Agilent 7200 Accurate-Mass Quadrupole Time-of-Flight GC/MS System

Operation Manual

Agilent Technologies

Notices

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Safety Notices

CAUTION

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

WARNING

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

About This Manual

This manual contains information for operating and maintaining the Agilent 7200 Accurate-Mass Quadrupole Time-of-Flight GC/MS system.

1 "Introduction"

Chapter 1 describes general information about the 7200 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 Electron Impact (EI) Mode"

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

4 "Operating in Chemical Ionization (CI) Mode"

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

5 "Using the Removable Ion Source"

Chapter 5 shows you how to change the removable ion source.

6 "General Maintenance"

Chapter 6 describes general maintenance procedures for the 7200 Q-TOF GC/MS.

Hardware User Information

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

The hardware user information DVDs that ship with your instrument provide an extensive collection of online help, videos, and books for the Agilent **7890A GC**, **7200 Q-TOF GC/MS**, **7693 ALS**, and the **7683B ALS**. Included are localized versions of the information you need most, such as:

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

Contents

1
Introduction

This section provides general information about the 7200 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
BFB	Bromofluorobenzene (calibrant)
CC	Collision cell
CI	Chemical ionization
DC	Direct current
DFTPP	Decafluorotriphenylphosphine (calibrant)
EI	Electron impact
EPC	Electronic pneumatic control
eV	Electron volt
GC	Gas chromatograph
id	Inside diameter
LAN	Local Area Network
m/z	Mass to charge ratio
MFC	Mass flow controller
MS	Mass spectrometer
NCI	Negative chemical ionization
OFN	Octafluoronaphthalene (sample)
PCI	Positive chemical ionization
PFDTD	Perfluoro-5,8-dimethyl-3,6,9-trioxydodecane (calibrant)
PFTBA	Perfluorotributylamine (calibrant)

Table 1 Abbreviations (continued)

Abbreviation	Definition
Q-TOF	Quadrupole time-of-flight
Quad	Quadrupole mass filter
RF	Radio frequency
RFPA	Radio frequency power amplifier
TOF	Time-of-flight
Torr	Unit of pressure, 1 mm Hg
Turbo	Turbomolecular vacuum pump

The 7200 Accurate-Mass Quadrupole Time-of-Flight GC/MS System

The 7200 Accurate-Mass Quadrupole Time-of-Flight (Q-TOF) GC/MS System is a standalone capillary GC detector for use with the Agilent 7890A Series gas chromatograph. The 7200 Q-TOF features:

- Three turbomolecular vacuum pumps
- Rotary vane foreline pump
- Independently MS-heated EI or CI ion source
- Removable ion source (RIS) probe with RIS bayonet and cooling chamber, which allows quick change from EI to CI ion source with minimal loss of vacuum in the instrument
- 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 with automatic retraction during ion source removal

Physical description

The 7200 Q-TOF GC/MS is approximately 48 cm high, 71 cm wide, and 89 cm deep excluding the flight tube and RIS handle. The flight tube extends 84 cm up over the top of the instrument. The RIS handle, when attached, extends 48 cm from the front of the instrument.

The weight of the instrument is 152 kg for the mainframe. The attached foreline (roughing) pump weighs an additional 11 kg.

The basic components of the instrument are: the frame/cover assemblies, the vacuum system, the GC/MS interface, the removable ion source, the flight tube electronics, the quadrupole mass filter, the collision cell, and the detector.

Vacuum gauge

The 7200 Q-TOF GC/MS is equipped with four ion vacuum gauges. The MassHunter Workstation can be used to read the pressure (high vacuum) in the vacuum manifold, the turbomolecular vacuum pump discharge, and the flight tube.

Ionization modes

The 7200 Accurate-Mass Q-TOF GC/MS can be purchased in two different ionization configurations:

- G3850AA 7200 Q-TOF GC/MS EI RIS Bundle
- G3851AA 7200 Q-TOF GC/MS EI/CI RIS Bundle

An upgrade kit is available, which allows the user to bring an EI system to an EI/CI system. This kit adds to the 7200 Q-TOF GC/MS:

- CI ion source
- Reagent gas flow control module plus associated tubing and cabeling
- Utilities panel update
- New ribs for cover support
- New routing for the IRM assembly

For the CI system, a methane/isobutane gas purifier is provided and is required. It removes oxygen, water, hydrocarbons, and sulfur compounds.

The MS CI system has been optimized to achieve the relatively high ion source pressure required for CI while still maintaining high vacuum in the collision cell, quadrupole, and TOF tube. Special seals along the flow path of the reagent gas and very small openings in the ion source keep the ion source gases in the ionization volume long enough for the appropriate reactions to occur. The interface has special plumbing for reagent gas. A retractable insulating seal fits onto the tip of the interface and is used for both CI and EI.

Switching back and forth between CI and EI ion sources takes less than 30 minutes with the new removable ion source. The RIS allows the instrument to remain under vacuum, and provides a cooling chamber with N2 purge for rapid ion source cooling without venting the machine. This saves hours in cycle time over the traditional unit.

Hardware Description

Figure 1 is an overview of a typical Agilent 7200 Accurate-Mass Q-TOF GC/MS system.

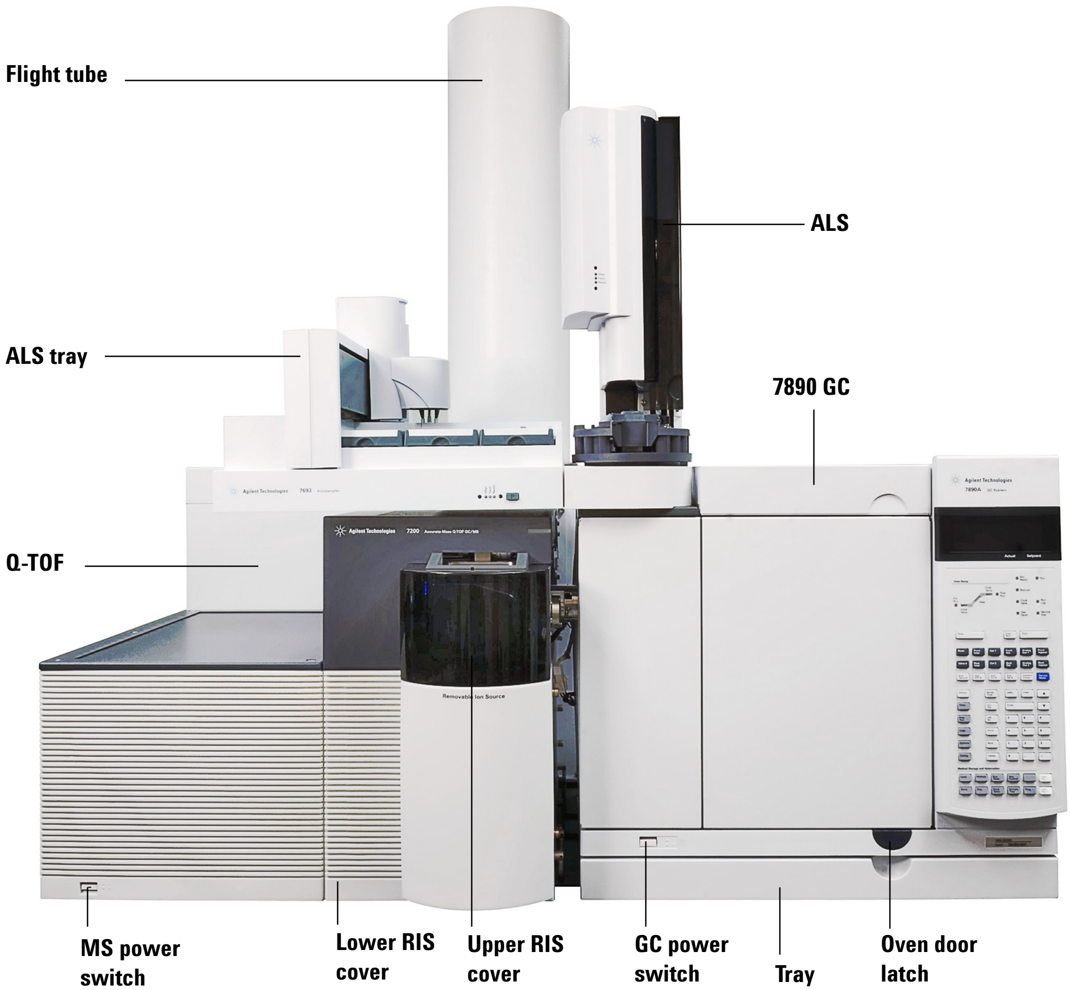


Figure 1 7200 Q-TOF GC/MS System

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.

Precautions to take to prevent an explosion

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

WARNING **You MUST make sure the top thumbscrew on the front analyzer side plate and the top thumbscrew on the rear analyzer side plate are both fastened finger-tight. Do not overtighten 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 MS interface

- Foreline pump
- GC/MS transfer line

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.**

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 standard foreline pump can be a fire hazard

Oily rags, paper towels, and similar absorbents in the oil pan could ignite and damage the pump and other parts of the 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, but do not leave absorbent material such as paper towels in it.**

Safety and Regulatory Certifications

The 7200 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 7200 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 7200 Q-TOF GC/MS is designed and manufactured under a quality system registered to ISO 9001.

Information

The Agilent Technologies 7200 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.

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.

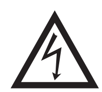
Symbols

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.

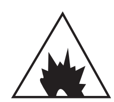
Indicates a hot surface.

Indicates hazardous voltages.

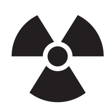


Indicates earth (ground) terminal.

Indicates potential explosion hazard.



Indicates radioactivity hazard.



Indicates electrostatic discharge hazard.

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

Electromagnetic compatibility

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

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

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

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

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

Sound emission declaration

Sound pressure

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

Liquid Spills

Do not spill liquids on the MS.

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 7200 Q-TOF GC/MS Troubleshooting and Maintenance Manual.*

2
Installing GC Columns

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.

Columns

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 the Column” on page 58 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 for EI mode and Table 6 for CI mode.

Table 2 Gas flows

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

* Total gas flow into the MS = column flow + collision cell gas flow + reagent gas flow (if applicable) + Agilent Quick Swap flow (if applicable)

† 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.

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 7200 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.**

To Prepare a Capillary Column for Installation

Materials needed

- Capillary column
- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Ferrules
    - 0.27 mm id, for 0.10 mm id columns (5062-3518)
    - 0.37 mm id, for 0.20 mm id columns (5062-3516)
    - 0.40 mm id, for 0.25 mm id columns (5181-3323)
    - 0.5 mm id, for 0.32 mm id columns (5062-3514)
    - 0.8 mm id, for 0.53 mm id columns (5062-3512)
- Gloves, clean
    - Large (8650-0030)
    - Small (8650-0029)
- Inlet column nut (5181-8830 for Agilent 7890A)
- Loupe
- Septum (may be old, used inlet septum)

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.**

Procedure

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

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

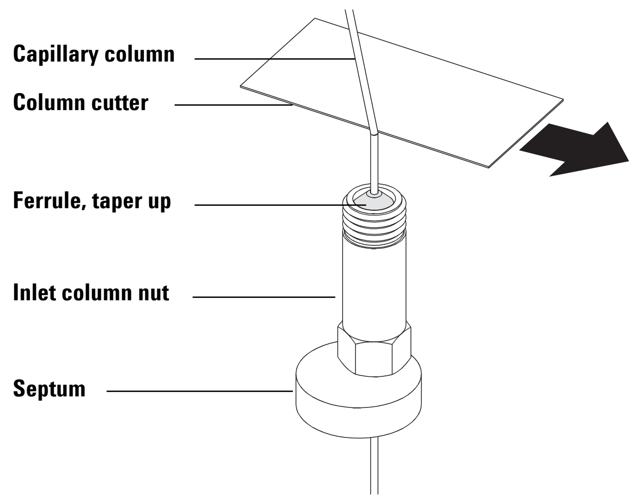


Figure 2 Preparing a capillary column for installation

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.

To Install a Capillary Column in a Split/Splitless Inlet

Materials needed

- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Metric ruler
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)

To install columns in other types of inlets, refer to your gas chromatograph 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. Prepare the column for installation. (See “To Prepare a Capillary Column for Installation” on page 29.)
2. Position the septum under the column nut so that the column extends 4 to 6 mm past the end of the ferrule (Figure 3).
3. Insert the column in the inlet.
4. Slide the nut up the column to the inlet base and finger-tighten the nut.
5. Adjust the column position so the septum is even with the bottom of the column nut.
6. Tighten the column nut an additional 1/4 to 1/2 turn. The column should not slide with a gentle tug.
7. Start carrier gas flow.
8. Verify flow by submerging the free end of the column in isopropanol. Look for bubbles.

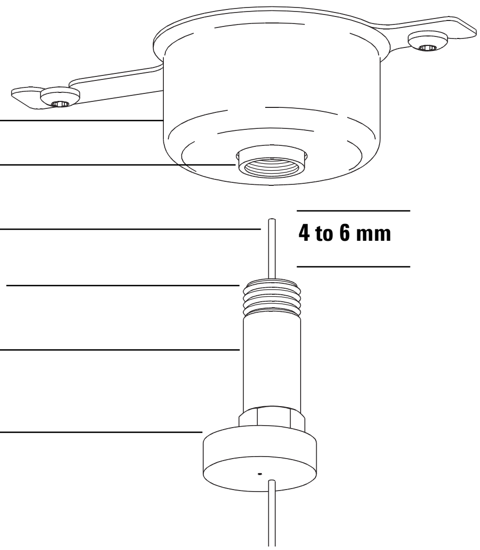


Figure 3 Installing a capillary column for a split/splitless inlet

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 31.)
2. Set a minimum velocity of 30 cm/s, or as recommended by the column manufacturer. Allow gas to flow through the column at room temperature for 15 to 30 minutes to remove air.
3. Program the oven from room temperature to the maximum temperature limit for the column.
4. Increase the temperature at a rate of 10 to 15 °C/min.
5. Hold at the maximum temperature for 30 minutes.

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

6. Set the GC oven temperature to 30 °C and wait for the GC to become ready.
7. Attach the column to the GC interface. (See “To Install a Capillary Column in the GC/MS Interface” on page 35.)

See also

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

To Install a Capillary Column in the GC/MS Interface

This procedure is for the installation of a capillary column directly to the transfer line with a column nut. If you are using the Agilent capillary flow technology Quick Swap accessory, or a Purged Ultimate Union (PUU) see “To Prepare the Column Ends for a CFT Fitting” on page 38.

Agilent 7890A GC

Materials needed

- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Ferrules
  - 0.3 mm id, for 0.10 mm id columns (5062-3507)
  - 0.4 mm id, for 0.20 and 0.25 mm id columns (5062-3508)
  - 0.5 mm id, for 0.32 mm id columns (5062-3506)
  - 0.8 mm id, for 0.53 mm id columns (5062-3512)
- Flashlight
- Magnifying loupe
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Interface column nut (05988-20066)
- Safety glasses
- Wrench, open-end, 1/4-inch and 5/16-inch (8710-0510)
- Column measuring tool

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

Procedure

1 Condition the column. (See “To Condition a Capillary Column” on page 33.)

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

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

2 If you are not using Quick Swap, vent the MS. To vent the MS, see “To Vent the MS” on page 69.

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

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

4 Insert the column into the column measuring tool and allow the end of the column to project 10–15 mm past the end of the tool.

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

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

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

8 Wipe the end with alcohol.

9. Slide the column so that the end projects 1–2 mm past the end of the tool.
10. Finger tighten the fitting.
11. Tighten 1/4 to 1/2 turn to fix the ferrule to the column.
12. Slide the column into the GC/MS interface.
13. Hand-tighten the nut. Make sure the position of the column does not change as you tighten the nut.
14. Check the GC oven to be sure that the column does not touch the oven walls.
15. Tighten the nut 1/4 to 1/2 turn.
16. Check the nut's tightness after one or two heat cycles; retighten as appropriate.

To Prepare the Column Ends for a CFT Fitting

Materials needed

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

Procedure

1 Obtain the appropriate metal ferrule.

2 Pass the tubing end through the internal nut and SilTite ferrule leaving approximately 1 cm of fused silica tubing protruding beyond the ferrule. Thread the ferrule pre-swaging tool onto the internal nut with the tube protruding (see Figure 4).

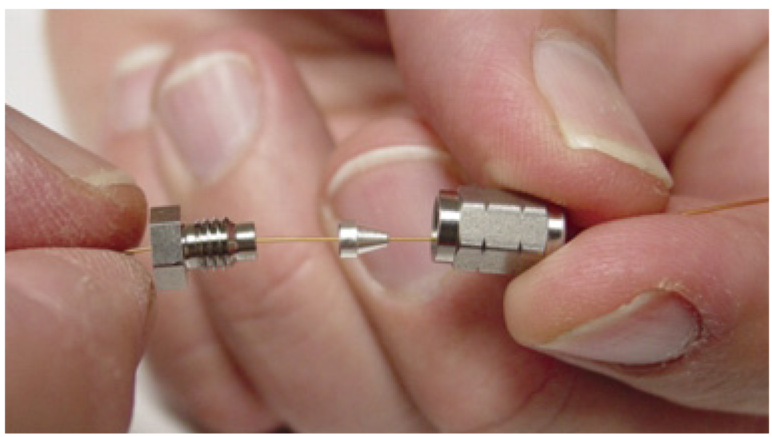


Figure 4 Assemble the internal nut, ferrule, and swaging nut

CAUTION The SilTite ferrules are delicate. Follow the instructions in the next step very carefully to avoid overtightening.

3 Using the wrench and ferrule pre-swaging tool, tighten the nut a little at a time, occasionally checking to see if the ferrule is gripping the tube (see Figure 5). When the ferrule just starts to grip, notice position of the nut and then tighten by turning 45 to 60 degrees of rotation, but no more than 60 degrees (one flat). If you can pull the column free, it is not tight enough.

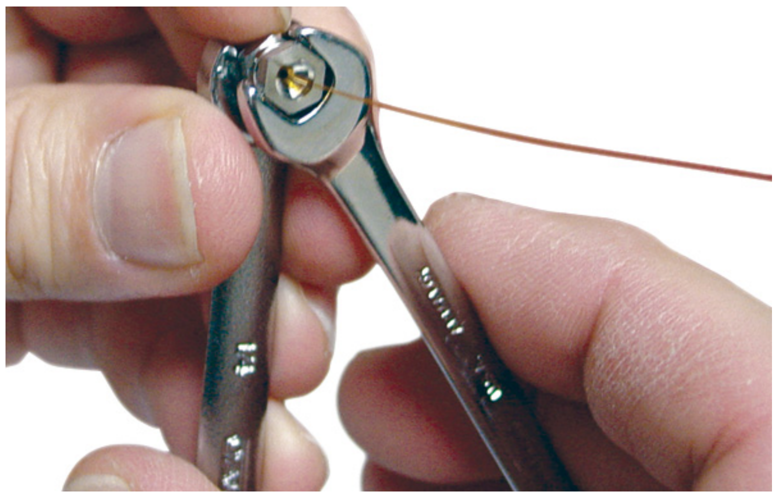


Figure 5 Tightening the internal nut

4 Remove the swaging tool (Figure 6).

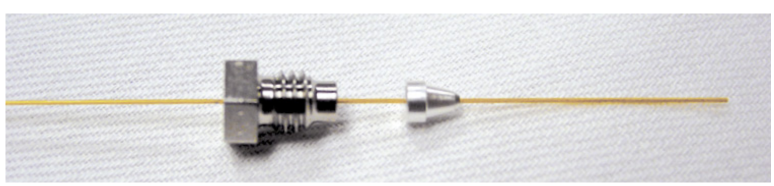


Figure 6 Removed ferrule pre-swaging tool

5 Using a ceramic column cutter, trim the tubing at the small end of the ferrule leaving approximately 0.3 mm of tubing extending beyond the ferrule (Figure 7).

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

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

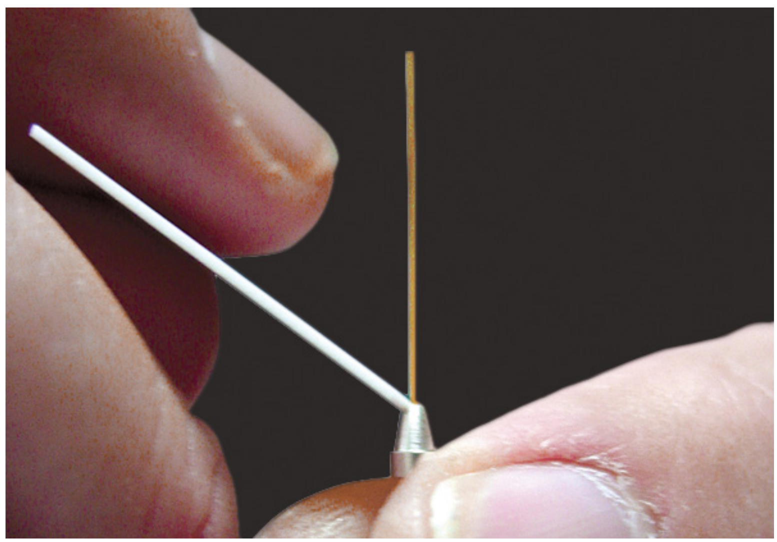


Figure 7 Ceramic column cutter and ferrule

6 Check the end of the tube with a magnifier. The end of the tube need not be perfectly square, but should not have cracks which extend under the ferrule. Figure 8 shows a completed tube end.

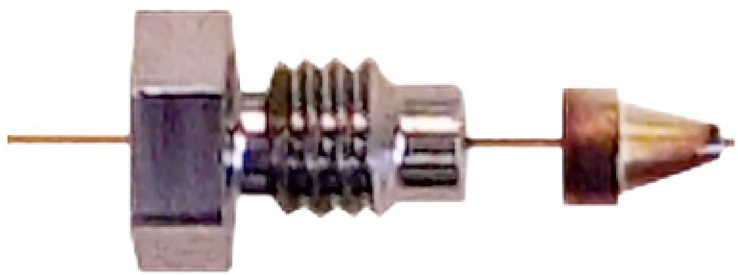


Figure 8 Tube end with internal nut and swaged SilTite ferrule

7 Connect column to the Quick Swap or PUU with internal nuts and preswaged SilTite ferrules (Figure 9). Finger-tighten the nuts. Further tighten with a wrench only 15 to 20 degrees.

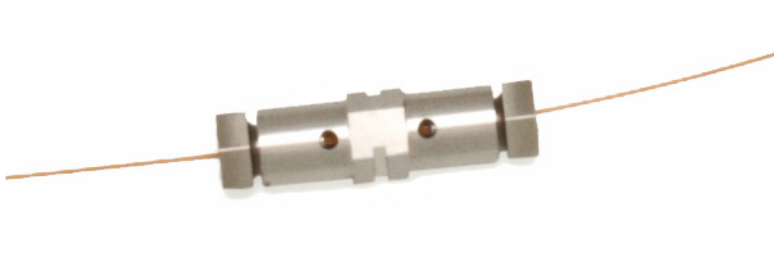


Figure 9 Completed Ultimate union with columns

3
Operating in Electron Impact (EI) Mode

This chapter will explain how to perform some routine operating procedures for the 7200 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, removing the ion source, 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 the steps in these procedures do not match your MassHunter Workstation software, refer to the manuals and online help supplied with the software for more information.

Your 7200 Q-TOF GC/MS is available in two configurations. The 7200 Q-TOF GC/MS EI RIS Bundle (G3850AA) operates in EI mode with the high sensitivity extractor EI removable ion source (RIS). The 7200 Q-TOF GC/MS EI/CI RIS Bundle (G3851AA) provides the capability of operating in either EI or CI modes, using the new RIS technology. You must specify the type of ionization you are using in the Acquisition software tune file used by your method.

The GC/MS Interface

The GC/MS interface (Figure 10) is a heated conduit into the MS for the capillary column. 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 1 to 2 mm of the capillary column extend past the end of the guide tube and into the ionization chamber. When changing the removable ion source, the interface is automatically retracted from the ion source by the MassHunter software. This allows the ion source to slide freely in and out of the analyzer 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 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 35.

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

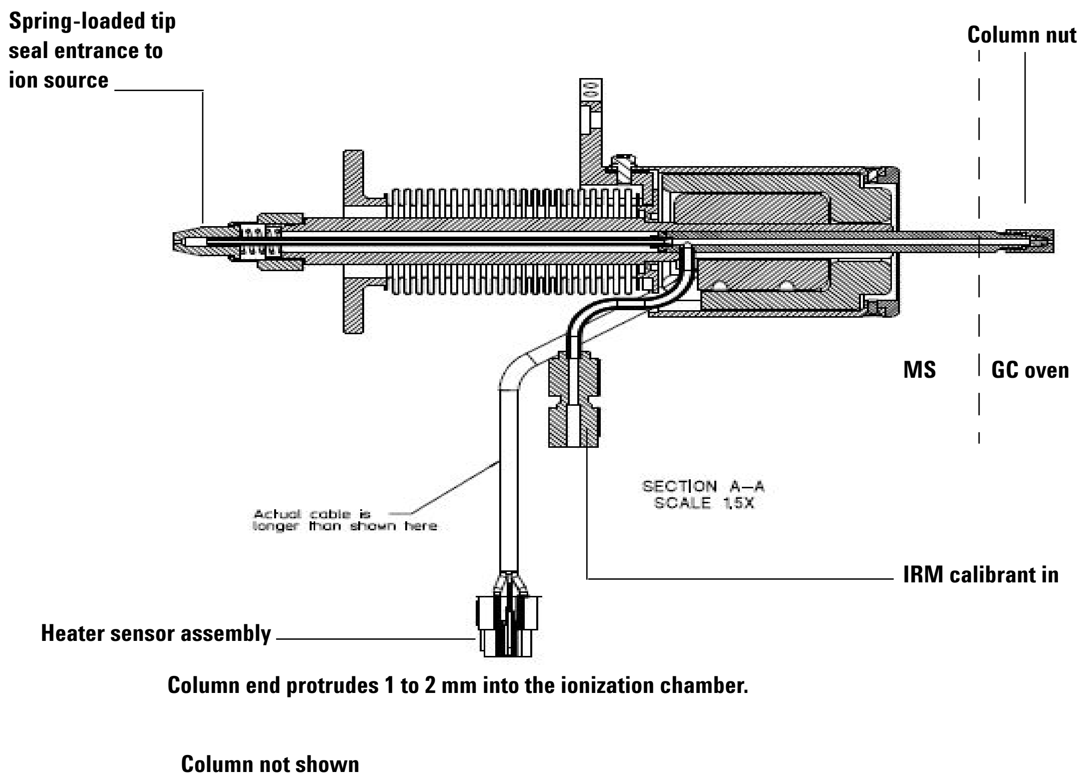


Figure 10 The GC/MS interface

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 or reagent gases are being used.
- 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. Be sure to 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

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 keypad.

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.

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 the Column” on page 58.

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. The gas pressure at the EPC outlet controls the flow of the gas. This pressure is controlled by the MassHunter Data Acquisition Workstation or directly at the GC keypad. See “To Set the Collision Cell Gas Flow Rate” on page 61.

Venting the MS

A program in the data system automates the venting process. It turns off the GC and MS heaters and the turbo pump at the correct time.

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

WARNING

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 vent valve or remove the column nut and column.

Do not exceed the maximum recommended total gas flow. See Table 2.

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

	CC Gas On N2 = 1.5 mL/min			CC Gas Off		
Column Flow (mL/min)	**Rough Vac**	**Quad Vac**	**TOF Vac**	**Rough Vac**	**Quad Vac**	**TOF Vac**
0.5	$1.16 * 10^{-1}$	$3.41 * 10^{-5}$	$4.20* 10^{-7}$	$8.29 * 10^{-2}$	$3.17 * 10^{-7}$	$2.15 * 10^{-7}$
0.7	$1.18 * 10^{-1}$	$3.41 * 10^{-5}$	$4.20 * 10^{-7}$	$8.67 * 10^{-2}$	$3.45 * 10^{-7}$	$2.15 * 10^{-7}$
1	$1.22 * 10^{-1}$	$3.43 * 10^{-5}$	$4.22 * 10^{-7}$	$9.17 * 10^{-2}$	$3.79 * 10^{-7}$	$2.16 * 10^{-7}$
1.2	$1.24 * 10^{-1}$	$3.43 * 10^{-5}$	$4.22 * 10^{-7}$	$9.49 * 10^{-2}$	$4.06 * 10^{-7}$	$2.16 * 10^{-7}$
2	$1.32 * 10^{-1}$	$3.44 * 10^{-5}$	$4.22 * 10^{-7}$	$1.07 * 10^{-1}$	$5.17 * 10^{-7}$	$2.17 * 10^{-7}$
3	$1.42 * 10^{-1}$	$3.44 * 10^{-5}$	$4.25 * 10^{-7}$	$1.19 * 10^{-1}$	$6.51 * 10^{-7}$	$2.20 * 10^{-7}$
4	$1.50 * 10^{-1}$	$3.46 * 10^{-5}$	$4.27 * 10^{-7}$	$1.29 * 10^{-1}$	$7.84 * 10^{-7}$	$2.21 * 10^{-7}$

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 In the **Instrument Control** panel of the MassHunter Workstation Acquisition software, click on the Method icon.

2 Select **Method > Edit Monitors** to display the **Select Monitors** dialog box. See Figure 11.

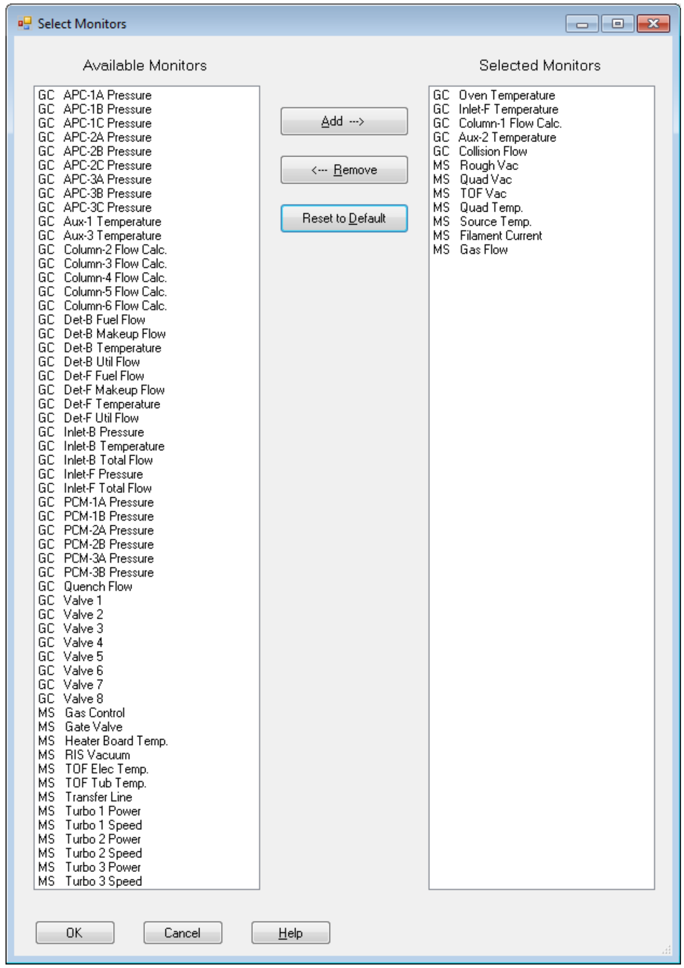


Figure 11 Select Monitors dialog box

3 In the **Available Monitors** column, select **MS Rough Vac** and click the **Add** button to move the selection to the **Selected Monitors** column.

4 In the **Available Monitors** column, select **MS Quad Vac** and click the **Add** button to move the selection to the **Selected Monitors** column.

5 In the **Available Monitors** column, select **MS TOF Vac** and click the **Add** button to move the selection to the **Selected Monitors** column.

6 In the **Available Monitors** column, select **MS Quad Temp** and click the **Add** button to move the selection to the **Selected Monitors** column.

7 In the **Available Monitors** column, select **MS Source Temp** and click the **Add** button to move the selection to the **Selected Monitors** column.

8 Select any other monitors you want and add them to the **Selected Monitors** column.

9 Click **OK**. The new monitors will be stacked on top of each other in the lower right corner of the **Instrument Control** window.

10 Select **Window > Arrange Plots and Monitors**, or click and drag each monitor to the desired position. See Figure 12 for one way of arranging the monitors.

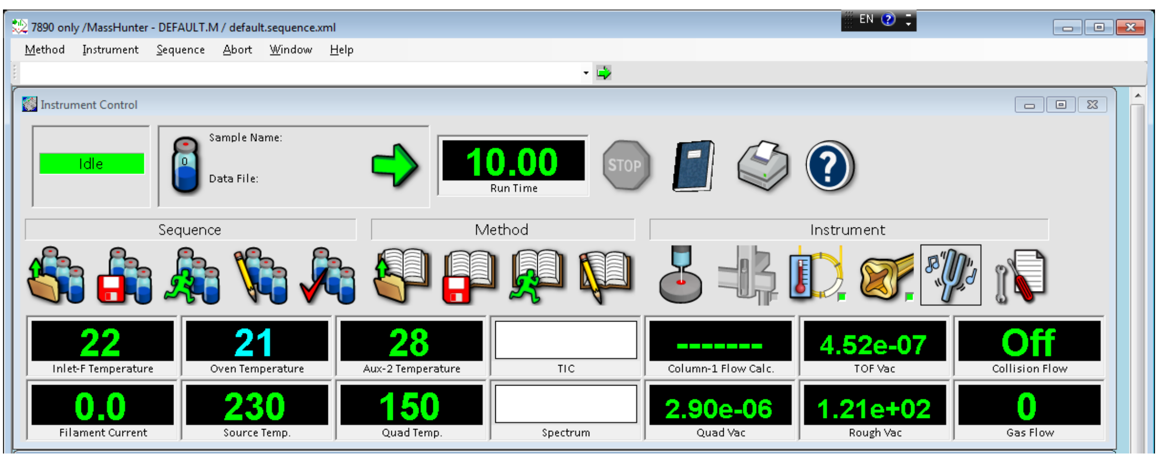


Figure 12 Arranging monitors

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

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.
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
MS Quad	150 °C

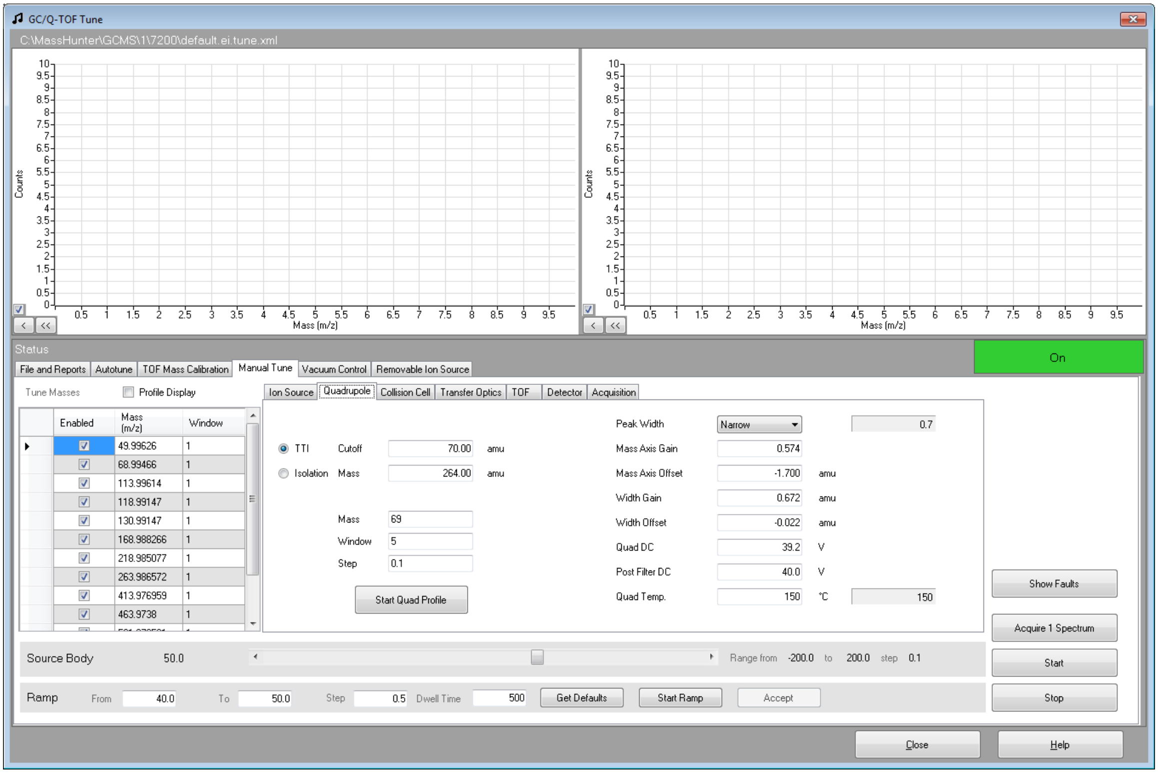


Figure 13 Setting temperatures

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.

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

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 the **Aux Temperatures** icon to edit the interface temperature (Figure 14). This example has the GC/MS interface temperature configured as **Thermal Aux 2**.

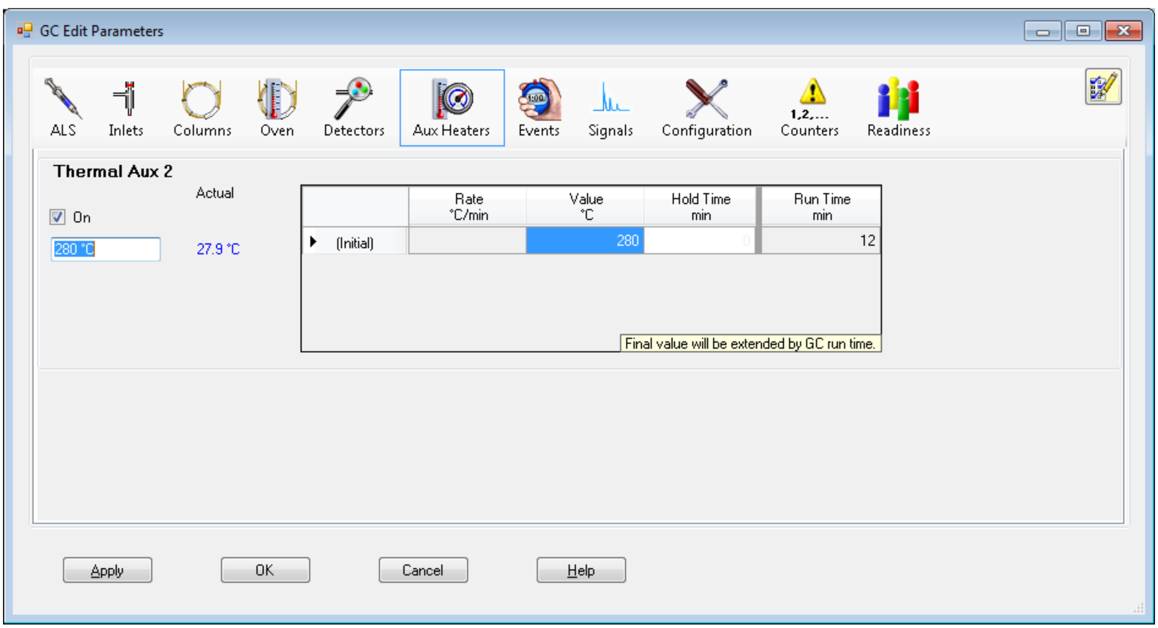


Figure 14 Setting the interface temperature

CAUTION Make sure 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.

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

3 Check the heater **On** 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 the Column

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

Procedure

1 Set Data Acquisition for splitless manual injection and set up a real time plot to monitor *m/z* 28.

2 Press **[Prep Run]** on the GC keypad.

3 Inject 1 µL of air into the GC inlet and press **[Start Run]**

4 Wait until a peak elutes at *m/z* 28. Note the retention time.

5 In the **Instrument Control** panel, select  .

6 Select the **Configuration** tab.

7 Select the **Column** tab and click on the **Inventory** button and verify that the column you are using is in inventory. Select the column to be calibrated and click **Install Selected Column**.

8 Highlight the column in the inventory list and select the **Calibrate** button.

9 Select the **Calc Length** button.

10 In the **Calculate Column Length** dialog box, enter the recorded retention time in the **Holdup Time** field. 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.

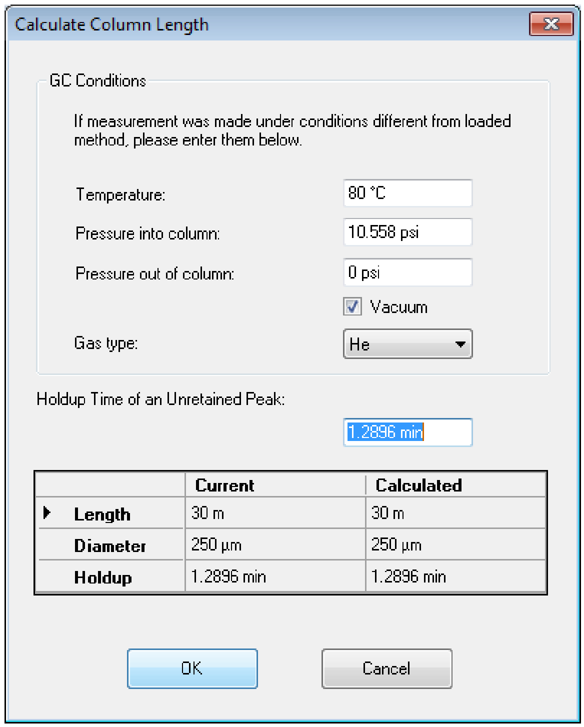
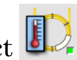


Figure 15 Calculate Column Length dialog box

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.

To Configure the Collision Cell Gas

1 From the **MassHunter Data Acquisition Workstation Instrument Control** panel, select .

2 Select the **Modules** tab to display the screen. See Figure 16.

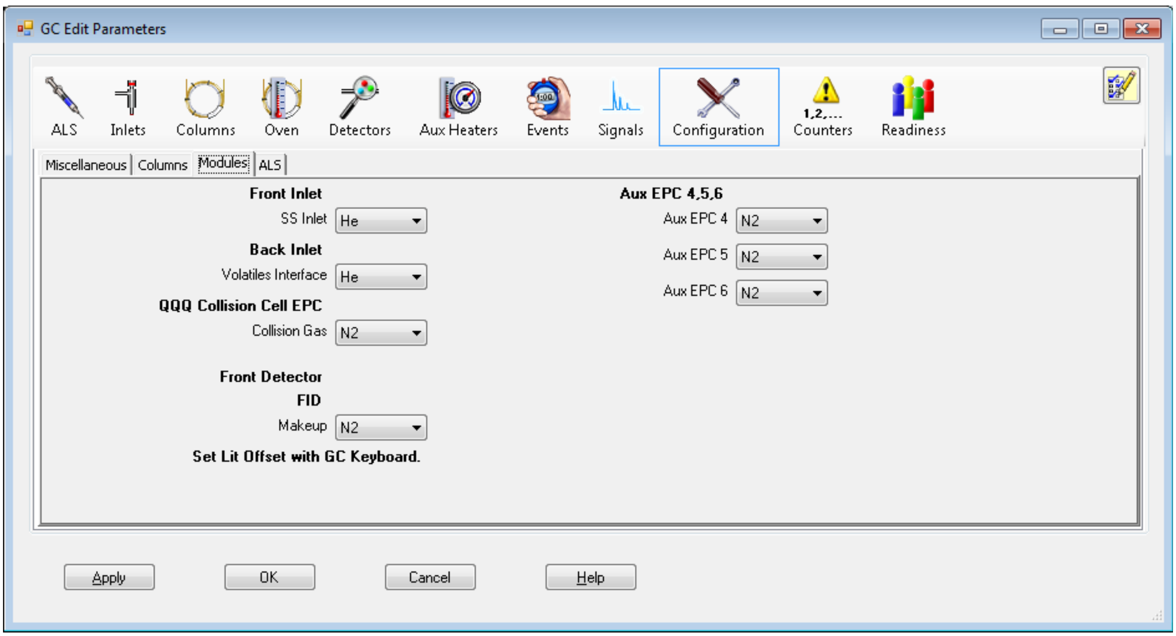


Figure 16 Configure collision cell gas

3 From the **Collision Cell EPC** drop-down menu, select **N2** as the collision cell gas.

4 Click **OK** to save the configuration.

To Set the Collision Cell Gas Flow Rate

1 From the MassHunter Data Acquisition Workstation **Instrument Control** panel, select **Instrument > GC Parameters**.
2 Click the **CFT** icon to display the **CFT** screen. See Figure 17.
3 Select **Collision Cell EPC** in the description list.
4 Enter the required gas flow rate in the **N2 Collision Gas** field.
5 Click the **N2 Collision Gas On** check box to allow the N2 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.

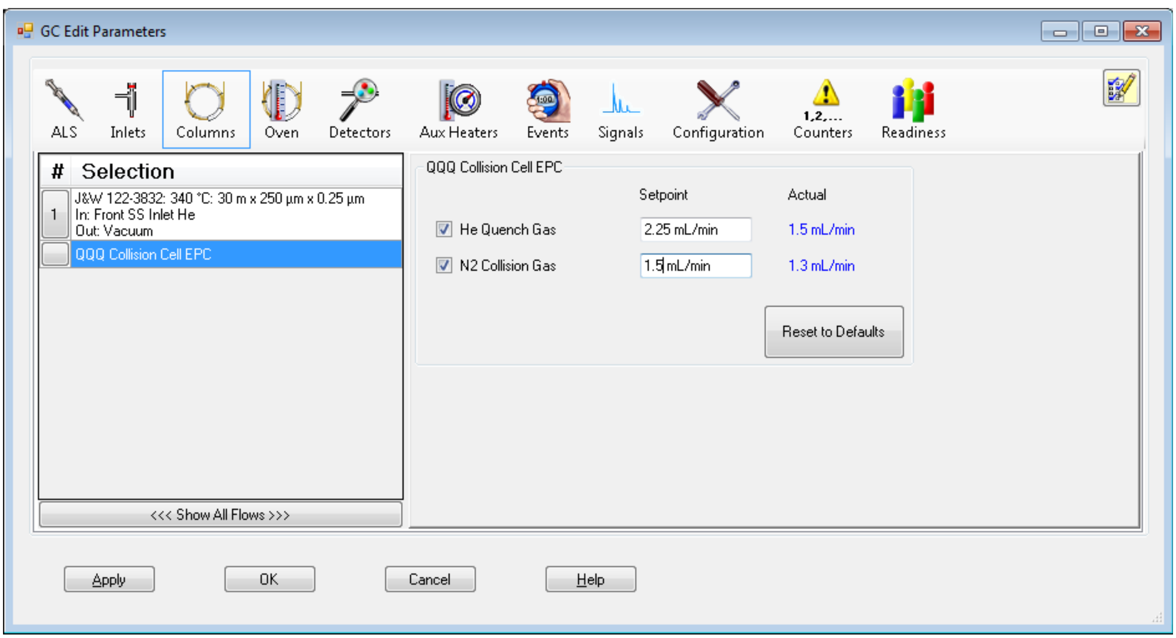


Figure 17 Setting collision cell gas flow rate

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 and 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 box.
3 The current tune file is displayed in the upper left corner of the **GC/Q-TOF Tune** dialog box. 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 on the **Load** button in the **Tune File** area. Select a tune file and click the **OK** button.

   The tune file must match the type of ion source in the analyzer. If you are using an EI ion source, select a tune file created for an EI ion source.
5 Click the **Autotune** tab and select an **EI high sensitivity autotune** for the high sensitivity EI ion source with a variable voltage extractor.
6 Select the **Tune from default settings** check box 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.
7 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.
8 Select the **Print autotune report** check box to automatically print a tune report.
9 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.

10 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.

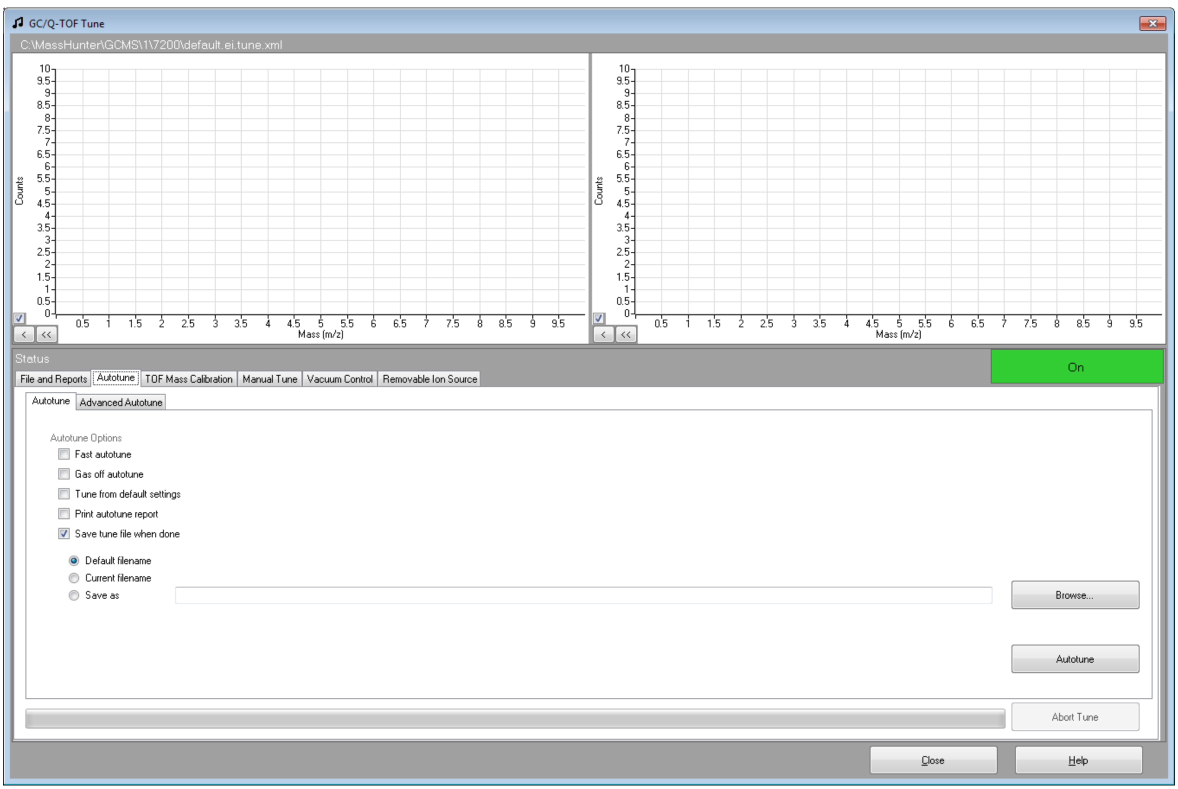


Figure 18 EI Autotune

To Remove the Upper RIS Cover

The upper RIS cover is in front of the instrument (Figure 19). Remove it to access the latches on the analyzer cover, attach the RIS probe, or access the EI calibration vial. Do not remove the upper RIS cover for any other reasons.

Procedure

1 Grasp the cover by the outside corners.

2 Pull the cover straight up, and remove it from the instrument.

Figure 19 Upper RIS cover

To Open the Analyzer Cover for Access to the Analyzer Sideplate

Open the analyzer cover (Figure 20) to access the analyzer sideplate only. This is necessary during pump down or to access the analyzer sideplate thumbscrews. You should not open the analyzer cover for any other reason.

Procedure

1 Remove the upper RIS cover to expose the analyzer cover latches. See “To Remove the Upper RIS Cover” on page 64.
2 Open the latches on the analyzer cover at the front of the instrument.
3 Swing the analyzer cover open.

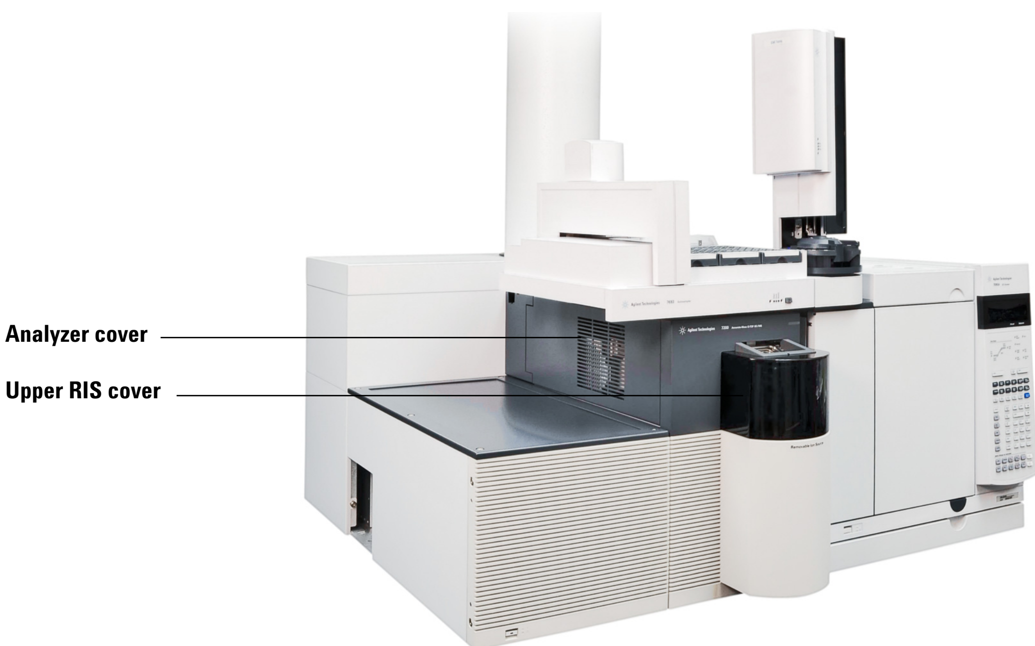


Figure 20 Covers

To Pump Down the MS

WARNING **Make sure 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 65.

2 Plug the power cord into a grounded electrical outlet.

3 Turn on the Q-TOF power switch (Figure 20).

4 Press lightly on the metal box on the quad driver board to ensure a correct seal.

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. If the Q-TOF was configured for both an EI and a CI ion source, you are prompted for the ion source type that is currently installed. Click on an EI or CI ion source type if prompted.

6 Select the **MS Tune** icon from the **Instrument Control** panel.

7 Select the **Vacuum Control** tab.

8 Click the **Pumpdown** button.

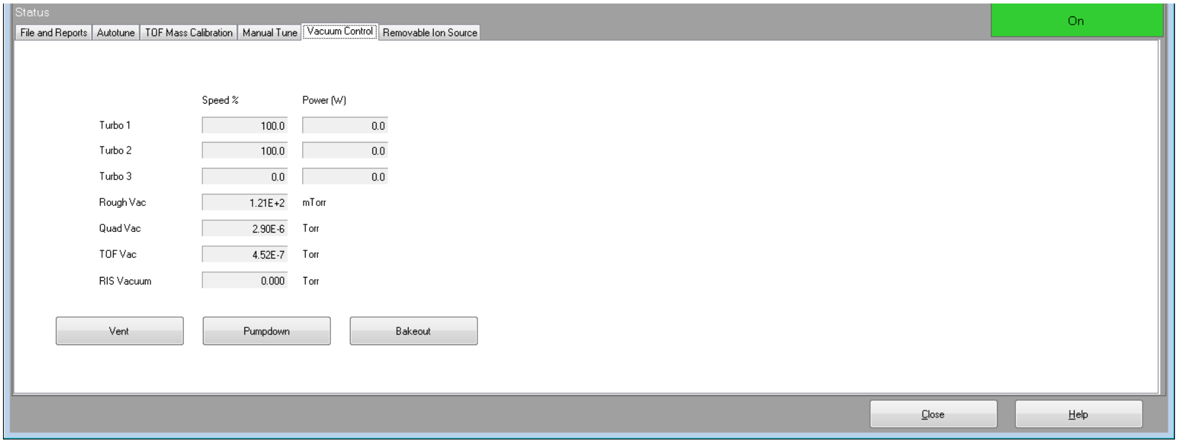


Figure 21 Vacuum Control tab

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% (Figure 21). 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.

9 Turn on the carrier gas flow after the vacuum pumps have been running for 15 minutes.

10 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.

11 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.

12 Tune the MS. (See “To Autotune the MS for EI Mode” on page 62 or “To Perform a CI Autotune” on page 88.)

To Vent the MS

Procedure

1 Click the **MS Tune** icon from the **Instrument Control** panel.
2 Select the **Vacuum Control** tab. See Figure 21.
3 Set the GC/MS interface heater and the GC oven temperatures to ambient (room temperature).
4 When the GC temperatures have reached 30 °C, turn off the flow of carrier gas.
5 Click the **Vent** button.

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

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

6 Turn off the MS by pressing the power switch. (See Figure 1.)
7 Unplug the MS power cord.

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.

4
Operating in Chemical Ionization (CI) Mode

This chapter provides information and instructions for operating the 7200 QTOF 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.

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.
- Make sure the CI ion source is installed.
- Make sure the reagent gas plumbing has no air leaks. This is determined in PCI mode, checking for *m/z* 32 after the methane pretune.
- Make sure the reagent gas inlet line is equipped with gas purifiers (not applicable for ammonia).

The CI GC/MS Interface

The CI GC/MS interface (Figure 22) 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 1 to 2 millimeters of the capillary column extend past the end of the guide tube and into the ionization chamber. When changing the removable ion source, the interface is automatically retracted from the ion source with the MassHunter software. This allows the source to slide freely in and out of the analyzer chamber.

Reagent gas is also 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.

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.

This interface is also be used for EI operation.

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

See Also

"To Install a Capillary Column in the GC/MS Interface" on page 35.

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

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

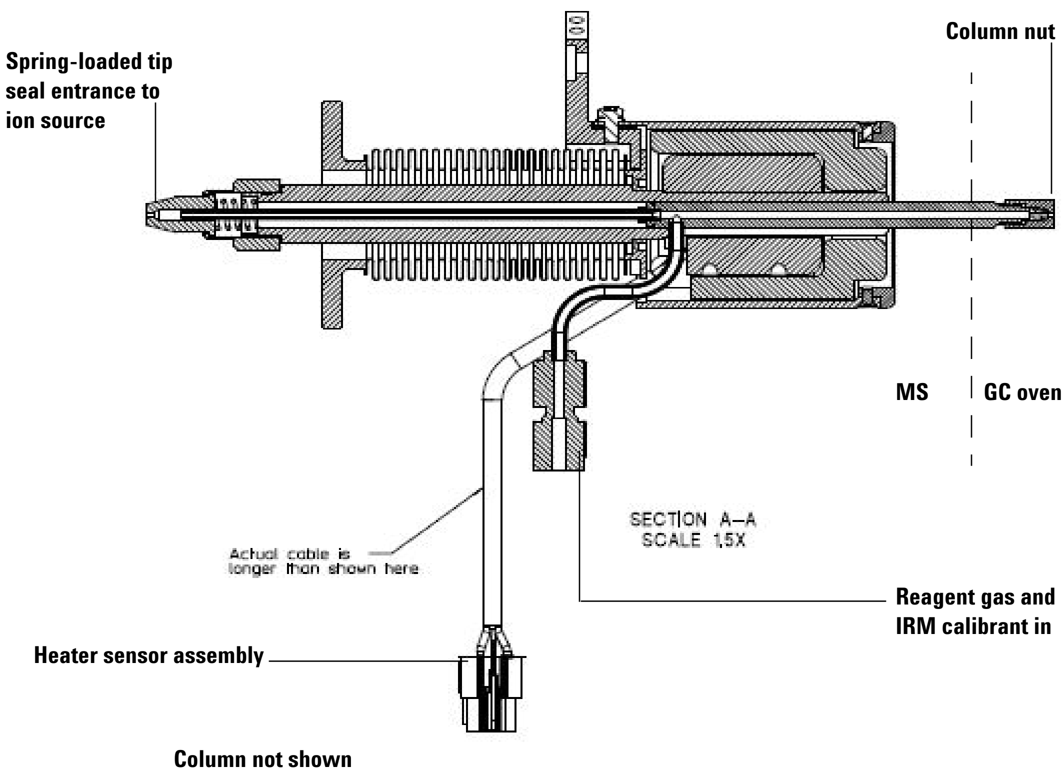


Figure 22 The CI GC/MS interface

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 (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 (Table 6). They were taken with the following set of conditions. Note that these are typical PCI temperatures:

Source temperature	300 °C
Quad temperature	150 °C
Interface temperature	280 °C to 320 °C
Helium carrier gas flow	1 mL/min

Table 6 Typical analyzer vacuum with reagent gas flow

	Collision cell gas flow on N2 = 1.5 mL/min		Collision cell gas flow off	
MFC (%)	**Rough vac**	**High vac**	**Rough vac**	**High vac**
10	1.77×10^{-1}	7.15×10^{-5}	1.33×10^{-1}	2.56×10^{-6}
15	1.86×10^{-1}	7.19×10^{-5}	1.43×10^{-1}	3.00×10^{-6}
20	1.94×10^{-1}	7.23×10^{-5}	1.53×10^{-1}	3.45×10^{-6}
25	2.02×10^{-1}	7.27×10^{-5}	1.63×10^{-1}	3.86×10^{-6}

Table 6 Typical analyzer vacuum with reagent gas flow (continued)

	Collision cell gas flow on N2 = 1.5 mL/min		Collision cell gas flow off	
MFC (%)	**Rough vac**	**High vac**	**Rough vac**	**High vac**
30	2.10×10^{-1}	7.31×10^{-5}	1.71×10^{-1}	4.30×10^{-6}
35	2.18×10^{-1}	7.39×10^{-5}	1.80×10^{-1}	4.76×10^{-6}
40	2.25×10^{-1}	7.43×10^{-5}	1.88×10^{-1}	5.18×10^{-6}

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 7200 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 7200 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_4H_{10}) is commonly used for chemical ionization when less fragmentation is desired in the chemical ionization spectrum. This is because the proton affinity of isobutane is higher than that of methane; hence less energy is transferred in the ionization reaction.

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

Ammonia CI

Ammonia (NH_3) is commonly used for chemical ionization when less fragmentation is desired in the chemical ionization spectrum. This is because the proton affinity of ammonia is higher than that of methane; hence less energy is transferred in the ionization reaction.

Because many compounds of interest have insufficient proton affinities, ammonia chemical-ionization spectra often result from the addition of NH_4^+ and then, in some cases, from the subsequent loss of water. Ammonia reagent ion spectra have principal ions at *m/z* 18, 35, and 52, corresponding to NH_4^+, $NH_4(NH_3)^+$, and $NH_4(NH_3)_2^+$.

CAUTION Use of ammonia affects the maintenance requirements of the MS. See Chapter 6, "General Maintenance"" 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 7200 Q-TOF GC/MS Troubleshooting and Maintenance Manual.)

CAUTION When running ammonia for 5 or more hours a day, the foreline pump must be ballasted (flushed with air) for at least 1 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 (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 Always verify MS performance in EI before switching to CI operation.

Table 7 CI tune default settings

Parameter	Methane		Isobutane		Ammonia		EI
Ion polarity	Positive	Negative	Positive	Negative	Positive	Negative	N/A
Emission	150 µA	50 µA	150 µA	50 µA	150 µA	50 µA	35 µA
Electron energy	150 eV	150 eV	150 eV	150 eV	150 eV	150 eV	70 eV
Filament	1	1	1	1	1	1	1 or 2
Repeller	3 V	3 V	3 V	3 V	3 V	3 V	30 V
Ion focus	130 V	130 V	130 V	130 V	130 V	130 V	90 V
Entrance lens offset	20 V	20 V	20 V	20 V	20 V	20 V	25 V
Shutoff valve	Open	Open	Open	Open	Open	Open	Closed
Suggested flow	20%	40%	20%	40%	20%	40%	N/A
Source temp	250 °C	150 °C	250 °C	150 °C	250 °C	150 °C	230 °C

Table 7 CI tune default settings (continued)

Parameter	Methane		Isobutane		Ammonia		EI
Quad temp	150 °C	150 °C	150 °C	150 °C	150 °C	150 °C	150 °C
Interface temp	280 °C	280 °C	280 °C	280 °C	280 °C	280 °C	280 °C
Autotune	Yes	Yes	No	Yes	No	Yes	Yes

N/A Not available

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 gases at 25 to 30 psi (170 to 205 kPa).

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 Change the source. See “To Change the Ion Source” on page 94.

2 Load a suitable PCI or NCI method for use with the CI source.

3 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.

4 Select the **Tune from default settings** check box because you have changed the ion source.

5 Select **Print autotune report** check box to automatically print a tune report.

6 Click the **Autotune** button to start the autotune. At the completion of the autotune, a Tune Report is printed.

7 Review the Tune Report. If the results are acceptable, save the autotune by clicking the **Files and Report** tab, then click the **Save** button.

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

Reagent gas	**Methane**		**Ammonia**	
Ion polarity	Positive	Negative	Positive	Negative
Abundance target	$1x10^6$	$1x10^6$	N/A	$1x10^6$
Peakwidth target	0.7	0.7	N/A	0.7
Maximum repeller	4	4	N/A	4
Maximum emission current, µA	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 peakwidth values give better sensitivity, lower values give better resolution.
- **Maximum emission current** Optimum emission current maximum for NCI is very compound-specific and must be selected empirically. Optimum emission current for pesticides, for example, may be about 200 µA.

To Operate the Reagent Gas Flow Control Module

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.

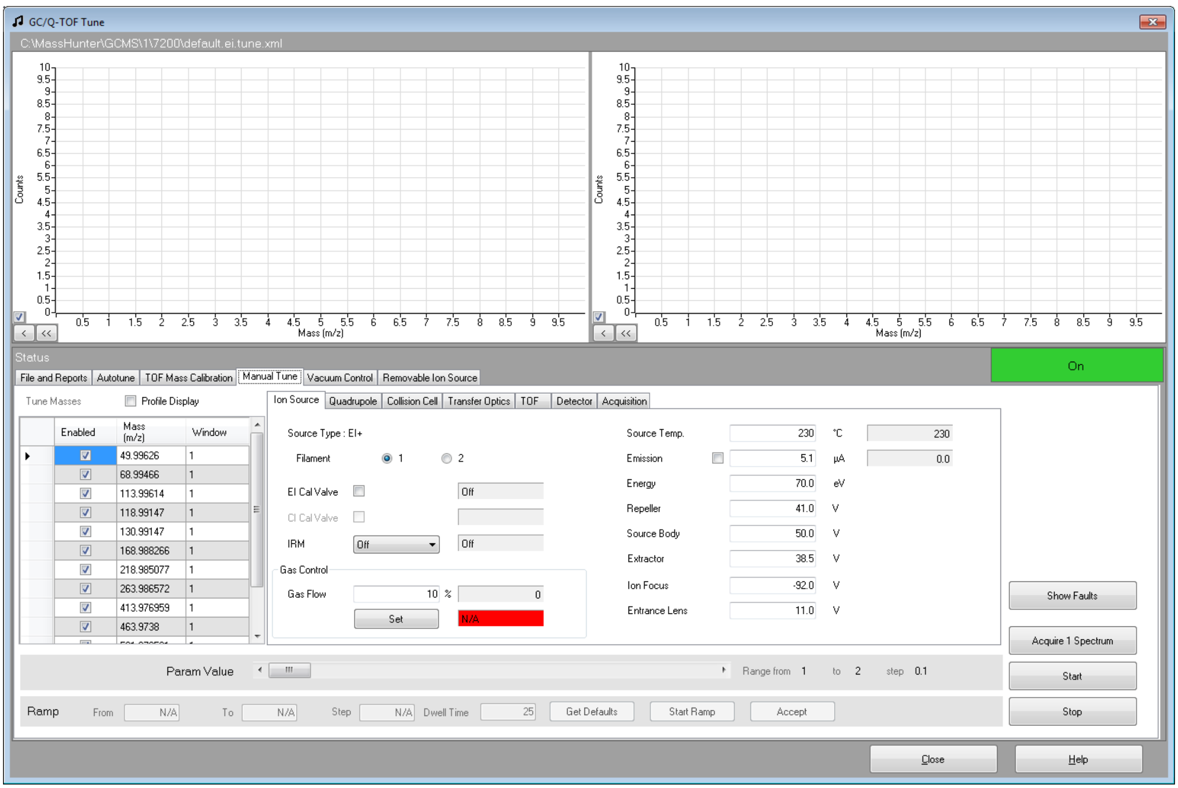


Figure 23 CI flow control

2 Use the parameters in the **CI Reagent Gas Control** area to control reagent gas flow.

CI Reagent - Selects the reagent gas from the dropdown menu.

CI 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 button - Opens the selected reagent gas supply valve and controls the reagent gas flow to the entered setpoint.

Off button - Turns off the reagent gas flow.

Purge button - Opens the selected reagent gas valve for 6 minutes to clear the system of unwanted compounds.

Pumpout button - Closes the reagent gas valve for 4 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 2 hours before tuning.

CAUTION Continuing with CI autotune if the MS has an air leak or large amounts of water will result in ***severe*** ion source contamination. If this happens, you 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 **CI Reagent Gas Control** area, select the CI reagent gas you are using.

3 Enter the reagent gas flow setpoint in the **CI 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 the **Set** button. 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 the **Save** button 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 Verify that the MS performs correctly in EI mode first.

2 Click the **MS Tune** icon in the **Instrument Control** panel to display the **GC/Q-TOF Tune** dialog box.

3 If necessary, load a new tune file by clicking on the **Files and Report** tab then click the **Load** button in the **Tune File** area. Select a tune file and click the **OK** button.

   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.

4 Click the **Autotune** tab and select **PCI source** for a positive CI source or **NCI source** for a negative CI source.

5 Select the reagent gas you are using from the dropdown menu in the **CI Reagent Gas** area.

6 If a log file and associated data files of the tune are required, click the **Files and Reports** tab and in the **Log Files** section, click on the **Browse** button to create a directory and files for the logs. Click on the required log and data files check boxes.

7 Click on the **Manual Tune** tab and select the **Ion source** tab. In the **CI Reagent Gas** section, select the reagent gas and then enter a **CI Gas Flow** rate of 20% for a PCI source or 40% for an NCI source. Click on the **Autotune** tab to return to Autotune.

8 Select the **Tune from default settings** check box 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.

9 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.

10 Select the **Print autotune report** check box to automatically print a tune report.

11 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 autotune parameter selection, click the **Abort Autotune** button. The parameters from the last successful autotune are used.

12 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.

5
Using the Removable Ion Source

This chapter will explain how to use the features of the removable ion source.

The Removable Ion Source

The removable ion source (RIS) is a feature of the Agilent 7200 Q-TOF GC/MS that allows changing of the ion source without venting the instrument through the use of the RIS probe extractor tool. The source must be removed from the MS periodically for routine cleaning and maintenance, or for changing ionization modes, and the new RIS minimizes the downtime necessary to complete these tasks. This process is guided by the MassHunter Workstation software, which performs the purging, valve operation, and pump down steps automatically.

The RIS probe extraction tool consists of a RIS bayonet that attaches to the source, a handle for moving the source in and out of the instrument, and a cooling chamber for cooling the source prior to removal (Figure 24). The entire process is guided by the MassHunter Workstation Software.

The RIS cooling chamber is attached to the instrument to provide a place to vent and purge the source with nitrogen during source changing. An automatic gate valve closes off the cooling chamber from the instrument to eliminate the need for venting the instrument during this process. Only the small RIS cooling chamber is vented.

CAUTION

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

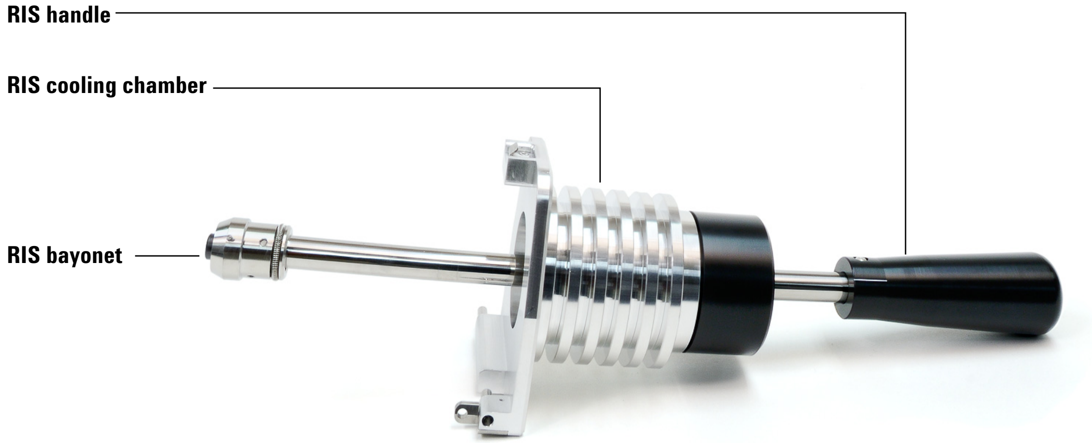


Figure 24 RIS probe extraction tool

To Change the Ion Source

The steps necessary for changing the ion source are provided here and also interactively in the MassHunter Workstation software. The process is mostly automated, with the exception of the use of the RIS probe extraction tool to remove and install the ion source. Purging the chamber, cooling the source, and pumping down are all performed with the use of the Workstation software.

Procedure

1 From MassHunter Data Acquisition software control view, click the **MS Tune** icon to display the **GC/Q-TOF Tune** dialog box.

2 Click the **Removable Ion Source** tab in the **GC/Q-TOF Tune** screen. (Figure 25)

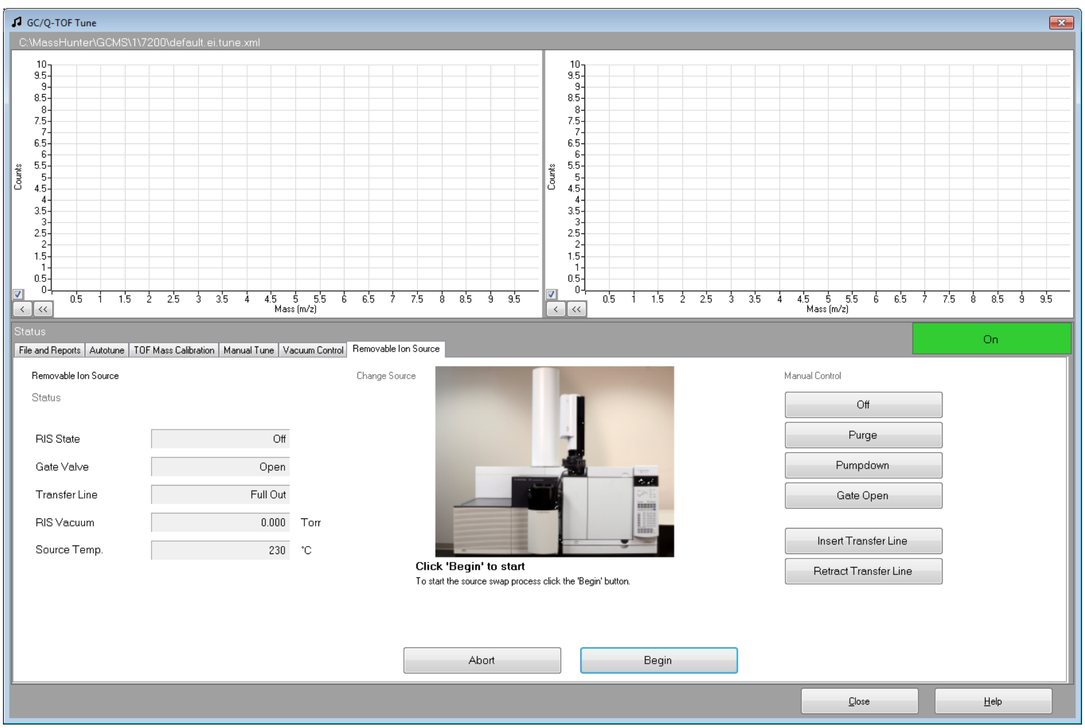


Figure 25 GC/Q-TOF Tune dialog box

3 Click the **Begin** button to purge the instrument.

4 When prompted, remove the RIS chamber door and attach the RIS probe extraction tool. See "To Install the RIS Probe Extraction Tool" on page 97.

5 Click **Next** to purge the chamber and pump down.

6 When the system is ready for source removal, click **Next** to retract the transfer line and open the gate valve.

The RIS status area will note when the **Gate Valve** is **Open** and the **Transfer Line** is **Full Out**.

7 When prompted, remove the source. See "To Remove the Source from the Analyzer Chamber" on page 99.

8 Once the source is retracted fully into the chamber, click **Next** to close the gate valve and purge the cooling chamber. The purge takes approximately 10 minutes.

9 When prompted, open the cooling chamber and change the ion source. See "To Change the Ion Source on the RIS Probe Extraction Tool" on page 101.

10 After replacing the source and securing the cooling chamber door, click **Next** to purge and pump down.

11 When prompted that the system is ready for source installation, click **Next** to retract the transfer line and open the gate valve.

12 When prompted, install the ion source in the analyzer chamber. See "To Install the Ion Source in the Analyzer Chamber" on page 103.

13 Verify in the RIS **Status** that the ion source temperature readback shows a source temperature. This ensures that the source is fully inserted into its socket.

CAUTION If the ion source is not fully installed, it may interfere with the gate valve as it closes. Damage to the RIS, the valve, or the transfer line may occur.

14 Click **Next** to close the gate valve and vent the RIS cooling chamber.

15 Remove the RIS probe extraction tool and replace the RIS chamber cover. See “To Remove the RIS Probe Extraction Tool from the Instrument” on page 104.

16 Click **Next** to pump down the chamber behind the RIS chamber cover.

17 When prompted, click **Finish**.

To Install the RIS Probe Extraction Tool

This procedure is part of an automated series of steps controlled by MassHunter. Do not attempt this procedure unless prompted by MassHunter to remove the RIS cover door.

Procedure

1 When prompted by MassHunter, remove the upper RIS cover (see “To Remove the Upper RIS Cover” on page 64).

2 Open the latch on the right side of the RIS cover door.

3 Swing the door open.

4 Lift the RIS cover door straight up to free it from the hinges, and remove.

CAUTION Do not leave the instrument with the RIS cover off. If you remove the cover, you must install the RIS probe as soon as possible to minimize contamination in the instrument.

5 Holding the RIS probe extraction tool by the handle, position the RIS cooling chamber up against the instrument RIS chamber. Hold the handle perpendicular to the front of the instrument so that the outside edge of the cooling chamber is flush with the RIS chamber opening on the front instrument.

6 Line up the pins on the probe hinge with the slots on the instrument hinge.

7 Lower the pins into the hinge.

8 Close the cooling chamber door, and secure the door with the latch on the right side.

9 Return to step 5 in the “To Change the Ion Source” on page 94.

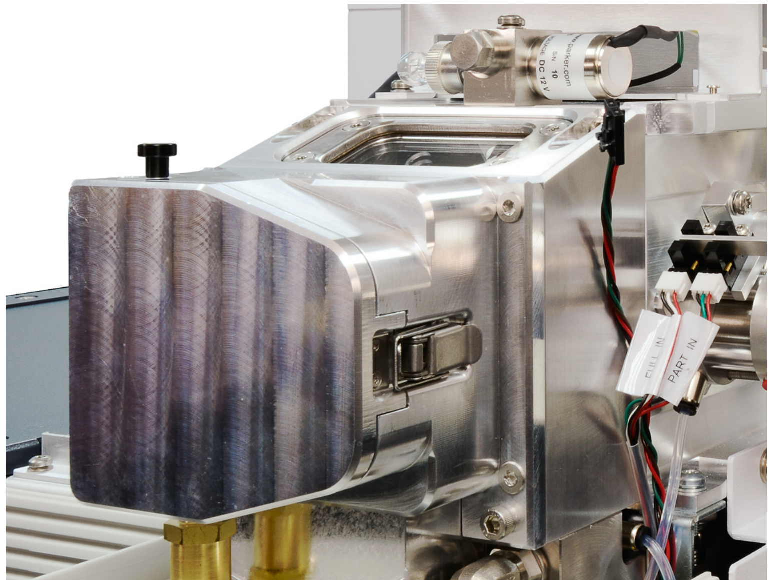


Figure 26 RIS cover door

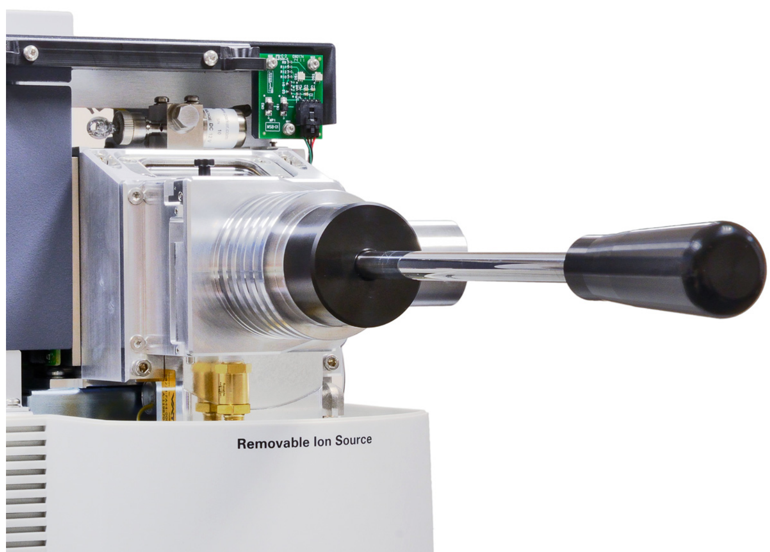


Figure 27 RIS probe installed

To Remove the Source from the Analyzer Chamber

This procedure is part of an automated series of steps controlled by MassHunter. Do not attempt this procedure unless prompted by MassHunter. The RIS **Status** area on the **GC/Q-TOF Tune** tab must show the **Gate Valve** is **Open** and the **Transfer Line** is **Full Out**.

Procedure

1 Holding the RIS handle, turn the alignment guide, which is the white line on the RIS handle, to the two o'clock position from the top of the handle. This ensures that the RIS bayonet is aligned properly to attach to the source.

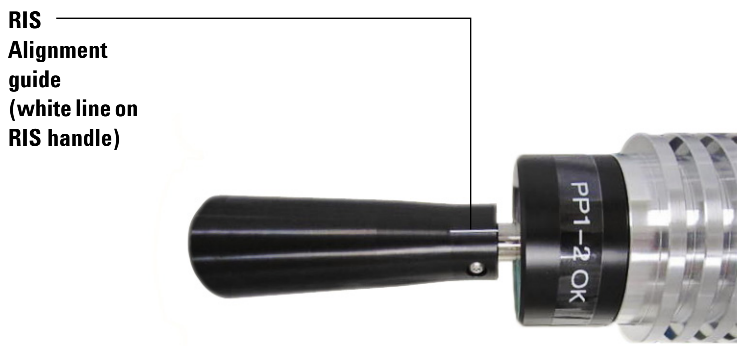


Figure 28 RIS Alignment guide

CAUTION While the RIS handle is fully extended, do not bump or push it from the side. Doing so will break the seal and allow air into the cooling chamber.

CAUTION Do not try to guide the ion source in manually. Push the RIS handle gently and allow the RIS bayonet to slide on its own. The RIS bayonet will slide in straight if it is allowed to move naturally.

2 Keeping the RIS handle in position, push the RIS handle gently and allow the RIS bayonet to extend into the analyzer chamber. Make sure the RIS handle extends fully, to ensure that the RIS bayonet seats in the holes on the ion source body properly.

3 When the ion source is properly seated, turn the RIS handle counter clockwise until the alignment guide is in the twelve o’clock position, to lock the ion source in place.

4 Pull the RIS handle toward you, keeping the alignment guide at the twelve o’clock position, to retract the ion source into the RIS cooling chamber.

5 Return to step 8 in the “To Change the Ion Source” on page 94.

To Change the Ion Source on the RIS Probe Extraction Tool

This procedure is part of an automated series of steps controlled by MassHunter. Do not attempt this procedure unless prompted by MassHunter. The RIS **Status** area on the **GC/Q-TOF Tune** tab must show the **Gate Valve** is **Open** and the **RIS vacuum** as **0.000 Torr**.

CAUTION If not done correctly, source maintenance can introduce contaminants into the MS. Always wear clean rubber gloves when handling the source.

1 Undo the latch on the right edge of the RIS cooling chamber.
2 Swing open the RIS cooling chamber until the RIS handle is parallel with the front of the instrument.
3 Lock the RIS cooling chamber in the open position by opening it enough to engage the locking pin on the hinge.

WARNING **The 7200 Q-TOF GC/MS operates at high temperatures. Make sure the source is cooled to a safe temperature before attempting to remove it from the RIS cooling chamber.**

4 Grasp the RIS handle with one hand, and the ion source body with the other hand.
5 While holding the ion source steady, turn the RIS handle counter clockwise so that the alignment guide is in the two o’clock position. This guides the pin on the RIS bayonet out of the track on the ion source body.
6 Gently pull the ion source free from the RIS bayonet and set aside.
7 Remove the new ion source from its storage container. The ion source remains connected to the lid. Twist the lid clockwise to separate the ion source from the lid.
8 Turn the RIS handle so that the alignment guide is in the two o’clock position.

9 Guide the pin on the RIS bayonet into the tracks on the end of the ion source body.

10 While holding the ion source securely, turn the RIS handle counter clockwise so that the alignment guide is in the twelve o'clock position, to lock the ion source securely on the bayonet.

11 Retract the RIS handle fully so that the ion source is located completely in the cooling chamber.

12 Raise the locking pin on the RIS cooling chamber hinge and swing it shut.

13 Secure the RIS cooling chamber to the instrument with the latch on the right side of the door.

14 Return to step 10 in the "To Change the Ion Source" on page 94.

To Install the Ion Source in the Analyzer Chamber

This procedure is part of an automated series of steps controlled by MassHunter. Do not attempt this procedure unless prompted by MassHunter. The RIS **Status** area on the **GC/Q-TOF Tune** tab must show the **Gate Valve** is **Open** and the **Transfer Line** is **Full Out**.

CAUTION Take care when walking past the front of the instrument when the RIS handle is fully extended, to avoid damage to the instrument. When closed, the RIS handle extends 19 inches out from the front of the instrument.

CAUTION Do not try to guide the ion source in manually. Push the RIS handle gently and allow the RIS bayonet to slide on its own. The RIS bayonet will slide in straight if it is allowed to move naturally.

Procedure

1 Make sure the alignment guide on the RIS handle is in the twelve o'clock position. Push the RIS handle gently towards the analyzer chamber and allow it to extend into the analyzer chamber. This will seat the ion source securely into its socket.

2 Once the RIS handle is fully extended, turn the RIS handle clockwise until the alignment guide is in the two o'clock position to release the ion source body from the RIS bayonet.

3 Pull the RIS handle toward you until it is fully retracted into the RIS cooling chamber.

4 Return to step 13 in the "To Change the Ion Source" on page 94.

To Remove the RIS Probe Extraction Tool from the Instrument

This procedure is part of an automated series of steps controlled by MassHunter. Do not attempt this procedure unless prompted by MassHunter. The RIS **Status** area on the **GC/Q-TOF Tune** tab must show the **Gate Valve** is **Open** and the **RIS vacuum** as **0.000 Torr**.

WARNING **The 7200 Q-TOF GC/MS operates at high temperatures. Make sure the RIS cooling chamber is at a safe temperature before attempting to remove it from the instrument.**

1 Undo the latch on the right side of the RIS probe extraction tool and open the door.
2 Lift the RIS probe extraction tool up to release it from its hinges.
3 Replace the RIS probe extraction tool with the RIS chamber door.
4 Line up the hinge pins on the RIS chamber door with the slots on the instrument hinge, and lower the pins into the hinge.
5 Swing the RIS chamber door closed and secure the door with the latch on the right side.
6 Return to step 16 in the "To Change the Ion Source" on page 94.

6
General Maintenance

Before Starting

You can perform much of the maintenance required by your MS. 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
Tune the MS				X
Check the foreline pump oil level	X			
Check the calibration vial(s)		X		
Replace the foreline pump oil*		X		
Check the foreline pump				X
Clean the ion source				X
Check the carrier gas trap(s) on the GC and MS				X
Replace the worn out parts				X
Check gasket on RIS probe			X	
Check the IRM vial		X		
Replace GC gas supplies				X

* Or as needed.

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 (120 VAC or 200/240 VAC) exists on the wiring and fuses between where the power cord enters the instrument and the power switch.

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

- Electronic circuit boards
- Toroidal transformer
- Wires and cables between 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 **Perform no maintenance with the MS turned on or plugged into its power source unless you are instructed to do so by one of the procedures 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. Be sure to comply with your local air quality regulations.

WARNING **The oil trap supplied with the standard foreline pump stops only foreline pump oil. It does not trap or filter out toxic chemicals. If you are using toxic solvents or analyzing toxic chemicals, use a hose to vent to a safe location.**

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

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

Ion source cleaning

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

WARNING **Always perform any maintenance procedures using hazardous solvents under a fume hood. Be sure to operate the MS in a well-ventilated room.**

Ammonia

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

Always purge the MS with methane after using ammonia.

Be sure to install the ammonia so the tank is in an upright position. This will help prevent liquid ammonia from getting into the flow module.

Maintaining the Vacuum System

Periodic maintenance

As listed in Table 9, some maintenance tasks for the vacuum system must be performed periodically. These include:

- Checking the foreline pump fluid color and level (every week)
- Checking the calibration vial(s) (every 6 months)
- Replacing the foreline pump oil and oil mist filter (every 6 months or as needed)

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 7200 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 7200 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 7200 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 filaments

The Agilent 7200 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. There are no customer serviceable parts accessible from this door.

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

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

CAUTION Electrostatic discharges to analyzer components are conducted to the quad driver board where they can damage sensitive components. Wear a grounded antistatic wrist strap and take other antistatic precautions when working on the analyzer.

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. The HED ceramic insulator must never be touched.

CAUTION Incorrect handling or cleaning of the mass filter can damage it and have a serious, negative effect on instrument performance. Never open the analyzer door, and do not touch the HED ceramic insulator.

More information is available

If you need more information about the locations or functions of analyzer components, refer to the *Agilent 7200 Accurate-Mass Q-TOF GC/MS Concepts Guide*.

To Disassemble the EI Ion Source

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Hex ball driver, 1.5 mm (8710-1570)
- Hex ball driver, 2.0 mm (8710-1804)
- Wrench, open-end, 10 mm (8710-2353)
- Nut driver, 5.5 mm (8710-1220)
- Tweezers (8710-2460)

Procedure

1 Remove the ion source. See “To Change the Ion Source” on page 94.

2 Remove the filaments. See “To Remove a Filament” on page 126.

3 Separate the ion focus and insulator assembly from the source body. (See Figure 29.)

4 Remove the two screws from the ion focus insulator and separate it from the ion focus lens.

5 Remove the extractor lens and its insulator from the source body.

6 Remove the two screws from the RIS bayonet mount on the bottom of the source body, and remove the RIS bayonet mount.

7 Remove the repeller assembly from the source body.

8 Disassemble the repeller assembly by separating the repeller, the repeller cap, ring heater and sensor assembly. (See Figure 29.)

9 Remove the clocking button from the repeller cap by removing the screw that holds it in place. (See Figure 29.)

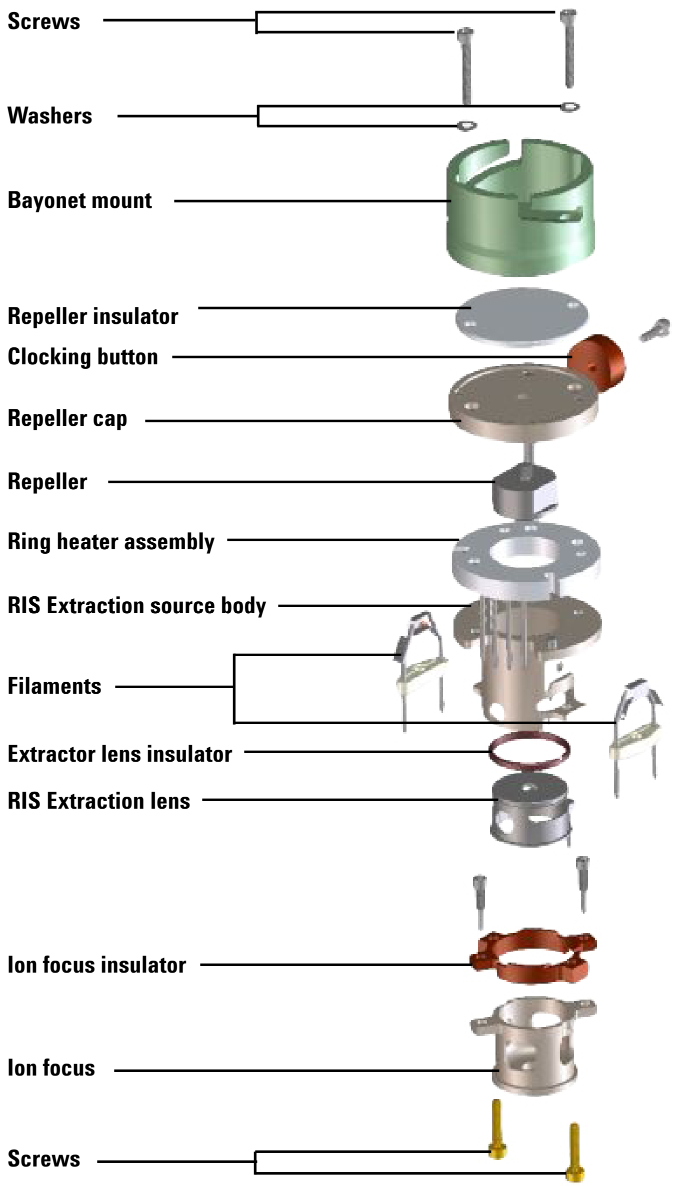


Figure 29 Disassembling the EI ion source

To Assemble the EI Ion Source

Materials needed

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

Procedure

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

1. Assemble the repeller assembly, by attaching the repeller to the repeller cap and connecting the clocking button with the screw.
2. Align the heater sensor with the source body.
3. Slide the repeller cap onto the heater sensor, and place the ceramic repeller insulator on top, aligning the screw holes.
4. Place the RIS bayonet mount on top of the ceramic repeller insulator, and attach with the washers and screws.
5. Attach the extractor lens insulator to the extractor lens, and slide them onto the other end of the source body (Figure 29).
6. Place the ion focus insulator on the ion focus lens and attach with the two screws.
7. Set the ion focus assembly on top of the extractor lens assembly on the source body.
8. Attach the filaments with the two screws.

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

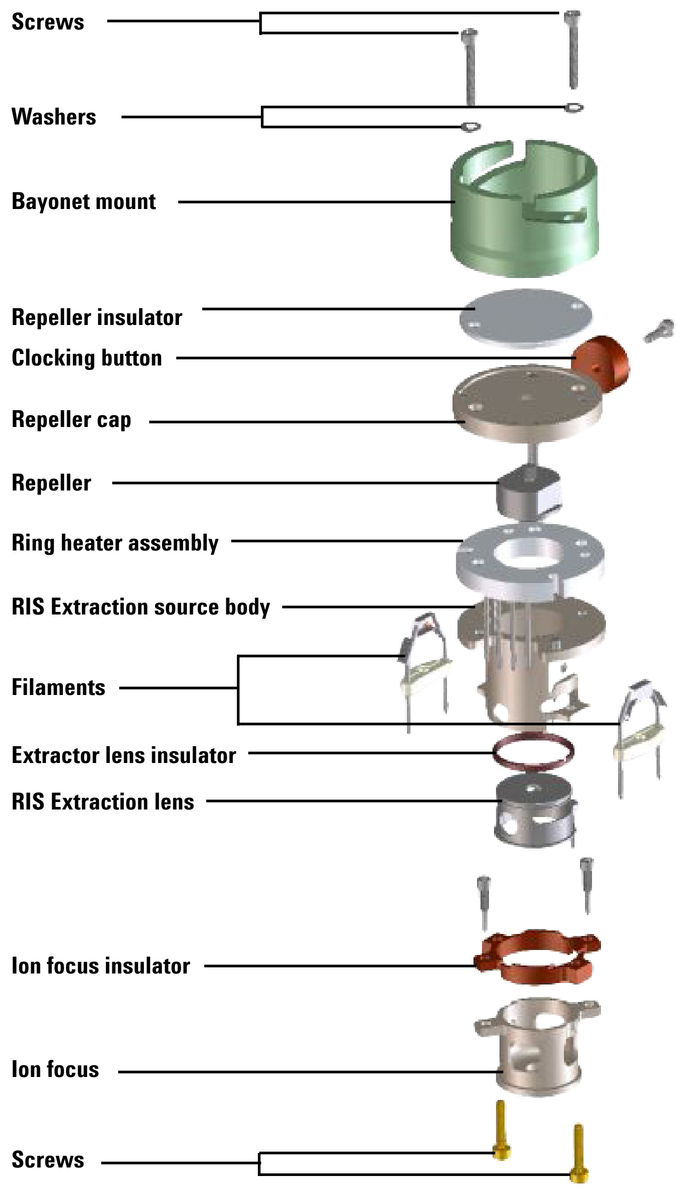


Figure 30 Assembling the EI source

To Disassemble the CI Ion Source

Materials needed

- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Hex ball driver, 1.5 mm (8710-1570)
- Hex ball driver, 2.0 mm (8710-1804)
- Wrench, open-end, 10 mm (8710-2353)
- Nut driver, 5.5 mm (8710-1220)
- Tweezers (8710-2460)

Procedure

1 Remove the CI ion source. See “To Change the Ion Source” on page 94.

2 Remove the filament. See “To Remove a Filament” on page 126.

3 Remove the ion focus lens and insulator. (See Figure 31.)

4 Remove the two screws from the ion focus lens insulator and separate it from the ion focus lens.

5 Remove the two screws from the RIS bayonet mount and remove the repeller assembly.

6 Disassemble the repeller assembly by removing the ceramic insulator and heater assembly from the repeller. (See Figure 31.)

7 Take the repeller off the source body.

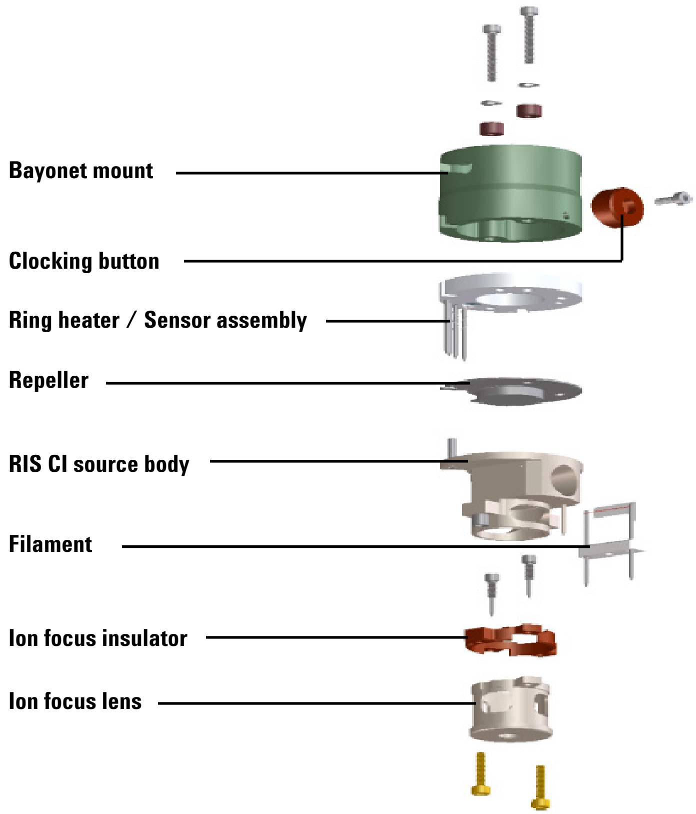


Figure 31 Disassembling the CI ion source

To Assemble the CI Ion Source

Materials needed

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

Procedure

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

1 Place the ceramic heater assembly on top of the repeller, aligning the grooves on the repeller with the slot on the heater assembly. (Figure 32).

2 Place the repeller on the source body. (Figure 32).

3 Place the assembled parts into the RIS bayonet mount, heater side first.

4 Align the interface socket in the source body with the alignment groove in the RIS bayonet mount. Attach with the two screws.

5 Place the ion focus and ion focus insulator together and attach with the two screws.

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

6 Set the filament in place.

7 Place the ion focus assembly on the source body, with the insulator against the source body. Make sure all pins are facing in the same direction.

8 Attach with the two screws.

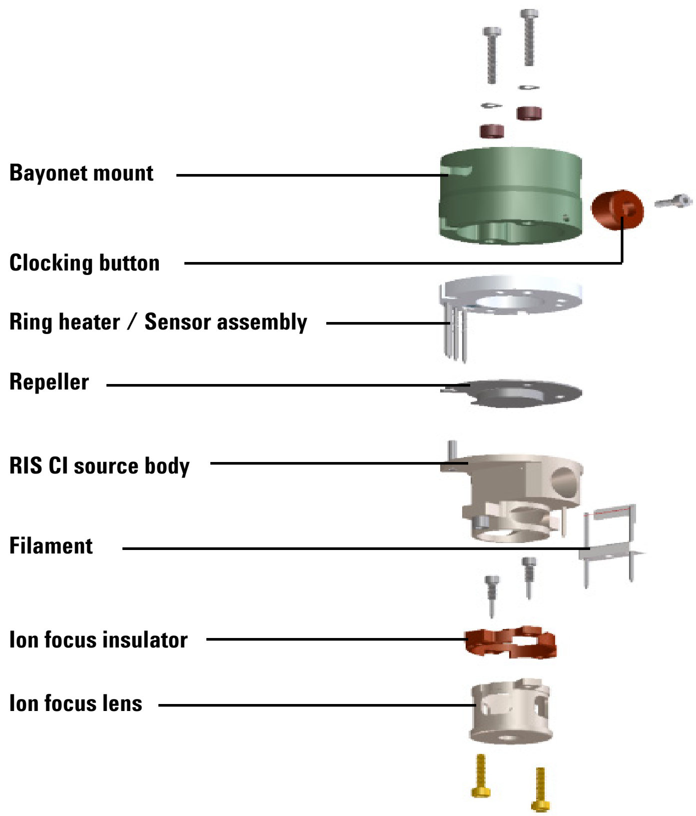


Figure 32 Assembling the CI ion source

To Clean the Ion Source

Frequency of cleaning

Because the CI ion source operates at much higher pressures than the EI ion source, it will probably require more frequent cleaning than the EI ion source. Cleaning of the source is not a scheduled maintenance procedure. The source should be cleaned whenever there are performance anomalies that are associated with a dirty ion source. See the 7200 Q-TOF GC/MS Troubleshooting and Maintenance Manual for symptoms that indicate a dirty ion source.

Visual appearance is not an accurate guide to cleanliness of the CI ion source. The CI ion source can show little or no discoloration yet still need cleaning. Let analytical performance be your guide.

Materials needed

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

Preparation

1 Disassemble the ion source. See "To Disassemble the EI Ion Source" on page 114 or "To Disassemble the CI Ion Source" on page 118.

2 Collect the following parts to be cleaned if you are cleaning a high sensitivity extractor EI ion source: (Figure 33)

- Repeller
- Source body
- Extractor lens
- Ion focus lens

Collect the following parts to be cleaned if you are cleaning a CI source:

- Repeller
- Source body
- Ion focus lens

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

CAUTION If insulators are dirty, clean them with a cotton swab dampened with reagent-grade methanol. If that does not clean the insulators, replace them. Do not abrasively or ultrasonically clean the insulators.

Cleaning the CI ion source is very similar to cleaning the EI ion source, with the following exceptions:

- The CI ion source may not look dirty but deposits left by chemical ionization are very difficult to remove. Clean the CI ion source thoroughly.
- Use a round wooden toothpick to gently clean out the electron entrance hole in the source body.
- Do not use halogenated solvents. Use hexane for the final rinse.

CAUTION Do not use halogenated solvents to clean the CI ion source.

Figure 33 Extractor EI Source parts to be cleaned

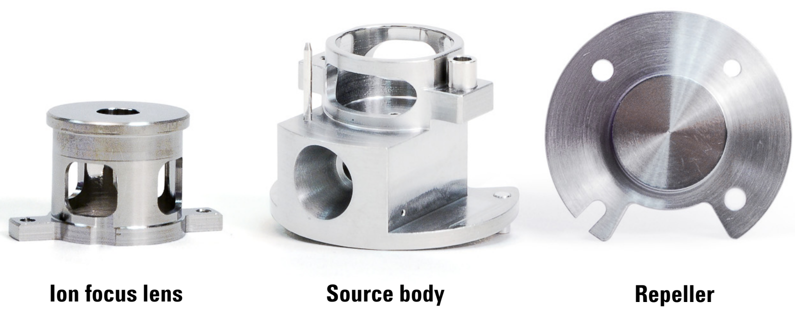


Figure 34 CI Source parts to be cleaned

CAUTION

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

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

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

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

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

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

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

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

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

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

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

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

9 Dry the cleaned parts in an oven at 100 °C for 5–6 minutes.

To Remove a Filament

Materials needed

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

Procedure

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

1 Remove the ion source. See “To Change the Ion Source” on page 94.
2 Remove the screw holding the filament to the ion source body. (See Figure 35.)
3 Slide the filament off the ion source assembly. (See Figure 35.)

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.**

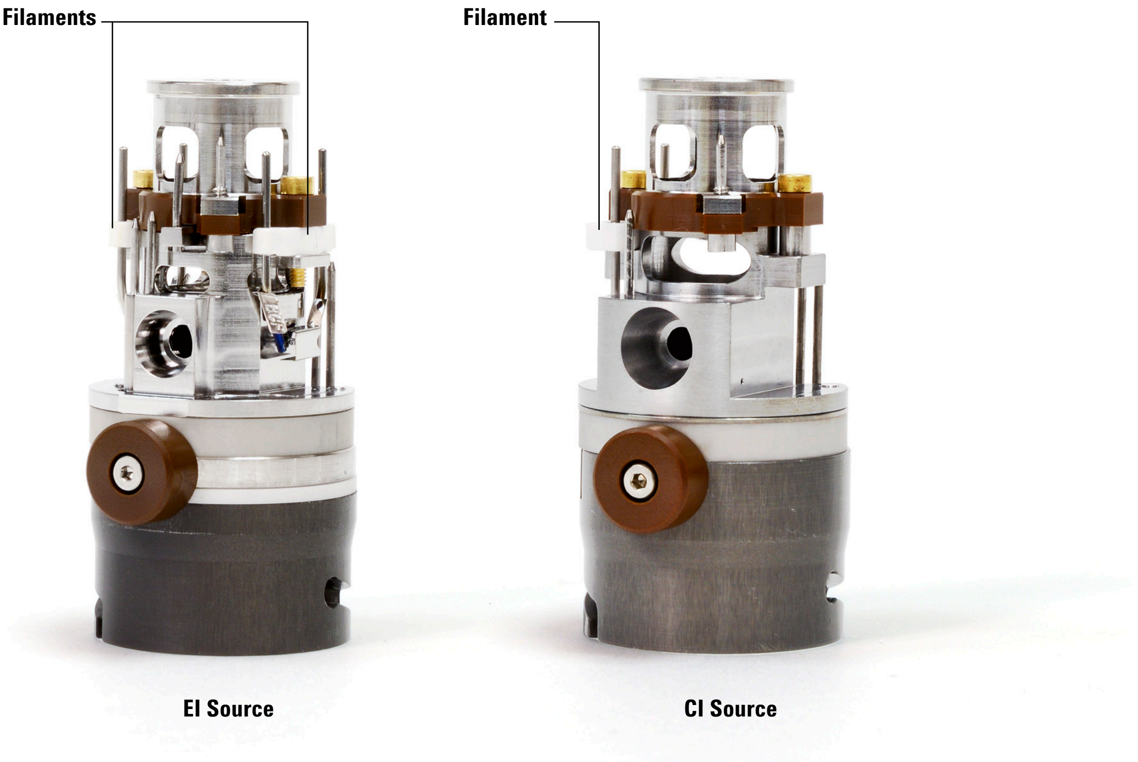


Figure 35 Changing the filament

To Install a Filament

Materials needed

- Filament assembly, EI (G3170-60050)
- Filament assembly, CI (G1099-80053)
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Tweezers (8710-2460)

Procedure

1. Remove the old filament. (See “To Remove a Filament” on page 126.)
2. Place the new filament into its position on the ion source body. (See Figure 35.)
3. Secure the filament to the ion source body with the screw.
4. After installing the filament, verify that it is not grounded to source body.
5. Reinstall the ion source. (See “To Change the Ion Source” on page 94.)
6. Autotune the MS.

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

Agilent Technologies

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