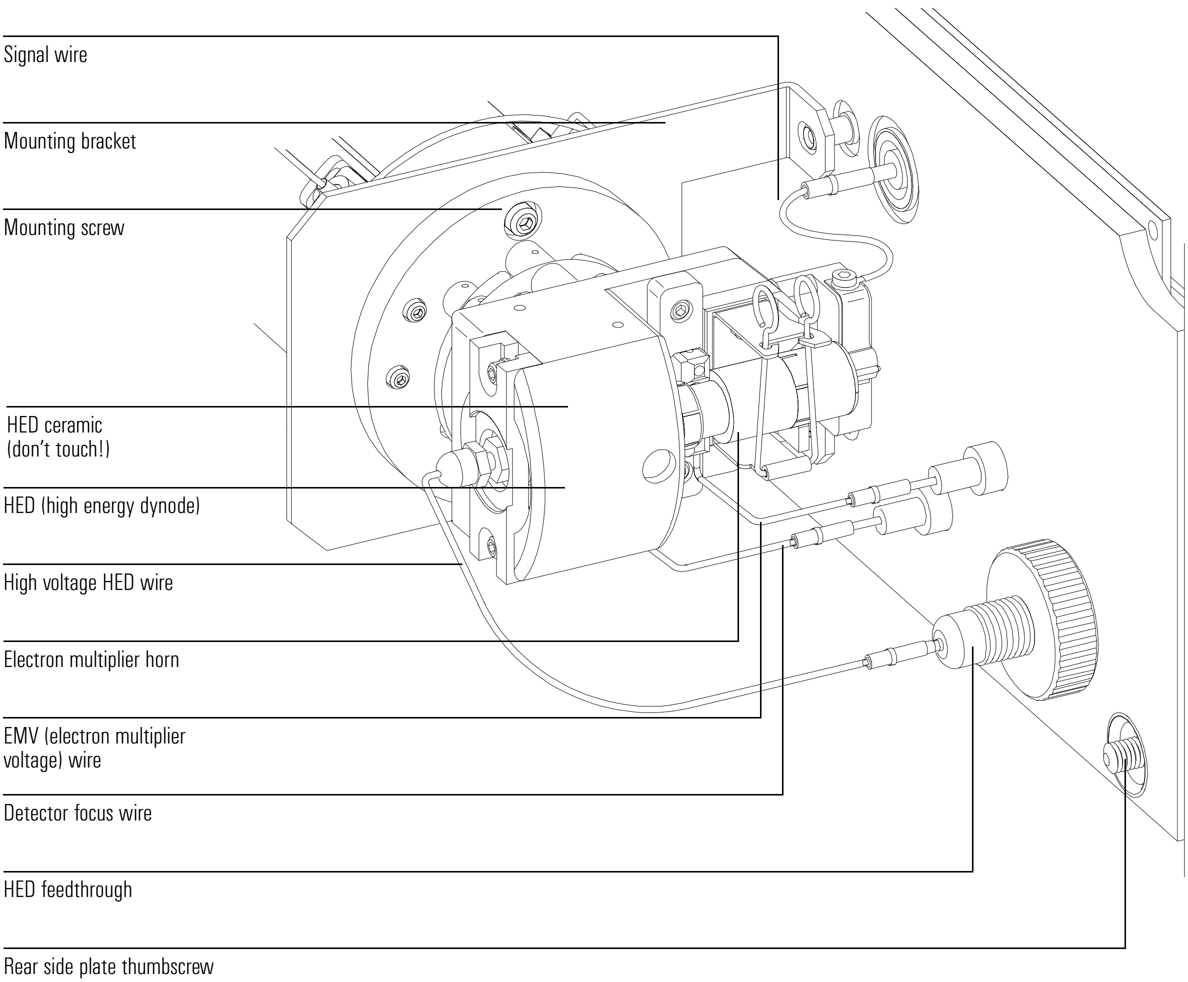
The electron multiplier horn carries a voltage of up to -3000 volts at its opening and 0 volts at the other end. The electrons emitted by the HED strike the EM horn and cascade through the horn, liberating more electrons as they go. At the far end of the horn, the current generated by the electrons is carried through a shielded cable outside the analyzer to the signal amplifier board.

The voltage applied to the electron multiplier horn determines the gain. The voltage is adjustable from 0 to -3000 VDC. Use the electron multiplier voltage found in autotune as a baseline for the electron multiplier voltage setting.

- To increase signal strength, increase the electron multiplier voltage.
- For concentrated samples where less signal strength is needed, decrease the electron multiplier voltage.

As the EM horn ages, the voltage (**EMVolts**) required by the electron multiplier increases over time. If the electron multiplier voltage must always be set at or near -3000 VDC to complete Autotune, with no other probable cause, it may need to be replaced. Check your tune charts for gradual degradation, which indicates wearing out. Select **View Tunes** from the Qualify menu in the Instrument Control view to see the tune charts. Sudden changes usually indicate a different type of problem.

See Also *Troubleshooting (5973 inert MSD)* in the online help for more information about symptoms that may indicate electron multiplier problems.



Analyzer heaters and radiators

The ion source and mass filter are housed in cylindrical aluminum tubes called radiators. The radiators control the distribution of heat in the analyzer. They also provide electrical shielding for analyzer components. The source heater and temperature sensor are mounted in the source heater block. The mass filter (quad) heater and temperature sensor are mounted on the mass filter radiator. Analyzer temperatures can be set and monitored from the MSD ChemStation.

In selecting the temperatures to use, consider the following:

- Higher temperatures help keep the analyzer clean longer.
- Higher ion source temperatures result in more fragmentation and therefore lower high-mass sensitivity.

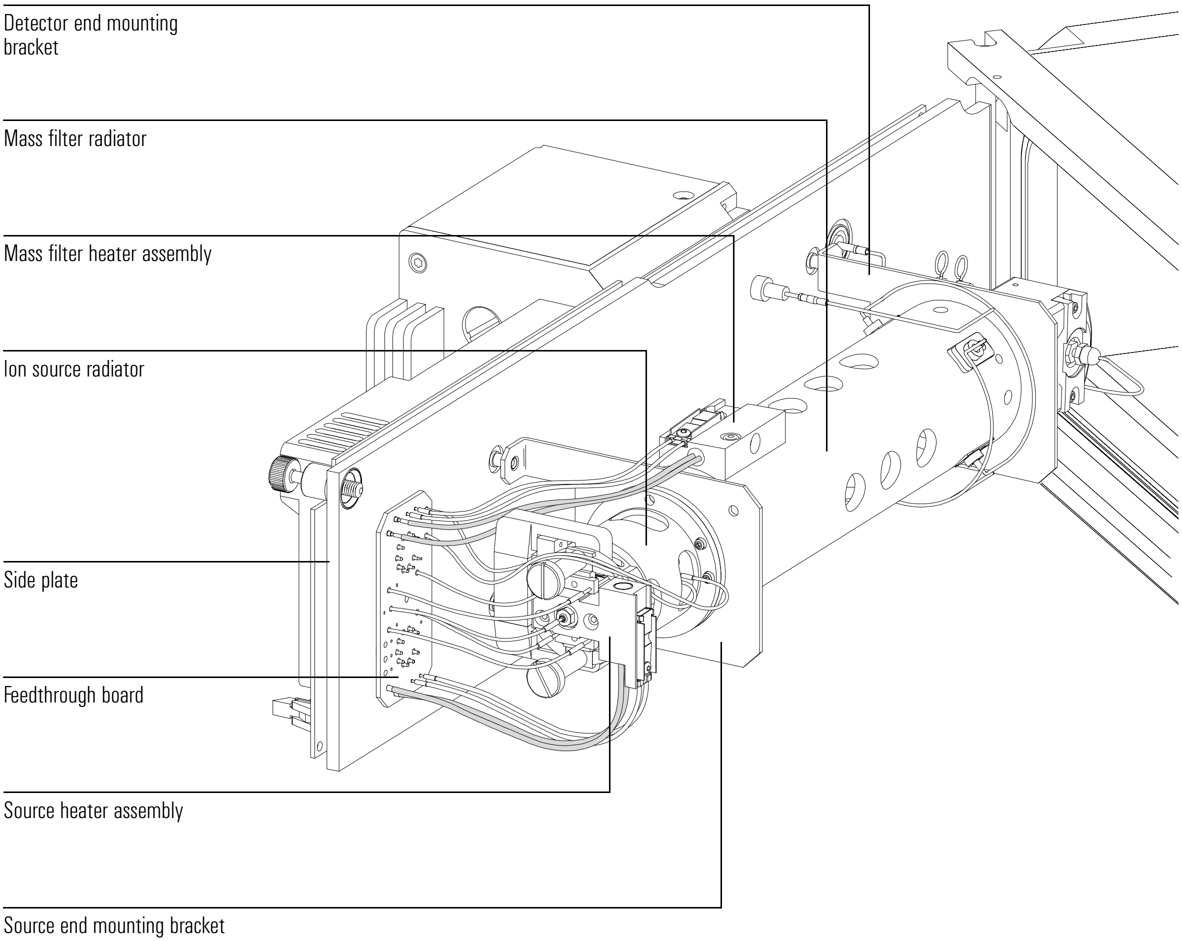
After pumpdown, it takes at least 2 hours for the analyzer to reach thermal equilibrium. Data acquired sooner may not be reproducible.

Recommended settings (for EI operation):

- Ion source 230°C
- Quadrupole 150°C

CAUTION Do not exceed 200°C on the quadrupole or 300°C on the ion source.

The GC/MSD interface, ion source, and mass filter (quad) heated zones interact. The analyzer heaters may not be able to accurately control temperatures if the setpoint for one zone is much lower than that of an adjacent zone.



Detector end mounting bracket
Mass filter radiator
Mass filter heater assembly
Ion source radiator
Side plate
Feedthrough board
Source heater assembly
Source end mounting bracket

11

Electronics

This chapter describes the MSD electronics

Electronics

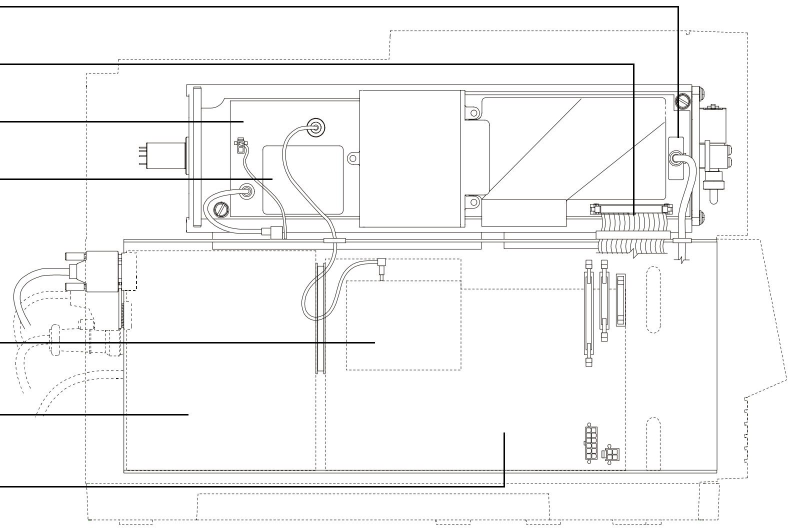
The following assemblies make up the MSD electronics:

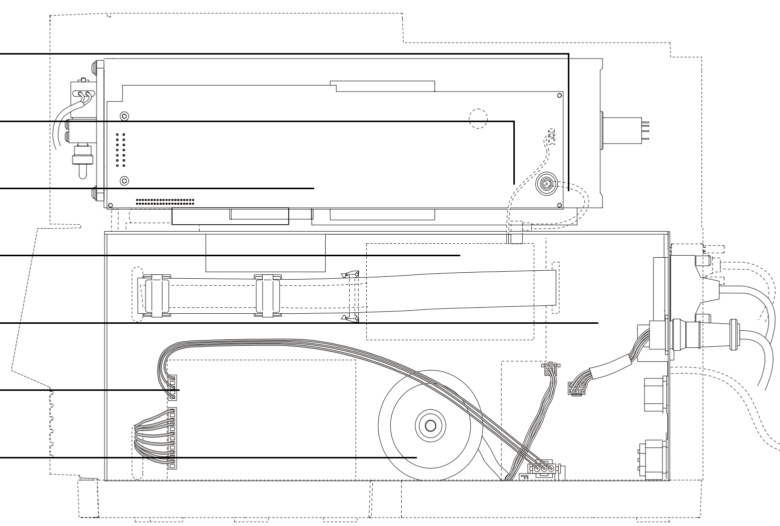
- Control panel and power switch
- Electronics module
- Main board
- Signal amplifier board
- LAN/MS control card
- AC board
- Turbo pump controller
- Low voltage (ac-dc) power supply
- High voltage (HED) power supply
- Toroid transformer assembly
- Back panel connectors
- Side board

Each is discussed in this chapter. Except for the *Back panel and connectors*, *Status display and power switch*, and *Interfacing to other devices* sections, most of this material is not essential for day-to-day operation of the MSD. It may be of interest to persons responsible for servicing the MSD.

WARNING

Dangerous voltages are present under the safety covers. Do not remove safety covers. Refer servicing to your Agilent Technologies service representative.


Source power cable
Side board control cable
Side board (front)
Signal cable
Signal amplifier board
MS control card
Main board


HED power cable
Detector focus cable
Side board (rear)
High voltage (HED) power supply
AC board
Low voltage power supply
Toroid transformer

Control panel and power switch

Control panel

The MSD has a control panel on the front of the instrument.

You can view MSD system status, and perform some control functions from the control panel.

Functions available through the control panel include

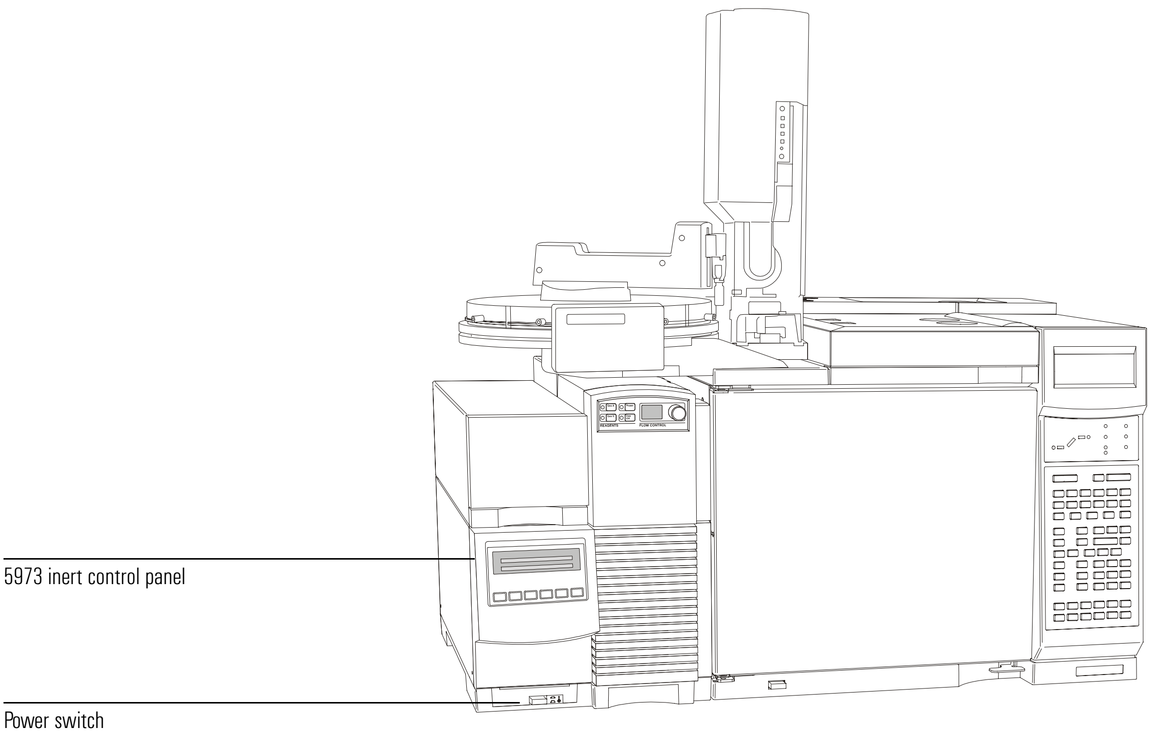
- Prepare to vent (cool analyzer and turn off high vacuum pump)
- Monitor MSD status
- Run autotune
- Run method
- Run sequence
- View and set analyzer temperatures

See also The *5973N and 5973 inert Mass Spectrometer Detector Local Control Panel (LCP) Quick Reference* (G2589-90072)

Power switch

The power switch is part of the electronics module, and is located on the lower left of the front of the MSD. It is used to switch the MSD and foreline pump on and off.

CAUTION Do not switch the MSD off unless it has completed the vent program. Incorrect shutdown can seriously damage the MSD.


5973 inert control panel
Power switch

Side board

The side board is mounted on the side plate. It performs the following functions:

- Provides the 1 MHz reference clock for the RF amplifier.
- Generates the RF component of the voltage applied to the quadrupole mass filter according to a signal from the main board. The amplitude of this voltage is proportional to the mass selected.
- Generates the dc component of the voltage applied to the quadrupole mass filter. The magnitude of this voltage is proportional to the RF voltage.
- Passes voltages generated on the main board, and the detector focus voltage from the HED power supply, to elements in the ion source and the detector.
- Generates and adjusts filament emission current and electron energy as controlled by the main board.
- Switches the filament power from one filament to the other.
- Monitors for RF faults and shuts down the analyzer one is detected.

Electronics module

Gloves, clean, lint-free

Most of the electronics in the MSD are contained in the electronics module. The whole electronics module can be replaced, if necessary, by your Agilent Technologies service representative.

The electronics module contains:

- Main board
- Signal amplifier board
- LAN/MS control card
- AC board (power distribution / vacuum control board)
- Low voltage (ac-dc) power supply
- High voltage (HED) power supply
- Toroid transformer assembly

Main board

The main board is mounted on the outer side of the electronics module. The main board performs the following functions:

- Receives and decodes digital instructions from the LAN/MS control card.
- Sends digital information to the LAN/MS control card.
- Generates voltages for the ion source lenses.
- Generates control signals for alternate filament selection, filament emission current, and electron energy. Generates control signals for quadrupole RF drive, dc/RF ratio adjustment, dc polarity selection, and all detector voltages.
- Performs analog-to-digital conversion for the coil DIP signal, ion source and mass filter temperature signals, and foreline pressure or turbo pump speed signal.
- Monitors the signals from the vacuum system and fans; and monitors the filament status, HV fault and RF fault signals from the side board. Activates the shutdown line when the analyzer electronics must be disabled.
- Generates the control signals (on and off) used by the ac board for the high vacuum pump and calibration valve.
- Generates ± 280 VDC (nominal) power for main board lens amplifiers and side board dc amplifiers.
- Supplies and controls the power for the ion source and quadrupole (mass filter) heaters.
- Provides 24 VDC power for the cooling fans.

Signal amplifier board

The signal amplifier board amplifies the output of the detector. The signal amplifier circuit produces an output voltage of 0 to 10 volts dc, proportional to the logarithm of the input current of 3 picoamps to 50 microamps.

An analog-to-digital converter converts the amplifier output voltage to digital information. The LAN/MS control card “unlogs” the data into abundance counts proportional to the detector signal current.

AC board

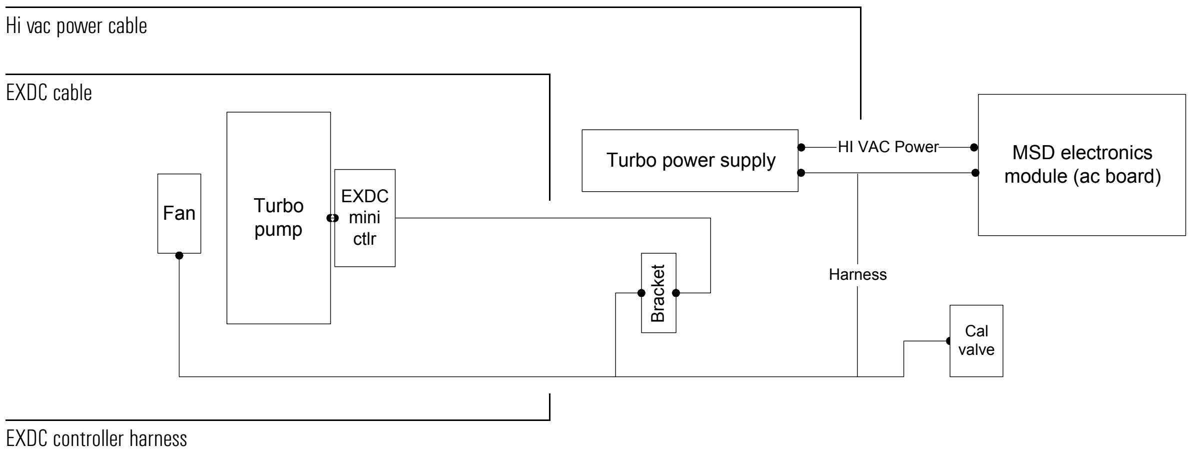
The ac board is mounted on the opposite side of the electronics panel from the LAN/MSD control card. The ac board is also sometimes called the power distribution / vacuum control board. The ac board performs the following functions:

- Provides input voltage transparency for the MSD.
- Distributes ac line power to the ac/dc power supply, the foreline pump, and the turbo pump controller.
- Turns the calibration valve on or off as directed to by the main board.
- Provides the voltage for the calibration valve.
- Provides logic interface to turbo controller.
- Controls the diffusion pump (not applicable for the 5973 inert MSDs):
  - Controls the foreline gauge.
  - Turns on the diffusion pump once the foreline pressure is low enough, as directed by the main board.
  - Regulates the ac power to the diffusion pump heater.
  - Turns off the diffusion pump if the foreline pressure is too high or if the diffusion pump is too hot.
- Passes the foreline pressure signal from the foreline gauge or turbo pump speed and other vacuum status information to the main board.
- Turns off the foreline pump in case of a problem with pumpdown.

Turbo pump control

The ac board sends control signals to, and receives turbo pump status information from, the turbo pump controller. The turbo pump controller provides power to the turbomolecular pump and regulates pump speed. If the pump fails to reach 80% speed within 7 minutes after beginning pumpdown, or if the speed drops below 50% during operation, the controller shuts off the turbo pump and the ac board shuts off the foreline pump.

Your MSD is equipped with turbo pump power supply and a separate EXDC mini-controller.



Turbo pump control with separate power supply and EXDC mini controller

Pumpdown failure shutdown

The ac board will shut down both the high vacuum and the foreline pump if the system fails to pump down correctly. One condition that triggers shutdown is:

- Turbo pump MSD: turbo pump speed below 80% after 7 minutes

This is usually because of a ***large*** air leak: either the sideplate has not sealed correctly or the vent valve is still open. This feature helps prevent the foreline pump from sucking air through the system, which can damage the analyzer and pump.

To correct the problem, power cycle the MSD, and troubleshoot. You have seven minutes to find and correct the air leak before the system shuts down again. Be sure to press on the sideplate when turning on the MSD power to ensure a good seal.

LAN/MSD control card

The LAN/MS control card is located to the left of the main board on the electronics panel. The LAN/MS control card has two main functions:

- Providing a communication interface between the MSD and the data system.
- Providing real-time control of the MSD, freeing the data system for other tasks.

Functional areas of the LAN/MS control card include:

- Instrument controller
- Data processor
- Main processor
- Serial communication processor
- Network communication controller
- Remote start processor
- Random access memory (RAM)
- Status LEDs
- Control panel firmware

LEDs on the LAN/MSD control card are visible on the rear panel. The upper two LEDs indicate network communication.

The two bottom LEDs are the power (good, digital 5V) and the “heartbeat” indicator. The flashing heartbeat LED indicates that the operating system of the MSD is functioning. In case of catastrophic loss of flash memory, the heartbeat flashes in an “SOS” pattern.

Power supplies

Low voltage (ac-dc) power supply

The low voltage power supply is mounted next to the toroid transformer in the electronics module. A universal input power supply, it converts ac line voltage into the dc voltages used by the rest of the electronics. The power supply generates the following dc voltages:

- +24 V (nominal)
- +15 V (nominal)
- -15 V (nominal)
- +5 V (nominal)

High voltage (HED) power supply

The high voltage power supply provides the -10,000 volts dc for the high energy dynode (HED) in the detector for the EI MSD. The EI/PCI/NCI MSD requires a bipolar power supply that can also provide +10,000 volts for NCI operation. The HED power supply also provides the 350 volts dc for the detector focus lens. Due to the high impedance of this circuit, measuring the detector focus voltage (350 volts) with a handheld voltmeter will give a typical reading of 90 to 100 volts where the polarity matches that of the HED voltage.

Toroid transformer

The toroid transformer is mounted next to the ac board. It provides 24 VAC for the mass filter and source heater circuits. The input wires take 120 VAC or 200 – 260 VAC from the ac board. The AC board samples the line voltage and uses a relay to appropriately strap the toroid primary. The output wires connect to the main board.

Back panel and connectors

The back panel contains several connectors, the primary fuses, several status LEDs. Most of these components are part of the ac board or the LAN/MS control card, and extend through the back panel.

*High vacuum control (***HIVAC SIGNAL***) connector*

The high vacuum signal connector is on the ac board. See Turbo pump control, 316.

*High vacuum power (***HIVAC POWER***) connector*

The high vacuum power connector carries power for the turbo controller from the ac board.

Primary fuses

The primary fuses limit current into the MSD in case of a short circuit in the fore-line pump. The primary fuses are on the ac board.

Power cord receptacle

The ac power cord brings in all electrical power for the MSD. The power cord can be detached from the MSD.

Foreline pump power cord receptacle

The foreline pump power cord receptacle provides ac power for the foreline pump. If the power switch is off, no power is supplied to the foreline pump.

Remote start connector

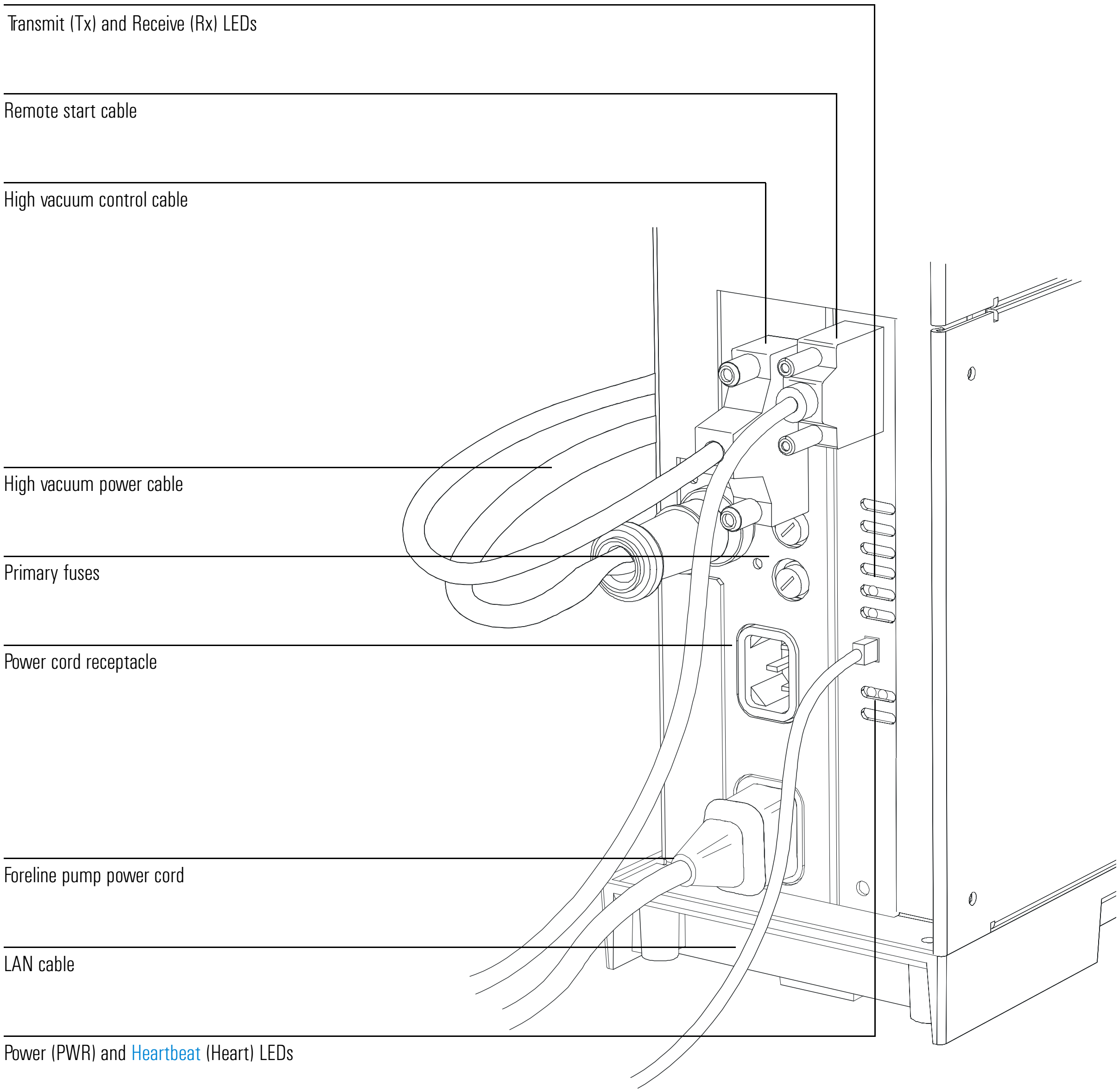
The remote start connector is the external connector for the remote start circuitry on the LAN/MS control card. It receives remote start signals from the GC.

LAN (I/O) connector

The LAN cable from the data system is connected to the I/O LAN connector. This cable carries all data communication between the PC and the MSD.

LAN/MSD control card LEDs

The upper two LEDs indicate network communication. The two bottom LEDs are the power and the "heartbeat" indicator.


Transmit (Tx) and Receive (Rx) LEDs
Remote start cable
High vacuum control cable
High vacuum power cable
Primary fuses
Power cord receptacle
Foreline pump power cord
LAN cable
Power (PWR) and Heartbeat (Heart) LEDs

Interfacing to external devices

Remote control processor

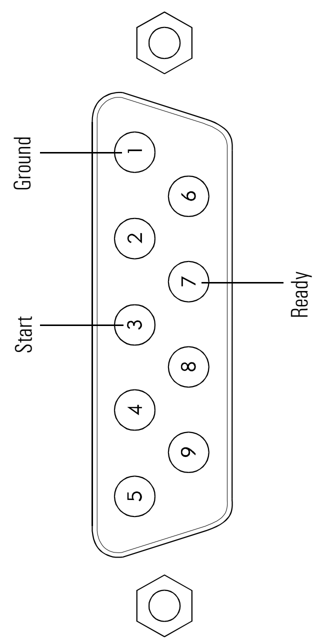
The remote control processor on the LAN/MS control card synchronizes start-run signals with GCs and other devices. The functions of the remote control processor are extended to the remote start (**Remote)** connector on the back panel of the MSD. The remote start cable connects the GC and the MSD.

Remote start signals

It is often necessary to communicate with external devices (for example, a purge-and-trap) during a run. Typically, these communications are:

- Requests to send a system ready signal
- Receive a start run signal from an external device
- Program the timing of events during a run

MSD remote start connector



System ready

When interfacing to an external device, it is often desirable to send a system ready signal to the device. In the case of a multi-sample Tekmar purge-and-trap, each sample is purged onto a trap where it waits for a ready signal. On receipt of the ready signal, the desorbtion cycle begins. When a specific temperature has been reached, the purge-and-trap closes a contact to indicate the run has started.

The ready pin on the remote start connector on the GC is held low at all times except when the GC, MSD, and data system are all ready. On system ready, a logic high of 5 VDC is present between that pin and any ground. This same high can be detected between the ready and ground pins on the remote start connector on the MSD.

Start run input

The best way to generate a start run signal is to use the remote start connector on the GC. Since remote start cables are made for most common devices, this is often the simplest way. A general-purpose remote start cable (05890-61080), which is also available, terminates in spade lugs. Care must be taken to ensure that the system is actually ready before the start run signal is sent.

If necessary, the remote start connector on the back of the MSD can be used to send the start run signal. A contact closure between the start and ground pins will start the run if the system is ready.

12

Parts

This chapter lists parts that can be ordered for your MSD

General Parts

This chapter lists parts that can be ordered for use in maintaining your 5973 inert MSD. It includes most of the parts or assemblies in the MSDs. This chapter is organized so that related parts are grouped together.

Some of the parts listed are not user-replaceable. They are listed here for the convenience of Agilent Technologies service representatives.

To order parts

To order parts for your MSD, address the order or inquiry to your local Agilent Technologies office. Supply them with the following information:

- Model and serial number of your MSD, located on a label on the lower left side near the front of the instrument. See page 19.
- Part number(s) of the part(s) needed
- Quantity of each part needed

Some parts are available as rebuilt assemblies

Rebuilt assemblies pass all the same tests and meet all the same specifications as new parts. Rebuilt assemblies can be identified by their part numbers. The first two digits of the last five digits of the part number are 69 or 89 (i.e., XXXXX-69XXX). Rebuilt assemblies are available on an exchange-only basis. When you return the original part to Agilent Technologies (after you receive the rebuilt assembly) you will receive a credit.

If you cannot find a part you need

If you need a part that is not listed in this chapter, check the Agilent Technologies Analytical Supplies Catalog or the on-line catalogue on the worldwide web at <**http://www.agilent.com**>. If you still cannot find it, contact your Agilent Technologies service representative or your Agilent Technologies office.

Electronics

The printed circuit boards in the MSD are available only as complete assemblies. Individual electronic components are not available. This section contains the following parts: cables, fuses, printed circuit boards (electronic assemblies).

Table 8 **External cables**

Description	Part number
Remote start cable	G1530-60930
LAN cable (shielded)	8121-0008
Power cord, Australia, China	8120-1369
Power cord, Denmark	8120-2956
Power cord, Europe	8120-1689
Power cord, India / South Africa	8120-4211
Power cord, Japan (200 V)	G2025-60189
Power cord, Switzerland	8120-2104
Power cord, UK, Hong Kong, Singapore	8120-1351
Power cord, US	8120-6825
Triode gauge cable (triode gauge tube to gauge controller)	8120-6573

Table 9 **Fuses and power switch**

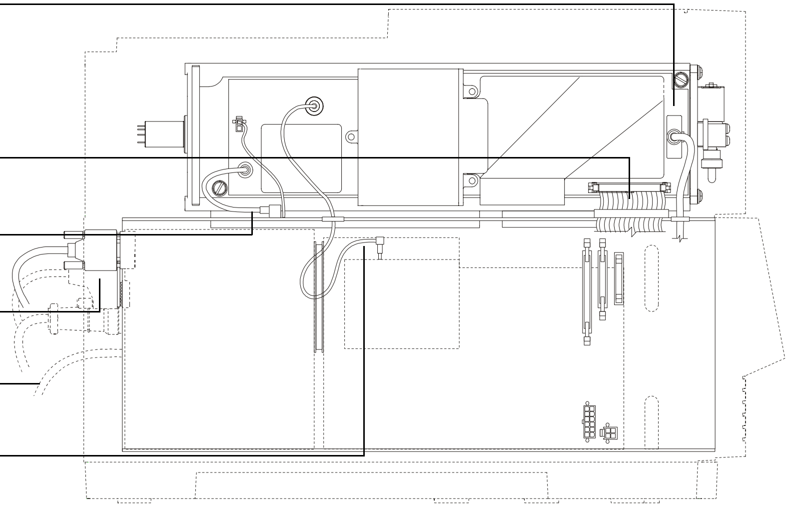
Description	Part number
Fuse 4A, 250 V, fast-acting, low breaking (ac board and main board)[1]	2110-0734
Fuse, 8A, 250 V, time-lag, high breaking capacity (primary fuses)	2110-0969
Power button[1]	5041-1203
Power switch extender rod[1] and switch adapter[1]	G1099-40007

1 Not a user-replaceable part. Refer service to your Agilent Technologies service representative.

Table 10 **Internal Cables**

Description	Part number
AC board control cable (AC board to main board)[1]	G1099-60422
Chassis ground wire[1]	G1099-60433
Control panel ribbon cable	G2589-60030
Electronics module fan cable[1]	G1099-60560
Fan (high vacuum) cable	G1099-60561
Feedthrough board (inside analyzer chamber)	G1099-60425
HED control cable	G1099-60430
HED power cable	G1099-60431
High vacuum power extender cable (AC board to back panel)[1]	G1099-60436
Low voltage power supply input cable (AC board to LV PS)[1]	G1099-60426
Low voltage power supply output cable (LV PS to main board)[1]	G1099-60427
Mass filter contact cable kit (inside analyzer chamber)	G1099-60130
Side board control (ribbon) cable (main board to side board)[1]	G1099-60410
Signal cable (signal feedthrough on side plate to signal amplifier board)[1]	G1099-60416
Source power cable (main board to side board)[1]	G1099-60428
Turbo pump control cable (back panel to turbo controller)	G1099-60438
Turbo pump power cable (back panel to turbo controller)	G1099-60435

1 Not a user-replaceable part. Refer service to your Agilent Technologies service representative.



Source power cable
G1099-60428

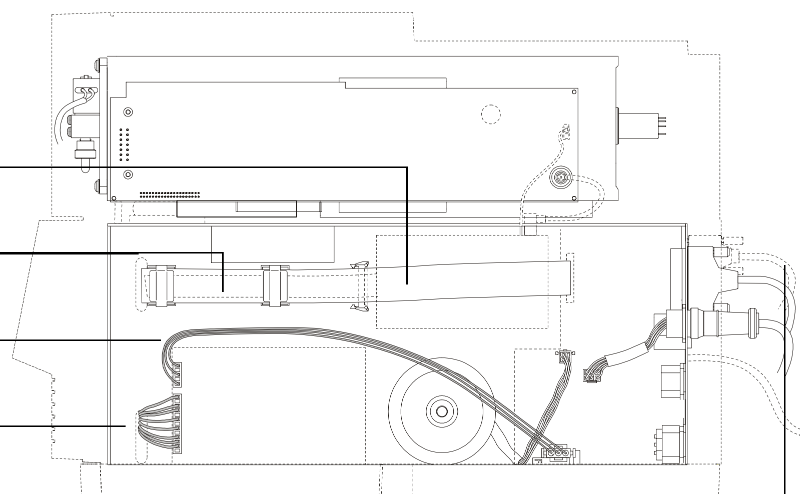
Side board control cable
G1099-60410

HED power supply output cable
G1099-60431

Remote start cable
G1530-60930

LAN cable, shielded
8121-0008

Signal cable
G1099-60416



AC board control cable
G1099-60422

HED control cable
G1099-60430

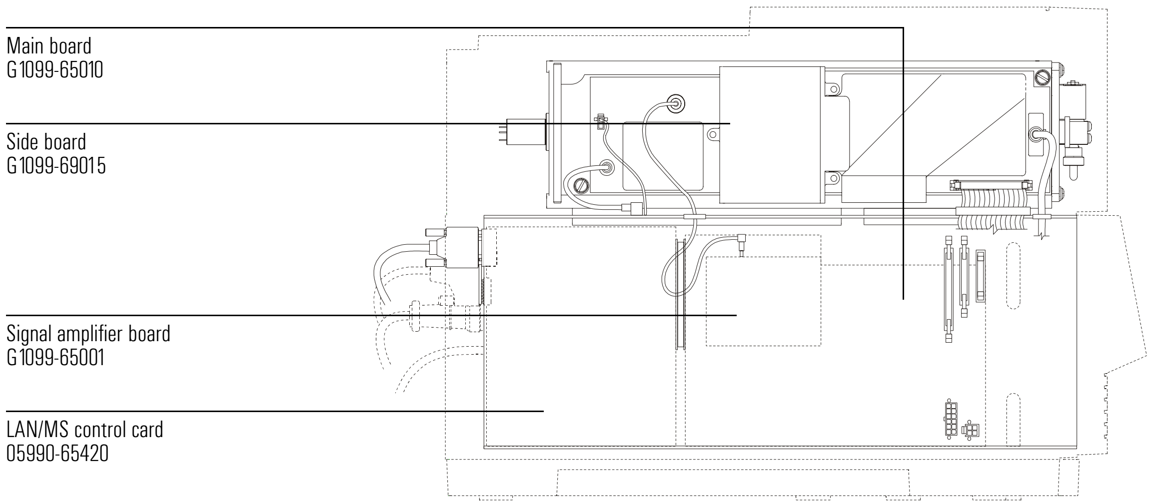
Low voltage PS input cable
G1099-60426

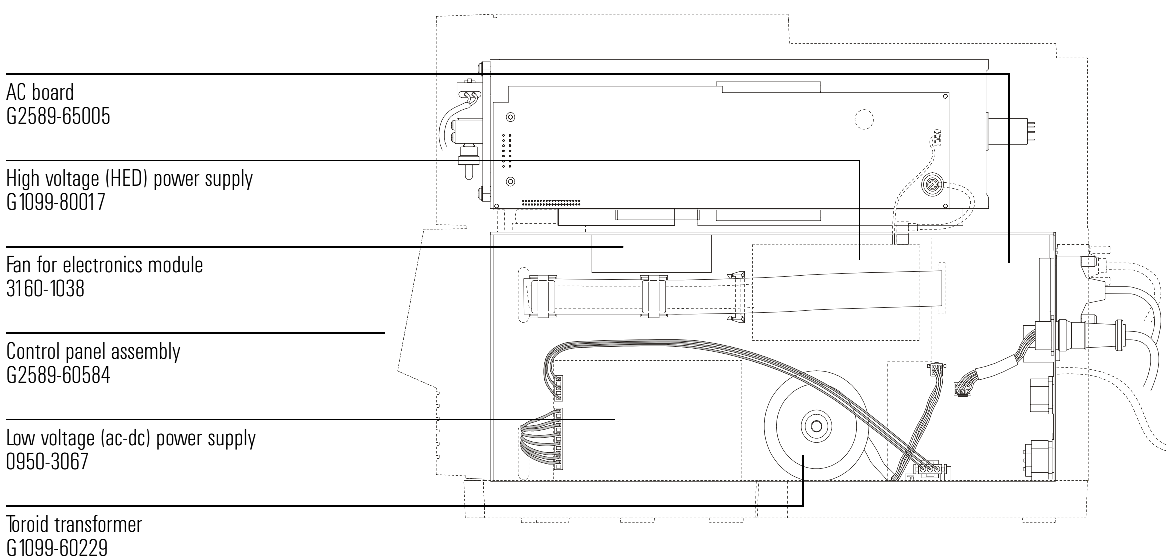
Low voltage PS output cable
G1099-60427

Table 11 **Printed circuit boards**[1]

Description	Part number
ac board	G2589-65005
fan for electronics module[1]	3160-1038
HED power supply	G1099-80017
bipolar HED power supply (EI/PCI/NCI MSDs only)	G1099-80018
LAN/MS control card–SC3	05990-65420
80–pin SIMM	05990-80040
72–pin SIMM	E4401-60289
low voltage (ac-dc) power supply	0950-3067
main board	G1099-65010
signal amplifier board	G1099-65001
toroid transformer	G1099-60229
Control panel assembly	G2589-60584
Side board	G1099-69015

1 None of the parts in this table are user-replaceable parts. Refer service to your Agilent Technologies service representative.


Main board
G1099-65010
Side board
G1099-69015
Signal amplifier board
G1099-65001
LAN/MS control card
05990-65420


AC board
G2589-65005
High voltage (HED) power supply
G1099-80017
Fan for electronics module
3160-1038
Control panel assembly
G2589-60584
Low voltage (ac-dc) power supply
0950-3067
Toroid transformer
G1099-60229

Vacuum system

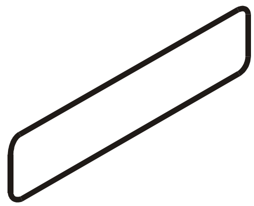
This section lists replacement parts available for the vacuum system. It includes: clamps, O-rings and seals, foreline pump and related components, diffusion pump vacuum system components, and turbomolecular pump vacuum system components.

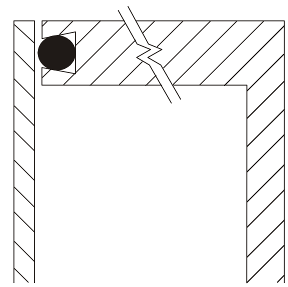
Table 12 **O-rings and seals**

Description	Part number
Calibration valve O-ring (1/4-inch)	0905-1217
End plate O-ring (for front and rear end plates)	0905-1441
GC/MSD interface O-ring	0905-1405
HED feedthrough	G1099-80012
HED feedthrough O-ring	0905-0490
KF10/16 seal (foreline pump inlet)	0905-1463
KF25 O-ring assembly (turbo pump outlet)	0100-1551
KF elbow adapter for standard turbo pump outlet	G2589-20041
O-ring, for standard turbo pump inlet[1]	0905-1443
Seal, performance turbo pump inlet[1]	0100-1879
Side plate O-ring	0905-1442
Triode gauge tube O-ring	0905-1070
Vent valve O-ring (1/4-inch)	0905-1217
Drain plug for the foreline pump	0100-2041
O-ring for the foreline pump drain plug	0905-1515

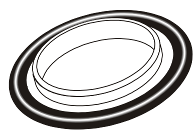
1 The turbo pump and its seal are not user-replaceable parts. Refer service to your Agilent Technologies service representative.

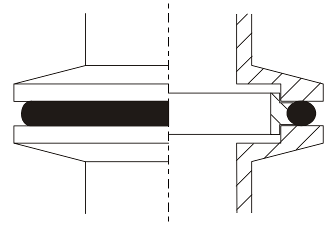
Face seal



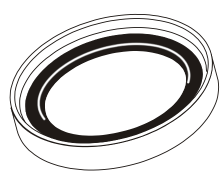


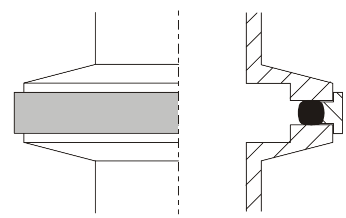
KF seal with internal centering ring



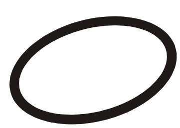


KF seal with external centering ring





Compression seal



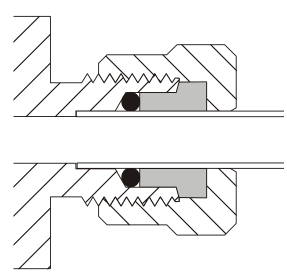


Table 13 **Foreline pump and related parts**

Description	Part number
Foreline hose assembly (hose and internal spring)	05971-60119
Foreline pump	
120 V	G1099-89023
230 V	G1099-89024
Foreline pump inlet seal (KF10/16)	0905-1463
Hose clamp	1400-1234
KF10/16 clamp (foreline inlet)	0100-1397
KF16 hose adapter	G1099-20531
KF25 clamp (turbo pump end of foreline hose – not shown)	0100-0549
KF25 hose adapter (turbo pump end of foreline hose – not shown)	G1099-20532
Exhaust oil mist trap (not shown)[1]	G1099-80037
Oil drip tray	G1099-00015

[1] Do ***not*** use an exhaust oil mist trap if you are analyzing hazardous samples, or if you are using hazardous carrier gas, or if you are running CI. Plumb the pump exhaust to a fume (exhaust) hood.

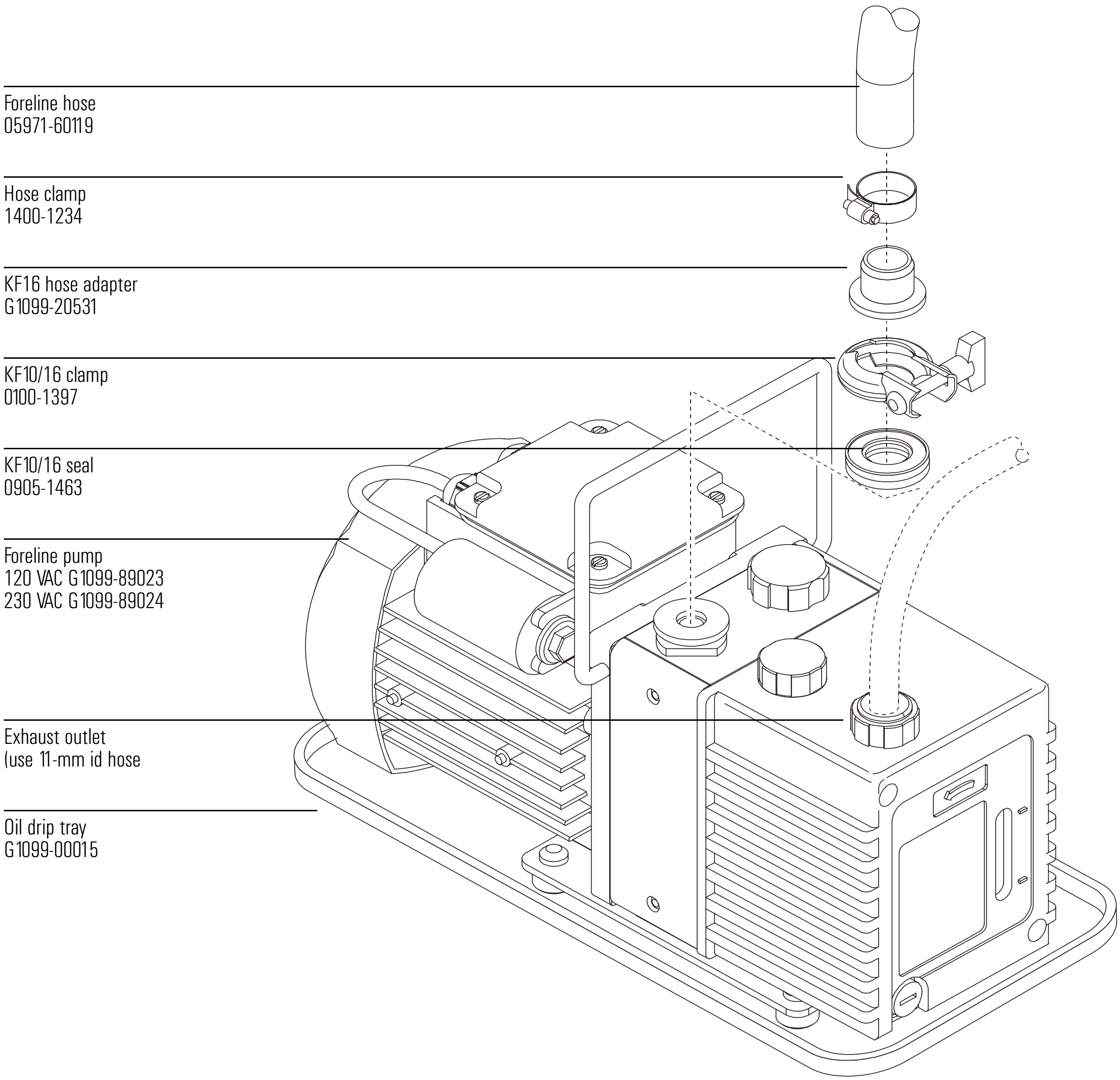
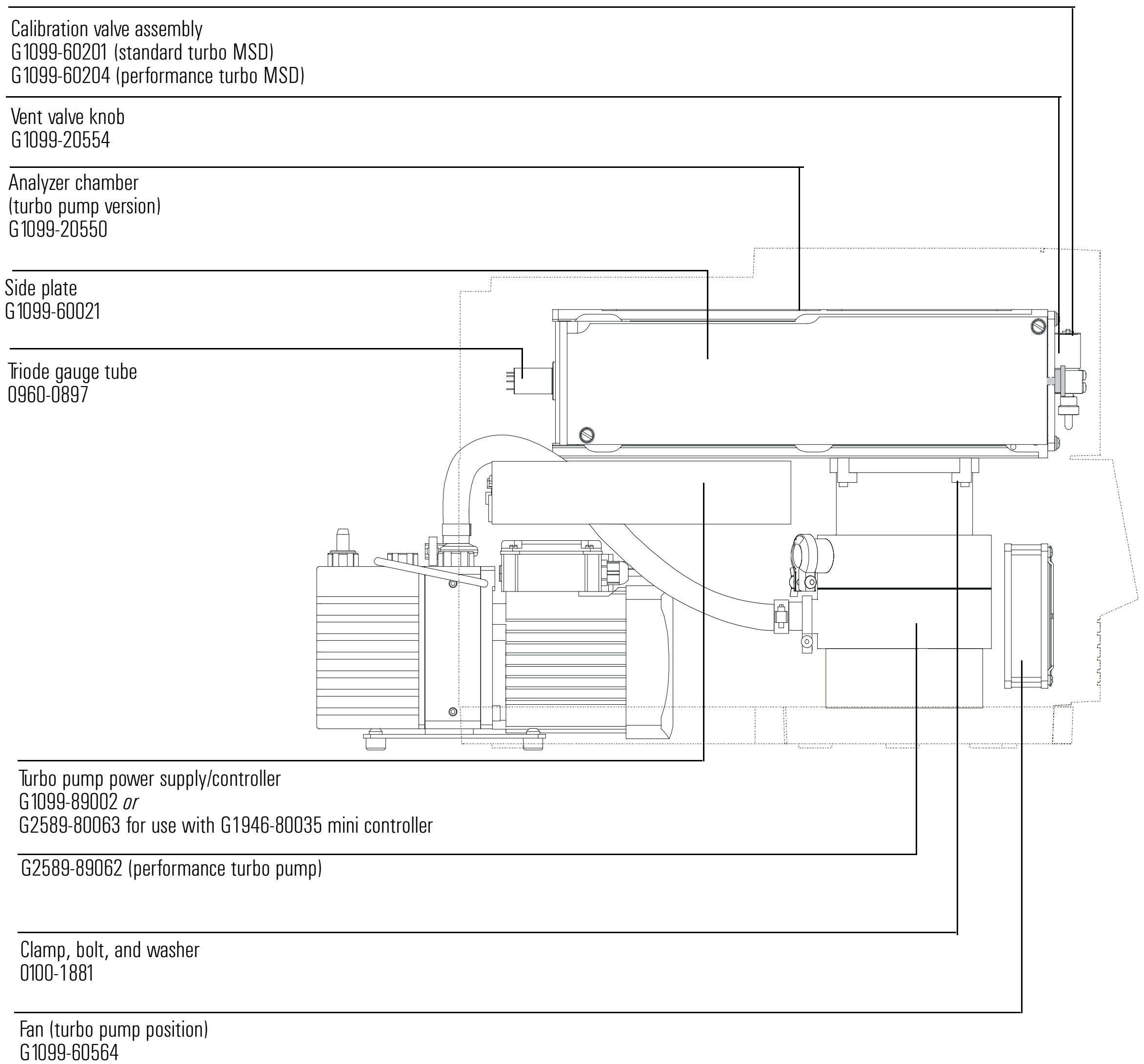

Foreline hose
05971-60119
Hose clamp
1400-1234
KF16 hose adapter
G1099-20531
KF10/16 clamp
0100-1397
KF10/16 seal
0905-1463
Foreline pump
120 VAC G1099-89023
230 VAC G1099-89024
Exhaust outlet
(use 11-mm id hose
Oil drip tray
G1099-00015

Table 14 **Foreline pump and related parts**

Description	Part number
Baffle (inside stem of triode gauge tube – not shown)	05972-00015
Calibration valve assembly for performance turbo MSD	G1099-60204
Calibration valve assembly for standard turbo (and diffusion pump) MSDs	G1099-60201
Calibrant vial	05980-20018
Claw clamps for turbo pump	0100-1881
Collar for triode gauge tube	05972-60210
Fan	3160-1037
KF25 clamp (for turbo pump outlet)	0100-0549
KF25 O-ring assembly (for turbo pump outlet)	0100-1551
Manifold end plates	
front	G1099-20552
rear	G1099-20553
Shield for triode gauge tube port	G1099-00003
Side plate (includes thumbscrews)	G1099-60021
Triode gauge tube	0960-0897
Performance turbomolecular pump[1]	G2589-89062
Standard turbomolecular pump	G2589-89061
Elbow, foreline, for the standard turbo pump	G2589-20041
Turbo pump seal, performance pump[1]	0100-1879
Turbo pump seal, standard pump[1]	0905-1443
Analyzer chamber– turbo	G1099-20550
Vent valve knob	G1099-20554
Turbo pump power supply/ controller	G1099-89002
Turbo pump harness for integrated PS/controller	G1099-60438
Turbo pump power supply for use with EXDC mini turbo controller	G2589-80063
EXDC mini turbo pump controller	G1946-80035
Turbo harness for EXDC mini controller	G2589-60034

1 The turbo pumps and their seals are not user-replaceable parts. Refer service to your Agilent Technologies service representative.


Calibration valve assembly
G1099-60201 (standard turbo MSD)
G1099-60204 (performance turbo MSD)
Vent valve knob
G1099-20554
Analyzer chamber
(turbo pump version)
G1099-20550
Side plate
G1099-60021
Triode gauge tube
0960-0897
Turbo pump power supply/controller
G1099-89002 *or*
G2589-80063 for use with G1946-80035 mini controller
G2589-89062 (performance turbo pump)
Clamp, bolt, and washer
0100-1881
Fan (turbo pump position)
G1099-60564

Analyzer

This table lists the replacement parts for the analyzer. Analyzer screws and the individual ion source parts are listed the next tables.

Table 15 **Analyzer parts**

Description	Part number
Analyzer (complete, tested, with side board)	G2589-65720
Analyzer (complete, rebuilt)	G2589-69720
detector (complete)	G1099-80001
electron multiplier horn	05971-80103
feedthrough board	G1099-60425
HED feedthrough	G1099-80012
O-ring, viton for HED feedthrough	0905-0490
ion source (complete, new)	G2589-65710
ion source (complete, rebuilt)	G2589-69710
magnet assembly	05971-60160
mass filter cable kit	G1099-60130
mass filter contacts (4 required)	G1099-60142
mass filter ceramic support, detector end	G1099-20124
mass filter ceramic support, source end	G1099-20123
mass filter heater assembly	G1099-60172
mass filter radiator	G1099-20121
mounting bracket, detector end	G1099-00002
mounting bracket, source end	G1099-00001
pins for source and detector end mounting brackets	G1099-20137
side plate (includes thumbscrews)	G1099-60021
source radiator	G1099-20122

Table 16 **Analyzer screws**

Description	Part number
Heater/sensor (quadrupole) set screw	0515-1446
Ion source thumbscrew	G1099-20138
Magnet mounting screws	0515-1046
Screw to attach magnet bracket to source radiator	0515-1602
Screws to attach source radiator and detector to quadrupole radiator	0515-1052
Screws for mass filter contact assembly and heater block	0515-0319
Screws for radiator mounting brackets and for side board	0515-0430
Source radiator screws	0515-1052

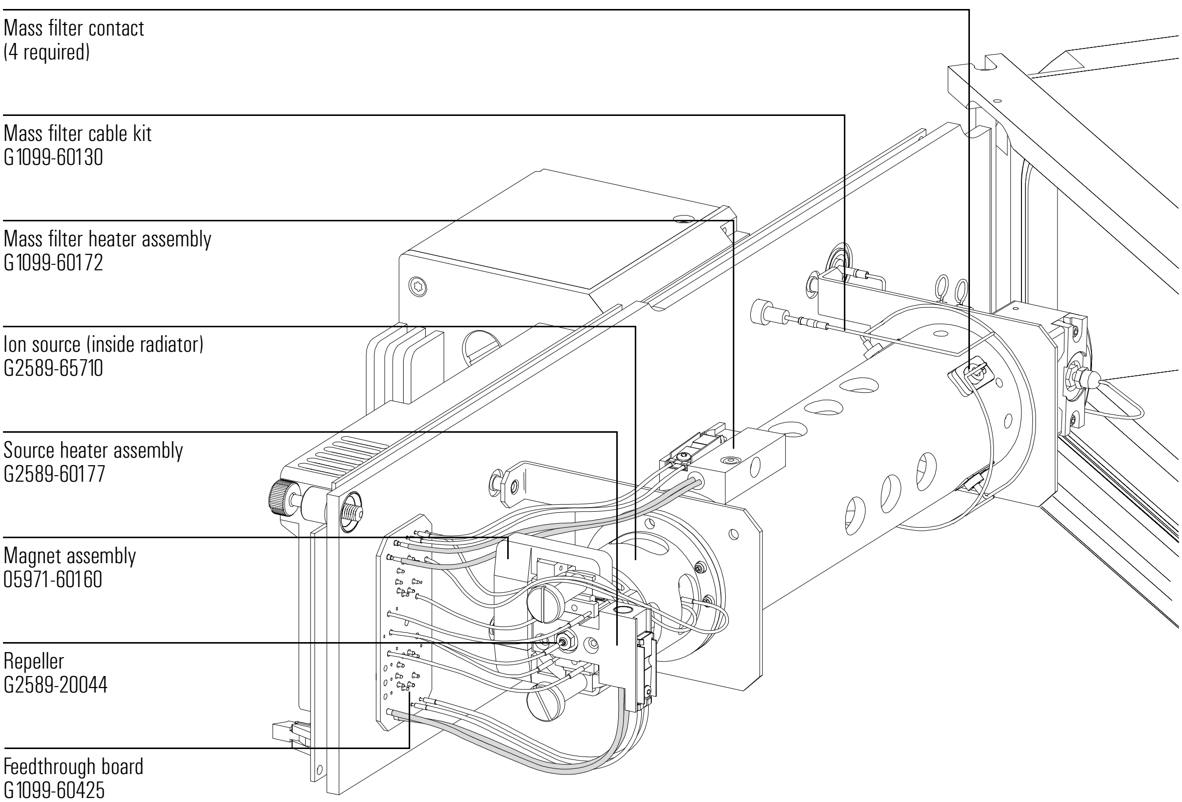


Table 17 **EI Ion source parts**

Description	Part number
Inert ion source (complete, new)	G2589-65710
Inert ion source (complete, rebuilt)	G2589-69710
drawout cylinder	G1072-20008
inert drawout plate (standard, 3mm)	G2589-20100
inert drawout plate (G2860 kit, 6mm)	G2589-20045
entrance lens	05971-20126
high temp EI filament (2 required)	G2590-60053
interface socket	G1099-20136
ion focus lens	05971-20143
lens insulator (pair)	05971-20130
repeller assembly (complete)	G2589-60102
screws	
for filaments and holding repeller assembly on source	G1999-20021
setscrew for lens stack	G1999-20022
inert source body	G2589-20043
Ion source sensor	G1099-60104
Quad sensor	G1099-60108

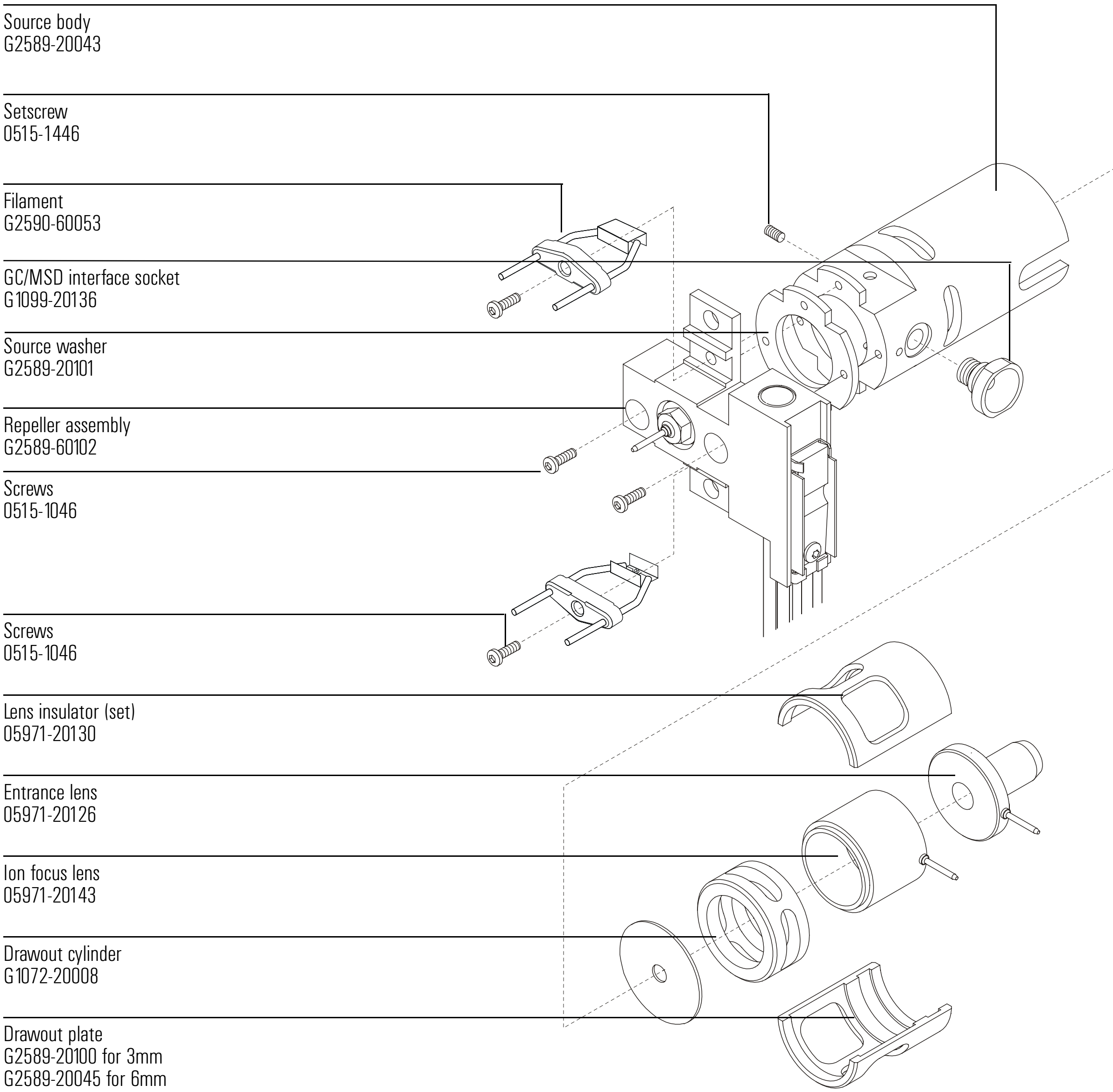
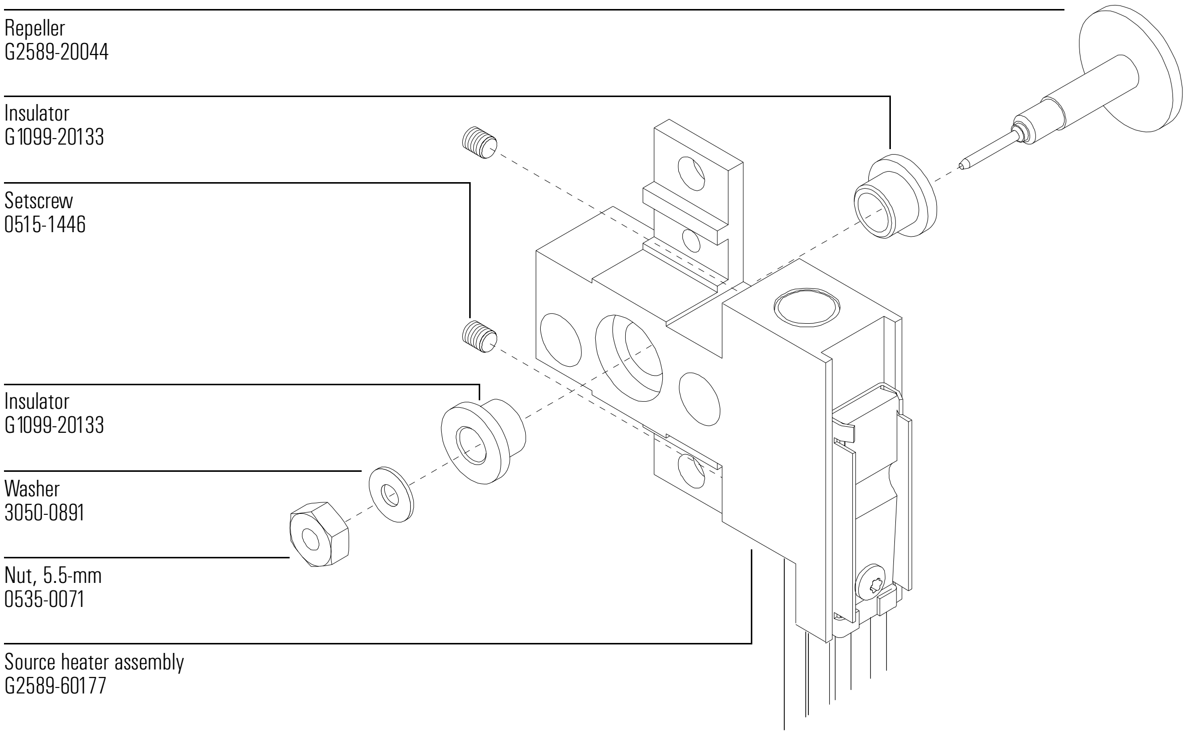

Source body
G2589-20043
Setscrew
0515-1446
Filament
G2590-60053
GC/MSD interface socket
G1099-20136
Source washer
G2589-20101
Repeller assembly
G2589-60102
Screws
0515-1046
Screws
0515-1046
Lens insulator (set)
05971-20130
Entrance lens
05971-20126
Ion focus lens
05971-20143
Drawout cylinder
G1072-20008
Drawout plate
G2589-20100 for 3mm
G2589-20045 for 6mm

Table 18 Repeller assembly parts

Description	Part number
Repeller assembly	G2589-60102
insulator (2 required)	G1099-20133
nut, 5.5-mm	0535-0071
ultra repeller	G2589-20044
setscrew	0515-1446
source heater (includes heater, sensor, heater block, ss washer)	G2589-60177
washer for nut, 5.5mm	3050-0891
repeller sensor	G1099-60104
Source washer	G2589-20101

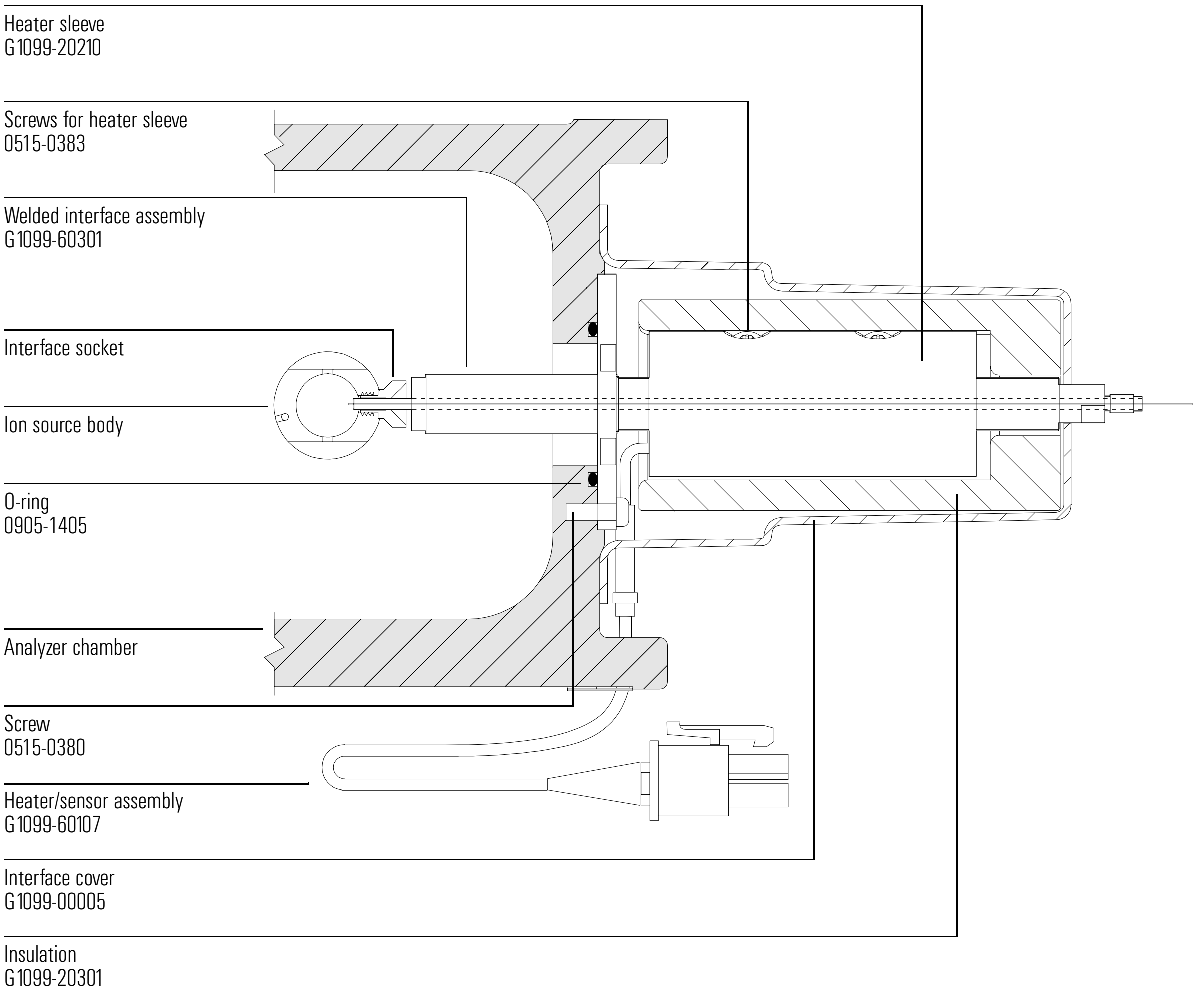

Repeller
G2589-20044
Insulator
G1099-20133
Setscrew
0515-1446
Insulator
G1099-20133
Washer
3050-0891
Nut, 5.5-mm
0535-0071
Source heater assembly
G2589-60177

EI GC/MSD interface

This table lists the replacement parts related to the GC/MSD interface.

Table 19 **GC/MSD interface**

Description	Part number
GC/MSD interface (complete)	G1099-60300
interface column nut (not shown)	05988-20066
heater sleeve	G1099-20210
heater/sensor assembly	G1099-60107
insulation	G1099-20301
setscrew for heater/sensor assembly (not shown)	0515-0236
screws, M4 x 0.7 panhead, for heater sleeve	0515-0383
welded interface assembly	G1099-60301
GC/MSD interface O-ring	0905-1405
Interface cover	G1099-00005
Screws for mounting interface and cover to analyzer chamber	0515-0380


Heater sleeve
G1099-20210
Screws for heater sleeve
0515-0383
Welded interface assembly
G1099-60301
Interface socket
Ion source body
O-ring
0905-1405
Analyzer chamber
Screw
0515-0380
Heater/sensor assembly
G1099-60107
Interface cover
G1099-00005
Insulation
G1099-20301

Consumables and maintenance supplies

This section lists parts available for cleaning and maintaining your MSD.

Table 20 **Maintenance supplies**

Description	Part number
Abrasive paper, 30 µm	5061-5896
Alumina powder	
1 kg sample	5061-5896
Cloths, clean (package of 300)	05980-60051
Cloths, cleaning (package of 300)	9310-4828
Cotton swabs (package of 100)	5080-5400
Foreline pump oil, Inland 45, 1 liter	6040-0834
Gloves, clean	
large	8650-0030
small	8650-0029
Grease, Apiezon L, high vacuum	6040-0289
Paint, touch-up, Glacier Gray	6010-1497
One-year maintenance kit	5183-0296

Table 21 **Tools**

Description	Part number
Column installation tool	G1099-20030
Tool kit	G1099-60566
Ball drivers	
1.5-mm	8710-1570
2.0-mm	8710-1804
2.5-mm	8710-1681
Hex nut driver, 5.5-mm	8710-1220
Pliers, long-nose (1.5-inch nose)	8710-1094
Screwdrivers	
flat-blade, large	8730-0002
TORX, T-10	8710-1623
TORX, T-15	8710-1622
TORX, T-20	8710-1615
Shipping kits	
5973 inert MSD	G2589-60500
6890 Series GC	G1530-60865
Tweezers, non-magnetic	8710-0907
Wrenches, open-end	
1/4-inch x 5/16-inch	8710-0510
10-mm	8710-2353
Wrist strap, anti-static	
small	9300-0969
medium	9300-1257
large	9300-0970

Table 22 Ferrules

Description	Part number
Blank, graphite-vespel	5181-3308
GC/MSD interface	
0.3-mm id, 85% Vespel 15% graphite, for 0.10-mm id columns	5062-3507
0.4-mm id, 85% Vespel 15% graphite, for 0.20-mm id and 0.25-mm id columns	5062-3508
0.5-mm id, 85% Vespel 15% graphite, for 0.32-mm id columns	5062-3506
0.8-mm id, 85% Vespel 15% graphite, for 0.53-mm id columns	5062-3538
Injection port	
0.27-mm id, 90% Vespel 10% graphite, for 0.10-mm id columns	5062-3518
0.37-mm id, 90% Vespel 10% graphite, for 0.20-mm id columns	5062-3516
0.40-mm id, 90% Vespel 10% graphite, for 0.25-mm id columns	5181-3323
0.47-mm id, 90% Vespel 10% graphite, for 0.32-mm id columns	5062-3514
0.74-mm id, 90% Vespel 10% graphite, for 0.53-mm id columns	5062-3512

Table 23 **Miscellaneous parts and samples**

Description	Part number
Electron multiplier horn	05971-80103
Filament assembly (EI)	G2590-60053
Filament assembly (CI)	G1099-80053
Foreline pump oil (1 liter)	6040-0834
Foreline exhaust oil mist trap[1]	G1099-80037
Heater/sensor assemblies	
GC/MSD interface	05972-60106
ion source	G2589-60177
mass filter	G1099-60172
Octafluoronapthalene (OFN), 1 pg/ul	8500-5441
Perfluorotributylamine (PFTBA), certified (10 gram)	8500-0656
Perfluorotributylamine (PFTBA) sample kit	05971-60571
Sample, evaluation A, hydrocarbons	05970-60045
Triode gauge tube	0960-0897
Covers	
EI top cover	G1099-40002
CI top front cover	G1999-20422
CI top rear cover	G1999-00411
Base cover	G2589-60036
Analyzer covers	
EI analyzer cover	G2589-60007
CI analyzer cover	G2589-60008

[1] Do ***not*** use an exhaust oil mist trap if you are analyzing hazardous samples, or if you are using hazardous carrier gas, or if you are running CI. Plumb the pump exhaust to a fume hood.

CI Parts

This section lists parts that may be required to maintain the 5973 inert MSD with CI. The parts listed in this section are related directly to the accessory; other parts for the MSD can be found in the previous section of this chapter.

Table 24 **Miscellaneous parts for CI MSD**

Description	Part number
Benzophenone, 100 pg/µl	8500-5440
Octafluronapthalene (OFN), 1 pg/µL	8500-5441
Bipolar HED power supply (EI/PCI/NCI MSDs only)	G1099-80018
Foreline pump secondary containment tray	G1099-00015
Methane/isobutane gas purifier	G1999-80410
PFDTD calibrant	8500-8130
Reagent gas line, 20-ft 1/8” ID stainless steel, cleaned	7157-0210
Wipes, industrial, 300/package	9310-4828
Swagelok fittings for gas purifier and inlet to flow module	
Ferrule, front, for 1/8-inch tubing, 20/package	5180-4110
Ferrule, rear, for 1/8-inch tubing, 20/package	5180-4116
Nut, for 1/8-inch tubing, 20/package	5080-8751
Nut and both front and rear ferrules, 20 sets/package	5080-8751
Tubing cutter for stainless steel tubing	8710-1709
Tubing cutter replacement blades	8710-1710
Stainless steel tubing, 1/8-inch, 20 feet	7157-0210
CI shipping kit	G2589-60505

Table 25 **CI Flow control module parts**

Description	Part number
CI flow control module (complete)	G1999-65460 (not available)
Calibration valve assembly	G1999-60452
PFDTD calibrant	8500-8130
Sample vial	05980-20018
Sample vial O-ring, 1/4-inch Viton	0905-1217
Solenoid valve and cable	G1999-80405
CI main power harness cable	G1999-60462
Display module	G1999-65461
Flow control knob	0370-3401
Flow control PCA	G1999-65005
Mass flow controller	0101-1006
Isolation valve	G1999-80402
Mass flow controller cable	G1999-60464
Reagent gas select valve	G1999-80401
VCR gasket, 1/4-inch, with retainer, one use only	0100-1436
VCR gasket, 1/8-inch, one use only	0100-0468
CI analyzer cover	G2589-60008
Front flow module cover	G1999-20422
Methane/isobutane gas purifier	G1999-80410
Reagent gas supply tubing, stainless steel, 1/8-inch	7157-0210
Rear flow module cover	G1999-00411
Swagelok fittings for gas purifier and inlet to flow module	
Ferrule, front, for 1/8-inch tubing, 10/package	5180-4110
Ferrule, rear, for 1/8-inch tubing, 10/package	5180-4116
Nut, for 1/8-inch tubing, 10/package	5180-4104
Nut and both front and rear ferrules, 10 sets/package	5080-8751

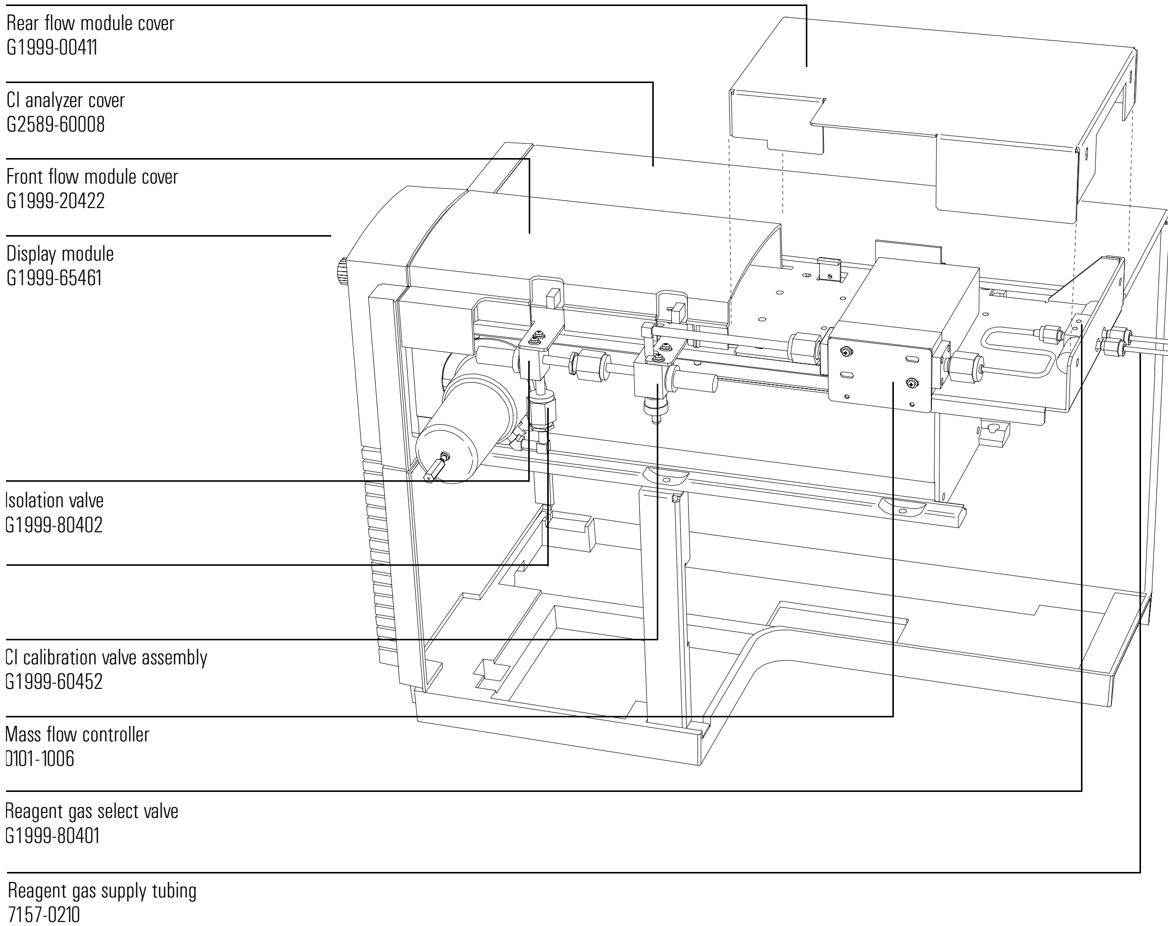

Rear flow module cover
G1999-00411
CI analyzer cover
G2589-60008
Front flow module cover
G1999-20422
Display module
G1999-65461
Isolation valve
G1999-80402
CI calibration valve assembly
G1999-60452
Mass flow controller
0101-1006
Reagent gas select valve
G1999-80401
Reagent gas supply tubing
7157-0210

Table 26 **CI ion source parts**

Description	Part number
Box for ion source shipping and storage	G1999-65001
CI ion source (tested)	G1999-65402
CI drawout cylinder	G1999-20444
CI drawout plate	G1999-20446
CI filament	G1099-80053
CI heater block	G1999-20431
CI interface tip seal	G1999-60412
CI ion focus lens	G1999-20443
CI lens insulators (set)	G1999-20445
CI repeller	G1999-20432
CI repeller insulator	G1999-20433
CI source body	G1999-20430
CI source heater assembly	G1999-60414
Dummy filament	G1999-60454
Entrance lens	05971-20126
Screw, socket-head cap for mounting filaments	G1999-20021
Setscrew for mounting heater and lens stack	G1999-20022
Screw, M3 × 4L socket head for mounting RTD	0515-2903
Screw, M2 × 8L mounts source to radiator	0515-1046

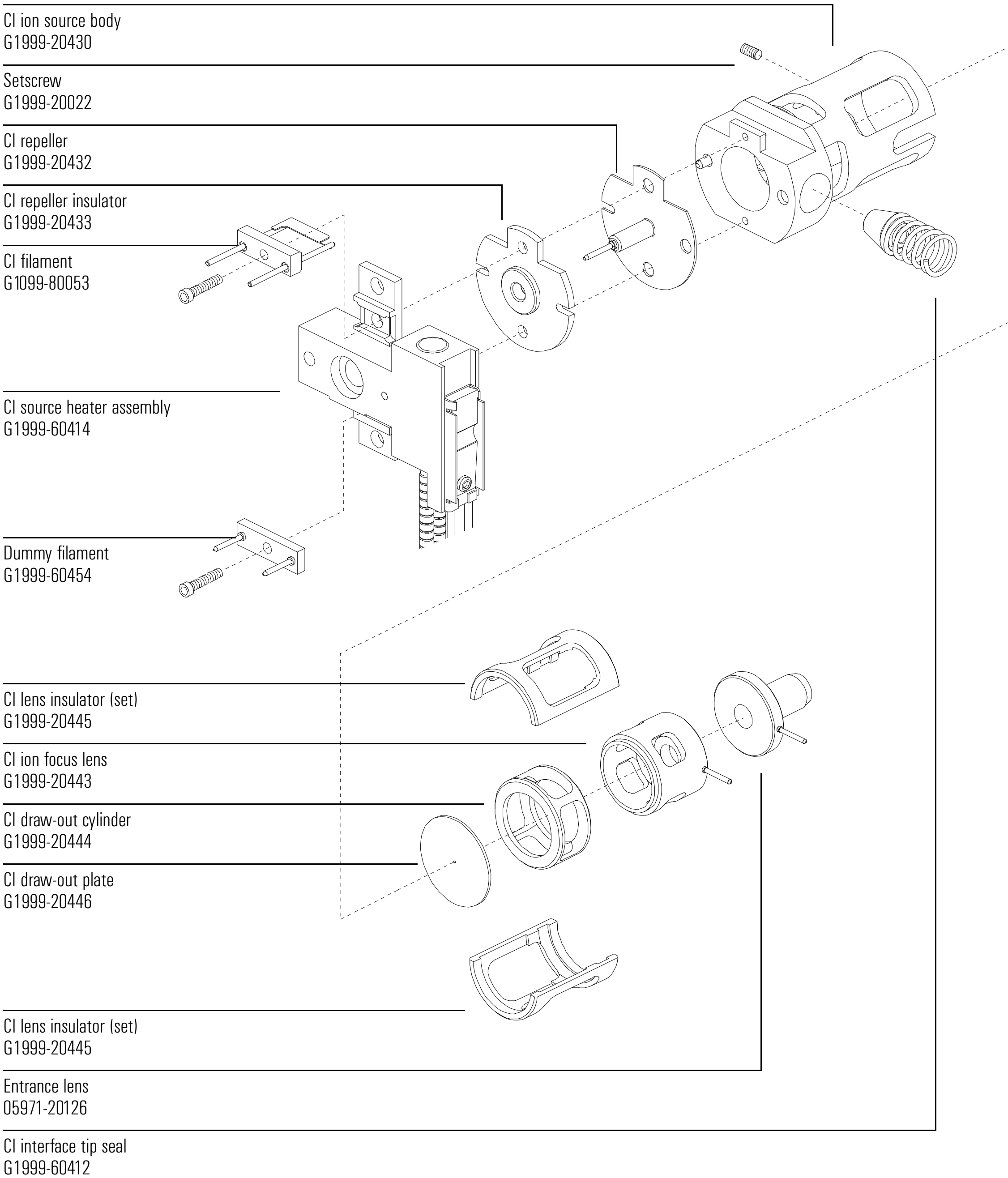
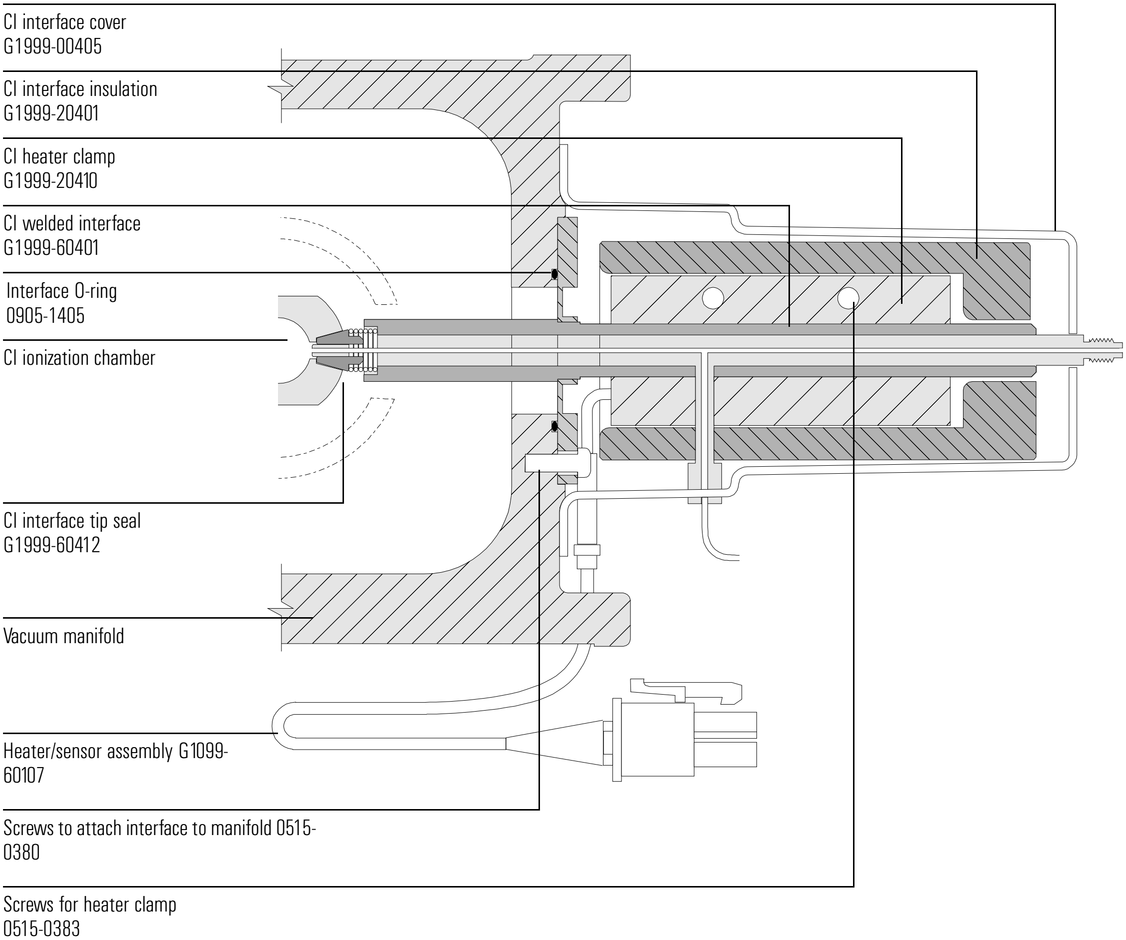

CI ion source body
G1999-20430
Setscrew
G1999-20022
CI repeller
G1999-20432
CI repeller insulator
G1999-20433
CI filament
G1099-80053
CI source heater assembly
G1999-60414
Dummy filament
G1999-60454
CI lens insulator (set)
G1999-20445
CI ion focus lens
G1999-20443
CI draw-out cylinder
G1999-20444
CI draw-out plate
G1999-20446
CI lens insulator (set)
G1999-20445
Entrance lens
05971-20126
CI interface tip seal
G1999-60412

Table 27 **EI/CI GC/MSD interface parts**

Description	Part number
CI/EI GC/MSD interface assembly	G1999-65400
Heater clamp	G1999-20410
Heater/sensor assembly	G1099-60107
Interface cover	G1999-00405
Interface insulation (two pieces)	G1999-20401
Screws for heater clamp	0515-0383
Screws to attach interface to manifold	0515-0380
Welded interface	G1999-60401
Interface tip seal	G1999-60412
Vespel blank	5181-3308


CI interface cover
G1999-00405
CI interface insulation
G1999-20401
CI heater clamp
G1999-20410
CI welded interface
G1999-60401
Interface O-ring
0905-1405
CI ionization chamber
CI interface tip seal
G1999-60412
Vacuum manifold
Heater/sensor assembly G1099-60107
Screws to attach interface to manifold 0515-0380
Screws for heater clamp
0515-0383

CI Parts

Appendix A

Chemical Ionization Theory

Chemical ionization overview

Chemical ionization (CI) is a technique for creating ions used in mass spectrometric analyses. There are significant differences between CI and electron ionization (EI). This section describes the most common chemical ionization mechanisms.

In EI, relatively high-energy electrons (70 eV) collide with molecules of the sample that is to be analyzed. These collisions produce (primarily) positive ions. Upon ionization, the molecules of a given substance fragment in fairly predictable patterns. EI is a direct process: energy is transferred collisionally from electrons to the sample molecules.

For CI, in addition to the sample and carrier gas, large amounts of reagent gas are introduced into the ionization chamber. Since there is so much more reagent gas than sample, most of the emitted electrons collide with reagent gas molecules, forming reagent ions. These reagent-gas ions react with each other, in primary and secondary reaction processes that establish an equilibrium. They also react in various ways with sample molecules to form sample ions. CI ion formation involves much lower energy, and is much more "gentle", than electron ionization. Since CI results in much less fragmentation, CI spectra usually show high abundance of the molecular ion. For this reason, CI is often used to determine the molecular weights of sample compounds.

Methane is the most common CI reagent gas. It yields certain characteristic ionization patterns. Other reagent gases yield different patterns and may result in better sensitivity for some samples. Common alternative reagent gases are isobutane and ammonia. Carbon dioxide is often used in negative CI. Less common reagent gases are carbon dioxide, hydrogen, freon, trimethylsilane, nitric oxide, and methylamine. Different ionization reactions occur with each reagent gas.

WARNING

Ammonia is toxic and corrosive. Use of ammonia requires special maintenance and safety precautions.

Water contamination in reagent gases may decrease CI sensitivity dramatically. A large peak at *m/z* 19 (H_30^+) in positive CI is a diagnostic symptom of water contamination. In high enough concentrations, especially when combined with calibrant, water contamination will result in a heavily contaminated ion source. Water contamination is most common immediately after new reagent gas tubing or reagent gas cylinders are connected. This contamination will often decrease if the reagent gas is allowed to flow for a few hours, purging the system.

References on chemical ionization

A. G. Harrison, *Chemical Ionization Mass Spectrometry*, 2nd Edition, CRC Press, INC. Boca Raton, FL (1992) ISBN 0-8493-4254-6.

W. B. Knighton, L. J. Sears, E. P. Grimsrud, “High Pressure Electron Capture Mass Spectrometry”, Mass Spectrometry Reviews (1996), 14, 327-343.

E. A. Stemmler, R. A. Hites, *Electron Capture Negative Ion Mass Spectra of Environmental Contaminants and Related Compounds*, VCH Publishers, New York, NY (1988) ISBN 0-89573-708-6.

Positive CI theory

Positive CI occurs with the same analyzer voltage polarities as EI. For PCI, the reagent gas is ionized by collision with emitted electrons. The reagent gas ions react chemically with sample molecules (as proton donors) to form sample ions. PCI ion formation is more “gentle” than electron ionization, producing less fragmentation. This reaction usually yields high abundance of the molecular ion, and is therefore often used for determining molecular weights of samples.

The most common reagent gas is methane. Methane PCI produces ions with almost any sample molecule. Other reagent gases, such as isobutane or ammonia, are more selective, and cause even less fragmentation. Because of the high background from the reagent gas ions, PCI is not especially sensitive, and detection limits are generally high.

There are four fundamental ionization processes that take place during positive chemical ionization at ion source pressures in the 0.8 – 2.0 Torr range. These are:

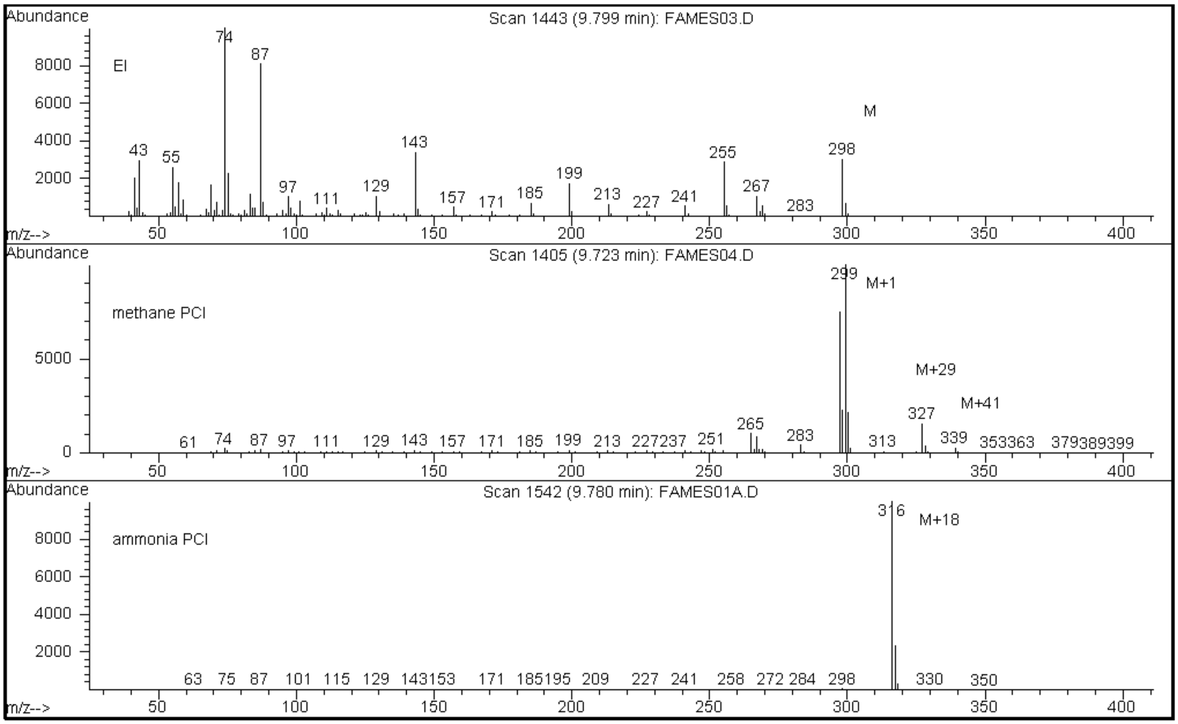
- Proton transfer
- Hydride abstraction
- Addition
- Charge exchange

Depending on the reagent gas used, one or more of these four processes can be used to explain the ionization products observed in the resulting mass spectra.

EI, methane PCI, and ammonia PCI spectra of methyl stearate are shown opposite. The simple fragmentation pattern, large abundance of the $[MH]^+$ ion, and the presence of the two adduct ions are characteristic of positive chemical ionization using methane as a reagent gas.

The presence of air or water in the system, especially in the presence of PFDTD calibrant, quickly contaminates the ion source.

Methyl stearate (MW = 298): EI, methane PCI, and ammonia PCI



Proton transfer

Proton transfer can be expressed as

$$BH^+ + M \rightarrow MH^+ + B$$

where the reagent gas B has undergone ionization resulting in protonation. If the proton affinity of the analyte (sample) M is greater than that of the reagent gas, then the protonated reagent gas will transfer its proton to the analyte, forming a positively charged analyte ion.

The most frequently used example is the proton transfer from CH_5^+ to the molecular analyte, which results in the protonated molecular ion MH^+.

The relative proton affinities of the reagent gas and the analyte govern the proton transfer reaction. If the analyte has a greater proton affinity than the reagent gas, then proton transfer can take place. Methane (CH_4) is the most common reagent gas because its proton affinity is very low.

Proton affinities can be defined according to the reaction:

$$B + H^+ \rightarrow BH^+$$

where the proton affinities are expressed in kcal/mole. Methane's proton affinity is 127 kcal/mole. The following tables list the proton affinities of several possible reagent gases and of several small organic compounds with various functional groups.

The mass spectrum generated by a proton-transfer reaction depends on several criteria. If the difference in proton affinities is large (as with methane), substantial excess energy may be present in the protonated molecular ion. This can result in subsequent fragmentation. For this reason, isobutane, with a proton affinity of 195 kcal/mole, may be preferred to methane for some analyses. Ammonia has a proton affinity of 207 kcal/mole, making it less likely to protonate most analytes. Proton-transfer chemical ionization is usually considered to be “soft” ionization, but the extent of the softness is dependent on the proton affinities of both the analyte and the reagent gas, as well as on other factors, including ion source temperature.

Reagent gas proton affinities

Species	Proton affinity kcal/mole	Reactant ion formed
H_2	100	H_3^+ (*m/z* 3)
CH_4	127	CH_5^+ (*m/z* 17)
C_2H_4	160	$C_2H_5^+$ (*m/z* 29)
H_2O	165	H_3O^+ (*m/z* 19)
H_2S	170	H_3S^+ (*m/z* 35)
CH_3OH	182	$CH_3OH_2^+$ (*m/z* 33)
t-C_4H_{10}	195	t-$C_4H_9^+$ (*m/z* 57)
NH_3	207	NH_4^+ (*m/z* 18)

Proton affinities of selected organic compounds for PCI

Molecule	Proton affinity (kcal/mole)	Molecule	Proton affinity (kcal/mole)
Acetaldehyde	185	Methyl amine	211
Acetic acid	188	Methyl chloride	165
Acetone	202	Methyl cyanide	186
Benzene	178	Methyl sulfide	185
2-Butanol	197	Methyl cyclopropane	I80
Cyclopropane	179	Nitroethane	185
Dimethyl ether	190	Nitromethane	180
Ethane	121	n-Propyl acetate	207
Ethyl formate	198	Propylene	179
Formic acid	175	Toluene	187
Hydrobromic acid	140	trans-2-Butene	180
Hydrochloric acid	141	Trifluoroacetic acid	167
Isopropyl alcohol	190	Xylene	187
Methanol	182		

Hydride abstraction

In the formation of reagent ions, various reactant ions can be formed that have high hydride-ion (H^-) affinities. If the hydride-ion affinity of a reactant ion is higher than the hydride-ion affinity of the ion formed by the analyte's loss of H^-, then the thermodynamics are favorable for this chemical ionization process. Examples include the hydride abstraction of alkanes in methane chemical ionization. In methane CI, both CH_5^+ and $C_2H_5^+$ are capable of hydride abstraction. These species have large hydride-ion affinities, which results in the loss of H^- for long-chain alkanes, according to the general reaction

$$R^+ + M \rightarrow [M\text{-}H]^+ + RH$$

For methane, R^+ is CH_5^+ and $C_2H_5^+$, and M is a long-chain alkane. In the case of CH_5^+, the reaction proceeds to form $[M\text{-}H]^+ + CH_4 + H_2$. The spectra resulting from hydride abstraction will show an M-1 amu peak resulting from the loss of H^-. This reaction is exothermic so fragmentation of the $[M\text{-}H]^+$ ion is often observed.

Often, both hydride-abstraction and proton-transfer ionization can be evident in the sample spectrum. One example is the methane CI spectrum of long-chain methyl esters, where both hydride abstraction from the hydrocarbon chain and proton transfer to the ester function occur. In the methane PCI spectrum of methyl stearate, for example, the MH^+ peak at m/z = 299 is created by proton transfer, and the $[M\text{-}1]^+$ peak at m/z 297 is created by hydride abstraction.

Addition

For many analytes, proton-transfer and hydride-abstraction chemical ionization reactions are not thermodynamically favorable. In these cases, reagent gas ions are often reactive enough to combine with the analyte molecules by condensation or association (addition reactions). The resulting ions are called adduct ions. Adduct ions are observed in methane chemical ionization by the presence of $[M+C_2H_5]^+$ and $[M+C_3H_5]^+$ ions, which result in M+29 and M+41 amu mass peaks.
Addition reactions are particularly important in ammonia CI. Because the NH_3 has a high proton affinity, few organic compounds will undergo proton transfer with ammonia reagent gas. In ammonia CI, a series of ion-molecule reactions takes place, resulting in the formation of NH_4^+, $[NH_4NH_3]^+$, and $[NH_4(NH_3)_2]^+$. In particular, the ammonium ion, NH_4^+, will give rise to an intense $[M+NH_4]^+$ ion observed at M+18 amu, either through condensation or association. If this resulting ion is unstable, subsequent fragmentation may be observed. The neutral loss of H_2O or NH_3, observed as a subsequent loss of 18 or 17 amu, respectively, is also common.

Charge exchange

Charge-exchange ionization can be described by the reaction:

$$X^{+\bullet} + M \rightarrow M^{+\bullet} + X$$

where X^+ is the ionized reagent gas, and M is the analyte of interest. Examples of reagent gases used for charge exchange ionization include the noble gases (helium, neon, argon, krypton, xenon, and radon), nitrogen, carbon dioxide, carbon monoxide, hydrogen, and other gases that do not react “chemically” with the analyte. Each of these reagent gases, once ionized, has a recombination energy expressed as:

$$X^{+\bullet} + e^- \rightarrow X$$

or simply the recombination of the ionized reagent with an electron to form a neutral species. If this energy is greater than the energy required to remove an electron from the analyte, then the first reaction above is exothermic and thermodynamically allowed.

Charge-exchange chemical ionization is not widely used for general analytical applications. It can, however, be used in some cases when other chemical ionization processes are not thermodynamically favored.

Negative CI theory

Negative chemical ionization (NCI) is performed with analyzer voltage polarities reversed to select negative ions. There are several chemical mechanisms for negative chemical ionization. Not all mechanisms provide the dramatic increases in sensitivity often associated with negative chemical ionization. The four most common mechanisms (reactions) are:

- Electron capture
- Dissociative electron capture
- Ion pair formation
- Ion-molecule reactions

In all of the cases except the ion-molecule reactions, the reagent gas serves a function different from the function it serves in positive chemical ionization. In negative CI, the reagent gas is often referred to as the buffer gas. When the reagent gas is bombarded with high energy electrons from the filament, the following reaction occurs:

$$\text{Reagent gas} + e^-_{\text{(230eV)}} \rightarrow \text{Reagent ions} + e^-_{\text{(thermal)}}$$

If the reagent gas is methane, the reaction is:

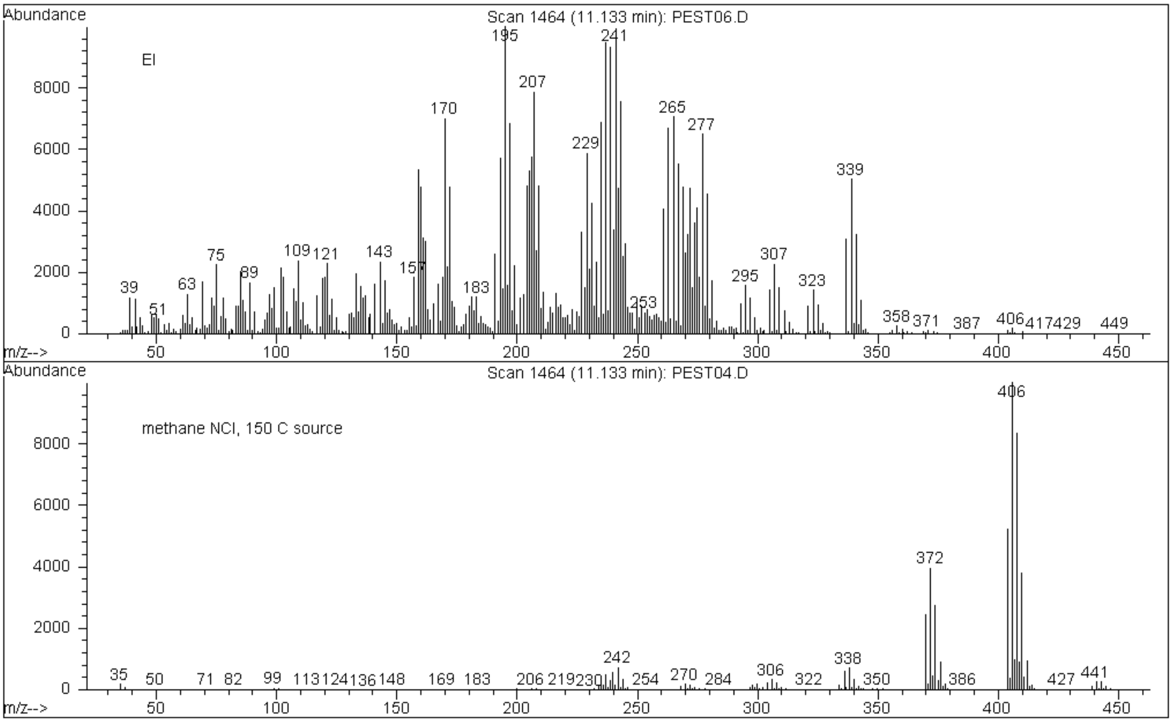
$$CH_4 + e^-_{\text{(230eV)}} \rightarrow CH_4^+ + 2e^-_{\text{(thermal)}}$$

The thermal electrons have lower energy levels than the electrons from the filament. It is these thermal electrons that react with the sample molecules.

There are no negative reagent gas ions formed. This prevents the kind of background that is seen in PCI mode, and is the reason for the much lower detection limits of NCI. The products of negative chemical ionization can only be detected when the MSD is operating in negative ion mode. This operating mode reverses the polarity of all the analyzer voltages.

Carbon dioxide is often used as a buffer gas in negative CI. It has obvious cost, availability, and safety advantages over other gases.

Endosulfan I (MW = 404): EI and methane NCI



Electron capture

Electron capture, is the primary mechanism of interest in negative CI. Electron capture (often referred to as high-pressure electron capture mass spectrometry, or HPECMS) provides the high sensitivity for which NCI is known. For some samples, and under ideal conditions, electron capture can provide sensitivity as much as 10 to 1000 times higher than positive ionization.

Note that all the reactions associated with positive CI will also occur in NCI mode, usually with contaminants. The positive ions formed do not leave the ion source because of the reversed lens voltages, and their presence can quench the electron capture reaction.

The electron capture reaction is described by:

$$MX + e^{-}_{\text{(thermal)}} \rightarrow MX^{-\bullet}$$

where MX is the sample molecule and the electron is a thermal (slow) electron generated by the interaction between high energy electrons and the reagent gas.

In some cases, the $MX^{-\bullet}$ radical anion is not stable. In those cases the reverse reaction can occur:

$$MX^{-\bullet} \rightarrow MX + e^{-}$$

The reverse reaction is sometimes called autodetachment. This reverse reaction generally occurs very quickly. Thus, there is little time for the unstable anion to be stabilized through collisions or other reactions.

Electron capture is most favorable for molecules that have hetero-atoms. For example: nitrogen, oxygen, phosphorus, sulfur, silicon, and especially the halogens: fluorine, chlorine, bromine, and iodine.

The presence of oxygen, water, or almost any other contaminant interferes with the electron-attachment reaction. Contaminants cause the negative ion to be formed by the slower ion-molecule reaction. This generally results in less sensitivity. All potential contamination sources, especially oxygen (air) and water sources, must be minimized.

Dissociative electron capture

Dissociative electron capture is also known as dissociative resonance capture. It is a process similar to electron capture. The difference is that during the reaction, the sample molecule fragments or dissociates. The result is typically an anion and a neutral radical. Dissociative electron capture is illustrated by the reaction equation:

$$MX + e^-_{(thermal)} \rightarrow M^\bullet + X^-$$

This reaction does not yield the same sensitivity as electron capture, and the mass spectra generated typically have lower abundance of the molecular ion.

As with electron capture, the products of dissociative electron capture are not always stable. The reverse reaction sometimes occurs. This reverse reaction is sometimes called an associative detachment reaction. The equation for the reverse reaction is:

$$M^\bullet + X^- \rightarrow MX + e^-$$

Ion pair formation

Ion pair formation is superficially similar to dissociative electron capture. The ion pair formation reaction is represented by the equation:

$$MX + e^-_{(thermal)} \rightarrow M^+ + X^- + e^-$$

As with dissociative electron capture, the sample molecule fragments. Unlike dissociative electron capture, however, the electron is not captured by the fragments. Instead, the sample molecule fragments in such a way that the electrons are distributed unevenly and positive and negative ions are generated.

Ion-molecule reactions

Ion-molecule reactions occur when oxygen, water, and other contaminants are present in the CI ion source. Ion-molecule reactions are 2 – 4 times slower than electron-attachment reactions and do not provide the high sensitivity associated with electron capture reactions. Ion-molecule reactions can be described by the general equation:

$$M + X^- \rightarrow MX^-$$

where X^- is most often a halogen or hydroxyl group that has been created by ionization of contaminants by electrons from the filament. Ion-molecule reactions compete with electron capture reactions. The more ion-molecule reactions that occur, the fewer electron capture reactions occur.

Numerics

A

B

D

E

H

I

N

O

P

Q

W

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