

Agilent 5977B Series MSD

Operating Manual



Agilent Technologies

Notices

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Manual Part Number

G7077-90026

Edition

First edition, September 2016

Printed in USA

Agilent Technologies, Inc.
5301 Stevens Creek Boulevard
Santa Clara, CA 95051

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WARNING

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

About This Manual

This manual contains information for operating and maintaining the Agilent 5977B Series Mass Selective Detector (MSD).

1 “Introduction”

Chapter 1 describes general information about the 5977B Series MSDs, including a hardware description, general safety warnings, and hydrogen safety information.

2 “Installing 7890 GC Columns”

Chapter 2 shows you how to prepare a capillary column for use with the MSD, install it in the GC oven, and connect it to the MSD using the GC/MSD interface.

3 “Intuvo 9000 GC Column Maintenance”

Chapter 3 shows you how to install an Agilent Intuvo column, connect a flow path from the column through the MS transfer line and into the ion source, and maintain the column guard chip.

4 “Operating in EI Mode”

Chapter 3 describes basic tasks such as setting temperatures, monitoring pressures, tuning, venting, and pumpdown. Much of the information in this chapter also applies to CI operation.

5 “Operating in CI Mode”

Chapter 4 describes additional tasks necessary to operate in CI mode.

6 “General Maintenance”

Chapter 5 describes maintenance procedures common to both EI and CI instruments.

7 “CI Maintenance”

Chapter 6 describes maintenance procedures unique to CI MSDs.

Hardware User Information

Accompanying your hardware and software is a comprehensive collection of manuals, videos, user applications, and method development tools. These are located on the:

- Agilent GC and GC/MS User Manuals and Tools DVD set
- Agilent GC/MS Software Information and Manuals memory stick

See the Agilent 5977B MSD with MassHunter Quick Start document for information on finding and installing the documentation located on this USB and DVDs.

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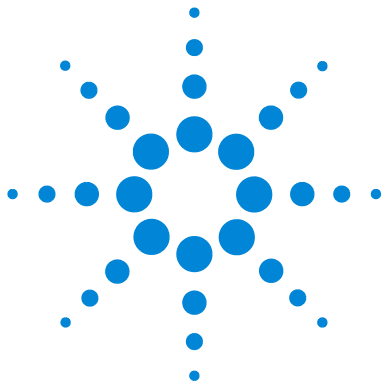
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1 Introduction

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This chapter describes general information about the MSD, including a hardware description, general safety warnings, and hydrogen safety information.

If you are using the *Agilent Intuvo 9000 Gas Chromatograph* with your MS, Chemical Ionization (CI) and the JetClean system are not currently supported.



5977B Series MSD Version

The 5977B Series MSDs are equipped with a turbomolecular (turbo) pump and a choice of four foreline pumps or a diffusion pump paired with a Pfeiffer DUO 2.5 foreline pump. There are three types of electron ionization (EI) sources available on the 5977B Series MSD, a standard EI stainless steel (SS) source, an EI Extractor (XTR) source available on the Inert+ MSD model, and a high efficiency source (HES). A chemical ionization (CI) source includes a reagent flow control system, a CI calibration system, and other required hardware features. The serial number label displays a product number that indicates what type of MSD you have. (See [Table 1](#).)

Table 1 Available high vacuum pumps

| Model name | Product number | Description | Ionization mode/Type |
|---------------------------|----------------|----------------|-----------------------------------|
| 5977B MSD Diff Pump | G7080B | Diffusion pump | EI/SS |
| 5977B MSD Turbo Pump | G7081B | Turbo pump | EI/SS |
| 5977B Inert+ MSD EI Turbo | G7077B | Turbo pump MSD | EI/XTR |
| 5977B EI/CI MSD | G7078B | Turbo pump MSD | EI /XTR CI /PCI, NCI |
| 5977B HES MSD | G7079B | Turbo pump MSD | EI HES field upgradeable to CI |

Abbreviations Used

The abbreviations in [Table 2](#) are used in discussing this product. They are collected here for convenience.

Table 2 Abbreviations

| Abbreviation | Definition |
|---------------------|--|
| AC | Alternating current |
| ALS | Automatic liquid sampler |
| BFB | Bromofluorobenzene (calibrant) |
| CI | Chemical ionization |
| DA | Data analysis |
| DC | Direct current |
| DFTPP | Decafluorotriphenylphosphine (calibrant) |
| DIP | Direct insertion probe |
| DS | Data System |
| EI | Electron ionization |
| EM | Electron multiplier (detector) |
| EMV | Electron multiplier voltage |
| EPC | Electronic pneumatic control |
| eV | Electron volt |
| GC | Gas chromatograph |
| HED | High-energy dynode (refers to detector and its power supply) |
| HES | High Efficiency Source. New generation EI source constructed from inert material |
| Inert | Standard EI source constructed from inert materials |
| Inert+ | MSD model designation equipped with an EI XTR source |
| id | Inside diameter |
| LAN | Local Area Network |

Table 2 Abbreviations (continued)

| Abbreviation | Definition |
|---------------------|---|
| LVDS | Low-voltage differential signaling |
| <i>m/z</i> | Mass to charge ratio |
| MFC | Mass flow controller |
| MSD | Mass selective detector |
| NCI | Negative CI |
| OFN | Octafluoronaphthalene (calibrant) |
| PCI | Positive CI |
| PFDTD | Perfluoro-5,8-dimethyl-3,6,9-trioxydodecane (calibrant) |
| PFHT | 2,4,6-tris(perfluoroheptyl)-1,3,5-triazine (calibrant) |
| PFTBA | Perfluorotributylamine (calibrant) |
| Quad | Quadrupole mass filter |
| RF | Radio frequency |
| RFPA | Radio frequency power amplifier |
| SS | Stainless steel |
| Torr | Unit of pressure, 1 mm Hg |
| Turbo | Turbomolecular (pump) |
| WUI | Web user interface |
| XTR | EI Extractor source |

The 5977B Series MSD

The 5977B Series MSD is a stand-alone capillary GC detector for use with either an Agilent 7890B Series or an Agilent 7820 GC. The MSD features:

- An optional JetClean system for cleaning the ion source in place under vacuum
- WEB User Interface (WUI) for locally monitoring and operating the MSD
- A turbo vacuum pump with one of four different foreline pumps or a diffusion vacuum pump with a Pfeiffer DUO 2.5 foreline pump
- Four different types of independently heated EI sources available:
 - Standard source in SS material
 - Standard source in Inert material
 - XTR source
 - HES
- Turbo pump models with 7890B GCs are field upgradeable to CI (PCI/NCI) modes. The upgrade adds an independently heated CI source, reagent gas MFC and plumbing, and CI tuning calibration
- Independently MSD heated hyperbolic Quad
- HED EM
- Independently GC heated GC/MSD interface

Physical description

The 5977B Series MSD housing is approximately 41 cm high, 30 cm wide, and 54 cm deep. The weight is 39 kg for the diffusion pump mainframe, 44 kg for the standard EI turbo pump mainframe, and 46 kg for the EI/CI turbo pump mainframe. The foreline (roughing) pump weighs an additional 11 kg (standard pump), and is usually located on the floor behind the MSD.

The basic components of the instrument are:

- Frame/cover assemblies
- Vacuum system
- GC interface
- Electronics
- Analyzer

Vacuum gauge

The MSD may be equipped (or ordered) with an ion vacuum gauge. The Agilent MassHunter GC/MS Acquisition software can be used to read the pressure (high vacuum) in the vacuum manifold. Operation of the gauge controller is described in this manual. The gauge is **required** for CI operation.

Table 3 5977B Series MSD features

| Feature | Diffusion | Turbo |
|--|-----------------------|---|
| High vacuum pump | Diffusion | Turbo |
| Optimal He column flow mL/min | 1 | 1 to 2 |
| Maximum recommended gas flow mL/min [†] | 1.5 | 4 |
| Maximum gas flow, mL/min [†] | 2 | 6.5 |
| Max column id | 0.25 mm (30 m) | 0.53 mm (30 m) |
| CI capability [‡] | No | Yes |
| Inert ion sources available | Yes | Yes |
| GC compatibility | 6890/7820/7890 Series | 7890 Series |
| Foreline pumps available | Pfeiffer Duo 2.5 | Pfeiffer Duo 2.5, MVP-070-3, MVP-070-3C, IDP3 24V |
| DIP ^{**} capability (3rd party) | Yes | Yes |

* Total gas flow into the MSD: column flow plus reagent gas flow (if applicable). Based on helium gas use. For other gases the maximum flow will vary.

† Expect degradation of spectral performance and sensitivity.

‡ Turbo pump models are field upgradeable to CI.

** Direct insertion probe.

MSD Hardware Description

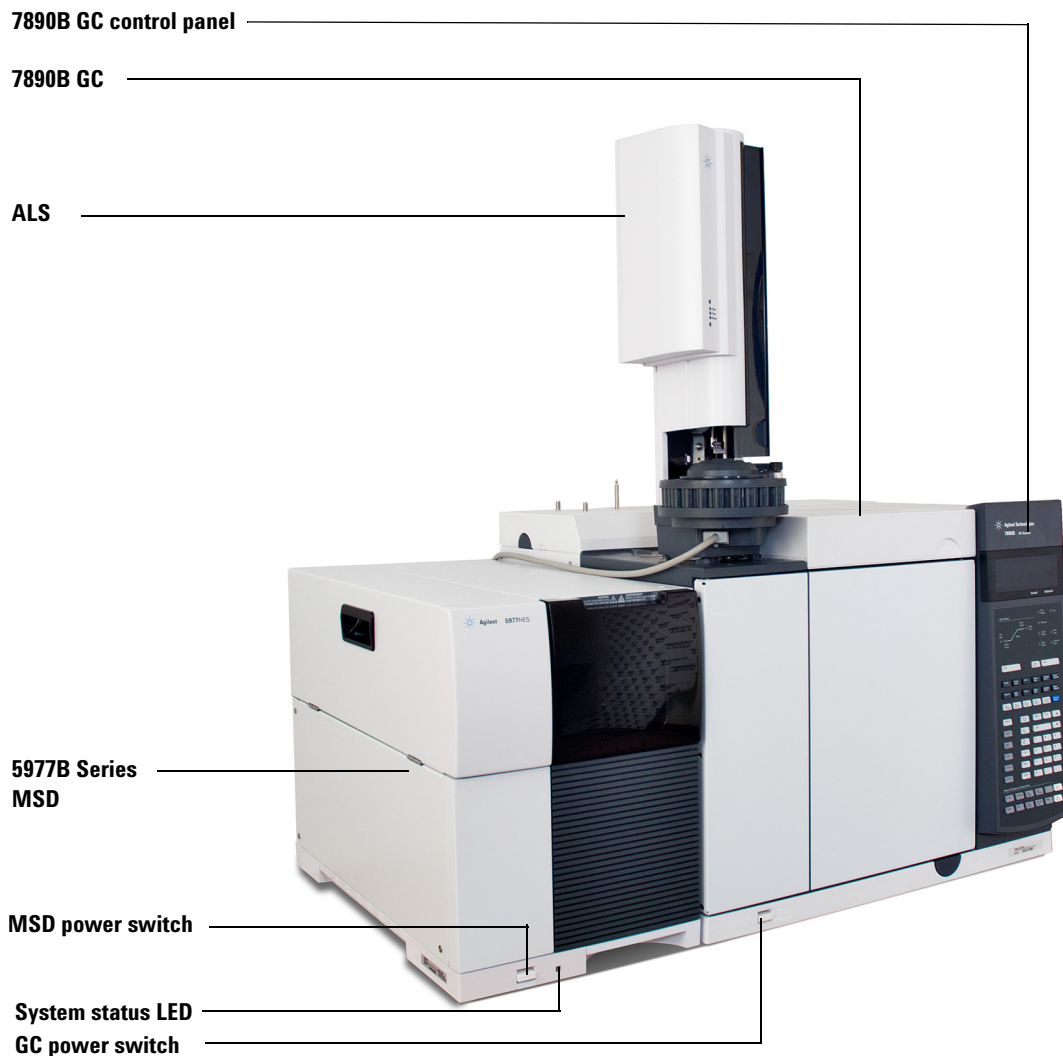


Figure 1 5977B Series GC/MSD system, shown with an Agilent 7890B GC

In this manual, the term “CI MSD” refers to the G7078B MSD or to the G7077B, G7079B, and G7081B models that were field upgraded for CI operation. It also applies, unless otherwise specified, to the flow modules for these instruments.

The CI hardware upgrade allows the MSD to produce high-quality, classical CI spectra, which include molecular adduct ions. A variety of reagent gases can be used. (See [Chapter 5](#), “Operating in CI Mode” on [page 119](#).)

The 5977B Series CI system field upgrade adds the following to the 5977B Series MSD:

- EI/CI GC/MSD interface
- JetClean option that shares the same MFC system EI/CI GC/MSD interface
- CI source with the new common interface tip seal that can also be used with an EI XTR source or HES
- Reagent gas MFC
- Bipolar HED power supply for PCI and NCI operation

A methane/isobutane gas purifier is provided and is **required**. It removes oxygen, water, hydrocarbons, and sulfur compounds.

A high vacuum gauge controller (G3397B) is integrated in the system. Its use is **required** for CI, and it is recommended for EI also.

The CI system has been optimized to achieve the relatively high CI source pressure required for CI while still maintaining high vacuum in the quadrupole and detector. 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 CI interface has special plumbing for reagent gas. A spring-loaded insulating seal fits onto the end of the GC/MSD interface.

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

Important Safety Warnings

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

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
- 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.

If one of the primary fuses has failed, the MSD will already be off, but for safety, 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.

Electrostatic discharge is a threat to MSD electronics

The printed circuit 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.

Many parts are dangerously hot

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

- The GC inlets
- The GC oven and its contents including the column nuts attaching the column to a GC inlet, GC/MS interface, or GC detector
- The GC detector
- The GC valve box
- The foreline pump
- The diffusion pump
- The heated MSD ion source, interface, and quadrupole

Always cool these areas of the system 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 GC emits hot exhaust which can cause burns.

WARNING

The insulation around the GC 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**

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

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

WARNING

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

Hydrogen Safety

WARNING

The use of hydrogen as a GC carrier gas, detector fuel gas, or in the optional JetClean system, is potentially dangerous.

WARNING

When using hydrogen (H₂) as the carrier gas or fuel gas, be aware that hydrogen can flow into the GC oven and create an explosion hazard. Therefore, ensure that the supply is turned off until all connections are made and that the inlet and detector column fittings are either connected to a column or capped when hydrogen is supplied to the instrument.

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

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

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

GC precautions

When using hydrogen as a carrier gas, remove the large round plastic cover for the MSD transfer line located on the GC left side panel. In the unlikely event of an explosion, this cover may dislodge.

Dangers unique to GC/MSD operation

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

- Combustion of leaking hydrogen
- Combustion due to rapid expansion of hydrogen from a high-pressure cylinder
- Accumulation of hydrogen in the GC oven and subsequent combustion (see your GC documentation and the label on the top edge of the GC oven door)
- Accumulation of hydrogen in the MSD and subsequent combustion

Hydrogen accumulation in an MSD

WARNING

The MSD cannot detect leaks in inlet 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.

WARNING

The MS cannot detect leaks in the valves for the optional JetClean system. It is possible that hydrogen can leak into the MS from this cleaning system. Always turn off the JetClean system, close the manual hydrogen shutoff valve to the JetClean MFC, and ensure good vacuum before venting the MS.

All users should be aware of the mechanisms by which hydrogen can accumulate and know what precautions to take if they know or suspect that hydrogen has accumulated. (See [Table 4](#) on page 25.) Note that these mechanisms apply to **all** mass spectrometers, including the MSD.

Table 4 Hydrogen accumulation mechanisms

| Mechanism | Results |
|---|--|
| Mass spectrometer turned off | A mass spectrometer can be shut down deliberately. It can also be shut down accidentally by an internal or external failure. There is a safety feature that will shut down the flow of carrier gas in the event of an MSD foreline pump shut down. However, if this feature fails, hydrogen may slowly accumulate in the mass spectrometer. |
| MS automated shutoff valves closed | The mass spectrometers are equipped with automated shutoff valves for the calibration vial, optional JetClean system, and the reagent gases. Deliberate operator action or various failures can cause the shutoff valves to close. Shutoff valve closure does not shut off the flow of carrier gas. As a result, hydrogen may slowly accumulate in the MS. |
| Mass spectrometer automated shutoff valves closed | Some mass spectrometers are equipped with automated diffusion pump shutoff valves. In these instruments, deliberate operator action or various failures can cause the shutoff valves to close. Shutoff valve closure does not shut off the flow of carrier gas. As a result, hydrogen may slowly accumulate in the mass spectrometer. |
| Mass spectrometer manual shutoff valves closed | Some mass spectrometers are equipped with manual diffusion pump shutoff valves. In these instruments, the operator can close the shutoff valves. Closing the shutoff valves does not shut off the flow of carrier gas. As a result, hydrogen may slowly accumulate in the mass spectrometer. |
| GC off | A GC can be shut down deliberately. It can also be shut down accidentally by an internal or external failure. Different GCs react in different ways. If a 7890 Series GC equipped with Electronic Pressure Control (EPC) is shut off, the EPC stops the flow of carrier gas. If a GC's carrier flow is not under EPC control, the flow increases to its maximum. This flow may be more than some mass spectrometers can pump away, resulting in the accumulation of hydrogen in the mass spectrometer. If the mass spectrometer is shut off at the same time, the accumulation can be fairly rapid. |
| Power failure | If the power fails, both the GC and mass spectrometer shut down. The carrier gas, however, is not necessarily shut down. In some GCs, a power failure may cause the carrier gas flow to be set to maximum. As a result, hydrogen may accumulate in the mass spectrometer. |

WARNING

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

WARNING

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

Precautions

Take the following precautions when operating a GC/MSD system or GC/MS system with hydrogen carrier gas or when operating the MSD with the JetClean option that supplies hydrogen to the MSD analyzer.

WARNING

You must remove the plastic cover over the glass window on the front of a 5977B Series MSD. In the unlikely event of an explosion, this cover may dislodge.

WARNING

You MUST make sure the top thumbscrew on the analyzer side plate is fastened finger-tight. Do not over tighten the thumbscrew; this can cause air leaks.

WARNING

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

General laboratory precautions

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

Operating precautions

- Turn off the hydrogen at its source every time you shut down the GC or MSD.
- Turn off the hydrogen at its source every time you vent the MSD. (Do not heat the capillary column without carrier gas flow.)
- Turn off the hydrogen at its source every time shutoff valves in an MSD are closed. (Do not heat the capillary column without carrier gas flow.)
- Turn off the hydrogen at its source if a power failure occurs.

- If a power failure occurs while the GC/MSD system is unattended, even if the system has restarted by itself:
 - 1 Immediately turn off the hydrogen at its source.
 - 2 Turn off the GC.
 - 3 Turn off the MSD, and allow it to cool for 1 hour.
 - 4 Eliminate **all** potential sources of ignition in the room.
 - 5 Open the vacuum manifold of the MSD to atmosphere.
 - 6 Wait at least 10 minutes to allow any hydrogen to dissipate.
 - 7 Start up the GC and MSD as normal.


When using hydrogen, 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. Ensure the foreline pump exhaust and GC injection port vents are both vented into a fume hood.

Safety and Regulatory Certifications

The 5977B Series MSD conforms to the following safety standards:

- Canadian Standards Association (CSA): CAN/CSA-C222 No. 61010-1-04
- CSA/Nationally Recognized Test Laboratory (NRTL): UL 61010-1
- International Electrotechnical Commission (IEC): 61010-1
- EuroNorm (EN): 61010-1

The 5977B Series MSD conforms to the following regulations on Electromagnetic Compatibility (EMC) and Radio Frequency Interference (RFI):

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

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



The 5977B Series MSD is designed and manufactured under a quality system registered to ISO 9001.

The 5977B Series MSD is RoHS compliant.

Information

The Agilent Technologies 5977B Series MSD meets the following IEC classifications: Equipment Class I, Laboratory Equipment, Installation Category II, Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards. It 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 MSD has been compromised, disconnect the unit from all power sources, and secure the unit against unintended operation.

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

Symbols

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

See accompanying instructions for more information.



Indicates a hot surface.



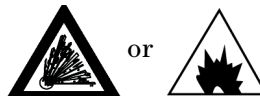
Indicates hazardous voltages.



Indicates earth (ground) terminal.



Indicates potential explosion hazard.



Indicates radioactivity hazard.



Indicates electrostatic discharge hazard.



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



Electromagnetic compatibility

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

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

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment on and off, 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 Ensure that all peripheral devices are also certified.
- 5 Ensure 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.

Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

Sound emission declaration

Sound pressure

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

Sound pressure L_p <70 dB according to EN ISO 3744:1995

Intended Use

Agilent products must be used only in the manner described in the Agilent product user guides. Any other use may result in damage to the product or personal injury. Agilent is not responsible for any damages caused, in whole or in part, by improper use of the products, unauthorized alterations, adjustments, or modifications to the products, failure to comply with procedures in Agilent product user guides, or use of the products in violation of applicable laws, rules, or regulations.

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.

Accidental Liquid Spillage

Do not spill liquids on the MSD. If liquid is accidentally spilled on the MSD, first, cut the power. Once the MSD is disconnected from all power sources, dry all affected parts. If the liquid spillage affects the electronics, wait at least 24 hours, depending upon the ambient humidity. While waiting for the parts to dry, please call your local Agilent service representative.

Moving or Storing the MSD

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, 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 Replace the Primary Fuses

Materials needed

- Fuse, T12.5A, 250 V (2110-1398) – 2 required
- Screwdriver, flat-blade (8730-0002)

The most likely cause of primary fuse failure is a problem with the foreline pump. If the primary fuses in your MSD fail, check the foreline pump.



Procedure

- 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, 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 carrier gas or for the JetClean system, the hydrogen 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 “Hydrogen Safety” on page 23 before operating the MSD with hydrogen gas.

- 2 Turn one of the fuse holders counterclockwise until it pops out. The fuse holders are spring loaded. (See [Figure 2](#) on page 33.)
- 3 Remove the old fuse from the fuse holder.
- 4 Install a new fuse in the fuse holder.
- 5 Reinstall the fuse holder.

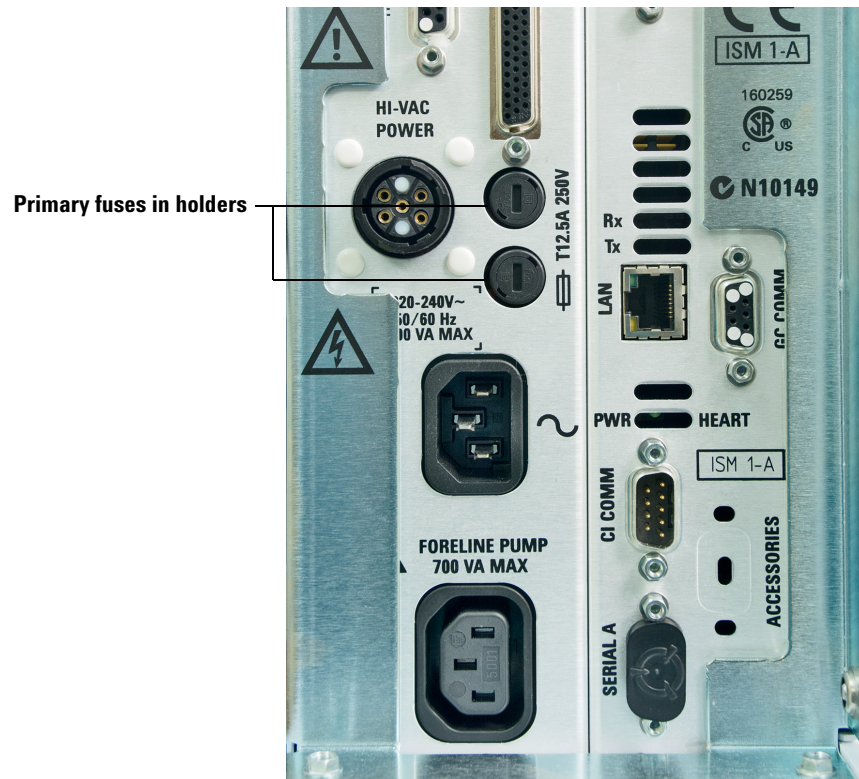
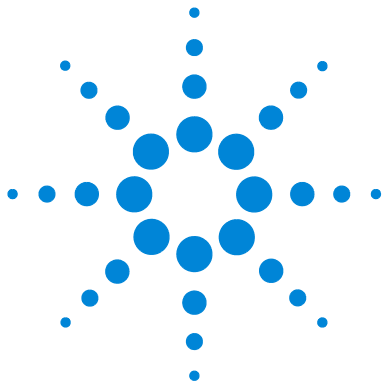


Figure 2 Primary fuses

- 6 Repeat steps 3 through 5 for the other fuse. Always replace both fuses.
- 7 Reconnect the MSD power cord to the electrical outlet.
- 8 Pumpdown the MSD.

1 Introduction



2 Installing 7890 GC Columns

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| To Condition a Capillary Column | 41 |
| To Install a Capillary Column in the GC/MS Interface Using the Self-Tightening Column Nut | 42 |
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| To Calibrate Column Flow Linear Velocity | 52 |
| The GC/MSD Interface for a 7890B Series GC | 55 |

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



Columns

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, diameter, and flow. Exceeding recommended flow results in degradation of mass spectral and sensitivity performance.

Remember that column flows vary greatly with oven temperature. (See “[To Calibrate Column Flow Linear Velocity](#)” on page 52 for instructions on how to measure actual flow in your column.) Use the Flow Calculation software and [Table 5](#) to determine whether a given column will give acceptable flow with realistic head pressure.

Table 5 Gas flows

| Feature | Gas flows | |
|--------------------------------------|-------------------|-------------------|
| High vacuum pump | Diffusion | Turbo |
| Optimal He column flow mL/min | 1 | 1 to 2 |
| Maximum recommended gas flow mL/min* | 1.5 | 4 |
| Maximum gas flow, mL/min† | 2 | 6.5 |
| Max column id | 0.53 mm (30 m) | 0.53 mm (30 m) |
| CI capability | No | Yes |
| CI reagent flows, mL/min | NA | 1 to 2 |
| JetClean option H ₂ flow | NA | 0.4 mL/min |

* Total gas flow into the MSD: column flow plus reagent gas flow (if applicable) plus JetClean H₂ flow (if applicable). Based on helium gas use. For other gases the maximum flow will vary.

† Expect degradation of spectral performance and sensitivity.

Conditioning columns

Conditioning a column before it is connected to the GC/MSD interface is essential. (See “[To Condition a Capillary Column](#)” on page 41.)

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

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

Conditioning ferrules

Heating ferrules to their maximum expected operating temperature a few times before they are installed can reduce chemical bleed from the ferrules. Thermal cycling ferrules to their maximum operating temperatures, prior to running your application, will help reduce leaks from the assembly.

Tips and hints

- The column installation procedures for the 5977B Series MSDs may be different from that for previous MSDs. Using the procedure from another instrument may *not* work, and may damage the column or the MSD.
- Always use carrier gas that is at least 99.9995% pure.
- Because of thermal expansion, new ferrules may loosen after heating and cooling a few times. Check for tightness after two or three heating cycles or use the self tightening column nuts.
- 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 or for the JetClean system, the hydrogen 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 “[Hydrogen Safety](#)” on page 23 before operating the MSD with hydrogen gas.

WARNING

Always wear safety glasses when handling capillary columns. Avoid puncturing your skin with the end of the column.

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 and 5/16-inch (8710-0510)
- Capillary column
- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- 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)
 - 0.5-mm id, for 0.32-mm id columns (5062-3514)
 - 0.8-mm id, for 0.53-mm id columns (5062-3512)
- Inlet column nut (5181-8830 for Agilent 7890 Series and 7820)
- Magnifying loupe
- Septum (may be old, used inlet septum)

To install columns in other types of inlets, refer to your GC User Information.

WARNING

The GC operates at high temperatures. Do not touch any parts of the GC until you are sure they are cool.

WARNING

Always wear safety glasses when handling capillary columns. Avoid puncturing your skin with the end of the column.

CAUTION

Always wear clean gloves while handling any parts that go inside the GC or analyzer chambers.



Procedure

- 1 Cool the oven and inlet to room temperature.
- 2 Wearing clean gloves, press the column through the septum (this takes a bit of pressure). Then slide the column nut and conditioned ferrule onto the free end of the column. (See [Figure 3](#).) The tapered end of the ferrule should point away from the column nut.

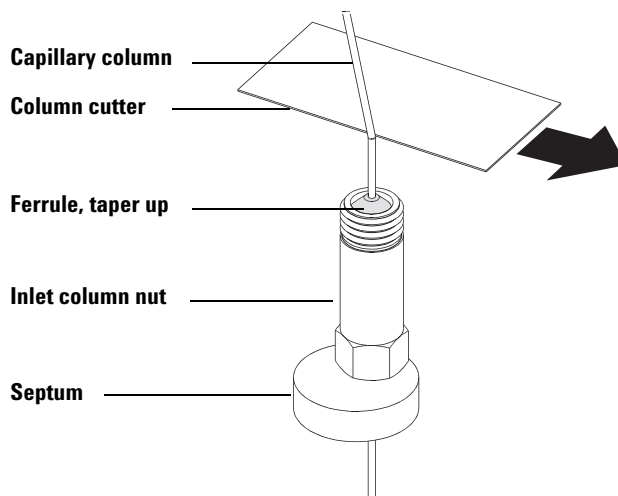


Figure 3 Preparing a capillary column for installation

- 3 Use the column cutter to score the column 2 cm or more from the end.
- 4 While holding the column, break the column end off at the score.
- 5 Inspect the end for jagged edges or burrs. If the break is not clean and even, repeat steps 3 and 4.
- 6 Wipe the outside of the free end of the column with a lint-free cloth moistened with methanol.

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

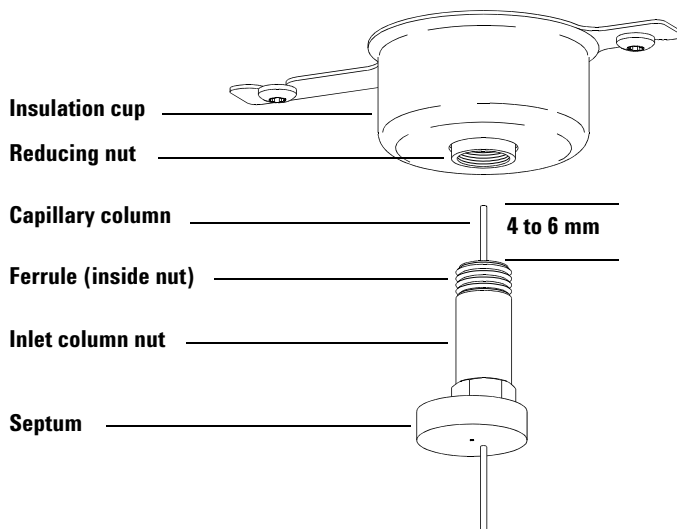


Figure 4 Installing a capillary column for a split/splitless inlet

- 8 Slide the septum up to the bottom of the nut to fix the correct column insertion length.
- 9 Insert the column in the inlet.
- 10 Slide the nut up the column to the inlet base, and finger-tighten the nut.
- 11 Adjust the column position so the septum is even with the bottom of the column nut.
- 12 Tighten the column nut an additional 1/4 to 1/2 turn. The column should not slide with a gentle tug.
- 13 Start carrier gas flow.
- 14 Verify flow by submerging the free end of the column in isopropanol. Look for bubbles.

See also

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

To Condition a Capillary Column

Materials needed

- Carrier gas, (99.9995% pure or better)
- Wrench, open-end, 1/4-inch and 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.

WARNING

The GC operates under high temperatures. Do not touch any GC parts unless you are certain they are cool.



Procedure

- 1 Install the column in the GC inlet. (See “To Install a Capillary Column in a Split/Splitless Inlet” on page 38.)
- 2 Set a minimum velocity of 30 cm/s, or as recommended by the column manufacturer. Allow the carrier gas to flow through the column at room temperature for 15 to 30 minutes to remove air.
- 3 Program the oven from room temperature to the maximum temperature limit for the column.
- 4 Increase the temperature at a rate of 10 to 15 °C/min.
- 5 Hold at the maximum temperature for 30 minutes.

CAUTION

Never exceed the maximum column temperature, either in the GC/MS interface, the GC oven, or the inlet.

- 6 Set the GC oven temperature to 30 °C, and wait for the GC to become ready.
- 7 Attach the column to the GC interface. (See “To Install a Capillary Column in the GC/MS Interface Using the Self-Tightening Column Nut” on page 42.)

To Install a Capillary Column in the GC/MS Interface Using the Self-Tightening Column Nut

This procedure is for the installation of a capillary column directly into the analyzer using the Agilent recommended self-tightening column nut.

Materials needed

- Interface tip seal (G3870-20542) (See [Figure 6](#) on page 44.) (Not used with EI SS or Inert source)
- Tip seal spring (G7005-20024)
- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Flashlight
- Magnifying loupe
- Gloves, clean
 - Large (8650-0030)
 - Small (8650-0029)
- Self Tightening column nut for GC\MS interface (5190-5233)
- Ferrules, Vespel
 - 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)
 - 0.5 mm id, for 0.32 mm id columns (5062-3514)
 - 0.8 mm id, for 0.53 mm id columns (5062-3512)
- Septum (may be old, used inlet septum)
- Safety glasses



Procedure

CAUTION

Always wear clean gloves while handling any parts that go inside the GC or the analyzer chambers.

WARNING

The analyzer, GC/MS 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.

1 Condition the column. (See “To Condition a Capillary Column” on page 41.)

WARNING

Dangerous voltages exist inside the analyzer chamber, which can result in fatal injury. Do not open the analyzer chamber door for any reason. If access is required, trained service personnel must first disconnect the instrument from the building power source.

2 Vent the MSD and open the analyzer chamber. (See “To Vent the MSD” on page 113 and “To Open the Analyzer Chamber” on page 152.) Be sure you can see the end of the GC/MS interface.

WARNING

The GC operates under high temperatures. Do not touch any GC parts until you are certain they are cool.

- 3 If installed, remove the isolation tip from the end of the GC/MS interface.
- 4 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.
- 5 Slide the column into the GC/MS interface.
- 6 Use the column cutter to score the column 2 cm from its end.
- 7 While holding the column against the column cutter with your thumb, break the column against the edge of the column cutter.
- 8 Inspect the end for jagged edges or burrs. If the break is not clean and even, repeat steps 6 and 7.
- 9 Wipe the end with alcohol.

2 Installing 7890 GC Columns

10 Adjust the column so it extends this specified distance from the end of the transfer line.

For an EI XTR, SS, Inert or CI Source Installation (See [Figure 5.](#)), the column extends about 1 mm.

For an EI HES Installation (See [Figure 6.](#)), the column extends 4 to 5 mm.

Use the flashlight and magnifying loupe, if necessary, to see the end of the column inside the analyzer chamber. Do not use your finger to feel for the column end.



Figure 5 Installing a capillary column in the GC/MS interface for an EI EXT, SS, Inert, or CI source.

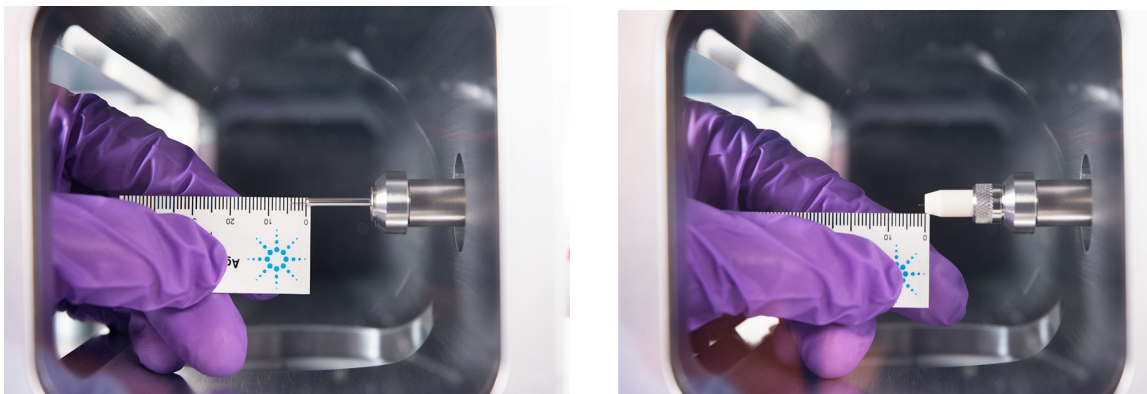


Figure 6 Installing a capillary column in the GC/MS interface for an EI HES source.

- 11 Hand-tighten the nut. Ensure the position of the column does not change as you tighten the nut. Do not over tighten the nut.
- 12 Tighten the nut in the clockwise direction. Continue to tighten until you feel the ferrule grip the column.
- 13 Check the GC oven to ensure that the column does not touch the oven walls.



Figure 7 Installing a capillary column in the GC/MS interface

CAUTION

Use care when placing the isolation tip on the GC/MS interface to avoid damaging the column.

- 14 Install the tip seal and spring on the GC/MS interface. (See “[To Install the Interface Tip Seal](#)” on page 50.) Do not use a tip seal or spring with an EI SS or Inert source. The interface socket on these sources does not allow the tip seal installation to fit. (See [Figure 56](#) on page 175.)

15 *Gently* check the alignment of the ion source and the interface tip seal.

When the ion source is aligned correctly, the analyzer chamber can be closed all the way with no resistance except the spring tension from the interface tip seal.

CAUTION

Forcing the analyzer door closed if these parts are misaligned will damage the tip seal or the interface or the ion source, or will keep the sideplate from sealing.

16 You can align the ion source and interface tip seal by wiggling the side plate on its hinge. If the door still will not close, contact your Agilent Technologies service representative.

17 Close the analyzer chamber door. (See “[To Close the Analyzer Chamber](#)” on page 196.)

To Install a Capillary Column in the GC/MS Interface Using a Standard Column Nut

This procedure is for the installation of a capillary column directly into the analyzer. There are two types of column nuts that can be used in the GC/MS interface: The standard column nut explained here, and the self tightening column nut explained in [“To Install a Capillary Column in the GC/MS Interface Using the Self-Tightening Column Nut”](#) on page 42.

Materials needed

- Interface tip seal (G3870-20542) (See [Figure 5](#) on page 44 and [Figure 6](#) on page 44.) (Not used with EI SS or Inert source)
- Tip seal spring (G7005-20024) (Not used with SS or Inert EI source)
- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Flashlight
- Magnifying loupe
- Gloves, clean
 - Large (8650-0030)
 - Small (8650-0029)
- Interface column nut (05988-20066)
- 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 (5062-3512)
- Septum (may be old, used inlet septum)
- Safety glasses
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)



Procedure

CAUTION

Always wear clean gloves while handling any parts that go inside the GC or the analyzer chambers.

WARNING

The analyzer, GC/MS 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.

1 Condition the column. (See “[To Condition a Capillary Column](#)” on page 41.)

WARNING

Dangerous voltages exist inside the analyzer chamber, which can result in fatal injury. Do not open the analyzer chamber door for any reason. If access is ever required, trained service personnel must first disconnect the instrument from the building power source.

2 Vent the MSD and open the analyzer chamber. (See “[To Vent the MSD](#)” on page 113 and “[To Open the Analyzer Chamber](#)” on page 152.) Be sure you can see the end of the GC/MS interface.

WARNING

The GC operates under high temperatures. Do not touch any GC parts unless you are certain they are cool.

- 3 If installed, remove the isolation tip from the end of the GC/MS interface.
- 4 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.
- 5 Slide the column into the GC/MS interface.
- 6 Use the column cutter to score the column 2 cm from the end.
- 7 While holding the column against the column cutter with your thumb, break the column against the edge of the column cutter.
- 8 Inspect the end for jagged edges or burrs. If the break is not clean and even, repeat steps 6 and 7.
- 9 Wipe the end with alcohol.
- 10 Adjust the column so it extends this specified distance from the end of the transfer line.

For an XTR, SS, Inert, or CI Source Installation (See [Figure 5](#) on page 44.), the column extends about 1 mm.

For an EI HES Installation (See [Figure 6](#) on page 44.), the column extends 4 to 5 mm.

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

- 11** Hand-tighten the nut. Ensure the position of the column does not change as you tighten the nut. Do not over tighten the nut.
- 12** Check the GC oven to be sure that the column does not touch the oven walls.
- 13** Tighten the nut 1/4 to 1/2 turn.
- 14** Check the nut's tightness after one or two heat cycles; tighten additionally as appropriate.

CAUTION

Use care when placing the isolation tip on the end of the GC/MS interface to avoid damaging the column.

-
- 15** Install the tip seal and spring on the GC/MS interface. (See [“To Install the Interface Tip Seal”](#) on page 50.) Do not use a tip seal or spring with an EI SS or Inert source. The interface socket on these sources does not allow the tip seal installation to fit. (See [Figure 56](#) on page 175.)

- 16** *Gently* check the alignment of the ion source and the interface tip seal.

When the ion source is aligned correctly, the analyzer chamber can be closed all the way with no resistance except the spring tension from the interface tip seal.

CAUTION

Forcing the analyzer door closed if these parts are misaligned will damage the tip seal or the interface or the ion source, or will keep the sideplate from sealing.

-
- 17** You can align the ion source and interface tip seal by wiggling the side plate on its hinge. If the door still will not close, contact your Agilent Technologies service representative.
 - 18** Close the analyzer chamber door. (See [“To Close the Analyzer Chamber”](#) on page 196.)

To Install the Interface Tip Seal

Materials needed

- Interface tip seal (G3870-20542)

The interface tip seal must be in place for the CI source, the EI XTR source, and HES.

CAUTION

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



Procedure

- 1 Verify that the CI source, EI XTR source, or HES is installed. This tip seal and its spring must not be installed when an EI SS or Inert source is installed.
- 2 Place the tip seal spring over the end of the interface.
- 3 Remove the interface tip seal from the ion source storage box, and place it over the end of the interface.

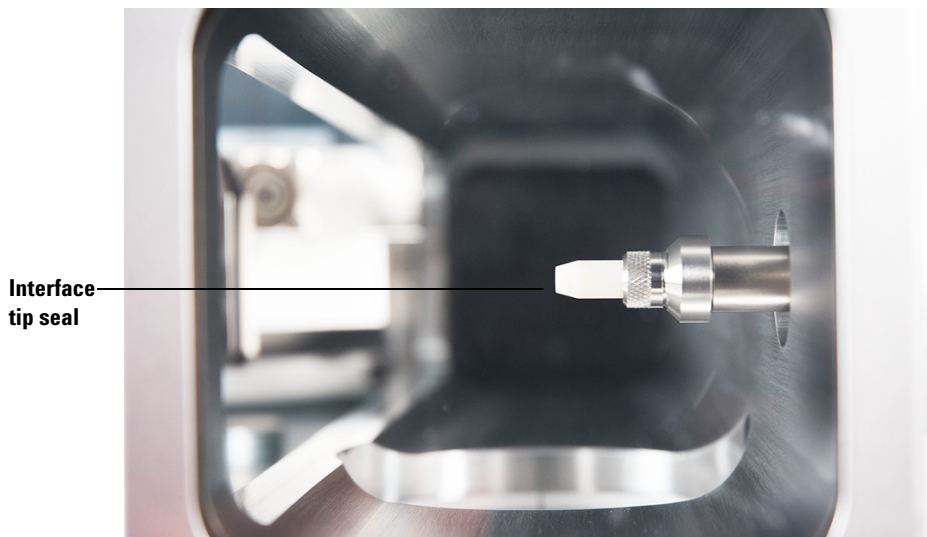


Figure 8 Interface tip seal

- 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 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 will not close, contact your Agilent Technologies service representative.

To Calibrate Column Flow Linear Velocity

The steps listed below apply to 7890 and 6890 series GCs only. Capillary columns must be calibrated prior to use with the MSD.

Procedure

- 1 Set Data Acquisition for splitless manual injection, and set up a real time plot to monitor m/z 28.
- 2 Press [**Prep Run**] on the GC keypad.
- 3 Inject 1 μ L of air into the GC inlet and press [**Start Run**].
- 4 Wait until a peak elutes at m/z 28. Note the retention time.
- 5 In the **Instrument Control** view, select **GC Parameters** from the **Instrument** menu.
- 6 Select the **Configuration** tab and then select the **Columns** tab.
- 7 Select your installed column from the table.
- 8 Click **Calibrate** to display the **Calibrate Column** dialog.

- 9 Click **Calc Length** in the **If unretained peak holdup time is known** section to display the **Calculate Column Length** dialog. (See Figure 9.)

Calculate Column Length

GC Conditions

If measurement was made under conditions different from loaded method, please enter them below.

Temperature: 75 °C

Pressure into column: 14.443 psi

Pressure out of column: 0 psi

Vacuum

Gas type: He

Holdup Time of an Unretained Peak: 1.1069 min

| | Current | Calculated |
|----------|------------|------------|
| ▶ Length | 30 m | 30 m |
| Diameter | 250 μm | 250 μm |
| Holdup | 1.1069 min | 1.1069 min |

OK Cancel

Figure 9 Calibrate column length dialog

- 10 Verify that the parameters listed (temperature, inlet and outlet pressures, and gas type) are those used in the method to determine the holdup time. Change any parameters that are different than those used in your method.
- 11 Enter the recorded retention time in the **Holdup Time** field. Move the cursor to another parameter's field and the calibrated column length appears.
- 12 Click **OK** to save the changes and exit the dialog.
- 13 Click **OK** on the **Calibrate Columns** dialog to save the calibration.

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

Calculation for average linear velocity

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

where:

L = Length of the column in meters

t = Retention time in seconds

Calculation for volumetric flow rate

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

where:

D = Internal column diameter in millimeters

L = Column length in meters

t = Retention time in minutes

The GC/MSD Interface for a 7890B Series GC

The GC/MSD interface is a heated conduit into the MSD for the capillary column. (See [Figure 10](#).) It is bolted onto the right side of the analyzer chamber, with an O-ring seal. It has a protective cover which should be left in place.

One end of the interface passes through the side of the GC and extends into the oven. This end is threaded to allow connection of the column with a nut and ferrule. The other end of the interface fits into the ion source. The end of the capillary column extends slightly past the end of the column guide tube and into the ionization chamber.

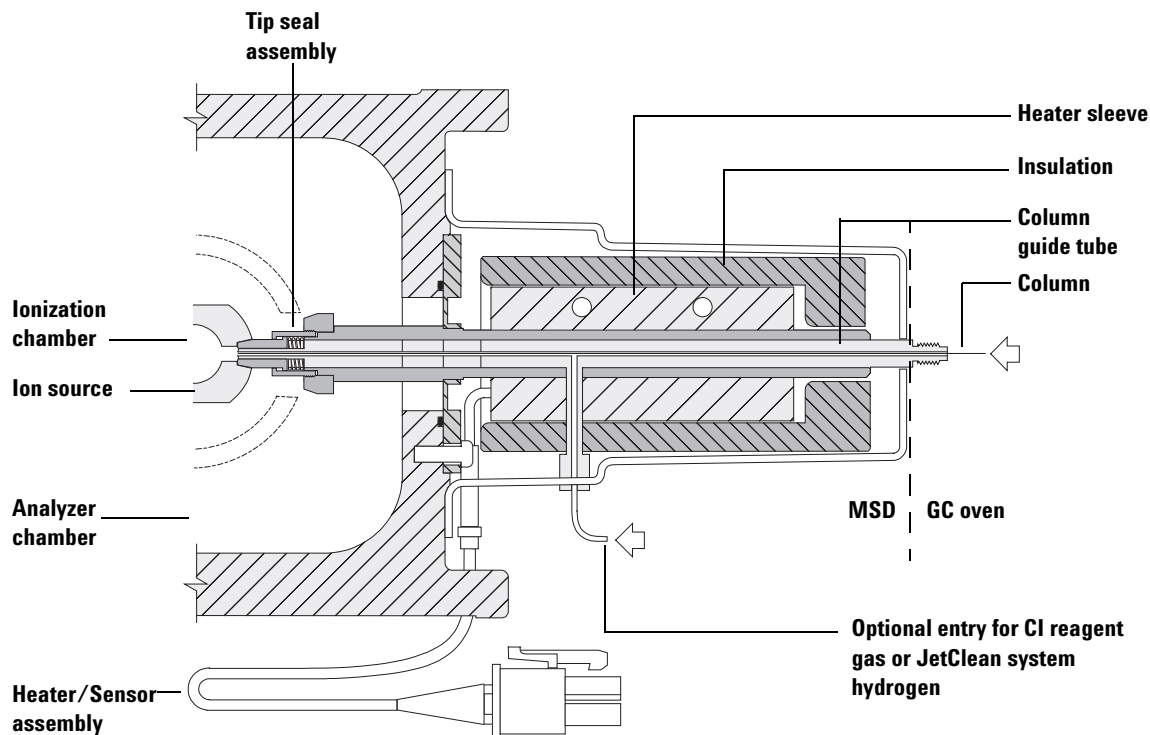


Figure 10 GC/MSD interface for a 7890B GC with EI XTR, HES, and CI sources

2 Installing 7890 GC Columns

The GC/MSD interface is heated by an electric cartridge heater. Normally, the heater is powered and controlled by Thermal Aux #2 heated zone of the GC. The interface temperature can be set from Agilent MassHunter GC/MS Acquisition software or from the GC. A thermocouple sensor in the interface monitors the temperature.

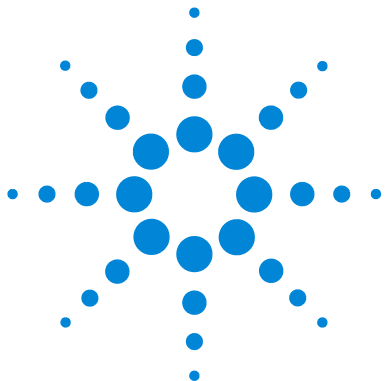
The GC/MSD interface should be operated in the range of 250 ° to 350 °C. The interface temperature should be slightly higher than the maximum GC oven temperature, but *never* higher than the maximum column temperature.

The GC/MSD interface can be used with both EI and CI sources. The EI XTR, HES, and CI sources require a tip seal assembly. The tip seal and spring interfere with, and must be removed when using the EI SS and Inert sources.

(See “To Install a Capillary Column in the GC/MS Interface Using the Self-Tightening Column Nut” on page 42, and “To Install a Capillary Column in the GC/MS Interface Using a Standard Column Nut” on page 47.)

WARNING

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



3 Intuvo 9000 GC Column Maintenance

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This chapter shows you how to install an Agilent Intuvo column, connect a flow path through the GC/MS Interface into the ion source, and maintain the column guard chip.

If you are using the *Agilent Intuvo 9000 Gas Chromatograph* with your MS, Chemical Ionization (CI) and the JetClean system are not currently supported.



Columns

Many types of Intuvo 9000 GC columns can be used with the MS, but there are some restrictions.

During tuning or data acquisition, the rate of column flow into the MS 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 temperature, requiring measurements of the actual flow to be taken. Use [Table 6](#) to determine an acceptable column flow.

Table 6 Gas flows

| Feature | Gas flows |
|---|-----------------------|
| High-vacuum pump | Turbo |
| Optimal He column gas flow, mL/min (carrier gas) | 1 to 2 |
| Maximum recommended gas flow, mL/min [*] | 4 to 6 |
| Maximum gas flow, mL/min [†] | 6.5 |
| Maximum column id | 0.53 mm (30 m length) |

* Total gas flow into the MS = column flow

† Expect degradation of spectral performance and sensitivity

Conditioning columns

Conditioning a column before it is connected to the GC/MS 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 MS ion source. This decreases MS 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/MS interface. (See “[To Condition an Intuvo Capillary Column](#)” on page 74.)

Tips and hints

- Always use carrier gas that is at least 99.9995% pure.
- Always wear clean gloves when handling a component's click and run connectors.
- Always wear clean gloves when handling a gasket.
- Always wear clean gloves when handling the 9000 GC/MS Tail.

WARNING

If you are using hydrogen as a carrier gas, the hydrogen gas flow must be off before turning off the MS power. If the foreline pump is off, hydrogen will accumulate in the MS and an explosion may occur. Read “[Hydrogen Safety](#)” on page 23 before operating the MS with hydrogen gas.

Handling the Intuvo 9000GC Column and Bus Components

The Agilent Intuvo 9000 Gas Chromatograph (Intuvo 9000 GC) does not use traditional ferrules and nuts for most column and flow path seals. In a traditional gas chromatography connection, the seal is made by deforming a soft ferrule around the periphery of a column or tube, with a second seal made between the ferrule and the fitting. Instead, the Intuvo 9000 GC click and run connections use a sealing system based on contact between flat surfaces. Compared to traditional ferrule seals, these connections are leak-free and easy to make.

When making these seals, follow a few simple guidelines:

- Do not touch the click and run sealing surfaces with bare skin or dirty gloves. Skin oils and dirt can contaminate the flow path surfaces.
- Use only the provided Intuvo 9000 GC torque driver to tighten Intuvo compression bolts.
- Avoid scratching or deforming click and run sealing surfaces.
- If you need to clean a sealing surface, use clean, compressed air.
- Use a new gasket each time you install a column or Intuvo chip.

To Replace an Intuvo GC Column

This procedure applies to GCs with a single column. For a 2 column replacement see the *Agilent Intuvo 9000 Gas Chromatograph Maintaining Your GC* manual.

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Tweezers (8710-2460)
- Intuvo gasket, polyimide 5/pk, for temperatures < 350 °C (5190-9072)
- Intuvo gasket, nickel 5/pk, for temperatures 350 °C to 450 °C (5190-9073)
- Intuvo torque driver stored on oven door (5190-9571)



Procedure

WARNING

The inlet, detector, bus components, and column can be hot enough to cause burns. Cool heated zones to a safe handling temperature before continuing.

CAUTION

Always wear clean gloves while handling any parts that are in the flow path going to the analyzer or parts that are located inside the analyzer.

- 1 Prepare the GC for maintenance.
From the GC panel select **Maintenance > Column > Perform Maintenance > Install Column > Start Maintenance**. This procedure cools the inlet, detector, column, Guard Chip, and other components in the flow path heated zones to < 40 °C and configures the GC. Follow the on-screen GC prompts.
- 2 From MassHunter Data Acquisition, Vent the MS (See “To Vent the MSD” on page 113.)
- 3 If you are using hydrogen or other flammable gas as a carrier gas, close the manual gas supply valve to the instrument before turning off the MS power.

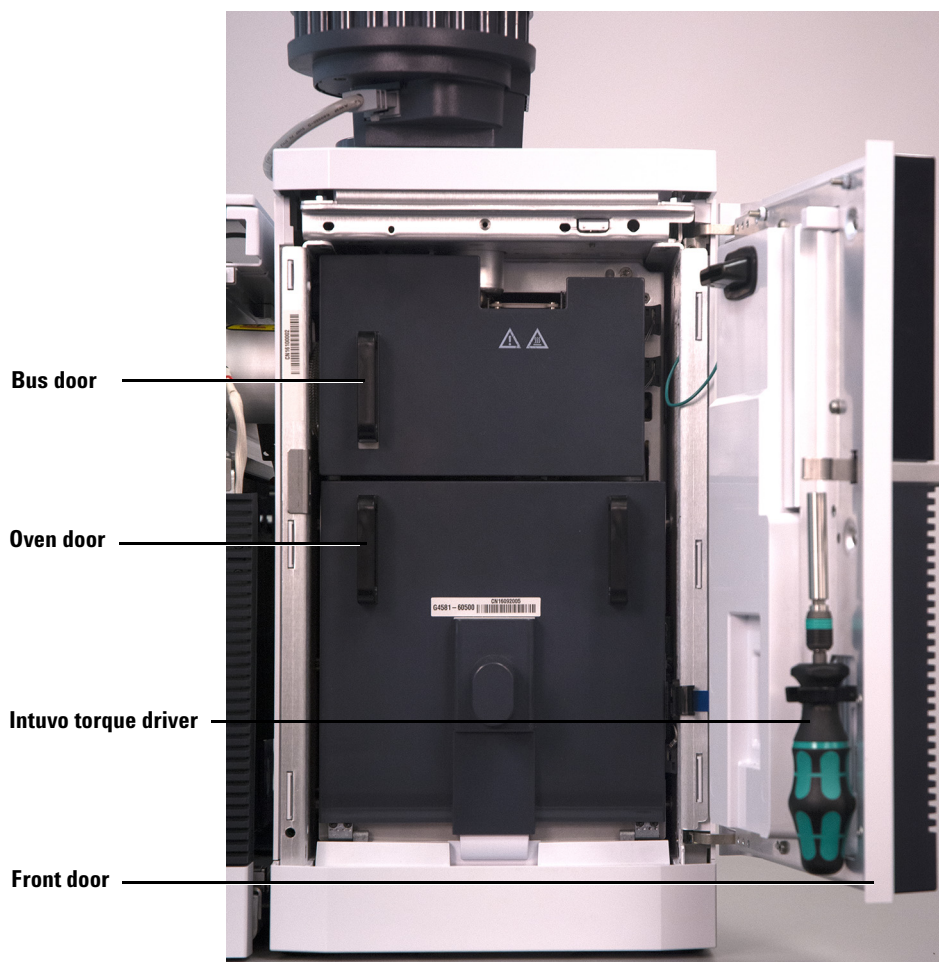


Figure 11 9000 GC front door, bus door, oven door, and Intuvo torque driver

- 4 Open the GC front door.
- 5 Open the bus door and remove it by lifting the door vertically off its hinge pins.
- 6 Lower the oven door.
- 7 Using the Intuvo torque driver, rotate all four column clamps off the column's retainer ring.

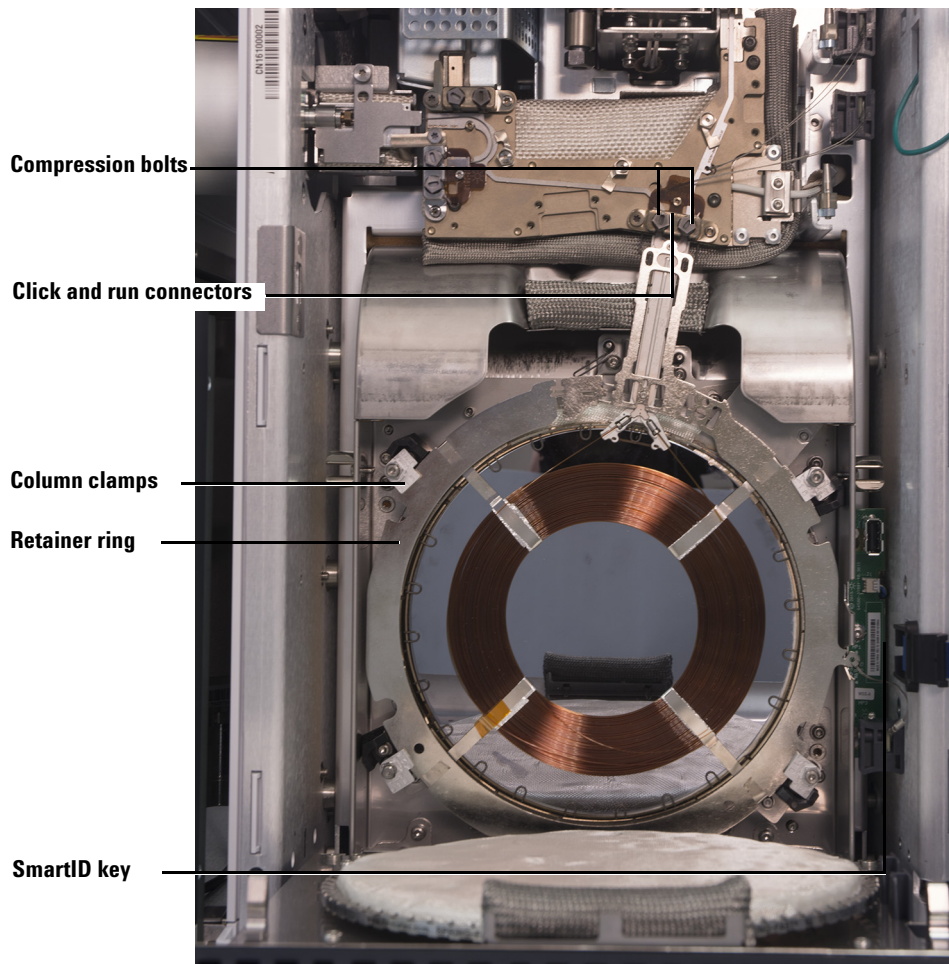


Figure 12 9000 GC column and related parts

- 8** Unplug the column SmartID key from the lower USB port.
- 9** Using the Intuvo torque driver, remove the two compression bolts that seal the column's click and run connectors to the bus and store for later use.
- 10** Remove and store the column for later use in accordance with the column manufacturer's recommendations.

- 11 Replace the gasket with a new gasket rated for the maximum column temperature expected with your method. (See “To Replace an Intuvo 9000 GC Gasket” on page 65.)
- 12 Verify that all Intuvo gaskets in the flow path are rated for the maximum column temperature expected with your method. Replace any gaskets that have a lower temperature rating with one that has a temperature rating that is compatible with your method.
- 13 Place the column’s click and run connectors into the single column (right) bus fitting. See the GC manual for installing 2 columns in the GC.
- 14 Insert the column's attached Intuvo SmartID Key into the lower USB connection shown.
- 15 Secure the new column by rotating the **1 c** tab of the 4 column clamps over the column retaining ring using the Intuvo torque driver.
- 16 Verify that the column’s click and run fittings sit flat against the gasket.
- 17 Loosely install the two compression bolts.

CAUTION

Use the Intuvo torque driver to tighten the compression bolt until you hear one click. Over-tightening can damage the flow path, strip the fittings, and cause leaks.

-
- 18 Tighten the compression bolts until you hear a click from the Intuvo torque driver.
 - 19 Close the column door.
 - 20 Install the bus door.
 - 21 Close the GC front door.

To Replace an Intuvo 9000 GC Gasket

This procedure assumes you have already removed the column, 9000 GC/MS Tail, or other part that sits on top of the gasket and that the instrument components are below 40 °C.

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Tweezers (8710-2460)
- Intuvo gasket, polyimide 5/pk, for temperatures < 350 °C (5190-9072)
- Intuvo gasket, nickel 5/pk, for temperatures 350 °C to 450 °C (5190-9073)

Procedure

WARNING

The inlet, detector, bus components, and column can be hot enough to cause burns. Cool heated zones to a safe handling temperature before continuing.

CAUTION

Always wear clean gloves while handling any parts that are in the flow path going to the analyzer or parts that are located inside the analyzer.

- 1 Remove the gasket tab from the alignment stud and discard the old gasket. Tweezers help when you are wearing the required gloved.
- 2 If needed, install any inlet or detector chips. All chips must be installed before installing the new gasket.
- 3 Carefully remove the new gasket from its packaging. Inspect the gasket to be sure it is not deformed. The two round lobes are the sealing surfaces.
- 4 Carefully insert the round gasket lobes into the bus click and run fitting. Note that the gasket is double-sided.
- 5 Place the hole in the gasket over the alignment stud in the bus fitting, and press the gasket body flat against the bus.
- 6 Check that the gasket's round lobes fit flat against the bus click and run fitting.

The new gasket is ready for the chip or column attachment.

To Install a Column Guard or Jumper Chip

The column Guard Chip and the Jumper Chip are both single-use, consumable parts. Installation deforms part of the Chip to make a good seal, so that a mis-installed Chip cannot be re-used. The Guard Chip cannot be cleaned or conditioned.

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Tweezers (8710-2460)
- Split/Splitless inlet Intuvo guard chip, 2/pk (G4587-60565)
- Multimode inlet Intuvo guard chip, 2/pk (G4587-60665)
- Split/Splitless inlet Intuvo jumper chip, 2/pk (G4587-60575)
- Multimode Inlet Intuvo jumper chip, 2/pk (G4587-60675)
- Intuvo gasket, polyimide 5/pk, for temperatures < 350 °C (5190-9072)
- Intuvo gasket, nickel 5/pk, for temperatures 350 °C to 450 °C (5190-9073)
- Intuvo torque driver stored on oven door (5190-9571)
- 7/16-inch open-end wrench



Procedure

WARNING

The inlet, detector, bus components, and column can be hot enough to cause burns. Cool heated zones to a safe handling temperature before continuing.

CAUTION

Always wear clean gloves while handling any parts that are in the flow path going to the analyzer or parts that are located inside the analyzer.

- 1 Prepare the GC for maintenance. From the GC panel select **Maintenance > Inlets > Guard Chip > Prepare for Maintenance > Replace Liner and Guard Chip > Start Maintenance**. This procedure cools the inlet, detector, column, Guard Chip, and other components in the flow path heated zones to < 40 °C and configures the GC. Follow the on-screen GC prompts.

- 2 From MassHunter Data Acquisition, Vent the MS (See “To Vent the MSD” on page 113.)
- 3 Wait until the GC reaches the ready state indicating the components are cooled below 40 °C and the instrument is vented before continuing the steps in this procedure.
- 4 If you are using hydrogen or other flammable gas as a carrier gas or for the JetClean system, close the manual gas supply valve to the instrument before turning off the MS power.
- 5 If installed, remove the ALS injector from the inlet.
- 6 Remove the inlet cover.

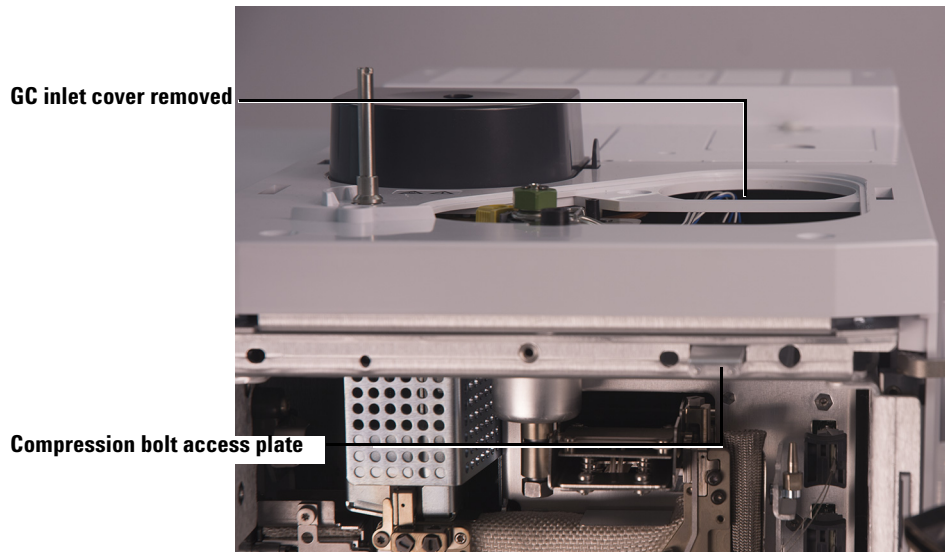


Figure 13 GC inlet cover and compression bolt access plate

- 7 Open the GC front door.
- 8 Open the bus door and remove it by lifting the door vertically off its hinge pins.
- 9 Pull the compression bolt access plate out to allow the torque driver to access the Guard chip compression bolt.

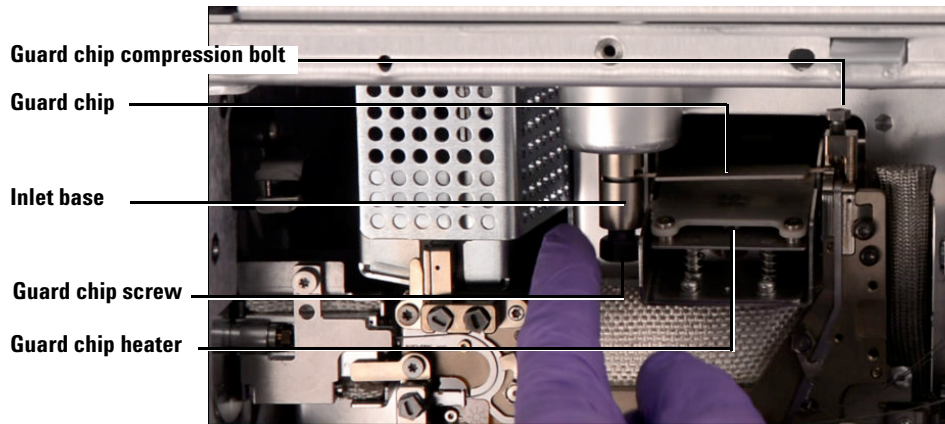


Figure 14 Guard chip and related parts

- 10** Use a 7/16- inch open-end wrench to loosen the Guard Chip screw at the base of the inlet.
- 11** Use your finger to gently rotate the front of the Guard Chip heater assembly down and expose the Guard Chip.
- 12** Loosen the Guard Chip compression bolt with the Intuvo torque driver.
- 13** Lift the right side of the Guard Chip tab over the boss then rotate it out of the bus connection.
- 14** Remove the left side of the Guard Chip from the inlet base.
- 15** Install a new Guard Chip. The larger end of the Guard Chip inserts into the inlet base first, then the smaller end is rotated into the bus connection, lifting its tab over the boss and into the bus mounting slot.
- 16** Finger tighten the compression bolt.
- 17** Raise the Guard Chip heater.
- 18** Finger tighten the Guard Chip screw in the inlet base.
- 19** Tighten the Guard Chip screw in the inlet base with a 7/16-inch open end wrench.

CAUTION

Use the Intuvo torque driver to tighten the compression bolt until you hear one click. Over-tightening can damage the flow path, strip the fittings, and cause leaks.

- 20 Tighten the Guard Chip compression bolt using the provided torque driver until you hear one click.
- 21 Install the inlet cover.
- 22 Install the bus door on its hinges and close it.
- 23 Close the GC front door.
- 24 If removed, install the ALS injector.

To Replace the 9000 GC/MS Tail

This procedure is necessary if you are switching to a source that requires a different 9000 GC/MS Tail, replacing a leaking gasket or a contaminated 9000 GC/MS Tail, or separating the 9000 GC from the MS.

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Tweezers (8710-2460)
- 9000 GC/MS Tail, used with a standard source (G4590-60009)
- 9000 GC/MS Tail, used with a HES (G4590-60109)
- Intuvo gasket, polyimide 5/pk, for temperatures < 350 °C (5190-9072)
- Intuvo gasket, nickel 5/pk, for temperatures 350 °C to 450 °C (5190-9073)
- Intuvo torque driver stored on oven door (5190-9571)
- 7/16-inch open-end wrench



Procedure

WARNING

The inlet, detector, bus components, and column can be hot enough to cause burns. Cool heated zones to a safe handling temperature before continuing.

CAUTION

Always wear clean gloves while handling any parts that are in the flow path going to the analyzer or parts that are located inside the analyzer.

- 1 Vent the MS (See “To Vent the MSD” on page 113.) When prompted, set the temperature for the ion source, quads, inlet, detector, column, Guard Chip, 9000 GC/MS Tail, and other components in the flow path heated zones to < 40 °C.
- 2 If you are using hydrogen or other flammable gas as a carrier gas, close the manual gas supply valve to the instrument before turning off the MS power.

- 3 Wait until the GC reaches the ready state indicating the components are cooled below 40 °C before continuing the steps in this procedure.
- 4 Open the analyzer chamber. (See “To Open the Analyzer Chamber” on page 152.)
- 5 Unscrew the tip seal from the tip seal holder and remove the tip seal and spring from the GC/MS interface. (See “Interface tip seal” on page 76.)
- 6 Ensure the MS is properly aligned with the GC. If the GC/MS is not properly aligned, the transfer line clamp screw will be difficult to tighten.
- 7 Open the GC front door.
- 8 Open the bus door and remove it by lifting the door vertically off its hinge pins.
- 9 Using the Intuvo torque driver, remove the compression bolt that secures the 9000 GC/MS Tail click and run bus connection. Also remove the bolt from the empty bus connection.

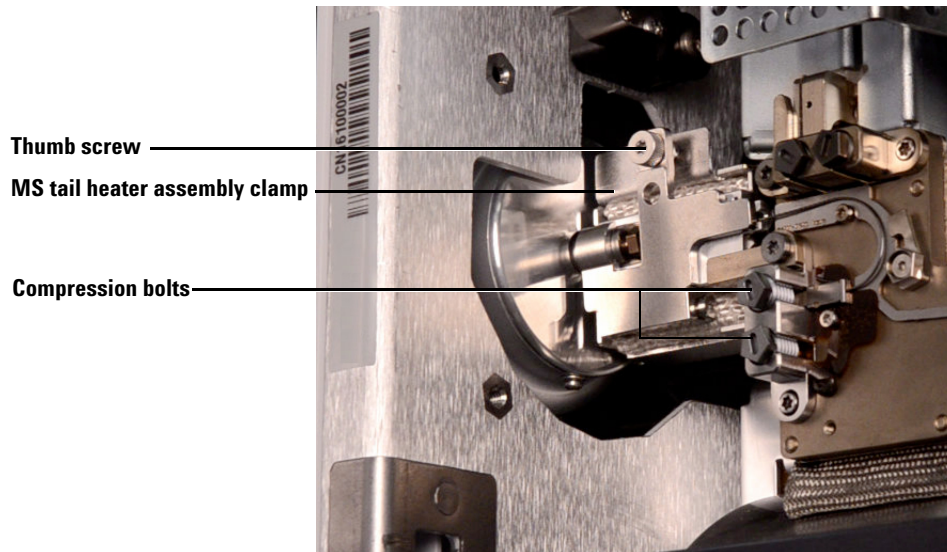


Figure 15 MS tail heater assembly clamp closed

- 10 Open the MS tail heater assembly by loosening the thumb screw at the top of the clamp and rotating the clamp down.

- 11 Push the MS tail heater assembly back a few millimeters. A magnet will hold the heater assembly away from the 9000 GC/MS Tail.

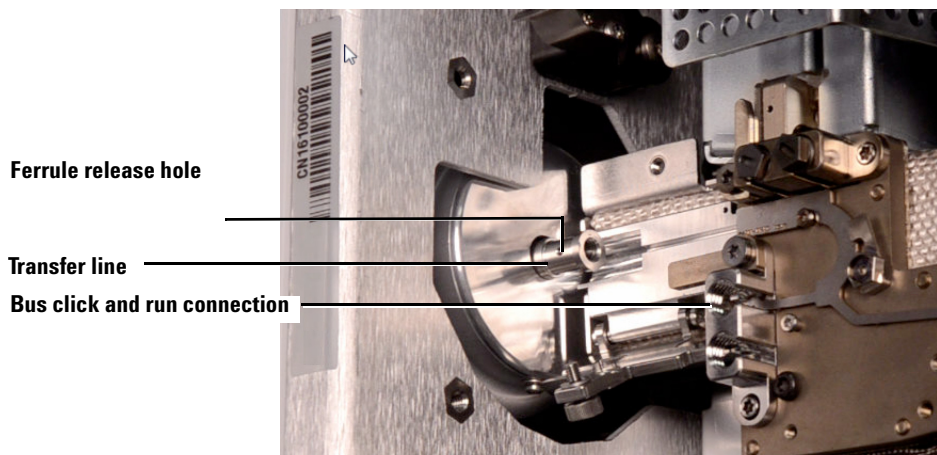


Figure 16 MS tail heater assembly clamp open with 9000 GC/MS Tail removed

- 12 Remove the 9000 GC/MS Tail from the transfer line and bus.
If the ferrule becomes stuck, press a pointed object, like a paper clip, into the ferrule release hole at the end of the transfer line.



Figure 17 9000 GC/MS Tail removed from GC/MS Interface

- 13 Replace the gasket. (See “To Replace an Intuvo 9000 GC Gasket” on page 65.)
- 14 Carefully slide the 9000 GC/MS Tail into the GC/MS interface, and gently place the click and run connection into the bus connection.
- 15 Check that the 9000 GC/MS Tail click and run fitting sits flat against the gasket in the bus connection.

- 16 Screw the 9000 GC/MS Tail column nut to the transfer line threaded connector finger tight, then use a ¼-inch wrench to tighten the nut an additional 20 to 30 degrees.
- 17 Loosely install the two compression bolts.

CAUTION

Use the Intuvo torque driver to tighten the compression bolt until you hear one click. Over-tightening can damage the flow path, strip the fittings, and cause leaks.

- 18 Tighten the single compression bolt until you hear a click from the Intuvo torque driver.
- 19 Loosely install the other compression bolt on the empty click and run bus connector.
- 20 Pull the MS tail heater assembly a few millimeters towards the 9000 GC/MS Tail until contact is made.
- 21 Close the MS tail heater assembly clamp, and hand tighten the thumb screw to secure it.
- 22 Install the bus door on its hinges and close it.
- 23 Close the GC front door.
- 24 Install the tip seal and spring on the GC/MS interface. Align, gently slide, and screw the tip seal into the tip seal holder. (See “[To Install the Interface Tip Seal](#)” on page 76.)
- 25 **Gently** check the alignment of the ion source and the interface tip seal.

When the ion source is aligned correctly, the front analyzer chamber can be closed all the way with no resistance except the spring tension from the interface tip seal.

CAUTION

Forcing the analyzer door closed if these parts are misaligned will damage the tip seal or the interface or the ion source, or will keep the sideplate from sealing.

- 26 You can align the ion source and interface tip seal by wiggling the side plate on its hinge. If the door still will not close, contact your Agilent Technologies service representative.
- 27 Close the analyzer chamber. (See “[To Close the Analyzer Chamber](#)” on page 196.)

To Condition an Intuvo Capillary Column

Materials needed

- Carrier gas, (99.9995% pure or better)
- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Tweezers (8710-2460)
- Intuvo torque driver (5190-9571)
- Intuvo gasket, polyimide 5/pk, for temperatures < 350 °C (5190-9072)
- Intuvo gasket, nickel 5/pk, for temperatures 350 °C to 450 °C (5190-9073)
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)

Procedure

WARNING

The inlet, detector, bus components, and column can be hot enough to cause burns. Cool heated zones to a safe handling temperature before continuing.

CAUTION

Always wear clean gloves while handling any parts that are in the flow path going to the analyzer or parts that are located inside the analyzer.

- 1 Install the column that requires conditioning. (See “To Replace an Intuvo GC Column” on page 61.)
- 2 Set a minimum velocity of 30 cm/s, or as recommended by the column manufacturer. Allow gas to flow through the column at room temperature for 15 to 30 minutes to remove air.
- 3 Increase the column temperature to 120 °C.
- 4 Hold at the this temperature for 30 minutes.
- 5 Use MassHunter to run an air and water check. Proceed to the next step if the air and water are within limits.
- 6 Program the column temperature to increase from 120 °C to the maximum temperature limit for the column at a rate of 10 to 15 °C/min.

CAUTION

Never exceed the maximum column temperature, in the GC/MS interface, the GC oven, or the inlet.

7 Hold at the maximum temperature for 30 minutes.

The column is conditioned and ready to be used with your method.

To Install the Interface Tip Seal

Materials needed

- Interface tip seal (G3870-20542)

The interface tip seal must be in place for the CI source, the EI XTR source, and HES.

CAUTION

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



Procedure

- 1 Verify that the CI source, EI XTR source, or HES is installed. This tip seal and its spring must not be installed when an EI SS or Inert source is installed. (See [Figure 18](#).)
- 2 Remove the interface tip seal, spring, and knurled nut retainer from the ion source storage box. In this order, slide the spring, tip seal, and knurled tip seal retainer over the column sleeve.
- 3 Thread the knurled tip seal retainer into the tip seal hold finger tight.

Interface tip seal

Knurled tip seal retainer

Tip seal holder

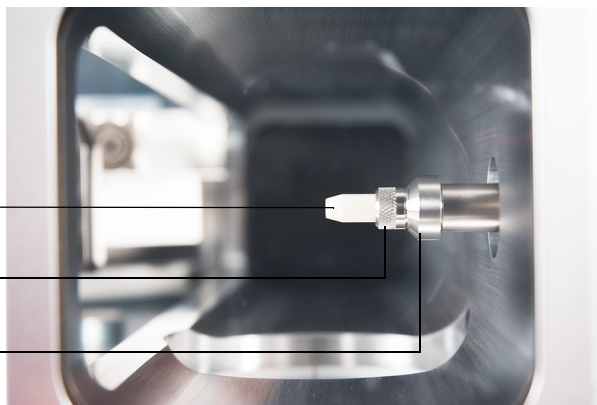


Figure 18 Interface tip seal

- 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 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 will not close, contact your Agilent Technologies service representative.

The GC/MS Interface for a 9000 Series GC

The GC/MS Interface is a heated conduit into the MS for maintaining MS vacuum and a suitable column effluent temperature. (See [Figure 19](#).) The GC/MS Interface is bolted onto the right side of the analyzer chamber, with an O-ring seal and has a protective cover which should be left in place.

One end of the interface passes through the side of the GC and is accessed from inside the GC front door. This allows connection of the 9000 GC/MS Tail column nut. The 9000 GC/MS Tail includes a swaged ferrule and column nut for attaching to the GC end of the GC/MS Interface. The 9000 GC/MS Tail transports the column effluent from a heated column bus connector in the GC, through the heated GC/MS Interface, and exits slightly past the end of the column guide tube and into the ionization chamber. The 9000 GC/MS Tail maintains temperature at several points along its length by a heated bus, an MS tail heater assembly, and the GC/MS Interface.

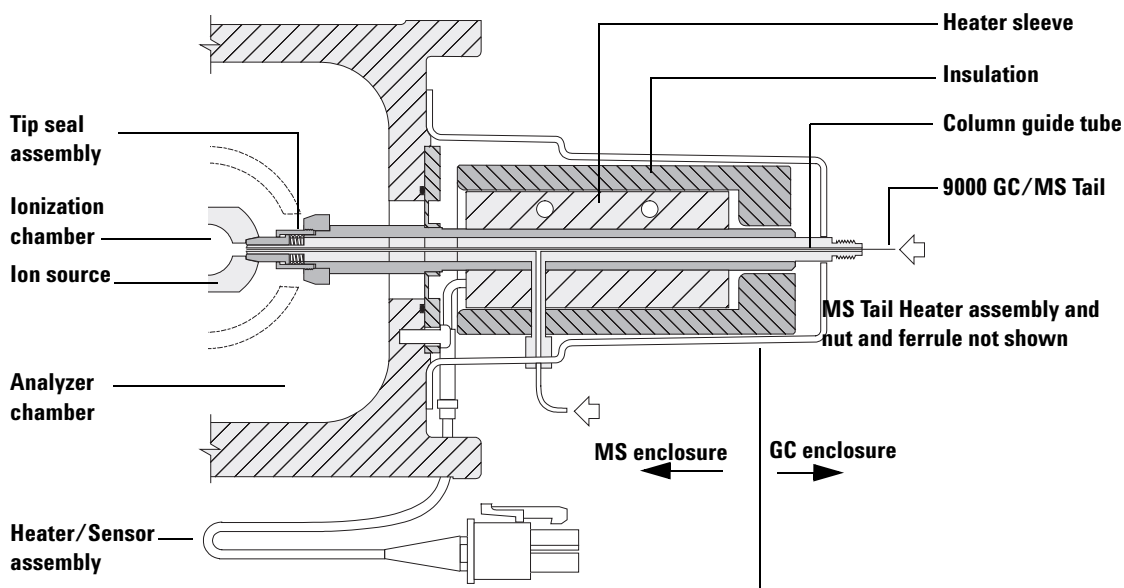
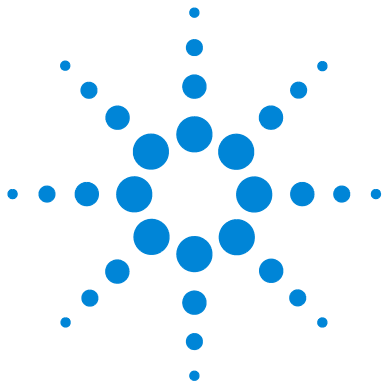


Figure 19 The GC/MS Interface for a 9000 GC (not to scale)

The 9000 GC/MS Tail is heated by an electric cartridge heater. The heater is powered and controlled by a heated zone of the 9000 GC. The 9000 GC/MS Tail temperature can be set from the Agilent MassHunter GC/MS Acquisition software or from the GC. A sensor (thermocouple) in the interface monitors the temperature.

The transfer line tip seal assembly is required when using the EI XTR source or the HES.

The 9000 GC/MS Tail should be operated in the 250 ° to 350 °C range. Subject to that restriction, the transfer line temperature should be slightly higher than the column temperature, but *never* higher than the maximum column temperature.



4 Operating in EI Mode

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This chapter describes how to perform some basic operating procedures for the Agilent 5977B Series GC/MSD using EI.

If you are using the *Agilent Intuvo 9000 Gas Chromatograph* with your MS, Chemical Ionization (CI) and the JetClean system are not currently supported.



Operating the MSD from the DS

The Agilent MassHunter GC/MS Acquisition software automates tasks such as pumping down, monitoring settings, setting temperatures, tuning, and venting the MSD. These tasks are described in this chapter. Additional information is described in the manuals and online help supplied with the Agilent MassHunter GC/MS Acquisition software.

CAUTION

The software and firmware are revised periodically. If the steps in these procedures do not match your Agilent MassHunter GC/MS Acquisition software, refer to the manuals and online help supplied with the software for more information.

Operating the MSD from the GC control panel

The GC control panel on a 7890A+ or 7890B GC can show the actual temperature and pressure of the MSD or initiate a task on the MSD without using the Agilent MassHunter GC/MS Acquisition software. You can access functions, such as venting and setting temperatures, right from the GC control panel. Limited features are available from the GC control panel. The Agilent MassHunter GC/MS Acquisition software is the full-featured controller for most instrument control operations.

MSD designated key on the GC control panel

The **MS/Aux Det** key on the 7890B GC control panel allows access to control and configuration parameters on the MSD. (See [Figure 20](#).) For 7890A+ models with updated firmware, this key is labeled **Aux Det #** which can be substituted for the **MS/Aux Det** key in the procedures that follow.



Figure 20 7890B GC control panel

To enable/disable LVDS on MSD

- 1 Press **Config**, and then press **MS/Aux Det** to bring up the CONFIGURE MS DETECTOR menu.
- 2 Press the down arrow to scroll to **LVDS communication**.
If you want to enable the LVDS, press **On/Yes**.
If you want to disable the LVDS, press **Off/No**.
- 3 Wait for the MSD to confirm the change on the GC control panel.

To change MSD temperatures from the GC control panel

- 1 Press **MS/Aux Det** to display the 5977B MSD menu.
- 2 Press the down arrow to scroll to **Quad temp, Source temp** or **Transfer line**.
- 3 Use GC keypad to enter the desired temperature.
- 4 Press **Enter** to apply the changes.

To view MSD vacuum pressure and Turbo speed/Foreline Pressure from the GC control panel

- 1 Press **MS/Aux Det** to display the 5977B MSD menu.
- 2 Press the down arrow to scroll to **HiVac Pressure**, or **Turbo Speed % of full / Foreline Pressure**.

To vent the MSD from the GC control panel

- 1 With the MSD pumped down, press **MS/Aux Det** to bring up the 5977B MSD menu.
- 2 Press the down arrow to scroll to **Start MSD Vent?**. (Press **Off/No** to cancel the vent cycle and pumpdown the MSD).
- 3 Press **On/Yes** to start the venting cycle.
- 4 When prompted, open the vent valve.

To pumpdown the MSD from the GC control panel

- 1 Press **MS/Aux Det** to display the 5977B MSD menu.
- 2 Press the down arrow to scroll to **Start MSD Pumpdown?**.
- 3 Press **On/Yes** to start the pumpdown cycle.

To view the firmware version of the MSD from the GC control panel

- 1 Press **MS/Aux Det** to bring up the 5977B MSD menu.
- 2 Press the down arrow to scroll to **Firmware**.

To view the serial number of the MSD from the GC control panel

- 1 Press **MS/Aux Det** to bring up the 5977B MSD menu.
- 2 Press the down arrow to scroll to **Serial#**.

To configure the network settings for the MSD from the GC control panel

- 1 Press **Config**, and then press **MS/Aux Det** to bring up the CONFIGURE MS DETECTOR menu.
- 2 To configure the **IP:** parameter, use the GC keypad to enter the new IP address for the MSD, then press **Enter** to complete the entry.
- 3 Wait for the GC to display the new IP address. Reboot the MSD or proceed to the gateway address with the down arrow.
- 4 Press the down arrow to scroll to **GW:** and use the GC keypad to enter the new gateway address for the LAN and press **Enter** to complete the entry.
- 5 Press the down arrow to scroll to **SW:** and use the GC keypad to enter the new subnet mask for the LAN and press **Enter** to complete the entry.
- 6 Reboot the MSD. (See below.)

To reboot the MSD from the GC control panel

- 1 Press **Config**, and then press **MS/Aux Det** to bring up the CONFIGURE MS DETECTOR menu.
- 2 Press the down arrow to scroll to **Request MSD Reboot?**.
- 3 Press **On/Yes** to reboot the MSD and wait for the MSD to complete this cycle before trying to access it. The LED turns red until the reboot cycle is completed.

To enable/disable BOOTP on MSD

By default, the BOOTP is disabled. If your LAN uses a BootP server, Enabling BOOTP causes the server to automatically assign an IP address to the MSD.

- 1 Press **Config**, and then press **MS/Aux Det** to bring up the CONFIGURE MS DETECTOR menu.
- 2 Press the down arrow to scroll to **MSD BOOTP**.
- 3 To enable BOOTP, press **On/Yes**.
To disable BOOTP, press **Off/No**.
- 4 Wait for the MSD to confirm the change on the GC control panel.
- 5 Reboot the MSD. (See “To reboot the MSD from the GC control panel” on page 83.)

Configuring the MSD through the Web User Interface (WUI)

If your GC does not support LVDS communications with an Agilent GC, you can use the WUI to configure the MSD network settings. Reasons that a GC does not support configuring a 5977B MSD's network settings from the GC control panel include any of the following:

- LVDS communication does not exist between the GC and MSD.
- The GC is not an Agilent 7890A+ or 7890B model with the correct firmware.

To change the network settings of the MSD

This procedure assumes that the operator has access to a PC located on the same LAN subnet as the MSD.

- 1** Open the top hinged cover on the MSD for accessing the analyzer to view the eModule mini display readout.
- 2** Press the **MSD On/Off** switch to start the instrument. When the instrument has completed its startup initialization it displays the current IP address information in the mini display readout and cycles through it for about 10 minutes.
- 3** Copy the IP address, gateway, and subnet mask from the mini display readout. The defaults for each are 192.168.254.12, 0.0.0.0, and 255.255.255.0 respectively.
- 4** Enter the IP address into a PC web browser URL to display the WUI page. (See [Figure 21](#) on page 86.)

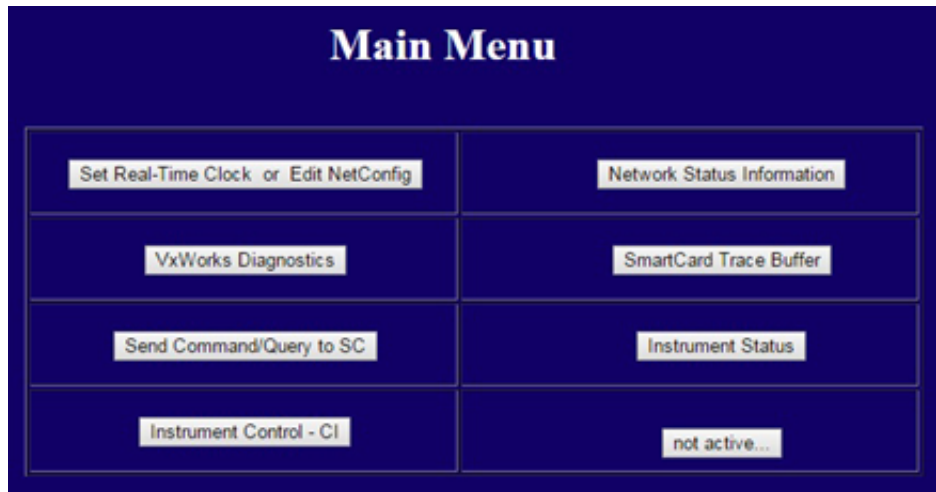


Figure 21 WUI main menu

- 5 Click **Set Real-Time Clock or Edit NetConfig**, and go to **Edit NetConfig (MSD network configuration)**. (See Figure 22.)

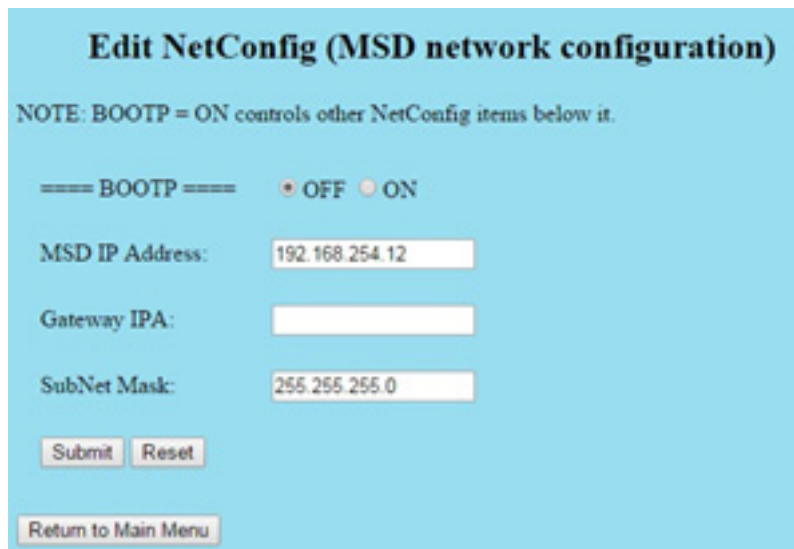


Figure 22 WUI Edit NetConfig

- 6 Confirm that **BootP** is set to **OFF**. If your LAN assigns IP addresses using a BootP server, click **ON** and skip the next step.
- 7 To update the **MSD IP address**, **Gateway IPA**, and **SubNet Mask** enter the new values. Before clicking submit, you can recover the previous settings by clicking **Return to Main Menu** and then returning here.
- 8 Click **Submit** to upload this net configuration to the MSD.

A dialog opens to confirm that the network configuration process has started. (See Figure 23.)

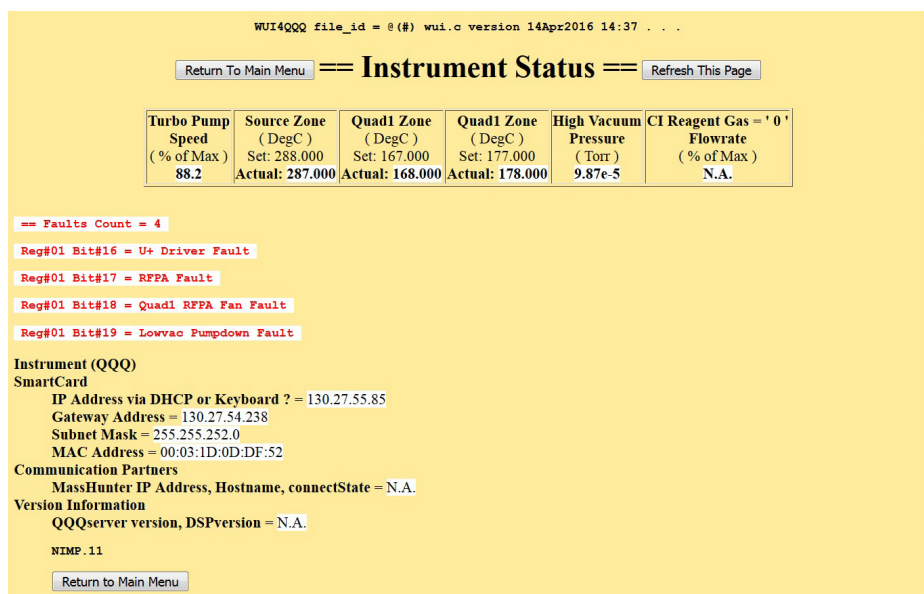


Figure 23 IP address configuration started message

- 9 Click **OK** to close the dialog and wait for the prompt requesting to **Manually reboot of the MSD/SmartCard to activate the new Settings**.
- 10 Use the **MSD on/off** switch to reboot the MSD SmartCard.

eModule Mini Display Readout

The eModule mini display, accessible when the analyzer door cover is open, allows the operator to view the LAN configuration of the instrument including its IP address, subnet mask, default gateway, and MAC address. This LAN configuration can be changed using the GC control panel or the web user interface (WUI) from a web browser.

Front Panel Instrument Status LED

Through the front panel instrument status LED, the operator can view the current status of the instrument using color codes and LED on/off timing. (See [Table 7.](#))

Table 7 Front panel Instrument Status LED codes

| Instrument status | LED code |
|---|-------------------------|
| Ready | Solid green |
| Acquiring data | Blinking green (<2 sec) |
| Not ready | Solid yellow |
| JetClean Acquire & Clean operation | Blinking magenta |
| JetClean Clean Only operation | Solid magenta |
| Ready and not connected to DS | Solid blue |
| Start up (prior to FW load) | Blinking red (<2 sec) |
| Fault | Solid red |

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 front side plate screw should not be tightened, unless hazardous carrier, JetClean system hydrogen, or reagent gases 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 GC inlet, and the oven are off.
- Carrier gas of at least 99.9995% purity is plumbed to the GC with the recommended traps. The regulator, plumbing, traps, EPC module, inlet, and column have been purged of any gas to remove oxygen from the system.
- If hydrogen is used as carrier gas, or JetClean system supply, the carrier gas and JetClean system supply shutoff valves must be closed, and the analyzer top 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. If using the standard foreline pump, it also contains traces of pump oil. If you are using toxic solvents or analyzing toxic chemicals, remove the oil trap (standard pump) and install a hose to take the foreline pump exhaust outside or to a fume (exhaust) hood. Be sure to comply with local regulations. The oil trap supplied with the standard pump stops only pump oil. It does not trap or filter out toxic chemicals.

WARNING

If hydrogen is used as a carrier gas or JetClean system supply, the carrier and JetClean system supply shutoff valves must be closed before turning off the MS power. If the foreline pump is off, hydrogen will accumulate in the MS and an explosion may occur. Read “Hydrogen Safety” on page 23 before operating the MSD with hydrogen gas.

Pumping Down

The DS or 7890B GC control panel helps you pumpdown the MSD. The process is primarily automated. After you turn on the MSD by pressing the **On/Off** switch, close the analyzer door, and close the vent valve once you hear hissing (while pressing on the sideplate), the MSD pumps down by itself. The Agilent MassHunter GC/MS Acquisition software monitors and displays system status during pumpdown. When the pressure is low enough, the program turns on the ion source and mass filter heaters and prompts you to turn on the GC/MSD interface heater. The MSD will shut down if it cannot pumpdown correctly.

Using the menus or MSD monitors, the DS can display:

- Motor speed for turbo pump MSDs (percent spin speed)
- Foreline pressure for diffusion pump MSDs
- Analyzer chamber pressure (vacuum) for MSDs with the optional G3397B Micro-Ion Gauge Controller

The 7890A+ and 7890B GC control panels also display this data.

Controlling Temperatures

MSD temperatures are controlled through the DS. 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 DS or from the 7890B GC control panel.

Normally, the GC/MSD interface heater is powered and controlled by the Thermal Aux #2 heated zone of the GC. The GC/MSD interface temperature can be set and monitored from the DS or from the 7890B GC control panel.

Controlling Column Flow

Carrier gas flow is controlled by inlet pressure in the GC. Set the column mode to **Constant Pressure** to maintain a constant inlet pressure, and the column flow will decrease as the GC oven temperature increases. With EPC and the column mode set to **Constant Flow**, the same column flow is maintained regardless of temperature.

The MSD can be used to measure actual column flow. You inject a *small* amount of air or other unretained chemical, and time how long it takes to reach the MSD. With this time measurement, you can calculate the column flow. (See “[To Calibrate Column Flow Linear Velocity](#)” on page 52.)

Controlling JetClean Hydrogen Flow

The JetClean option uses hydrogen gas for cleaning the ion source. The JetClean system MFC sends hydrogen to the annular space surrounding the column in the GC/MS interface where it then enters the ion source body. The flow rate is controlled by the MassHunter GC/MS Acquisition software. The JetClean system is only supported on turbo based systems using an EI Inert source, or an EI HES. (See “[To Set the Operation Mode for the Optional JetClean System](#)” on page 106.)

Venting the MSD

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

The MSD *will* be damaged by incorrect venting. A 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. A temperature of 100 °C is hot enough to burn skin; always wear cloth gloves when handling analyzer parts.

WARNING

If hydrogen is used as a carrier gas or JetClean system supply, the carrier and JetClean system supply shutoff valves must be closed before turning off the MS power. If the foreline pump is off, hydrogen will accumulate in the MS and an explosion may occur. Read “[Hydrogen Safety](#)” on page 23 before operating the MSD with hydrogen gas.

WARNING

Never open the vent valve or shut down the vacuum pumps without first closing the shutoff valves for all hydrogen flows to the analyzer. This includes hydrogen carrier gas and hydrogen for the JetClean system.

CAUTION

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

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

Do not exceed the maximum recommended total gas flow. (See [Table 3](#) on page 18.)

To View MSD Temperature and Vacuum in Manual Tune

You can also use the GC Control Panel to perform this task. (See “Operating the MSD from the GC control panel” on page 81.)

Procedure

- 1 In **Instrument Control** view, select **Edit Tune Parameters** from the Instrument menu to display the **Manual Tune** dialog.
- 2 Click the **Values** tab to view the MSD Temperatures and Vacuum. (See [Figure 24](#) and [Figure 25](#) on page 94.)

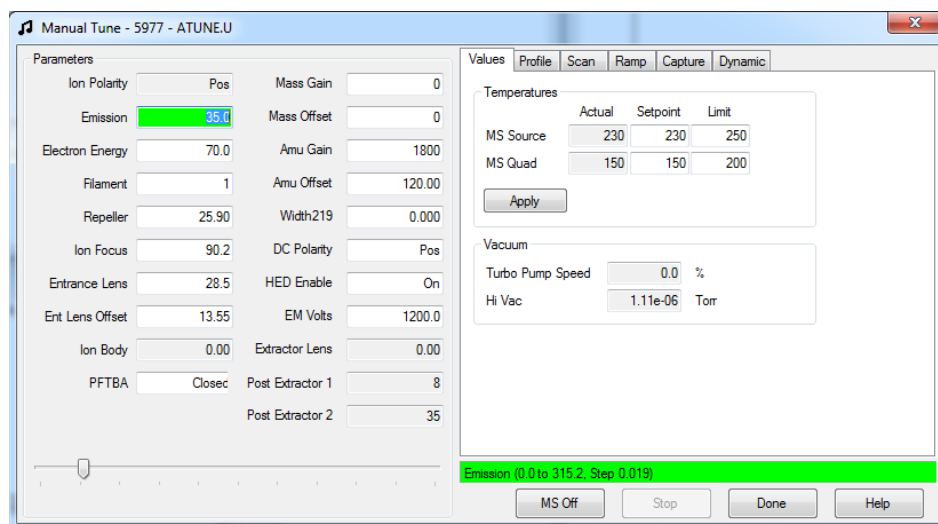


Figure 24 Manual tune values tab

4 Operating in EI Mode

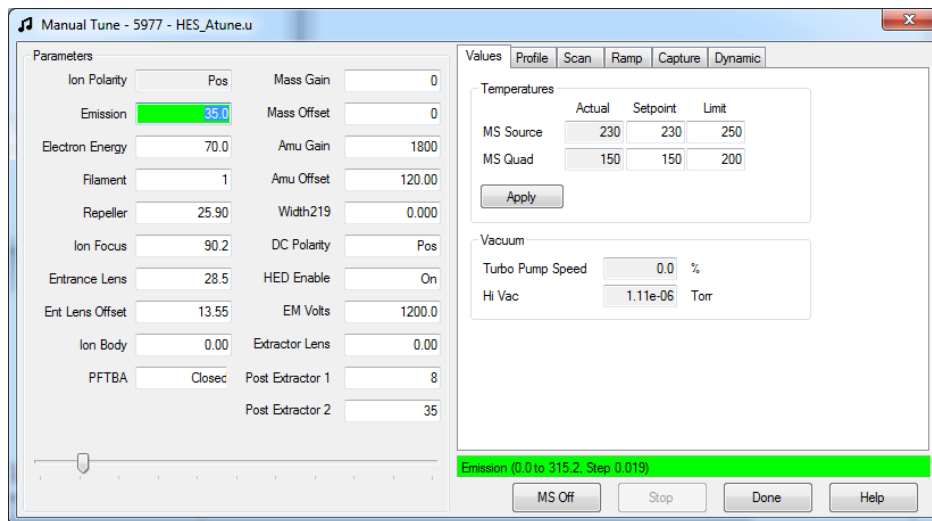


Figure 25 HES manual tune values tab

- 3 To change a temperature **Setpoint** or **Limit** enter the new parameters and click **Apply**.

Temperature setpoints or limits can not be changed until the foreline pressure is less than 300 mTorr, or the turbo pump is running at least 80% speed. MSD heaters remain off as long as the diffusion pump is cold or the turbo pump is operating at less than 80%. Normally, the foreline pressure will be below 100 mTorr, or the turbo pump speed will be at 100%.

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

To Set Monitors for MSD Temperature and Vacuum Status

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

Procedure

- 1 In **Instrument Control** view, select **Edit Monitors** from the **Instrument** menu to display the **Select Monitors** dialog. (See [Figure 26](#).)

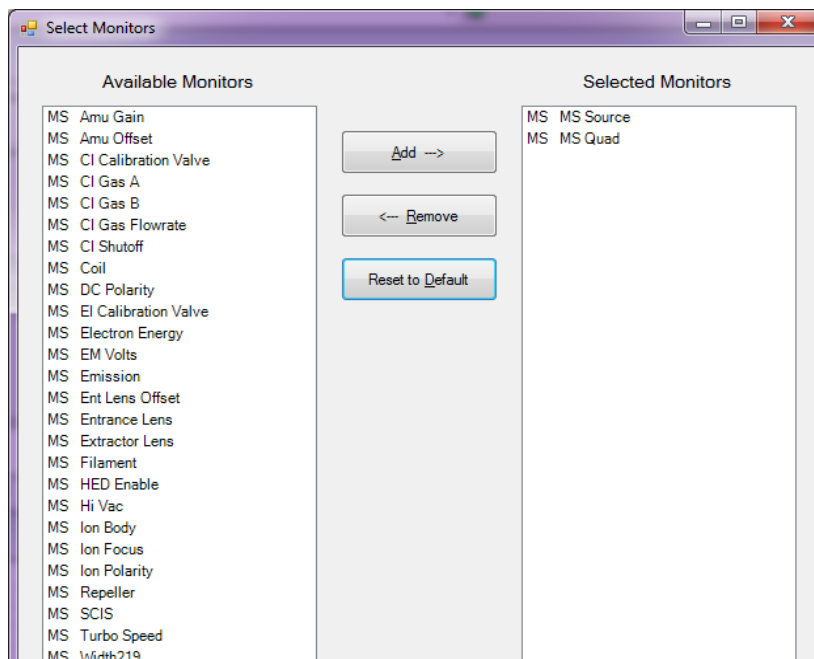


Figure 26 Select monitors dialog

- 2 In the **Available Monitors** column, select a monitor, and click **Add** to move the selection to the **Selected Monitors** column. Repeat for additional monitors.
- 3 Click **OK**. The new monitors will be stacked on top of each other in the lower right corner of the **Instrument Control** window.

4 Operating in EI Mode

- 4 Select **Window > Arrange Monitors**, or click and drag each monitor to the desired position. (See Figure 27.)

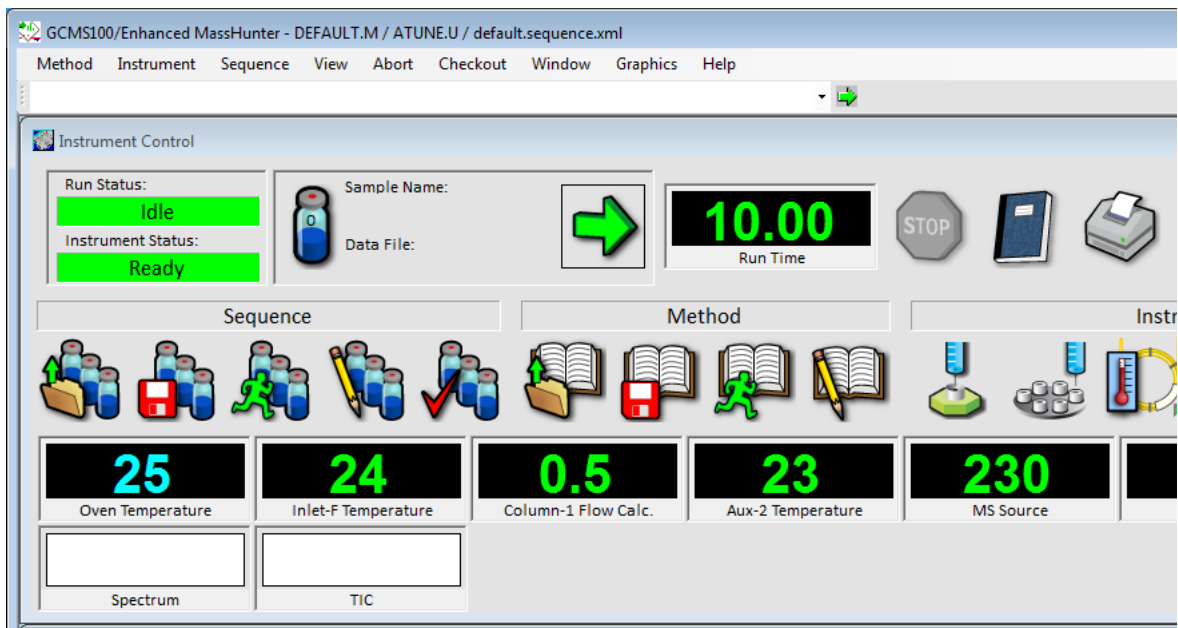
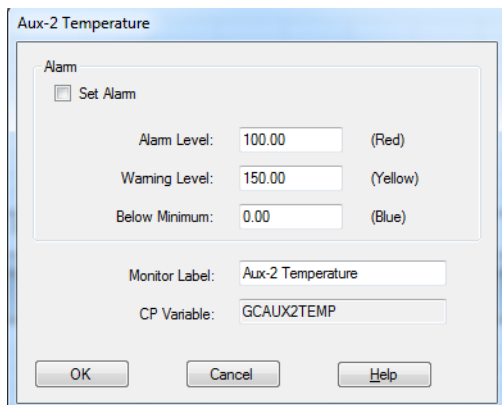


Figure 27 Arranging monitors

- 5 To set a monitor's alarm, double-click a monitor displayed in the Instrument Control view to open that monitor's dialog for setting alarms. (See Figure 28.)



Aux-2 Temperature

Alarm

Set Alarm

Alarm Level: 100.00 (Red)

Warning Level: 150.00 (Yellow)

Below Minimum: 0.00 (Blue)

Monitor Label: Aux-2 Temperature

CP Variable: GCAUX2TEMP

OK Cancel Help

Figure 28 Monitor's alarm settings

- a Select the **Set Alarm** check box.
 - b Set the **Warning Level**, **Alarm Level**, and **Below Minimum** to appropriate values.
 - c Enter descriptive text in the **Monitor Label** field if the default label is not appropriate.
 - d Click **OK** to finish the monitor's alarm configuration.
- 6 To make the new settings part of the method, save the Method.

To Set Analyzer Temperatures from the Instrument Control View

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

Procedure

- 1 In **Instrument Control** view, select **MS Temperatures** from the **Instrument** menu. (See [Figure 29](#).)

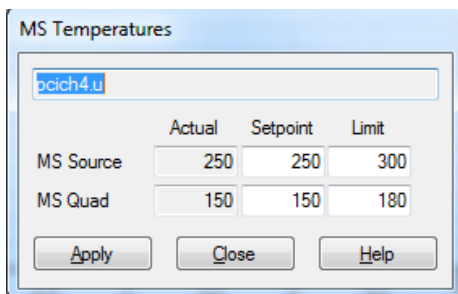


Figure 29 MS temperatures dialog

- 2 Enter the **MS Source** and **MS Quad** (mass filter) temperatures in the **Setpoint** and **Limit** fields. (See [Table 8](#).)

Table 8 Recommended temperature settings

| | El operation | PCI operation | NCI operation |
|-----------|--------------|---------------|---------------|
| MS Source | 250 | 250 | 150 |
| MS Quad | 150 | 150 | 150 |

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

CAUTION

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

- 3 To send the new temperature parameters to the currently loaded tune file and download these parameters to the MSD, click **Apply**.
- 4 Click **Close** to exit the dialog. If changes were made to any parameters the **Save MS Tune File** dialog displays. Click **OK** to save your changes to the same file, or type a new file name and click **OK**. Click **Cancel** to discard the edit made to any parameter.

To Set the MassHunter 7890 GC/MSD Interface Temperature

Procedure

- 1 From **Instrument Control** view select **Instrument>GC Edit Parameters**.
- 2 Click the **Aux Heaters** icon to edit the interface temperature. (See Figure 30.)

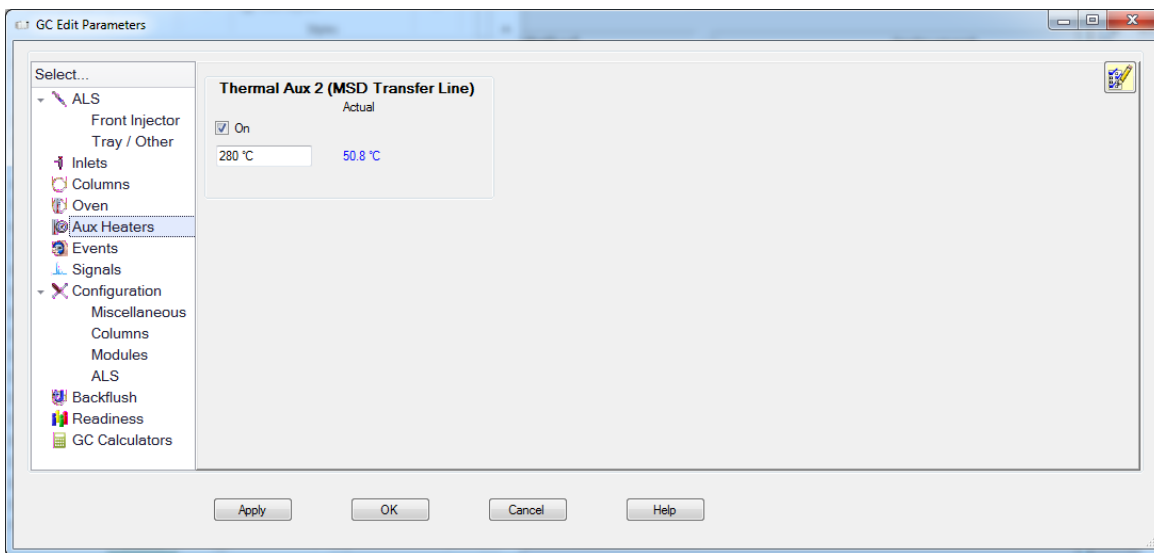


Figure 30 GC edit parameters window

- 3 Select **On** to turn on the heater and type the setpoint in the **Value °C** column. The typical setpoint is 280 °C.

CAUTION

Ensure that the carrier gas is turned on and the column has been purged of air before heating the GC/MS interface or the GC oven.

When setting the GC/MS interface temperature, never exceed the maximum for your column.

- 4 Click **Apply** to download setpoints, or click **OK** to download setpoints and close the window.
- 5 To make the new settings part of the method, select **Save** from the Method menu.

To Monitor High Vacuum Pressure

Pressure monitoring requires an optional G3397B Micro-Ion vacuum gauge.

WARNING

If you are using hydrogen as a carrier gas or for the JetClean system, the hydrogen 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 “[Hydrogen Safety](#)” on page 23 before operating the MSD with hydrogen gas.

Procedure

- 1 Ensure that the hydrogen supply shutoff valves are closed if you are using hydrogen as a carrier gas or for the JetClean system.
- 2 Start up and pumpdown the MSD. (See “[To Pumpdown the MSD](#)” on page 116.)
- 3 In the **Tune and Vacuum Control** view, select **Turn Vacuum Gauge on/off** from the **Vacuum** menu.
- 4 Select **Manual Tune** from the **Parameters** menu to display the Manual Tune dialog.
- 5 Select the **Values** tab to view the HiVac reading. (See [Figure 31](#) on page 102.)

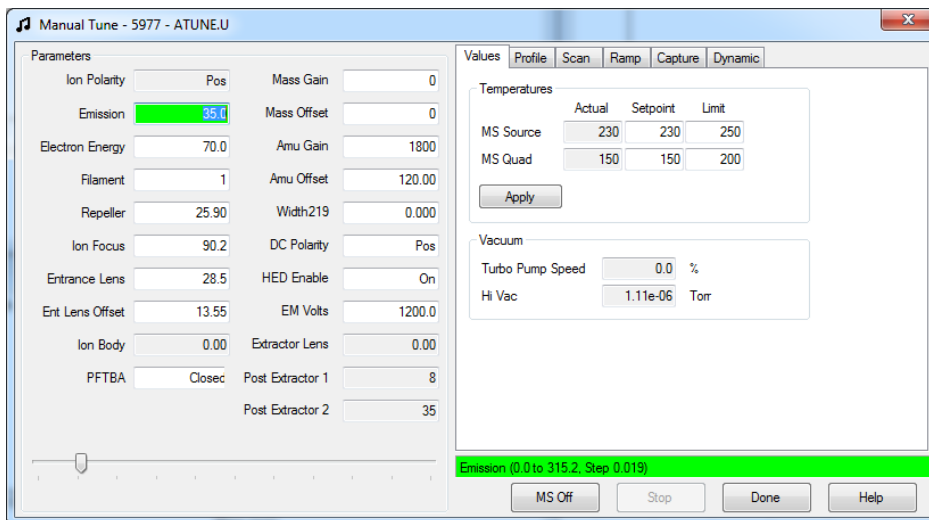


Figure 31 Manual Tune Values tab HiVac reading

The largest influence on operating pressure in EI mode is the carrier gas (column) flow. [Table 9](#) 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 9 Ion vacuum gauge reading

| Column flow rate, mL/min | Optional gauge reading, Torr <i>Turbo pump</i> | Gauge reading, Torr <i>Diffusion pump</i> | Foreline reading, mTorr <i>Diffusion pump</i> |
|--------------------------|---|--|--|
| 0.5 | 3.18E-06 | 2.18E-05 | 34.7 |
| 0.7 | 4.42E-06 | 2.59E-05 | 39.4 |
| 1 | 6.26E-06 | 3.66E-05 | 52.86 |
| 1.2 | 7.33E-06 | 4.46E-05 | 60.866 |
| 2 | 1.24E-05 | 7.33E-05 | 91.784 |
| 3 | 1.86E-05 | 1.13E-04 | 125.76 |
| 4 | 2.48E-05 | | |
| 6 | 3.75E-05 | | |

If the pressure is consistently higher than those listed, refer to the online help in the Agilent MassHunter GC/MS Acquisition software for information on troubleshooting air leaks and other vacuum problems.

In the **Instrument Control** view, you can set up an MSD Monitor for displaying this vacuum reading. The vacuum can also be read on the 7890A+ or 7890B GC series control panel or from the Manual Tune screen.

To Tune the MSD in EI Mode

You can also use the 7890B GC control panel to run the autotune that is currently loaded in MassHunter. (See “Operating the MSD from the GC control panel” on page 81.)

Procedure

- 1 Load the method that will be used for data acquisition.
- 2 In the **Instrument Control** view, verify the correct tune file is displayed in the tile bar. For most applications, **Autotune** gives good results.
- 3 To select a different tune file select **MS Tune File** from the **Instrument** menu to display the **Select Tune File** dialog. The **Settings** area displays the important parameters for a selected tune file.
The tune file must match the type of ion source in the analyzer. If you are using an EI source, select a tune file created for an EI source.
- 4 Click the **MS Tune** icon to display the **Select Tune Type** dialog. (See Figure 32.)

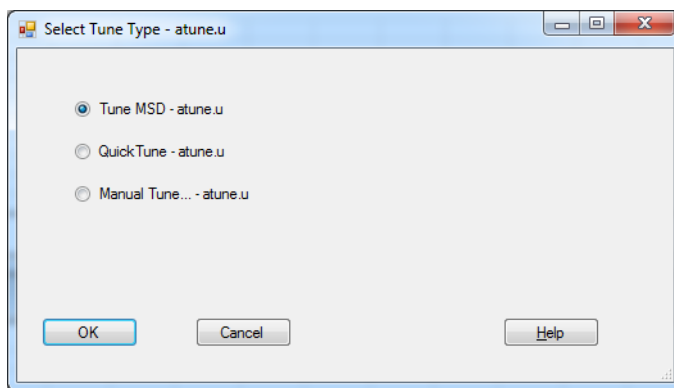


Figure 32 Select tune type dialog

- 5 Select **Tune MSD** to perform a complete autotune, or select **Quick Tune** to adjust peak width, mass assignment, and abundance, without changing ion ratios.

- 6 Click **OK** to close this dialog and start the tune. If the MSD temperatures are not stable, you are prompted to wait or override the wait by clicking **Override**.
- 7 Wait for the tune to complete, and generate the report.
- 8 To evaluate the tune results, select **Evaluate Tune** from the **Checkout** menu in **Instrument Control** view.

To view history of tune results, in the **Instrument Control** view select **Checkout>View Previous Tunes....**

To manually tune your MSD or to perform special autotunes, from the **View** menu select **Tune and Vacuum Control** view. See the manuals and online help provided with your Agilent MassHunter GC/MS Acquisition software for additional information about tuning.

To Set the Operation Mode for the Optional JetClean System

Procedure:

- 1 In **Instrument Control** view, click the **MS Parameters** icon.
- 2 In the Single Quadrupole MS Method Editor navigation panel, select the **JetClean** tab. (See [Figure 33](#).)
- 3 From the **Operation** dropdown menu, select a mode.

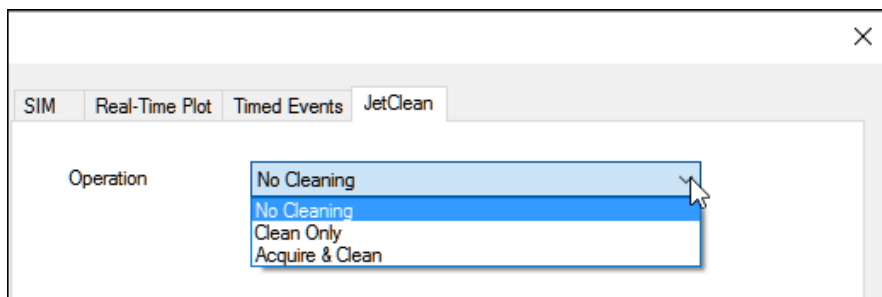


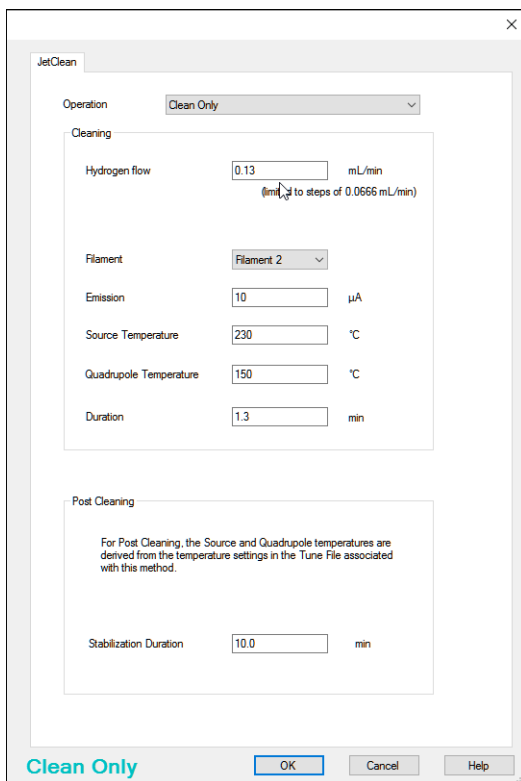
Figure 33 Selecting the JetClean Operation mode

For detailed information on the JetClean system see the *JetClean Operating manual*.

To Set the JetClean Parameters for the Clean Only Mode

Procedure:

- 1 In **Instrument Control** view, click the **MS Parameters** icon.
- 2 In the Single Quadrupole MS Method Editor, select the **JetClean** tab. (See [Figure 34](#).)
- 3 From the **Operation** dropdown menu, select **Clean Only**.



The screenshot shows the JetClean dialog box with the following settings:

- Operation:** Clean Only (dropdown menu)
- Cleaning:**
 - Hydrogen flow:** 0.13 mL/min (with a note: (1ml to steps of 0.0666 mL/min))
 - Filament:** Filament 2 (dropdown menu)
 - Emission:** 10 μ A
 - Source Temperature:** 230 $^{\circ}$ C
 - Quadrupole Temperature:** 150 $^{\circ}$ C
 - Duration:** 1.3 min
- Post Cleaning:**
 - For Post Cleaning, the Source and Quadrupole temperatures are derived from the temperature settings in the Tune File associated with this method.
 - Stabilization Duration:** 10.0 min

Buttons at the bottom: Clean Only (highlighted), OK, Cancel, Help.

Figure 34 JetClean set to Clean Only mode

- 4 Enter the parameters supplied by your method developer.
- 5 Save the method.

To Verify EI System Performance

Materials needed for an EI HES

- 10 fg/ μ L OFN Checkout standard 3 \times 1 mL (5190-0585)

Materials needed for an EI XTR, SS, or Inert source

- 1 pg/ μ L OFN EI Checkout standard 3 \times 1 mL (5188-5348)

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 **Checkout** menu. The software will perform an autotune and print the report.
- 4 When the autotune has completed, save the method, and then select **Evaluate Tune** from the **Checkout** menu.
The software will evaluate the last autotune, and print a System Verification – Tune report.

Verify the sensitivity performance

- 1 Set up to inject 1 μ L of the checkout sample shown in the Materials needed for your source type above, either with the ALS or manually.
- 2 In the **Instrument Control** view, select **Sensitivity Check** from the **Checkout** menu. The system displays an **Alert** dialog reminder about resolving the OFN_SN method and placing the OFN sample in vial 1 when an ALS is configured.
- 3 If necessary, resolve your hardware with this method and place the sample in the vial 1 position.
- 4 Click **OK** to run the method.

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

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

To Perform High-Mass Testing

Materials needed

- PFHT High Mass checkout sample 3 × 1 mL (5188-5357)

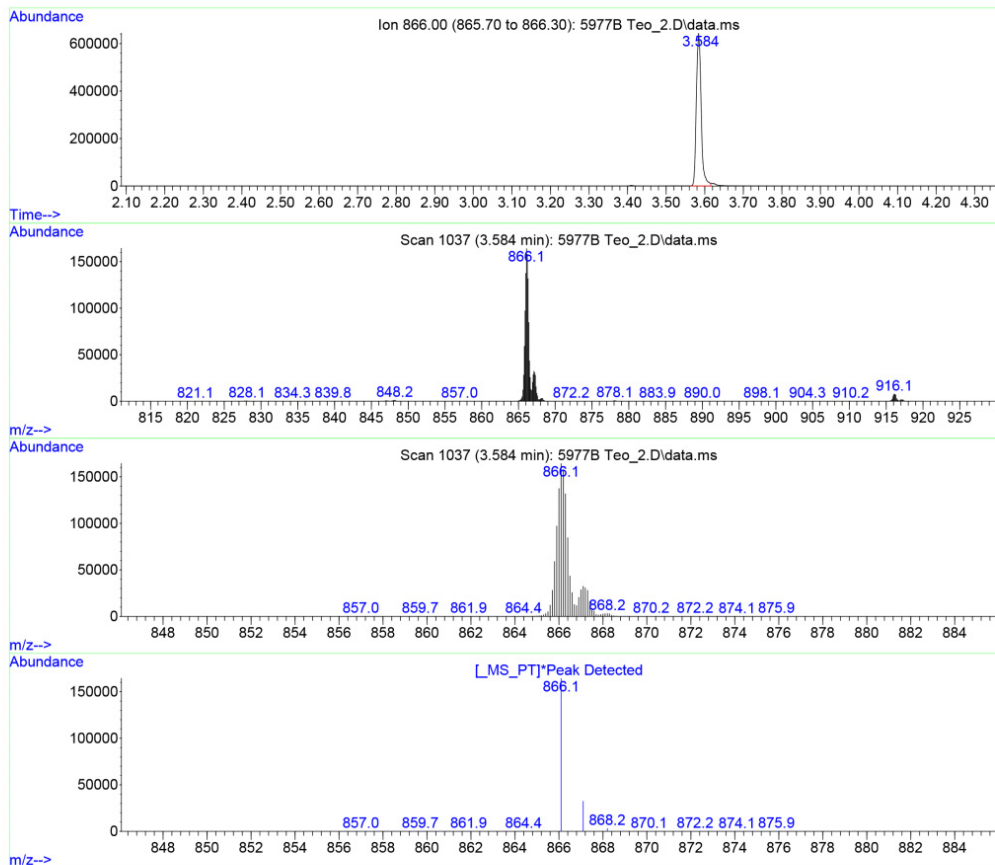
Procedure

- 1 Load the autotune file (atune.u, HES_atune.u, etune.u) then auto tune the MSD. (See “[To Tune the MSD in EI Mode](#)” on page 104.)
- 2 Resolve the PFHT.M method under MassHunter\MSD\x\methods\checkout\PFHT.M where x is the instrument number being used.
- 3 Update and save the method.
- 4 Load the PFHT calibration sample into a vial and place in position 2.
- 5 In **Instrument Control** view select **High Mass Check** from the **Checkout** menu.
- 6 Follow the instructions on screen.
- 7 The Run is completed, and results are printed within 5 minutes. (See [Figure 35](#) on page 110.)

4 Operating in EI Mode

PFHT High Mass Report

Data File : E:\MassHunter\GCMS\3\DATA\5977B Teo_2.D Vial: 1
 Acq On : 21 Mar 2016 08:29 Operator:
 Sample : Inst : 5977B HES
 Misc : Multiplr: 1.00
 Barcode : Expected= <none> Actual=ERR Sample Amount:0.00
 MS Integration Params: NA



| Mass | Actual | Isotope | Abund | Isotope | Ratio | Relative | Wid50% | Wid10% | Valley |
|--------|--------|---------|--------|---------|-------|----------|--------|--------|--------|
| 866.00 | 866.10 | 867.10 | 164416 | 32352 | 19.68 | 100.00 | 0.545 | 1.04 | 1686 |
| 867.00 | 867.10 | 868.20 | 32352 | 3114 | 9.63 | 19.68 | 0.545 | 1.04 | 1686 |
| 916.00 | 916.10 | 917.00 | 7702 | 1869 | 24.27 | 4.68 | 0.000 | 0.00 | 0 |

Figure 35 PFHT high mass report

Results

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

Adjustments

- 1 Verify the autotune file (atune.u, HES_atune.u, etune.u) has been loaded.
- 2 In **Instrument Control** view select **Edit Tune Parameters** from the **Instrument** menu to display the **Manual Tune** dialog.
- 3 Click the **Dynamic** tab and then click the **Amu Offset** sub-tab.
- 4 Select the **Enable This Lens** checkbox.
- 5 Enter the recommend dynamic offset **Voltage (V)**, and click **OK**.
- 6 Click **Save** to save this dynamic **Amu Offset** for the high-mass.
You can overwrite the existing autotune file to include high-mass adjustment or save this file to a new name, for example, ATUNEHIGH.U.
When an autotune is performed, it will overwrite this dynamic **Amu Offset** that was entered. This is why you might want to rename the tune.
- 7 Click **Done** to close the Manual Tune dialog.
- 8 Load the PFHT.M, then load the saved tune file, and then save the method.
- 9 Rerun test mixture (repeat high-mass checkout). If the correction is within 5 units, no further adjustments are required.

To Open the MSD Covers

If you need to open one of the MSD covers, follow these procedures.



To remove the analyzer window cover

Press down on the rounded area on the top of the window, tilt the window slightly forward and lift it off the MSD. (See [Figure 36](#).)

CAUTION

Do not use excessive force or the plastic tabs that hold the cover to the mainframe will break off.

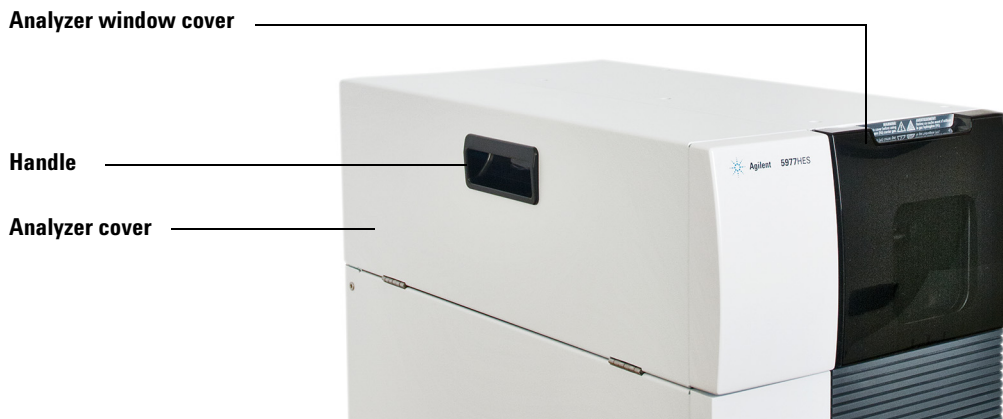


Figure 36 5977B MSD covers



Pull the handle on the side of the MSD to the left and down to release the magnetic latch and open the cover. The cover is held in place by its hinges. (See [Figure 36](#).)

WARNING

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

To Vent the MSD

Agilent MassHunter GC/MS Acquisition software allows you to specify parameters in a GC method that will automate and speed up the vent process if direct communication (DCOMM) with the 7890B GC is established. You must predefine a Fast Vent method to use this option. See the Agilent MassHunter GC/MS Acquisition software Online Help for information on how to configure a Fast Vent method.



Procedure

Agilent recommends that you create and save a Vent method, to ensure that safe temperatures are applied to the GC/MS transfer line and MS.

- 1 If you do not have LVDS or DCOMM communication between the GC and MS, in **Instrument Control** view select **GC Parameters** from the **Instrument** menu to display the **GC Edit Parameters** dialog. Select **Oven**, and set the oven temperature to room temperature. Also select **Aux Heaters (MSD Transfer line) and Inlets**, and set those temperatures to room temperature. Click **OK** to close the dialog and send this temperature to the GC.

WARNING

If hydrogen is used as a carrier gas or JetClean system supply, the carrier and JetClean system supply shutoff valves must be closed before turning off the MS power. If the foreline pump is off, hydrogen will accumulate in the MS and an explosion may occur. Read “Hydrogen Safety” on page 23 before operating the MSD with hydrogen gas.

CAUTION

Ensure the GC oven and the GC/MSD interface are cool before turning off carrier gas flow to prevent damage to the column.

- 2 In the **Instrument Control** view, **Instrument** menu, select **MS Vacuum Control** to display the **Vacuum Control** dialog. (See [Figure 37](#) on page 114.)

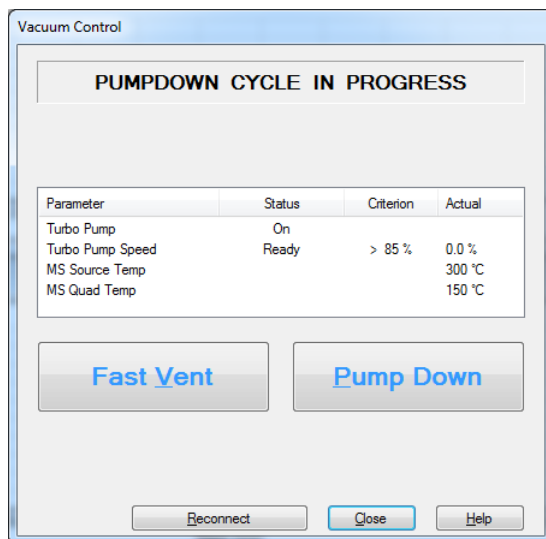


Figure 37 Vacuum control dialog

- 3 Remove the analyzer window cover. (See “To Open the MSD Covers” on page 112.)
- 4 Click **Fast Vent** if your system supports DCOMM communication between the GC and MS, or click **Vent** if it does not. If your system supports DCOMM, MassHunter will load the Fast Vent method to turn off the GC oven and interface heaters, the source and quad heaters, and the turbo pump. If your system does not support DCOMM, or if DCOMM has been disabled, when prompted set the GC/MS interface heater and the GC oven temperatures to ambient (room temperature).
- 5 Turn off the MS by pressing the power switch. (See [Figure 1](#) on page 19.)
- 6 Unplug the MS power cord.

WARNING

When the MS is vented, do not put the Agilent MassHunter GC/MS Acquisition software into Instrument Control view. Doing so will turn on the interface heater.

WARNING

Never open the vent valve without first closing the hydrogen supply line to all possible sources of hydrogen entry to the analyzer. This requires closing the hydrogen shutoff valve to the carrier gas flow module and the shutoff valve to the JetClean system supply if either of these hydrogen sources exist on the instrument.

- 7 Close shutoff valves to hydrogen sources that enter the analyzer if the instrument is so equipped.
- 8 When prompted, turn the vent valve knob counterclockwise **only** 3/4 turns or until you hear the hissing sound of air flowing into the analyzer chamber. (See Figure 38.)



Figure 38 Vent valve knob

Do **not** turn the knob too far, or the O-ring may fall out of its groove.

To Pumpdown the MSD

You can also use the 7890B GC control panel to perform this task. (See “Operating the MSD from the GC control panel” on page 81.)

WARNING

Make sure your MSD meets all the conditions listed in the introduction to this chapter before starting up and pumping down the MSD. (See “Before You Turn On the MSD” on page 89.) Failure to do so can result in personal injury.

WARNING

Never open the vent valve without first closing the hydrogen supply line to all possible sources of hydrogen entry to the analyzer. This requires closing the hydrogen shutoff valve to the carrier gas flow module and the shutoff valve to the JetClean system supply if either of these hydrogen sources exist on the instrument. Read “Hydrogen Safety” on page 23 before operating the MSD with hydrogen gas.

CAUTION

Agilent does not recommend using hydrogen as a carrier gas on systems equipped with an IDP3 rough pump.



Procedure

- 1 Remove the analyzer window cover. (See “To Open the MSD Covers” on page 112.)
- 2 Verify that the foreline pump inlet valve is open.

WARNING

Do not open the vent valve without verifying that the JetClean system hydrogen supply shutoff valve is closed if the optional JetClean system is installed. Refer to the JetClean system Operating manual for warnings regarding hydrogen safety when the hydrogen shutoff valve is open.

- 3 Verify that the vent valve is open by turning it clockwise until closed. (See Figure 39 on page 117.)
- 4 Open the vent valve by turning it counterclockwise 45 degrees. (See Figure 39 on page 117.)



Figure 39 Vent valve knob

- 5 Verify that the MSD power cord is plugged into a grounded building's main receptacle.
- 6 Turn on the MSD by pressing the **On/Off** switch on the front of the MSD.
- 7 Press lightly on the side board to ensure a correct seal. Press on the metal box on the side board.
- 8 Close the vent valve when it makes a hissing sound. (See [Figure 39](#).)
The foreline pump will make a gurgling noise. This noise should stop within a minute. If the noise continues, there is a **large** air leak in your system, probably at the side plate seal, the interface column nut, or the vent valve.
- 9 Start the Agilent MassHunter GC/MS Acquisition software.
- 10 In the **Instrument Control** view, from the Instrument menu, select **MS Vacuum Control** to display the **Vacuum Control** dialog. (See [Figure 40](#) on page 118.)

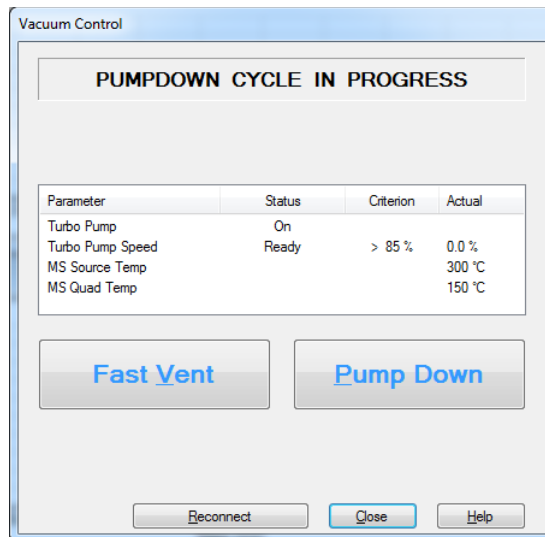


Figure 40 Vacuum control dialog

11 Click **Pump Down** in the **Vacuum Control** dialog, and follow the system prompts.

CAUTION

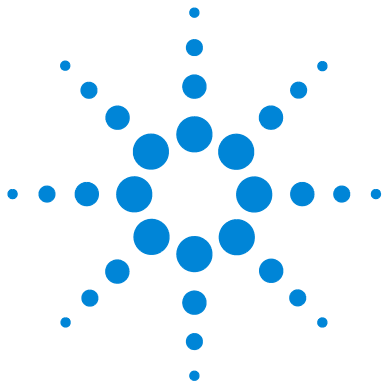
Do not turn on any GC heated zones until carrier gas flow is on. Heating a column with no carrier gas flow will damage the column.

12 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.

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

14 If using the JetClean system, prepare the hydrogen system for use. Refer to the JetClean system Operating manual for warnings regarding hydrogen safety.



5 Operating in CI Mode

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This chapter provides information and instructions for operating the 5977B Series CI MSDs in CI mode. Most of the information in the preceding chapter is also relevant.

Most of the material is related to methane CI but one section discusses the use of other reagent gases.

The software contains 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.

If you are using the *Agilent Intuvo 9000 Gas Chromatograph* with your MS, Chemical Ionization (CI) and the JetClean system are not currently supported.



General Guidelines

- Always use the highest purity methane (and other reagent gases, if applicable). Methane must be at least 99.9995% pure.
- Always verify the MSD is performing well in EI mode before switching to CI. (See “To Verify EI System Performance” on page 108.)
- Ensure the CI source and GC/MSD interface tip seal are installed.
- Ensure the reagent gas plumbing has no air leaks. This is determined in PCI mode, checking for m/z 32 after the methane pretune.
- Ensure the reagent gas inlet line is equipped with gas purifiers (not applicable for ammonia).

CI Autotune

After the reagent gas flow is adjusted, the lenses and electronics of the MSD should be tuned. (See “[Reagent gas settings](#)” on page 122.) 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 source is changed from EI to CI, 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.

EMV at or above 2600 V, however, indicates a problem. If your method requires EMV 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 “[To Verify EI System Performance](#)” on page 108.) Always set up the CI MSD in PCI first, even if you are going to run NCI.

5 Operating in CI Mode

Table 10 Reagent gas settings

| Reagent gas | Methane | | Isobutane | | Ammonia | | EI |
|----------------------|-------------|------------|-------------|------------|-------------|------------|------------|
| | Positive | Negative | Positive | Negative | Positive | Negative | |
| Ion polarity | Positive | Negative | Positive | Negative | Positive | Negative | N/A* |
| Emission | 150 μ A | 50 μ A | 150 μ A | 50 μ A | 150 μ A | 50 μ A | 35 μ A |
| 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 |
| EM volts | 1200 | 1400 | 1200 | 1400 | 1200 | 1400 | 1300 |
| Shutoff valve | Open | Open | Open | Open | Open | Open | Closed |
| Gas select | 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 | 280 °C | 280 °C | 280 °C | 280 °C | 280 °C | 280 °C | 280 °C |
| Autotune | Yes | Yes | No | Yes | No | Yes | Yes |

* N/A Not Available

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, and electron energy may need to be optimized for your specific analyte. (See Table 11.)

Table 11 Temperatures for CI operation

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

Start the system in PCI mode

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).
- Determine if a gross air leak is present by monitoring the ions at m/z 19 (protonated water) and 32.
- Confirm that 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. 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.

Depending upon the application, use the following reagent gas flow rates during system startup:

- PCI mode set reagent gas flow to 20 (1 mL/min)
- NCI mode set reagent gas flow to 40 (2 mL/min)

To Pumpdown the MSD in CI Mode

This procedure assumes that the instrument will eventually be PCI tuned using methane after the system is stable.

Procedure

- 1 Follow the instructions for the EI MSD. (See “To Pumpdown the MSD” on page 116.)

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

- 2 In the **Manual Tune** dialog, click the **Values** tab to monitor that the pressure is decreasing (Hi-Vac gauge option installed).
- 3 In the **Manual Tune** dialog, click the **CI Gas** tab, then in the **Valve Settings** area close **Gas Valve A**, **Gas Valve B**, and the **ShutOff Valve**.
- 4 Verify that **PCICH4.U** is loaded (top left in **Manual Tune** dialog) and click the **Values** tab to 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 280 °C. (See [Table 11](#) on page 123.)
- 6 Set **Gas A (methane)** to 20%.
- 7 Let the system bake out and purge for at least 2 hours. If you will be running NCI, for best sensitivity, bake out the MSD overnight.

To Set Up the Software for CI Operation

CAUTION

Always verify GC/MS performance in EI before switching to CI operation.

Procedure

- 1 From the **Tune and Vacuum Control** view, select **Load Tune Parameters** from the **File** menu, and load the tune file **PCICH4.U**.
- 2 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 [Table 12](#) for default values for the Tune Control Limits box.) These limits are used by Autotune only. They should **not** be confused with the parameters set in Edit MS Parameters or with those appearing on the tune report.

Table 12 Default Tune Control Limits, used by CI autotune only

| Reagent gas | Methane | | Isobutane | | Ammonia | |
|-----------------------------------|-------------------|-------------------|-----------|-------------------|----------|-------------------|
| | Positive | Negative | Positive | Negative | Positive | Negative |
| Abundance target | 1x10 ⁶ | 1x10 ⁶ | N/A | 1x10 ⁶ | N/A | 1x10 ⁶ |
| Peakwidth target | 0.6 | 0.6 | N/A | 0.6 | N/A | 0.6 |
| Maximum repeller | 4 | 4 | N/A | 4 | N/A | 4 |
| Maximum emission current, μ A | 240 | 50 | N/A | 50 | N/A | 50 |
| Max electron energy, eV | 240 | 240 | N/A | 240 | N/A | 240 |

Notes for Table 12:

- N/A: Not available. There are no PFDTD ions formed in PCI with any reagent gas but methane, hence, CI autotune is not available with these configurations.

- **Ion polarity:** Always set up in PCI with methane first, then switch to your desired ion polarity and reagent gas.
- **Abundance target:** Adjust higher or lower to get the desired signal abundance. Higher signal abundance also gives higher noise abundance. This is adjusted for data acquisition by setting the EMV in the method.
- **Peakwidth target:** Higher peakwidth values give better sensitivity, lower values give better resolution.
- **Maximum emission: current** 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

CAUTION

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

CAUTION

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

Procedure

- 1 In the **Manual Tune** dialog, click the **CI Gas** tab to access the parameter settings for controlling the CI gas flow. (See [Figure 41](#).)

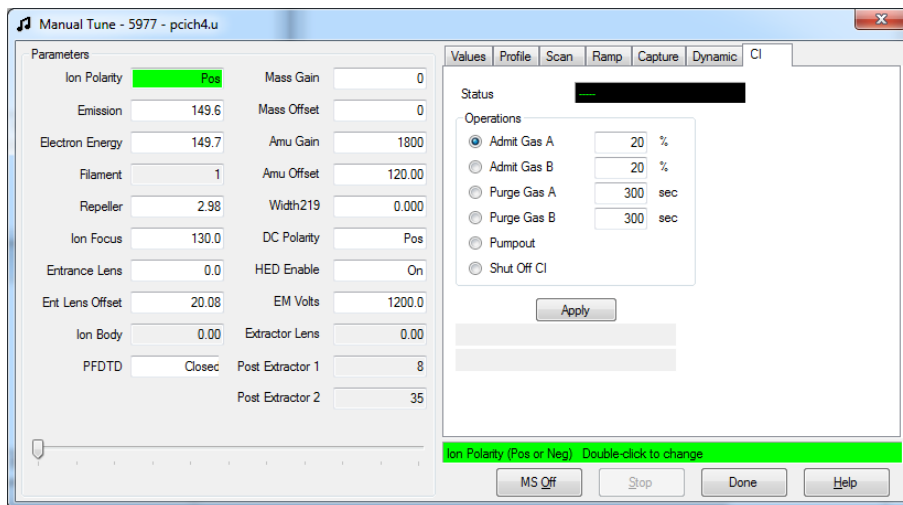


Figure 41 Manual tune CI tab

- 2 In the **Operations** area, select admit a reagent gas for the current tune file.
The system evacuates the gas lines for 6 minutes, then turns on the selected gas (A or B). This is to reduce cross-mixing of the gases in the lines.
- 3 Enter the reagent gas flow setpoint in the **Flow** field. This value is entered as a percentage of maximum flow rate. The recommended flow is 20% for a PCI source and 40% for an NCI source.
The flow control hardware 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 the last time.
- 4 To begin reagent gas flow, select **Shutoff CI**.
The system turns off the present gas flow while leaving the shutoff valve open. (See [Figure 42](#) on page 129.) This is to remove any residual gas in the lines. Typical evacuation time is 6 minutes and then the shutoff valve is closed.

The CI flow control module

The CI reagent gas flow control module regulates the flow of reagent gas into the CI GC/MSD interface. The flow module consists of a MFC, gas select valves, CI calibration valve, shutoff valve, control electronics, and plumbing. (See [Figure 42](#) and [Table 13](#) on page 129.)

The back panel provides Swagelok inlet fittings for methane (**CH₄**) and one **OTHER** reagent gas. The software refers to them as **Gas A** and **Gas B**, respectively. If you are not using a second reagent gas, cap the **OTHER** fitting to prevent accidental admission of air to the analyzer. Supply reagent gases at 25 to 30 psi (170 to 205 kPa).

The shutoff valve prevents contamination of the flow control module by atmosphere while the MSD is vented or by PFTBA during EI operation.

When a CI system is installed along with a JetClean system, the MFC is shared by both systems. By design, its use is restricted to one of these systems at a time. In this case, the Gas B supply is dedicated to hydrogen used for source cleaning. For detailed information on the JetClean system refer to the JetClean Operating manual installed on your PC along with this manual.

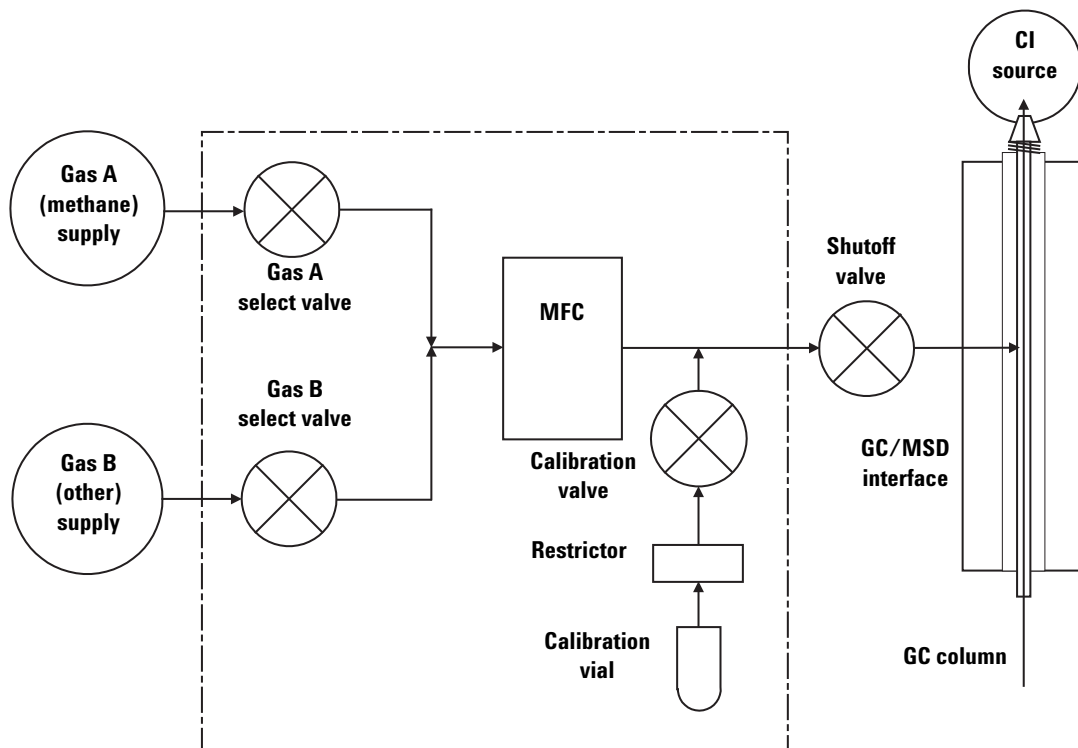


Figure 42 Reagent gas flow control module schematic

Table 13 Flow control module state diagram

| Result | Gas A flow | Gas B flow | Purge with Gas A | Purge with Gas B | Pump out flow module | Standby, vented, or EI mode |
|---------------|---------------|---------------|------------------|------------------|----------------------|-----------------------------|
| Gas A | Open | Closed | Open | Closed | Closed | Closed |
| Gas B | Closed | Open | Closed | Open | Closed | Closed |
| MFC | On → setpoint | On → setpoint | On → 100% | On → 100% | On → 100% | Off → 0% |
| Shutoff valve | Open | Open | Open | Open | Open | Closed |

The **Open** and **Closed** states are shown in the monitors as **Open** and **Closed** respectively.

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 PCI mode. 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, pretuning 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.

Procedure

- 1 Using an EI source, perform the standard autotune, save the report, and note the reported pressure. (See “[To Tune the MSD in EI Mode](#)” on page 104.)
- 2 Vent the system. (See “[Venting the MSD](#)” on page 91.)
- 3 Install the CI source. (See “[To Install the CI Source](#)” on page 225.)
- 4 Pump out the system. (See “[To Pumpdown the MSD in CI Mode](#)” on page 124.)
- 5 Wait until the pressure is near the previously recorded pressure for the EI autotune. (See “[To Monitor CI Mode High Vacuum Pressure](#)” on page 141.)
- 6 Select **Bake out MSD** from the **Manual Tune** view in the **Execute** menu to display the **Specify Bake Out parameters** dialog. Set a minimum time of 2 hours, adjust the other parameters, and click **OK** to begin the bake out.

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.

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 need to **vent the MSD** and **clean the ion source**.

-
- 7 Select **Methane Pretune** from the **Setup** menu and follow the system prompts. See the Agilent MassHunter GC/MS Acquisition software online help for additional information.

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

- 8 Examine the displayed profile scan of the reagent ions. (See Figure 43.)
 - There should be no visible peak at m/z 32. A peak there indicates an air leak. Repair the leak before proceeding. Operating in the CI mode with an air leak will rapidly contaminate the ion source.
 - The peak at m/z 19 (protonated water) is less than 50% of the peak at m/z 17.
- 9 When prompted, click **OK** to perform the methane Flow Adjust.

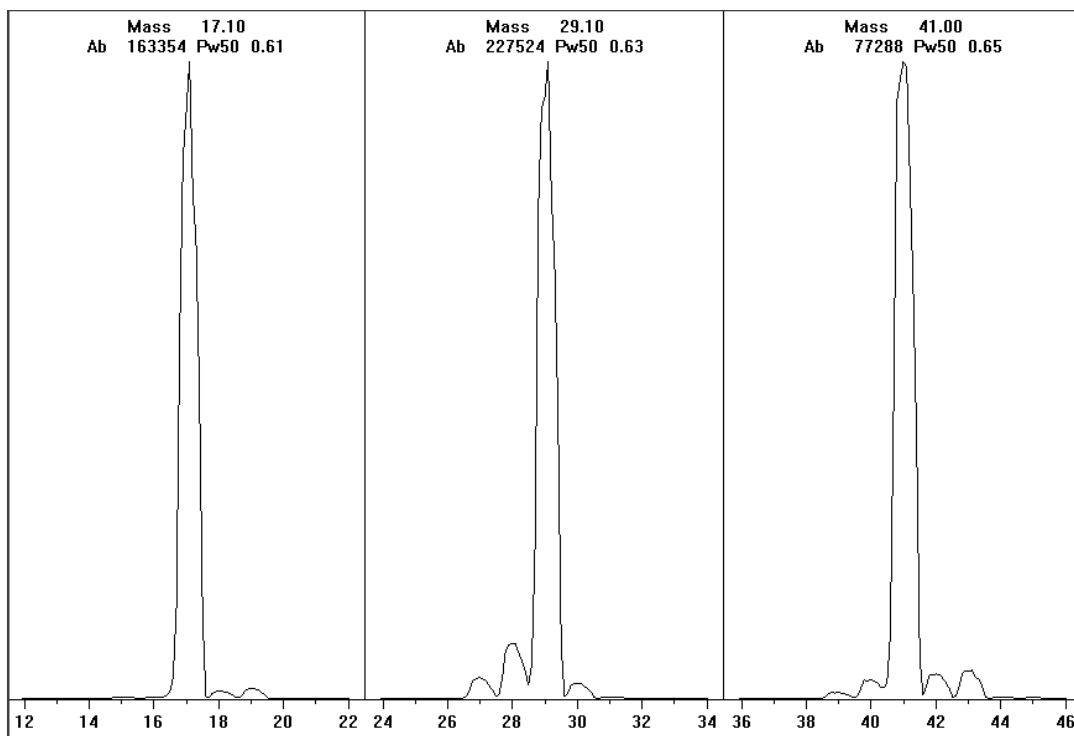


Figure 43 Reagent ion scans after a very long bake out

Methane pretune 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.

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 5977B Series 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 CI reactions encountered are described in general in the 5977B Series Concepts Guide. If you are not experienced with CI, we suggest reviewing that material before you proceed.

CAUTION

Not all setup operations can be performed in all modes with all reagent gases. (See [Table 14](#) for details.)

Table 14 Reagent gases

| Reagent gas/mode | Reagent ion masses | PFDTD Calibrant ions | Flow adj ions: Ratio |
|------------------|--------------------|-------------------------|--|
| | | | EI/PCI/NCI MSD Turbo pump Recommended flow: 20% PCI 40% NCI |
| Methane/PCI | 17, 29, 41* | 41, 267, 599 | 28/27: 1.5 – 5.0 |
| Methane/NCI | 17, 35, 235† | 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 |

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

† There are no **negative** reagent gas ions formed. To pretune in negative mode, use background ions: 17 (OH-), 35 (Cl-), and 235 (ReO3-). These ions cannot be used for reagent gas flow adjustment. Set the 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 CI when less fragmentation is desired in the CI 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 CI when less fragmentation is desired in the CI 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 CI 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 CI, use the following procedure:

Procedure

- 1 Perform a standard PCI autotune with methane and PFDTD. (See “[To Perform a PCI Autotune \(Methane Only\)](#)” on page 135.)
- 2 In the **Tune and Vacuum Control** view from the **Tune** menu, click **Tune Wizard**, and when prompted, select **Isobutane** or **Ammonia**. This will change the menus to use the selected gas and select appropriate default tune parameters.
- 3 When prompted, select **Gas B**. (the port where Isobutane or Ammonia is plumbed). Continue to the prompts from the Tune Wizard and set the gas flow to 20%.

If you use an existing tune file, be sure to save it with a new name if you do not want to overwrite the existing values. Accept the default temperature and other settings.

4 Click **Isobutane** (or **Ammonia**) **Flow Adjust** on the **Execute** 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 an existing NCI tune file for the specific gas. For more information on CI operation using ammonia, refer to Agilent Application Note “[Implementation of Ammonia Reagent Gas for Chemical Ionization on the 5975 Series MSDs](#)” (5989-5170EN).

CAUTION

Use of ammonia affects the maintenance requirements of the MSD. (See “[CI Maintenance](#)” on page 197 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. Refer to the *Agilent 5977B Series MSD Troubleshooting and Maintenance Manual*.

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 CI while minimizing its negative effects.

Carbon Dioxide CI

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

To Perform a PCI Autotune (Methane Only)

CAUTION

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

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

Procedure

- 1 Verify that the MSD performs correctly in EI mode first. (See “To Verify EI System Performance” on page 108.)
- 2 From the **Tune and Vacuum Control** view, 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 do not want to overwrite the existing values.
- 3 Accept the default settings.
- 4 Perform methane setup. (See “To Set Up Methane Reagent Gas Flow” on page 130.)
- 5 Under the **Tune** menu, click **CI Autotune**.

There are no tune performance criteria. If autotune completes, it passes. (See [Figure 44](#) on page 136.) If the tune sets the EMV at or above 2,600 V, however, you may not be able to acquire data successfully if your method sets EMV to “+400” or higher.

The autotune report contains information about air and water in the system. (See “PCI autotune report” on page 136.)

The 19/29 ratio shows the abundance of water.

The 32/29 ratio shows the abundance of oxygen.

5 Operating in CI Mode

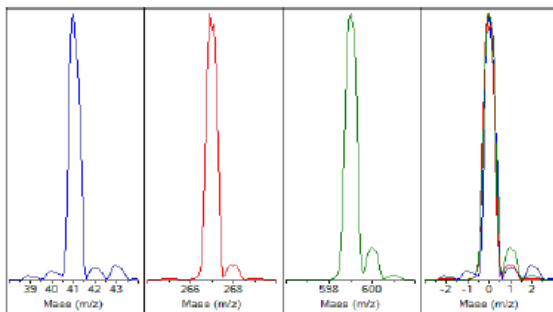
Methane@17 CI Autotune - 5977

8/5/2015 7:41:30 PM (UTC-4:00)

CT12

D:\MASSHUNTER\GCMS\1\5977\PCICH4.U

US1531Nxxx



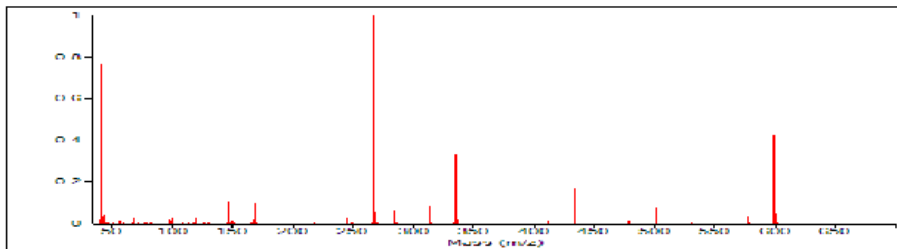
| | | | |
|-----------------|-------|----------------|--------|
| Ion Polarity | Pos | Mass Gain | -384 |
| Emission | 240.1 | Mass Offset | -35 |
| Electron Energy | 65.0 | Amu Gain | 1511 |
| Filament | 1 | Amu Offset | 118.38 |
| Repeller | 2.89 | Width219 | -0.021 |
| Ion Focus | 129.9 | DC Polarity | Pos |
| Entrance Lens | 13.1 | HED Enable | On |
| Ent Lens Offset | 16.46 | EM Volts | 858.8 |
| Ion Body | 0.00 | Extractor Lens | 0.00 |
| | | Scan Speed | 3 |
| | | Averages | 3 |
| PFTD | Open | Step Size | 0.10 |

| Actual m/z | Abund | Rel Abund | Pw50 |
|------------|---------|-----------|------|
| 41.00 | 410,787 | 100.0% | 0.61 |
| 266.90 | 529,995 | 129.0% | 0.62 |
| 599.00 | 221,740 | 54.0% | 0.62 |

Temperatures and Pressures

| | | |
|-----------|-----------------|----------|
| MS Source | 300 Turbo Speed | 100.0 |
| MS Quad | 150 Hi Vac | 7.51e-05 |

| Low | High | Step | Speed | Threshold | Peaks | Base | Abundance | Total Ion |
|-------|--------|------|-------|-----------|-------|--------|-----------|-----------|
| 35.00 | 700.00 | 0.10 | 3 | 1 | 680 | 267.00 | 508,096 | 1,956,041 |



| Target m/z | Actual m/z | Abund | Rel Abund | Iso m/z | Iso Abund | Iso Ratio |
|------------|------------|---------|-----------|---------|-----------|-----------|
| 41.00 | 41.00 | 388,096 | 76.4% | 42.00 | 16,696 | 4.3% |
| 267.00 | 267.00 | 508,096 | 100.0% | 268.00 | 27,480 | 5.4% |
| 599.00 | 599.00 | 217,920 | 42.9% | 600.00 | 25,448 | 11.7% |

Air/Water Check: H2O ~N/A% N2 ~N/A% O2 ~N/A% CO2 ~N/A% N2/H2O ~N/A%

Column(1) Flow: 1.19 Column(2): 0.00 ml/min Interface Temp: 280

Ramp Criteria:

Ion Focus maximum N/A volts using ion N/A; Electron Multiplier Gain 6470.073

Repeller maximum N/A volts using ion N/A; Gain Factor 0.0647

Mass Gain Values(Scan Speed): -384(3) -384(2) -384(1) -384(0) -281(FS1) -280(FS2)

| TARGET MASS: | 50 | 69 | 131 | 219 | 414 | 502 | 1050 |
|----------------------|-------|-------|-------|-------|-------|-------|-------|
| Amu Offset | 118.4 | 118.4 | 118.4 | 118.4 | 118.4 | 118.4 | 118.4 |
| Entrance Lens Offset | 16.5 | 16.5 | 16.5 | 16.5 | 16.5 | 16.5 | 16.5 |

Figure 44 PCI autotune report

To Perform an NCI Autotune (Methane Reagent Gas)

CAUTION

Always verify MSD performance in EI before switching to CI operation. (See “To Verify EI System Performance” on page 108.) Always set up the CI MSD in PCI with methane as the reagent gas first, even if you are going to be using a different reagent gas or going to run NCI.

Procedure

- 1 From the **Tune and Vacuum Control** view, load **NCICH4.U** (or an existing tune file for the reagent gas you are using).
- 2 From the **Setup** menu, select the **CI Tune Wizard** and follow the system prompts.
Accept the default temperature and other settings.
If you use an existing tune file, be sure to save it with a new name if you don't want to overwrite the existing values.
- 3 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. (See [Figure 45](#) on page 138.) If the tune sets the EMV at or above 2,600 V, however, you may not be able to acquire data successfully if your method sets EMV to “+400” or higher.

5 Operating in CI Mode

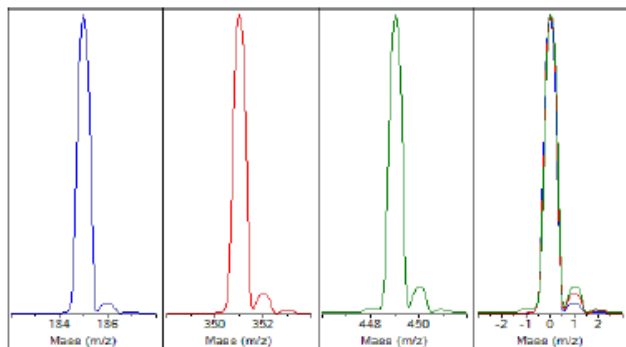
Methane@40 CI Autotune - 5977

8/5/2015 8:47:11 PM (UTC-4:00)

CT12

D:\MASSHUNTER\GCMS\1\5977\NCICH4.U

US1531Nxxx



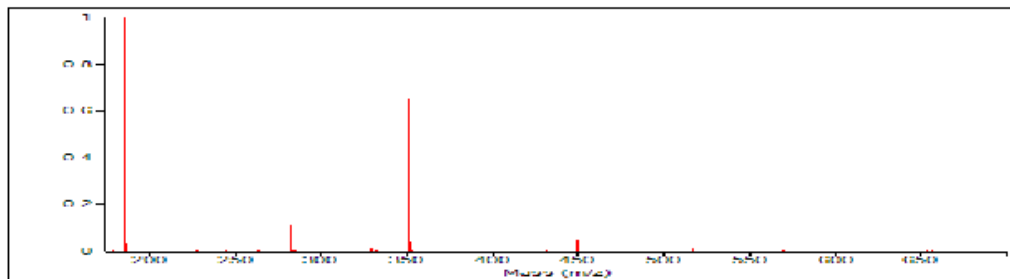
| | | | |
|-----------------|-------|----------------|--------|
| Ion Polarity | Neg | Mass Gain | -376 |
| Emission | 50.0 | Mass Offset | -34 |
| Electron Energy | 139.9 | Amu Gain | 1499 |
| Filament | 1 | Amu Offset | 122.13 |
| Repeller | 2.79 | Width219 | 0.000 |
| Ion Focus | 144.9 | DC Polarity | Neg |
| Entrance Lens | 15.9 | HED Enable | On |
| Ent Lens Offset | 14.18 | EM Volts | 1047.1 |
| Ion Body | 0.00 | Extractor Lens | 0.00 |
| | | Scan Speed | 3 |
| | | Averages | 3 |
| PFTD | Open | Step Size | 0.10 |

| Actual m/z | Abund | Rel Abund | Pw50 |
|------------|---------|-----------|------|
| 185.00 | 542,810 | 100.0% | 0.61 |
| 351.00 | 349,809 | 64.4% | 0.61 |
| 449.00 | 25,371 | 4.7% | 0.61 |

Temperatures and Pressures

| | | |
|-----------|-----------------|----------|
| MS Source | 150 Turbo Speed | 100.0 |
| MS Quad | 150 Hi Vac | 1.49e-04 |

| Low | High | Step | Speed | Threshold | Peaks | Base | Abundance | Total Ion |
|--------|--------|------|-------|-----------|-------|--------|-----------|-----------|
| 175.00 | 700.00 | 0.10 | 3 | 100 | 77 | 185.00 | 520,128 | 1,040,425 |



| Target m/z | Actual m/z | Abund | Rel Abund | Iso m/z | Iso Abund | Iso Ratio |
|------------|------------|---------|-----------|---------|-----------|-----------|
| 185.00 | 185.00 | 520,128 | 100.0% | 186.00 | 17,184 | 3.3% |
| 351.00 | 351.00 | 340,416 | 65.4% | 352.00 | 22,112 | 6.5% |
| 449.00 | 449.00 | 25,088 | 4.8% | 450.00 | 2,179 | 8.7% |

Air/Water Check: H2O ~N/A% N2 ~N/A% O2 ~N/A% CO2 ~N/A% N2/H2O ~N/A%

Column(1) Flow: 1.20 Column(2): 0.00 ml/min Interface Temp: 250

Ramp Criteria:

Ion Focus maximum N/A volts using ion N/A; Electron Multiplier Gain 30460.038

Repeller maximum N/A volts using ion N/A; Gain Factor 0.3046

Mass Gain Values(Scan Speed): -377(3) -377(2) -377(1) -377(0) -273(FS1) -273(FS2)

Figure 45 NCI autotune

To Verify PCI Performance

Materials needed

- Benzophenone, 100 pg/ μ L 5 \times 1 mL (8500-5440)

CAUTION

Always verify MSD performance in EI before switching to CI operation. (See “To Verify EI System Performance” on page 108.) Always set up the CI MSD in PCI first, even if you are going to run NCI.

Procedure

- 1 Verify that the MSD performs correctly in EI mode.
- 2 Verify that the **PCICH4.U** tune file is loaded.
- 3 Select **Gas A** and set flow to 20%.
- 4 In **Tune and Vacuum Control** view, perform CI setup. (See “To Perform a PCI Autotune (Methane Only)” on page 135.)
- 5 Run CI Autotune. (See “CI Autotune” on page 121.)
- 6 Run the PCI sensitivity method **BENZ_PCI.M** using 1 μ L of 100 pg/ μ L benzophenone.
- 7 Verify that the system conforms to the published sensitivity specification. Please refer to the Agilent Web site at www.agilent.com/chem for specifications.

To Verify NCI Performance

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

Materials needed

- OFN, 100 fg/ μ L 3 \times 1 mL (5188-5347)

CAUTION

Always verify MSD performance in EI before switching to CI operation. (See “To Verify EI System Performance” on page 108.) Always set up the CI MSD in PCI first, even if you are going to run NCI.

Procedure

- 1 Verify that the MSD performs correctly in EI mode.
- 2 Load the **NCICH4.U** tune file, and accept the temperature setpoints.
- 3 Select **Gas A** and set flow to 40%.
- 4 In **Tune and Vacuum Control** view, run CI Autotune. (See “To Perform an NCI Autotune (Methane Reagent Gas)” on page 137.)
Note that there are no criteria for a “passing” Autotune in CI. If the Autotune completes, it passes.
- 5 Run the NCI sensitivity method: OFN_NCI.M using 2 μ L of 100 fg/ μ L OFN.
- 6 Verify that the system conforms to the published sensitivity specification. Please refer to the Agilent Web site at www.agilent.com/chem for specifications.

To Monitor CI Mode High Vacuum Pressure

WARNING

If hydrogen is used as a carrier gas or JetClean system supply, the carrier and JetClean system supply shutoff valves must be closed before turning off the MS power. If the foreline pump is off, hydrogen will accumulate in the MS and an explosion may occur. Read “Hydrogen Safety” on page 23 before operating the MSD with hydrogen gas.

Procedure

- 1 Start up and pumpdown the MSD. (See “To Pumpdown the MSD in CI Mode” on page 124.)
- 2 In the **Tune and Vacuum Control** view select **Turn Vacuum Gauge on/off** from the **Vacuum** menu.
- 3 In the **Instrument Control** view you can set up an MS Monitor for reading. The vacuum can also be read on the LCP or from the Manual Tune screen.

The gauge controller will not turn on if the pressure in the MSD is above approximately 8×10^{-3} 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. Typical pressures for various helium carrier gas flows are approximate and vary from instrument to instrument. (See [Table 15](#) on page 142.)

Typical pressure readings

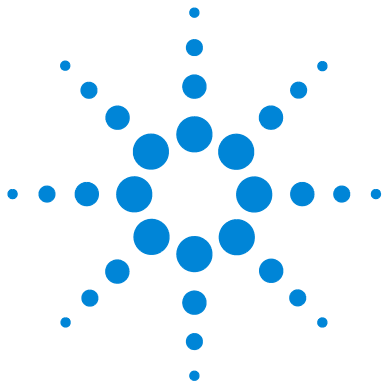
Use the G3397B Micro-Ion vacuum gauge. Note that the MFC is calibrated for methane and the vacuum gauge is calibrated for nitrogen, so these measurements are not accurate, but are intended as a guide to typical observed readings. (See [Table 15](#) on page 142.) They were taken with the following set of conditions. Note that these are typical PCI temperatures:

| | |
|--------------------------------|----------|
| Source temperature | 250 °C |
| Quad temperature | 150 °C |
| Interface temperature | 280 °C |
| Helium carrier gas flow | 1 mL/min |

Table 15 MFC settings and typical pressure readings

| MFC (%) | Pressure (Torr) | |
|---------|-----------------------------|-----------------------------|
| | Methane | Ammonia |
| | EI/PCI/NCI MSD (Turbo pump) | EI/PCI/NCI MSD (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.



6 General Maintenance

| | |
|---|-----|
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| Maintaining the Analyzer | 150 |
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| To Remove the EI HES | 154 |
| To Connect/Disconnect Wiring to the EI HES | 156 |
| To Disassemble the EI HES | 157 |
| To Clean the EI HES | 160 |
| To Assemble the EI HES | 163 |
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| To Install the EI HES Filament | 170 |
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| To Remove the EI XTR, SS, or Inert Source | 172 |
| To Connect/Disconnect Wiring from the EI XTR, SS, and Inert Sources | 173 |
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| To Close the Analyzer Chamber | 196 |

If you are using the *Agilent Intuvo 9000 Gas Chromatograph* with your MS, Chemical Ionization (CI) and the JetClean system are not currently supported.



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.

Table 16 Maintenance schedule

| Task | Frequency |
|---|------------------|
| Check the foreline pump oil level | Every week |
| Check the calibration vial(s) | Every 6 months |
| Replace the foreline pump oil* | Every 6 months |
| Replace the diffusion pump fluid | Every year |
| Change the dry foreline pump tip seal | Every year |
| Check the dry foreline pump | As needed |
| Tune the MSD | As needed |
| Change the foreline pump exhaust filter | As needed |
| Clean the ion source | As needed |
| Check the carrier gas trap(s) on the GC and MSD | As needed |
| Replace the worn out parts | As needed |
| Lubricate sideplate or vent valve O-rings† | As needed |
| Replace CI Reagent gas supply | As needed |
| Replace GC gas supplies | As needed |
| Leak check system | As needed |

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

† 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.

Scheduled maintenance

Performing the common maintenance tasks when scheduled can reduce operating problems, prolong system life, and reduce overall operating costs. (See [Table 16](#) on page 144.)

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.

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.

High voltage precautions

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

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

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

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

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

WARNING

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

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

Dangerous temperatures

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

- GC inlet
- GC oven and its contents
- GC detector
- GC valve box
- Foreline pump
- Heated MSD ion source, interface, and quadrupole

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 a thermal 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.

WARNING

The foreline pump can cause burns if touched when operating. It has a safety shield to prevent the user from touching it.

The GC inlets 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

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

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

WARNING

The oil trap supplied with the standard foreline pump stops only foreline pump oil. It does not trap or filter out toxic chemicals. If you are using toxic solvents or analyzing toxic chemicals, remove the oil trap. 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 diffusion pump and standard 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.

Ion source cleaning

The main effect of operating the MS 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 MS in a well-ventilated room.

Electrostatic discharge

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

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

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

CAUTION

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

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

Maintaining the Vacuum System

Periodic maintenance

Some maintenance tasks for the vacuum system must be performed periodically. (See [Table 16](#) on page 144.) These include:

- Checking the foreline pump fluid (every week)
- Replacing the tipseal (annually)
- Checking for leaks (every month)
- Checking the calibration vial(s) (every 6 months)
- 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)
- Replacing the diffusion pump fluid (once a year)
- Replacing the dry foreline pump seals (once a year)

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

Other procedures

Tasks such as replacing a foreline vacuum gauge or Micro-Ion vacuum gauge should be performed only when needed. Refer to the *Agilent 5977B Series MSD Troubleshooting and Maintenance* manual and the online help in Agilent MassHunter GC/MS Acquisition 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, refer to the *Agilent 5977B Series MSD Troubleshooting and Maintenance* manual.

Most of the procedures in this chapter are illustrated with video clips on the Agilent GC and GC/MS User Manuals and Tools DVD set and the Agilent GC/MS Software Information and Manuals memory stick.

Maintaining the Analyzer

Scheduling

None of the analyzer components require 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

The *Agilent 5977B Series MSD Troubleshooting and Maintenance Manual* provides information about symptoms that indicate the need for analyzer maintenance. The troubleshooting material in the online help in the Agilent MassHunter GC/MS Acquisition software provides more extensive information.

Precautions

Cleanliness

Keep components clean during analyzer maintenance. Analyzer maintenance involves opening the analyzer chamber and removing parts from the analyzer. During analyzer maintenance procedures, take care to avoid contaminating the analyzer or the interior of the analyzer chamber. Wear clean gloves during all analyzer maintenance procedures. After cleaning, parts must be thoroughly baked out before they are reinstalled. After cleaning, analyzer parts should be placed only on clean, lint-free cloths.

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 “[Electrostatic discharge](#)” on page 148 for more information.)

CAUTION

Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded antistatic wrist strap (See “[Electrostatic discharge](#)” on page 148.) and take other antistatic precautions **before** you open the analyzer chamber.

Some analyzer parts should not be disturbed

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

CAUTION

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

More information is available

If you need more information about the locations or functions of analyzer components, refer to the *Agilent 5977B Series MSD Troubleshooting and Maintenance Manual*.

Many procedures in this chapter are illustrated with video clips.

To Open the Analyzer Chamber

The analyzer chamber should only be opened to clean or replace the ion source, change the detector's EM, or to change a filament.

Materials needed

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

CAUTION

Electrostatic discharges to analyzer components are conducted to the quad driver board, where they can damage sensitive components. Wear a grounded antistatic wrist strap and take other antistatic precautions (See “[Electrostatic discharge](#)” on page 148.) before you open the analyzer chamber.



Procedure

- 1 Vent the MS. (See “[To Vent the MSD](#)” on page 113.)
- 2 Open the left side panel. (See “[To Open the Analyzer Chamber](#)” on page 152.)

WARNING

The analyzer, GC/MS 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.

- 3 Loosen the analyzer side plate thumbscrews if they are fastened. (See Figure 46 on page 153.)

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

CAUTION

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

- 4 *Gently* swing the side plate out.

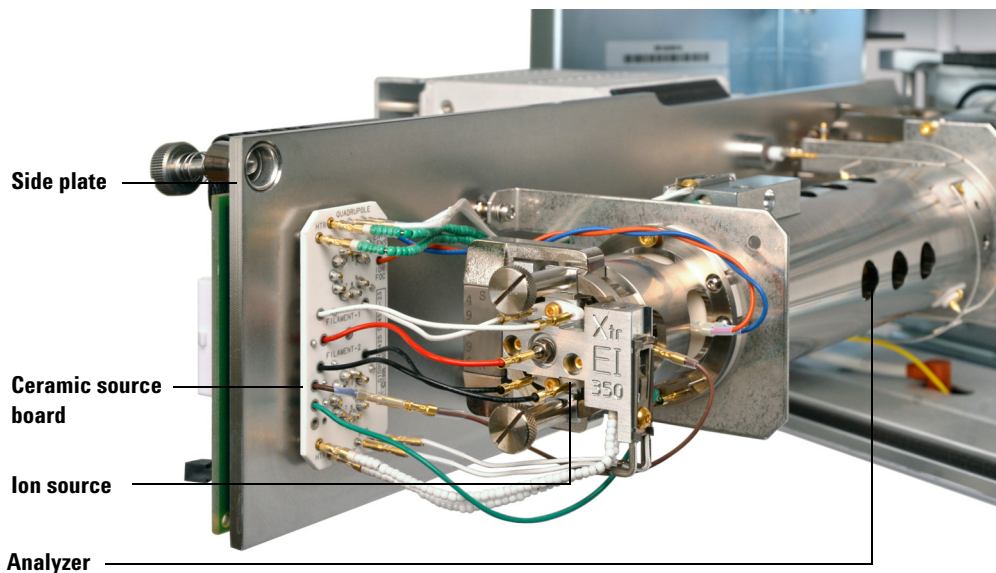


Figure 46 The analyzer chamber for an Inert+ model MSD

To Remove the EI HES

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Tweezers (8710-2460)



Procedure

- 1 Vent the MS. (See [“To Vent the MSD”](#) on page 113.)

WARNING

The analyzers, GC/MS 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.

- 2 Open the analyzer chamber. (See [“To Open the Analyzer Chamber”](#) on page 152.)

CAUTION

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

CAUTION

When disconnecting leads, pull on the connectors, not on the wires.

- 3 Remove the two large thumbscrews that hold the ion source in place. (See [Figure 47](#) on page 155.)
- 4 Disconnect the wires from the EI HES. (See [Figure 47](#) on page 155.) Do not bend the wires any more than necessary. (See [“To Connect/Disconnect Wiring to the EI HES”](#) on page 156.)

To Connect/Disconnect Wiring to the EI HES

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Pliers, long-nose (8710-1094)
- Tweezers (8710-2460)



Procedure

- 1 Use tweezers or needle nose pliers to connect/disconnect the ceramic board wire leads (red, white, black, and gray) at the source connectors. (See Figure 48.)

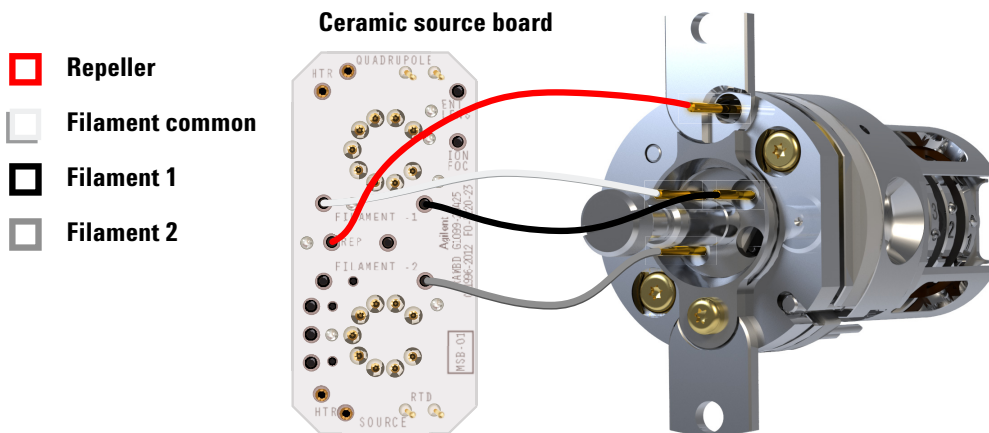


Figure 48 Wiring between the ceramic source board and the EI HES

To Disassemble the EI HES

Materials needed

- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- Cloths, clean (05980-60051)
- T6 Torx driver, (8710-2548)
- Tweezers (8710-2460)



Procedure

Refer to the exploded parts view [Figure 49](#) and the EI HES parts list [Table 17](#) on page 158 while using this procedure.

- 1 Place a clean cloth on your work surface to hold the ion source parts.
- 2 Use a T6 Torx screwdriver to remove the screw securing the filament block to the source mount, and using the finger grip, remove the filament block.
- 3 Remove the dual filament from the filament block by lifting the source body up off of the filament block, while holding the filament block so that the dual filament will not fall and become damaged.
- 4 Remove the finger grip from the filament block.
- 5 Use a T6 Torx screwdriver to remove the two screws securing the source mount to the source body.
- 6 Use the finger grip to remove the source mount from the lens body.
- 7 Remove the repeller and ring heater sensor assembly from the source body.
- 8 Separate the repeller from the ring heater assembly.
- 9 Use a T6 Torx screwdriver to remove the screw and locking ring for the lens insulator that secures the lens stack in the source body, then remove the lens stack.
- 10 If necessary, use gravity to remove the lens stack ceramic insulator from the source body.

CAUTION

Use care when removing the lenses from the lens insulator casing. Putting excessive stress on this casing can break or crack it. If this happens, do not attempt to operate with a defective lens insulator, it must be replaced.

11 Remove the five lenses from the lens insulator/holder.

CAUTION

Use care when removing the filament from the filament block. Putting excessive stress on this can break or crack it. If this happens, do not attempt to operate with a defective filament, it must be replaced.

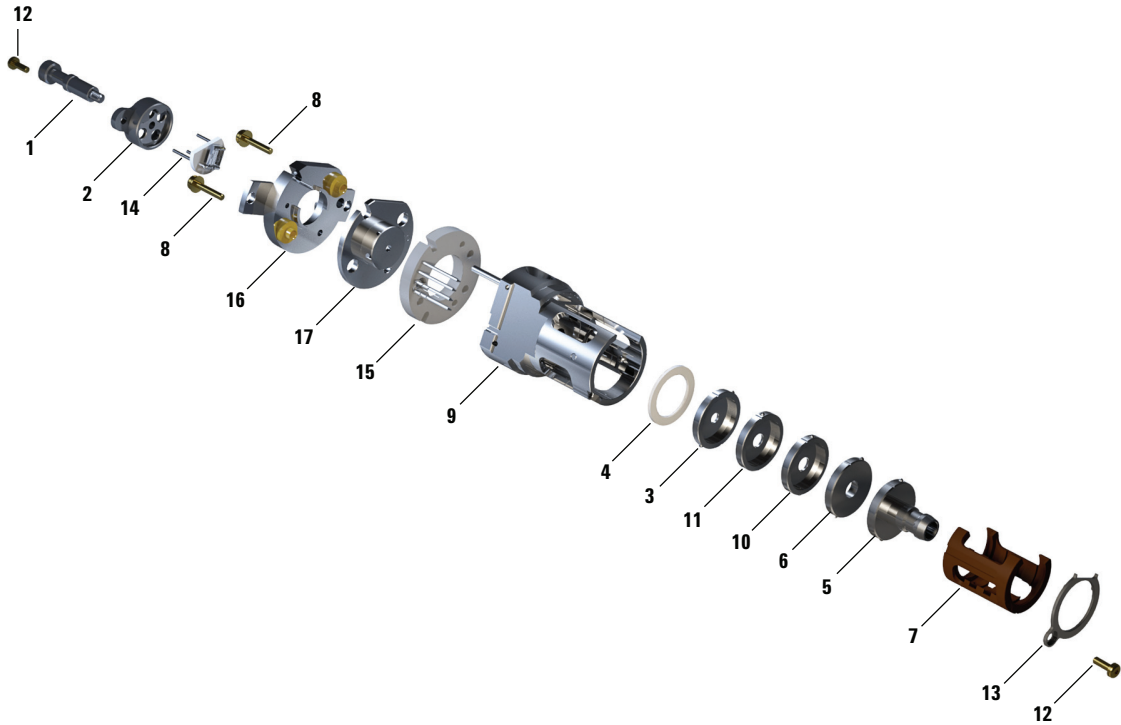


Figure 49 Exploded parts view of the EI HES

Table 17 Parts list for EI HES (Figure 49)

| Item number | Item description | Part number |
|-------------|--|-------------|
| 1 | Source finger grip | G7002-20008 |
| 2 | Filament block | G7002-20019 |
| 3 | Extractor lens (5)*, with 3 mm opening | G7002-20061 |
| 4 | Ceramic insulator for extractor | G7002-20064 |

Table 17 Parts list for EI HES (Figure 49) (continued)

| Item number | Item description | Part number |
|--------------------|---|--------------------|
| 5 | Entrance lens assembly, Extended, HES (1)* | G7002-20165 |
| 6 | Ion focus lens (2)* | G7002-20068 |
| 7 | Lens insulator/holder | G7002-20074 |
| 8 | M2 x 0.4 screw x 12 mm long gold plated screw | G7002-20083 |
| 9 | Source body | G7002-20084 |
| 10 | Post extractor lens 2 (3)* | G7002-20090 |
| 11 | Post extractor lens 1 (4)* | G7002-20104 |
| 12 | M2 x 6 mm gold plated screw | G7002-20109 |
| 13 | Locking ring lens insulator | G7002-20126 |
| 14 | High efficiency dual filament | G7002-60001 |
| 15 | Ring heater/sensor assembly | G7002-60043 |
| 16 | Source mount 1.5 mm | G7002-60053 |
| 17 | Repeller assembly | G7002-60057 |
| Not shown | HES assembly | G7004-67055 |

* The number in parenthesis is the number engraved on the lens

To Clean the EI HES

Materials needed

- Abrasive paper (5061-5896)
- Alumina abrasive powder (393706201)
- 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
 - Methanol (reagent-grade)
 - Methylene chloride (reagent-grade)
 - Acetone (reagent-grade)
- Ultrasonic bath



Procedure

- 1 Disassemble the EI HES. (See “To Disassemble the EI HES” on page 157.)
- 2 Collect the following parts to be cleaned: (See [Figure 50](#) on page 161.)
 - Filament mount
 - Source mount (does not get abrasively or ultrasonically cleaned)
 - Repeller
 - Source body
 - Extractor lens (5)
 - Post extractor lens 1 (4)
 - Post extractor lens 2 (3)
 - Ion focus lens (2)
 - Entrance lens (1)

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.

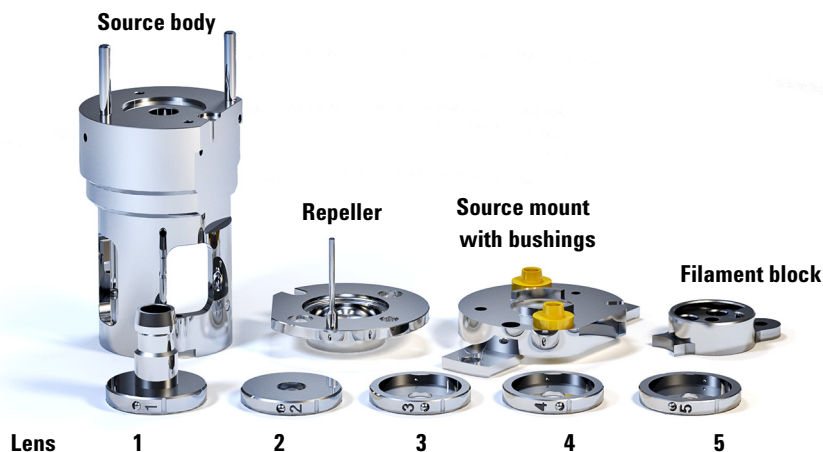


Figure 50 EI HES parts to be cleaned

CAUTION

The filaments, source heater assembly, and insulators cannot be cleaned ultrasonically. Replace these components if major contamination occurs.

- 3** If the contamination is serious, such as an oil backflow into the analyzer, seriously consider replacing the contaminated parts

CAUTION

Do not use the abrasive slurry on the source mount bushings.

- 4 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.

- 5 Rinse away all abrasive residue with reagent-grade methanol.

Ensure *all* abrasive residue is rinsed away *before* ultrasonic cleaning. If the methanol becomes cloudy or contains visible particles, rinse again.

- 6 Separate the parts that were abrasively cleaned from the parts that were not abrasively cleaned.

CAUTION

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

- 7 Ultrasonically clean the parts (each group separately) for 15 minutes in each of the following solvents:

- 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.

- 8 Place the parts in a clean beaker. *Loosely* cover the beaker with clean aluminum foil (dull side down).
- 9 Dry the cleaned parts in an oven at 100 °C for 5–6 minutes.

To Assemble the EI HES

Materials needed

- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- T6 Torx driver, (8710-2548)
- Tweezers (8710-2460)



Procedure

CAUTION

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

Refer to the exploded parts view [Figure 53](#) and the EI HES parts list [Table 18](#) on page 166 while using this procedure.

- 1** Assemble the five lenses inside the lens insulator. (See [Figure 51](#) on page 164.) The lens number is engraved into the outer circumference of each lens.
 - a** Starting with the entrance lens 1, set the lens into the end groove in the lens insulator, and rotate the lens until you feel the ball seat into the circular recess.
 - b** Insert the next 4 lenses, in numerical order, into the lens insulator. The lens chamber open end always faces the entrance lens 1. Rotate each lens until you feel the ball seat into the circular recess.

It is easier to insert lens 5 on an angle because the lens stack at this point makes the lens insulator less flexible.

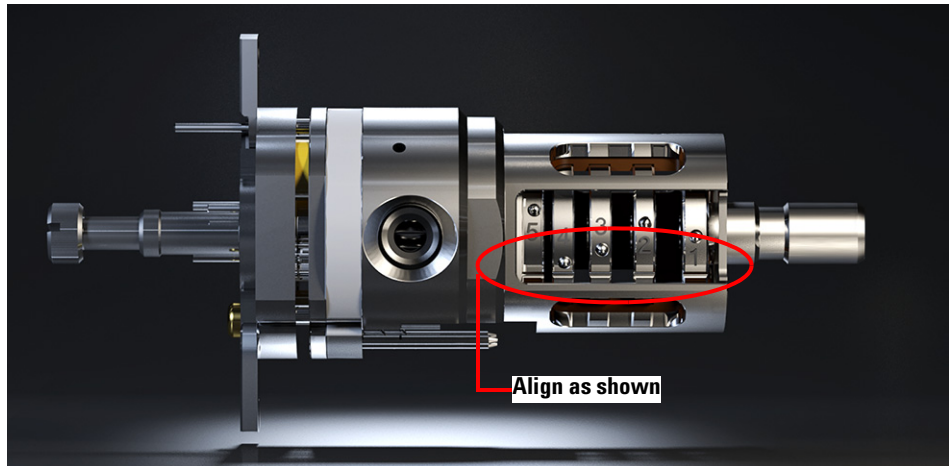


Figure 51 Assembled EI HES

- 2 Insert the extractor ceramic insulator into the source body.

CAUTION

The ceramic insulator must be positioned flat against the source body when inserting the lens stack in the next step.

- 3 Insert the lens stack encased in the insulator into the source body. (See [Figure 53](#) on page 166.) The engraved numbers face the full opening extending to the end of the source body. Verify that the ceramic is sitting flush at the end of the source body.
- 4 Using the T6 Torx driver, install and secure the gold plated setscrew and lock ring lens insulator that holds the lens assembly in place.



Figure 52 Secure the lens screw and lock ring insulator

- 5 Place the heater/sensor assembly over the guide pins on the source body with the four electrical pins oriented down the flat side of the source body.
- 6 Place the repeller onto the heater/sensor assembly with the flat side of the repeller circumference aligned with the interface socket in the source body.
- 7 Place the source mount over the repeller.
- 8 Finger tighten the two gold plated screws using a T6 Torx screwdriver to secure the source mount to the source body.

CAUTION

Do not overtighten the screws in the source body. Damage may occur to the repeller.

- 9 Thread the finger grip into the filament block.
- 10 On the side of the filament block opposite the finger grip, orient the dual filament ceramic holder so that it lines up with the flat of the filament block. Fully insert the three filament leads into the filament block.
- 11 Inset the filament block into the source holder, and use a T6 Torx screwdriver to secure it to the holder with the gold plated screw.

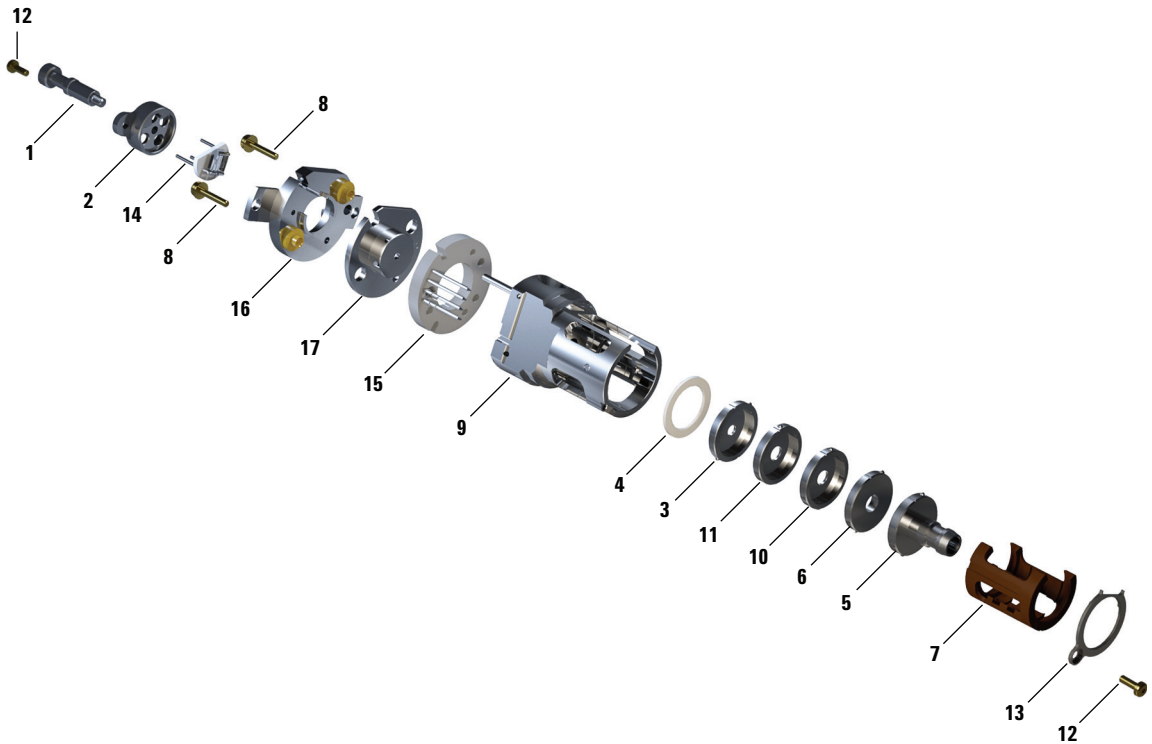


Figure 53 Assembling the EI HES

Table 18 Parts list for EI HES (Figure 53)

| Item number | Item description | Part number |
|-------------|--|-------------|
| 1 | Source finger grip | G7002-20008 |
| 2 | Filament block | G7002-20019 |
| 3 | Extractor lens (5)*, with 3 mm opening | G7002-20061 |
| 4 | Ceramic insulator for extractor | G7002-20064 |
| 5 | Entrance lens assembly, Extended, HES (1)* | G7002-20165 |
| 6 | Ion focus lens (2)* | G7002-20068 |
| 7 | Lens insulator/holder | G7002-20074 |

Table 18 Parts list for EI HES (Figure 53) (continued)

| Item number | Item description | Part number |
|-------------|---|-------------|
| 8 | M2 x 0.4 screw x 12 mm long gold plated screw | G7002-20083 |
| 9 | Source body | G7002-20084 |
| 10 | Post extractor lens 2 (3)* | G7002-20090 |
| 11 | Post extractor lens 1 (4)* | G7002-20104 |
| 12 | M2 x 6 mm gold plated screw | G7002-20109 |
| 13 | Locking ring lens insulator | G7002-20126 |
| 14 | High efficiency dual filament | G7002-60001 |
| 15 | Ring heater/sensor assembly | G7002-60043 |
| 16 | Source mount 1.5 mm | G7002-60053 |
| 17 | Repeller assembly | G7002-60057 |
| Not shown | HES assembly | G7004-67055 |

* The number in parenthesis is the number engraved on the lens

CAUTION

Use care when inserting the lenses into the lens insulator casing. Putting excessive stress on this casing can break or crack it. If this happens, do not attempt to operate with a defective lens insulator, it must be replaced.

To Remove the EI HES filaments

Materials needed

- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- Cloths, clean (05980-60051)
- T6 Torx driver, (8710-2548)



Procedure

- 1 Vent the MS. (See “To Vent the MSD” on page 113.)

CAUTION

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

WARNING

The analyzer, GC/MS 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.

- 2 Open the analyzer chamber. (See “To Open the Analyzer Chamber” on page 152.)
- 3 Remove the ion source and place it on a clean cloth on your work surface. (See “To Remove the EI HES” on page 154.)
- 4 Remove the screw securing the filament block to the source mount. (See [Figure 54](#) on page 169.)

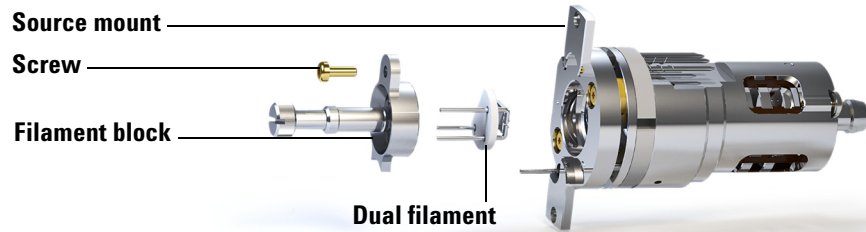


Figure 54 Replacing the dual filament

- 5 Use the hand grip on the filament block to remove the filament block from the source.

CAUTION

Use extra caution when removing the dual filament, as it is extremely brittle.

- 6 Remove the dual filament from the filament block by lifting the source body up off of the filament block, while holding the filament block so that the dual filament will not fall and become damaged.

To Install the EI HES Filament

Materials needed

- Filament assembly, high efficiency dual filament (G7001-60061)
- Hex ball driver, 1.5 mm (8710-1570)
- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- T6 Torx driver, (8710-2548)



Procedure

- 1 Remove the old filament. (See “To Remove the EI HES filaments” on page 168.)
- 2 Insert the 3 pins on the dual filament through the rear of the filament block. (See [Figure 54](#) on page 169.)
- 3 Place the filament block in the source mount.
- 4 Use a T6 Torx screwdriver to tighten the screw securing the filament block to the source mount.
- 5 Reinstall the ion source. (See “To Install the EI HES” on page 171.)
- 6 Close the analyzer chamber. (See “To Close the Analyzer Chamber” on page 196.)
- 7 Pumpdown the MSD. (See “To Pumpdown the MSD” on page 116.)
- 8 Autotune the MSD.

To Install the EI HES

Materials needed

- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- Tweezers (8710-2460)



Procedure

CAUTION

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

- 1 Align the EI HES so that the slotted opening for the lens stack, where the lens numbers are visible, is on the right side. Also, position it so the two attachment tabs of the source mount align with the corresponding slots in the source radiator. Slide the ion source into the source radiator until resistance is felt.
- 2 Snap the source into place so that the source mount tabs are flush against the source mount attachment surface of radiator. Some force is required to overcome the resistance of the electrical contacts spring loaded surfaces.
- 3 Connect the EI HES wires. (See [“To Connect/Disconnect Wiring to the EI HES”](#) on page 156.)
- 4 Install and hand-tighten the source thumbscrews. Do not overtighten the thumbscrews.
- 5 Close the analyzer chamber. (See [“To Close the Analyzer Chamber”](#) on page 196.)
- 6 Pumpdown the MSD. (See [“To Pumpdown the MSD”](#) on page 116.)
- 7 Tune the MSD. (See [“To Tune the MSD in EI Mode”](#) on page 104.)

To Remove the EI XTR, SS, or Inert Source

Materials needed

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



Procedure

- 1 Vent the MSD. (See “To Vent the MSD” on page 113.)

WARNING

The analyzers, GC/MS 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

- 2 Open the analyzer chamber. (See “To Open the Analyzer Chamber” on page 152.)

CAUTION

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

CAUTION

When disconnecting leads, pull on the connectors, not the wires.

- 3 Disconnect the wires from the EI source. Do not bend the wires any more than necessary. (See “To Connect/Disconnect Wiring from the EI XTR, SS, and Inert Sources” on page 173.)
- 4 Remove the thumbscrews that hold the ion source in place. (See Figure 62 on page 193.)
- 5 Pull the ion source out of the source radiator.

To Connect/Disconnect Wiring from the EI XTR, SS, and Inert Sources

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Pliers, long-nose (8710-1094)
- Tweezers (8710-2460)



Procedure

- 1 Use tweezers or needle nose pliers to connect/disconnect the ceramic source board wire leads at the source connectors. (See [Figure 55](#).)
- 2 Use tweezers or needle nose pliers to connect/disconnect the source heater wire leads at the ceramic source board.

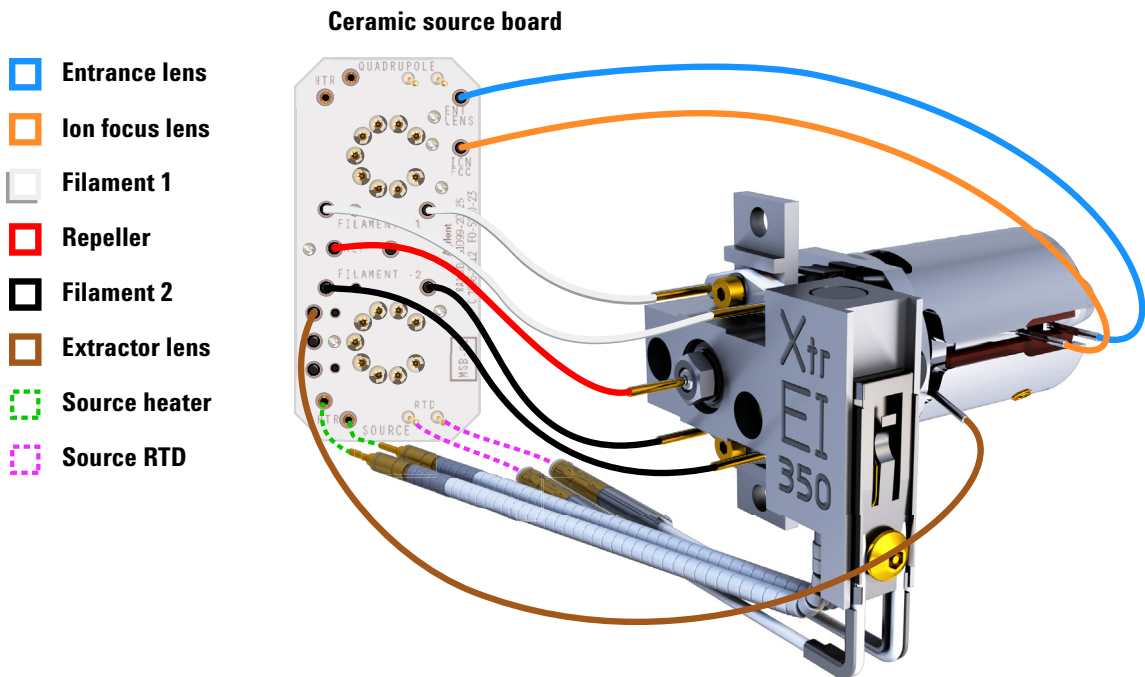


Figure 55 Wiring for the EI XTR Source

To Disassemble the EI SS or EI Inert 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)
- Wrench, open-end, 10 mm (8710-2353)



Procedure

Refer to the exploded parts view [Figure 56](#) and the EI SS and EI Inert parts list [Table 19](#) on page 175 while using this procedure.

- 1 Remove the EI source. (See “[To Remove the EI HES](#)” on page 154 or “[To Remove the EI XTR, SS, or Inert Source](#)” on page 172.)
- 2 Remove the two gold plated screws from the filaments and remove the filaments from the source.
- 3 Loosen the two gold plated screws from the source heater block assembly, and separate the repeller assembly from the source body. The repeller assembly includes the source heater block assembly, repeller, and related parts.
- 4 Remove the repeller nut and washers then remove the repeller from the source heater block assembly.
- 5 Remove the repeller insulators and the repeller block insert from the source heater block assembly.
- 6 Remove the gold plated setscrew from the side of the source body.
- 7 Push on the drawout plate to remove the entrance lens, ion focus lens, drawout cylinder, and drawout plate from the other end of the source body.
- 8 Unscrew the interface socket. A 10-mm open-end wrench fits the flats on the interface socket.
- 9 Remove the entrance lens and ion focus lens from the lens insulator.

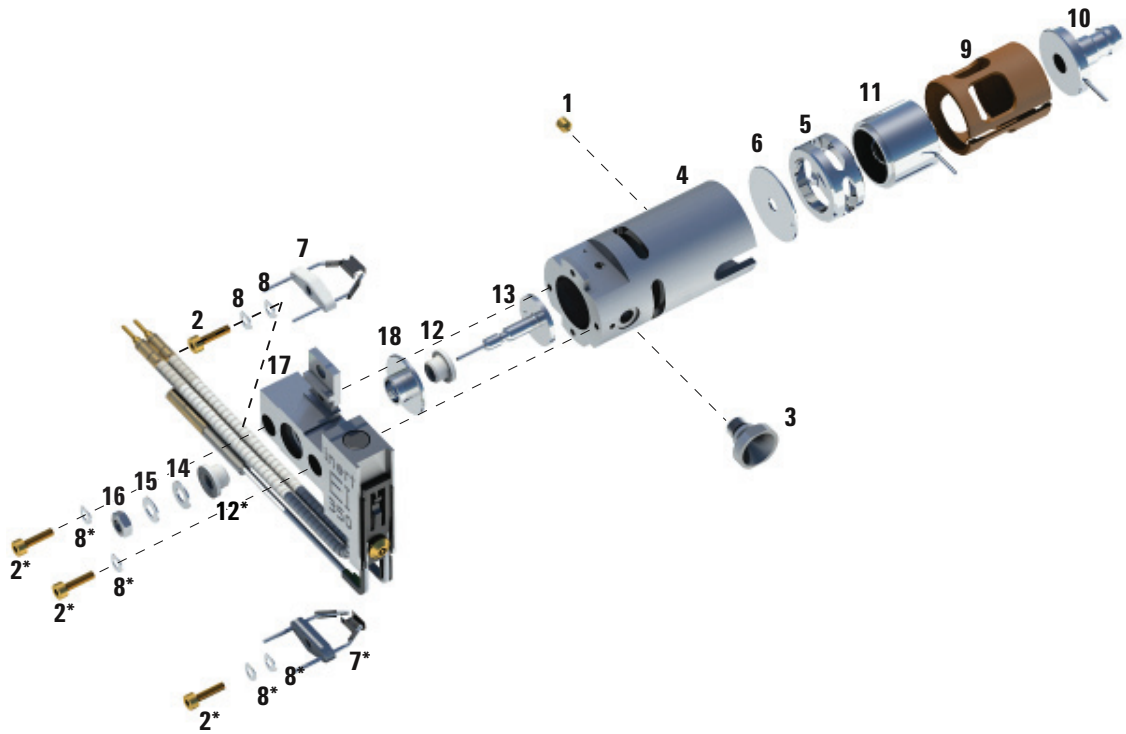


Figure 56 Disassembling the EI SS or Inert source

Table 19 Parts list for the EI SS or Inert source (Figure 56)

| Item number | Item description | Part number (SS) | Part number (Inert) |
|-------------|-----------------------|------------------|---------------------|
| 1 | Gold plated set screw | G1999-20022 | G1999-20022 |
| 2 | Gold plated screw | G3870-20021 | G3870-20021 |
| 3 | Interface socket | G1099-20136 | G1099-20136 |
| 4 | Source body | G1099-20130 | G2589-20043 |
| 5 | Drawout cylinder | G1072-20008 | G1072-20008 |
| 6 | Drawout plate | 05971-20134 | G2589-20100 |
| 7 | 4-turn filament | G7005-60061 | G7005-60061 |

Table 19 Parts list for the EI SS or Inert source (Figure 56) (continued)

| Item number | Item description | Part number (SS) | Part number (Inert) |
|-------------|------------------------------|------------------|---------------------|
| 8 | Spring washer | 3050-1374 | 3050-1374 |
| 8 | Flat washer | 3050-0982 | 3050-0982 |
| 9 | Lens insulator | G3170-20530 | G3170-20530 |
| 10 | Entrance lens | G3170-20126 | G3170-20126 |
| 11 | Ion focus lens | 05971-20143 | 05971-20143 |
| 12 | Repeller insulator | G1099-20133 | G1099-20133 |
| 13 | Repeller | G3870-60172 | G3870-60173 |
| 14 | Flat washer | 3050-0627 | 3050-0627 |
| 15 | Belleville spring washer | 3050-1301 | 3050-1301 |
| 16 | Repeller nut | 0535-0071 | 0535-0071 |
| 17 | Source heater block assembly | G3870-60180 | G3870-60179 |
| 18 | Repeller block insert | G3870-20135 | G3870-20135 |

To Disassemble the EI XTR 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)
- Wrench, open-end, 10 mm (8710-2353)



Procedure

Refer to the exploded parts view [Figure 57](#) and the EI EXT parts list [Table 20](#) on page 178 while using this procedure.

- 1** Remove the EI XTR source. (See “[To Remove the EI XTR, SS, or Inert Source](#)” on page 172.)
- 2** Remove the filaments by removing the two gold plated screws and separating the filaments from the source.
- 3** Loosen the two gold plated screws on the source heater block assembly, and separate the repeller assembly from the source body. The repeller assembly includes the source heater block assembly, repeller, and related parts.
- 4** Remove the gold plated setscrews from the side of the source body.
- 5** Pull the entrance lens and ion focus lens to remove them from the source body.
- 6** Remove the extractor lens and insulator.
- 7** Separate the entrance lens and ion focus lens from the lens insulator.
- 8** Remove the repeller nut, washers, and insulator from the front side of the source heater block assembly, then remove the repeller, insulator, and repeller block insert from the opposite side.

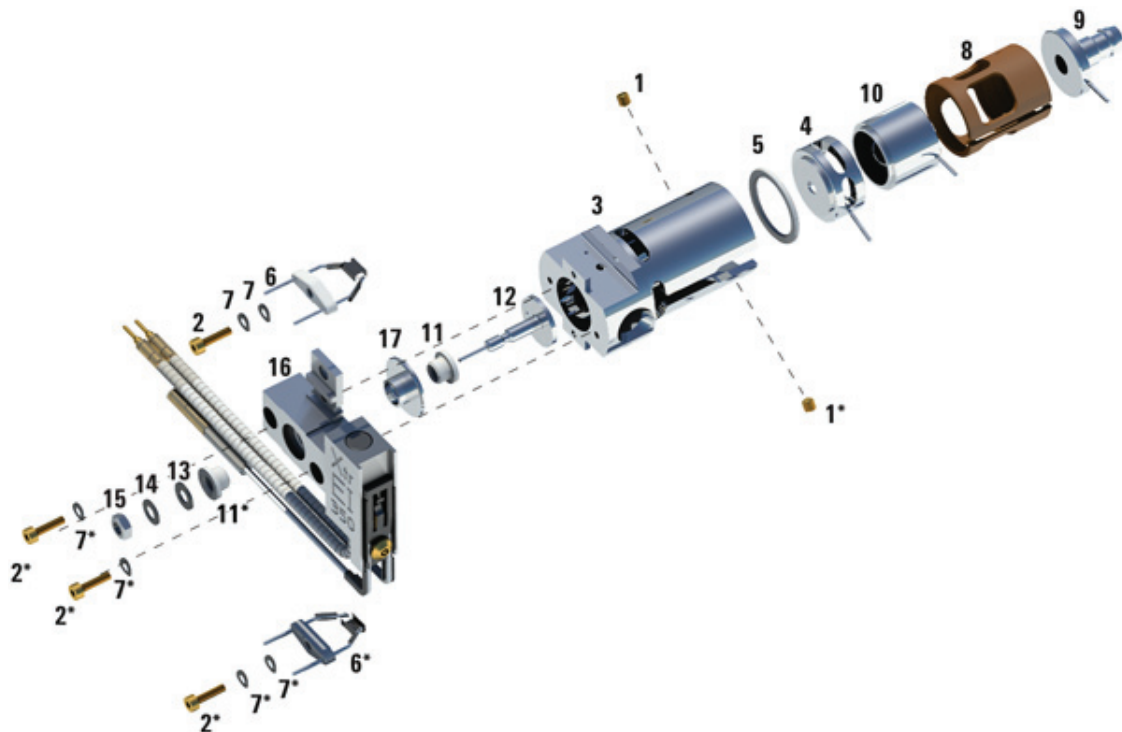


Figure 57 Disassembling the EI XTR source

Table 20 Parts list for EI XTR source (Figure 57)

| Item | Description | Part number |
|------|--------------------------|-------------|
| 1 | Setscrews | G3870-20446 |
| 2 | Screws | G3870-20021 |
| 3 | Source body | G3870-20440 |
| 4 | Extractor lens | G3870-20444 |
| 5 | Extractor lens insulator | G3870-20445 |
| 6 | Filaments | G7005-60061 |
| 7 | Spring washer | 3050-1301 |

Table 20 Parts list for EI XTR source (Figure 57) (continued)

| Item | Description | Part number |
|-------------|----------------------------------|--------------------|
| 7 | Flat washer | 3050-0982 |
| 8 | Lens insulator | G3870-20530 |
| 9 | Entrance lens assembly, Extended | G7000-20026 |
| 10 | Ion focus lens | 05971-20143 |
| 11 | Repeller insulator | G1099-20113 |
| 12 | Repeller | G3870-60171 |
| 13 | Flat washer | 3050-0891 |
| 14 | Belleville spring washer | 3050-1301 |
| 15 | Repeller nut | 0535-0071 |
| 16 | Source heater block assembly | G3870-60177 |
| 17 | Repeller block insert | G3870-20135 |
| Not shown | EI XTR source assembly | G7003-67720 |

To Clean an EI XTR, SS, or Inert 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

Preparation

- 1 Disassemble the ion source. (See “To Disassemble the EI SS or EI Inert Source” on page 174 or “To Disassemble the EI XTR Source” on page 177.)
- 2 Collect the following parts to be cleaned for an EI SS or Inert source: (See [Figure 58](#) on page 182.)
 - Repeller
 - Interface socket
 - Source body
 - Repeller block insert
 - Drawout plate
 - Drawout cylinder
 - Ion focus lens
 - Entrance lens

3 Collect the following parts to be cleaned for an EI XTR source: (See Figure 58 on page 182.)

- Repeller
- Repeller block insert
- Source body
- Extractor lens
- 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.



Figure 58 Source parts to be cleaned

**CAUTION****Procedure**

The filaments, source heater assembly, and insulators cannot be cleaned ultrasonically. Replace these components if major contamination occurs.

- 1 If the contamination is serious, such as an oil backflow into the analyzer, seriously consider replacing the contaminated parts.
- 2 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.
- 3 Rinse away all abrasive residue with reagent-grade methanol.
Make sure **all** abrasive residue is rinsed **before** ultrasonic cleaning. If the methanol becomes cloudy or contains visible particles, rinse again three times.
- 4 Separate the parts that were abrasively cleaned from the parts that were not abrasively cleaned.
- 5 Ultrasonically clean the parts (each group separately) for 15 minutes. For dirty parts, use all three solvents in the order shown, cleaning 15 minutes with each of the following solvents:
 - Methylene chloride (reagent-grade)
 - Acetone (reagent-grade)
 - Methanol (reagent-grade)

For routine cleaning, cleaning with methanol is sufficient.

WARNING

All of these solvents are hazardous. Work in a fume hood and take all appropriate precautions.

- 6 Place the parts in a clean beaker. **Loosely** cover the beaker with clean aluminum foil (dull side down).
- 7 Dry the cleaned parts in an oven at 100 °C for 5–6 minutes.

WARNING

Let the parts cool before you handle them.

6 General Maintenance

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 Assemble an EI SS or Inert 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)
- Wrench, open-end, 10 mm (8710-2353)



Procedure

Refer to the exploded parts view [Figure 59](#) and EI SS and EI Inert parts list [Table 21](#) on page 186 while using this procedure.

- 1 Assemble the repeller assembly.
 - a Install the repeller block insert into the source heater block assembly.
 - b Install the repeller insulators into the source heater block assembly and repeller block insert.
 - c Install the repeller through the repeller insulators, then put the flat washer followed by the belleville spring washer onto the end of the repeller shaft, and secure finger tight with the repeller nut.
- 2 Insert the drawout plate and the drawout cylinder into the source body.
- 3 Assemble the ion focus lens, entrance lens, and lens insulators.
- 4 Slide these assembled parts into the source body.
- 5 Install the setscrew that holds the lenses in place.

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 Install the interface socket.
- 7 Attach the repeller assembly to the source body using the two gold plated screws and spring washers.
- 8 Install the filaments using the two gold plated screws and spring washers.

CAUTION

Do not overtighten the interface socket. Overtightening could strip the threads.

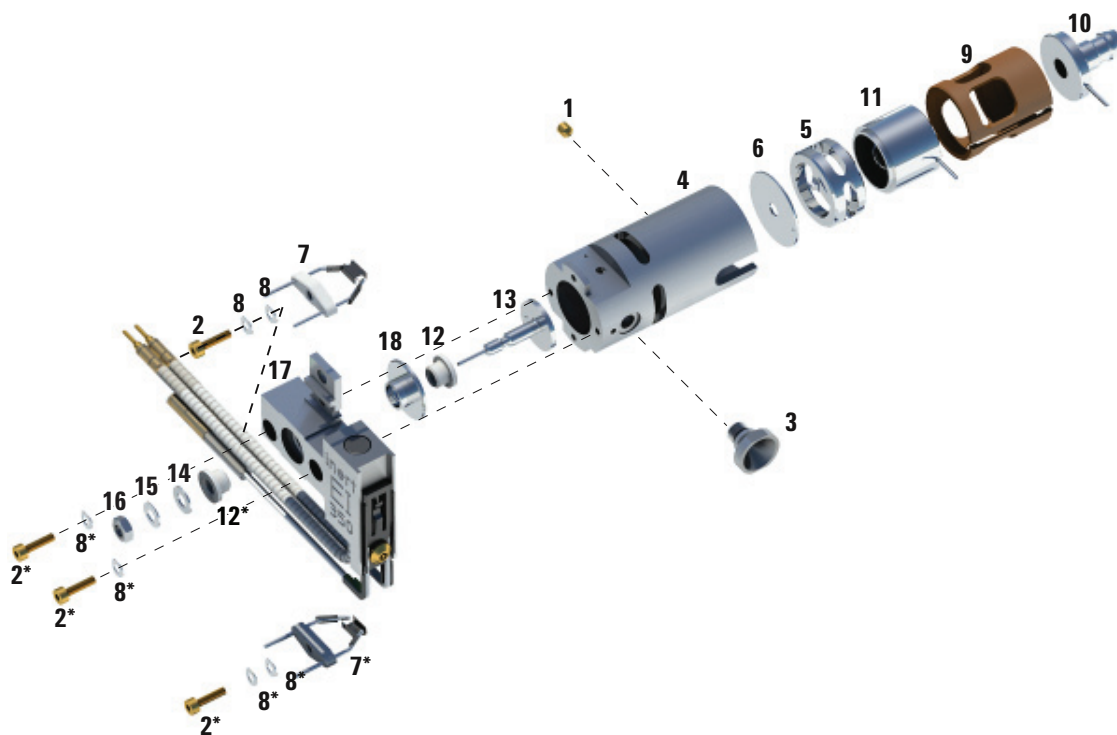


Figure 59 Assembling the EI standard or EI Inert source

Table 21 Parts list for the EI standard or EI Inert source (Figure 59)

| Item number | Item description | Part number (SSL) | Part number (Inert) |
|-------------|-----------------------|-------------------|---------------------|
| 1 | Gold plated set screw | G1999-20022 | G1999-20022 |
| 2 | Gold plated screw | G3870-20021 | G3870-20021 |
| 3 | Interface socket | G1099-20136 | G1099-20136 |
| 4 | Source body | G1099-20130 | G2589-20043 |

Table 21 Parts list for the EI standard or EI Inert source (Figure 59) (continued)

| Item number | Item description | Part number (SSL) | Part number (Inert) |
|-------------|------------------------------|-------------------|---------------------|
| 5 | Drawout cylinder | G1072-20008 | G1072-20008 |
| 6 | Drawout plate | 05971-20134 | G2589-20100 |
| 7 | 4-turn filament | G7005-60061 | G7005-60061 |
| 8 | Spring washer | 3050-1374 | 3050-1374 |
| 8 | Flat washer | 3050-0982 | 3050-0982 |
| 9 | Lens insulator | G3170-20530 | G3170-20530 |
| 10 | Entrance lens | G3170-20126 | G3170-20126 |
| 11 | Ion focus lens | 05971-20143 | 05971-20143 |
| 12 | Repeller insulator | G1099-20133 | G1099-20133 |
| 13 | Repeller | G3870-60172 | G3870-60173 |
| 14 | Flat washer | 3050-0627 | 3050-0627 |
| 15 | Belleville spring washer | 3050-1301 | 3050-1301 |
| 16 | Repeller nut | 0535-0071 | 0535-0071 |
| 17 | Source heater block assembly | G3870-60180 | G3870-60179 |
| 18 | Repeller block insert | G3870-20135 | G3870-20135 |

To Assemble the EI XTR 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)
- Wrench, open-end, 10 mm (8710-2353)



Procedure

Refer to the exploded parts view [Figure 60](#) and EI XTR parts list [Table 22](#) on page 189 while using this procedure.

- 1 Slide the ceramic washer into the source body.
- 2 Insert the extractor lens into the source body, flat side first.
- 3 Insert the entrance lens and ion focus lens into the insulator in the order shown in [Figure 60](#) on page 189.
- 4 Slide the insulator containing the ion focus and entrance lens into the source body, with the ion focus lens against the extractor lens.
- 5 Install the two gold plated setscrews that holds the lenses in place.
- 6 Assemble the repeller assembly.
 - a Install the repeller block insert into the source heater block assembly.
 - b Install the repeller insulators into the source heater block assembly and repeller block insert.
 - c Install the repeller through the repeller insulators, then put the flat washer followed by the belleville spring washer onto the end of the repeller shaft and secure finger tight with the repeller nut.

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.

- 7 Attach the repeller assembly to the source body using the two gold plated screws and spring washers.
- 8 Install the filaments using the two gold plated screws and spring washers.

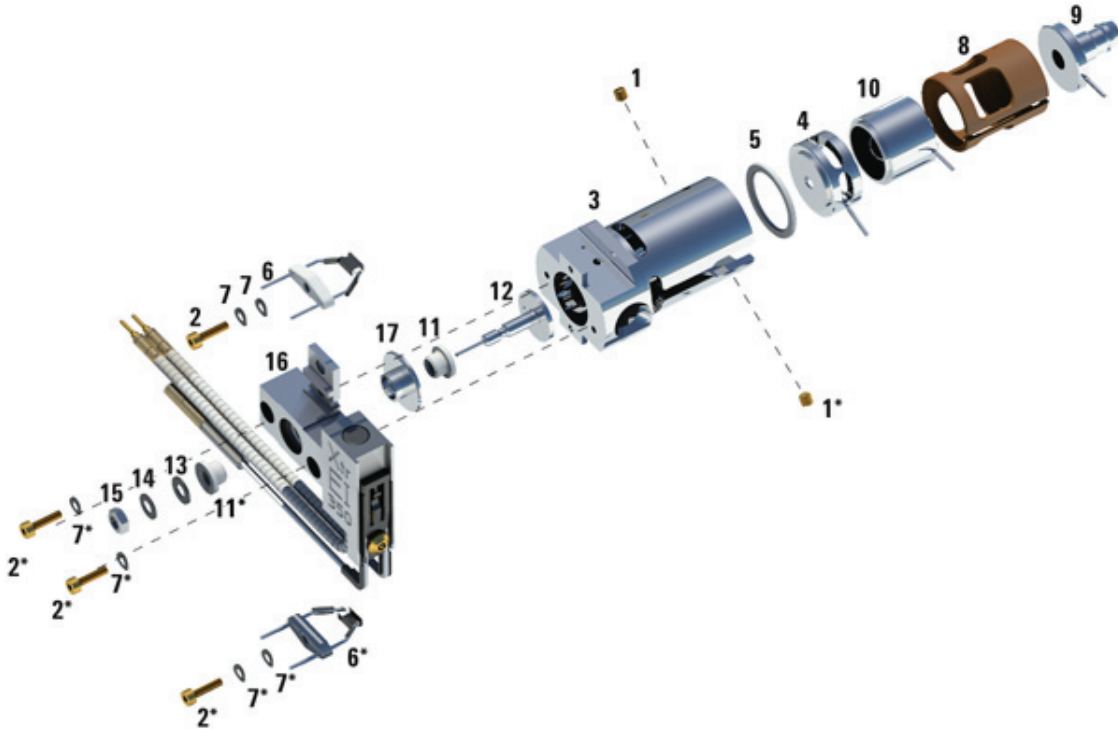


Figure 60 Assembling the EI XTR source

Table 22 Parts list for EI XTR source (Figure 60)

| Item | Description | Part number |
|------|-------------|-------------|
| 1 | Setscrews | G3870-20446 |
| 2 | Screws | G3870-20021 |
| 3 | Source body | G3870-20440 |

Table 22 Parts list for EI XTR source (Figure 60) (continued)

| Item | Description | Part number |
|-------------|----------------------------------|--------------------|
| 4 | Extractor lens | G3870-20444 |
| 5 | Extractor lens insulator | G3870-20445 |
| 6 | Filaments | G7005-60061 |
| 7 | Spring washer | 3050-1301 |
| 7 | Flat washer | 3050-0982 |
| 8 | Lens insulator | G3870-20530 |
| 9 | Entrance lens assembly, Extended | G7000-20026 |
| 10 | Ion focus lens | 05971-20143 |
| 11 | Repeller insulator | G1099-20113 |
| 12 | Repeller | G3870-60171 |
| 13 | Flat washer | 3050-0891 |
| 14 | Belleville spring washer | 3050-1301 |
| 15 | Repeller nut | 0535-0071 |
| 16 | Source heater block assembly | G3870-60177 |
| 17 | Repeller block insert | G3870-20135 |
| Not shown | EI XTR source assembly | G7003-67720 |

To Replace a Filament in an EI XTR, SS, or Inert Source

Materials needed

- Filament assembly (G7005-60061)
- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- Hex ball driver, 1.5-mm (8710-1570)



Procedure

- 1 Vent the MSD. (See “Venting the MSD” on page 91.)

WARNING

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

- 2 Open the analyzer chamber. (See “To Open the Analyzer Chamber” on page 152.)
- 3 Remove the ion source. (See “To Remove the EI XTR, SS, or Inert Source” on page 172.)
- 4 Remove the gold plated screw and washer for the filaments. (See Figure 61 on page 192.)

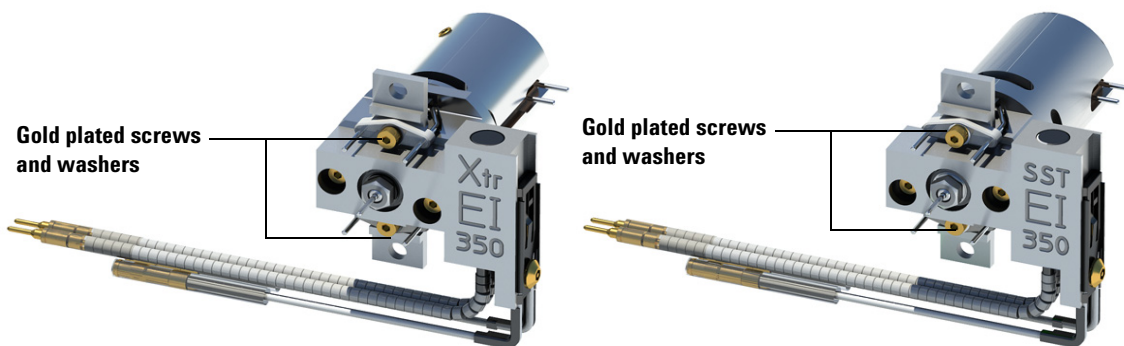


Figure 61 Changing the filament

- 5 Secure the new filaments with the gold plated screw and washer. (See [Figure 61](#).)
- 6 After installing the filament, verify that it is not grounded to the source body.
- 7 Install the EI source. (See “[To Install the EI XTR, SS, or Inert Source](#)” on page 193.)
- 8 Close the analyzer chamber. (See “[To Close the Analyzer Chamber](#)” on page 196.)
- 9 Pumpdown the MSD. (See “[To Pumpdown the MSD](#)” on page 116.)
- 10 Autotune the MSD. (See “[To Tune the MSD in EI Mode](#)” on page 104.)
- 11 In the Manual Tune dialog, the **Filament** parameter allows you to enter **1** or **2** for the filament number. Whichever number was present during the previous autotune enter the other filament number.
- 12 Autotune the MSD again.
- 13 Enter the filament number that gave the best results.
If you decide to use the first filament number, run Autotune again to make sure the tune parameters are compatible with the filament.
- 14 Select **Save Tune Parameters** from the **File** menu.

To Install the EI XTR, SS, or Inert Source

Materials needed

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



Procedure

- 1 Slide the ion source into the source radiator. (See Figure 62.)

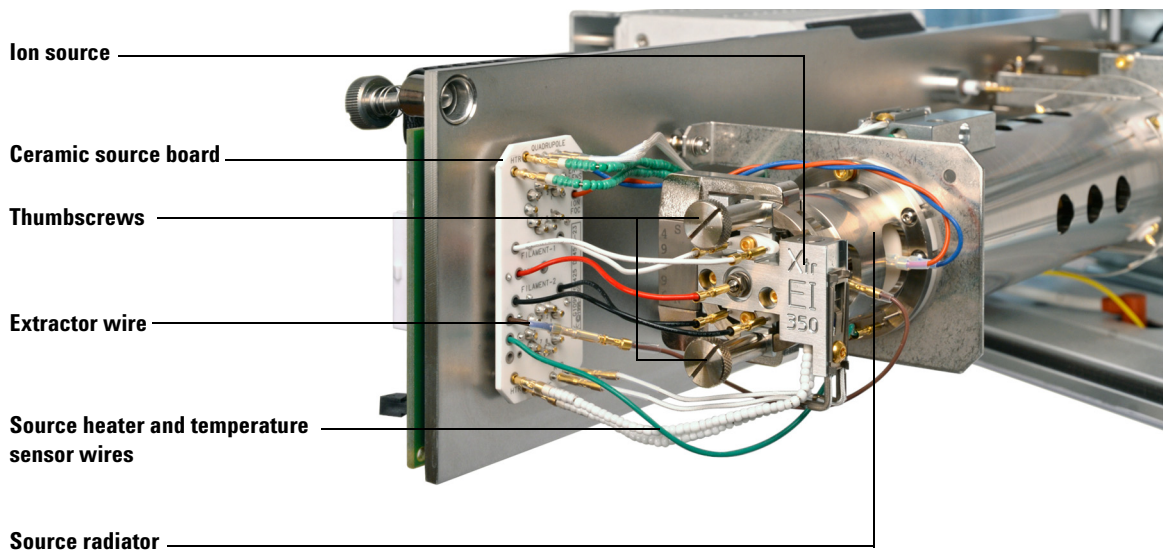


Figure 62 Installing the EI source

- 2 Insert and hand tighten the source thumbscrews. Do not overtighten the thumbscrews. (See Figure 62.)
- 3 Connect the ion source wires. (See “To Connect/Disconnect Wiring from the EI XTR, SS, and Inert Sources” on page 173.)
- 4 Close the analyzer chamber. (See “To Close the Analyzer Chamber” on page 196.)

To Replace the Electron Multiplier Horn

The replacement EM horn part number for this Series 2 detector is stamped on the front face of the detector. You can determine which series detector you have without having to directly check the detector. The detector series is displayed as “Triple Axis Series 2” in the detector tab of manual tune, in the detector section on the second page of the tune report and in the pumpdown window.

Materials needed

- Electron multiplier horn (Series 2 Detector G7002-80103)
- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- Pliers, long-nose (8710-1094)



Procedure

- 1 Vent the MS. (See “[To Vent the MSD](#)” on page 113.)

WARNING

The analyzer, GC/MS 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.

- 2 Open the analyzer chamber. (See “[To Open the Analyzer Chamber](#)” on page 152.)
- 3 Open the retaining clip. (See [Figure 63](#) on page 195.) Lift the arm of the clip up and swing the clip away from the electron multiplier horn.
- 4 Slide the blue signal wire from the connector in the sideplate.
- 5 Remove the electron multiplier horn.
- 6 Hold the new horn with blue signal wire end down, and attach the signal wire to the connector in the sideplate.

- 7 Slide the electron multiplier horn into position.
- 8 Close the retaining clip.
- 9 Close the analyzer chamber. (See “To Close the Analyzer Chamber” on page 196.)

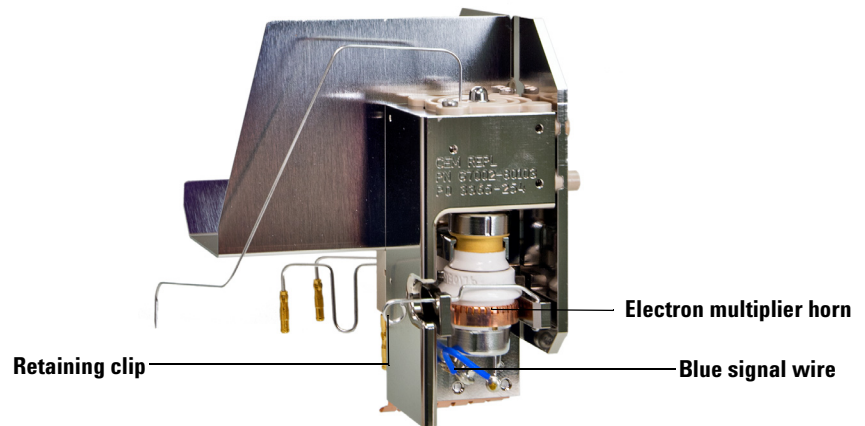


Figure 63 Replacing the EM horn on a Series II Detector



Figure 64 Electron multiplier horn

To Close the Analyzer Chamber



Procedure

- 1 Ensure all the internal analyzer electrical leads are correctly attached. Wiring is not the same for both the EI and CI sources. (See “To Connect/Disconnect Wiring from the EI XTR, SS, and Inert Sources” on page 173.)

Check the side plate O-ring.

Make sure the O-ring has a *very* light coat of Apiezon L high vacuum grease. If the O-ring is very dry, it may not seal well. If the O-ring looks shiny, it has too much grease on it. Refer to the *5977 Series MS Troubleshooting and Maintenance Manual* for lubricating instructions.

CAUTION

Do not force the analyzer door when closing, or you may damage the quadrupole.

- 2 Swing the analyzer side plate closed.
- 3 Ensure the vent valve is closed.
- 4 If hydrogen or other flammable or toxic substance is used for carrier gas, *gently* hand-tighten the top thumbscrew on the analyzer side plate.
- 5 Pumpdown the MSD. (See “To Pumpdown the MSD” on page 116.)

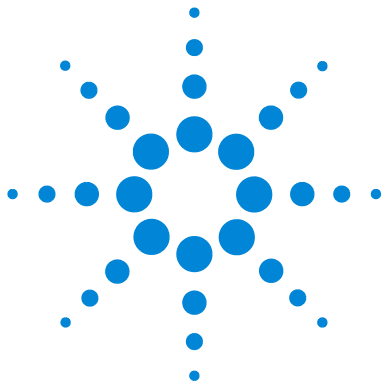
WARNING

The top thumbscrew must be fastened if hydrogen (or other hazardous gas) is being used as the GC carrier gas or if hydrogen is used for the JetClean system. 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.

- 6 Once the MSD has pumped down, close the left analyzer cover and replace the window cover.
- 7 Tune the MSD.



7 CI Maintenance

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This chapter describes maintenance procedures and requirements that are unique to 5977B Series MSDs equipped with the CI hardware.

If you are using the *Agilent Intuvo 9000 Gas Chromatograph* with your MS, Chemical Ionization (CI) and the JetClean system are not currently supported.



General Information

Ion source cleaning

The main 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-ventilated room.

Ammonia

Ammonia, used as a reagent gas, increases the need for foreline pump maintenance. Ammonia causes foreline pump oil to break down more quickly. Therefore, the oil in the standard foreline vacuum pump must be checked and replaced more frequently.

Always purge the MSD with methane after using ammonia.

Be sure to install the ammonia with the tank 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 MSD for operation in CI mode requires special care to avoid contamination and air leaks.

Guidelines

- Before venting in EI mode for the installation of the CI source, verify that the GC/MSD system is performing correctly. (See “[To Verify EI System Performance](#)” on page 108.)
- 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 Switch from the EI HES to the CI Source

Procedure

CAUTION

Always verify GC/MS performance in EI before switching to CI operation.

- 1 Vent the MS. (See “[To Vent the MSD](#)” on page 113.) The software will prompt you for the appropriate actions.
- 2 Open the left side access panel. (See “[To Open the Analyzer Chamber](#)” on page 152.)

CAUTION

Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded antistatic wrist strap. (See “[Electrostatic discharge is a threat to MSD electronics](#)” on page 21.) Take antistatic precautions **before** you open the analyzer chamber.

- 3 Open the analyzer chamber. (See “[To Open the Analyzer Chamber](#)” on page 152.)
- 4 Remove the EI HES. (See “[To Remove the EI HES](#)” on page 154.)
- 5 Place the EI HES in the storage container.
- 6 Remove the EI HES radiator. (See “[To Remove the EI HES Radiator](#)” on page 200.)
- 7 Place the EI HES radiator in the storage container.
- 8 Remove the CI source radiator from the storage container.
- 9 Install the CI source radiator. (See “[To Install the CI Source Radiator](#)” on page 202.)
- 10 Remove the CI source from the storage container.
- 11 Install the CI source. This requires shortening the column so that it protrudes from the transfer line by 1 to 2 mm. (See “[To Install the CI Source](#)” on page 225.)
- 12 Pumpdown the MSD. (See “[To Pumpdown the MSD in CI Mode](#)” on page 124.)

To Remove the EI HES Radiator

Materials needed

- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- Tweezers (8710-2460)
- T10 Torx screwdriver (5182-3466)



WARNING

Procedure

The analyzers, GC/MS 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.
 - Make sure you use an antistatic wrist strap and take other antistatic precautions before touching analyzer components.
 - When disconnecting leads, pull on the connectors, not on the wires
-

- 1 Remove the EI HES. (See [“To Remove the EI HES”](#) on page 154.)
- 2 Disconnect the wires from the EI HES radiator. (See [“To Connect/Disconnect Wiring to the EI HES Radiator”](#) on page 201.)
- 3 Use a T10 Torx screwdriver to loosen the 2 screws that secure the radiator to the analyzer, and place the radiator in its storage container.

To Connect/Disconnect Wiring to the EI HES Radiator

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Pliers, long-nose (8710-1094)
- Tweezers (8710-2460)

Procedure

- 1 Use tweezers or needle nose pliers to connect/disconnect the green ground wire and the 5 lens wires from the radiator. Do not bend the wires any more than necessary. (See Figure 65.)
- 2 Use tweezers or needle nose pliers to connect/disconnect the 2 purple source heater wires and the 2 gray RTD wires from the ceramic source board.

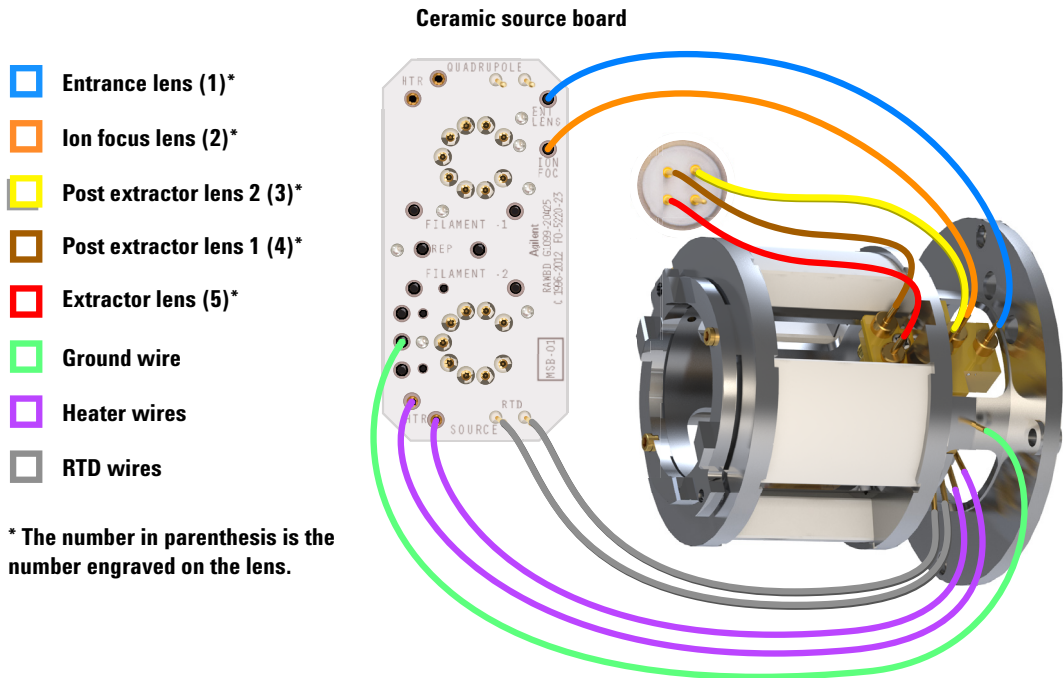


Figure 65 Wiring between the ceramic source board and the EI HES radiator

To Install the CI Source Radiator

Materials needed

- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- Tweezers (8710-2460)
- T10 Torx screwdriver (5182-3466)



Procedure

WARNING

The analyzers, GC/MS 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

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

CAUTION

When disconnecting leads, pull on the connectors, not on the wires.

- 1 Align the radiator over the two guide pins on the analyzer support, and secure it using the two retained screws with a number T10 Torx screwdriver.

- 2 Connect the green ground wire to the radiator. (See Figure 66.)

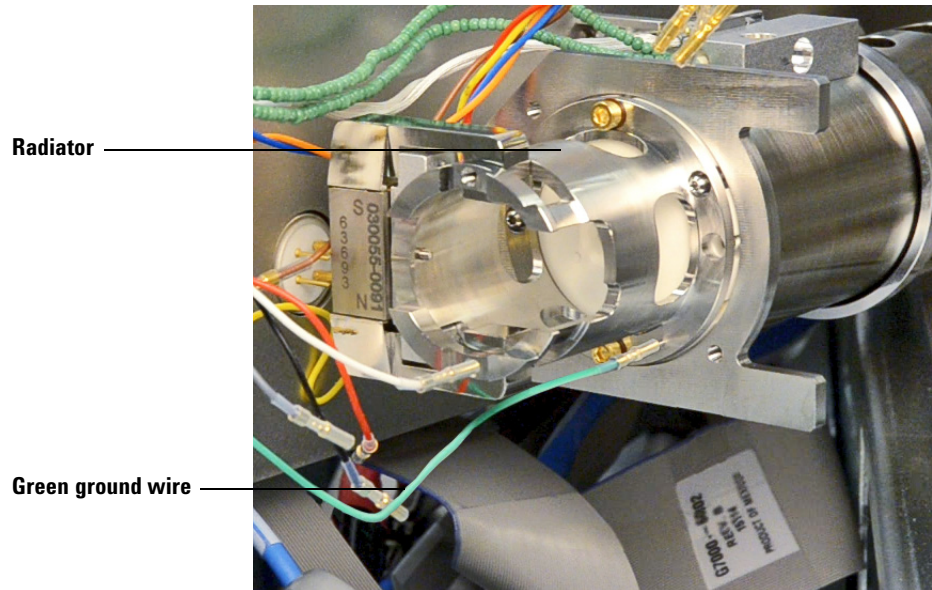


Figure 66 CI Source radiator

To Switch from the CI Source to the EI HES

Procedure

- 1 Vent the MS. (See [“Venting the MSD”](#) on page 91.) The software will prompt you for the appropriate actions.

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 antistatic wrist strap and take other antistatic precautions **before** you open the analyzer chamber. (See [“Electrostatic discharge is a threat to MSD electronics”](#) on page 21.)

- 2 Open the left side access panel. (See [“To Open the MSD Covers”](#) on page 112.)
- 3 Open the analyzer chamber door. (See [“To Open the Analyzer Chamber”](#) on page 152.)
- 4 Remove the CI source. (See [“To Remove the CI Source”](#) on page 211.)
- 5 Place the CI source in the storage container.
- 6 Remove the CI source radiator. The Radiator should not be removed or installed with the CI source in place. (See [“To Remove the CI Source Radiator”](#) on page 206.)
- 7 Place the CI source radiator in the storage container.
- 8 Loosen the column nut and remove the column from the GC/MS Interface.
- 9 Cut the column on the tapered side of the ferrule to remove the ferrule.
- 10 Install the column into the GC/MS interface extending 4 to 5 mm past the analyzer end of the transfer line. (See [“To Install a Capillary Column in the GC/MS Interface Using the Self-Tightening Column Nut”](#) on page 42 or [“To Install a Capillary Column in the GC/MS Interface Using a Standard Column Nut”](#) on page 47.)
- 11 Remove the EI HES radiator from the storage container.

- 12** Install the EI HES radiator. (See “To Install the EI HES Radiator” on page 208.)
- 13** Remove the EI HES from the storage container.
- 14** Install the EI HES. (See “To Install the EI HES” on page 171.)
- 15** Pumpdown the MSD. (See “To Pumpdown the MSD” on page 116.)

To Remove the CI Source Radiator

Materials needed

- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- Tweezers (8710-2460)
- T10 Torx screwdriver (5182-3466)



Procedure

WARNING

The analyzers, GC/MS 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

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

CAUTION

When disconnecting leads, pull on the connectors, not on the wires.

- 1 Remove the CI source. (See “To Remove the CI Source” on page 211.)
- 2 Disconnect the green ground wire from the radiator. (See Figure 67.)
- 3 Use a T-10 Torx screwdriver to loosen the two retained screws that secure the radiator to the analyzer and place the radiator in its storage container.

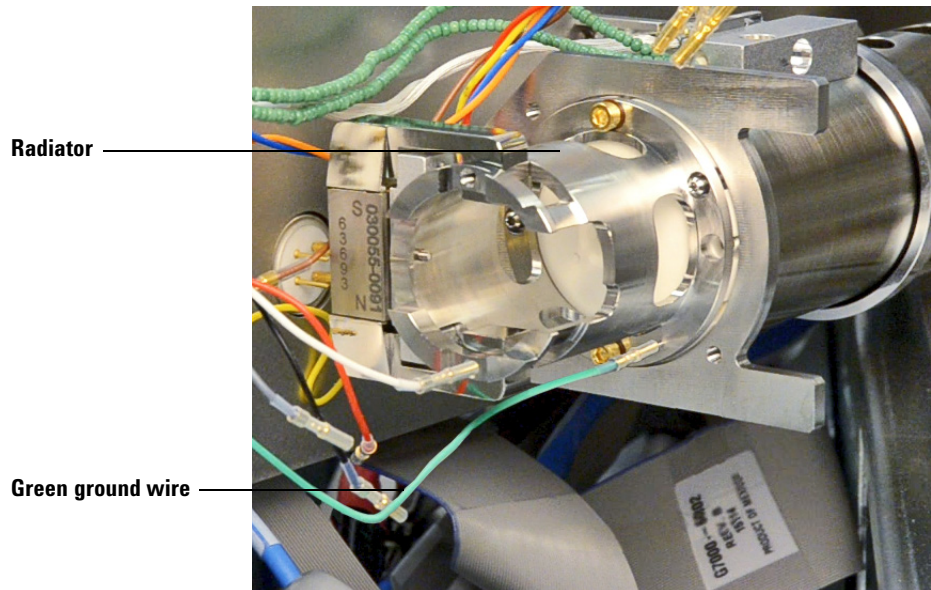


Figure 67 CI Source radiator

To Install the EI HES Radiator

Materials needed

- Gloves, clean, lint-free
 - Large (8650-0030)
 - Small (8650-0029)
- T10 Torx screwdriver (5182-3466)
- Tweezers (8710-2460)



Procedure

WARNING

The analyzers, GC/MS 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

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

- 1 Place the radiator over the guide pins on the analyzer support and use a T10 Torx screwdriver to secure it using the (2) M3 x 12 gold plated screws (part number G7002-20110).
- 2 Use tweezers to disconnect the wires from the EI HES radiator. (See “[To Connect/Disconnect Wiring to the EI HES Radiator](#)” on page 201.)

To Switch from an EI XTR, SS, or Inert Source to the CI Source

CAUTION

Always verify MSD performance in EI before switching to CI operation.

Always set up the CI MSD in PCI first, even if you are going to run NCI.

Procedure

- 1 Vent the MSD. (See “[To Vent the MSD](#)” on page 113.)
- 2 Open the analyzer. (See “[To Open the Analyzer Chamber](#)” on page 152.)
- 3 Remove the EI source. (See “[To Remove the EI XTR, SS, or Inert Source](#)” on page 172.)
- 4 If removing an XTR source, remove the brown extractor wire from the ceramic source board and store it with the EI XTR source. (See [Figure 55](#) on page 173.)

CAUTION

Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded antistatic wrist strap. (See “[Electrostatic discharge](#)” on page 148.) Take antistatic precautions **before** you open the analyzer chamber.

- 5 Install the CI source. (See “[To Install the CI Source](#)” on page 225.)
- 6 Install the CI/Extractor interface tip seal if it is not already installed (p/n G3870-20542). (See “[To Install the Interface Tip Seal](#)” on page 50.)
- 7 Close the analyzer. (See “[To Close the Analyzer Chamber](#)” on page 196.)
- 8 Pumpdown the MSD. (See “[To Pumpdown the MSD in CI Mode](#)” on page 124.)

To Switch from the CI Source to an EI XTR, SS, or Inert Source

Procedure

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 antistatic wrist strap, and take other antistatic precautions **before** you open the analyzer chamber. (See “[Electrostatic discharge](#)” on page 148.)

- 1 From the Tune and Vacuum Control view, vent the MSD. (See “[To Vent the MSD](#)” on page 113.) The software will prompt you for the appropriate actions.
- 2 Open the analyzer. (See “[To Open the Analyzer Chamber](#)” on page 152.)
- 3 Remove interface tip seal and spring if switching to an EI SS or EI Inert source.
- 4 Install the EI source. (See “[To Install the EI XTR, SS, or Inert Source](#)” on page 193.)
- 5 If installing an EI XTR source, locate the brown extractor wire from storage and connect it to the extractor lens and the source board.
- 6 Place the CI source in the ion source storage box.
- 7 If installing an EI SS or Inert source, remove the interface tip seal and spring and put it in the CI storage container.
- 8 Pumpdown the MSD. (See “[To Pumpdown the MSD](#)” on page 116.)

To Remove the CI Source

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Tweezers (8710-2460)



Procedure

- 1 Vent the MS. (See [“To Vent the MSD”](#) on page 113.)

WARNING

The analyzers, GC/MS 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

- 2 Open the analyzer chamber. (See [“To Open the Analyzer Chamber”](#) on page 152.)
- Make sure you use an antistatic wrist strap, and take other antistatic precautions before touching analyzer components.

CAUTION

When disconnecting leads, pull on the connectors, not on the wires.

- 3 Use tweezers to disconnect the wires from the CI source. Do not bend the wires any more than necessary. (See [“To Connect/Disconnect non-HES Model Wiring from the CI Source”](#) on page 212.)

Trace the wires for the ion source heater and temperature sensor to the ceramic source board, and disconnect them there. (See [“To Connect/Disconnect EI HES Model Wiring to the CI Source”](#) on page 213.)

- 4 Remove the two large thumbscrews that hold the ion source in place.
- 5 Pull the ion source out of the source radiator, and place it in its storage container.

To Connect/Disconnect non-HES Model Wiring from the CI Source

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Pliers, long-nose (8710-1094)
- Tweezers (8710-2460)



Procedure

- 1 Use pliers to connect/disconnect the ceramic board wire leads at the source connectors. (See Figure 68.)
- 2 Use pliers to connect/disconnect the source heater wire leads at the ceramic source board. (See Figure 68.)

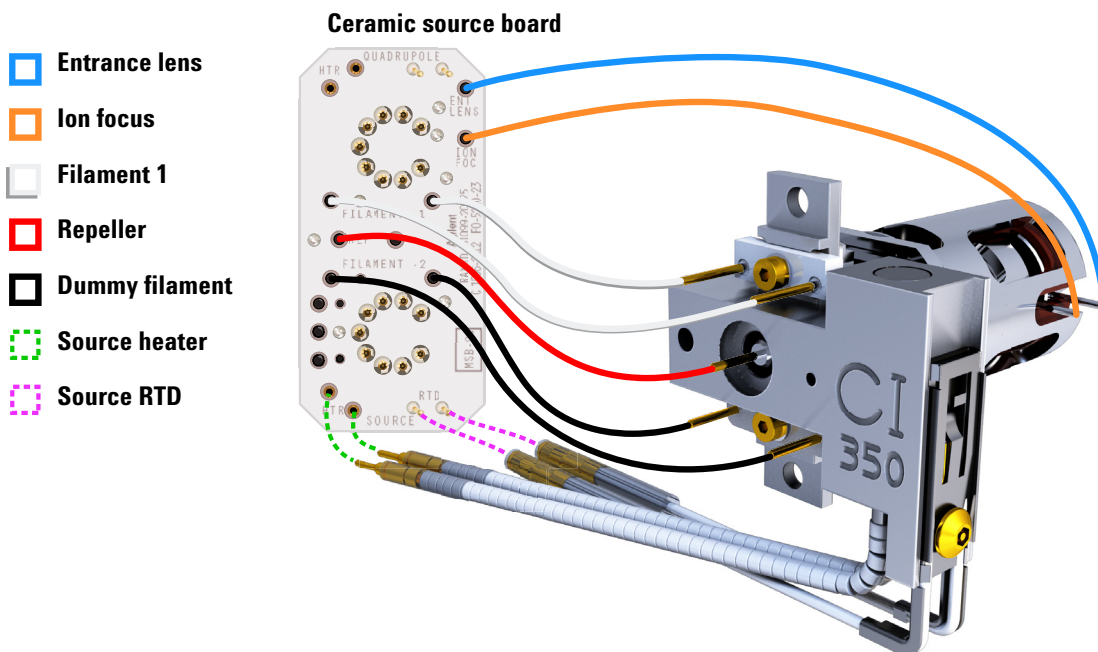


Figure 68 Wiring between the ceramic source board and the source

To Connect/Disconnect EI HES Model Wiring to the CI Source

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Pliers, long-nose (8710-1094)
- Tweezers (8710-2460)



Procedure

- 1 Use pliers to connect/disconnect the ceramic board wire leads (red, white, black, and gray) at the source connectors. (See Figure 69.)
- 2 Use pliers to connect/disconnect the source heater wire leads at the ceramic source board. (See Figure 69.)

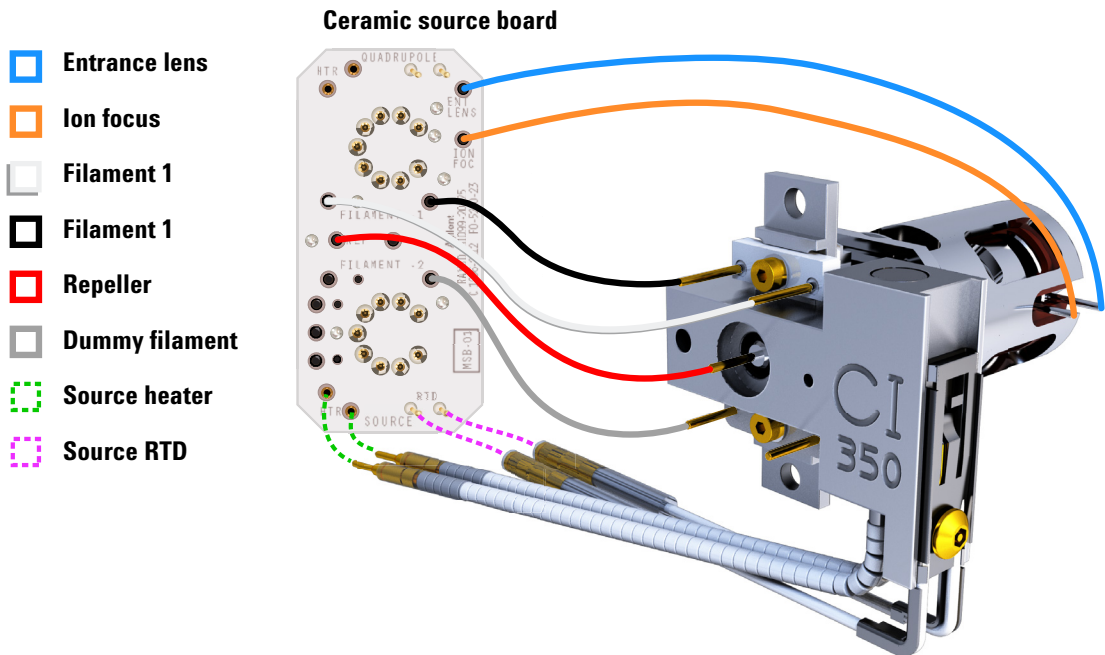


Figure 69 Wiring between the ceramic source board and the source

To Disassemble the CI 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)
- Wrench, open-end, 10 mm (8710-2353)
- Nut driver, 5.5 mm (8710-1220)
- Tweezers (8710-2460)

Procedure

Refer to the exploded parts view [Figure 70](#) and CI source parts list [Table 23](#) on page 215 while using this procedure.

- 1** Remove the CI source. (See [“To Remove the CI Source”](#) on page 211.)
- 2** Remove the filaments. (See [“To Remove the CI Source Filament”](#) on page 223.)
- 3** Separate the source heater assembly from the source body. The source heater assembly includes the source heater, repeller, and related parts.
- 4** Disassemble the repeller assembly by removing the ceramic insulator from the repeller.
- 5** Remove the setscrew securing the lenses to the source body.
- 6** Pull the lenses out of the source body and separate the lens insulator, ion focus lens, drawout cylinder, drawout lens, and entrance lens.

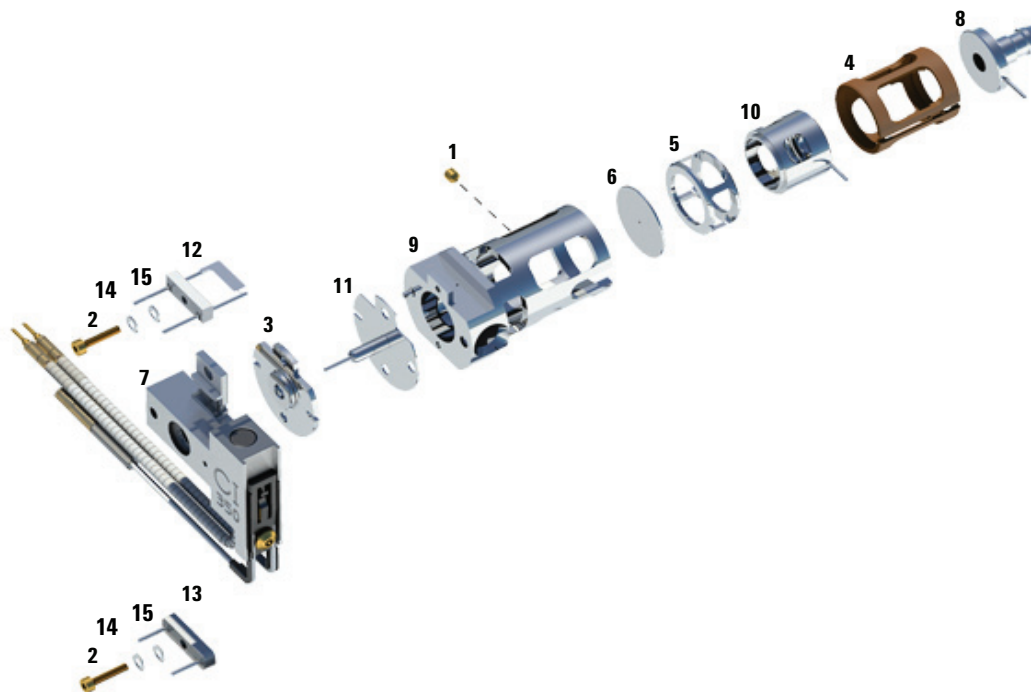


Figure 70 Disassembling the CI source

Table 23 Parts list for CI source (Figure 70)

| Item | Description | Part number |
|------|---------------------------------|-------------|
| 1 | Set screw | G1999-20022 |
| 2 | Filament screw | G1999-20021 |
| 3 | CI repeller insulator | G1999-20433 |
| 4 | CI lens insulator | G3170-20540 |
| 5 | CI drawout cylinder | G1999-20444 |
| 6 | CI drawout plate | G1999-20446 |
| 7 | CI source heater block assembly | G3870-60415 |
| 8 | Entrance lens | G7000-20026 |

Table 23 Parts list for CI source (Figure 70) (continued)

| Item | Description | Part number |
|-------------|--|--------------------|
| 9 | CI source body | G3170-20430 |
| 10 | Ion focus lens | G1999-20443 |
| 11 | CI repeller | G7077-20432 |
| 12 | CI filament- 2PK | G7005-60072 |
| 13 | Dummy filament | G1999-60454 |
| 14 | Washer spring curved 2.2 mm-ID 4.5 mm-OD, Qty. 2 | 3050-1374 |
| 15 | Flat washer | 3050-9082 |
| Not shown | Package, GC/MS source Clamshell | G7002-80008 |
| Not shown | Bracket, GC/MS source, Clamshell | G7002-00008 |
| Not shown | CI source assembly | G7002-67404 |
| Not shown | CI source assembly (without tip seal) | G7077-67404 |

To Clean the CI 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

Preparation

- 1 Disassemble the CI source. (See “To Disassemble the CI Source” on page 214.)
- 2 Collect the following parts to be cleaned for a CI source: (See [Figure 71](#) on page 218.)
 - Repeller
 - 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 the CI repeller insulator is dirty, clean it with a cotton swab dampened with reagent-grade methanol. If that does not clean the insulator, replace it. Do not abrasively or ultrasonically clean the insulator.



Figure 71 CI source parts to be cleaned

**Procedure**

- 1 If the contamination is serious, such as an oil backflow into the analyzer, seriously consider replacing the contaminated parts.
- 2 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.
- 3 Rinse away all abrasive residue with reagent-grade methanol.
Ensure **all** abrasive residue is rinsed **before** ultrasonic cleaning. If the methanol becomes cloudy or contains visible particles, rinse again three times.
- 4 Separate the parts that were abrasively cleaned from the parts that were not abrasively cleaned.

5 Ultrasonically clean the parts (each group separately) for 15 minutes. For dirty parts, use all three solvents in the order shown, cleaning 15 minutes with each of the following solvents:

- Methylene chloride (reagent-grade)
- Acetone (reagent-grade)
- Methanol (reagent-grade)

For routine cleaning, cleaning with methanol is sufficient.

WARNING

All of these solvents are hazardous. Work in a fume hood and take all appropriate precautions.

6 Place the parts in a clean beaker. *Loosely* cover the beaker with clean aluminum foil (dull side down).

7 Dry the cleaned parts in an oven at 100 °C for five to six minutes.

WARNING

Let the parts cool before you handle them.

NOTE

Take care to avoid contaminating 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 Assemble the CI 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)
- Wrench, open-end, 10 mm (8710-2353)



Procedure

Refer to the exploded parts view [Figure 72](#) and the CI source parts list [Table 24](#) on page 221 while using this procedure.

CAUTION

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

- 1 Assemble the ion focus lens, entrance lens, and lens insulator.
- 2 Slide the drawout plate and the drawout cylinder into the source body.
- 3 Slide the parts assembled in step 1 into the source body.
- 4 Install the setscrew that holds the lenses in place.
- 5 Attach the ceramic disk to the repeller and place on top of the source body.

CAUTION

While installing, do not overtighten the repeller nut or the ceramic repeller insulator will break when the source heats up. The nut should only be finger-tight.

- 6 Place the heater block assembly on top of the source body.
- 7 Reinstall the dummy filament, and the filament and attach with the setscrews.

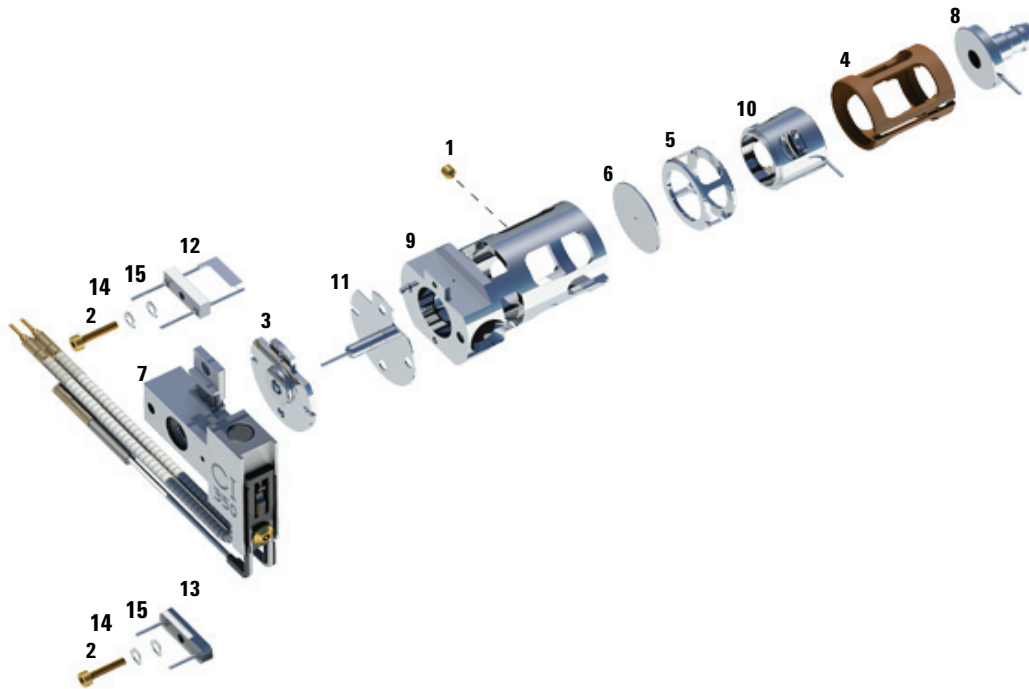


Figure 72 Assembling the CI source

Table 24 Parts list for CI source (Figure 72)

| Item | Description | Part number |
|------|---------------------------------|-------------|
| 1 | Set screw | G1999-20022 |
| 2 | Filament screw | G1999-20021 |
| 3 | CI repeller insulator | G1999-20433 |
| 4 | CI lens insulator | G3170-20540 |
| 5 | CI drawout cylinder | G1999-20444 |
| 6 | CI drawout plate | G1999-20446 |
| 7 | CI source heater block assembly | G3870-60415 |
| 8 | Entrance lens | G7000-20026 |

Table 24 Parts list for CI source (Figure 72) (continued)

| Item | Description | Part number |
|-------------|--|--------------------|
| 9 | CI source body | G3170-20430 |
| 10 | Ion focus lens | G1999-20443 |
| 11 | CI repeller | G7077-20432 |
| 12 | CI filament- 2PK | G7005-60072 |
| 13 | Dummy filament | G1999-60454 |
| 14 | Washer spring curved 2.2 mm-ID 4.5 mm-OD, Qty. 2 | 3050-1374 |
| 15 | Flat washer | 3050-9082 |
| Not shown | Package, GC/MS source Clamshell | G7002-80008 |
| Not shown | Bracket, GC/MS source, Clamshell | G7002-00008 |
| Not shown | CI source assembly | G7002-67404 |
| Not shown | CI source assembly (without tip seal) | G7077-67404 |

To Remove the CI Source Filament

Materials needed

- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Hex ball driver, 1.5 mm (8710-1570)
- Tweezers (8710-2460)



Procedure

- 1 Vent the MS. (See “To Vent the MSD” on page 113.)

CAUTION

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

WARNING

The analyzer, GC/MS 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.

- 2 Open the analyzer chamber. (See “To Open the Analyzer Chamber” on page 152.)
- 3 Remove the CI source. (See “To Remove the CI Source” on page 211.)
- 4 Remove the screw holding the filament to the CI source body. (See Figure 73.)
- 5 Slide the filament off the CI source assembly. (See Figure 73.)

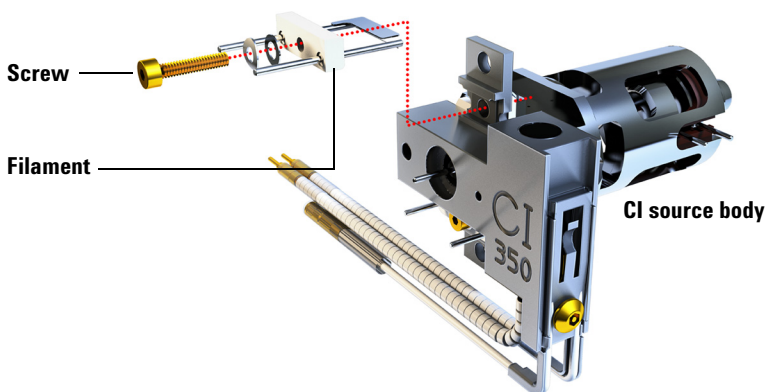


Figure 73 Changing the CI source filament

To Install a CI Source Filament

Materials needed

- Filament assembly, 2-pk, CI (G7005-60072)
- Gloves, clean, lint-free (Large 8650-0030) (Small 8650-0029)
- Tweezers (8710-2460)



Procedure

- 1 Remove the old filament. (See “To Remove the CI Source Filament” on page 223.)
- 2 Place the new filament into its position on the ion source body. (See [Figure 73](#) on page 223.)
- 3 Secure the filament to the ion source body with the screw. (See [Figure 73](#) on page 223.)
- 4 After installing the filament, verify that it is not grounded to source body.
- 5 Reinstall the CI source. (See “To Install the CI Source” on page 225 or “General Information” on page 198.)
- 6 Pumpdown the MSD. (See “To Pumpdown the MSD” on page 116.)
- 7 Autotune the MSD.

To Install the CI Source

CAUTION

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



Procedure

- 1 Vent the MS. (See “To Vent the MSD” on page 113.)
- 2 Open the analyzer chamber. (See “To Open the Analyzer Chamber” on page 152.)
- 3 Slide the CI source into the radiator. (See Figure 74.)
- 4 Install the thumbscrews. (See Figure 74.)
- 5 Connect the wiring to the CI source. (See “To Connect/Disconnect non-HES Model Wiring from the CI Source” on page 212.)

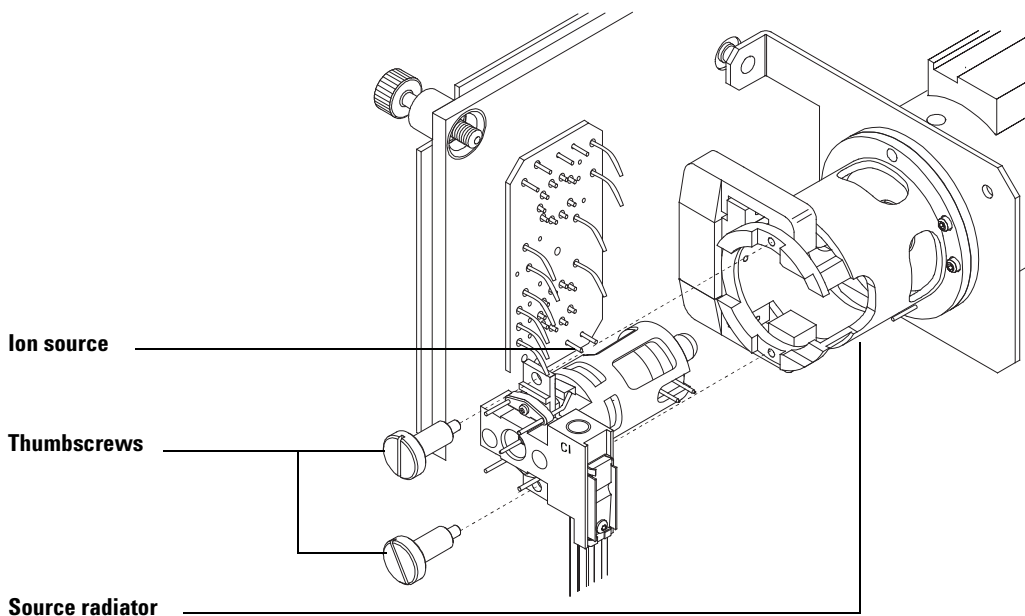


Figure 74 Installing the CI source

7 CI Maintenance

- 6 Close the analyzer door. (See “To Close the Analyzer Chamber” on page 196.)
- 7 Pumpdown the MSD. (See “To Pumpdown the MSD in CI Mode” on page 124.)
- 8 Tune the MSD. (See “CI Autotune” on page 121.)



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Printed in USA, June 2016



G7077-90026