Notices

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Safety Notices

CAUTION

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

WARNING

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

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

1  “Introduction”

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

2  “Installing 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  “Operating in Electron Ionization (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.

4  “Operating in Chemical Ionization (CI) Mode”

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

5  “General Maintenance”

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

6  “CI Maintenance”

Chapter 6 describes maintenance procedures unique to CI MSDs.
Online User Information

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

The software DVD that ships with your instrument provides an extensive collection of online help, videos, and books for the Agilent 7890 Series GC, 7820 GC, 5977 Series MSD, and the 7693A. Included are localized versions of the information you need most, such as:

- Getting Familiar documentation
- Safety and Regulatory guides
- Installation information
- Operating guides
- Maintenance information
- Troubleshooting details
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1 Introduction

This chapter describes general information about the 5977 Series MSDs, including a hardware description, general safety warnings, and hydrogen safety information.
5977 Series MSD Version

5977 Series MSDs are equipped with a high performance turbomolecular (turbo) pump or a diffusion pump, and a choice of three foreline pumps. In addition, there are two types of analyzers (stainless steel or inert) and four types of ion sources. The serial number label displays a product number (Table 1) that indicates what type of MSD you have.

Table 1   Available high vacuum pumps

<table>
<thead>
<tr>
<th>Model name</th>
<th>Product number</th>
<th>Description</th>
<th>Ionization mode/Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>5977E MSD Diff Pump for 7820 GC</td>
<td>G7035A</td>
<td>Diffusion pump</td>
<td>Electron ionization (EI)/Stainless Steel</td>
</tr>
<tr>
<td>5977E MSD Turbo Pump for 7820 GC</td>
<td>G7036A</td>
<td>Performance Turbo pump</td>
<td>Electron ionization (EI)/Stainless Steel</td>
</tr>
<tr>
<td>5977A VL inert MSD</td>
<td>G7037A</td>
<td>Diffusion pump MSD</td>
<td>Electron ionization (EI)/Inert</td>
</tr>
<tr>
<td>5977A inert MSD El Turbo for 7890 Series GC</td>
<td>G7038A</td>
<td>Performance turbo pump MSD</td>
<td>Electron ionization (EI)/Inert</td>
</tr>
<tr>
<td>5977A extractor MSD El Perf Turbo for 7890 Series GC</td>
<td>G7039A</td>
<td>Performance turbo pump MSD</td>
<td>Electron ionization (EI)/Extractor</td>
</tr>
<tr>
<td>5977A EI/CI MSD for 7890 Series GC</td>
<td>G7040A</td>
<td>Performance turbo pump MSD</td>
<td>Electron ionization (EI)/Extractor Chemical ionization /PCI, NCI</td>
</tr>
</tbody>
</table>
Abbreviations Used

The abbreviations in Table 2 are used in discussing this product. They are collected here for convenience.

Table 2  Abbreviations

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC</td>
<td>Alternating current</td>
</tr>
<tr>
<td>ALS</td>
<td>Automatic liquid sampler</td>
</tr>
<tr>
<td>BFB</td>
<td>Bromofluorobenzene (calibrant)</td>
</tr>
<tr>
<td>CI</td>
<td>Chemical ionization</td>
</tr>
<tr>
<td>DC</td>
<td>Direct current</td>
</tr>
<tr>
<td>DFTPP</td>
<td>Decafluorotriphenylphosphine (calibrant)</td>
</tr>
<tr>
<td>DIP</td>
<td>Direct insertion probe</td>
</tr>
<tr>
<td>DP</td>
<td>Diffusion pump</td>
</tr>
<tr>
<td>EI</td>
<td>Electron ionization</td>
</tr>
<tr>
<td>EM</td>
<td>Electron multiplier (detector)</td>
</tr>
<tr>
<td>EMV</td>
<td>Electron multiplier voltage</td>
</tr>
<tr>
<td>EPC</td>
<td>Electronic pneumatic control</td>
</tr>
<tr>
<td>eV</td>
<td>Electron volt</td>
</tr>
<tr>
<td>GC</td>
<td>Gas chromatograph</td>
</tr>
<tr>
<td>HED</td>
<td>High-energy dynode (refers to detector and its power supply)</td>
</tr>
<tr>
<td>id</td>
<td>Inside diameter</td>
</tr>
<tr>
<td>LAN</td>
<td>Local Area Network</td>
</tr>
<tr>
<td>LCP</td>
<td>Local control panel (on the MSD)</td>
</tr>
<tr>
<td>m/z</td>
<td>Mass to charge ratio</td>
</tr>
<tr>
<td>MFC</td>
<td>Mass flow controller</td>
</tr>
<tr>
<td>MSD</td>
<td>Mass selective detector</td>
</tr>
</tbody>
</table>
Table 2  Abbreviations (continued)

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>NCI</td>
<td>Negative CI</td>
</tr>
<tr>
<td>OFN</td>
<td>Octafluoronaphthalene (calibrant)</td>
</tr>
<tr>
<td>PCI</td>
<td>Positive CI</td>
</tr>
<tr>
<td>PFDTD</td>
<td>Perfluoro-5,8-dimethyl-3,6,9-trioxydodecane (calibrant)</td>
</tr>
<tr>
<td>PFHT</td>
<td>2,4,6-tris(perfluoroheptyl)-1,3,5-triazine (calibrant)</td>
</tr>
<tr>
<td>PFTBA</td>
<td>Perfluorotributylamine (calibrant)</td>
</tr>
<tr>
<td>Quad</td>
<td>Quadrupole mass filter</td>
</tr>
<tr>
<td>RF</td>
<td>Radio frequency</td>
</tr>
<tr>
<td>RFPA</td>
<td>Radio frequency power amplifier</td>
</tr>
<tr>
<td>Torr</td>
<td>Unit of pressure, 1 mm Hg</td>
</tr>
<tr>
<td>Turbo</td>
<td>Turbomolecular (pump)</td>
</tr>
</tbody>
</table>
The 5977 Series MSD

The 5977 Series MSD is a stand-alone capillary GC detector for use with an Agilent Series Gas Chromatograph (Table 3 on page 14). The MSD features:

- Local control panel (LCP) for locally monitoring and operating the MSD
- One of two different high vacuum pumps
- One of three different foreline pumps
- Three different types of independently MSD heated electron-ionization (EI) sources available: standard (stainless), inert, and extraction
- Optional chemical ionization (PCI/NCI) modes available that add a chemical-ionization (CI) source, reagent gas flow controller and plumbing, and CI tuning calibration
- Independently MSD heated hyperbolic quadrupole mass filter
- High-energy dynode (HED) electron multiplier detector
- Independently GC heated GC/MSD interface

Physical description

The 5977 Series MSD housing is approximately 41 cm high, 30 cm wide, and 54 cm deep. The weight is 39 kg for the diffusion pump models, 44 kg for the standard EI performance turbo pump mainframe, and 46 kg for the EI/CI performance 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: the frame/cover assemblies, the local control panel, the vacuum system, the GC interface, the electronics, and the analyzer.

Local control panel

The local control panel allows local monitoring and operation of the MSD. You can tune the MSD, run a method or a sequence, and monitor instrument status.
1 Introduction

Vacuum gauge

The 5977 Series MSD may be equipped with an ion vacuum gauge. The MassHunter Data 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 chemical ionization (CI) operation.

### Table 3 5977 series MSD models and features

<table>
<thead>
<tr>
<th>Feature</th>
<th>G7035A</th>
<th>G7036A</th>
<th>G7037A</th>
<th>G7038A</th>
<th>G7039A</th>
<th>G7040A</th>
</tr>
</thead>
<tbody>
<tr>
<td>High vacuum pump</td>
<td>Diffusion</td>
<td>Performance turbo</td>
<td>Diffusion</td>
<td>Performance turbo</td>
<td>Performance turbo</td>
<td>Performance turbo</td>
</tr>
<tr>
<td>Optimal He column flow mL/min</td>
<td>1</td>
<td>1 to 2</td>
<td>1</td>
<td>1 to 2</td>
<td>1 to 2</td>
<td>1 to 2</td>
</tr>
<tr>
<td>Maximum recommended gas flow mL/min</td>
<td>1.5</td>
<td>4</td>
<td>1.5</td>
<td>4</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Maximum gas flow, mL/min†</td>
<td>2</td>
<td>6.5</td>
<td>2</td>
<td>6.5</td>
<td>6.5</td>
<td>6.5</td>
</tr>
<tr>
<td>Max column id</td>
<td>0.25 mm (30 m)</td>
<td>0.53 mm (30 m)</td>
<td>0.25 mm (30 m)</td>
<td>0.53 mm (30 m)</td>
<td>0.53 mm (30 m)</td>
<td>0.53 mm (30 m)</td>
</tr>
<tr>
<td>CI capability</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>Inert material</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>GC compatibility</td>
<td>7820</td>
<td>7820</td>
<td>7890 Series</td>
<td>7890 Series</td>
<td>7890 Series</td>
<td>7890 Series</td>
</tr>
<tr>
<td>Separate interface tip seal</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>Foreline pumps available</td>
<td>DS42 MVP55</td>
<td>DS42, MVP55</td>
<td>DS42, MVP55</td>
<td>DS42, DS42i, MVP55, IDP3-C</td>
<td>DS42i, MVP55, IDP3-C</td>
<td>DS42i, MVP55, IDP3-C</td>
</tr>
<tr>
<td>DIP‡ capability (3rd party)</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
</tr>
</tbody>
</table>

* Total gas flow into the MSD: column flow plus reagent gas flow (if applicable).
† Expect degradation of spectral performance and sensitivity.
‡ Direct insertion probe.
MSD Hardware Description

Figure 1  5977 Series GC/MSD system, shown with Agilent 7890B GC
The CI hardware allows the 5977 Series MSD to produce high-quality, classical CI spectra, which include molecular adduct ions. A variety of reagent gases can be used.

In this manual, the term “CI MSD” refers to the G7040A MSD and upgraded G7038A and G7039A MSDs. It also applies, unless otherwise specified, to the flow modules for these instruments.

The 5977 Series CI system adds to the 5977 Series MSD:
- EI/CI GC/MSD interface
- CI ion source with an interface tip seal that can also be used with and extractor EI ion source
- Reagent gas flow control module
- 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 MSD and is recommended for EI also.

The MSD CI system has been optimized to achieve the relatively high CI ion 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 ion sources takes less than an hour, although 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, and the wiring from the AC power supply to the power switch.

With the power switch on, potentially dangerous voltages also exist on:
- All electronics boards in the instrument
- The internal wires and cables connected to these boards
- The wires for any heater (oven, detector, inlet, or valve box)

**WARNING**

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

**WARNING**

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

If one of the primary fuses has failed, the MSD will already be off, but for safety you should switch off the MSD and unplug the power cord. It is not necessary to allow air into the analyzer chamber.

**WARNING**

Never replace the primary fuses while the MSD is connected to a power source.

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 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 standard foreline pump can be a fire hazard**

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

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

**WARNING**

Using hydrogen as a GC carrier gas 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, be sure that the supply is turned off until all connections are made and ensure that the inlet and detector column fittings are either connected to a column or capped at all times when hydrogen 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 non luminous 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.
Introduction

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 and/or detector gas streams. For this reason, it is vital that column fittings should always be either connected to a column or have a cap or plug installed.

All users should be aware of the mechanisms by which hydrogen can accumulate (Table 4 on page 21) and know what precautions to take if they know or suspect that hydrogen has accumulated. Note that these mechanisms apply to all mass spectrometers, including the MSD.
Table 4  Hydrogen accumulation mechanisms

<table>
<thead>
<tr>
<th>Mechanism</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass spectrometer turned off</td>
<td>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.</td>
</tr>
<tr>
<td>Mass spectrometer automated shutoff valves closed</td>
<td>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.</td>
</tr>
<tr>
<td>Mass spectrometer manual shutoff valves closed</td>
<td>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.</td>
</tr>
<tr>
<td>GC off</td>
<td>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.</td>
</tr>
<tr>
<td>Power failure</td>
<td>If the power fails, both the GC and mass spectrometer shut down. The carrier gas, however, is not necessarily shut down. As described previously, 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.</td>
</tr>
</tbody>
</table>
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.

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

**Precautions**

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

**Equipment precaution**

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

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

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

**General laboratory precautions**

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

- Turn off the hydrogen at its source every time you shut down the 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. Always make sure the vent line is vented into a fume hood.
Safety and Regulatory Certifications

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

Information

The Agilent Technologies 5977 Series MSD meets the following IEC (International Electro-technical Commission) classifications: Equipment Class I, Laboratory Equipment, Installation Category II, Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and is designed for use 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.
1 Introduction

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

Sound emission declaration

Sound pressure

Sound pressure $L_p < 70$ dB according to EN 27779:1991.
Cleaning/Recycling the Product

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

Liquid Spillage

Do not spill liquids on the MSD.

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, a few additional precautions are required. The MSD must remain upright at all times; this requires special caution when moving. The MSD should not be left vented to atmosphere for long periods.
To 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 failure of the primary fuses 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 you should switch off the MSD and unplug the power cord. It is not necessary to allow air into the analyzer chamber.

2. Turn one of the fuse holders (Figure 2 on page 29) counterclockwise until it pops out. The fuse holders are spring loaded.
3. Remove the old fuse from the fuse holder.
4. Install a new fuse in the fuse holder.
5. Reinstall the fuse holder.

**WARNING** Never replace the primary fuses while the MSD is connected to a power source.

**WARNING** If you are using hydrogen as a GC carrier gas, a power failure may allow it to accumulate in the analyzer chamber. In that case, further precautions are required. See “Hydrogen Safety” on page 19.
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 Pump down the MSD.
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. The serial number tag on the lower front of the left side panel shows the model number.


2 Installing GC Columns

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 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 66 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>
<thead>
<tr>
<th>Table 5</th>
<th>Gas flows</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Feature</strong></td>
<td><strong>Model</strong></td>
</tr>
<tr>
<td>High vacuum pump</td>
<td>Diffusion</td>
</tr>
<tr>
<td>Optimal He column flow mL/min</td>
<td>1</td>
</tr>
<tr>
<td>Maximum recommended gas flow mL/min*</td>
<td>1.5</td>
</tr>
<tr>
<td>Maximum gas flow, mL/min†</td>
<td>2</td>
</tr>
<tr>
<td>Max column id</td>
<td>0.25 mm (30 m)</td>
</tr>
<tr>
<td>CI capability</td>
<td>No</td>
</tr>
<tr>
<td>GC compatibility</td>
<td>7820</td>
</tr>
</tbody>
</table>

* Total gas flow into the MSD: column flow plus reagent gas flow (if applicable).
† 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 37.

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

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

Conditioning ferrules

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

Tips and hints

- The column installation procedures for the 5977 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.
- You can remove old ferrules from column nuts with an ordinary push pin.
- Always use carrier gas that is at least 99.9995% pure.
- Because of thermal expansion, new ferrules may loosen after heating and cooling a few times. Check for tightness after two or three heating cycles.
- Always wear clean gloves when handling columns, especially the end that will be inserted into the GC/MSD interface.

**WARNING** If you are using hydrogen as a carrier gas, do not start carrier gas flow until the column is installed in the MSD and the MSD has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the MSD and an explosion may occur. See “Hydrogen Safety” on page 19.

**WARNING** Always wear safety glasses when handling capillary columns. Use care to 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 Gas Chromatograph User Information.

**WARNING** The GC operates at high temperatures. In order to avoid burns, do not touch any parts of the GC until you are sure they are cool.

**WARNING** Always wear safety glasses when handling capillary columns. Use care to avoid puncturing your skin with the end of the column.
Always wear clean gloves while handling any parts that go inside the GC or analyzer chambers.

**Procedure**

1. Cool the oven to room temperature.
2. Wearing clean gloves, slide a septum, column nut, and conditioned ferrule onto the free end of the column (Figure 3). The tapered end of the ferrule should point away from the column nut.

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

---

**Figure 3** Preparing a capillary column for installation
7 Position the column so it extends 4 to 6 mm past the end of the ferrule (Figure 4).

8 Slide the septum to place the nut and ferrule in the correct position.
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. To avoid burns, 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 34.)
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. 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” on page 38.)
To Install a Capillary Column in the GC/MS Interface

This procedure is for the installation of a capillary column directly to the transfer line with a column nut. If you are using the Agilent capillary flow technology Quick Swap accessory, or a Purged Ultimate Union (PUU) see “To Prepare the Column Ends for a CFT Fitting” on page 41.

Agilent 7890 Series GC

Materials needed

- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- 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)
- Flashlight
- Magnifying loupe
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Interface column nut (05988-20066)
- Safety glasses
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)
- Column measuring tool

CAUTION

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

1 Condition the column. (See “To Condition a Capillary Column” on page 37.)

**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.

**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 If you are not using Quick Swap, vent the MS. To vent the MS, see “To Vent the MSD” on page 76.

**WARNING**
The GC operates under high temperatures. To avoid burns, do not touch any GC parts until you are certain they are cool.

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

4 Use the column cutter to score the column 2 cm from its end.

5 While holding the column against the column cutter with your thumb, break the column against the edge of the column cutter.

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

7 Wipe the end with alcohol.

8 Insert the column into the column measuring tool.

9 Slide the column so that the end projects 1–2 mm past the end of the tool.

10 Finger tighten the fitting.

11 Tighten 1/4 to 1/2 turn to fix the ferrule to the column.

12 Slide the column into the GC/MS interface.
2 Installing GC Columns

13 Hand-tighten the nut. Ensure the position of the column does not change as you tighten the nut.

14 Check the GC oven to be sure that the column does not touch the oven walls.

15 Tighten the nut 1/4 to 1/2 turn.

16 Check the nut’s tightness after one or two heat cycles; retighten as appropriate.
To Prepare the Column Ends for a CFT Fitting

Materials needed

- SilTite ferrules:
  - 0.1- to 0.25-mm columns, pkg of 10 (5188-5361)
  - 0.32-mm columns, pkg of 10 (5188-5362)
  - 0.53-mm columns, pkg of 10 (5188-5363)
- Swaging nut
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)

Procedure

1. Obtain the appropriate metal ferrule.
2. Pass the tubing end through the internal nut and SilTite ferrule leaving approximately 1 cm of fused silica tubing protruding beyond the ferrule. Thread the ferrule pre-swaging tool onto the internal nut with the tube protruding (see Figure 5).

![Figure 5](image)

Figure 5  Assemble the internal nut, ferrule, and swaging nut

CAUTION

The SilTite ferrules are delicate. Follow the instructions in the next step very carefully to avoid overtightening.
3 Using the wrench and ferrule pre-swaging tool, tighten the nut a little at a time, occasionally checking to see if the ferrule is gripping the tube (see Figure 6). When the ferrule just starts to grip, notice position of the nut and then tighten by turning 45 to 60 degrees of rotation, but no more than 60 degrees (one flat). If you can pull the column free, it is not tight enough.

Figure 6  Tightening the internal nut

4 Remove the swaging tool (Figure 7).

Figure 7  Removed ferrule pre-swaging tool
5 Using a ceramic column cutter, trim the tubing at the small end of the ferrule leaving approximately 0.3 mm of tubing extending beyond the ferrule (Figure 8 on page 43).

It is important that the ceramic column cutter have one side dedicated to only make contact with the column and the other side dedicated to riding on the edge of the metallic SilTite ferrule. This preserves the ceramic edge sharpness which is necessary to making good column cuts.

**NOTE**

It is important that the tube end does not extend beyond 0.5 mm from the end of the ferrule.

---

**Figure 8** Ceramic column cutter and ferrule
2 Installing GC Columns

6 Check the end of the tube with a magnifier. The end of the tube need not be perfectly square, but should not have cracks which extend under the ferrule. Figure 9 shows a completed tube end.

![Figure 9](image)

**Figure 9** Tube end with internal nut and swaged SilTite ferrule

7 Connect column to the Quick Swap or PUU with internal nuts and preswaged SilTite ferrules (Figure 10). Finger-tighten the nuts. Further tighten with a wrench only 15 to 20 degrees.

![Figure 10](image)

**Figure 10** Completed Ultimate union with columns
3 Operating in Electron Ionization (EI) Mode

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This chapter describes how to perform some basic operating procedures for the Agilent 5977 Series GC/MSD using electron ionization.
Operating the MSD from the Data System

The Agilent MassHunter Data Acquisition Workstation automates tasks such as pumping down, removing the ion source, 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 MassHunter Workstation software.

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

Operating the MSD from the LCP

The local control panel (LCP) shows the status of the MSD or initiates a task on the MSD without using the Agilent MassHunter Data Acquisition software. The Agilent MassHunter Data Acquisition software may be located anywhere on the site local area network (LAN), so the Data Acquisition software might not be near the instrument itself. Because the LCP communicates with the Data Acquisition software through the LAN, you can access Data Acquisition software functions, such as tuning and starting a run, right from the MSD. Only certain features are available from the LCP. The Data Acquisition software is the full-featured controller for most instrument control operations.

Modes of operation

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

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

*Menu* mode allows you to query various aspects of the GC/MSD and to initiate some actions like running a method or sequence, or preparing to vent the system.
To access a particular menu option:

- Press [Menu] until the desired menu appears.
- Press [Item] until the desired menu item appears.

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

- Use [Up] to increase the displayed value or to scroll up (such as in a message list).
- Use [Down] to decrease the displayed value or to scroll down (such as in a message list).
- Use [Yes/Select] to accept the current value.
- Use [No/Cancel] to return to the Status mode.

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

Pressing [Menu], then [No/Cancel], will always display the Status mode.
Pressing [No/Cancel] twice will always return to the Status mode.
LCP status messages

The following messages may be displayed on the LCP to inform you of the status of the MSD system. If the LCP is currently in Menu mode, cycle through the menus to return to Status mode. No messages will be displayed if an online instrument session is not currently running in MassHunter Data Acquisition.

**ChemStation Loading <timestamp>**
The Agilent MassHunter Data Acquisition software is starting up.

**Executing <type>tune**
A tuning procedure is in progress (type = QuickTune or Autotune).

**Instrument Available <timestamp>**
The Agilent MassHunter Data Acquisition software is not running.

**Loading Method <method name>**
Method parameters are being sent to the MSD.

**Loading MSD Firmware**
The MSD’s firmware is being initialized.

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

- **Server not Found**
- **Check LAN Connection**
- **Seeking Server**
- **Bootp Query xxx**

These messages indicate that the MSD has not received its unique IP address from the Windows Service. If the messages persist after you have logged onto your account in the MassHunter Data Acquisition program, consult the Troubleshooting section of the Software Installation manual.

**Loading OS**
The operating system of the instrument controller is being initialized.
<method> Complete <timestamp>

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

**Method Loaded <method name>**

Method parameters were sent to the MSD.

**MS Locked by <computer name>**

MS parameters can only be changed from the MassHunter Data Acquisition.

**Press Sideplate**

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

**Run: <method> Acquiring <datafile>**

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

**To view system status during startup**

1. The following messages are displayed on the LCP display during startup:
   - Press Sideplate
   - Loading OS
   - Press Sideplate
   - Loading MSD Firmware

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

To access a particular menu option, press [Menu] until the desired menu appears, then press [Item] until the desired menu item appears. Table 6 through Table 11 on page 52 list the menus and selections.

Table 6  ChemStation menu

<table>
<thead>
<tr>
<th>Action</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Run Method</td>
<td>Displays the current method name and starts an analysis.</td>
</tr>
<tr>
<td>Run Sequence</td>
<td>Displays the current sequence and starts a sequence.</td>
</tr>
<tr>
<td>Run Current Tune</td>
<td>Displays the current tune file and starts an autotune (EI mode only; CI tune must be started from the MassHunter Data Acquisition).</td>
</tr>
<tr>
<td># of Messages</td>
<td>Displays the number of messages and the text of the most recent message. Use the arrow keys to scroll through previous messages (up to 20).</td>
</tr>
<tr>
<td>Release ChemStation</td>
<td>Disassociates the MassHunter Data Acquisition from the MSD.</td>
</tr>
<tr>
<td>Connection Status</td>
<td>Displays the LAN connection status for the MSD.</td>
</tr>
<tr>
<td></td>
<td>Remote = connected to MassHunter Data Acquisition online session</td>
</tr>
<tr>
<td></td>
<td>Local = not connected to MassHunter Data Acquisition online session</td>
</tr>
<tr>
<td>Name of Instrument</td>
<td>Displays the name of the instrument if connected to MassHunter Data Acquisition online session. The name of the instrument is the name assigned to the MSD by the MassHunter Data Acquisition Configuration dialogue.</td>
</tr>
</tbody>
</table>
Table 7  Maintenance menu

<table>
<thead>
<tr>
<th>Action</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prepare to vent</td>
<td>Reminds you to shut down the GC then prepares the instrument for venting when [Yes/Select] is pressed.</td>
</tr>
<tr>
<td>Pumpdown</td>
<td>Initiates a pumpdown sequence.</td>
</tr>
<tr>
<td>Hi Vac Soft Start</td>
<td>Allows you to connect or disconnect the hi vac soft start feature. This feature slowly ramps up the turbo pump speed, and should be used after the pump has sat idle for a few weeks to minimize wear on the pump.</td>
</tr>
</tbody>
</table>

Table 8  MS Parameters menu

<table>
<thead>
<tr>
<th>Action</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>High Vacuum Pressure</td>
<td>Only with Micro-Ion vacuum gauge installed.</td>
</tr>
<tr>
<td>Turbo Pump Speed</td>
<td>Displays the turbo pump speed.</td>
</tr>
<tr>
<td>MSD Fault Status</td>
<td>Reports a summary fault status code (number) in 'dec’ (decimal) and 'hex’ (hexadecimal) format covering all possible fault combinations.</td>
</tr>
<tr>
<td>Ion Source Temp, °C</td>
<td>Displays and sets the ion source temperature.</td>
</tr>
<tr>
<td>Quadrupole Temp, °C</td>
<td>Displays and sets the quadrupole temperature.</td>
</tr>
<tr>
<td>CI Reagent</td>
<td>Displays CI reagent gas and flow rate (if installed).</td>
</tr>
</tbody>
</table>

**NOTE**

MS parameters cannot be set from the LCP while an online MassHunter Data Acquisition session is connected to the MSD.

Table 9  Network menu

<table>
<thead>
<tr>
<th>Action</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>MSD IP via keyboard</td>
<td>Displays the IP address for the MSD and is programmed via MassHunter Data Acquisition.</td>
</tr>
<tr>
<td>Gateway IP Address</td>
<td>Displays the gateway IP address for the MSD.</td>
</tr>
<tr>
<td>Subnet Mask</td>
<td>Displays the subnet mask for the MSD.</td>
</tr>
<tr>
<td>GC Comm IP Address</td>
<td>Displays the IP address</td>
</tr>
</tbody>
</table>
### Table 9  Network menu (continued)

<table>
<thead>
<tr>
<th>Action</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>ChemStation IP</td>
<td>Displays the IP address for the MassHunter Data Acquisition.</td>
</tr>
<tr>
<td>GC IP Address</td>
<td>Displays the IP address for the GC.</td>
</tr>
<tr>
<td>Ping gateway</td>
<td>Checks communication with the gateway.</td>
</tr>
<tr>
<td>Ping ChemStation</td>
<td>Checks communication with the MassHunter Data Acquisition.</td>
</tr>
<tr>
<td>Ping GC</td>
<td>Checks communication with the GC.</td>
</tr>
<tr>
<td>MAC Address</td>
<td>Displays the MAC address of the SmartCard in the MSD.</td>
</tr>
<tr>
<td>Reboot with new network settings</td>
<td>Reboots the system and saves the new network settings.</td>
</tr>
</tbody>
</table>

### Table 10  Version menu

<table>
<thead>
<tr>
<th>Action</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control firmware</td>
<td>Displays the MSD firmware version.</td>
</tr>
<tr>
<td>Operating system</td>
<td>Displays the MassHunter Data Acquisition operating system version.</td>
</tr>
<tr>
<td>Front panel</td>
<td>Displays the version of the LCP.</td>
</tr>
<tr>
<td>Log amplifier</td>
<td>Displays version information.</td>
</tr>
<tr>
<td>Sideboard</td>
<td>Displays the sideboard type.</td>
</tr>
<tr>
<td>Mainboard</td>
<td>Displays the mainboard type.</td>
</tr>
<tr>
<td>Serial number</td>
<td>Is assigned to the MSD by MassHunter Data Acquisition Configuration dialogue.</td>
</tr>
</tbody>
</table>

### Table 11  Controller menu

<table>
<thead>
<tr>
<th>Action</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reboot controller</td>
<td>Starts the LAN/MS control card.</td>
</tr>
<tr>
<td>Test LCP?</td>
<td>Initiates a diagnostic test of the two-line display.</td>
</tr>
<tr>
<td>Test HTTP link to GC/MSD ChemStation?</td>
<td>Checks the status of the HTTP server.</td>
</tr>
</tbody>
</table>
The EI GC/MSD Interface

The GC/MSD interface (Figure 11 on page 54) is a heated conduit into the MSD for the capillary column. It is bolted onto the right side of the analyzer chamber, with an O-ring seal. It has a protective cover which should be left in place.

One end of the GC/MSD interface passes through the side of the gas chromatograph and extends into the GC 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 last 1 to 2 mm of the capillary column extend past the end of the guide tube and into the ionization chamber.

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

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

The EI GC/MSD interface can only be used with an EI ion source. There are two types of tip seals that may be used with EI ion sources. The standard/inert EI ion sources do not need a tip seal. The extractor ion source on an EI only system uses a tip seal (G3870-20542). The extractor ion source and the CI ion source on an EI/CI system uses the CI tip seal (G1999-60412).

See also

“To Install a Capillary Column in the GC/MS Interface” on page 38.

WARNING

The GC/MSD interface operates at high temperatures. If you touch it when it is hot, it will burn you.
Column end protrudes 1 to 2 mm into the ionization chamber.

Figure 11  The EI GC/MSD interface
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 or reagent gasses are being used.
- The MSD is connected to a grounded power source.
- The GC/MSD interface extends into the GC oven.
- A conditioned capillary column is installed in the GC inlet and in the GC/MSD interface.
- The GC is on, but the heated zones for the GC/MSD interface, the GC inlet, and the oven are off.
- Carrier gas of at least 99.9995% purity is plumbed to the GC with the recommended traps.
- If hydrogen is used as carrier gas, carrier gas flow must be off and the front sideplate thumbscrew must be loosely fastened.
- The foreline pump exhaust is properly vented.

**WARNING**

The exhaust from the foreline pump contains solvents and the chemicals you are analyzing. 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 (11-mm id) 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 you are using hydrogen as a carrier gas, do not start carrier gas flow until the MSD has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the MSD and an explosion may occur. Read “Hydrogen Safety” on page 19 before operating the MSD with hydrogen carrier gas.
Pumping Down

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

Using the menus or MS monitors, the data system can display:

- 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 LCP can also display these data.

Controlling Temperatures

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

Normally, the GC/MSD interface heater is powered and controlled by the Thermal Aux #2 heated zone of the GC. For the 7820 Series GC’s, the heater is either connected to the rear inlet thermal zone for single inlet models or is connected to the manual valve thermal zone for dual inlet models. The GC/MSD interface temperature can be set and monitored from the data system or from the GC.

Controlling Column Flow

Carrier gas flow is controlled by inlet pressure in the GC. For a given inlet pressure, column flow will decrease as the GC oven temperature increases. With electronic pneumatic control (EPC) and the column mode set to **Constant Flow**, the same column flow is maintained regardless of temperature.
The MSD can be used to measure actual column flow. You inject a small amount of air or other unretained chemical and time how long it takes to reach the MSD. With this time measurement, you can calculate the column flow. See “To Calibrate Column Flow Linear Velocity” on page 66.

**Venting the MSD**

A program in the data system 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 you are using hydrogen as a carrier gas, the carrier gas flow must be off before turning off the MSD power. If the foreline pump is off, hydrogen will accumulate in the MSD and an explosion may occur. Read “Hydrogen Safety” on page 19 before operating the MSD with hydrogen carrier gas.

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

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

Do not exceed the maximum recommended total gas flow. See “5977 series MSD models and features” on page 14.
3 Operating in Electron Ionization (EI) Mode

To View MSD Temperature and Vacuum in Manual Tune

You can also use the Local Control Panel to perform this task. See “Operating the MSD from the LCP” on page 46.

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.

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

Unless you have just begun the pumpdown process, the foreline pressure should be less than 300 mTorr, or the turbo pump should be 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 the MSD zones are turned off.
To Set Monitors for MSD Temperature and Vacuum Status

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

Procedure

1. In Instrument Control view, select Edit Monitors from the Instrument menu to display the Select Monitors dialog.

2. In the Available Monitors column, select a monitor and click the Add button 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. Select Window > Arrange Monitors, or click and drag each monitor to the desired position.
Operating in Electron Ionization (EI) Mode

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.

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

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

Procedure

1. In Instrument Control view, select MS Temperatures from the Instrument menu.

2. Enter the MS Source and MS Quad (mass filter) temperatures in the Setpoint and Limit fields. See Table 12.

Table 12  Recommended temperature settings

<table>
<thead>
<tr>
<th></th>
<th>EI operation</th>
<th>PCI operation</th>
<th>NCI operation</th>
</tr>
</thead>
<tbody>
<tr>
<td>MS Source</td>
<td>230</td>
<td>250</td>
<td>150</td>
</tr>
<tr>
<td>MS Quad</td>
<td>150</td>
<td>150</td>
<td>150</td>
</tr>
</tbody>
</table>

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 GC/MSD Interface Temperature from MassHunter

**Procedure**

1. From **Instrument Control** view select **Instrument>GC Edit Parameters**.
2. Click the **Aux Heater** icon to edit the interface temperature.

3. Select **On** to turn on the heater and type the setpoint in the **Value °C** column.

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

   **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.
Operating in Electron Ionization (EI) Mode

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, do not turn on the Micro-Ion vacuum gauge if there is any possibility that hydrogen has accumulated in the analyzer chamber. Read “Hydrogen Safety” on page 19 before operating the MSD with hydrogen carrier gas.

**Procedure**

1. Start up and pump down the MSD (“To Pump Down the MSD in EI Mode” on page 84).
2. In the Tune and Vacuum Control view select **Turn Vacuum Gauge on/off** from the **Vacuum** menu.
3. Select **Manual Tune** from the **Parameters** menu to display the Manual Tune dialog.
4. Select the **Values** tab to view the HiVac reading.
The largest influence on operating pressure in EI mode is the carrier gas (column) flow. Table 13 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 13  Ion vacuum gauge reading

<table>
<thead>
<tr>
<th>Column flow rate, mL/min</th>
<th>Optional Gauge reading, Torr Performance turbo pump</th>
<th>Gauge reading, Torr Diffusion pump</th>
<th>Foreline reading, Torr Diffusion pump</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5</td>
<td>3.18E–06</td>
<td>2.18E–05</td>
<td>34.7</td>
</tr>
<tr>
<td>0.7</td>
<td>4.42E–06</td>
<td>2.59E–05</td>
<td>39.4</td>
</tr>
<tr>
<td>1</td>
<td>6.26E–06</td>
<td>3.66E–05</td>
<td>52.86</td>
</tr>
<tr>
<td>1.2</td>
<td>7.33E–06</td>
<td>4.46E–05</td>
<td>60.866</td>
</tr>
<tr>
<td>2</td>
<td>1.24E–05</td>
<td>7.33E–05</td>
<td>91.784</td>
</tr>
<tr>
<td>3</td>
<td>1.86E–05</td>
<td>1.13E–04</td>
<td>125.76</td>
</tr>
<tr>
<td>4</td>
<td>2.48E–05</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>3.75E–05</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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

In the **Instrument Control** view you can set up an MS Monitor for displaying this vacuum reading. The vacuum can also be read on the LCP or from the Manual Tune screen.
To Calibrate Column Flow Linear Velocity

Capillary columns must be calibrated prior to use with the MS.

Procedure

1. Set Data Acquisition for splitless manual injection and set up a real time plot to monitor $m/z$ 28.
3. Inject 1 µL of air into the GC inlet and press [Start Run].
4. Wait until a peak elutes at $m/z$ 28. Note the retention time.
5. 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 the Calibrate button to display the Calibrate Column dialog.
9 Click the **Calc Length** button in the **If unretained peak holdup time is known** section to display the **Calculate 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.
3 Operating in Electron Ionization (EI) Mode

**Calculation for average linear velocity**

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**

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

where:

\( D \) = Internal column diameter in millimeters

\( L \) = Column length in meters

\( t \) = Retention time in minutes
To Tune the MSD in EI Mode

You can also use the Local Control Panel to run the autotune that is currently loaded in MassHunter. See “Operating the MSD from the LCP” on page 46.

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, ATUNE.U (Autotune) gives good results. STUNE.U (Standard Tune) is not recommended as it may reduce sensitivity.

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 ion source, select a tune file created for an EI ion source.

4. Click the MS Tune icon to display the 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.

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 MassHunter Data Acquisition software for additional information about tuning.
To Verify System Performance

Materials needed

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

Verify the tune performance

1 Verify that the system has been pumping down for at least 60 minutes.
2 Set the 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 µL of OFN, 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. Please see the Agilent Web site at www.agilent.com/chem for specifications.
To Perform High-Mass Testing (5977 Series MSDs)

Materials needed

- PFHT calibration sample (5188-5357)

Procedure

1. Load tune file ATUNE.U then auto tune the MSD. See “To Tune the MSD in EI Mode” on page 69.
2. Resolve the PFHT.M method under x\5977\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 12 on page 73.
Results

*PFHT HIGH MASS REPORT

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

Figure 12  PFHT high mass report
Results will indicate the recommended amount to adjust AMU offset for high-mass. If your results are within 5 units of the targeted amount, there is no need to make adjustments.

Adjustments

1. Verify ATUNE.U has been loaded.
2. 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 ATUNE.U to include high-mass adjustment or save this file to a new name, for example, ATUNEHIGH.U.

   Anytime an ATUNE.U 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 off the MSD.

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

To open the analyzer cover

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.

WARNING
Do not remove any other covers. Dangerous voltages are present under other covers.
3 Operating in Electron Ionization (EI) Mode

To Vent the MSD

Procedure

1. 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.

2. In Instrument Control view select Edit Tune Parameters from the Instrument menu to display the Manual Tune dialog.

3. Select the Values tab and set the MS Source and MS Quad temperatures to ambient (room temperature) and click Apply to download these settings to the MSD.

**WARNING** If you are using hydrogen as a carrier gas, the carrier gas flow must be off before turning off the MSD power. If the foreline pump is off, hydrogen will accumulate in the MSD and an explosion may occur. Read “Hydrogen Safety” on page 19 before operating the MSD with hydrogen carrier gas.

**CAUTION** Be sure the GC oven and the GC/MSD interface are cool before turning off carrier gas flow to prevent damage to the column.
4. In the Instrument Control view, Instrument menu, select MS Vacuum Control to display the Vacuum Control dialog.

5. Remove the analyzer window cover (see “To Open the MSD Covers” on page 75).

6. Click Vent to begin the automated shutdown of the MSD. Follow the instructions presented.

7. 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.

Do not turn the knob too far or the O-ring may fall out of its groove. Be sure to close the vent valve before pumping down.
3 Operating in Electron Ionization (EI) Mode

To Open the Analyzer Chamber

Materials needed

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

**CAUTION**

Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded antistatic wrist strap and take other antistatic precautions (page 123) before opening the analyzer chamber.

Procedure

1 Vent the MSD (see “To Vent the MSD” on page 76).
2 Disconnect the side board control cable and the source power cable from the side board.
3 Loosen the side plate thumbscrews if they are fastened.

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

**CAUTION**

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

**WARNING**

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

**CAUTION**

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

4 *Gently* swing the side plate out.
To Close the Analyzer Chamber

Materials needed

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

Procedure

1. Ensure all the internal analyzer electrical leads are correctly attached. Wiring is the same for both the standard EI and CI ion sources. The extractor EI ion source has an extra wire leading to the extractor lens.

The wiring is described in Table 14 and illustrated in Figure 13 on page 81 and Figure 14 on page 82. The term “Board” in the table refers to the feedthrough board located next to the ion source.

Table 14  Source board wiring

<table>
<thead>
<tr>
<th>Wire description</th>
<th>Attached to</th>
<th>Connects to</th>
</tr>
</thead>
<tbody>
<tr>
<td>Green beaded (2)</td>
<td>Quad heater</td>
<td>Board, top left (HTR)</td>
</tr>
<tr>
<td>White with braided cover (2)</td>
<td>Quad sensor</td>
<td>Board, top (RTD)</td>
</tr>
<tr>
<td>White (2)</td>
<td>Board, center (FILAMENT-1)</td>
<td>Filament 1 (top)</td>
</tr>
<tr>
<td>Red (1)</td>
<td>Board, center left (REP)</td>
<td>Repeller</td>
</tr>
<tr>
<td>Black (2)</td>
<td>Board, center (FILAMENT-2)</td>
<td>Filament 2 (bottom)</td>
</tr>
<tr>
<td>Orange (1)</td>
<td>Board, top right (ION FOC)</td>
<td>Ion focus lens</td>
</tr>
<tr>
<td>Blue (1)</td>
<td>Board, top right (ENT LENS)</td>
<td>Entrance lens</td>
</tr>
<tr>
<td>White beaded (2)</td>
<td>Ion source heater</td>
<td>Board, bottom left (HTR)</td>
</tr>
<tr>
<td>White (2)</td>
<td>Ion source sensor</td>
<td>Board, bottom (RTD)</td>
</tr>
<tr>
<td>Brown (1)</td>
<td>Extractor lens</td>
<td>Board, middle left</td>
</tr>
<tr>
<td></td>
<td>(Extractor EI ion source only)</td>
<td></td>
</tr>
</tbody>
</table>
Figure 13  Feedthrough board wiring
Check the side plate O-ring. Ensure 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 MSD Troubleshooting and Maintenance Manual for lubricating instructions.

2 Close the side plate.
3 Reconnect the side board control cables.
4 Make sure the vent valve is closed.
5 Pump down the MSD. See “To Pump Down the MSD in EI Mode” on page 84.
6 If you are operating in CI mode, or if hydrogen or other flammable or toxic substance is used for carrier gas, *gently* hand tighten the front side plate thumbscrew.
Once the MSD has pumped down, close the analyzer cover.

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

**CAUTION** Do not overtighten the thumbscrew; it can cause air leaks or prevent successful pumpdown. Do not use a screwdriver to tighten the thumbscrew.
To Pump Down the MSD in EI Mode

You can also use the Local Control Panel to perform this task. See “Operating the MSD from the LCP” on page 46.

**WARNING**

Make sure your MSD meets all the conditions listed in the introduction to this chapter (page 53) before starting up and pumping down the MSD. Failure to do so can result in personal injury.

**WARNING**

If you are using hydrogen as a carrier gas, do not start carrier gas flow until the MSD has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the MSD and an explosion may occur. Read “Hydrogen Safety” on page 19 before operating the MSD with hydrogen carrier gas.

**Procedure**

1. Remove the analyzer window cover (see “To Open the MSD Covers” on page 75).
2. Close the vent valve by turning the knob clockwise.
3. Plug in the MSD power cord.
4. Press the **Power on** button on the front of the MSD.
5 Press lightly on the side board to ensure a correct seal. Press on the metal box on the side board.

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

6 Start the MassHunter Data Acquisition program.

7 In the Instrument Control view, from the Instrument menu, select MS Vacuum Control to display the Vacuum Control dialog.

8 In the Instrument Control view, Instrument menu, select MS Vacuum Control to display the Vacuum Control dialog.

9 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.

10 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.

11 After the message Okay to run appears, wait 2 hours for the MSD to reach thermal equilibrium. Data acquired before the MSD has reached thermal equilibrium may not be reproducible.
To Move or Store the MSD

Materials needed

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

Procedure

1. Vent the MSD (See “To Vent the MSD” on page 76).
2. Remove the column and install a blank ferrule and interface nut.
3. Tighten the vent valve.
4. Move the MSD away from the GC (see the 5977 Series MSD Troubleshooting and Maintenance Manual).
5. Unplug the GC/MSD interface heater cable from the GC.
6. Open the analyzer cover (See “To Open the MSD Covers” on page 75).
7. Finger-tighten the side plate thumbscrews.

**CAUTION**

Do not overtighten the side plate thumbscrews. Overtightening will strip the threads in the analyzer chamber. It will also warp the side plate and cause leaks.
8  Plug the MSD power cord in.
9  Switch the MSD on to establish a rough vacuum. Verify that the turbo pump speed is greater than 50%, or that the foreline pressure is ~1 Torr.
10  Switch the MSD off.
11  Close the analyzer cover.
12  Disconnect the LAN, remote, and power cables.

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

**CAUTION**

The MSD must remain upright at all times. If you need to ship your MSD to another location, contact your Agilent Technologies service representative for advice about packing and shipping.
3 Operating in Electron Ionization (EI) Mode
4
Operating in Chemical Ionization (CI) Mode

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To Switch from the Extractor EI Ion Source to the CI Ion Source 97
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This chapter provides information and instructions for operating the 5977 Series CI MSDs in Chemical Ionization (CI) mode. Most of the information in the preceding chapter is also relevant.

Most of the material is related to methane chemical ionization 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 positive CI (PCI) with methane reagent gas and for negative CI (NCI) with any reagent gas.
General Guidelines

- Always use the highest purity methane (and other reagent gases, if applicable). Methane must be at least 99.9995% pure.
- Always verify the MSD is performing well in EI mode before switching to CI. See “To Verify System Performance” on page 71.
- Make sure the CI ion source and GC/MSD interface tip seal are installed.
- Make sure the reagent gas plumbing has no air leaks. This is determined in PCI mode, checking for $m/z$ 32 after the methane pretune.
- Make sure the reagent gas inlet line is equipped with gas purifiers (not applicable for ammonia).
The CI GC/MSD Interface

The CI GC/MSD interface (Figure 15 on page 92) is a heated conduit into the MSD for the capillary column. It is bolted onto the right side of the analyzer chamber, with an O-ring seal and 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. It is threaded to allow connection of the column with a nut and ferrule. The other end of the interface fits into the ion source. The last 1 to 2 mm of the capillary column extend past the end of the guide tube and into the ionization chamber.

Reagent gas is plumbed into the interface. The tip of the interface assembly extends into the ionization chamber. A spring-loaded seal keeps reagent gases from leaking out of the CI ion source at the tip. The reagent gas enters the interface body and mixes with carrier gas and sample in the ion source.

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 MassHunter Data Acquisition or from the gas chromatograph. A sensor (thermocouple) in the interface monitors the temperature.

This interface is also used for EI operation in CI MSDs. The CI tip seal, necessary for CI operation, can stay in place when the EI extractor ion source is in use. It is easily replaced for the standard or inert EI ion source.

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

**CAUTION**

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

**WARNING**

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

“To Install a Capillary Column in the GC/MS Interface” on page 38.
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 94. 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. EMVolts (electron multiplier voltage) at or above 2600 V, however, indicates a problem. If your method requires EMVolts set at +400, you may not have adequate sensitivity in your data acquisition.

**CAUTION**

Always verify MSD performance in EI before switching to CI operation. See “To Verify System Performance” on page 71. Always set up the CI MSD in PCI first, even if you are going to run NCI.
### Operating in Chemical Ionization (CI) Mode

#### Table 15 Reagent gas settings

<table>
<thead>
<tr>
<th>Reagent gas</th>
<th>Methane</th>
<th>Isobutane</th>
<th>Ammonia</th>
<th>EI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ion polarity</td>
<td>Positive</td>
<td>Negative</td>
<td>Positive</td>
<td>Negative</td>
</tr>
<tr>
<td>Emission</td>
<td>150 μA</td>
<td>50 μA</td>
<td>150 μA</td>
<td>50 μA</td>
</tr>
<tr>
<td>Electron energy</td>
<td>150 eV</td>
<td>150 eV</td>
<td>150 eV</td>
<td>150 eV</td>
</tr>
<tr>
<td>Filament</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Repeller</td>
<td>3 V</td>
<td>3 V</td>
<td>3 V</td>
<td>3 V</td>
</tr>
<tr>
<td>Ion focus</td>
<td>130 V</td>
<td>130 V</td>
<td>130 V</td>
<td>130 V</td>
</tr>
<tr>
<td>Entrance lens offset</td>
<td>20 V</td>
<td>20 V</td>
<td>20 V</td>
<td>20 V</td>
</tr>
<tr>
<td>EM volts</td>
<td>1200</td>
<td>1400</td>
<td>1200</td>
<td>1400</td>
</tr>
<tr>
<td>Shutoff valve</td>
<td>Open</td>
<td>Open</td>
<td>Open</td>
<td>Open</td>
</tr>
<tr>
<td>Gas select</td>
<td>A</td>
<td>A</td>
<td>B</td>
<td>B</td>
</tr>
<tr>
<td>Suggested flow</td>
<td>20%</td>
<td>40%</td>
<td>20%</td>
<td>40%</td>
</tr>
<tr>
<td>Source temp</td>
<td>250 °C</td>
<td>150 °C</td>
<td>250 °C</td>
<td>150 °C</td>
</tr>
<tr>
<td>Quad temp</td>
<td>150 °C</td>
<td>150 °C</td>
<td>150 °C</td>
<td>150 °C</td>
</tr>
<tr>
<td>Interface temp</td>
<td>280 °C</td>
<td>280 °C</td>
<td>280 °C</td>
<td>280 °C</td>
</tr>
<tr>
<td>Autotune</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
</tr>
</tbody>
</table>

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 (Table 16), and electron energy may need to be optimized for your specific analyte.

**Table 16  Temperatures for CI operation**

<table>
<thead>
<tr>
<th></th>
<th>Ion source</th>
<th>Quadrupole</th>
<th>GC/MSD interface</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCI</td>
<td>250 °C</td>
<td>150 °C</td>
<td>280 °C</td>
</tr>
<tr>
<td>NCI</td>
<td>150 °C</td>
<td>150 °C</td>
<td>280 °C</td>
</tr>
</tbody>
</table>

**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).
- Tell 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)
4 Operating in Chemical Ionization (CI) Mode

To Switch from the Standard or Inert EI ion source to the CI Ion 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 76.
2. Open the analyzer. See “To Open the Analyzer Chamber” on page 78.
3. Remove the EI ion source. See “To Remove the EI Ion Source” on page 128.

**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 123. Take antistatic precautions before you open the analyzer chamber.

4. Install the CI ion source. See “To Install the CI Ion Source” on page 167.
5. Install the CI/Extractor interface tip seal (p/n G3870-20542). See “To Install the CI/Extractor Interface Tip Seal” on page 155.
6. Close the analyzer. See “To Close the Analyzer Chamber” on page 80.
7. Pump down the MSD. See “To Pump Down the MSD in CI Mode” on page 98.
To Switch from the Extractor EI Ion Source to the CI Ion 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.

**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 123. Take antistatic precautions before you open the analyzer chamber.

**Procedure**
1. Vent the MSD. See “To Vent the MSD” on page 76.
2. Open the analyzer. See “To Open the Analyzer Chamber” on page 78.
3. Remove the extractor EI ion source. See “To Remove the EI Ion Source” on page 128.
4. Remove the brown extractor wire from the feedthrough board and store it with the EI extractor ion source. See Figure 13 on page 81.
5. Install the CI ion source. See “To Install the CI Ion Source” on page 167.
6. Close the analyzer. See “To Close the Analyzer Chamber” on page 80.
7. Pump down the MSD. See “To Pump Down the MSD in CI Mode” on page 98.
To Pump Down 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 Pump Down the MSD in EI Mode” on page 84.

   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.

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

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 17 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 17**  Default Tune Control Limits, used by CI autotune only

<table>
<thead>
<tr>
<th>Reagent gas</th>
<th>Methane</th>
<th>Isobutane</th>
<th>Ammonia</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ion polarity</td>
<td>Positive</td>
<td>Negative</td>
<td>Positive</td>
</tr>
<tr>
<td>Abundance target</td>
<td>1x10^6</td>
<td>1x10^6</td>
<td>N/A</td>
</tr>
<tr>
<td>Peakwidth target</td>
<td>0.6</td>
<td>0.6</td>
<td>N/A</td>
</tr>
<tr>
<td>Maximum repeller</td>
<td>4</td>
<td>4</td>
<td>N/A</td>
</tr>
<tr>
<td>Maximum emission current, µA</td>
<td>240</td>
<td>50</td>
<td>N/A</td>
</tr>
<tr>
<td>Max electron energy, eV</td>
<td>240</td>
<td>240</td>
<td>N/A</td>
</tr>
</tbody>
</table>

**Notes for Table 17:**

- 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.
Operating in Chemical Ionization (CI) Mode

- **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 MS 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.

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 Valve**.

The system turns off the present gas flow while leaving the shutoff valve (Figure 16 on page 103) open. This is to remove any residual gas in the lines. Typical evacuation time is 6 minutes and then the shutoff valve is closed.

**The 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 mass flow controller (MFC), gas select valves, CI calibration valve, shutoff valve, control electronics, and plumbing. See Figure 16 and Table 18 on page 103.

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.
The Open and Closed states are shown in the monitors as 1 and 0 respectively.
Operating in Chemical Ionization (CI) Mode

To Set Up Methane Reagent Gas Flow

The reagent gas flow must be adjusted for maximum stability before tuning the CI system. Do the initial setup with methane in positive chemical ionization (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 ion source, perform the standard autotune, save the report, and note the reported pressure. See “To Tune the MSD in EI Mode” on page 69.
2. Vent the system. See “Venting the MSD” on page 57.
3. Install the CI ion source. See “To Install the CI Ion Source” on page 167.
4. Pump out the system. See “To Pump Down the MSD in CI Mode” on page 98.
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 117.
6. Select Bake out MSD from the Manual Tune view Execute menu to display the Specify Bake Out parameters dialog. Set for a minimum time of 2 hours, adjust the other parameters, and click OK to begin the bake out.

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 MassHunter 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.
Examine the displayed profile scan of the reagent ions.

* 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.

When prompted, click **OK** to perform the methane Flow Adjust.

![Reagent ion scans after a very long bake out](image)

**Figure 17** 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 5977 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 chemical ionization reactions encountered are described in general in the 5977 Series Concepts Guide. If you are not experienced with chemical ionization, we suggest reviewing that material before you proceed.

**CAUTION**

Not all setup operations can be performed in all modes with all reagent gases. See Table 19 for details.

### Table 19 Reagent gases

<table>
<thead>
<tr>
<th>Reagent gas/mode</th>
<th>Reagent ion masses</th>
<th>PFDTD Calibrant ions</th>
<th>Flow adj ions: Ratio</th>
<th>Flow adj ions: Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>EI/PCI/NCI MSD Performance turbo pump</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Recommended flow: 20% PCI 40% NCI</td>
</tr>
<tr>
<td>Methane/PCI</td>
<td>17, 29, 41</td>
<td>41, 267, 599</td>
<td>28/27: 1.5 – 5.0</td>
<td></td>
</tr>
<tr>
<td>Methane/NCI</td>
<td>17, 35, 235†</td>
<td>185, 351, 449</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Isobutane/PCI</td>
<td>39, 43, 57</td>
<td>N/A</td>
<td>57/43: 5.0 – 30.0</td>
<td></td>
</tr>
<tr>
<td>Isobutane/NCI</td>
<td>17, 35, 235</td>
<td>185, 351, 449</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Ammonia/PCI</td>
<td>18, 35, 52</td>
<td>N/A</td>
<td>35/18: 0.1 – 1.0</td>
<td></td>
</tr>
<tr>
<td>Ammonia/NCI</td>
<td>17, 35, 235</td>
<td>185, 351, 517</td>
<td>N/A</td>
<td></td>
</tr>
</tbody>
</table>

* 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 (C₄H₁₀) is commonly used for chemical ionization when less fragmentation is desired in the chemical ionization spectrum. This is because the proton affinity of isobutane is higher than that of methane; hence, less energy is transferred in the ionization reaction.

Addition and proton transfer are the ionization mechanisms most often associated with isobutane. The sample itself influences which mechanism dominates.

Ammonia CI

Ammonia (NH₃) is commonly used for chemical ionization when less fragmentation is desired in the chemical ionization spectrum. This is because the proton affinity of ammonia is higher than that of methane; hence, less energy is transferred in the ionization reaction.

Because many compounds of interest have insufficient proton affinities, ammonia chemical-ionization spectra often result from the addition of NH₄⁺ 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 NH₄⁺, NH₄(NH₃)⁺, and NH₄(NH₃)₂⁺.

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

Procedure

1 Perform a standard Positive CI autotune with methane and PFDTD. See “To Perform a PCI Autotune (Methane Only)” on page 111.

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.
Operating in Chemical Ionization (CI) Mode

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 Agilent 5977 Series MSDs” (5989-5170EN).

**CAUTION** Use of ammonia affects the maintenance requirements of the MSD. See “CI Maintenance” on page 153 for more information.

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

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

Ammonia tends to break down vacuum pump fluids and seals. Ammonia CI makes more frequent vacuum system maintenance necessary. (See the **5977 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 chemical ionization 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 Switch from the CI Ion Source to the Standard or Inert EI Ion 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 123.

1. From the Tune and Vacuum Control view, vent the MSD. See “To Vent the MSD” on page 76. The software will prompt you for the appropriate actions.
2. Open the analyzer. See “To Open the Analyzer Chamber” on page 78.
3. Remove the CI/Extractor interface tip seal. See “To Install the CI/Extractor Interface Tip Seal” on page 155.
4. Install the EI ion source. See “To Install the EI Ion Source” on page 150.
5. Place the CI ion source and interface tip seal in the ion source storage box.
6. Pump down the MSD. See “To Pump Down the MSD in EI Mode” on page 84.
7. Load your EI tune file.
To Switch from the CI Ion Source to the Extractor EI Ion Source

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

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 123.

Procedure

1. From the Tune and Vacuum Control view, vent the MSD. See “To Vent the MSD” on page 76. The software will prompt you for the appropriate actions.
2. Open the analyzer. See “To Open the Analyzer Chamber” on page 78.
3. Remove the CI ion source. See “To Remove the CI Ion Source” on page 157. There is no need to remove the tip seal, as in the standard or inert EI ion source. The CI interface tip seal will fit in the extractor EI ion source.
4. Install the EI extractor ion source. See “To Install the EI Ion Source” on page 150.
5. Locate the brown extractor wire from storage and connect it to the extractor lens and the source board.
6. Place the CI ion source in the ion source storage box.
7. Pump down the MSD. See “To Pump Down the MSD in EI Mode” on page 84.
8. Load your EI tune file.
To Perform a PCI Autotune (Methane Only)

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 System Performance” on page 71.

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

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

3. Accept the default settings.


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

   There are no tune performance criteria. If autotune completes, it passes (See **Figure 18** on page 112). If the tune sets the electron multiplier voltage (EMVolts) at or above 2600 V, however, you may not be able to acquire data successfully if your method sets EMVolts to “+400” or higher.

   The autotune report contains information about air and water in the system. See “PCI autotune report” on page 112.

   The 19/29 ratio shows the abundance of water.

   The 32/29 ratio shows the abundance of oxygen.
Operating in Chemical Ionization (CI) Mode

Figure 18  PCI autotune report
To Perform an NCI Autotune (Methane Reagent Gas)

Always verify MSD performance in EI before switching to CI operation. See “To Verify System Performance” on page 71. 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 19 on page 114). If the tune sets the electron multiplier voltage (EMVolts) at or above 2600 V, however, you may not be able to acquire data successfully if your method sets EMVolts to “+400” or higher.
Operating in Chemical Ionization (CI) Mode

Figure 19  NCI autotune
To Verify PCI Performance

Materials needed

- Benzophenone, 100 pg/µL (8500-5440)

CAUTION

Always verify MSD performance in EI before switching to CI operation. See “To Verify System Performance” on page 71. 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 Switch from the CI Ion Source to the Extractor EI Ion Source” on page 110.
5. Run CI Autotune. See “CI Autotune” on page 93.
6. Run the PCI sensitivity method `BENZ_PCI.M` using 1 µL of 100 pg/µL benzophenone.
7. Verify that the system conforms to the published sensitivity specification. Please see the Agilent Web site at www.agilent.com/chem for specifications.
4 Operating in Chemical Ionization (CI) Mode

To Verify NCI Performance

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

Materials needed

- Octafluoronaphthalene (OFN), 100 fg/µL (5188-5347)

CAUTION Always verify MSD performance in EI before switching to CI operation. See “To Verify System Performance” on page 71. 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%.

   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_NCLM using 2 µL of 100 fg/µL OFN.
6. Verify that the system conforms to the published sensitivity specification. Please see the Agilent Web site at www.agilent.com/chem for specifications.
To Monitor CI Mode High Vacuum Pressure

**WARNING**

If you are using hydrogen as a carrier gas, do not turn on the Micro-Ion vacuum gauge if there is any possibility that hydrogen has accumulated in the manifold. Read “Hydrogen Safety” on page 19 before operating the MSD with hydrogen carrier gas.

**Procedure**

1. Start up and pump down the MSD. See “To Pump Down the MSD in CI Mode” on page 98.

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 $8 \times 10^{-3}$ 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. Table 20 on page 118 lists typical pressures for various helium carrier gas flows. These pressures are approximate and vary from instrument to instrument.

**Typical pressure readings**

Use the G3397B Micro-Ion vacuum gauge. Note that the mass flow controller 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 20 on page 118). They were taken with the following set of conditions. Note that these are typical PCI temperatures:

<table>
<thead>
<tr>
<th>Source temperature</th>
<th>250 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quad temperature</td>
<td>150 °C</td>
</tr>
<tr>
<td>Interface temperature</td>
<td>280 °C</td>
</tr>
<tr>
<td>Helium carrier gas flow</td>
<td>1 mL/min</td>
</tr>
</tbody>
</table>
Table 20  Mass flow controller settings and typical pressure readings

<table>
<thead>
<tr>
<th>MFC (%)</th>
<th>Methane</th>
<th>Ammonia</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>EI/PCI/NCI MSD (Turbo pump)</td>
<td>EI/PCI/NCI MSD (Turbo pump)</td>
</tr>
<tr>
<td>10</td>
<td>$5.5 \times 10^{-5}$</td>
<td>$5.0 \times 10^{-5}$</td>
</tr>
<tr>
<td>15</td>
<td>$8.0 \times 10^{-5}$</td>
<td>$7.0 \times 10^{-5}$</td>
</tr>
<tr>
<td>20</td>
<td>$1.0 \times 10^{-4}$</td>
<td>$8.5 \times 10^{-5}$</td>
</tr>
<tr>
<td>25</td>
<td>$1.2 \times 10^{-4}$</td>
<td>$1.0 \times 10^{-4}$</td>
</tr>
<tr>
<td>30</td>
<td>$1.5 \times 10^{-4}$</td>
<td>$1.2 \times 10^{-4}$</td>
</tr>
<tr>
<td>35</td>
<td>$2.0 \times 10^{-4}$</td>
<td>$1.5 \times 10^{-4}$</td>
</tr>
<tr>
<td>40</td>
<td>$2.5 \times 10^{-4}$</td>
<td>$2.0 \times 10^{-4}$</td>
</tr>
</tbody>
</table>

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.
5 General Maintenance

Before Starting  120
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Maintaining the Analyzer  126
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To Disassemble the Standard or Inert EI Ion Source  131
To Disassemble the Extractor EI Ion Source  134
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To Assemble a Standard or Inert EI Ion Source  142
To Assemble the Extractor EI Ion Source  145
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5  General Maintenance

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 21  Maintenance schedule

<table>
<thead>
<tr>
<th>Task</th>
<th>Every week</th>
<th>Every 6 months</th>
<th>Every year</th>
<th>As needed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tune the MSD</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Check the foreline pump oil level</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Check the calibration vial(s)</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replace the foreline pump oil*</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Replace the diffusion pump fluid</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Check the dry foreline pump</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Change the dry foreline pump tip seal</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Change the foreline pump exhaust filter</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Clean the ion source</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Check the carrier gas trap(s) on the GC and MSD</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Replace the worn out parts</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Lubricate sideplate or vent valve O-rings†</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Replace CI Reagent gas supply</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Replace GC gas supplies</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
</tbody>
</table>

* 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

Common maintenance tasks are listed in Table 21. Performing these tasks when scheduled can reduce operating problems, prolong system life, and reduce overall operating costs.
Keep a record of system performance (tune reports) and maintenance operations performed. This makes it easier to identify variations from normal operation and to take corrective action.

**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.

---

Electrostatic discharge

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

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

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

**CAUTION**

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

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

**Periodic maintenance**

As listed earlier in Table 21 on page 120, some maintenance tasks for the vacuum system must be performed periodically. These include:

- Checking the foreline pump fluid (every week)
- 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. See the 5977 Series MSD Troubleshooting and Maintenance manual and see the online help in MassHunter Data 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, see the 5977 Series MSD Troubleshooting and Maintenance manual.

Most of the procedures in this chapter are illustrated with video clips on the Agilent GC/GCMSD Hardware User Information & Instrument Utilities and 5977 Series MSD User Information disks.
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 5977 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 MassHunter software provides more extensive information.

Precautions

Cleanliness

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

CAUTION

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

WARNING

The analyzer operates at high temperatures. Do not touch any part until you are sure it is cool.
Some parts can be damaged by electrostatic discharge

The wires, contacts, and cables connected to the analyzer components can carry electrostatic discharges (ESD) to the electronics boards to which they are connected. This is especially true of the mass filter (quadrupole) contact wires which can conduct ESD to sensitive components on the side board. ESD damage may not cause immediate failure but will gradually degrade performance and stability. See page 123 for more information.

Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded antistatic wrist strap (see page 123) 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.

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 5977 Series MSD Troubleshooting and Maintenance Manual.

Many procedures in this chapter are illustrated with video clips.
To Remove the EI Ion 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 76.

2 Open the analyzer chamber. See “To Open the Analyzer Chamber” on page 78.

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

3 If you are using a standard or inert EI ion source, disconnect the seven wires from the ion source. Do not bend the wires any more than necessary (See Figure 20 on page 130 and Table 22).

Table 22  Standard EI ion source wires

<table>
<thead>
<tr>
<th>Wire color</th>
<th>Connects to</th>
<th>Number of leads</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blue</td>
<td>Entrance lens</td>
<td>1</td>
</tr>
<tr>
<td>Orange</td>
<td>Ion focus</td>
<td>1</td>
</tr>
<tr>
<td>White</td>
<td>Filament 1 (top filament)</td>
<td>2</td>
</tr>
<tr>
<td>Red</td>
<td>Repeller</td>
<td>1</td>
</tr>
<tr>
<td>Black</td>
<td>Filament 2 (bottom filament)</td>
<td>2</td>
</tr>
</tbody>
</table>

CAUTION Pull on the connectors, not on the wires.
4 If you are using an extractor EI ion source, disconnect the eight wires from the ion source. Do not bend the wires any more than necessary (See Figure 20 on page 130 and Table 23).

Table 23  Extractor EI ion source wires

<table>
<thead>
<tr>
<th>Wire color</th>
<th>Connects to</th>
<th>Number of leads</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blue</td>
<td>Entrance lens</td>
<td>1</td>
</tr>
<tr>
<td>Orange</td>
<td>Ion focus</td>
<td>1</td>
</tr>
<tr>
<td>White</td>
<td>Filament 1 (top filament)</td>
<td>2</td>
</tr>
<tr>
<td>Red</td>
<td>Repeller</td>
<td>1</td>
</tr>
<tr>
<td>Black</td>
<td>Filament 2 (bottom filament)</td>
<td>2</td>
</tr>
<tr>
<td>Brown</td>
<td>Extractor lens</td>
<td>1</td>
</tr>
</tbody>
</table>

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

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

7 Pull the ion source out of the source radiator.

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

Figure 20 Removing the EI ion source

- Ion source
- Source feedthrough board
- Thumbscrews
- Extractor wire
- Source heater and temperature sensor wires
- Source radiator
To Disassemble the Standard or Inert EI Ion Source

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Hex ball driver, 1.5 mm (8710-1570)
- Hex ball driver, 2.0 mm (8710-1804)
- Wrench, open-end, 10 mm (8710-2353)

Procedure

1. Remove the ion source. See “To Remove the EI Ion Source” on page 128.
2. Remove the two gold plated screws from the filaments and remove the filaments from the source. See Figure 21 on page 132.
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 21  Disassembling the standard or inert EI ion source

Table 24  Parts list for the standard or inert EI ion source (Figure 21)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Gold plated set screw</td>
</tr>
<tr>
<td>2</td>
<td>Gold plated screw</td>
</tr>
<tr>
<td>3</td>
<td>Interface socket</td>
</tr>
<tr>
<td>4</td>
<td>Source body</td>
</tr>
<tr>
<td>5</td>
<td>Drawout cylinder</td>
</tr>
<tr>
<td>6</td>
<td>Drawout plate</td>
</tr>
<tr>
<td>7</td>
<td>4-turn filament</td>
</tr>
</tbody>
</table>
### Table 24  Parts list for the standard or inert EI ion source (Figure 21) (continued)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>Spring washer</td>
</tr>
<tr>
<td>9</td>
<td>Lens insulator</td>
</tr>
<tr>
<td>10</td>
<td>Entrance lens</td>
</tr>
<tr>
<td>11</td>
<td>Ion focus lens</td>
</tr>
<tr>
<td>12</td>
<td>Repeller insulator</td>
</tr>
<tr>
<td>13</td>
<td>Repeller</td>
</tr>
<tr>
<td>14</td>
<td>Flat washer</td>
</tr>
<tr>
<td>15</td>
<td>Belleville spring washer</td>
</tr>
<tr>
<td>16</td>
<td>Repeller nut</td>
</tr>
<tr>
<td>17</td>
<td>Source heater block assembly</td>
</tr>
<tr>
<td>18</td>
<td>Repeller block insert</td>
</tr>
</tbody>
</table>
To Disassemble the Extractor EI Ion Source

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Hex ball driver, 1.5 mm (8710-1570)
- Hex ball driver, 2.0 mm (8710-1804)
- Wrench, open-end, 10 mm (8710-2353)

Procedure

1. Remove the ion source. See “To Remove the EI Ion Source” on page 128.
2. Remove the filaments by removing the two gold plated screws and separating the filaments from the source. See Figure 22 on page 135.
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 insulators from the source heater block assembly, then remove the repeller.
Figure 22  Disassembling the extractor EI ion source

Table 25  Parts list for extractor ion source (Figure 22)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Setscrews</td>
</tr>
<tr>
<td>2</td>
<td>Screws</td>
</tr>
<tr>
<td>3</td>
<td>Source body</td>
</tr>
<tr>
<td>4</td>
<td>Extractor lens</td>
</tr>
<tr>
<td>5</td>
<td>Extractor lens insulator</td>
</tr>
<tr>
<td>6</td>
<td>Filaments</td>
</tr>
</tbody>
</table>
### Table 25  Parts list for extractor ion source (Figure 22) (continued)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>Spring washer</td>
</tr>
<tr>
<td>8</td>
<td>Lens insulator</td>
</tr>
<tr>
<td>9</td>
<td>Entrance lens</td>
</tr>
<tr>
<td>10</td>
<td>Ion focus lens</td>
</tr>
<tr>
<td>11</td>
<td>Repeller insulator</td>
</tr>
<tr>
<td>12</td>
<td>Repeller</td>
</tr>
<tr>
<td>13</td>
<td>Flat washer</td>
</tr>
<tr>
<td>14</td>
<td>Belleville spring washer</td>
</tr>
<tr>
<td>15</td>
<td>Repeller nut</td>
</tr>
<tr>
<td>16</td>
<td>Source heater block assembly</td>
</tr>
<tr>
<td>17</td>
<td>Insulator</td>
</tr>
</tbody>
</table>
To Clean the EI Ion Source

Materials needed

- Abrasive paper (5061-5896)
- Alumina abrasive powder (8660-0791)
- Aluminum foil, clean
- Cloths, clean (05980-60051)
- Cotton swabs (5080-5400)
- Glass beakers, 500 mL
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Solvents
  - Acetone, reagent grade
  - Methanol, reagent grade
  - Methylene chloride, reagent grade
- Ultrasonic bath

Preparation

1. Disassemble the ion source. See “To Disassemble the Standard or Inert EI Ion Source” on page 131 or “To Disassemble the Extractor EI Ion Source” on page 134.

2. Collect the following parts to be cleaned for a standard or inert EI ion source: (See Figure 23 on page 139.)
   - Repeller
   - Interface socket
   - Source body
   - Drawout plate
   - Drawout cylinder
   - Ion focus lens
   - Entrance lens
3 Collect the following parts to be cleaned for an extractor EI ion source: (See Figure 23 on page 139.)
   • Repeller
   • Insulator
   • 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 23  Source parts to be cleaned
General Maintenance

Procedure

CAUTION

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.

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 a Standard or Inert EI Ion Source

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Hex ball driver, 1.5 mm (8710-1570)
- Hex ball driver, 2.0 mm (8710-1804)
- Wrench, open-end, 10 mm (8710-2353)

Procedure

1. Assemble the repeller assembly.
   a. Install the repeller block insert into the source heater block assembly.
      (See Figure 24 on page 143.)
   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.
   (See Figure 24 on page 143.)

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 24  Assembling the standard or inert EI ion source

Table 26  Parts list for the standard or inert EI ion source (Figure 24)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Set screw</td>
</tr>
<tr>
<td>2</td>
<td>Set screw</td>
</tr>
<tr>
<td>3</td>
<td>Interface socket</td>
</tr>
</tbody>
</table>
### General Maintenance

**Table 26** Parts list for the standard or inert EI ion source *(Figure 24)* (continued)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>Source body</td>
</tr>
<tr>
<td>5</td>
<td>Drawout cylinder</td>
</tr>
<tr>
<td>6</td>
<td>Drawout plate</td>
</tr>
<tr>
<td>7</td>
<td>4-turn filament</td>
</tr>
<tr>
<td>8</td>
<td>Spring washer</td>
</tr>
<tr>
<td>9</td>
<td>Lens insulator</td>
</tr>
<tr>
<td>10</td>
<td>Entrance lens</td>
</tr>
<tr>
<td>11</td>
<td>Ion focus lens</td>
</tr>
<tr>
<td>12</td>
<td>Repeller insulator</td>
</tr>
<tr>
<td>13</td>
<td>Repeller</td>
</tr>
<tr>
<td>14</td>
<td>Belleville spring washer</td>
</tr>
<tr>
<td>15</td>
<td>Flat washer</td>
</tr>
<tr>
<td>16</td>
<td>Repeller nut</td>
</tr>
<tr>
<td>17</td>
<td>Source heater block</td>
</tr>
<tr>
<td>18</td>
<td>Repeller block insert</td>
</tr>
</tbody>
</table>
To Assemble the Extractor EI Ion Source

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Hex ball driver, 1.5 mm (8710-1570)
- Hex ball driver, 2.0 mm (8710-1804)
- Wrench, open-end, 10 mm (8710-2353)

Procedure

1. Slide the ceramic washer into the source body.
2. Insert the extractor lens into the source body, flat side first (See Figure 25 on page 146).
3. Insert the entrance lens and ion focus lens into the insulator in the order shown (Figure 25 on page 146).
4. Slide the insulator containing the ion focus and entrance lens into the source body, with the ion focus lens against the extractor lens (See Figure 25 on page 146).
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. (See Figure 24 on page 143.)
   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.
5 General Maintenance

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 25  Assembling the extractor EI ion source

Table 27  Parts list for the extractor EI ion source (Figure 25)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Set screws</td>
</tr>
<tr>
<td>2</td>
<td>Screws</td>
</tr>
<tr>
<td>3</td>
<td>Source body</td>
</tr>
</tbody>
</table>
**Table 27**  Parts list for the extractor EI ion source (Figure 25) (continued)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>Extractor lens</td>
</tr>
<tr>
<td>5</td>
<td>Extractor lens insulator</td>
</tr>
<tr>
<td>6</td>
<td>Filaments</td>
</tr>
<tr>
<td>7</td>
<td>Spring washer</td>
</tr>
<tr>
<td>8</td>
<td>Lens insulator</td>
</tr>
<tr>
<td>9</td>
<td>Entrance lens</td>
</tr>
<tr>
<td>10</td>
<td>Ion focus lens</td>
</tr>
<tr>
<td>11</td>
<td>Repeller insulator</td>
</tr>
<tr>
<td>12</td>
<td>Repeller</td>
</tr>
<tr>
<td>13</td>
<td>Flat washer</td>
</tr>
<tr>
<td>14</td>
<td>Belleville spring washer</td>
</tr>
<tr>
<td>15</td>
<td>Repeller nut</td>
</tr>
<tr>
<td>16</td>
<td>Source heater block assembly</td>
</tr>
<tr>
<td>17</td>
<td>Repeller block insert</td>
</tr>
</tbody>
</table>
To Replace a Filament in an EI Ion Source

Materials needed

- Filament assembly (G2590-60053)
- 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 57.

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 78.

3. Remove the ion source. See “To Remove the EI Ion Source” on page 128.

4. Remove the gold plated screw and washer for the filament(s).
5 Secure the new filament(s) with the gold plated screw and washer.
6 After installing the filament, verify that it is not grounded to the source body.
7 Install the ion source. See “To Install the EI Ion Source” on page 150.
8 Close the analyzer chamber. See “To Close the Analyzer Chamber” on page 80.
9 Pump down the MSD. See “To Pump Down the MSD in EI Mode” on page 84.
10 Autotune the MSD. See “To Tune the MSD in EI Mode” on page 69.
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 Ion Source

Materials needed

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

Procedure

1. Slide the ion source into the source radiator.

2. Install and hand tighten the source thumbscrews. Do not overtighten the thumbscrews.

3. Connect the ion source wires as shown in Figure 14 on page 82.

4. Close the analyzer chamber. See “To Close the Analyzer Chamber” on page 80.
To Replace the Electron Multiplier Horn

Materials needed

- Electron multiplier horn (G3170-80103)
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)

Procedure

1. Vent the MSD. See “To Vent the MSD” on page 76.
2. Open the analyzer chamber. See “To Open the Analyzer Chamber” on page 78.
3. Open the retaining clip. Lift the arm of the clip up and then swing the clip away from the electron multiplier horn.

Figure 28  Electron multiplier horn
4 Remove the electron multiplier horn.
5 Install the new electron multiplier horn.
6 Close the retaining clip.

The signal pin on the horn must rest **on the outside** of the loop in the contact strip. **Do not** put the signal pin on the inside of the loop in the contact strip. Incorrect installation will result in poor sensitivity or no signal.

7 Close the analyzer chamber. See “To Close the Analyzer Chamber” on page 80.

8 Pump down the MSD. See “To Pump Down the MSD in EI Mode” on page 84.
This chapter describes maintenance procedures and requirements that are unique to 5977 Series MSDs equipped with the chemical ionization hardware.
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.

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 ion source, verify that the GC/MSD system is performing correctly. See “To Verify System Performance” on page 71.
- Make sure the reagent gas inlet line(s) are equipped with gas purifiers (not applicable for ammonia).
- Use extra-high purity reagent gases; 99.99% or better for methane and as pure as is available for other reagent gases.
To Install the CI/Extractor Interface Tip Seal

Materials needed

- Interface tip seal (G1999-60412)

The interface tip seal must be in place for the CI ion source and the Extraction source.

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 EI extractor ion source or CI ion source is installed. This tip seal must not be installed when a standard EI SST source or EI Inert source is installed.

2. Remove the CI/Extractor tip seal from the ion source storage box and place it over the end of the interface.
6 CI Maintenance

3 *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.

4 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 Remove the CI Ion 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 76.
2. Open the analyzer chamber. See “To Open the Analyzer Chamber” on page 78.

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

3. Disconnect the seven wires at the ion source. Use the pliers to pull on the metal connectors at the source. Do not bend the wires any more than necessary. See Table 28 on page 158 for color coding of the wires,
4 Trace the wires for the ion source heater and temperature sensor to the feedthrough board. Use the pliers to pull on the metal connectors to remove these four wires from the feedthrough board connections.

**CAUTION**
Pull on the connectors, not on the wires.

<table>
<thead>
<tr>
<th>Wire color</th>
<th>Connects to</th>
<th>Number of leads</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blue</td>
<td>Entrance lens</td>
<td>1</td>
</tr>
<tr>
<td>Orange</td>
<td>Ion focus</td>
<td>1</td>
</tr>
<tr>
<td>White</td>
<td>Filament 1 (top filament)</td>
<td>2</td>
</tr>
<tr>
<td>Red</td>
<td>Repeller</td>
<td>1</td>
</tr>
<tr>
<td>Black</td>
<td>Filament 2 (bottom filament)</td>
<td>2</td>
</tr>
</tbody>
</table>

5 Remove the thumbscrews that hold the ion source in place.
6 Pull the ion source out of the source radiator.

**WARNING** The analyzer operates at high temperatures. Do not touch any part until you are sure it is cool.
To Disassemble the CI Ion Source

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Hex ball driver, 1.5 mm (8710-1570)
- Hex ball driver, 2.0 mm (8710-1804)
- Wrench, open-end, 10 mm (8710-2353)

Procedure

1. Remove the ion source. See “To Remove the CI Ion Source” on page 157.
2. Remove the filaments. Refer to Figure 29 on page 160.
3. 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 and ceramic insulator and separate them.
5. Remove the setscrew for the lenses.
6. Pull the lens assembly out of the source body.
7. Remove the drawout cylinder and drawout plate from the source body.
8. Separate the ion focus lens, entrance lens and insulator.
Figure 29  Disassembling the CI ion source

Table 29  Parts list for the CI ion source (Figure 29)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Setscrew</td>
</tr>
<tr>
<td>2</td>
<td>Filament screw</td>
</tr>
<tr>
<td>3</td>
<td>CI interface tip seal</td>
</tr>
<tr>
<td>4</td>
<td>CI repeller insulator</td>
</tr>
<tr>
<td>5</td>
<td>CI lens insulator</td>
</tr>
<tr>
<td>6</td>
<td>CI drawout cylinder</td>
</tr>
<tr>
<td>7</td>
<td>CI drawout plate</td>
</tr>
<tr>
<td>Item number</td>
<td>Item description</td>
</tr>
<tr>
<td>-------------</td>
<td>-----------------------------------</td>
</tr>
<tr>
<td>8</td>
<td>CI ion source heater block assembly</td>
</tr>
<tr>
<td>9</td>
<td>Entrance lens</td>
</tr>
<tr>
<td>10</td>
<td>CI ion source body</td>
</tr>
<tr>
<td>11</td>
<td>CI ion focus lens</td>
</tr>
<tr>
<td>12</td>
<td>CI repeller</td>
</tr>
<tr>
<td>13</td>
<td>CI filament</td>
</tr>
<tr>
<td>14</td>
<td>Dummy filament</td>
</tr>
<tr>
<td>14</td>
<td>Tool box</td>
</tr>
</tbody>
</table>
To Clean the CI Ion Source

Materials needed

- Abrasive paper (5061-5896)
- Alumina abrasive powder (8660-0791)
- Aluminum foil, clean
- Cloths, clean (05980-60051)
- Cotton swabs (5080-5400)
- Glass beakers, 500 mL
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Solvents
  - Acetone, reagent grade
  - Methanol, reagent grade
  - Methylene chloride, reagent grade
- Ultrasonic bath

Preparation

1. Disassemble the ion source. See “To Disassemble the CI Ion Source” on page 159.

2. Collect the following parts to be cleaned for a CI ion source: (Figure 30 on page 163)
   - 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.
3 Clean the parts as described in “To Clean the EI Ion Source” on page 137.

**CAUTION**

If the 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 30**  CI ion source parts to be cleaned
To Assemble the CI Ion Source

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Hex ball driver, 1.5 mm (8710-1570)
- Hex ball driver, 2.0 mm (8710-1804)
- Wrench, open-end, 10 mm (8710-2353)

Procedure

1. Assemble the ion focus lens, entrance lens, and lens insulator.
2. Slide the drawout plate and the drawout cylinder into the source body (Figure 31 on page 165).
3. Slide the assembled lens parts into the source body.
4. Install the setscrew that holds the lenses in place.
5. Install the repeller, repeller insulators, washer, repeller nut and source heater block onto the source body.

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. Reinstall the filaments using the gold plated screws and spring washers.
**Figure 31**  Assembling the CI ion source

**Table 30**  Parts list for the CI ion source *(Figure 31)*

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Setscrew</td>
</tr>
<tr>
<td>2</td>
<td>Filament screw</td>
</tr>
<tr>
<td>3</td>
<td>CI interface tip seal</td>
</tr>
<tr>
<td>4</td>
<td>CI repeller insulator</td>
</tr>
<tr>
<td>5</td>
<td>CI lens insulator</td>
</tr>
<tr>
<td>6</td>
<td>CI drawout cylinder</td>
</tr>
</tbody>
</table>
### Table 30  Parts list for the CI ion source (Figure 31) (continued)

<table>
<thead>
<tr>
<th>Item number</th>
<th>Item description</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>CI drawout plate</td>
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<tr>
<td>8</td>
<td>CI ion source heater block assembly</td>
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<td>9</td>
<td>Entrance lens</td>
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<td>CI ion source body</td>
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<td>11</td>
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<td>13</td>
<td>CI filament</td>
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<td>14</td>
<td>Dummy filament</td>
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<td>15</td>
<td>Tool box</td>
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</tbody>
</table>
To Install the CI Ion Source

Materials needed

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

Procedure

1. Slide the ion source into the source radiator.

2. Install and hand tighten the source thumbscrews. Do not overtighten the thumbscrews.

3. Connect the ion source wires as shown in “To Close the Analyzer Chamber” on page 80.

4. Close the analyzer chamber. See “To Close the Analyzer Chamber” on page 80.
To Replace a Filament in a CI Ion Source

Materials needed
- Filament assembly (G2590-60053)
- 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 57.

**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 78.
3 Remove the ion source. See “To Remove the CI Ion Source” on page 157.
4 Remove the gold plated screw and washer for the filament.
5 Secure the new filament with the gold plated screw and washer.
6 After installing the filament, verify that it is not grounded to source body.
7 Install the ion source. See “To Install the CI Ion Source” on page 167.
8 Close the analyzer chamber. See “To Close the Analyzer Chamber” on page 80.
9 Pump down the MSD. See “To Pump Down the MSD in CI Mode” on page 98.
10 Perform a PCI autotune with methane. See “To Perform a PCI Autotune (Methane Only)” on page 111.
11 Select **Save Tune Parameters** from the **File** menu.