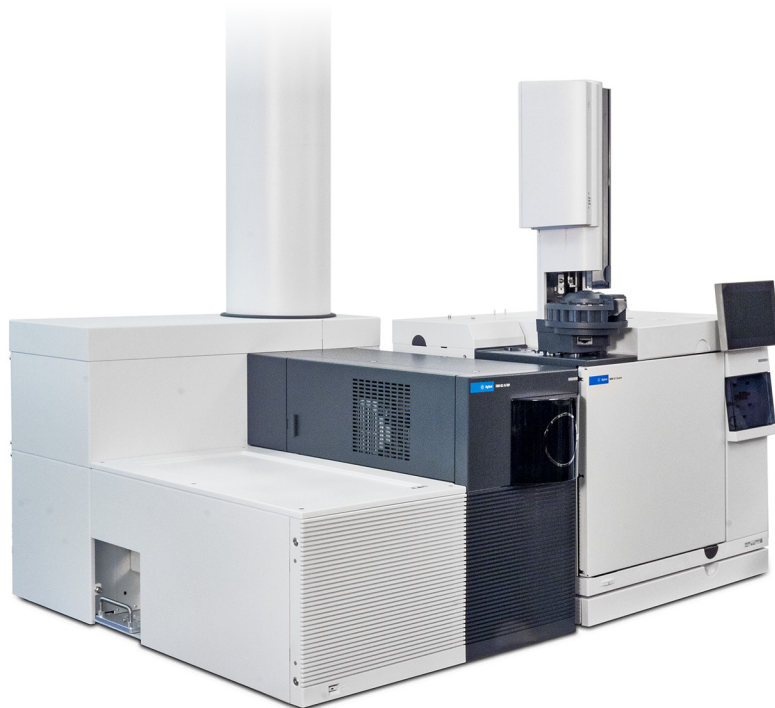




# Agilent 7250 Accurate-Mass Quadrupole Time-of-Flight GC/MS System

## Operating Manual



# Notices

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### 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 7250 Accurate-Mass Quadrupole Time-of-Flight GC/MS system.

## **1 “Introduction”**

Chapter 1 describes general information about the 7250 Q-TOF GC/MS, including a hardware description, and general safety warnings.

## **2 “Installing GC Columns”**

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

## **3 “Operating in EI Mode”**

Chapter 3 describes routine operations in EI mode such as setting temperatures, monitoring pressures, tuning, venting, and pumpdown.

## **4 “Operating in CI Mode”**

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

## **5 “General Maintenance”**

Chapter 5 describes general maintenance procedures for the 7250 Q-TOF GC/MS.

## **6 “CI Maintenance”**

Chapter 6 describes maintenance procedures unique to CI MS.

# Hardware User Information

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

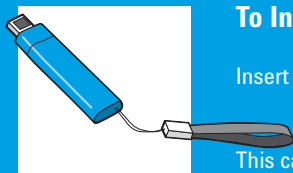
- Agilent GC and GC/MS User Manuals and Tools DVD set
- Agilent GC/MS Software Information and Manuals memory stick



## To Install Your Hardware Library

Insert Disk 1 into your DVD drive and follow the prompts.

This can be installed by anyone who has authority to copy information onto the receiving computer.



## To Install Your Software Library

Insert the memory stick into a USB port and follow the prompts.

This can be installed by anyone who has authority to copy information onto the receiving computer.

See the *Agilent 7200/7250 Accurate Mass Q-TOF GC/MS System Quick Start* document for more details on how to install this information on your computer.

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# 1

## Introduction

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This chapter provides general information about the Agilent 7250 Accurate-Mass Quadrupole Time-of-Flight (Q-TOF) GC/MS System, including a hardware description, and general safety warnings.

# Abbreviations Used

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

**Table 1** Abbreviations

Abbreviation	Definition
AC	Alternating current
ALS	Automatic liquid sampler
CC	Collision cell
CI	Chemical ionization
DC	Direct current
EI	Electron impact
EPC	Electronic pneumatic control
eV	Electron volt
GC	Gas chromatograph
GC/MS	Gas chromatograph mass spectrometer
id	Inside diameter
IDP	Isolated dry pump
LAN	Local Area Network
LE-EI	Low energy EI
<i>m/z</i>	Mass-to-charge ratio
MFC	Mass flow controller
MS	Mass spectrometer
OFN	Octafluoronaphthalene (sample)
PFTBA	Perfluorotributylamine (calibrant)
Q-TOF	Quadrupole time-of-flight
Quad	Quadrupole mass filter
RFI	Radio frequency interference
RFPA	Radio frequency power amplifier
TOF	Time-of-flight
Torr	Unit of pressure, 1 mm Hg
Turbo	Turbomolecular vacuum pump

## 1 Introduction

### The 7250 Accurate-Mass Quadrupole Time-of-Flight GC/MS System

# The 7250 Accurate-Mass Quadrupole Time-of-Flight GC/MS System

The 7250 Accurate-Mass Quadrupole Time-of-Flight (Q-TOF) GC/MS System is a standalone capillary GC detector for use with Agilent 8890 and 7890 Series gas chromatographs. The 7250 Q-TOF features:

- Three turbomolecular vacuum pumps
- Foreline pump
  - Rotary vane pump
  - Optional isolated dry pump (IDP)
- Independently MS-heated LE-EI ion source
- Independently MS-heated hyperbolic quadrupole mass filter, which can be heated to high temperatures, minimizing the contamination typical with low temperature analyses
- Single hexapole collision cell
- Ion-focusing slicer
- Vacuum-insulated flight tube with dual-stage ion mirror
- Fast electronics, allowing fast sampling rates
- Analog-to-digital detector
- Independently GC-heated GC/MS interface

## Physical description

The 7250 Q-TOF GC/MS is approximately 53.5 cm high, 69 cm wide, and 92 cm deep excluding the flight tube. The flight tube extends 136 cm over the top of the instrument.

The weight of the instrument mainframe is 159 kg. The weight of the attached foreline (roughing) pump is 25 kg for the DS202 rotary vane pump model, and 45 kg for the IDP model.

The basic components of the instrument are the:

- Frame/cover assemblies
- Vacuum system
- GC/MS interface

## 1 Introduction

### Vacuum gauge

- Ion source
- Flight tube electronics
- Quadrupole mass filter
- Collision cell
- Detector
- Analyzer

## Vacuum gauge

The 7250 Q-TOF GC/MS is equipped with three ion vacuum gauges:

- Vacuum manifold chamber
- TOF vacuum manifold chamber
- Turbomolecular vacuum pumps exhaust

The MassHunter Workstation can be used to read the pressure (high vacuum) in the vacuum manifold, at the turbomolecular vacuum pump discharge, and the flight tube.

## Ionization modes

The 7250 Accurate-Mass Q-TOF GC/MS can operate in EI mode using the standard LE-EI ion source or in CI mode using the optional CI MS system. In EI mode, the Q-TOF can operate in standard or low-energy modes.

# Hardware Description

**Figure 1** is an overview of a typical Agilent 7250 Accurate-Mass Q-TOF GC/MS system.

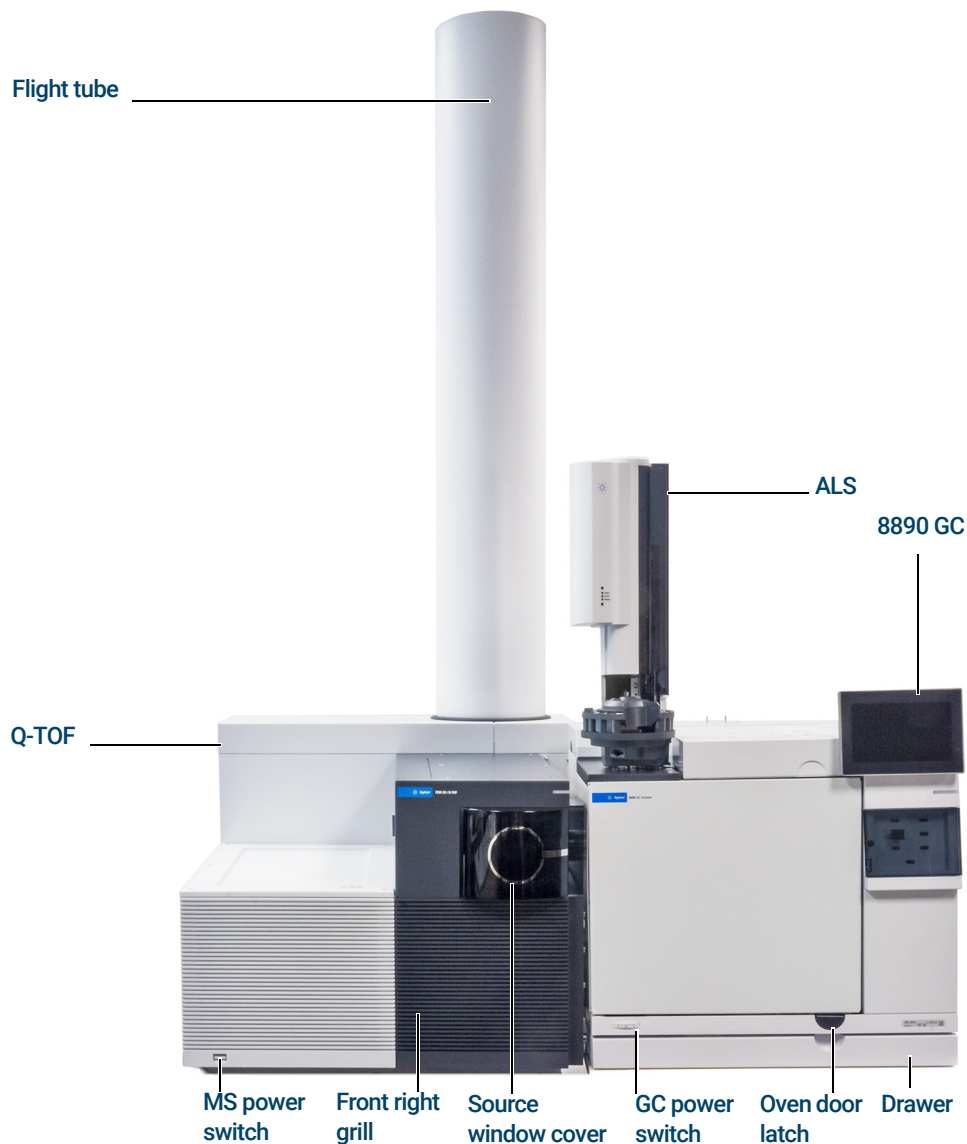


Figure 1. 7250 Accurate-Mass Q-TOF and 8890 GC/MS system

## Important Safety Warnings

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

### Many internal parts of these instruments carry dangerous voltages

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

- The wiring between the instrument power cord and the AC power supply
- The AC power supply itself
- The wiring from the AC power supply to the power switch

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

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

#### WARNING

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

#### WARNING

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

### Electrostatic discharge is a threat to instrument electronics

The printed circuit boards in these instruments 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.



## 1 Introduction

### Precautions to take to prevent an explosion

## Precautions to take to prevent an explosion

### **WARNING**

The use of hydrogen gas is specifically prohibited with this product.

### **WARNING**

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

You **MUST** leave the collision cell chamber top plate shipping brackets fastened. Do not remove the shipping brackets from the top plate for normal operation; they secure the top plate in the event of an explosion.

### **WARNING**

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

## Many parts are dangerously hot

Many parts of these instruments operate at temperatures high enough to cause serious burns. These parts include, but are not limited to the:

- Inlet
- Oven and its contents
- Valve box
- Column nuts attaching the column to an inlet, detector, or GC/MS interface
- Foreline pump
- GC/MS interface
- Quadrupole
- Ion source

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 cooldown cycles, the GC emits hot exhaust that can cause burns.

## 1 Introduction

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

### **WARNING**

The foreline pump can cause burns if touched when operating.

### **WARNING**

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

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

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

### **WARNING**


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

# Safety and Regulatory Certifications

The 7250 Q-TOF GC/MS conforms to the following safety standards:

- Canadian Standards Association (CSA): CAN/CSA-C22.2 No. 61010-1-04
- CSA/Nationally Recognized Test Laboratory (NRTL): UL 61010-1
- International Electrotechnical Commission (IEC): 61010-1
- EuroNorm (EN): 61010-1

The 7250 Q-TOF GC/MS conforms to the following regulations on Electromagnetic Compatibility (EMC) and Radio Frequency Interference (RFI):

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

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



The 7250 Q-TOF GC/MS is designed and manufactured under a quality system registered to ISO 9001.

The 7250 Q-TOF GC/MS is RoHS compliant.

## South Korean Class A EMC Declaration

A 급 기기 ( 업무용 방송통신기자재 )

This equipment is Class A suitable for professional use and is for use in electromagnetic environments outside of the home.

이 기기는 업무용 (A 급) 전자파적합기기로서 판 매자 또는 사용자는 이 점을 주의 하 시기 바라 며 , 가정외의 지역에서 사용하는 것을 목 적으 로 합 니 다 .

## Information

The Agilent Technologies 7250 Accurate-Mass Q-TOF GC/MS meets the following IEC (International Electrotechnical Commission) classifications: Equipment Class I, Laboratory Equipment, Installation Category II, and Pollution Degree 2.

## 1 Introduction

### Symbols

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 MS 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.



or



Indicates electrostatic discharge hazard.



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



## Electromagnetic compatibility

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

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

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

- Relocate the radio or antenna.
- Move the device away from the radio or television.
- Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
- Ensure that all peripheral devices are also certified.
- Ensure that appropriate cables are used to connect the device to peripheral equipment.
- Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.

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

## Sound emission declaration

### **Sound pressure**

Sound pressure  $L_p < 70$  dB according to EN 27779:1991 and EN ISO 3744:1995.

### **Schalldruckpegel**

Schalldruckpegel  $LP < 70$  dB nach EN 27779:1991 und EN ISO 3744:1995.

## Intended Use

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

## Cleaning/Recycling the Product

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

## Accidental Liquid Spillage

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

## Moving or Storing the MS

The best way to keep your MS functioning properly is to keep it pumped down and hot, with carrier gas flow. If you plan to move or store your MS, a few additional precautions are required. The MS must remain upright at all times; this requires special caution when moving. The MS should not be left vented to atmosphere for long periods. For more information, see "To Move or Store the MS" in the *Agilent 7250 Q-TOF GC/MS Troubleshooting and Maintenance Manual*.

## 2

# Installing GC Columns

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To Condition a Capillary Column 29

To Install a Capillary Column in the GC/MS Interface 30

To Install the GC/MS Interface Tip Seal 33

Before you can operate your GC/MS system, you must select, install, and condition a GC column. This chapter shows you how to install and condition a column. This chapter applies to Agilent 8890 and 7890 GCs.

# Columns

The material in this chapter applies to Agilent 8890 and 7890 GCs.

Many types of GC columns can be used with the MS, but there are some restrictions. During tuning or data acquisition the rate of column flow into the MS should not exceed the maximum recommended flow. Therefore, there are limits to column length and flow. Exceeding recommended flow will result in degradation of mass spectral and sensitivity performance.

Remember that column flows vary greatly with oven temperature. See **“To Calibrate a 7890 GC Column”** on page 48 for instructions on how to measure actual flow in your column. Use the flow calculator in the Agilent Instrument Utilities software, and **Table 2** to determine an acceptable column flow. For expected column outlet flow pressures, use the values shown in **Table 3**, “Influence of carrier and collision cell gas flows on vacuum,” on page 42.

**Table 2 Gas flows**

Feature	Gas flows
High-vacuum pump 1	Split-flow turbo
High-vacuum pump 2	Turbo
High-vacuum pump 3	Turbo
Optimal carrier gas flow, mL/min	1.0 to 1.5
Collision cell Nitrogen gas flow, mL/min	1
Collision cell Helium gas flow, mL/min	4
Maximum recommended gas flow, mL/min*	2.0
Maximum gas flow, mL/min <sup>†</sup>	2.4
Maximum column id	0.32 mm (30 m length)

\* Total gas flow into the MS = column flow + collision cell gas flow.

† Expect degradation of spectral performance and sensitivity.



## Conditioning columns

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

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

Column bleed is most common in new or poorly cross-linked columns. It is much worse if there are traces of oxygen in the carrier gas when the column is heated. To minimize column bleed, all capillary columns should be conditioned before they are installed in the GC/MS interface. See **“To Condition a Capillary Column”** on page 29.

## 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 procedure for the 7250 Q-TOF GC/MS is different from that for other MSs. Using the procedure from another instrument may not work and may damage the column or the MS.
- You can remove old ferrules from column nuts with an ordinary pushpin.
- 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/MS interface.

### **WARNING**

**The use of hydrogen gas is specifically prohibited with this product.**

### **WARNING**

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

## 2 Installing GC Columns

### To Install a Capillary Column in a Split/Splitless Inlet

# To Install a Capillary Column in a Split/Splitless Inlet

#### Materials needed

- Gloves, clean, lint-free
  - 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)
- Self-tightening column nut (5190-6194)
- Alternate: Standard Inlet column nut (5181-8830, for use with Vespel ferrules)
- Ferrules, Vespel
  - 0.27 mm id, for 0.10 mm id columns (5062-3518)
  - 0.37 mm id, for 0.20 mm id columns (5062-3516)
  - 0.40 mm id, for 0.25 mm id columns (5181-3323)
  - 0.5 mm id, for 0.32 mm id columns (5062-3514)
  - 0.8 mm id, for 0.53 mm id columns (5062-3512)
- Magnifying loupe
- Septum (may be old, used inlet septum)

To install columns in other types of inlets, refer to your GC user information.



#### Procedure

#### **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.

- 1 Cool the oven to room temperature.

#### **WARNING**

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

## 2 Installing GC Columns

### To Install a Capillary Column in a Split/Splitless Inlet

#### CAUTION

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

- 2 Wearing clean gloves, slide a septum, column nut, and conditioned ferrule onto the free end of the column. **Figure 2** shows a traditional inlet column nut but the nut is similarly positioned for a self-tightening column nut. The tapered end of the ferrule should point away from the column nut for a column attachment to an inlet.

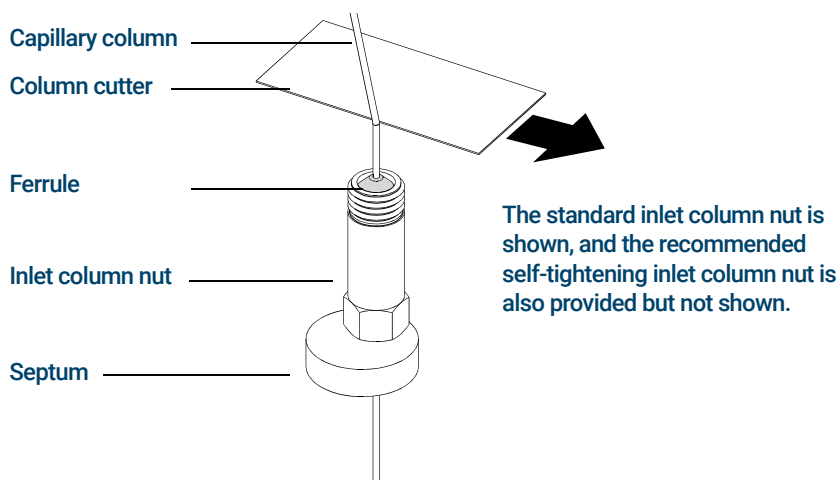


Figure 2. Preparing a capillary column for installation

- 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.
- 7 Position the septum under the column nut so that the column extends 4 to 6 mm past the end of the ferrule. (See **Figure 3**, "Installing a capillary column for a split/splitless inlet," on page 28.)
- 8 Insert the column in the inlet.
- 9 Slide the nut up the column to the inlet base and finger-tighten the nut.

## 2 Installing GC Columns

### To Install a Capillary Column in a Split/Splitless Inlet

- 10 Adjust the column position so the septum is even with the bottom of the column nut.

#### CAUTION

Never use a wrench to tighten a self-tightening nut. This may result in damage to the nut or instrument.

- 11 For the self-tightening column nut, finger-tighten until it begins to grip the column then turn an additional 30 to 60 degrees until tight.

For the standard column nut, tighten the nut an additional 1/4 to 1/2 turn. The column should not slide with a gentle tug.

- 12 Start carrier gas flow.

- 13 Verify flow by submerging the free end of the column in isopropanol. Look for bubbles.

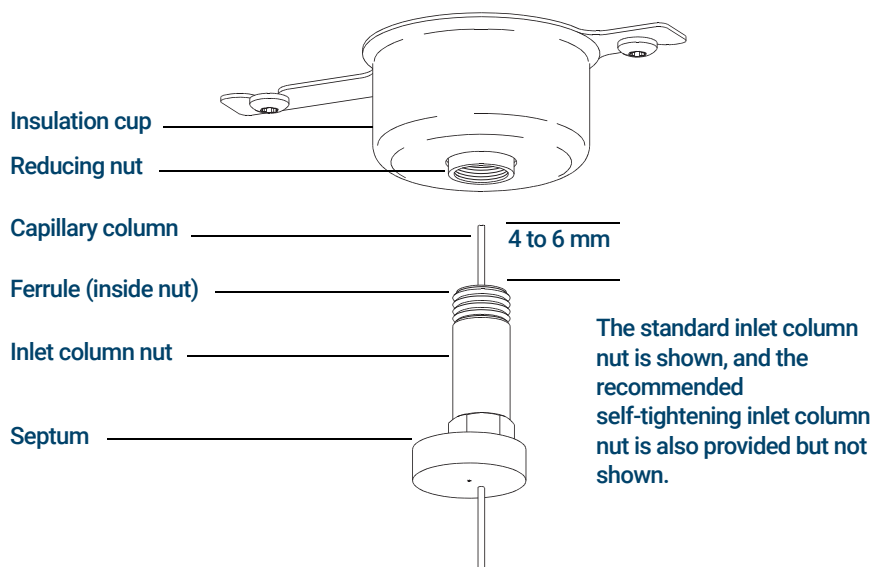


Figure 3. Installing a capillary column for a split/splitless inlet

# 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

The use of hydrogen gas is specifically prohibited with this product.

### 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 26.)
- 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 Install the column in the GC/MS interface. (See **“To Install a Capillary Column in the GC/MS Interface”** on page 30.)

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.

## 2 Installing GC Columns

### To Install a Capillary Column in the GC/MS Interface

# To Install a Capillary Column in the GC/MS Interface

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

#### Materials needed

- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Flashlight
- Magnifying loupe
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Septum (may be old, used inlet septum)
- Safety glasses

#### Self-tightening column nut materials (recommended)

- Self-tightening GC/MS interface column nut (5190-5233)
- Ferrules, Vespel for self-tightening column nut
  - 0.4 mm VG 0.1-0.25 col 10 pk (5181-3323)
  - 0.5 mm VG 0.32 col 10 pk (5062-3514)

#### Standard column nut materials

- Standard GC/MS interface column nut (05988-20066)
- Ferrules, Vespel for standard column nut
  - 0.4 mm VG 0.1-0.25 col 10 pk (5181-3323)
  - 0.5 mm VG 0.32 col 10 pk (5062-3514)

## 2 Installing GC Columns

To Install a Capillary Column in the GC/MS Interface



### Procedure

#### CAUTION

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

- 1 Condition the column. (See **"To Condition a Capillary Column"** on page 29.)

#### 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 Vent the MS. See **"To Vent the MS"** on page 57.
- 3 Open the analyzer chamber. (See **"To Open the Analyzer Cover for Access to the Analyzer Sideplate"** on page 106.)

#### WARNING

Use an antistatic wrist strap and take other antistatic precautions before touching analyzer components.

#### WARNING

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

- 4 Remove the interface tip seal, spring, and knurled tip seal retainer from the GC/MS interface. (See **Figure 5**, "GC/MS interface tip seal," on page 34.)
- 5 Slide the self-tightening or standard column nut and conditioned ferrule onto the free end of the GC column. The tapered end of the ferrule must point towards the nut.
- 6 Slide the column into the GC/MS interface.
- 7 Lightly thread the column nut onto the GC/MS interface.
- 8 Use the column cutter to score the column 2 cm from the end.

## 2 Installing GC Columns

### To Install a Capillary Column in the GC/MS Interface

#### CAUTION

**Do not break the column off inside the vacuum manifold. Pieces of column could fall or be pulled into the turbo pump and damage it.**

- 9 While holding the column against the column cutter with your thumb, break the column against the edge of the column cutter.
- 10 Inspect the end for jagged edges or burrs. If the break is not clean and even, repeat steps 7 and 8.
- 11 Adjust the column so it extends 4 to 5 mm past the end of the transfer line.

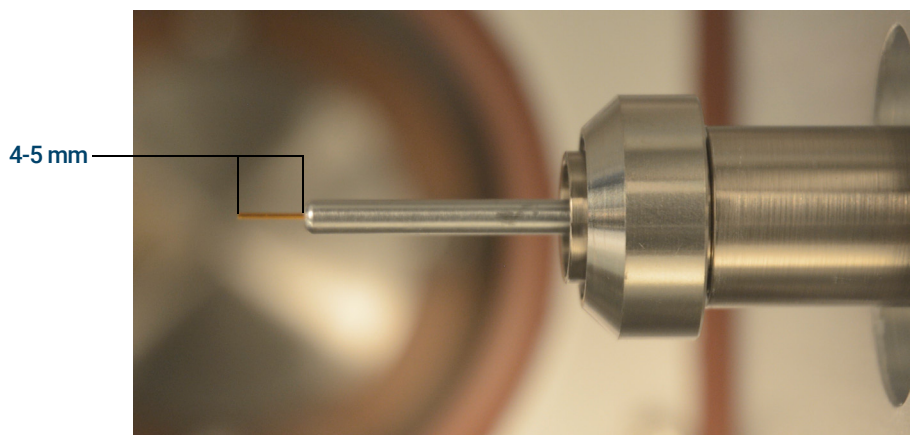


Figure 4. Installing a capillary column in the GC/MS interface

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

For the self-tightening column nut, finger tighten until it begins to grip the column, then turn an additional 30 to 60 degrees until tight.

For the standard column nut, tighten the nut an additional 1/4 to 1/2 turn. The column should not slide with a gentle tug.

- 13 Check the GC oven to ensure that the column does not touch the oven walls.
- 14 Install the interface tip seal, spring, and knurled tip seal retainer on the GC/MS interface. (See **"To Install the GC/MS Interface Tip Seal"** on page 33.)
- 15 Check the nut's tightness after one or two heat cycles; retighten as appropriate.



## 2 Installing GC Columns

### To Install the GC/MS Interface Tip Seal

# To Install the GC/MS Interface Tip Seal

#### Materials needed

- Interface tip seal (G3870-20542)
- Tip seal spring (G7005-20024)
- Knurled tip seal retainer (G3870-20547)
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)

The interface tip seal must be in place for the LE-EI ion source.

#### CAUTION

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

#### Procedure

- 1 Remove the interface tip seal, spring, and knurled tip seal retainer from the ion source storage box. In this order, slide the spring, tip seal, and knurled tip seal retainer over the column sleeve.
- 2 Thread the knurled tip seal retainer into the tip seal holder finger tight.

## 2 Installing GC Columns

### To Install the GC/MS Interface Tip Seal

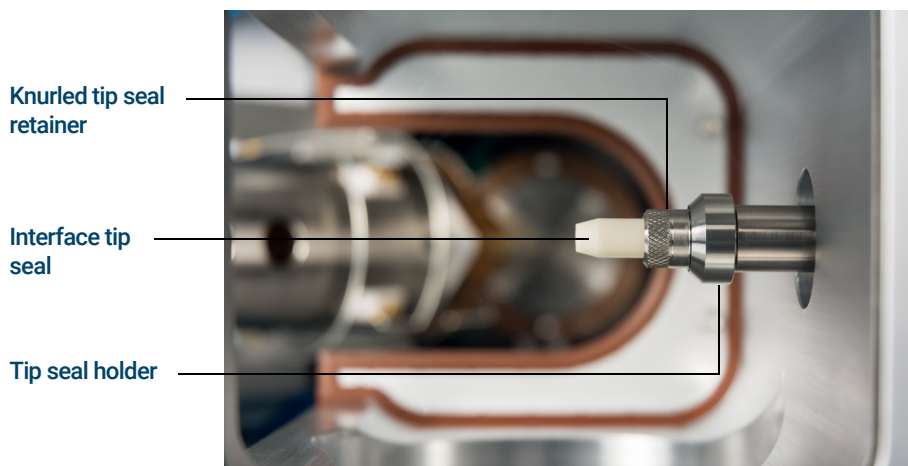


Figure 5. GC/MS interface tip seal

### 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.**

- 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.

- 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.

# 3

## Operating in EI Mode

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This chapter explains how to perform some routine operating procedures for the 7250 Accurate-Mass Q-TOF GC/MS in EI mode.

## Operating the MS from the Data System

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

### CAUTION

**The software and firmware are revised periodically. If this chapter's procedure steps do not match your MassHunter Workstation software, refer to the manuals and online help supplied with the software for more information.**

## The GC/MS Interface

The GC/MS interface is a heated conduit into the MS for the capillary column. (See **Figure 6**, "The GC/MS interface," on page 38.) The interface is bolted onto the right side of the front analyzer chamber and has an O-ring seal. It has a protective insulated cover that should be left in place.

One end of the GC/MS 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 4 to 5 mm of the capillary column extends past the end of the guide tube and into the ionization chamber.

The GC/MS interface is heated by an electric cartridge heater. Normally, the heater is powered and controlled by the Thermal Aux #2 heated zone of the GC. The interface temperature can be set from the MassHunter Workstation or from the gas chromatograph keypad. A sensor (thermocouple) in the interface monitors the temperature. The GC/MS interface generally operates in the 250 °C 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.

### See also

**"To Install a Capillary Column in the GC/MS Interface"** on page 30.

#### **WARNING**

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

### 3 Operating in EI Mode

See also

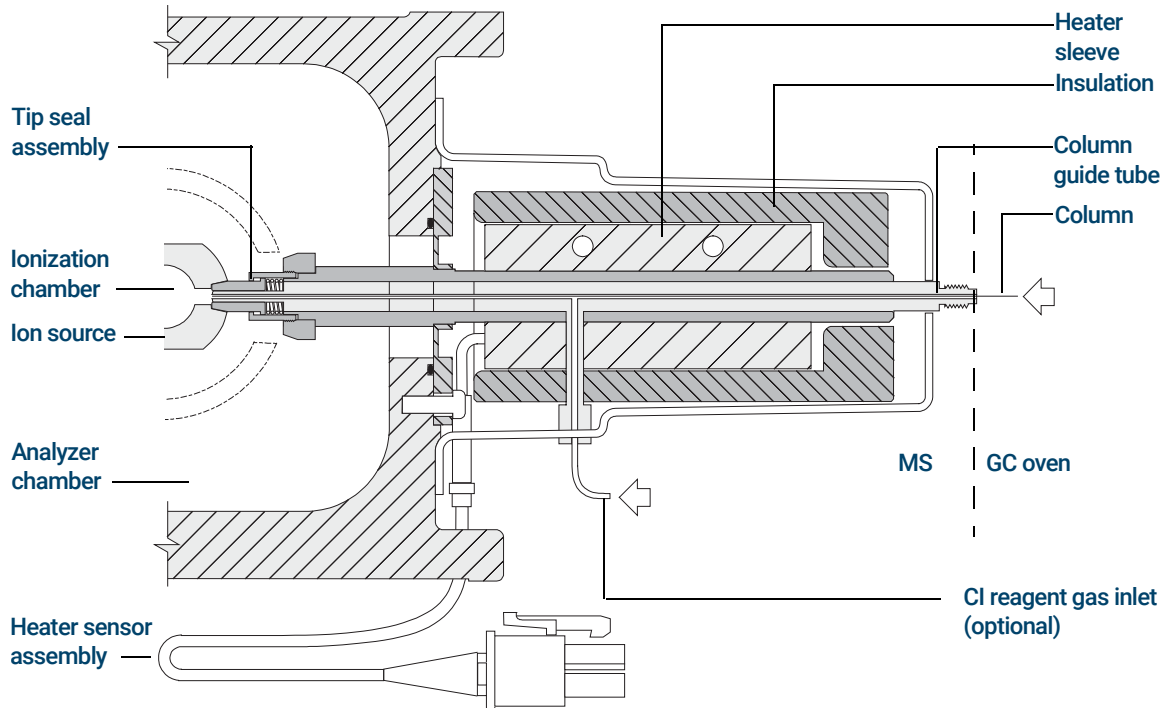


Figure 6. The GC/MS interface

## Before You Turn On the MS

Verify the following *before* you turn on or attempt to operate the MS.

- All vacuum seals and fittings must be in place and fastened correctly. The analyzer plate thumbscrew should be unfastened, unless hazardous carrier gases are being used, in which case the thumbscrew should be engaged and very lightly tightened.

### CAUTION

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

- The MS is connected to a grounded power source.
- The GC/MS interface extends into the GC oven.
- A conditioned capillary column is installed in the GC inlet and in the GC/MS interface.
- The GC is on, but the heated zones for the GC/MS interface, the GC inlet, and the oven are off.
- Carrier gas of at least 99.9995% purity is plumbed to the GC with the recommended traps.
- The use of hydrogen gas is specifically prohibited with this product.
- The foreline pump exhaust is properly vented.

### WARNING

**The exhaust from the foreline pump contains solvents and the chemicals you are analyzing. The standard foreline pump, also exhausts traces of pump oil. If you are using toxic solvents or analyzing toxic chemicals, attach a hose (11-mm id) to the oil mist filter to take the foreline pump exhaust outside or to a fume (exhaust) hood. Comply with local regulations. The oil mist filter supplied with the standard pump stops only pump oil. It does not trap or filter out toxic chemicals.**

### WARNING

**The use of hydrogen gas is specifically prohibited with this product.**

## Pumping Down

Pumping down is only required if the analyzer was vented.

The data system helps you pump down the MS. The process is mostly automated. When you turn on the main power switch (while pressing on the analyzer sideplate), the MS 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/MS interface heater. The MS will shut down if it cannot pump down correctly.

Using the MS monitors, the data system can display:

- Motor speed for each MS turbo pump
- Analyzer chamber pressure (vacuum)
- Quadrupole and ion source temperatures

## Controlling Temperatures

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

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

## Controlling Column Flow

Carrier gas flow is controlled by column 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.



### 3 Operating in EI Mode

#### Controlling Collision Cell Flow

The MS 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 MS. With this time measurement, you can calculate the column flow. (See **“To Calibrate a 7890 GC Column”** on page 48.)

## Controlling Collision Cell Flow

The collision cell gas flow rate is controlled by an EPC module located in the GC. The collision cell gas flow consists of nitrogen and helium. The gas pressure at the EPC outlets control the flow of the gases. This pressure is controlled by the MassHunter Data Acquisition Workstation or directly at the GC. (See **“To Set the Collision Cell Gas Flow Rate”** on page 51.)

## Venting the MS

A quick vent system fully automates the venting process. Once initiated, it turns off the ion gauge filaments and the turbo pumps. It then delivers dry nitrogen gas to the source region and the TOF manifold to break vacuum in the analyzer. When the turbo pumps stop rotating it turns off the foreline pump. (See **“To Vent the MS”** on page 57.)

### WARNING

The use of hydrogen gas is specifically prohibited with this product.

### CAUTION

Never vent the MS by allowing air in through either end of the foreline hose. Use the proper venting procedure, or remove the column nut and column.

Do not exceed the maximum recommended total gas flow. (See **Table 2**, “Gas flows,” on page 24.)

### 3 Operating in EI Mode

Typical Vacuum Pressures in EI Mode

## Typical Vacuum Pressures in EI Mode

The largest influences on operating pressure in EI mode are the carrier gas (column) and collision cell gas flows. **Table 3** lists typical pressures for various helium and nitrogen collision cell gas flows. These pressures are approximate and will vary from instrument to instrument by as much as 30%.

**Table 3** Influence of carrier and collision cell gas flows on vacuum

Column flow (mL/min)	CC Gas On N <sub>2</sub> = 1 mL/min He = 4 mL/min			CC Gas Off		
	Rough Vac (mTorr)	Quad Vac (Torr)	TOF Vac (Torr)	Rough Vac (mTorr)	Quad Vac (Torr)	TOF Vac (Torr)
0.7	1.25 E+02	3.50 E-05	2.61 E-07	2.70 E+01	5.78 E-07	1.58 E-07
1	1.30 E+02	3.49 E-05	2.61 E-07	3.61 E+01	6.21 E-07	1.58 E-07
1.2	1.34 E+02	3.49 E-05	2.61 E-07	4.13 E+01	6.55 E-07	1.58 E-07
2	1.48 E+02	3.51 E-05	2.61 E-07	6.18 E+01	8.09 E-07	1.59 E-07
3	1.63 E+02	3.52 E-05	2.61 E-07	8.45 E+01	9.99 E-07	1.60 E-07

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

## To Set Monitors for Temperature and Vacuum Status

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

### Procedure

- 1 Select **Instrument > Edit Monitors** to display the **Select Monitors** dialog box. (See [Figure 7.](#))

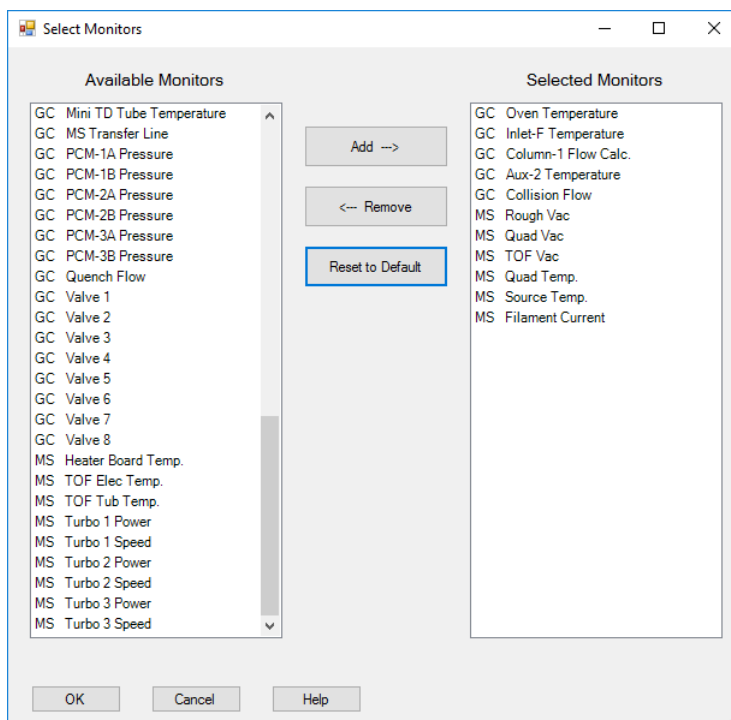


Figure 7. Select Monitors dialog box

- 2 In the **Available Monitors** column, select a monitor and click the **Add** button to move the selected monitor to the **Selected Monitors** column.

### 3 Operating in EI Mode

#### To Set Monitors for Temperature and Vacuum Status

- 3 Select any other monitors you want and add them to the **Selected Monitors** column.
- 4 Click **OK**. The new monitors will be stacked on top of each other in the lower right corner of the **Instrument Control** window.
- 5 Select **Window > Arrange Monitors**, or click and drag each monitor to the desired position. See **Figure 8** for one way of arranging the monitors.

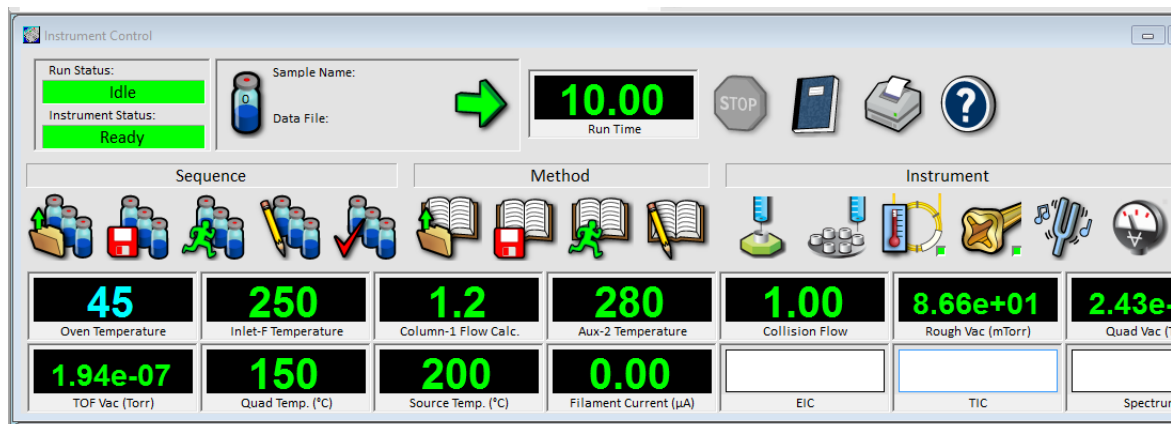


Figure 8. Arranging monitors

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


### 3 Operating in EI Mode

To Set the MS Analyzer Temperature

## To Set the MS Analyzer Temperature

Setpoints for the MS ion source and quad temperatures are stored in the current tune file. When a method is loaded, the setpoints in the tune file associated with that method are downloaded automatically.

### Procedure

- 1 In **Instrument Control** panel, select the **MS Tune** icon  to display the **Tune** dialog box. Select the **Manual Tune** tab then select the **Ion Source** tab to display the ion source parameters. (See **Figure 9**, "Setting temperatures," on page 46.)
- 2 Enter the temperature setpoint in the **Source Temp** field. See **Table 4** for recommended setpoints.
- 3 Select the **Quadrupole** tab to display the MS parameters.
- 4 Enter the temperature setpoint in the **Quad Temp** field. See **Table 4** for recommended setpoints.
- 5 Select the **Files and Reports** tab then click the **Save** button to save the tune file with these changes.

**Table 4** Recommended temperature settings

Zone	EI operation
MS Source	230 °C default. High limit 350 °C. Application dependent.
MS Quad	150 °C

### 3 Operating in EI Mode To Set the MS Analyzer Temperature

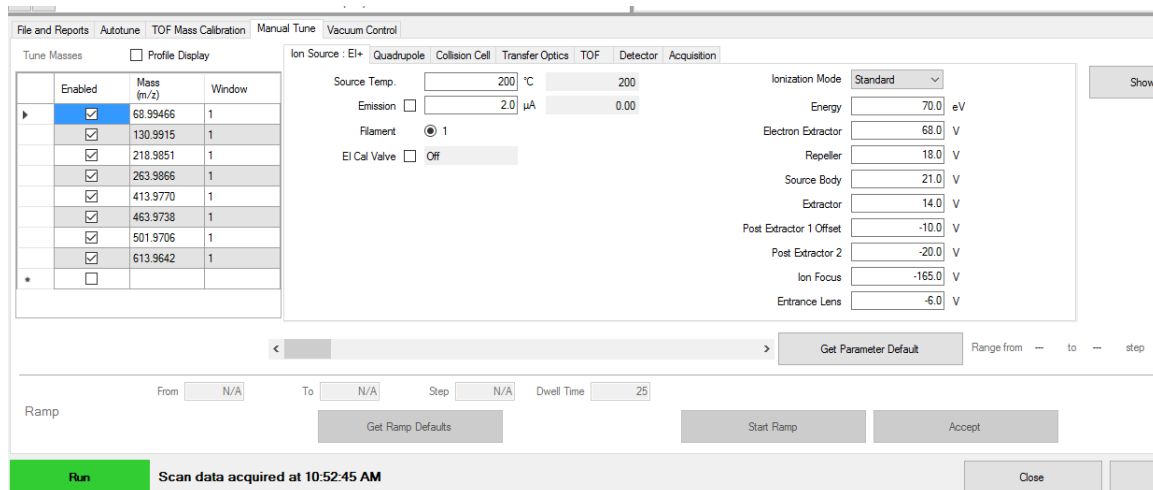


Figure 9. Setting temperatures

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

The software will not allow you to exceed 200 °C for the quadrupole, or 350 °C for the ion source.

### 3 Operating in EI Mode

To Set the GC/MS Interface Temperature from the MassHunter Workstation

## To Set the GC/MS Interface Temperature from the MassHunter Workstation

You can also use the **GC Control** panel to perform this task.

### Procedure

- 1 Select **Instrument > GC Parameters** from the **Instrument Control** panel.
- 2 Click **Aux Heaters** in the navigation pane to edit the interface temperature. The example in **Figure 10** has the GC/MS interface temperature configured as **Thermal Aux 2**.

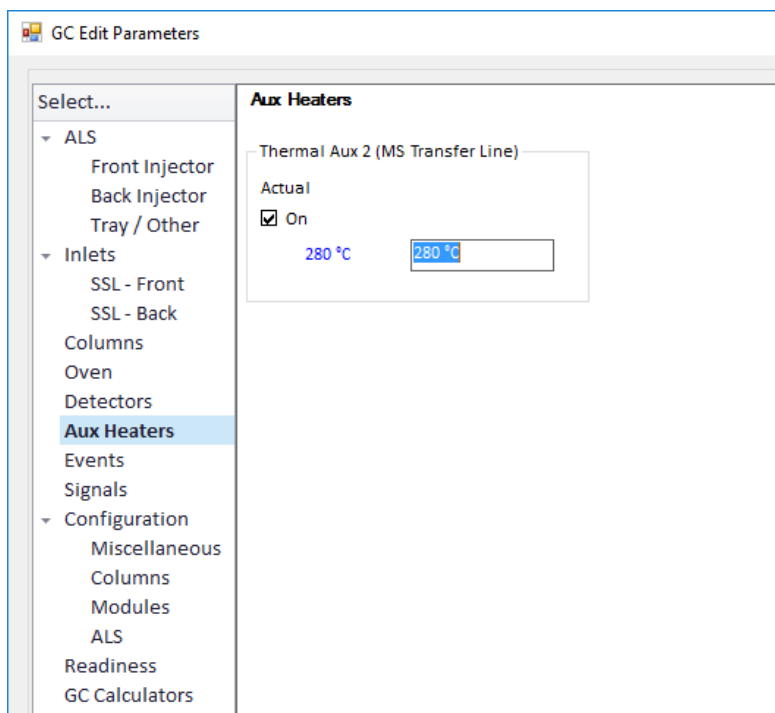


Figure 10. Setting the interface temperature

### 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.

### 3 Operating in EI Mode To Calibrate a 7890 GC Column

#### CAUTION


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

- 3 Click the heater **On** check box, and enter the setpoint in the **Value °C** column. The typical setpoint is 280 °C. The limits are 0 °C to 400 °C. A setpoint below ambient temperature turns off the interface heater.
- 4 Click **Apply** to download setpoints or click **OK** to download setpoints and close the window.
- 5 To make the new settings part of the method, select **Save** from the **Method** menu.

## To Calibrate a 7890 GC Column

Capillary columns must be calibrated prior to use with the MS. This procedure describes the process for an Agilent 7890 GC. For other GCs, see the documentation and online help supplied with the GC.

### Procedure

- 1 Set data acquisition for splitless manual injection, and set up a real time plot to monitor  $m/z$  28.
- 2 Press **[Prep Run]** on the GC keypad.
- 3 Inject 1  $\mu$ L of air into the GC inlet, and press **[Start Run]**.
- 4 Wait until a peak elutes at  $m/z$  28. Note the retention time.
- 5 In the **Instrument Control** panel, select .
- 6 Click **Configuration > Columns** from the navigation pane.
- 7 Select the column to be calibrated, and click **Calibrate**.
- 8 Click **Calc Length**.
- 9 In the **Calculate Column Length** dialog, enter the recorded retention time in the **Holdup Time of an Unretained Peak** field. (See **Figure 11**, "Calculate Column Length dialog," on page 49.)



### 3 Operating in EI Mode

#### To Calibrate a 7890 GC Column

- 10 Verify that the other 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.

Calculate Column Length ✕

GC Conditions

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

Temperature:

Pressure into column:

Pressure out of column:

Vacuum

Gas type:  ▾

Holdup Time of an Unretained Peak:

	Current	Calculated
▶ Length	30 m	30 m
Diameter	250 μm	250 μm
Holdup	1.2555 min	1.2555 min


Figure 11. Calculate Column Length dialog

- 11 When the new column length appears, click **OK** to save the changes.
- 12 Click **OK** on the **Calibrate Columns** screen to save the calibration.

### 3 Operating in EI Mode

To Configure the Collision Cell Gas

## To Configure the Collision Cell Gas

- 1 From the MassHunter Data Acquisition Workstation **Instrument Control** panel, select .
- 2 From the Navigation pane, click **Configuration > Modules** to display the screen. (See **Figure 12.**)

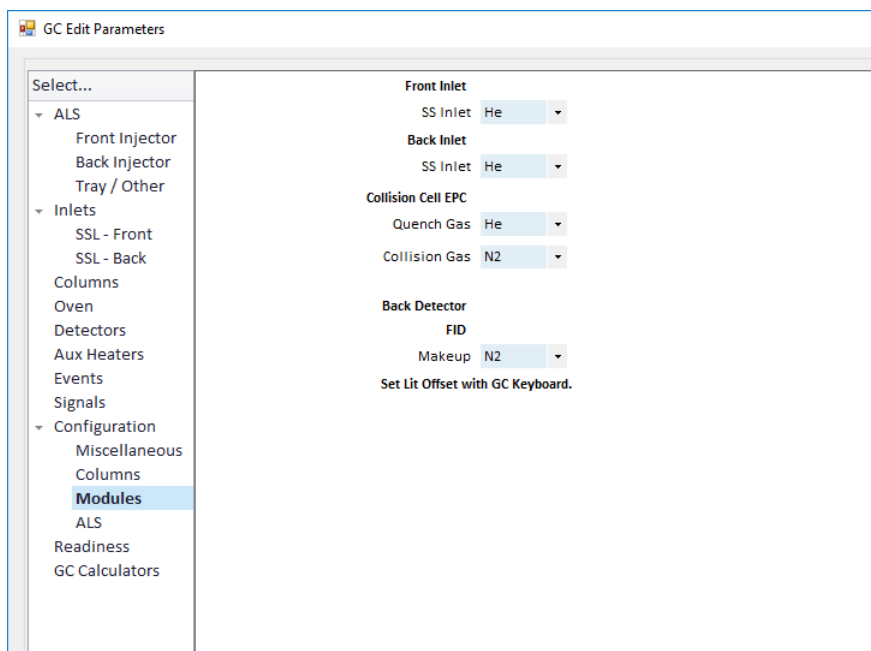


Figure 12. Configure the collision cell gas

- 3 From the **Collision Cell EPC** section, select **He** as the **Quench Gas** and **N2** as the **Collision Gas** from the drop-down menus.
- 4 Click **OK** to save the configuration.

### 3 Operating in EI Mode

To Set the Collision Cell Gas Flow Rate

## To Set the Collision Cell Gas Flow Rate

- 1 From the MassHunter Data Acquisition Workstation **Instrument Control** panel, select **Instrument > GC Parameters**.
- 2 From the Navigation pane, click **Columns** to display control parameter entry screen for column and Aux flow modules. (See **Figure 13** Click **Collision Cell PC** in the **Selection** list.
- 3 Select **He Quench Gas** to allow the He quench gas flow.
- 4 Enter the required gas flow rates in the appropriate fields.
- 5 Select **N2 Collision Gas** to allow the N<sub>2</sub> collision gas flow.
- 6 Click **Apply** to download the setpoints or **OK** to download the setpoints and close the window.
- 7 To make the new settings part of the method, select **Save** from the **Method** menu.

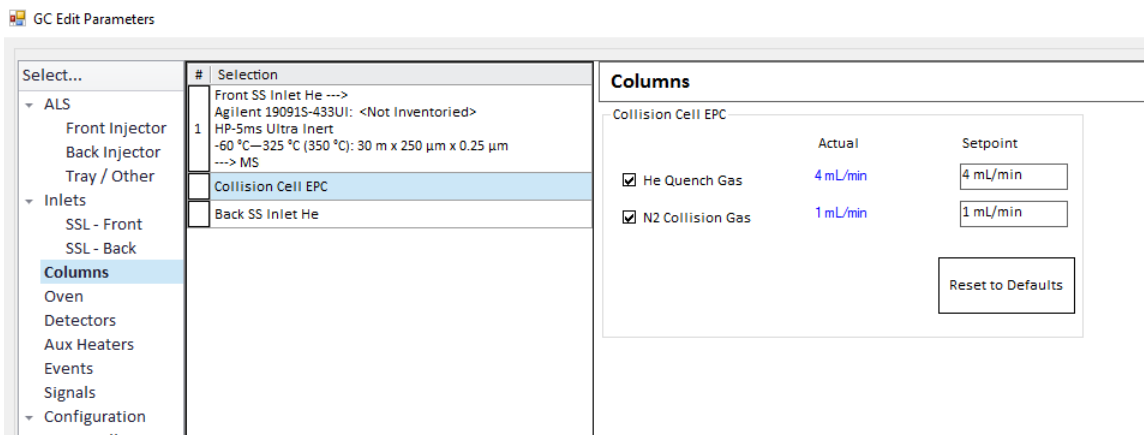



Figure 13. Setting the collision cell gas flow rate

## To Autotune the MS for EI Mode

The MS can be tuned using the MassHunter Workstation software.

### Procedure

- 1 Set the system to the same conditions (GC oven temperature, column flow, and MS analyzer temperature) that will be used for data acquisition.
- 2 In the **Instrument Control** panel, click the **MS Tune** icon  to display the **GC/Q-TOF Tune** dialog. (See **Figure 14**, "EI Autotune," on page 53.)
- 3 The current tune file is displayed in the upper left corner of the **GC/Q-TOF Tune** dialog. Verify that the correct tune file is loaded.
- 4 If necessary, load a new tune file by clicking on the **Files and Report** tab then click the **Load** button in the **Tune File** section. Select a tune file and click the **OK** button.
- 5 Click the **Autotune** tab and select the **Tune from default settings** if you are restarting the system after a system vent, major servicing, or a power outage. If you clear the **Tune from default settings** box, the autotune process starts using the previous tune values.
- 6 Select **Save tune file when done** check box to save the new tune parameters generated by the autotune. Do not select this item if you want to review the autotune report before saving the newly generated tune parameters.
- 7 Select the **Print autotune report** check box to automatically print a tune report.
- 8 Click the **Autotune** button to start the autotune. The **Status** line displays the current step in the autotune process and the plot of the tuned parameter for that step is shown in the top graph. If specified above, at the completion of the autotune, a Tune Report is printed.

To stop the autotune before it completes the automatic parameter selection, click the **Abort Tune** button. The parameters from the last successful autotune are used.

- 9 Review the Tune Report. If the results are acceptable and you did not select the **Save tune file when done** check box, save the autotune by clicking the **Files and Report** tab, then click the **Save** button.

See the manuals or online help provided with your MassHunter Data Acquisition Workstation software for additional information about tuning.

### 3 Operating in EI Mode

#### To Autotune the MS for EI Mode

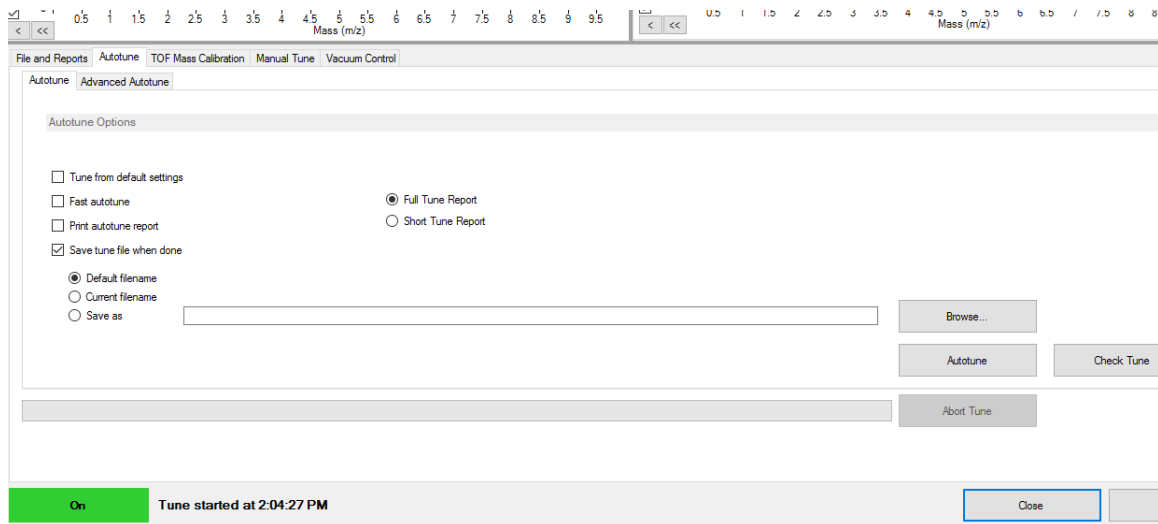


Figure 14. EI Autotune

## To Pump Down the MS

### WARNING

Ensure your MS meets all the conditions listed in the introduction to this chapter before starting up and pumping down the MS. Failure to do so can result in personal injury.

### WARNING

The use of hydrogen gas is specifically prohibited with this product.

### 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.

### Procedure

- 1 Open the analyzer cover to access the analyzer quad driver board. (See [“To Open the Analyzer Cover for Access to the Analyzer Sideplate”](#) on page 106.)
- 2 Plug the power cord into a grounded electrical outlet.
- 3 Turn on the Q-TOF power switch to start the pumpdown.

### 3 Operating in EI Mode To Pump Down the MS

- 4 Press lightly on the silver, vented metal box on the quad driver board to ensure a correct seal. See [Figure 15](#).




Figure 15. Pressing on the metal box

#### CAUTION

**Do not push on the filament board safety cover while pressing on the analyzer board. This cover was not designed to withstand this type of pressure.**

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 sideplate seal or the interface column nut.

- 5 Start the MassHunter Data Acquisition program.

- 6 Click the **Vacuum Control** icon  from the **Instrument Control** panel to monitor the instrument during the pumpdown. (See [Figure 16](#), "Vacuum Control tab," on page 56.)

### 3 Operating in EI Mode To Pump Down the MS

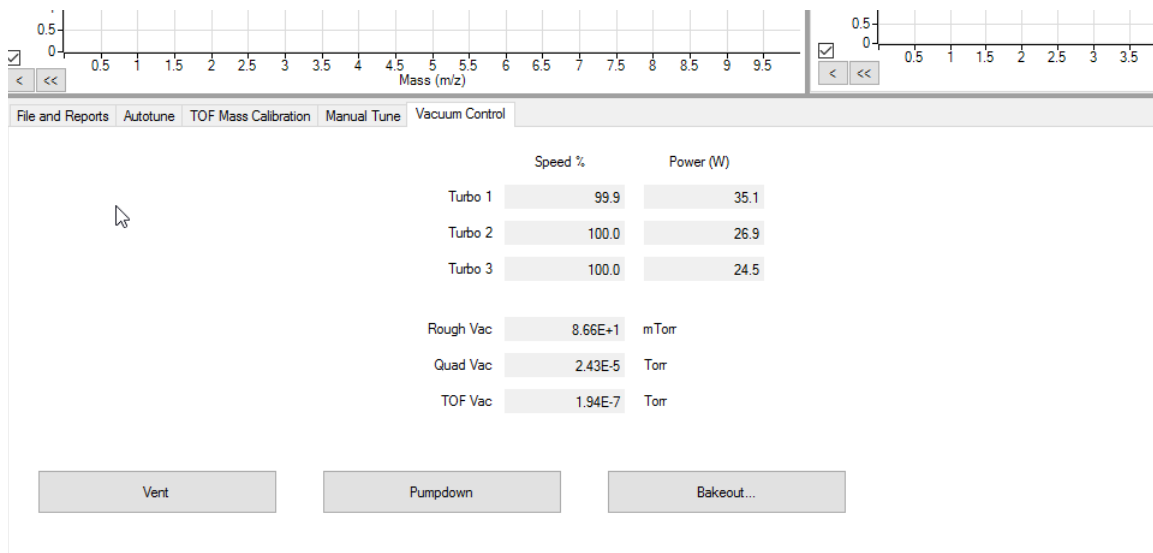


Figure 16. Vacuum Control tab

#### 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.**


- Within 10 to 15 minutes, the turbo pump speed should be up to 80%. (See [Figure 16](#).) The pump speed should eventually reach 95%. If these conditions are not met, the MS electronics will shut off the foreline pump. In order to recover from this condition, you must power cycle the MS. If the MS does not pump down correctly, see the manual or online help for information on troubleshooting air leaks and other vacuum problems.
- 7 Turn on the carrier gas flow after the vacuum pumps have been running for 15 minutes.
  - 8 When prompted, turn on the GC/MS interface heater and GC oven. Click **OK** when you have done so. The software will turn on the ion source and mass filter (quad) heater. The temperature setpoints are stored in the current autotune file.
  - 9 After the message **Okay to run** appears, wait 2 hours for the MS to reach thermal equilibrium. Data acquired before the MS has reached thermal equilibrium may not be reproducible.
  - 10 Tune the MS. (See [“To Autotune the MS for EI Mode”](#) on page 52.)



### 3 Operating in EI Mode To Vent the MS

## To Vent the MS

### Procedure

- 1 Click the **Vacuum Control** icon  from the **Instrument Control** panel. (See **Figure 16**, "Vacuum Control tab," on page 56.)
- 2 From the GC keypad, set the GC/MS interface heater and the GC oven temperatures to ambient (room temperature).
- 3 When the GC temperatures have reached 30 °C, turn off the flow of carrier gas.
- 4 Click the **Vent** button.

#### WARNING

The use of hydrogen gas is specifically prohibited with this product.

#### CAUTION

Ensure the GC oven and the GC/MS interface are cool before turning off carrier gas flow.

#### CAUTION

Do not power off the system. When powered off, the MS will shut off nitrogen flow.

#### WARNING

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

#### CAUTION

When the MS is vented, do not put the Workstation into Instrument Control view. Doing so will turn on the interface heater, which can damage the column.

### 3 Operating in EI Mode

To Vent the MS

## 4

# Operating in CI Mode

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To Perform a CI Autotune 74

This chapter provides information and instructions for operating the 7250 Q-TOF GC/MS system 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.

## 4 Operating in CI Mode

### Setting Up Your MS to Operate in CI Mode

# Setting Up Your MS to Operate in CI Mode

Setting up your MS for operation in CI mode requires special care to avoid contamination and air leaks.

- Always use the highest purity methane (and other reagent gases, if applicable). Methane must be at least 99.9995% pure.
- Always verify the MS is performing well in EI mode before switching to CI.
- Ensure the CI ion source is installed.
- Ensure the reagent gas plumbing has no air leaks. This is determined in PCI mode, checking for  $m/z$  32 after the methane pretune.
- Ensure the reagent gas inlet line is equipped with a gas purifier (not applicable for ammonia).

#### **WARNING**

**Use of hydrogen gas is specifically prohibited with this product.**

## The GC/MS Interface

The GC/MS interface (see **Figure 17**, “The GC/MS interface,” on page 62) is a heated conduit into the MS for the capillary column. It is bolted onto the right side of the analyzer chamber, with an O-ring seal and has a protective insulated 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 4 to 5 mm of the capillary column extend past the end of the guide tube and into the ionization chamber.

For CI operating mode, reagent gas is plumbed into the interface. A tip seal keeps reagent gases from leaking out around the tip. The reagent gas enters the interface body and mixes with carrier gas and sample in the ion source.

This GC/MS interface is also used for EI operation.

The GC/MS interface is heated by an electric cartridge heater. Normally, the heater is powered and controlled by the Thermal Aux #2 heated zone of the GC. The interface temperature can be set from the MassHunter Workstation or a GCs control panel. A sensor (thermocouple) in the interface monitors the temperature. The interface generally operates 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 in the GC/MS interface, the GC oven, or the GC inlet.**

## 4 Operating in CI Mode

### The GC/MS Interface

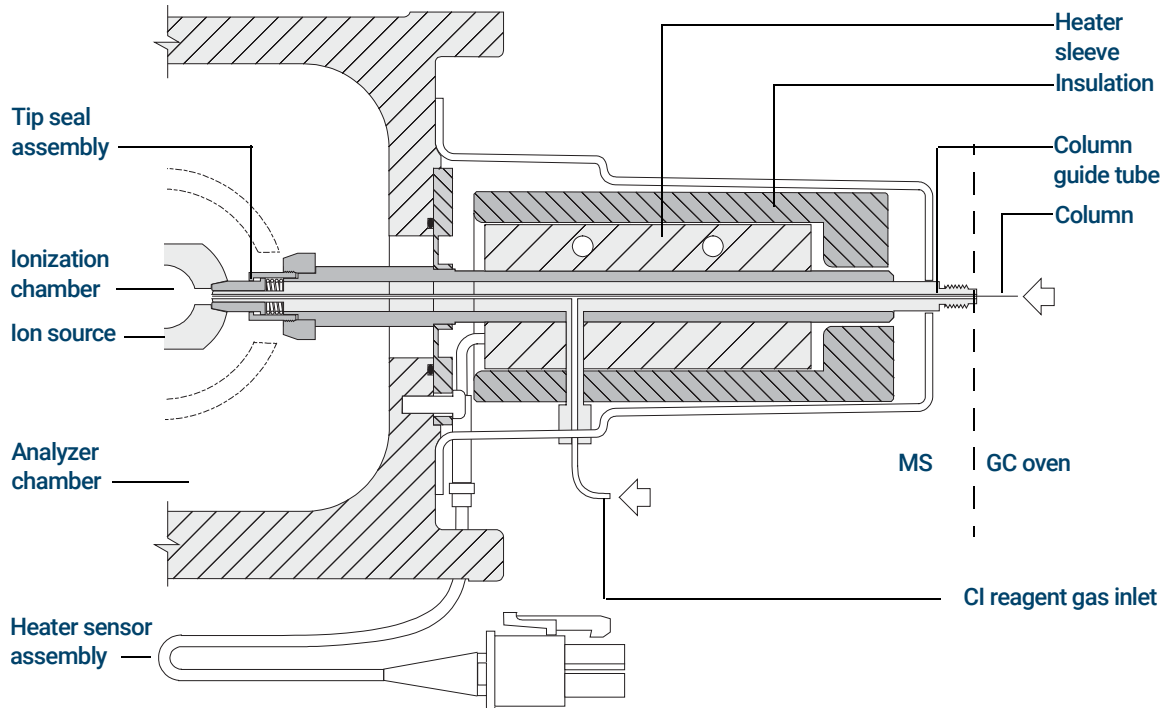


Figure 17. The GC/MS interface

## Operating the CI MS

Operating your GC/MS in the CI mode is slightly more complicated than operating in the EI mode. After tuning, gas flow, temperatures (see **Table 5**), and electron energy may need to be optimized for your specific analyte.

**Table 5** Temperatures for CI operation

	Ion source	Analyzer	GC/MS interface
PCI	300 °C	150 °C	280 °C
NCI	150 °C	150 °C	280 °C

### Start the system in CI mode

When starting up the system, you may begin in either PCI or NCI mode. Depending upon the application, use the following reagent gas flowrates 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)

## High Vacuum Pressure in CI Mode

The largest influences on operating pressure in CI mode are the reagent and collision cell gas flows. **Table 6** lists typical pressures for various reagent gas flows, depending upon the collision cell gas flowrate. 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 MS to the next.

### Analyzer vacuum with reagent gas flowing

Note that the mass flow controller (MFC) is calibrated for methane, and the vacuum gauge is calibrated for nitrogen, so these measurements are not accurate, but are intended as a guide to typical observed readings (see **Table 6**). They were taken with the following set of conditions. Note that these are typical PCI temperatures:

Source temperature	230 °C
Quad temperature	150 °C
GC/MS interface temperature	280 °C
GC inlet	250 °C
GC oven	100 °C
Helium carrier gas flow	1 mL/min

**Table 6** Typical analyzer vacuum with reagent gas flow

MFC (%)	Collision cell gas flow on N <sub>2</sub> = 1.5 mL/min			Collision cell gas flow off N <sub>2</sub> = 0 mL/min		
	Rough pump (mTorr)	Quadrupole (Torr)	Flight tube (Torr)	Rough pump (mTorr)	Quadrupole (Torr)	Flight tube (Torr)
0	1.36e+02	3.62e-05	3.35e-07	9.13e+01	5.98e-07	1.64e-07
10	1.36e+02	3.62e-05	3.37e-07	1.14e+01	1.27e-06	1.65e-07
15	1.43e+02	3.66e-05	3.37e-07	1.23e+01	1.62e-06	1.67e-07
20	1.5e+02	3.71e-05	3.39e-07	1.31e+01	1.96e-06	1.67e-07
25	1.57e+02	3.73e-05	3.41e-07	1.39e+01	2.32e-06	1.70e-07
30	1.63e+02	3.77e-05	3.41e-07	1.46e+01	2.64e-06	1.71e-07
35	1.69e+02	3.81e-05	3.41e-07	1.52e+01	3.00e-06	1.71e-07
40	1.74e+02	3.83e-05	3.43e-07	1.58e+01	3.34e-06	1.72e-07



## 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 7250 Q-TOF GC/MS 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 *Agilent 7250 Accurate-Mass Q-TOF GC/MS Concept Guide*. If you are not experienced with chemical ionization, we suggest reviewing that material before you proceed.

### Isobutane CI

Isobutane (C<sub>4</sub>H<sub>10</sub>) is commonly used for chemical ionization when less fragmentation is desired in the CI spectrum. This is because the proton affinity of isobutane is higher than that of methane; hence less energy is transferred in the ionization reaction.

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

### Ammonia CI

Ammonia (NH<sub>3</sub>) is commonly used for chemical ionization when less fragmentation is desired in the CI spectrum. This is because the proton affinity of ammonia is higher than that of methane; hence less energy is transferred in the ionization reaction.

Because many compounds of interest have insufficient proton affinities, ammonia chemical-ionization spectra often result from the addition of NH<sub>4</sub><sup>+</sup> and then, in some cases, from the subsequent loss of water. Ammonia reagent ion spectra have principal ions at *m/z* 18, 35, and 52, corresponding to NH<sub>4</sub><sup>+</sup>, NH<sub>4</sub>(NH<sub>3</sub>)<sup>+</sup>, and NH<sub>4</sub>(NH<sub>3</sub>)<sub>2</sub><sup>+</sup>.

## 4 Operating in CI Mode

### Carbon dioxide CI

#### CAUTION

Use of ammonia affects the maintenance requirements of the MS. See [Chapter 5, "General Maintenance,"](#) starting on page 77 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 *Agilent 7250 Q-TOF GC/MS Troubleshooting and Maintenance Manual*.)

#### CAUTION

**When running ammonia for five or more hours a day, the foreline pump must be ballasted (flushed with air) for at least one hour a day to minimize damage to pump seals. Always purge the MS with methane after flowing ammonia.**

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

## Carbon dioxide CI

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

## CI Autotune

After the reagent gas flow is set, the lenses and electronics of the MS should be tuned. This provides a good starting point for developing your application settings. Typical CI application settings are shown in **Table 7**.

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/MS interface by means of the gas flow control module. There are no tune performance criteria. If CI autotune completes, it passes.

### CAUTION

After the source is changed from EI to CI or vented for any other reason, the MS must be purged and baked out for at least two hours before tuning. Longer bakeout is recommended before running samples requiring optimal sensitivity.

### CAUTION

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

**Table 7** Typical CI application settings

Parameter	Methane		Isobutane		Ammonia	
	Positive	Negative	Positive	Negative	Positive	Negative
Emission	150 A	50 A	150 A	50 A	150 A	50 A
Electron energy	150 eV	150 eV	150 eV	150 eV	150 eV	150 eV
Filament	1	1	1	1	1	1
Repeller	3 V	3 V	3 V	3 V	3 V	3 V
Ion focus	130 V	130 V	130 V	130 V	130 V	130 V
Entrance lens offset	20 V	20 V	20 V	20 V	20 V	20 V
Shutoff valve	Open	Open	Open	Open	Open	Open
Suggested flow	20%	40%	20%	40%	20%	40%
Source temp	250 °C	150 °C	250 °C	150 °C	250 °C	150 °C
Quad temp	150 °C	150 °C	150 °C	150 °C	150 °C	150 °C
Interface temp	280 °C	280 °C	280 °C	280 °C	280 °C	280 °C
Autotune	Yes	Yes	No	Yes	No	Yes

## The Flow Control Module

The CI reagent gas flow control module regulates the flow of reagent gas into the CI GC/MS interface. The flow module consists of a mass flow controller (MFC), gas select valves, CI calibration valve, shutoff valve, control electronics, and plumbing.

The instrument provides Swagelok inlet fittings for connecting the CI reagent gas. The software refers to it as CI reagent gas. Supply reagent gas flow at 20 to 25 psi (138 to 172 kPa) for methane.

## To Switch from the EI Mode to CI Mode

**CAUTION**

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

**CAUTION**

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

### Procedure

- 1 Run Autotune or otherwise verify the Q-TOF GC/MS is operating correctly in EI mode.
- 2 Remove the LE-EI ion source. (See **"To Remove the LE-EI Ion Source"** on page 85.)
- 3 Install the Axial CI ion source. (See **"To Install the Axial CI Ion Source"** on page 122.)
- 4 Load a suitable PCI or NCI method for use with the CI source.
- 5 Click the **MS Tune** icon in the **Instrument Control** panel to display the Tune dialog box, and select the **Autotune** tab.

The method selects PCI or NCI autotune and the correct reagent gas.

- 6 Select the **Tune from default settings** check box because you have changed the ion source.
- 7 Select **Print autotune report** check box to automatically print a tune report.
- 8 Click the **Autotune** button to start the autotune. At the completion of the autotune, a Tune Report is printed.
- 9 Review the Tune Report. If the results are acceptable, save the autotune by clicking the **Files and Report** tab, then click the **Save** button.

## 4 Operating in CI Mode

### To Switch from the EI Mode to CI Mode

**Table 8** Default Tune Control Limits, used by CI autotune only

Reagent gas	Methane		Ammonia	
Ion polarity	Positive	Negative	Positive	Negative
Abundance target	1x10 <sup>6</sup>	1x10 <sup>6</sup>	N/A	1x10 <sup>6</sup>
Peakwidth target	0.7	0.7	N/A	0.7
Maximum repeller	4	4	N/A	4
Maximum emission current, $\mu$ A	240	50	N/A	50
Max electron energy, eV	240	240	N/A	240

#### Notes for Table 8

- **N/A:** Not available.
- **Abundance target:** Adjust higher or lower to get desired signal abundance. Higher signal abundance also gives higher noise abundance.
- **Peakwidth target:** Higher peak width 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  $\mu$ A.

# To Operate the Reagent Gas Flow Control Module

## Procedure

- 1 In **Instrument Control** panel, select the **MS Tune** icon to display the **GC/Q-TOF Tune** dialog box. Select the **Manual Tune** tab, then select the **Ion Source** tab to display the ion source parameters.

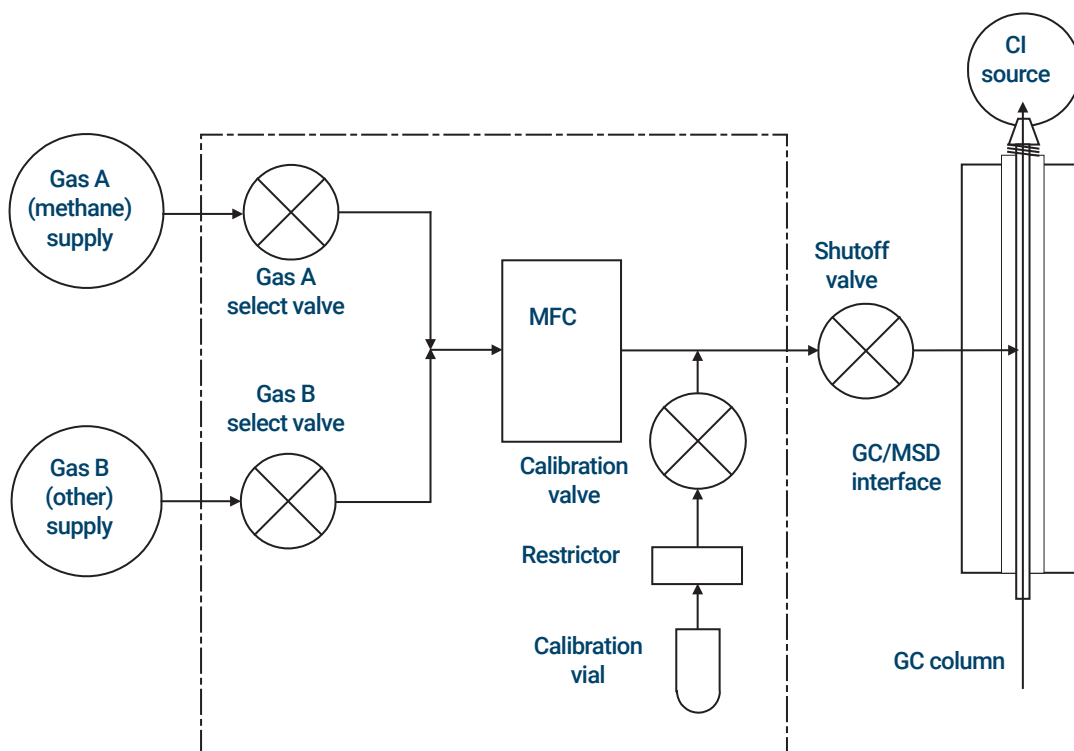


Figure 18. CI flow control

- 2 Use the parameters in the **Gas Control** area to control reagent gas flow.
  - **Methane** - Select if methane is the reagent gas.
  - **Ammonia or other** - Select if this is the reagent gas.

## 4 Operating in CI Mode

### To Operate the Reagent Gas Flow Control Module

- **Gas Flow** - Enter percent of maximum volumetric flow for the selected reagent gas. The actual % transmitted by the flow controller is shown next to this entry. 20% is a good value for PCI, and 40% is a good value for NCI.
- **Set** - Opens the selected reagent gas supply valve and controls the reagent gas flow to the entered setpoint.
- **Off** - Turns off the reagent gas flow.
- **Purge** - Opens the selected reagent gas valve for six minutes to clear the system of unwanted compounds.
- **Pumpout** - Closes the reagent gas valve for four minutes and evacuates the system of reagent gases. At the end of the pumpout time the selected reagent gas valve opens.



## To Set a Reagent Gas Flow

### 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 two 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 will need to vent the MS and clean the ion source.

### Procedure

- 1 In **Instrument Control** panel, select the **MS Tune** icon to display the **GC/Q-TOF Tune** dialog box. Select the **Manual Tune** tab then select the **Ion Source** tab to display the ion source parameters.
- 2 In the **Gas Control** area, select the CI reagent gas you are using.
- 3 Enter the reagent gas flow setpoint in the **Gas 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.
- 4 Click **Set**. The **Flow Set** indication is displayed.

The reagent gas is flowing into the ion source at the rate displayed next to the setpoint.

- 5 Click the **Files and Reports** tab, then click **Save** to save your changes to the currently loaded tune file.

## To Perform a CI Autotune

### CAUTION

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

### CAUTION

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

### Procedure

- 1 Click the **MS Tune** icon in the **Instrument Control** panel to display the **GC/Q-TOF Tune** window.
- 2 If necessary, load a new tune file by clicking on the **Files and Report** tab, then click **Load** in the **Tune File** area. Select a tune file and click **OK**.  
  
The tune file must match the type of ion source in the analyzer. For a CI ion source, select a tune file created for a positive or negative CI source.
- 3 In the **Autotune** \Autotune tab, select **Tune from default settings** if you are restarting the system after a system vent, major servicing, or a power outage. If you clear the **Tune from default settings** box, the Autotune process starts using previous tune values.
- 4 In the **Autotune** \Autotune tab, select **Include emission optimization** if you want to find the emission current required to optimize ion abundance.
- 5 In the **Autotune** \Autotune tab, if a tune report is required, select **Print autotune report**, then select **Full tune report** or **Short tune report**. The selected report type is generated automatically at the end of the autotune.
- 6 Click on the **Manual Tune** tab, and select the **Ion source** tab. In the **Gas Control** section, select the reagent gas, and then enter a **Gas Flow** rate of 20% for a PCI source or 40% for an NCI source. Click on the **Autotune** tab to return to Autotune.
- 7 Select **Save tune file when done** to save the new tune parameters generated by the autotune. Do not select this item if you want to review the autotune report before saving the newly generated tune parameters.

## 4 Operating in CI Mode

### To Perform a CI Autotune

- 8 Click **Autotune** to start the autotune. The **Waiting for instrument to be ready** dialog is displayed. Wait for the instrument's indicated parameters to maintain the required setpoints (green indicator) and enter ready state. When ready, the dialog closes and the autotune begins. When the Autotune starts, all other tabs in the Tune dialog are inaccessible.

If you selected **Use custom autotune parameters** the Custom Autotune dialog is displayed first when you click **Autotune**. Verify the entered custom values, and click **OK**.

To stop the autotune before it completes the autotune parameter selection, click **Abort Autotune**. The parameters from the last successful autotune are used.

- 9 Click **OK** when an information dialog is displayed indicating the autotune is finished running. The dialog can indicate that the autotune completed successfully or failed with an error message displayed.
- 10 Review the Tune Report. If the results are acceptable and you did not select **Save tune file when done**, save the autotune by clicking the **Files and Report** tab, then click **Save**.

## 4 Operating in CI Mode

To Perform a CI Autotune

## 5

# General Maintenance

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This chapter describes general maintenance procedures for the Agilent 7250 Accurate-Mass Quadrupole Time-of-Flight (Q-TOF) GC/MS System.

## Before Starting

You can perform most of the MS required maintenance that is explained in this chapter. For your safety, read all of the information in this introduction before performing any maintenance tasks.

### Scheduled maintenance

Common maintenance tasks are listed in **Table 9**. Performing these tasks when scheduled can reduce operating problems, prolong system life, and reduce overall operating costs.

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

**Table 9** Maintenance schedule

Task	Every week	Every 6 months	Every year	As needed
Check the foreline pump oil level	X			
Check the calibration vial(s)		X		
Replace the foreline pump oil*		X		
Replace the tip seals on the optional IDP			X	
Replace the filters on the optional IDP			X	
Clean the ion source				X
Check the carrier gas trap(s) on the GC and MS				X
Replace the worn out parts				X
Replace GC gas supplies				X
Replace CI reagent gas supplies				X
Check the foreline pump				X
Tune the MS				X
Leak check the system				X
Lubricate side plate or vent valve O-rings <sup>†</sup>				X

\* Or as needed, every 3 months if ammonia is being used as CI 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.

## 5 General Maintenance

### Tools, spare parts, and supplies

## Tools, spare parts, and supplies

Some of the required tools, spare parts, and supplies are included in the GC shipping kit, MS shipping kit, or MS 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 MS is plugged in, even if the power switch is off, potentially dangerous voltage (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 the boards
- Wires and cables between the boards and the connectors on the back panel of the MS
- 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**

**Do not perform maintenance with the MS turned on or plugged into its power source unless you are specifically instructed to do so by a procedure in this chapter.**

Some procedures in this chapter require access to the inside of the MS 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 MS operate at, or reach, temperatures high enough to cause serious burns. These parts include, but are not limited to:

- GC/MS interface
- Analyzer parts
- Vacuum pumps

### WARNING

The foreline pump can cause burns if touched when operating.

### WARNING

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

### WARNING

The GC/MS 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 MS is off. The GC/MS interface is well insulated. Even after it is turned off, it cools very slowly.

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 mist filter is supplied with the standard foreline pump. This filter stops *only* pump oil droplets. It *does not trap* any other chemicals. If you are using toxic solvents or analyzing toxic chemicals, install a hose from the mist filter outlet to the outdoors or into a fume hood vented to the outdoors. Comply with your local air quality regulations.



## 5 General Maintenance

### Chemical residue

#### **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, use a hose to vent to a safe location.

The fluids in the 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 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.

#### **WARNING**

The optional IDP foreline pump may contain significant quantities of dust. This dust can contain traces of the chemicals analyzed by the mass spectrometer. When replacing the pump's tip seal, do NOT use compressed air to blow out this dust. Using compressed air will contaminate your laboratory with potentially hazardous dust.

#### **WARNING**

Always perform any maintenance procedures using hazardous solvents under a fume hood. Operate the MS in a well-ventilated room.

# Maintaining the Vacuum System

## Periodic maintenance

As listed in **Table 9**, "Maintenance schedule," on page 78, some maintenance tasks for the vacuum system must be performed periodically. These include:

- Checking the foreline pump oil color and level (every week)
- Checking the calibration vial (every 6 months)
- Replacing the rotary vane foreline pump oil and oil mist filter (every 6 months or as needed)
- Replacing the tip seals in the optional IDP dry scroll foreline pump (yearly)

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

## Other procedures

Tasks such as replacing an ion vacuum gauge should be performed only when needed by a certified Agilent service personnel. See the *Agilent 7250 Accurate-Mass Q-TOF GC/MS Troubleshooting and Maintenance Manual* and see the online help in the MassHunter WorkStation software for symptoms that indicate this type of maintenance is required.

## More information is available

If you need more information about the locations or functions of vacuum system components, see the *Agilent 7250 Accurate-Mass Q-TOF GC/MS Troubleshooting and Maintenance Manual*.

Most of the procedures in this chapter are illustrated with video clips on the Agilent GC/MS Hardware User Information & Instrument Utilities and 7250 Q-TOF GC/MS User Information disks.

# Maintaining the Analyzer

## Scheduling

None of the analyzer components requires periodic maintenance. Some tasks, however, must be performed when MS behavior indicates they are necessary. These tasks include:

- Cleaning the ion sources
- Replacing filament

The *Agilent 7250 Accurate-Mass Q-TOF GC/MS 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 Workstation software provides more extensive information.

## Precautions

Keep components clean during analyzer maintenance. Never open the analyzer chamber, except when accessing the ion source or installing a GC column.

### CAUTION

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

### WARNING

**The analyzers operate at high temperatures. Do not touch any part until you are sure it is cool.**

### Electrostatic discharge

All of the printed circuit boards in the MS 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 quadrupole board. ESD damage may not cause immediate failure, but it will gradually degrade the performance and stability of your MS.

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

## 5 General Maintenance

More information is available

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, 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 MS. 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.**

### **Analyzer parts that should not be disturbed**

The mass filter (quadrupole) and the collision cell require 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.

### CAUTION

**Incorrect handling or cleaning of the mass filter can damage it and have a serious, negative effect on instrument performance.**

## More information is available

If you need more information about the locations or functions of analyzer components, refer to the *Agilent 7250 Accurate-Mass Q-TOF GC/MS Concepts Guide*.

## To Remove the LE-EI Ion Source

### Materials needed

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



### Procedure

- 1 Vent the MS. (See **“To Vent the MS”** on page 57.)

#### WARNING

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

#### CAUTION

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

#### CAUTION

Use an antistatic wrist strap, and take other antistatic precautions before touching analyzer components.

#### CAUTION

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

- 2 Open the analyzer chamber. (See **“To Open the Analyzer Cover for Access to the Analyzer Sideplate”** on page 106.)
- 3 Remove the two large thumbscrews that hold the ion source in place. (See **Figure 19**, “The LE-EI ion source before removal,” on page 86.)
- 4 Disconnect the black, white, and gray filament wires and the red repeller wire from the front of the ion source. Do not bend the wires any more than necessary. (See **“To Connect or Disconnect Wiring to the LE-EI Ion Source”** on page 87.)
- 5 Using the source finger grip, pull the ion source out of the source radiator.

## 5 General Maintenance

### To Remove the LE-EI Ion Source

- 6 The source contacts have spring loaded pins so some force must be applied to pull the source out. (See **Figure 19.**)

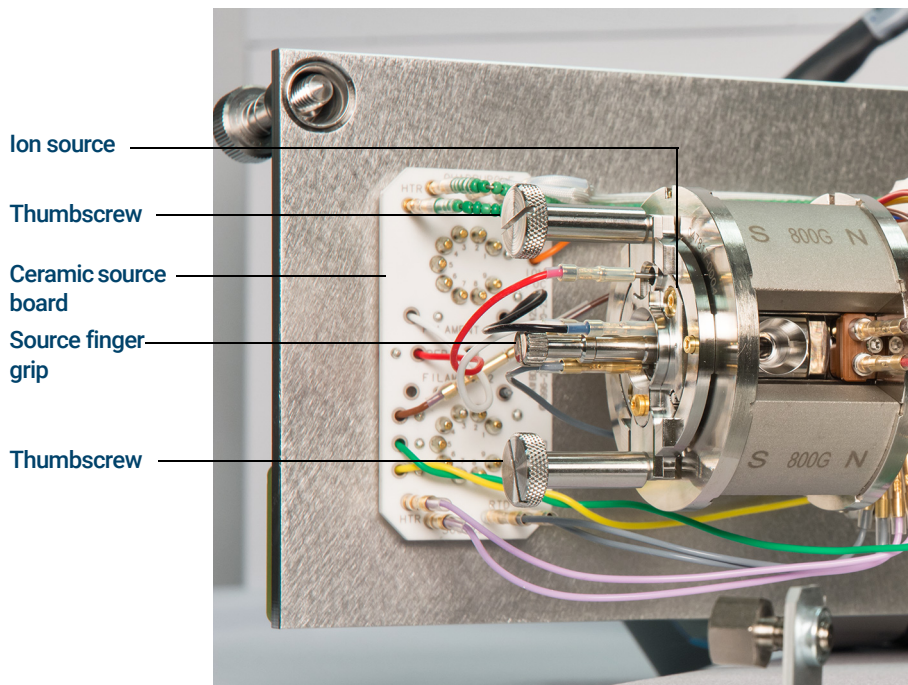


Figure 19. The LE-EI ion source before removal

## 5 General Maintenance

### To Connect or Disconnect Wiring to the LE-EI Ion Source

# To Connect or Disconnect Wiring to the LE-EI Ion Source

This topic is used to identify the wiring connections to the ion source. For detailed procedures and videos, see **“To Remove the LE-EI Ion Source”** on page 85, or **“To Install the LE-EI Ion Source”** on page 96.

### Materials needed

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

### Procedure

Use tweezers or pliers to connect or disconnect the ceramic board wire leads (red, white, and black) and the gray electron extractor wire at the source connectors. (See **Figure 20**.)

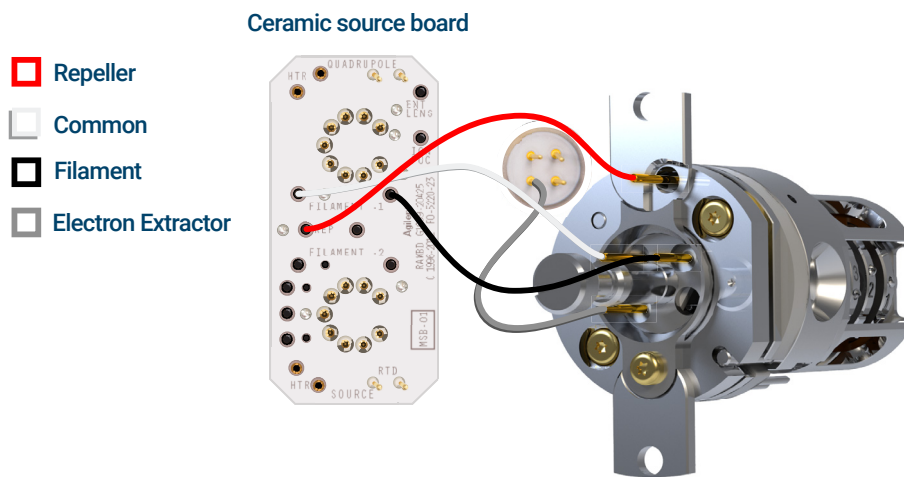


Figure 20. Wiring for the LE-EI ion source

## To Disassemble the LE-EI Ion Source

### Materials needed

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



### Procedure

Refer to **Figure 21**, “Exploded parts view of the LE-EI ion source,” on page 89, and **Table 10**, “Parts list for LE-EI ion source (**Figure 21**),” on page 90 while using this procedure.

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

### CAUTION

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



## 5 General Maintenance

### To Disassemble the LE-EI Ion Source

- 9 Remove the five lenses from the lens insulator/holder.

#### CAUTION

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

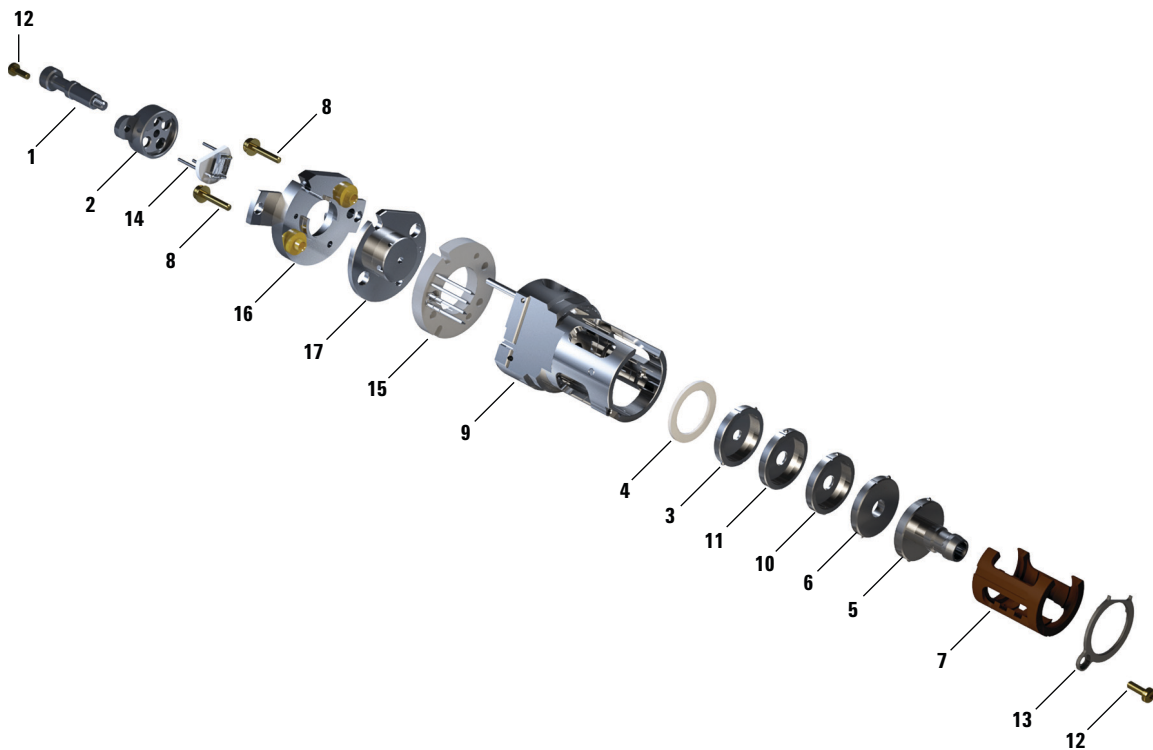


Figure 21. Exploded parts view of the LE-EI ion source

## 5 General Maintenance

### To Disassemble the LE-EI Ion Source

**Table 10** Parts list for LE-EI ion source (Figure 21)

Item number	Item description	Part number
1	Source finger grip	G7002-20008
2	Filament block	G7002-20019
3	Extractor lens (5)*, with 3 mm opening	G7004-20061
4	Ceramic insulator for extractor	G7002-20064
5	Entrance lens assembly (1)*	G7250-20075
6	Ion focus lens (2)*	G7004-20068
7	Lens insulator/holder	G7002-20074
8	M2 × 0.4 screw × 12 mm long gold plated screw	G7002-20083
9	Source body	G7002-20084
10	Post extractor lens 2 (3)*	G7004-20090
11	Post extractor lens 1 (4)*	G7004-20004
12	M2 × 6 mm gold plated screw	G7002-20109
13	Locking ring lens insulator	G7002-20126
14	Filament assembly, HE, Single	G3850-60021
15	Ring heater/sensor assembly	G7002-60043
16	Source mount 1.5 mm	G7002-60053
17	Repeller assembly	G7002-60057
Complete assembly	Ion Source Assy, LE-EI 7250A	G7250-67170

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

## To Assemble the LE-EI Ion Source

### Materials needed

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



### Procedure

#### CAUTION

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

Refer to **Figure 24**, “Exploded parts view of the LE-EI ion source,” on page 94, and **Table 11**, “Parts list for LE-EI ion source (**Figure 24**),” on page 95 while using this procedure.

- 1 Assemble the five lenses inside the lens insulator. (See **Figure 22**, “Assembled LE-EI ion source lens stack,” on page 92.) The lens number is engraved onto the outer circumference of each lens.
  - a Starting with the entrance lens 1, set the lens into the end groove in the lens insulator, and rotate the lens until you feel the ball seat into the circular recess.
  - b Insert the next 4 lenses, in numerical order, into the lens insulator. The lens chamber open end always faces the entrance lens 1. Rotate each lens until you feel the ball seat into the circular recess.

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

## 5 General Maintenance

### To Assemble the LE-EI Ion Source

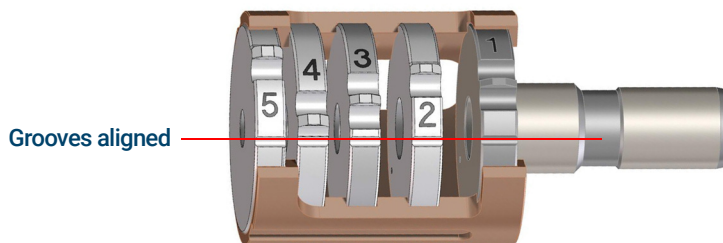


Figure 22. Assembled LE-EI ion source lens stack

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

#### CAUTION

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

- 3 Insert the lens stack encased in the insulator into the source body. (See [Figure 24](#), "Exploded parts view of the LE-EI ion source," on page 94.) The engraved numbers face the full opening extending to the end of the source body. Verify that the ceramic is sitting flush at the end of the source body.

## 5 General Maintenance

### To Assemble the LE-EI Ion Source

- Using the T6 Torx driver, install and secure the gold plated setscrew and lock ring lens insulator that holds the lens assembly in place. (See **Figure 23.**)



Figure 23. Secure the lens screw and lock ring insulator

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

#### CAUTION

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

- Insert the three filament leads into the side of the filament block opposite the finger grip.
- Orient the filament so that the ceramic filter fits flush with the filament block.
- Insert the filament block into the source holder, and use a T6 Torx screwdriver to secure it to the holder with the gold plated screw.

## 5 General Maintenance

### To Assemble the LE-EI Ion Source

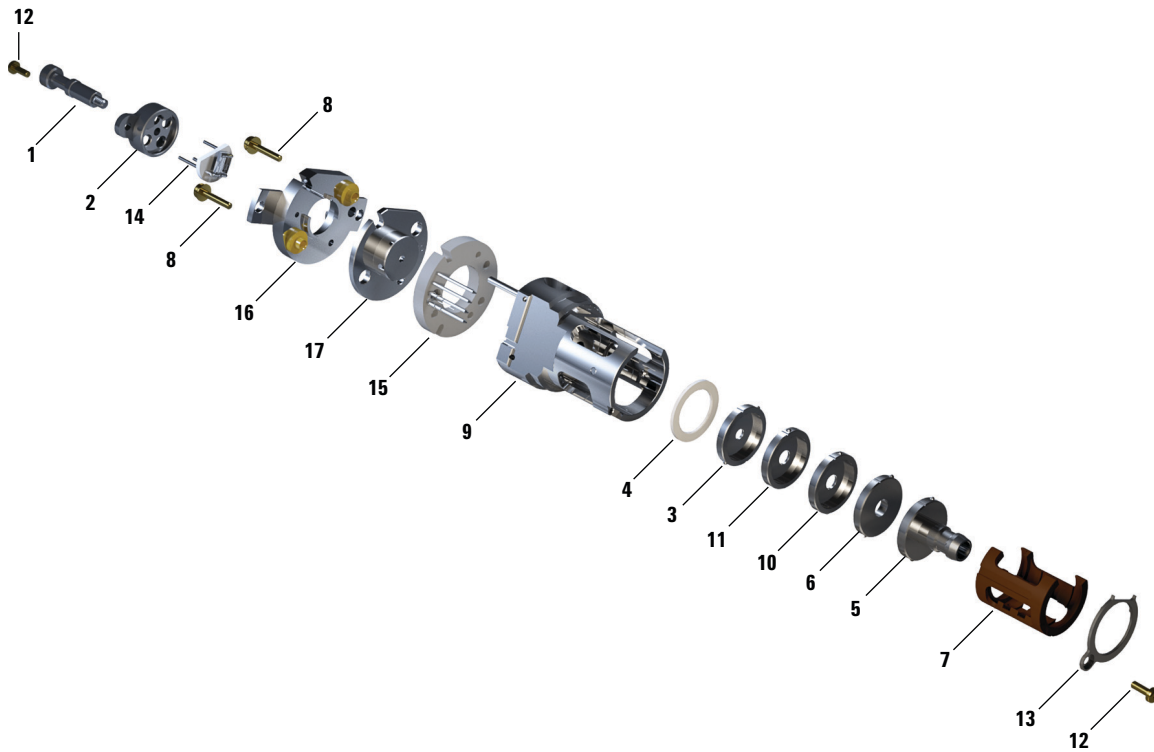


Figure 24. Exploded parts view of the LE-EI ion source

## 5 General Maintenance

### To Assemble the LE-EI Ion Source

**Table 11** Parts list for LE-EI ion source (Figure 24)

Item number	Item description	Part number
1	Source finger grip	G7002-20008
2	Filament block	G7002-20019
3	Extractor lens (5)*, with 3 mm opening	G7004-20061
4	Ceramic insulator for extractor	G7002-20064
5	Entrance lens assembly (1)*	G7250-20075
6	Ion focus lens (2)*	G7004-20068
7	Lens insulator/holder	G7002-20074
8	M2 × 0.4 screw × 12 mm long gold plated screw	G7002-20083
9	Source body	G7002-20084
10	Post extractor lens 2 (3)*	G7004-20090
11	Post extractor lens 1 (4)*	G7004-20004
12	M2 × 6 mm gold plated screw	G7002-20109
13	Locking ring lens insulator	G7002-20126
14	Filament assembly, HE, single	G3850-60021
15	Ring heater/sensor assembly	G7002-60043
16	Source mount 1.5 mm	G7002-60053
17	Repeller assembly	G7002-60057
Complete assembly	Ion Source Assy, LE-EI 7250A	G7250-67170

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

## To Install the LE-EI Ion Source

### Materials needed

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



### Procedure

#### CAUTION

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

- 1 Vent the MS. (See **“To Vent the MS”** on page 57.)
- 2 Open the analyzer chamber. (See **“To Open the Analyzer Cover for Access to the Analyzer Sideplate”** on page 106.)
- 3 Align the ion source so that the slotted opening for the lens stack, where the lens numbers are visible, is on the right side. Also, position it so the two attachment tabs of the source mount align with the corresponding slots in the source radiator. (See **Figure 25**, “The ion source radiator,” on page 97.) Slide the ion source into the source radiator until resistance is felt.



## 5 General Maintenance

### To Install the LE-EI Ion Source

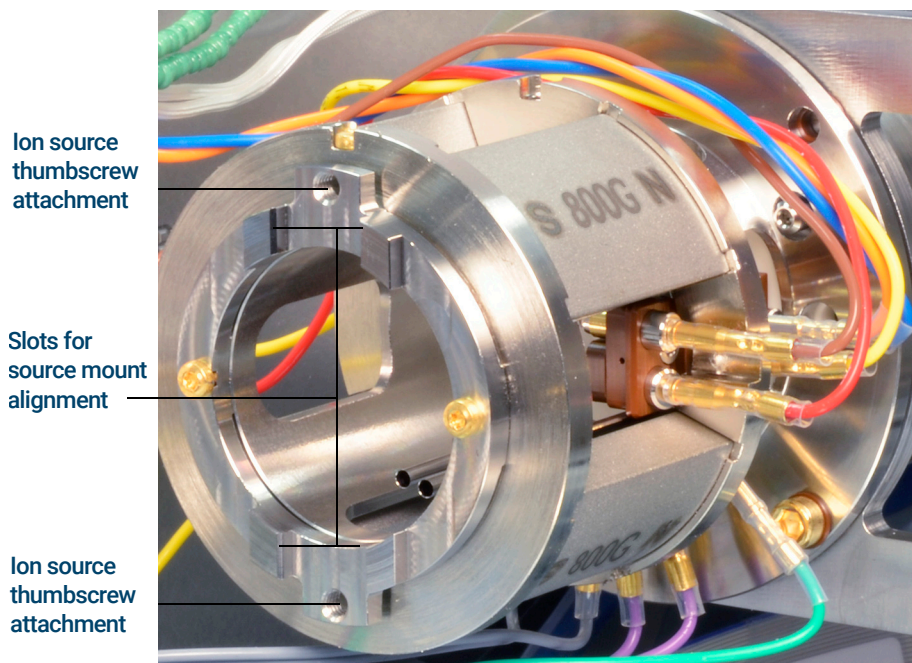


Figure 25. The ion source radiator

- 4 Snap the source into place so that the source mount tabs are flush against the source mount attachment surface of radiator. Some force is required to overcome the resistance of the electrical contacts spring loaded surfaces.
- 5 Connect the ion source wires. (See **“To Connect or Disconnect Wiring to the LE-EI Ion Source”** on page 87.)
- 6 Install and hand-tighten the source thumbscrews. (See **Figure 25.**) Do not overtighten the thumbscrews.
- 7 Close the analyzer chamber.
- 8 Pumpdown the MS. (See **“To Pump Down the MS”** on page 54.)
- 9 Mass calibrate the MS. (See **“To Autotune the MS for EI Mode”** on page 52.)

## To Clean the LE-EI Ion Source

### Materials needed

- Abrasive paper (5061-5896)
- Alumina abrasive powder, 100 g (393706201)
- Aluminum foil, clean
- Cloths, clean (05980-60051)
- Cotton swabs (5080-5400)
- Glass beakers, 500 mL
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Solvents
  - Acetone, reagent grade
  - Methanol, reagent grade
  - Methylene chloride, reagent grade
- Ultrasonic bath



### Procedure

- 1 Disassemble the LE-EI ion source. (See **“To Disassemble the LE-EI Ion Source”** on page 88.)
- 2 Collect the following parts to be cleaned: (See **Figure 26**, “LE-EI ion source parts to be cleaned,” on page 99.)
  - Filament mount
  - Source mount
  - Repeller
  - Source body
  - Extractor lens (5)
  - Post extractor lens 1 (4)
  - Post extractor lens 2 (3)
  - Ion focus lens (2)
  - Entrance lens (1)

These are the parts that contact the sample or ion beam. The other parts normally should not require cleaning.

## 5 General Maintenance

### To Clean the LE-EI Ion Source

#### 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.

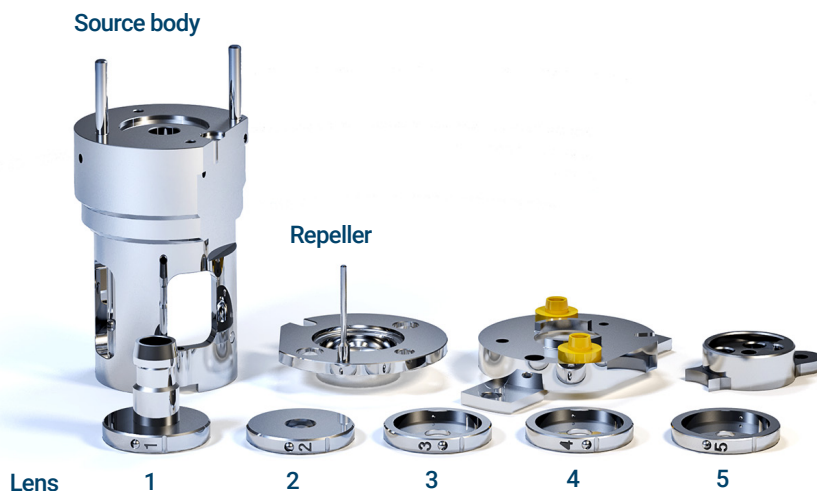


Figure 26. LE-EI ion source parts to be cleaned

#### CAUTION

The filament, source heater assembly, insulators, source mounting plate, and filament block cannot be cleaned ultrasonically. Replace these components if major contamination occurs.

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

#### CAUTION

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

- 4 Abrasively clean the surfaces that contact the sample or ion beam.

Use an abrasive slurry of alumina powder and reagent-grade methanol on a cotton swab. Use enough force to remove all discolorations. Polishing the parts is not necessary; small scratches will not harm performance. Also,

## 5 General Maintenance

### To Clean the LE-EI Ion Source

abrasively clean the discolorations where electrons from the filament enter the source body.

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

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

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

#### CAUTION

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

#### WARNING

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

- 7 Ultrasonically clean the parts, except the source mount, (each group separately) for 15 minutes in each of the following solvents:
  - Methylene chloride (reagent-grade)
  - Acetone (reagent-grade)
  - Methanol (reagent-grade)
- 8 Place the parts in a clean beaker. Loosely cover the beaker with clean aluminum foil (dull side down).
- 9 Dry the cleaned parts in an oven at 100 °C for 5–6 minutes.

## 5 General Maintenance

### To Remove the LE-EI Ion Source Filament Assembly

# To Remove the LE-EI Ion Source Filament Assembly

#### Materials needed

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



#### Procedure

- 1 Vent the MS. (See **"To Vent the MS"** on page 57.)

#### CAUTION

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

- 2 Open the analyzer chamber. (See **"To Open the Analyzer Cover for Access to the Analyzer Sideplate"** on page 106.)
- 3 Remove the ion source and place it on a clean cloth on your work surface. (See **"To Remove the LE-EI Ion Source"** on page 85.)
- 4 Remove the screw securing the filament block to the source mount. (See **Figure 27**, "Replacing the filament," on page 102.)

#### 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.

## 5 General Maintenance

### To Remove the LE-EI Ion Source Filament Assembly

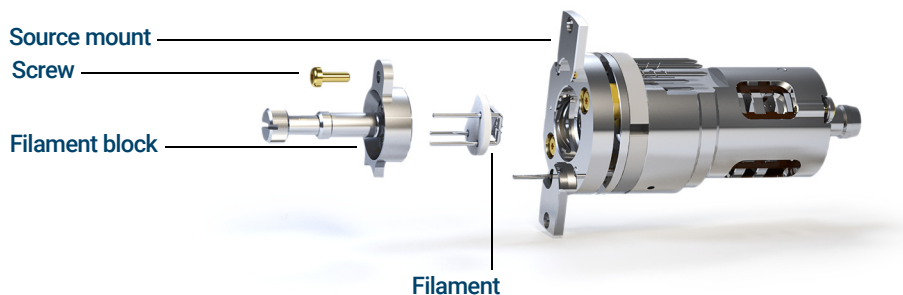


Figure 27. Replacing the filament

- 5 Use the hand grip on the filament block to remove the filament block from the source, by lifting the source body up off of the filament block, while holding the filament block so that the filament will not fall and become damaged.

### CAUTION

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

- 6 Remove the filament from the filament block.

## 5 General Maintenance

### To Install the LE-EI Ion Source Filament Assembly

# To Install the LE-EI Ion Source Filament Assembly

#### Materials needed

- Filament assembly, HE, single (G3850-60021)
- Hex ball driver, 1.5 mm (8710-1570)
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- T6 Torx driver (8710-2548)



#### Procedure

- 1 Remove the old filament. (See **“To Remove the LE-EI Ion Source Filament Assembly”** on page 101.)
- 2 Insert the 3 pins on the filament through the rear of the filament block. (See **Figure 27**, “Replacing the filament,” on page 102.)
- 3 Place the filament block in the source mount.
- 4 Use a T6 Torx screwdriver to tighten the screw securing the filament block to the source mount.
- 5 Reinstall the ion source. (See **“To Install the LE-EI Ion Source”** on page 96.)
- 6 Close the analyzer chamber.
- 7 Pumpdown the MS. (See **“To Pump Down the MS”** on page 54.)
- 8 Autotune the MS.

## 5 General Maintenance

### To Remove the Source Window Cover

# To Remove the Source Window Cover

The source window cover is in front of the instrument. (See **Figure 28**.) Remove it to access the EI calibration vial, or prior to removing the analyzer cover. Do not remove the source window cover for any other reason.



#### Procedure

- 1 Grasp the source window cover by the outside corners.
- 2 Pull the cover straight up, and remove it from the instrument.

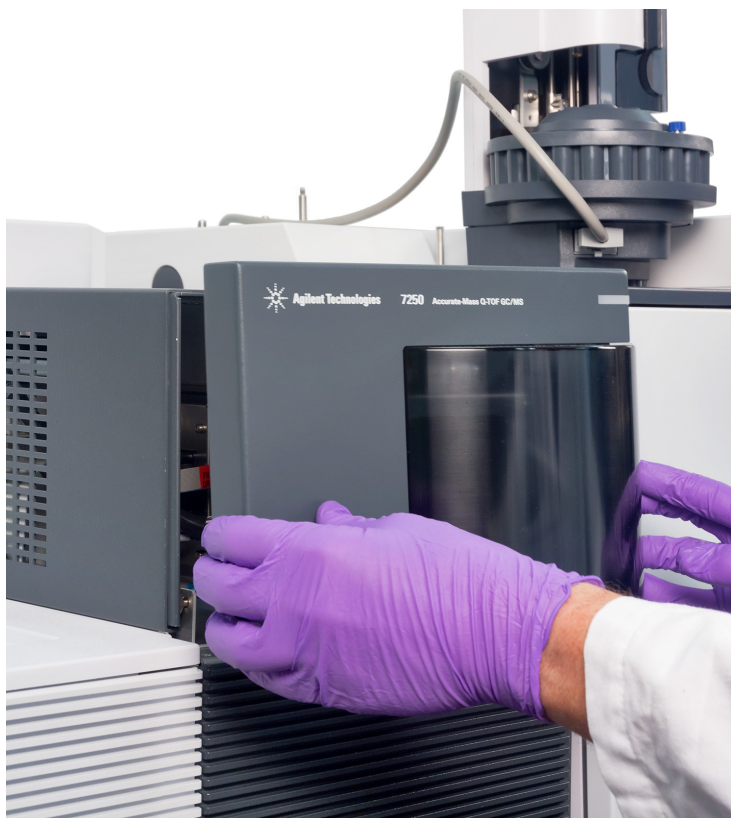


Figure 28. Removing the source window cover



## 5 General Maintenance

### To Install the Source Window Cover

# To Install the Source Window Cover

The source window cover is in front of the instrument. (See **Figure 29**.)



### Procedure

- 1 Grasp the source window cover by its side edges.
- 2 Place the cover on the top edge of the right front grille, and move the cover pins into the latch on the analyzer top cover frame.

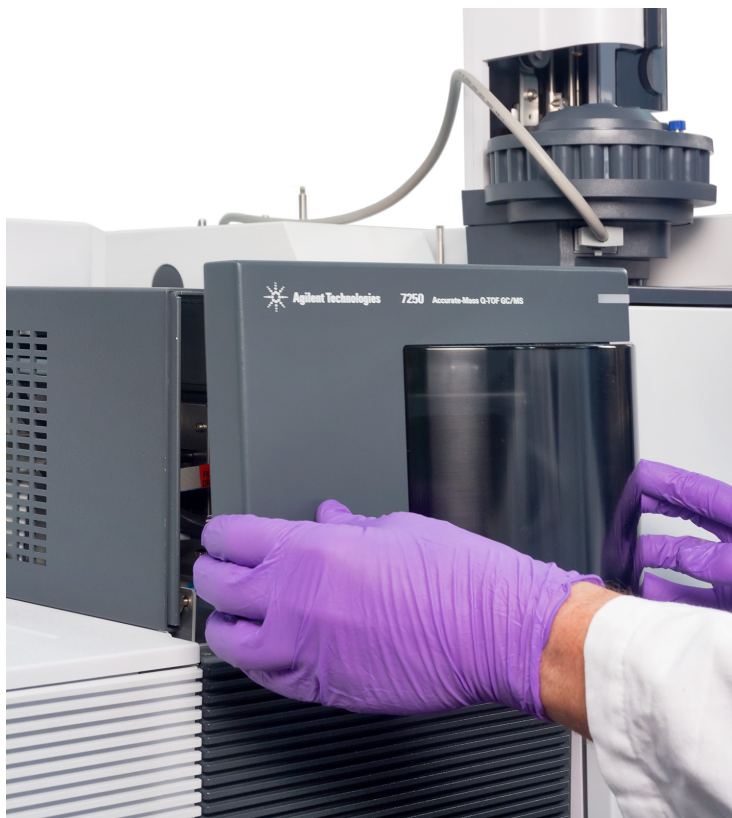


Figure 29. Installing the source window cover

## 5 General Maintenance

### To Open the Analyzer Cover for Access to the Analyzer Sideplate

# To Open the Analyzer Cover for Access to the Analyzer Sideplate

Open the analyzer cover to access the analyzer sideplate only. (See **Figure 30.**)

This is necessary during pump down, for adjusting the analyzer sideplate thumbscrews, and for removing the ion source for maintenance.



#### Procedure

- 1 Remove the source window cover. (See **“To Remove the Source Window Cover”** on page 104.)
- 2 Pull the analyzer cover pull to release the cover.
- 3 Move the cover away from the analyzer.



Figure 30. Covers

## To Install the Analyzer Cover



### Procedure

- 1 Hold the analyzer cover by its side edges, and bring it up to the position shown. (See **Figure 31**.)
- 2 Move the cover pins into the analyzer top cover frame holes where it is held in place by a magnet.
- 3 Install the source window cover. (See **“To Install the Source Window Cover”** on page 105.)



Figure 31. Installing the analyzer cover

## 5 General Maintenance

### To Install the Analyzer Cover

## 6

# CI Maintenance

General Information 110

To Remove the Axial CI Source 111

To Connect or Disconnect Wiring to the Axial CI Ion Source 113

To Disassemble the Axial CI Ion Source 114

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To Assemble the Axial CI Ion Source 120

To Install the Axial CI Ion Source 122

To Replace the Axial CI Ion Source Filament 124

This chapter describes maintenance procedures that are unique to an Agilent 7250 Accurate-Mass Quadrupole Time-of-Flight (Q-TOF) GC/MS System equipped with CI hardware.

## General Information

### Ion source cleaning

The main effect of operating the MS in CI mode is the need for more frequent ion source cleaning. In CI operation, the ion source chamber is subject to more rapid contamination than in EI operation because of the higher source pressures required for CI.

### 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 rotary vane foreline pump must be checked frequently and replaced every three months or as needed.

Always purge the MS with methane after using ammonia.

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 MS for CI operation

Setting up your MS for operation in CI mode requires special care to avoid contamination and air leaks.

#### **Guidelines**

- Before venting in EI mode for the installation of the CI source, verify that the GC/MS system is performing correctly.
- Ensure 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 Remove the Axial CI Source

### Materials needed

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



### Procedure

- 1 Vent the MS. (See **“To Vent the MS”** on page 57.)

#### WARNING

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

#### CAUTION

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

#### CAUTION

Use an antistatic wrist strap and take other antistatic precautions before touching analyzer components.

#### CAUTION

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

- 2 Open the analyzer chamber. (See **“To Open the Analyzer Cover for Access to the Analyzer Sideplate”** on page 106.)
- 3 Remove the two large thumbscrews that hold the ion source in place. (See **Figure 32**, “Axial CI ion source before removal,” on page 112.)
- 4 Disconnect the black, white, and gray filament wires and the red repeller wire from the ion source. Do not bend the wires any more than necessary. (See **“To Connect or Disconnect Wiring to the Axial CI Ion Source”** on page 113.)

## 6 CI Maintenance

### To Remove the Axial CI Source

- 5 Using the source finger grip, pull the ion source out of the source radiator. The source contacts have spring loaded pins so some force must be applied to pull the source out.

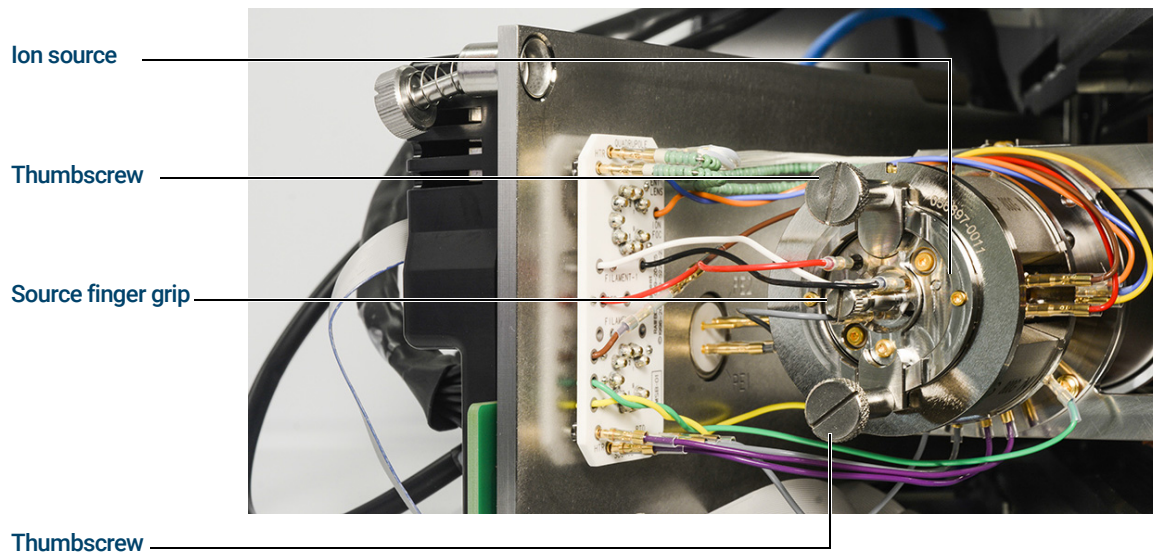


Figure 32. Axial CI ion source before removal



## 6 CI Maintenance

### To Connect or Disconnect Wiring to the Axial CI Ion Source

# To Connect or Disconnect Wiring to the Axial CI Ion Source

This topic is used to identify the wiring connections to the ion source. For detailed procedures and videos, see **“To Remove the Axial CI Source”** on page 111, or **“To Install the Axial CI Ion Source”** on page 122.

### Materials needed

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

### Procedure

Use tweezers or pliers to connect or disconnect the gray, white, and black filament leads and the red repeller lead from the source. (See **Figure 33.**)

#### Ceramic source board

- Filament (black)
- Common (white)
- Extractor (gray)
- Repeller (red)

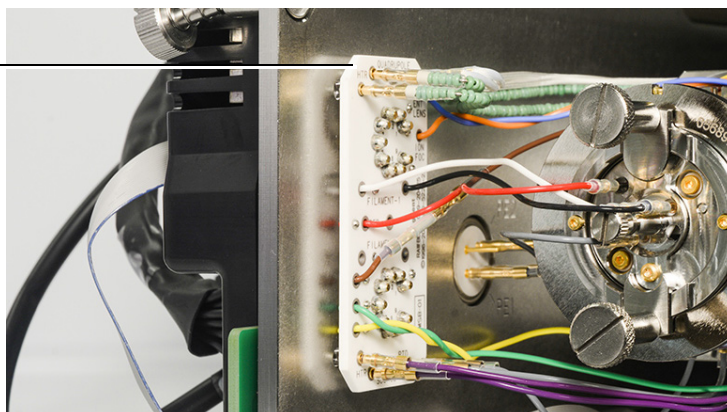


Figure 33. Wiring for the Axial CI ion source

## To Disassemble the Axial CI Ion Source

### Materials needed

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

Refer to **Figure 34**, “Exploded parts view of Axial CI source,” on page 115 and to **Table 12**, “Parts list for Axial CI ion source,” on page 116 while performing this procedure.

### CAUTION

Always wear clean gloves when working on the source to avoid contamination.



### Procedure

- 1 Place a clean cloth on your work surface to hold the ion source parts.
- 2 Use a T6 Torx screwdriver to remove the screw securing the filament block to the source mount, and using the finger grip, remove the filament block.

### CAUTION

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

- 3 Remove the filament from the filament block.
- 4 Use a T6 Torx screwdriver to remove the two gold screws securing the source mount to the source body. Do not lose the white ceramic insulators on the screws.
- 5 Remove the source mount, repeller, and heater sensor assembly from the source body. Carefully separate those parts.
- 6 Use a T6 Torx screwdriver to remove the gold screw and locking ring that secures the lens stack in the source body.
- 7 Remove the lens stack and drawout plate from the source body.

## 6 CI Maintenance

To Disassemble the Axial CI Ion Source

### CAUTION

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

- 8 Remove the cylinder from the lens insulator.
- 9 Remove the ion focus through the open side of the lens insulator. It does not slide out the end of the insulator.
- 10 Remove the entrance lens through the open side of the lens insulator. It does not slide out the end of the insulator.

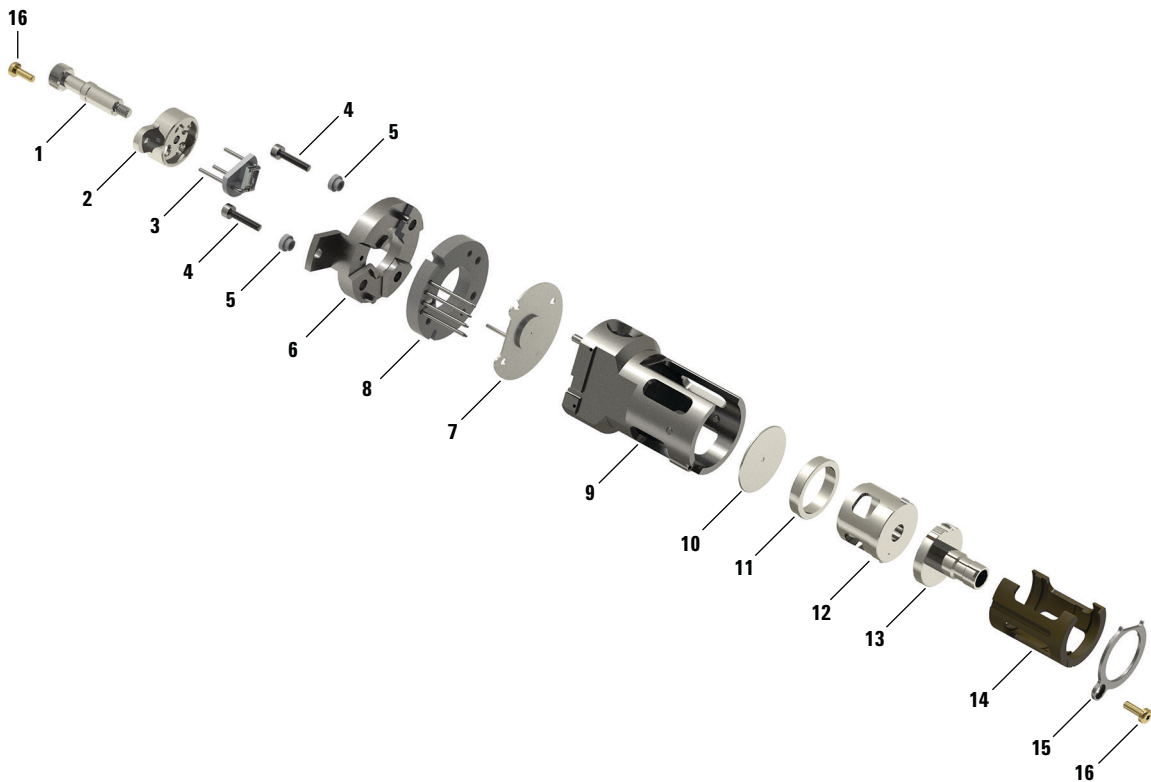


Figure 34. Exploded parts view of Axial CI source

## 6 CI Maintenance

### To Disassemble the Axial CI Ion Source

**Table 12** Parts list for Axial CI ion source

Item number	Item description	Part number
1	Source finger grip	G7002-20008
2	Filament block	G7002-20019
3	Filament, HES CI	G7250-60075
4	5973 Gold Fil Screw (M2 x 0.4 10MM-LG)	G1999-20021
5	in-W SHR-Shoulder 0.25 0.37 0.06 Alumina	3050-2670
6	HES CI source mount assembly	G7250-60009
7	HES CI repeller assy	G7250-60102
8	GCMS Ring heater/sensor assembly	G7002-60058
9	HES CI Body	G7250-20097
10	Drawout plate	G1999-20446
11	HES CI Drawout cylinder	G7250-20098
12	HES Ramp CI Ion focus	G7250-20096
13	Entrance lens assy, STD, Half-Ramped	G7250-20075
14	HES CI Lens insulator	G7250-20095
15	Locking ring for lens insulator	G7002-20126
16	Screw, M2 x 6 Pan Head Torx, gold plated	G7002-20109
Complete assembly	Axial CI Ion Source Assy (Support)	G7250-67404

# To Clean the Axial CI Ion Source

## Materials needed

- Abrasive paper (5061-5896)
- Alumina abrasive powder, 100 g (393706201)
- Aluminum foil, clean
- Cloths, clean (05980-60051)
- Cotton swabs (5080-5400)
- Glass beakers, 500 mL
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Solvents
  - Acetone, reagent grade
  - Methanol, reagent grade
  - Methylene chloride, reagent grade
- Ultrasonic bath

### CAUTION

Always wear clean gloves when working on the source to avoid contamination.



## Procedure

- 1 Disassemble the Axial CI ion source. (See **"To Disassemble the Axial CI Ion Source"** on page 114.)
- 2 Collect the following parts to be cleaned: (See **Figure 35**, "Axial CI ion source parts to be cleaned," on page 118.)
  - Entrance lens
  - Ion focus
  - Drawout plate
  - Source body
  - Repeller
  - Cylinder

## 6 CI Maintenance

### To Clean the Axial CI Ion Source

These are the parts that contact the sample or ion beam. The other parts normally should not require cleaning. If the contamination is serious, such as an oil backflow into the analyzer, consider replacing the contaminated parts.

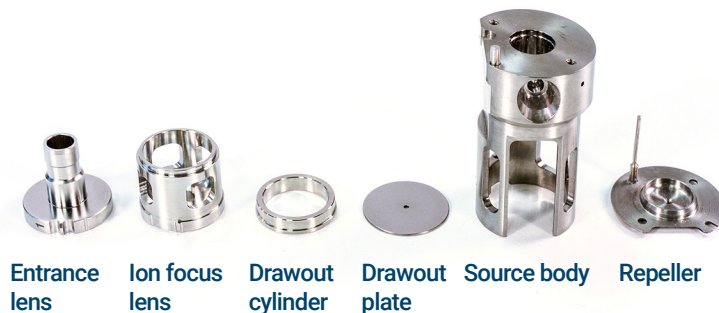


Figure 35. Axial CI ion source parts to be cleaned

#### 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.

#### CAUTION

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

- 3 Abrasively clean the surfaces that contact the sample or ion beam.

Use an abrasive slurry of alumina powder and reagent-grade methanol on a cotton swab. Use enough force to remove all discolorations. Polishing the parts is not necessary; small scratches will not harm performance. Also, abrasively clean the discolorations where electrons from the filament enter the source body.

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

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

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

## 6 CI Maintenance

### To Clean the Axial CI Ion Source

#### **WARNING**

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

- 6 Ultrasonically clean the parts, except the source mount, (each group separately) for 15 minutes in each of the following solvents:
  - Methylene chloride (reagent-grade)
  - Acetone (reagent-grade)
  - Methanol (reagent-grade)
- 7 Place the parts in a clean beaker. Loosely cover the beaker with clean aluminum foil (dull side down).
- 8 Dry the cleaned parts in an oven at 100 °C for 5-6 minutes.

## To Assemble the Axial CI Ion Source

### Materials needed

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

Refer to **Figure 33**, “Wiring for the Axial CI ion source,” on page 113 while performing this procedure.

### CAUTION

**Always wear clean gloves when working on the source to avoid contamination.**



### Procedure

### CAUTION

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

- 1 Install the entrance lens in the lens insulator. The entrance lens goes in through the open side of the insulator; it does not slide into the end.
- 2 Rotate the entrance lens until you feel the ball on the lens click into place.
- 3 Install the ion focus lens in the lens insulator. The ion focus goes in through the open side of the insulator; it does not slide into the end. The “closed” end of the ion focus should face the entrance lens.
- 4 Rotate the ion focus lens until you feel the ball on the lens click into place.
- 5 Slide the cylinder into the end of the lens insulator.
- 6 Slide the drawout plate into the source body.
- 7 Slide the lens stack into the source body. The open side of the lens insulator aligns with the open side of the source body.
- 8 Using the T6 Torx driver, install and secure the retaining ring and gold screw onto the end of the source body. Tighten the screw.



## 6 CI Maintenance

### To Assemble the Axial CI Ion Source

- 9 In order, install the repeller, heater sensor assembly, and source mount on the source body.

#### CAUTION

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

- 10 Make sure the white ceramic insulators are still present on the gold source mount screws, and then install and *gently* tighten the screws.
- 11 Insert the three filament leads into the side of the filament block opposite the finger grip.
- 12 Orient the filament so that the ceramic filter fits flush with the filament block.
- 13 Insert the filament block into the source holder, and use a T6 Torx screwdriver to secure it to the holder with the gold screw.

## To Install the Axial CI Ion Source

### Materials needed

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

### CAUTION

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

### CAUTION

Use an antistatic wrist strap and take other antistatic precautions before touching analyzer components.



### Procedure

- 1 If it is not already removed, remove the LE-EI ion source. (See **"To Remove the LE-EI Ion Source"** on page 85.)
- 2 Align the ion source so that the slotted opening for the lens stack, where the lens numbers are visible, is on the right side. Also, position it so the two attachment tabs of the source mount align with the corresponding slots in the source radiator. (See **Figure 36**, "The ion source radiator," on page 123.) Slide the ion source into the source radiator until resistance is felt.

## 6 CI Maintenance

### To Install the Axial CI Ion Source

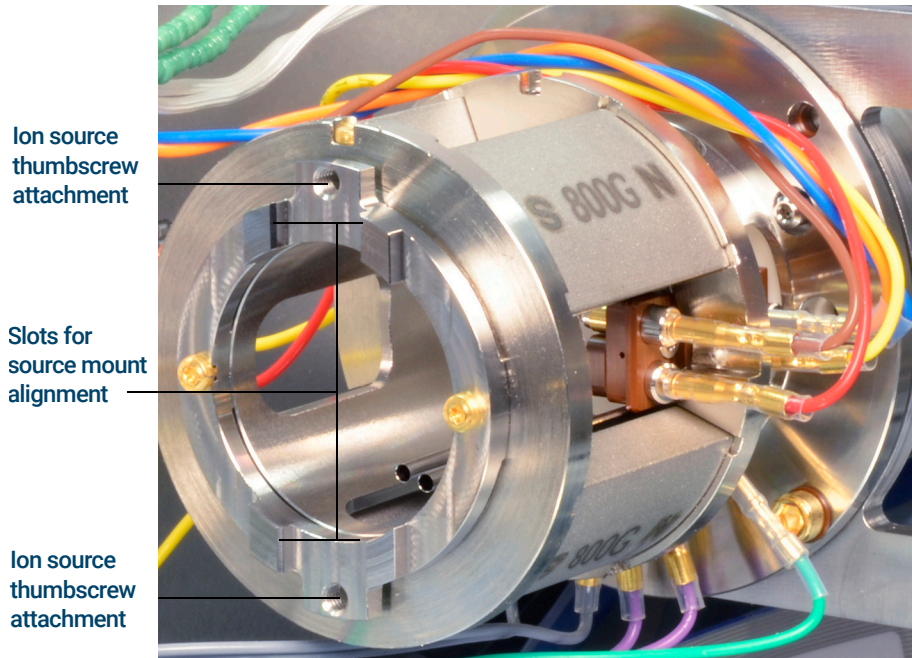


Figure 36. The ion source radiator

- 3 Snap the source into place so that the source mount tabs are flush against the source mount attachment surface of radiator. Some force is required to overcome the resistance of the electrical contacts' spring-loaded surfaces.
- 4 Connect the ion source wires. (See **"To Connect or Disconnect Wiring to the Axial CI Ion Source"** on page 113.)
- 5 Install and hand-tighten the source thumbscrews. Do not overtighten the thumbscrews.
- 6 Close the analyzer chamber.
- 7 Pump down the MS. (See **"To Pump Down the MS"** on page 54.)

With heated zones set to normal operating temperatures, allow the MS to bake out for at least two hours before running autotune or acquiring data. Bake out longer if samples being analyzed require maximum sensitivity.

- 8 Autotune the MS. (See **"CI Autotune"** on page 67 **"To Perform a CI Autotune"** on page 74.)

# To Replace the Axial CI Ion Source Filament

## Materials needed

- Filament assembly, 2-pk, CI (G7005-60072)
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Cloths, clean (05980-60051)
- T6 Torx driver (8710-2548)
- Tweezers (8710-2460)



## Procedure

- 1 Vent the MS. (See **“To Vent the MS”** on page 57.)

### WARNING

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

### CAUTION

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

- 2 Open the analyzer door. (See **“To Open the Analyzer Cover for Access to the Analyzer Sideplate”** on page 106.)
- 3 Remove the ion source and place it on a clean cloth on your work surface. (See **“To Remove the Axial CI Source”** on page 111.)
- 4 Remove the screw securing the filament block to the source mount. (See **Figure 37.**)

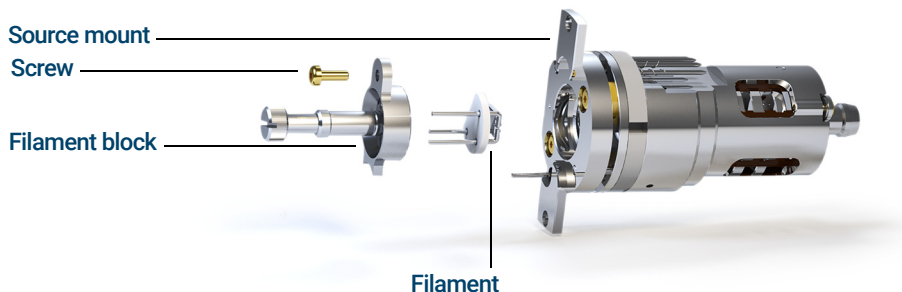


Figure 37. Replacing the filament

## 6 CI Maintenance

### To Replace the Axial CI Ion Source Filament

- 5 Use the hand grip on the filament block to remove the filament block from the source, by lifting the source body up off of the filament block, while holding the filament block so that the filament will not fall and become damaged.

#### CAUTION

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

- 6 Remove the filament from the filament block.
- 7 Insert the three pins on the filament through the rear of the filament block. (See **Figure 37**, "Replacing the filament," on page 124.)
- 8 Place the filament block in the source mount.
- 9 Use a T6 Torx screwdriver to tighten the screw securing the filament block to the source mount.
- 10 Reinstall the ion source. (See **"To Install the Axial CI Ion Source"** on page 122.)
- 11 Close the analyzer chamber.
- 12 Pump down the MS. (See **"To Pump Down the MS"** on page 54.)

With heated zones set to normal operating temperatures, allow the MS to bake out for at least two hours before running autotune or acquiring data. Bake out longer if samples being analyzed require maximum sensitivity.

- 13 Run PCI Autotune. (See **"To Perform a CI Autotune"** on page 74.) Verify performance first in PCI mode, even if you will ultimately be operating in NCI mode.

## 6 CI Maintenance

To Replace the Axial CI Ion Source Filament



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