Agilent 7000 Series Triple Quad GC/MS

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.

Contents

1 Introduction

Abbreviations Used 10

The 7000 Series Triple Quad GC/MS 12

Triple Quad GC/MS Hardware Description 14

Important Safety Warnings 15

Hydrogen Safety 17

Safety and Regulatory Certifications 22

Intended Use 25

Cleaning/Recycling the Product 25

Liquid Spills 25

Moving or Storing the MS 25

2 Installing GC Columns

Columns 28

To Prepare a Capillary Column for Installation 30

To Install a Capillary Column in a Split/Splitless Inlet 32

To Condition a Capillary Column 34

To Install a Capillary Column in the GC/MS Interface 35

3 Operating in Electron Impact (EI) Mode

Operating the MS from the Data System 40

Monitoring the MS from the LCP 40

LCP Menus 42

The EI GC/MS Interface 43

Before You Turn On the MS 45

Pumping Down 46

Controlling Temperatures 46

Controlling Column Flow 47

Controlling Collision Cell Flow 47

Venting the MS 48

High Vacuum Pressure in EI Mode 49

To Set Monitors for MS Temperature and Vacuum Status 50

To Set the MS Analyzer Temperatures 52

To Set the GC/MS Interface Temperature from the MassHunter Workstation 54

To Calibrate the Column 55

To Configure CC Gas 57

To Set the Collision Cell Gas Flow Rates 58

To Autotune the MS for EI Mode 59

To Open Left Side Panel for Access to the Analyzer Chambers 61

To Pump Down the MS 62

To Vent the MS 65

To Switch from the CI Source to the EI Source 67

To Move or Store the MS 69

To Set the Interface Temperature from the GC 71

To Save a Method on the GC 72

4 Operating in Chemical Ionization (CI) Mode

Setting Up Your MS to Operate in CI Mode 74

The CI GC/MS Interface 75

Operating the CI MS 77

High Vacuum Pressure in CI Mode 78

Other Reagent Gases 79

CI Autotune 81

The Flow Control Module 83

To Switch from the EI Source to the CI Source 85

To Operate the Reagent Gas Flow Control Module 87

To Set a Reagent Gas Flow 89

To Perform a CI Autotune 90

5 General Maintenance

Before Starting 94

Maintaining the Vacuum System 99

Maintaining the Analyzer 100

To Open the Front Analyzer Chamber 102

To Remove the EI Ion Source 105

To Disassemble the Standard EI Ion Source 108

To Disassemble the EI Extractor Ion Source 110

To Clean the EI Ion Source 112

To Assemble the Standard EI Ion Source 116

To Assemble the EI Extractor Ion Source 117

To Install the EI Ion Source 119

To Remove the CI Ion Source 121

To Disassemble the CI Ion Source 124

To Clean the CI Ion Source 126

To Assemble the CI Ion Source 128

To Install the CI Ion Source 130

To Install the CI Interface Tip Seal 132
To Remove a Filament 134
To Install a Filament 136
To Attach Wiring from the Ion Source to the Side Board 137
To Close the Front Analyzer Chamber 141
To Remove Left Rear Cover for Access to the Rear Analyzer Chamber 142
To Open the Rear Analyzer Chamber 144
To Replace the Electron Multiplier Horn 147
To Close the Rear Analyzer Chamber 149

A Chemical Ionization Theory

Chemical Ionization Overview 152
Positive CI Theory 154
Negative CI Theory 161

About This Manual

This manual contains information for operating and maintaining the Agilent 7000 Series Triple Quad Mass Spectrometer (MS) system.

1 “Introduction”

Chapter 1 describes general information about the 7000 Series Triple Quad GC/MS, including a hardware description, general safety warnings, and hydrogen safety information.

2 “Installing GC Columns”

Chapter 2 shows you how to prepare a capillary column for use with the 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 “General Maintenance”

Chapter 5 describes general maintenance procedures for the 7000 Series Triple Quad GC/MS.

Hardware User Information

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

The hardware user information DVD that ships with your instrument provides an extensive collection of online help, videos, and books for the Agilent **7890A GC**, **7000 Series 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

1

Introduction

Abbreviations Used 10
The 7000 Series Triple Quad GC/MS 12
Triple Quad GC/MS Hardware Description 14
Important Safety Warnings 15
Hydrogen Safety 17
Safety and Regulatory Certifications 22
Intended Use 25
Cleaning/Recycling the Product 25
Liquid Spills 25
Moving or Storing the MS 25

This section provides general information about the 7000 Series Triple Quad Gas Chromatograph (GC) / Mass Spectrometer (MS), including a hardware description, general safety warnings, and hydrogen safety information.

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
CID	Collision induced dissociation
DC	Direct current
DFTPP	Decafluorotriphenylphosphine (calibrant)
DIP	Direct insertion probe
EI	Electron impact
EM	Electron multiplier (detector)
EMV	Electron multiplier voltage
EPC	Electronic pneumatic control
eV	Electron volt
GC	Gas chromatograph
HED	High-energy dynode (refers to detector and its power supply)
id	Inside diameter
LAN	Local Area Network
LCP	Local control panel
m/z	Mass to charge ratio
MFC	Mass flow controller

Table 1 Abbreviations (continued)

Abbreviation	Definition
MRM	Multiple reaction monitoring
MS	Mass spectrometer
MS1	Front quadrupole
MS2	Rear quadrupole
NCI	Negative chemical ionization
OFN	Octafluoronaphthalene (sample)
PCI	Positive chemical ionization
PFDTD	Perfluoro-5,8-dimethyl-3,6,9-trioxydodecane (calibrant)
PFTBA	Perfluorotributylamine (calibrant)
QQQ	Triple quadrupole
Quad	Quadrupole mass filter
RF	Radio frequency
RFPA	Radio frequency power amplifier
Torr	Unit of pressure, 1 mm Hg
Turbo	Split flow turbomolecular vacuum pump

The 7000 Series Triple Quad GC/MS

The 7000 Series Triple Quad GC/MS is a standalone capillary GC detector for use with the Agilent 7890A Series gas chromatograph. The Triple Quad MS features:

- One split flow turbomolecular vacuum pump
- Rotary vane foreline pump
- Independently MS heated ion source
- Chemical and electron-ionization modes available (PCI/NCI/EI)
- Two independently MS heated hyperbolic quadrupole mass filters
- Single hexapole collision cell
- High-energy dynode (HED) electron multiplier detector
- Independently GC heated GC/MS interface
- Independently GC controlled collision cell gas flows
- High sensitivity electronic upgrades for the EI source and HED available
- Local control panel (LCP) for locally monitoring the MS

Physical description

The 7000 Series Triple Quad GC/MS is a rectangular box, approximately 47 cm high, 35 cm wide, and 86 cm deep. The weight is 59 kg for the turbo pump 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 ion source, the electronics, the collision cell, the detector, and the front and rear analyzers.

Local control panel

The local control panel allows local monitoring of the MS instrument status.

Vacuum gauge

The 7000 Series Triple Quad GC/MS is equipped with two ion vacuum gauges. The MassHunter Workstation can be used to read the pressure (high vacuum) in the vacuum manifold and at the turbomolecular vacuum pump discharge.

In this manual, the term “CI MS” refers to the 7000 Series Triple Quad GC/MS CI source system. It also applies, unless otherwise specified, to the flow modules for these instruments.

The 7000 Series Triple Quad GC/MS CI source system upgrade kit adds to the 7000 Triple Quad MS:

- EI/CI GC/MS interface
- High sensitivity EI source
- CI ion source and interface tip seal
- Reagent gas flow control module
- HED with high sensitivity electronics
- Bipolar HED power supply for PCI and NCI operation

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

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

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

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

Triple Quad GC/MS Hardware Description

Figure 1 is an overview of a typical 7000 Triple Quad GC/MS system.

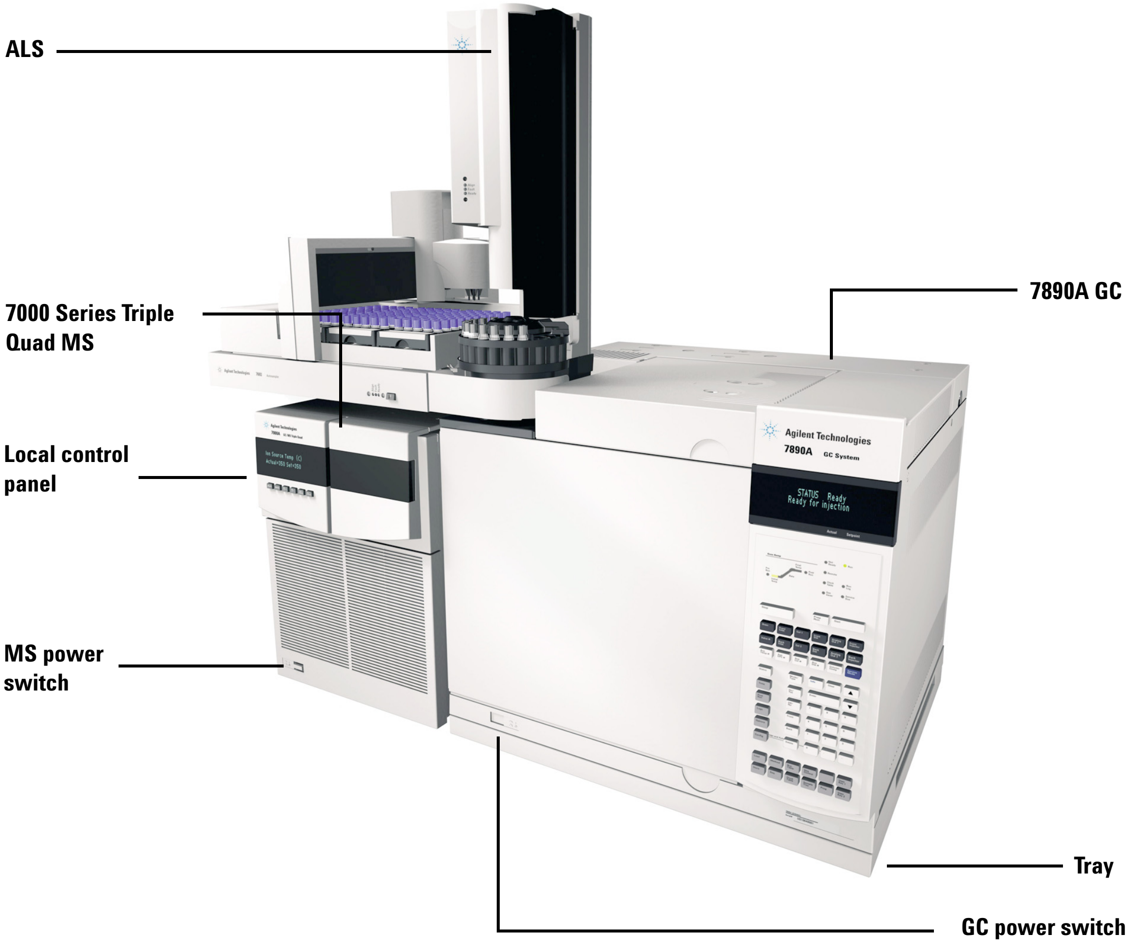


Figure 1 7000 Triple Quad MS

Important Safety Warnings

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

Many internal parts of the MS carry dangerous voltages

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

- The wiring between the MS 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 MS electronics

The printed circuit boards in the MS can be damaged by electrostatic discharge. Do not touch any of the boards unless it is absolutely necessary. If you must handle them, wear a grounded wrist strap and take other antistatic precautions.

Many parts are dangerously hot

Many parts of the GC/MS 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
- Detectors
- Column nuts attaching the column to an inlet or detector
- 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 cool-down cycles, the GC emits hot exhaust that can cause burns.**

WARNING **The insulation around the inlets, detectors, valve box, and the insulation cups is made of refractory ceramic fibers. To avoid inhaling fiber particles, we recommend the following safety procedures: ventilate your work area; wear long sleeves, gloves, safety glasses, and a disposable dust/mist respirator; dispose of insulation in a sealed plastic bag 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.**

Hydrogen Safety

WARNING **The use of hydrogen as a GC carrier gas is potentially dangerous.**

WARNING **When using hydrogen (H_2) as the carrier gas or fuel gas, be aware that hydrogen gas can flow into the GC oven and create an explosion hazard. Therefore, be sure that the hydrogen supply is turned off until all connections are made and ensure that the inlet and detector column fittings are either connected to a column or capped at all times when hydrogen gas is supplied to the instrument.**

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

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

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

Dangers unique to GC/MS operation

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

- Combustion of leaking hydrogen
- Combustion due to rapid expansion of hydrogen from a high-pressure cylinder

- Accumulation of hydrogen in the GC oven and subsequent combustion (See your GC documentation and the label on the top edge of the GC oven door.)
- Accumulation of hydrogen in the MS and subsequent combustion

Hydrogen accumulation in an MS

WARNING **The MS cannot detect leaks in inlet and/or detector gas streams. For this reason, it is vital that column fittings should always be either connected to a column or have a cap or plug installed.**

All users should be aware of the mechanisms by which hydrogen can accumulate (Table 2) and know what precautions to take if they are certain or suspect that hydrogen has accumulated. Note that these mechanisms apply to *all* mass spectrometers.

Table 2 Hydrogen accumulation mechanisms

Mechanism	Results
Mass spectrometer turned off	A mass spectrometer can be shut down deliberately. It can also be shut down accidentally by an internal or external failure. A mass spectrometer shutdown does not shut off the flow of carrier gas. As a result, hydrogen may slowly accumulate in the mass spectrometer.
Mass spectrometer automated shutoff valves closed	The mass spectrometers are equipped with automated shutoff valves for the calibration vial and the reagent gases. Deliberate operator action or various failures can cause the shutoff valves to close. Shutoff valve closure does not shut off the flow of carrier gas. As a result, hydrogen may slowly accumulate in the mass spectrometer.

Table 2 Hydrogen accumulation mechanisms (continued)

Mechanism	Results
GC off	A GC can be shut down deliberately. It can also be shut down accidentally by an internal or external failure. Different GCs react in different ways. If a 7890A GC equipped with Electronic Pressure Control (EPC) is shut off, the EPC stops the flow of carrier gas. If the carrier flow is *not* under EPC control, the flow increases to its maximum. This flow may be more than some mass spectrometers can pump away, resulting in the accumulation of hydrogen in the mass spectrometer. If the mass spectrometer is shut off at the same time, the accumulation can be fairly rapid.
Power failure	If the power fails, both the GC and mass spectrometer shut down. The flow of carrier gas, however, is not necessarily shut down. As described previously, in some GCs a power failure may cause the carrier gas flow to be set to maximum. As a result, hydrogen may accumulate in the mass spectrometer.

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

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

Precautions

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

Equipment precautions

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.

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

WARNING

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

General laboratory precautions

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

Operating precautions

- Turn off the hydrogen at its source every time you shut down the GC or MS.
- Do not use hydrogen as a collision cell gas.

- Turn off the hydrogen at its source every time you vent the MS (do not heat the capillary column without carrier gas flow).
- Turn off the hydrogen at its source every time shutoff valves in the MS are closed (do not heat the capillary column without carrier gas flow).
- Turn off the hydrogen at its source if a power failure occurs.
- If a power failure occurs while the GC/MS system is unattended, even if the system has restarted by itself:
    1. Immediately turn off the hydrogen at its source.
    2. Turn off the GC.
    3. Turn off the MS and allow it to cool for 1 hour.
    4. Eliminate *all* potential sources of ignition in the room.
    5. Open the vacuum manifold of the MS to atmosphere.
    6. Wait at least 10 minutes to allow any hydrogen to dissipate.
    7. Start up the GC and MS as normal.

When using hydrogen gas, check the system for leaks to prevent possible fire and explosion hazards based on local Environmental Health and Safety (EHS) requirements. Always check for leaks after changing a tank or servicing the gas lines. Always make sure the vent line is vented into a fume hood.

Safety and Regulatory Certifications

The 7000 Series Triple Quad GC/MS conforms to the following safety standards:

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

The 7000 Triple Quad 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
- AUS/NZ ✓

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

CE

The 7000 Series Triple Quad GC/MS is designed and manufactured under a quality system registered to ISO 9001.

Information

The Agilent Technologies 7000 Series Triple Quad 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.

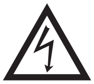
Symbols

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.

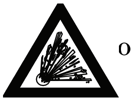
Indicates a hot surface.

Indicates hazardous voltages.



Indicates earth (ground) terminal.

Indicates potential explosion hazard.

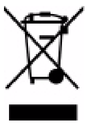


or

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" on page 69.

2

Installing GC Columns

Columns 28
To Prepare a Capillary Column for Installation 30
To Install a Capillary Column in a Split/Splitless Inlet 32
To Condition a Capillary Column 34
To Install a Capillary Column in the GC/MS Interface 35

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. For correct column and flow selection, you must know what type of vacuum system your MS has. The serial number tag on the lower front of the left side panel shows the model number.

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 55 for instructions on how to measure actual flow in your column. Use the flow calculator in the Agilent Instrument Utilities software, and Table 3 to determine an acceptable column flow. For expected column outlet flow pressures, use the values shown in Table 7 for EI mode and Table 10 for CI mode.

Table 3 Gas flows

Feature	**G7000**
High-vacuum pump	Split flow turbo
Optimal HE column gas flow, mL/min (carrier gas)	1 to 2
Reagent gas flow, mL/min	1 to 2
Collision cell gas flow	3 to 4
Maximum recommended gas flow, mL/min*	4
Maximum gas flow, mL/min†	6.5
Maximum column id	0.53 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 7000 Series Triple Quad GC/MS is different from that for previous 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 **If you are using hydrogen as a carrier gas, do not start carrier gas flow until the column is installed in the MS and the MS has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the MS and an explosion may occur. See "Hydrogen Safety".**

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.

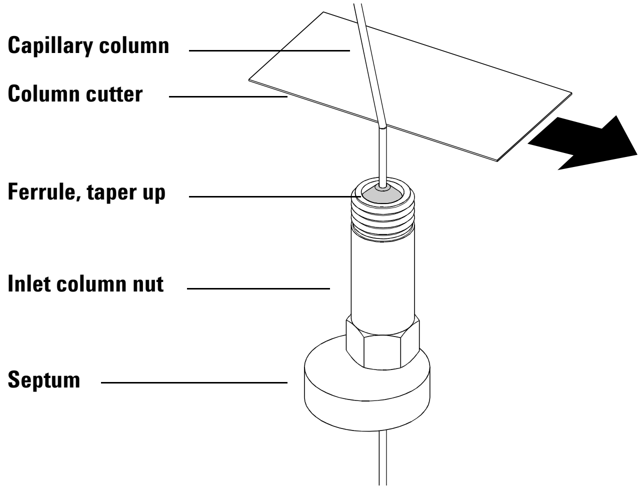


Figure 2 Preparing a capillary column for installation

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 30.)
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.

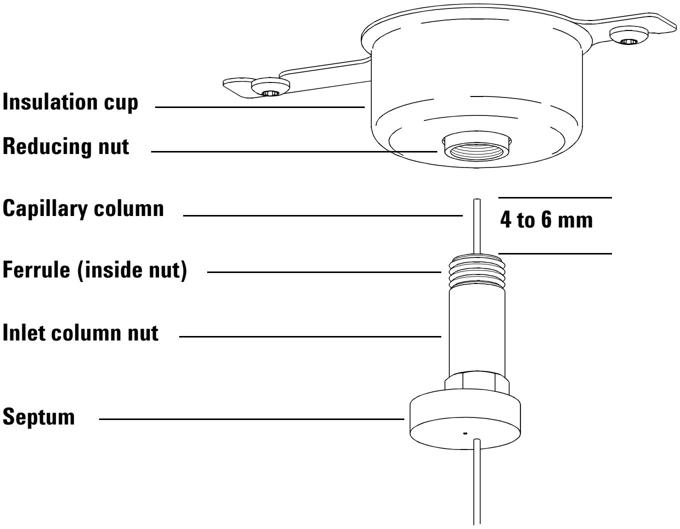


Figure 3 Installing a capillary column for a split/splitless inlet

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

Do not condition your capillary column with hydrogen. Hydrogen accumulation in the GC oven can result in an explosion. If you plan to use hydrogen as your carrier gas, first condition the column with ultrapure (99.999% or better) inert gas such as helium, nitrogen, or argon.

Procedure

WARNING

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

---

1. Install the column in the GC inlet. (See “To Install a Capillary Column in a Split/Splitless Inlet” on page 32.)
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 detector.

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 into the analyzer. If you are using the Agilent capillary flow technology Quick Swap accessory, or any other CFT device, please refer to the appropriate documentation.

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)

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 34.)

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

2 Vent the MS (See "To Vent the MS" on page 65) and open the front analyzer chamber (See "To Open the Front Analyzer Chamber" on page 102). Be sure you can see the end of the GC/MS interface.

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

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

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

7 Slide the column into the GC/MS interface (Figure 4). Adjust the column so it projects 1 to 2 mm past the end of the interface.

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

8 Hand-tighten the nut. Make sure the position of the column does not change as you tighten the nut.

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

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

11 Check the nut's tightness after one or two heat cycles; retighten as appropriate.

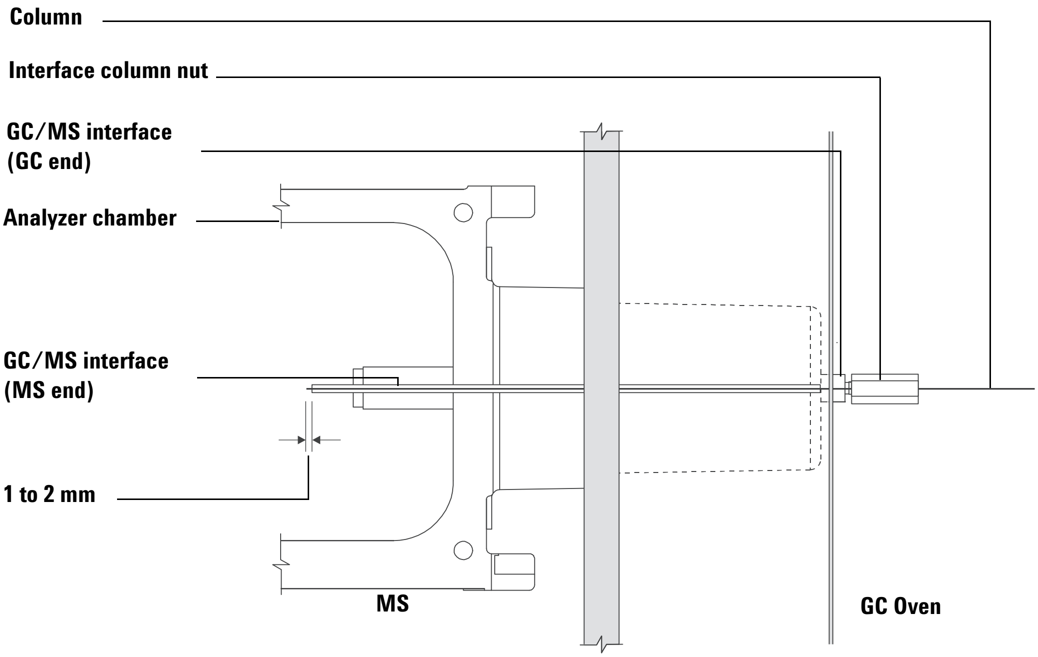


Figure 4 Installing a capillary column in the GC/MS interface

3

Operating in Electron Impact (EI) Mode

Operating the MS from the Data System 40
Monitoring the MS from the LCP 40
LCP Menus 42
The EI GC/MS Interface 43
Before You Turn On the MS 45
Pumping Down 46
Controlling Temperatures 46
Controlling Column Flow 47
Controlling Collision Cell Flow 47
Venting the MS 48
High Vacuum Pressure in EI Mode 49
To Set Monitors for MS Temperature and Vacuum Status 50
To Set the MS Analyzer Temperatures 52
To Set the GC/MS Interface Temperature from the MassHunter Workstation 54
To Calibrate the Column 55
To Configure CC Gas 57
To Set the Collision Cell Gas Flow Rates 58
To Autotune the MS for EI Mode 59
To Open Left Side Panel for Access to the Analyzer Chambers 61
To Pump Down the MS 62
To Vent the MS 65
To Switch from the CI Source to the EI Source 67
To Move or Store the MS 69
To Set the Interface Temperature from the GC 71
To Save a Method on the GC 72

This chapter will explain how to perform some routine operating procedures for the 7000 Series Triple Quad GC/MS in EI mode.

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.

Operating the MS from the Data System

The Agilent MassHunter Data Acquisition Workstation performs tasks such as pumping down, monitoring pressures, setting temperatures, tuning, and preparing to vent. These tasks are described in this chapter. Additional information is described in the manuals and online help supplied with the MassHunter Workstation software.

Your 7000 Triple Quad has the capability of operating in EI mode, with the choice of two EI sources. The standard EI source (G7008A) includes a drawout lens assembly. The High Sensitivity Extractor EI source (G7008B) is an upgrade to the standard source, with an extractor lens in place of the drawout plate and drawout cylinder. This provides higher sensitivity when ionizing the sample. You must specify the type of EI source you are using in the Acquisition software tune file used by your acquisition method.

Monitoring the MS from the LCP

The local control panel (LCP) shows the status of the MS without using the Agilent MassHunter Workstation.

The MassHunter Workstation may be located anywhere on the site's local area network (LAN), so the MassHunter Workstation might not be near the instrument itself. Because the LCP communicates with the MassHunter Workstation you can access MassHunter Workstation monitors, right from the MS.

NOTE

Only certain functions are available from the LCP; the GC/MS MassHunter Data Acquisition Workstation is the full-featured controller for most instrument control operations.

Operating the LCP

The LCP uses the Menu button to query various aspects of the GC/MS.

To access a particular menu option:

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

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

⬆/+	Use [**Up**] to increase the displayed value or to scroll up (such as in a message list).
⬇/–	Use [**Down**] to decrease the displayed value or to scroll down (such as in a message list).
Yes/ Select	Use [**Yes/Select**] to accept the current value.
No/ Cancel	Use [**No/Cancel**] to change the current value or return to the previous menu.

LCP Menus

To access a particular menu option, press [**Menu**] until the desired menu appears, then press [**Item**] until the desired menu item appears. Table 4 through Table 6 list the menus and selections.

Table 4 MS Parameters menu

Action	Description
High Vacuum Pressure	Displays the analyzer pressure.
Ion Source Temp, °C	Displays the actual ion source temperature and the setpoint.
Quad 1 Temp, °C	Displays the actual front mass filter temperature and the setpoint.
Quad 2 Temp, °C	Displays the actual rear mass filter temperature and the setpoint.
Turbo Speed % Full	Displays the turbo pump speed.

NOTE MS parameters cannot be set from the LCP. This must be accomplished by an online GC/MS MassHunter Workstation connected to the MS.

Table 5 Network menu

Action	Description
IP Address	Displays the IP address for the MS.
Subnet Mask	Displays the subnet mask for the MS.
Gateway	Displays “Not found”
MAC Address	Displays the MAC address of the SmartCard in the MS.
Install Standard Network Config	Select “Yes”, sets configuration back to factory default
Install Customized Network Config	Select “Yes”, use Telnet network configuration command to install customized configuration. This is a service modification.

Table 6 LCP Tests Menu

Action	Description
Test LCP Buttons?	Following directions on display, allows you to test each LCP button.
Test LCP Display?	Selecting "Yes" allows you to test the LCP display for dead pixels.
LCP Display Flow Control Test	Allows you to check for irregular patterns in the LCP display.
Test LCP Beep?	Display beeps after 3 seconds.

The EI GC/MS Interface

The GC/MS interface (Figure 5) 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 cover which 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.

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. A sensor (thermocouple) in the interface monitors the temperature.

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

The EI GC/MS interface can only be used with an EI ion source. However, the CI GC/MS interface can be used with either source.

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.**

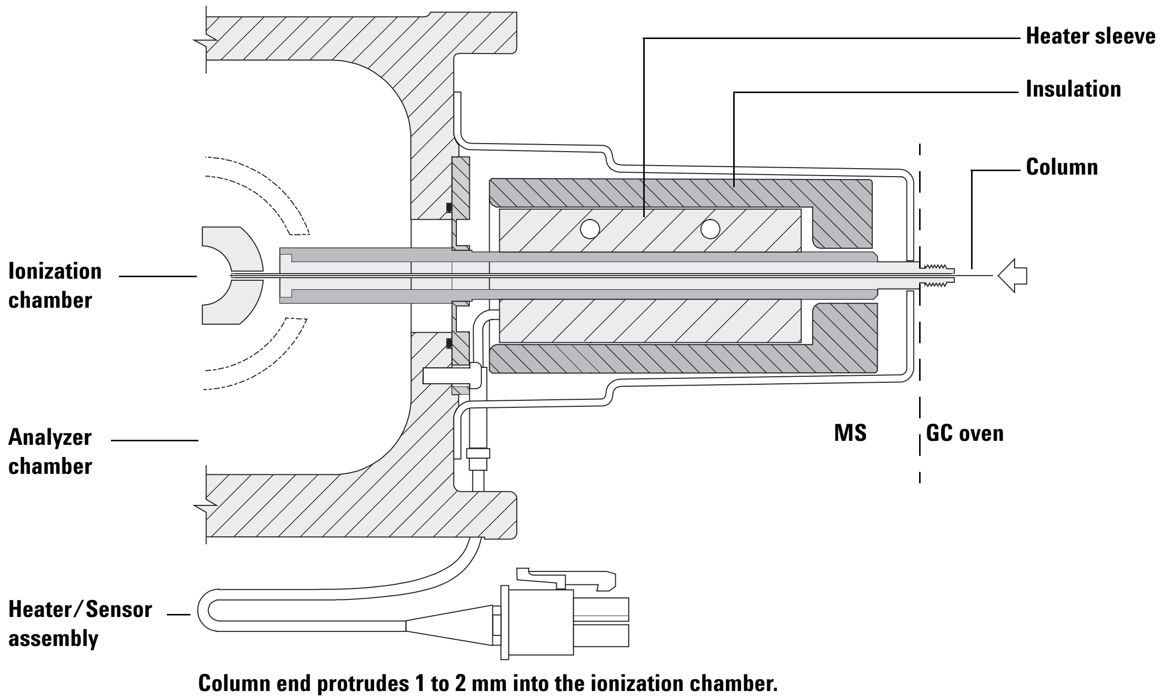


Figure 5 The EI GC/MS interface

Before You Turn On the MS

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

- The vent valve must be closed (the knob turned all the way clockwise). See "To Vent the MS" on page 65.
- All other vacuum seals and fittings must be in place and fastened correctly. All analyzer plate thumbscrews should be open, 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.
- If hydrogen is used as carrier gas, carrier gas flow must be off and the top thumbscrew on the front analyzer sideplate and the top thumbscrew on the rear analyzer sideplate must both be loosely fastened.
- The foreline pump exhaust is properly vented.

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

WARNING **If you are using hydrogen as a carrier gas, do not start carrier gas flow until the MS has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the MS and an explosion may occur. Read "Hydrogen Safety" before operating the MS with hydrogen carrier gas.**

CAUTION The 7000 Triple Quad MS has not been designed for the use of helium in the collision cell when hydrogen is being used as a carrier gas.

Pumping Down

The data system helps you pump down the MS. The process is mostly automated. Once you close the vent valve and turn on the main power switch (while pressing on both analyzer sideplates), 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 turbo pump MS
- Analyzer chamber pressure (vacuum)

The LCP can also display this data.

Controlling Temperatures

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

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

Controlling Column Flow

Carrier gas flow is controlled by column inlet pressure in the GC. For a given inlet pressure, column flow will decrease as the GC oven temperature increases. With electronic pneumatic control (EPC) and the column mode set to **Constant Flow**, the same column flow is maintained regardless of temperature.

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 55.

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 is a combination of two gases mixed at the EPC outlet and sent in a single tubing line to the MS. Typically, these two gases are nitrogen and helium. The gas pressure at the EPC outlet before the mixing tee controls the flow of each gas. These pressures are controlled by the MassHunter Data Acquisition Workstation or directly at the GC panel. See "To Set the Collision Cell Gas Flow Rates" on page 58.

Venting the MS

A program in the data system guides you through the venting process. It turns off the GC and MS heaters and the turbo pump at the correct time. It also lets you monitor temperatures in the MS and indicates when to vent the MS.

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.

The LCP can also display this data.

WARNING

Make sure the GC/MS interface and the analyzer zones are cool (below 100 °C) before you vent the MS. A temperature of 100 °C is hot enough to burn skin; always wear cloth gloves when handling analyzer parts.

WARNING

If you are using hydrogen as a carrier gas, the carrier gas flow must be off before turning off the MS power. If the foreline pump is off, hydrogen will accumulate in the MS and an explosion may occur. Read "Hydrogen Safety" before operating the MS with hydrogen carrier gas.

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 vent while the turbo pump is still spinning at more than 50%.

Do not exceed the maximum recommended total gas flow. See Table 3.

High Vacuum Pressure in EI Mode

The largest influences on operating pressure in EI mode are the carrier gas (column) and collision cell gas flows. Table 7 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 7 Influence of carrier and collision cell gas flows on ion vacuum gauge readings

	CC Gas On N2 = 1.5, He = 2.25		**CC Gas Off**		**CC Gas On N2 = 1.5, He off**	
Column Flow (mL/min)	**Rough Vac**	**High Vac**	**Rough Vac**	**High Vac**	**Rough Vac**	**High Vac**
0.5	$1.58 * 10^{-1}$	$1.11 * 10^{-4}$	$8.82 * 10^{-2}$	$6.05 * 10^{-7}$	$1.36 * 10^{-1}$	$1.31 * 10^{-4}$
0.7	$1.61 * 10^{-1}$	$1.10 * 10^{-4}$	$9.92 * 10^{-2}$	$7.75 * 10^{-7}$	$1.39 * 10^{-1}$	$1.31 * 10^{-4}$
1	$1.66 * 10^{-1}$	$1.10 * 10^{-4}$	$1.00 * 10^{-1}$	$8.38 * 10^{-7}$	$1.44 * 10^{-1}$	$1.31 * 10^{-4}$
1.2	$1.69 * 10^{-1}$	$1.10 * 10^{-4}$	$1.05 * 10^{-1}$	$9.38 * 10^{-7}$	$1.47 * 10^{-1}$	$1.31 * 10^{-4}$
2	$1.80 * 10^{-1}$	$1.11 * 10^{-4}$	$1.22 * 10^{-1}$	$1.36 * 10^{-6}$	$1.60 * 10^{-1}$	$1.32 * 10^{-4}$
3	$1.95 * 10^{-1}$	$1.12 * 10^{-4}$	$1.41 * 10^{-1}$	$1.82 * 10^{-6}$	$1.75 * 10^{-1}$	$1.32 * 10^{-4}$
4	$2.10 * 10^{-1}$	$1.12 * 10^{-4}$	$1.57 * 10^{-1}$	$2.33 * 10^{-6}$	$1.90 * 10^{-1}$	$1.31 * 10^{-4}$
6	$2.37 * 10^{-1}$	$1.13 * 10^{-4}$	$1.89 * 10^{-4}$	$3.29 * 10^{-6}$	$2.18 * 10^{-1}$	$1.34 * 10^{-1}$

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 MS Temperature and Vacuum Status

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

Procedure

1 Select **Method > Edit Monitors** to display the **Select Monitors** dialog box. See Figure 6.

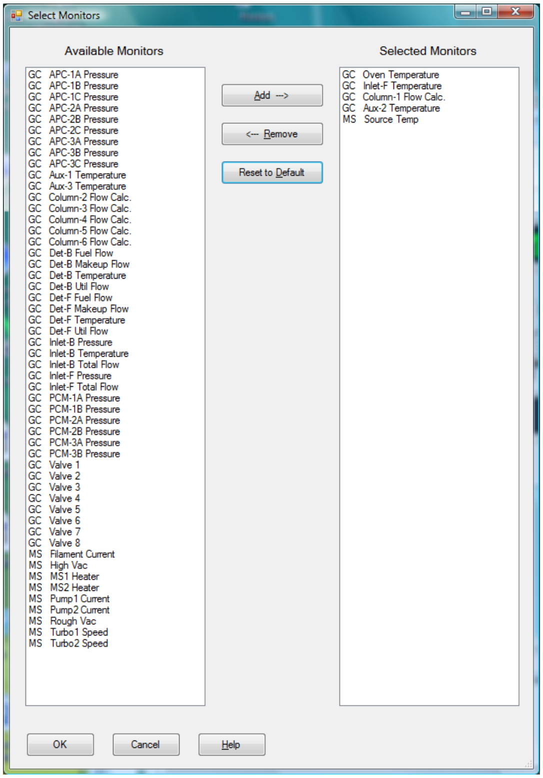


Figure 6 Select Monitors dialog box

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

3 In the **Available Monitors** column, select **MS 1 Heater** and click the **Add** button to move the selection to the **Selected Monitors** column.

4 In the **Available Monitors** column, select **MS 2 Heater** and click the **Add** button to move the selection to the **Selected Monitors** column.

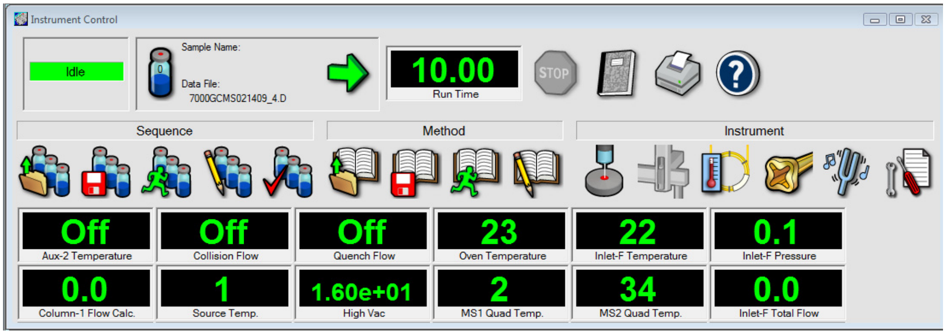
5 In the **Available Monitors** column, select **MS Turbo Speed** and click the **Add** button to move the selection to the **Selected Monitors** column.

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

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

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

9 Select **Window > Arrange Plots and Monitors**, or click and drag each monitor to the desired position. See Figure 7 for one way of arranging the monitors.



Idle

Sample Name:

Data File:
7000GCMS021409_4.D

10.00
Run Time

Off Aux-2 Temperature	Off Collision Flow	Off Quench Flow	23 Oven Temperature	22 Inlet-F Temperature	0.1 Inlet-F Pressure
0.0 Column-1 Flow Calc.	1 Source Temp.	1.60e+01 High Vac	2 MS1 Quad Temp.	34 MS2 Quad Temp.	0.0 Inlet-F Total Flow

Figure 7 Arranging monitors

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

To Set the MS Analyzer Temperatures

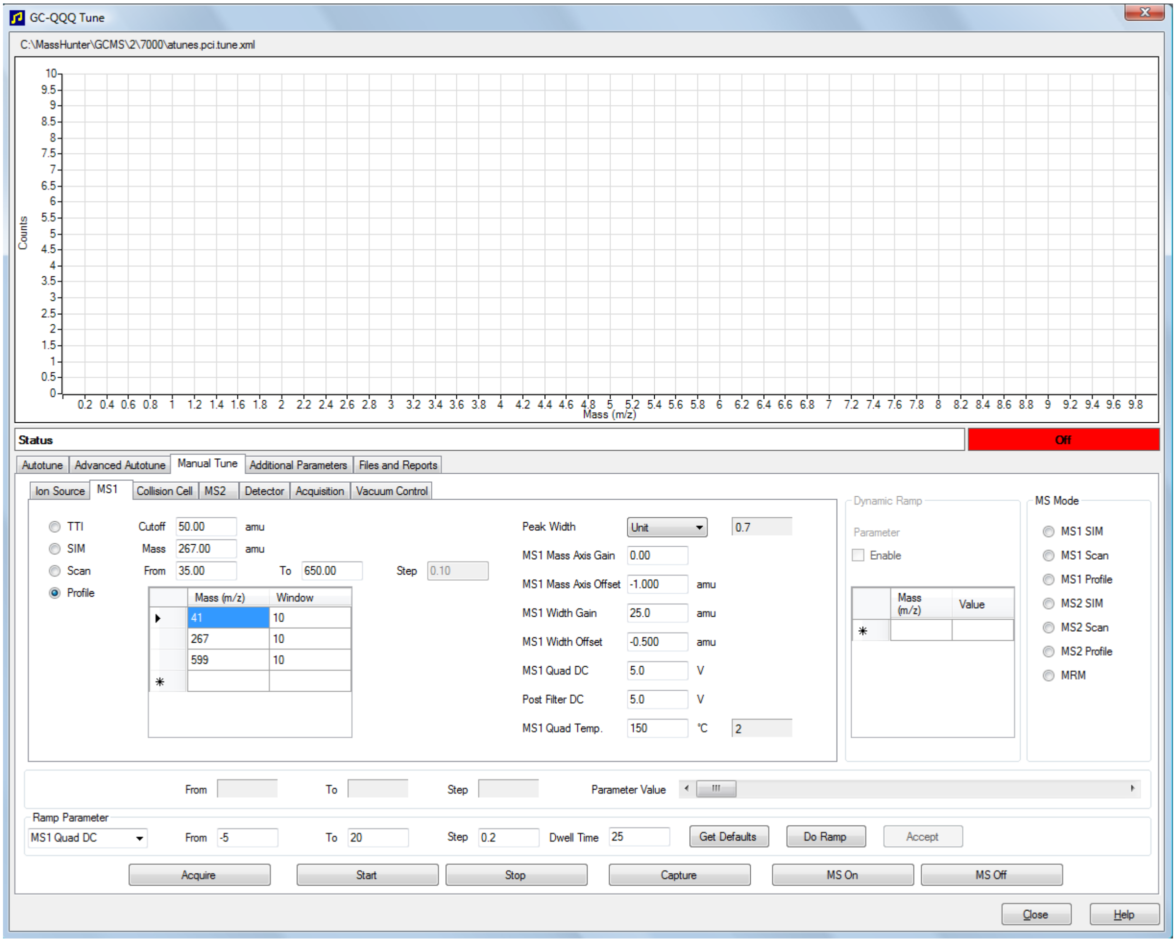
Setpoints for the MS ion source, front quad (MS1), rear quad (MS2), and temperatures are stored in the current tune file. When a method is loaded, the setpoints in the tune file associated with that method are downloaded automatically.

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 8 for recommended setpoints.
3. Select the **MS1** tab to display the MS1 parameters.
4. Enter the temperature setpoint in the **MS1 Quad temp** field. See Table 8 for recommended setpoints.
5. Select the **MS2** tab to display the MS2 parameters.
6. Enter the temperature setpoint in the **MS2 Quad temp** field. See Table 8 for recommended setpoint.
7. Select the **Files and Reports** tab then click the **Save** button to save the tune file with these changes.

Table 8 Recommended temperature settings

Zone	EI operation
MS Source	230 °C
MS Quad 1	150 °C
MS Quad 2	150 °C



C:\MassHunter\GCMS\2\7000\atunes.pci.tune.xml

Status: Off

○ TTI
○ SIM
○ Scan
● Profile

Cutoff 50.00 amu
Mass 267.00 amu
From 35.00 To 650.00 Step 0.10

Mass (m/z)	Window
41	10
267	10
599	10
*	

Peak Width: Unit 0.7
MS1 Mass Axis Gain: 0.00
MS1 Mass Axis Offset: -1.000 amu
MS1 Width Gain: 25.0 amu
MS1 Width Offset: -0.500 amu
MS1 Quad DC: 5.0 V
Post Filter DC: 5.0 V
MS1 Quad Temp.: 150 °C 2

Dynamic Ramp
Parameter
☐ Enable

Mass (m/z)	Value
*	

MS Mode
○ MS1 SIM
○ MS1 Scan
○ MS1 Profile
○ MS2 SIM
○ MS2 Scan
○ MS2 Profile
○ MRM

From To Step Parameter Value

Ramp Parameter: MS1 Quad DC From -5 To 20 Step 0.2 Dwell Time 25

Figure 8 Setting temperatures

The GC/MS interface, ion source, and the MS1 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 source.**

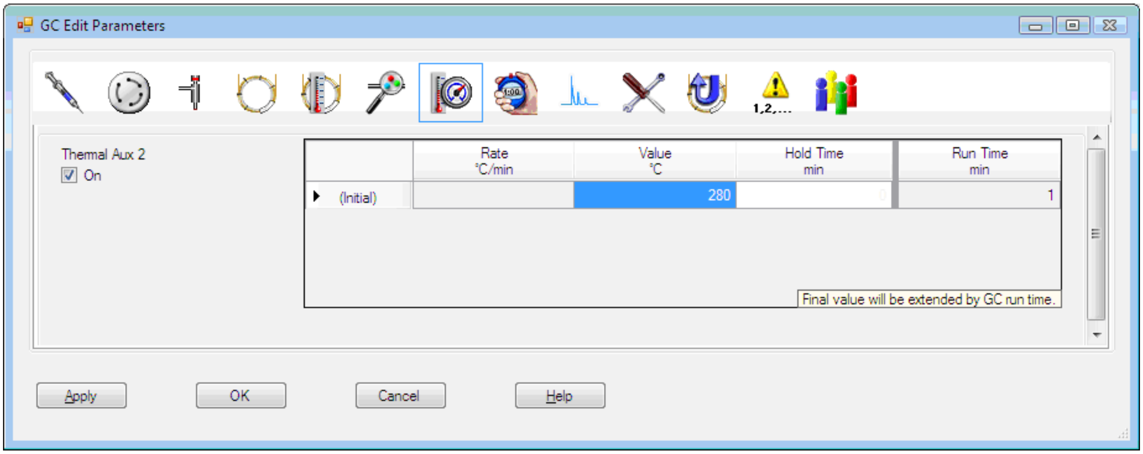
To Set the GC/MS Interface Temperature from the MassHunter Workstation

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** icon to edit the interface temperature (Figure 9). This example has the GC/MS interface temperature configured as Thermal Aux 2.



Thermal Aux 2

☑ On

	Rate °C/min	Value °C	Hold Time min	Run Time min
(Initial)		280	0	1

Final value will be extended by GC run time.

Figure 9 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.

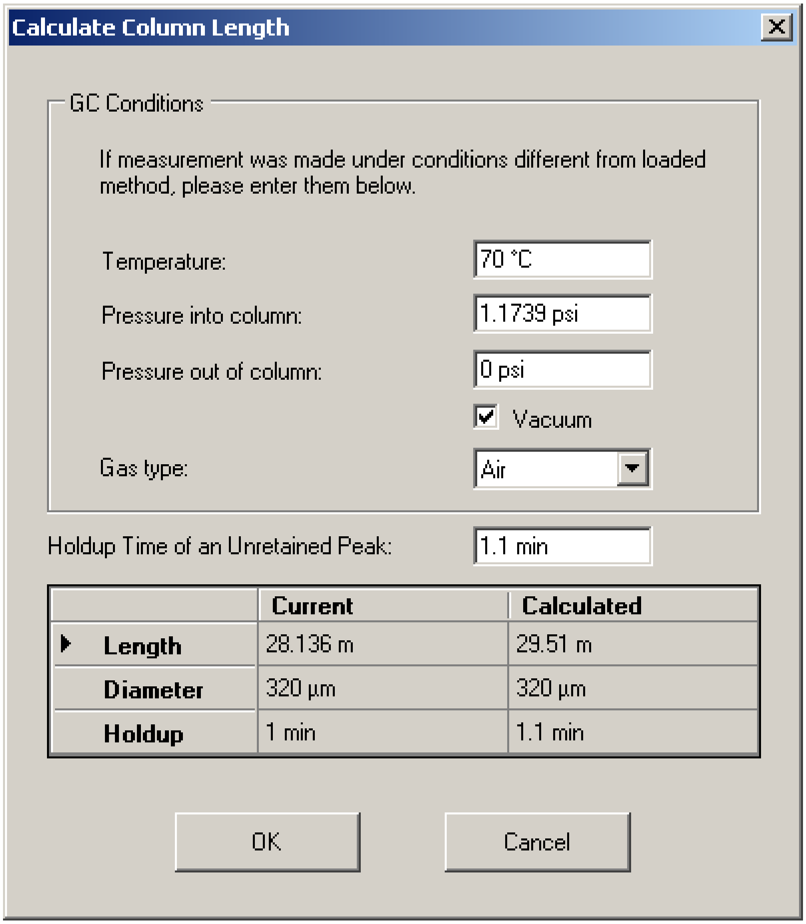
3. Check the heater **On** and type the setpoint in the **Value °C** column. The typical setpoint is 280 °C. The limits are 0 °C to 400 °C. A setpoint below ambient temperature turns off the interface heater.
4. Click **Apply** to download setpoints or click **OK** to download setpoints and close the 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 selected ion monitoring (SIM) of *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 **Instrument > GC Configuration**.
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.



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

Temperature: 70 °C

Pressure into column: 1.1739 psi

Pressure out of column: 0 psi

☑ Vacuum

Gas type: Air

Holdup Time of an Unretained Peak: 1.1 min

	Current	Calculated
Length	28.136 m	29.51 m
Diameter	320 µm	320 µm
Holdup	1 min	1.1 min

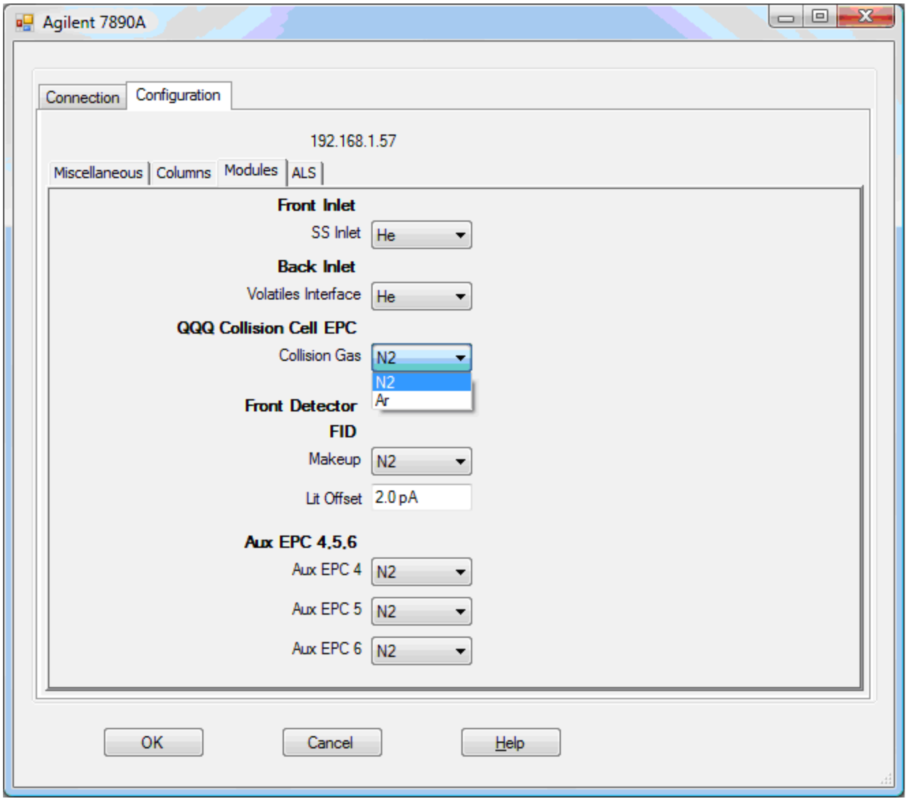
Figure 10 Calculate Column Length dialog box

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 CC Gas

1. From the **MassHunter Data Acquisition Workstation Instrument Control** panel, select **Instrument > Configuration**.
2. Select the **Modules** tab to display the screen. See Figure 11.



192.168.1.57

Front Inlet
SS Inlet: He

Back Inlet
Volatiles Interface: He

QQQ Collision Cell EPC
Collision Gas: N2

Front Detector
FID
Makeup: N2
Lit Offset: 2.0 pA

Aux EPC 4,5,6
Aux EPC 4: N2
Aux EPC 5: N2
Aux EPC 6: N2

Figure 11 Configure collision cell gas

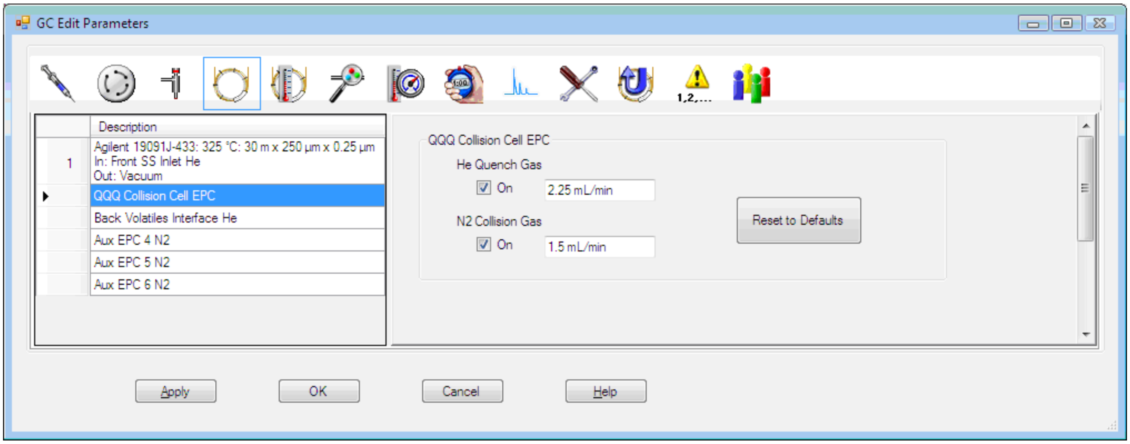
3. From the **QQQ Collision Cell EPC** drop-down menu, select the collision cell gas.
4. Click **OK** to save the configuration.

To Set the Collision Cell Gas Flow Rates

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 12.
3. Select **QQQ Collision Cell EPC** in the description list.
4. Enter the required gas flow rates in the appropriate field.

NOTE Helium should only be used as a quench gas in the collision cell when it is also used as the carrier gas. When hydrogen has been used as the carrier gas, turn off the helium flow to the collision cell, and cap off the helium input line with a leak-tight fitting.

5. Click the **He Quench Gas On** check box to allow the He quench gas flow. 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.



	Description
1	Agilent 19091J-433: 325 °C: 30 m x 250 µm x 0.25 µm In: Front SS Inlet He Out: Vacuum
	QQQ Collision Cell EPC
	Back Volatiles Interface He
	Aux EPC 4 N2
	Aux EPC 5 N2
	Aux EPC 6 N2

QQQ Collision Cell EPC

He Quench Gas
☑ On 2.25 mL/min

N2 Collision Gas
☑ On 1.5 mL/min

Figure 12 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 temperatures) that will be used for data acquisition.
2. In the **Instrument Control** panel, click the **MS Tune** icon to display the **GC-QQQ Tune** dialog box.
3. The current tune file is displayed in the upper left corner of the **GC-QQQ 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 source** for a standard EI ion source, or an **EI source with extractor** if you are using a high sensitivity EI ion source containing 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 Autotune** 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.

To Open Left Side Panel for Access to the Analyzer Chambers

The left side panel should only be opened to access the front and rear analyzer chambers or the analyzer side plates. This is necessary to pump down, clean or change the ion source, replace a filament, or replace the electron multiplier horn. If you need to open the left side panel (Figure 37), follow this procedure:

Procedure

1 Remove the front right analyzer window cover by pulling it out from the top then forward from the bottom to free the window. This cover is held in place by magnets.

2 Pull gently on the front left window and allow the left panel to slide forward and down.

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

Procedure

1. Remove the front analyzer window and open the left analyzer panel to access the vent valve and the analyzer quad driver boards. See "To Open Left Side Panel for Access to the Analyzer Chambers" on page 61.
2. Partially close the vent valve, leaving it slightly open, by turning it clockwise.
3. Plug the power cord into a grounded electrical outlet.
4. Turn on the Triple Quad power switch and wait for the word "Agilent" to display in the Local Control Panel.
5. Press lightly on the front and rear analyzer quad driver boards to ensure a correct seal. Press on the metal box on the quad driver board.

CAUTION Do not push on the filament board safety cover while pressing on the analyzer boards. 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, the interface column nut, or the vent valve.

6. Start the MassHunter Data Acquisition program. If the Triple Quad 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.

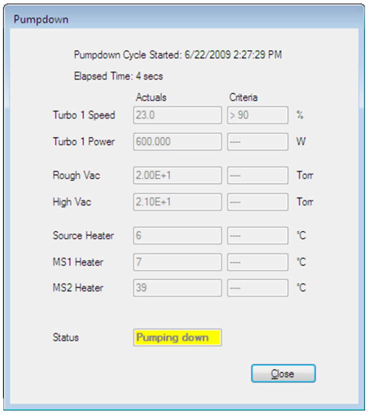
7 Select the **MS Tune** icon from the **Instrument Control** panel.

8 Select the **Manual Tune** tab.

9 Select the **Vacuum Control** tab.

10 Click the **Pumpdown** button.

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



Pumpdown Cycle Started: 6/22/2009 2:27:29 PM

Elapsed Time: 4 secs

	Actuals	Criteria	
Turbo 1 Speed	23.0	> 90	%
Turbo 1 Power	600.000	----	W
Rough Vac	2.00E+1	----	Torr
High Vac	2.10E+1	----	Torr
Source Heater	6	----	°C
MS1 Heater	7	----	°C
MS2 Heater	39	----	°C
Status	Pumping down		

Figure 13 Pumping down

CAUTION

Within 10 to 15 minutes the turbo pump speed should be up to 80% (Figure 13). 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.

CAUTION

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

12 When you hear a hissing sound from the vent valve, remove your hands from the side plates and close the vent valve.

13 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) heaters. The temperature setpoints are stored in the current autotune file.

14 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.

15 Tune the MS. (See “To Autotune the MS for EI Mode” on page 59 or “To Perform a CI Autotune” on page 90.)

To Vent the MS

Procedure

1. Click the **MS Tune** icon from the **Instrument Control** panel.
2. Select the **Manual Tune** tab.
3. Select the **Vacuum Control** tab.
4. Click the **Vent** button.
5. Set the GC/MS interface heater and the GC oven temperatures to ambient (room temperature).

WARNING

If you are using hydrogen as a carrier gas, the carrier gas flow must be off before turning off the MS power. If the foreline pump is off, hydrogen will accumulate in the MS and an explosion may occur. Read "Hydrogen Safety" before operating the MS with hydrogen carrier gas.

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

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

8. Remove the analyzer window cover (See "To Open Left Side Panel for Access to the Analyzer Chambers" on page 61).

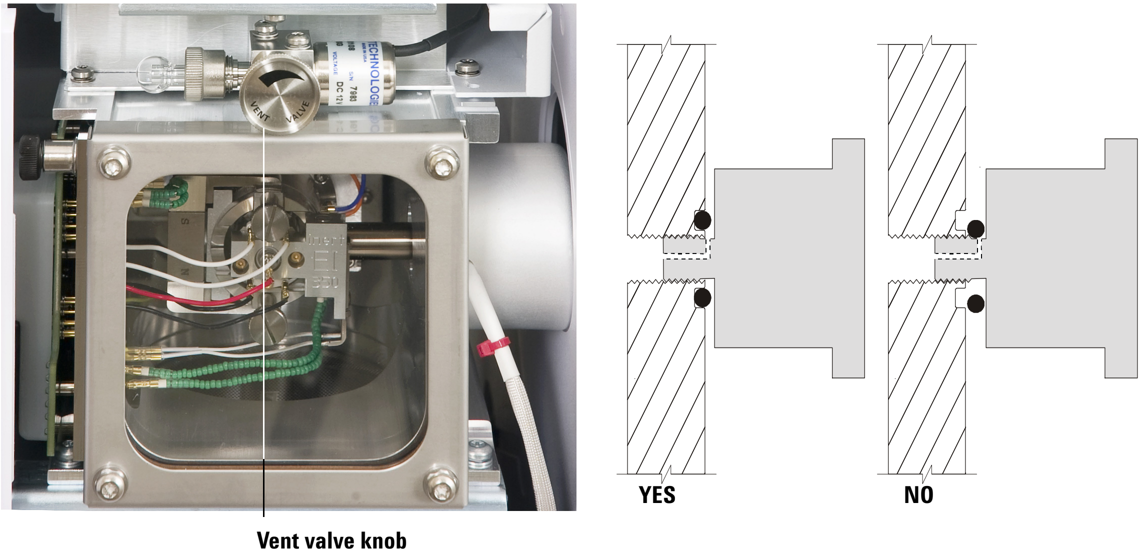


Figure 14 The MS vent valve knob

9 Turn the vent valve knob (Figure 14) counterclockwise *only* 3/4 turn or until you hear the hissing sound of air flowing into the analyzer chamber.

Do *not* turn the knob too far or the O-ring may fall out of its groove. Be sure to retighten the knob before pumping down.

WARNING **Allow the analyzers to cool to near room temperature before touching them.**

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

To Switch from the CI Source to the EI Source

Procedure

1. Vent the MS. See "To Vent the MS" on page 65. The software will prompt you for the appropriate actions.
2. Open the left side access panel. See "To Open Left Side Panel for Access to the Analyzer Chambers" on page 61.
3. Open the front analyzer chamber. See "To Open the Front Analyzer Chamber" on page 102.
4. Remove the CI interface tip seal. See "To Install the CI Interface Tip Seal" on page 132.
5. Remove the CI ion source. See "To Remove the CI Ion Source" on page 121.
6. Install the EI ion source. See "To Install the EI Ion Source" on page 119.
7. Place the CI ion source and interface tip seal in the ion source storage box.

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

CAUTION Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded antistatic wrist strap and take other antistatic precautions ***before*** you open the analyzer chamber. See "Electrostatic discharge is a threat to MS electronics" on page 15.

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8. If necessary, start the MassHunter Data Acquisition program and pump down the MS. (See"To Pump Down the MS" on page 62)
9. At pumpdown, for systems configured with both an EI and CI source, the user is asked to identify the source in the MS. Select the EI source.
10. Load a suitable method for a standard EI source, or for a high sensitivity EI source, depending upon the type of EI source you are using.
11. Click the **MS Tune** icon in the **Instrument Control** panel to display the **GC-QQQ Tune** dialog box and select the **Autotune** tab.

    The method selects the correct EI source.

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

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

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

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

To Move or Store the MS

Materials needed

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

Procedure

1. Vent the MS. (See "To Vent the MS" on page 65.)
2. Remove the column and install a blank ferrule and interface nut.
3. Move the MS away from the GC (See the *Agilent 7000 Series Triple Quad GC/MS Troubleshooting and Maintenance Manual*).
4. Unplug the GC/MS interface heater cable from the GC.
5. Install the interface nut with the blank ferrule.
6. Remove front analyzer window and open the side analyzer cover (See "To Open Left Side Panel for Access to the Analyzer Chambers" on page 61).
7. Finger-tighten the side plate thumbscrews for both analyzers. (See Figure 15).

CAUTION Do not overtighten the side plate thumbscrews. Overtightening will strip the threads in the analyzer chambers. It will also warp the side plate and cause leaks.

8. Plug the MS power cord in.
9. Switch the MS on to establish a rough vacuum. When you hear the hissing sound of the pump, close the vent valve. Wait about 2 to 3 minutes.
10. Switch the MS off.
11. Close the analyzer cover and replace front analyzer window.
12. Disconnect the LAN, remote, and power cables.

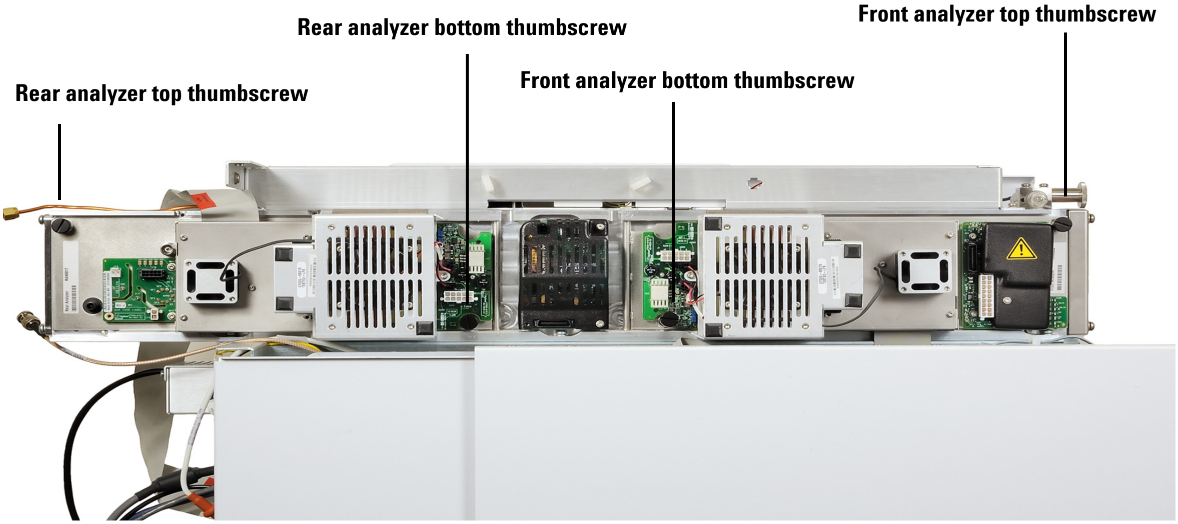


Figure 15 Side plate thumbscrews

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

CAUTION

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

To Set the Interface Temperature from the GC

The interface temperature can be set directly at the GC. For the Agilent 7890A GC, this is usually the Aux #2 temperature. See the *7890A GC Advanced User Guide* for details.

CAUTION Never exceed the maximum temperature of your column.

CAUTION Make sure that the carrier gas is turned on and the column has been purged of air before heating the GC/MS interface or the GC oven to avoid damaging the column.

Procedure

1. Press **[Aux Temp #]** and scroll to the interface temperature. Press **[Enter]**.
2. Scroll to **Temperature**. Enter a value and press **[Enter]**.
3. Scroll to **Initial time**. Enter a value and press **[Enter]**.
4. Scroll to **Rate 1**. Enter **0** to end the program here or enter a positive value to create a temperature program.

If you want the new setpoint saved with a method stored on the GC, press **OK** to save the method. You can also upload the GC method to the MassHunter Workstation to save the new setpoints made at the GC keypad. When a new method is loaded, all the setpoints in the new method will overwrite those currently in the GC.

To Save a Method on the GC

Procedure

1. Press **[Method]** and scroll to the specific method number.
2. Press **[Store]** and **[On/Yes]** to store the new method using the chosen number. Alternatively, press **[Off/No]** to return to the stored methods list without saving the method.

   A message is displayed if a method with the number you selected already exists.
3. Press **[On/Yes]** to *replace* the existing method or **[Off/No]** to return to the stored methods list without saving the method.

4

Operating in Chemical Ionization (CI) Mode

Setting Up Your MS to Operate in CI Mode 74
The CI GC/MS Interface 75
Operating the CI MS 77
High Vacuum Pressure in CI Mode 78
Other Reagent Gases 79
CI Autotune 81
The Flow Control Module 83
To Switch from the EI Source to the CI Source 85
To Operate the Reagent Gas Flow Control Module 87
To Set a Reagent Gas Flow 89
To Perform a CI Autotune 90

This chapter provides information and instructions for operating the 7000 Series Triple Quad 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 and GC/MS interface tip seal are installed.
- Make sure the reagent gas plumbing has no air leaks. This is determined in PCI mode, checking for *m/z* 32 after the methane pretune.
- Make sure the reagent gas inlet line(s) are equipped with gas purifiers (not applicable for ammonia).

The CI GC/MS Interface

The CI GC/MS interface (Figure 16) 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 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.

Reagent gas is plumbed into the interface. The tip of the interface assembly extends into the ionization chamber. A spring-loaded seal keeps reagent gases from leaking out around the tip. The reagent gas 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. A sensor (thermocouple) in the interface monitors the temperature.

This interface can also be used for EI operation.

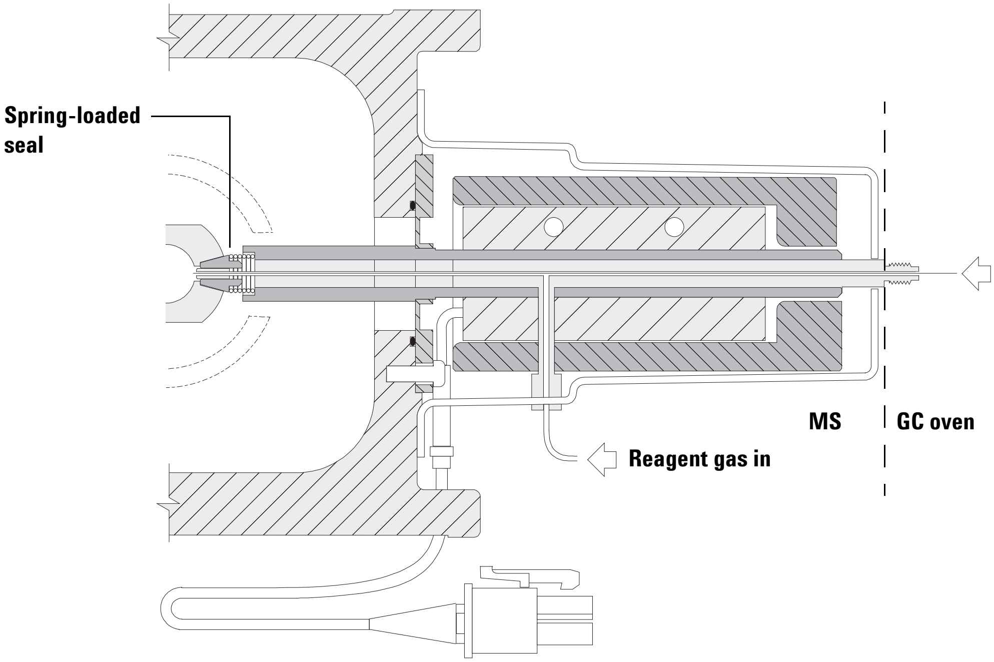
The interface should be operated in the 250 ° to 350 °C range. Subject to that restriction, the interface temperature should be slightly higher than the maximum GC oven temperature, but ***never*** higher than the maximum column temperature.

See Also

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

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.**



Column end protrudes 1 to 2 mm into the ionization chamber.

Figure 16 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, source temperature (Table 9), and electron energy may need to be optimized for your specific analyte.

Table 9 Temperatures for CI operation

	Ion source	Front analyzer	Rear analyzer	GC/MS interface
PCI	300 °C	150 °C	150 °C	280 °C
NCI	150 °C	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 10 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 10). They were taken with the following set of conditions. Note that these are typical PCI temperatures:

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

Table 10 Typical analyzer vacuum with reagent gas flow

	Collision cell gas flow on N2 = 1.5, He = 2.25		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}
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 7000 Series Triple Quad 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 *Appendix A*, “Chemical Ionization Theory. 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 5, “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 7000 Series Triple Quad 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 11). 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.

CAUTION

After the source is changed from EI to CI or vented for any other reason, the MS must be purged and baked out for at least 2 hours before tuning. Longer bakeout is recommended before running samples requiring optimal sensitivity.

There are no tune performance criteria. If CI autotune completes, it passes.

EMVolts (electron multiplier voltage) at or above 2600 V, however, indicates a problem. If your method requires EMVolts set at +400, you may not have adequate sensitivity in your data acquisition.

CAUTION

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

Table 11 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

Table 11 CI tune default settings (continued)

Parameter	**Methane**		**Isobutane**		**Ammonia**		**EI**
Entrance lens offset	20 V	20 V	20 V	20 V	20 V	20 V	25 V
EM volts	1200	1400	1200	1400	1200	1400	1300
Shutoff valve	Open	Open	Open	Open	Open	Open	Closed
Gas select	A	A	B	B	B	B	None
Suggested flow	20%	40%	20%	40%	20%	40%	N/A
Source temp	250 °C	150 °C	250 °C	150 °C	250 °C	150 °C	230 °C
Front quad temp	150 °C	150 °C	150 °C	150 °C	150 °C	150 °C	150 °C
Rear 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 (Figure 17 and Table 12) 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 back panel provides Swagelok inlet fittings for methane (**CH4**) and one **OTHER** reagent gas. The software refers to them as **Gas A** and **Gas B,** respectively. If you are not using a second reagent gas, cap the **OTHER** fitting to prevent accidental admission of air to the analyzer. Supply reagent gases at 25 to 30 psi (170 to 205 kPa).

The shutoff valve prevents contamination of the flow control module by atmosphere while the MS is vented or by PFTBA during EI operation. The MS monitors will reflect **On** as **1** and **Off** as **0** (see Table 12).

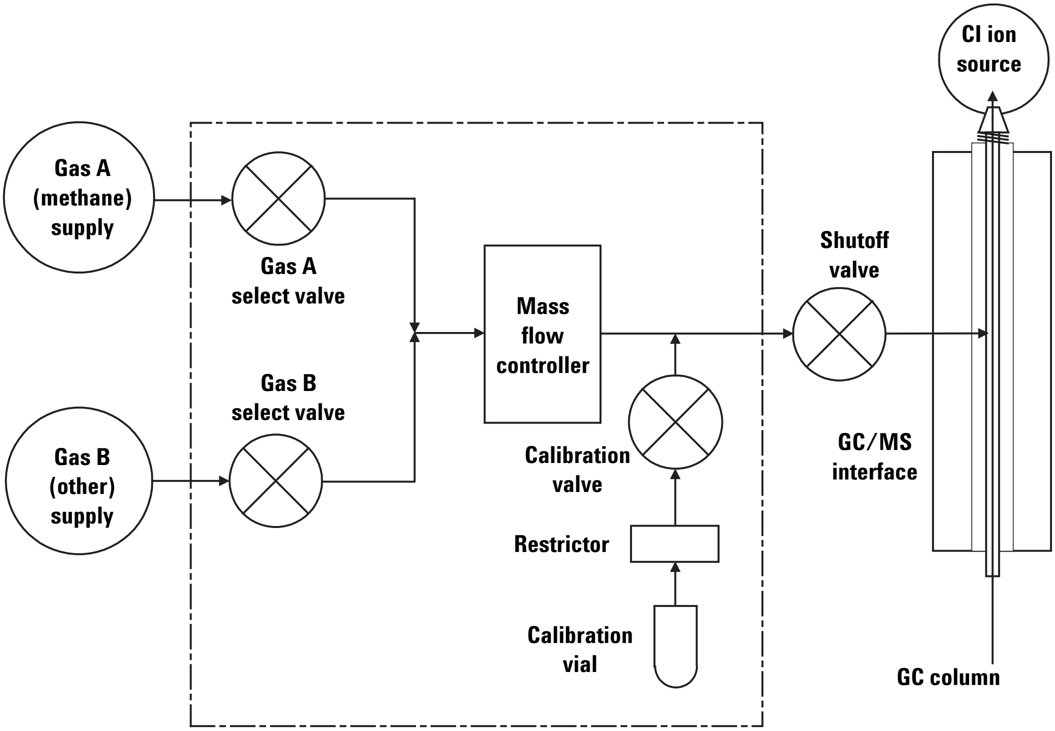


Figure 17 Reagent gas flow control module schematic

Table 12 Flow control module state diagram

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

The **Open** and **Closed** states are shown in the monitors as **1** and **0** respectively.

To Switch from the EI Source to the CI Source

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

Procedure

1. Vent the MS. See "To Vent the MS" on page 65.
2. Open the front analyzer chamber. See "To Open the Front Analyzer Chamber" on page 102.
3. Remove the EI ion source. See "To Remove the EI Ion Source" on page 105.

CAUTION Electrostatic discharges to analyzer components are conducted to the side board where they can damage sensitive components. Wear a grounded antistatic wrist strap. See "Electrostatic discharge is a threat to MS electronics" . Take antistatic precautions ***before*** you open the analyzer chamber.

---

4. Install the CI ion source. See "To Install the CI Ion Source" on page 130.
5. Install the interface tip seal. See "To Install the CI Interface Tip Seal" on page 132.
6. Close the analyzer.
7. Pump down the MS. See "To Pump Down the MS" on page 62. At pumpdown, for systems configured with both an EI and CI source, the user is asked to identify the source in the MS. Select the CI source.
8. Load a suitable PCI or NCI method for use with the CI source.
9. Click the **MS Tune** icon in the **Instrument Control** panel to display the **GC-QQQ Tune** dialog box and select the **Autotune** tab.

   The method selects the correct PCI or NCI source and reagent gas setting.
10. Select the **Tune from default settings** check box because you have changed the ion source.
11. Select **Print autotune report** check box to automatically print a tune report.

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

13 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 13 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

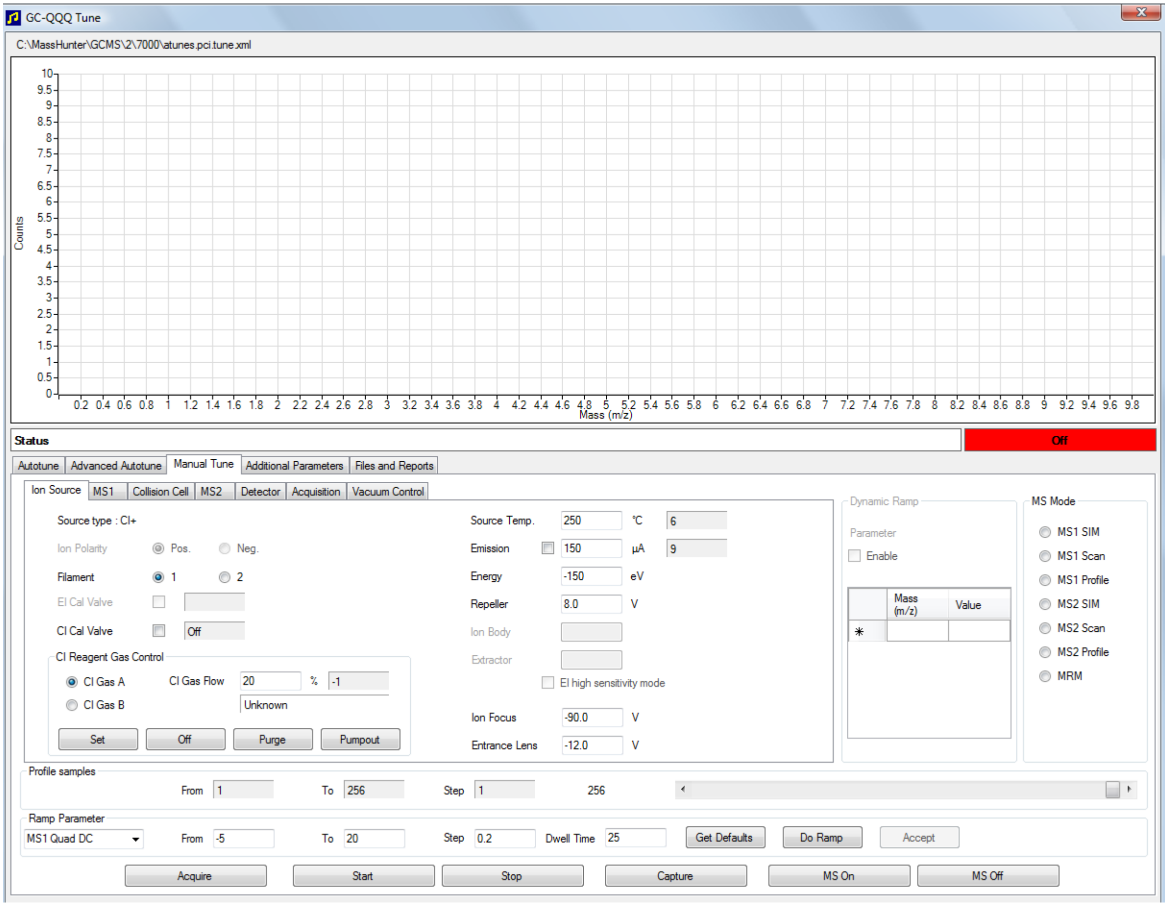
Notes for Table 13:

- **N/A** Not available.
- **Abundance target** Adjust higher or lower to get desired signal abundance. Higher signal abundance also gives higher noise abundance. This is adjusted for data acquisition by setting the EMV in the method.
- **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-QQQ Tune** dialog box. Select the **Manual Tune** tab then select the **Ion Source** tab to display the ion source parameters.



GC-QQQ Tune

C:\MassHunter\GCMS\2\7000\atunes.pci.tune.xml

Status: Off

Source type : CI+

Ion Polarity: ● Pos. ○ Neg.

Filament: ● 1 ○ 2

EI Cal Valve: ☐

CI Cal Valve: ☐ Off

CI Reagent Gas Control: ● CI Gas A ○ CI Gas B

CI Gas Flow: 20 % -1

Unknown

Source Temp.: 250 °C 6

Emission: ☐ 150 µA 9

Energy: -150 eV

Repeller: 8.0 V

☐ EI high sensitivity mode

Ion Focus: -90.0 V

Entrance Lens: -12.0 V

Dynamic Ramp: Parameter ☐ Enable

MS Mode: ○ MS1 SIM ○ MS1 Scan ○ MS1 Profile ○ MS2 SIM ○ MS2 Scan ○ MS2 Profile ○ MRM

Profile samples: From 1 To 256 Step 1 256

Ramp Parameter: MS1 Quad DC From -5 To 20 Step 0.2 Dwell Time 25

Figure 18 CI flow control

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

CI Gas A - Selects Methane as the reagent gas.

CI Gas B - Selects the gas connected to the B inlet on the gas flow controller as the reagent gas.

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 both reagent gas valves 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-QQQ 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 **CI Gas A** if you are using methane for the reagent gas or select **CI Gas B** to use the reagent gas attached to the CI reagent gas controller’s B gas inlet for the reagent gas.
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.

Procedure

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

---

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-QQQ 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. Click on **Methane** if you are using methane as the reagent gas or click on **Ammonia** if using the gas attached to the reagent gas controllers B port as the reagent gas.
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 **CI Gas A** or **CI Gas B** as 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

General Maintenance

Before Starting 94
Maintaining the Vacuum System 99
Maintaining the Analyzer 100
To Open the Front Analyzer Chamber 102
To Remove the EI Ion Source 105
To Disassemble the Standard EI Ion Source 108
To Disassemble the EI Extractor Ion Source 110
To Clean the EI Ion Source 112
To Assemble the Standard EI Ion Source 116
To Assemble the EI Extractor Ion Source 117
To Install the EI Ion Source 119
To Remove the CI Ion Source 121
To Disassemble the CI Ion Source 124
To Clean the CI Ion Source 126
To Assemble the CI Ion Source 128
To Install the CI Ion Source 130
To Install the CI Interface Tip Seal 132
To Remove a Filament 134
To Install a Filament 136
To Attach Wiring from the Ion Source to the Side Board 137
To Close the Front Analyzer Chamber 141
To Remove Left Rear Cover for Access to the Rear Analyzer Chamber 142
To Open the Rear Analyzer Chamber 144
To Replace the Electron Multiplier Horn 147
To Close the Rear Analyzer Chamber 149

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 14. 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 14 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
Lubricate sideplate or vent valve O-rings†				X
Replace GC gas supplies				X

* Or as needed.

† Vacuum seals other than the side plate O-ring and vent valve O-ring do not need to be lubricated. Lubricating other seals can interfere with their correct function.

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 **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.**

WARNING **The foreline pump can cause burns if touched when operating. It could have an optional safety shield to prevent the user from touching it.**

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

Chemical residue

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

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

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

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.

Electrostatic discharge

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

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

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

CAUTION

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

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

Maintaining the Vacuum System

Periodic maintenance

As listed in Table 14 on page 94, some maintenance tasks for the vacuum system must be performed periodically. These include:

- Checking the foreline pump fluid (every week)
- Checking the calibration vial(s) (every 6 months)
- Replacing the foreline pump oil (every 6 months or as needed)
- Tightening the foreline pump oil box screws (first oil change after installation)
- Replacing the foreline pump (typically every 3 years)

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. See the *Agilent 7000 Series Triple Quad 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 7000 Series Triple Quad 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 7000 Series 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
- Replacing the electron multiplier horn

The *Agilent 7000 Series Triple Quad 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

Cleanliness

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

CAUTION If not done correctly, analyzer maintenance can introduce contaminants into the 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. See “Electrostatic discharge” on page 98 for more information.

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 (see “Electrostatic discharge” on page 98) and take other antistatic precautions *before* you open the analyzer chambers.

Analyzer parts that should not be disturbed

The mass filters (quadrupoles) and the collision cell require no periodic maintenance. In general, the mass filters should never be disturbed. In the event of extreme contamination, they can be cleaned, but such cleaning should only be done by a trained Agilent Technologies service representative. The HED ceramic insulator must never be touched.

CAUTION

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

More information is available

If you need more information about the locations or functions of analyzer components, refer to the *Agilent 7000 Series Triple Quad GC/MS Troubleshooting and Maintenance Manual*.

To Open the Front Analyzer Chamber

The front analyzer chamber should only be opened to clean or replace the ion source or to change a filament.

Materials needed

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

CAUTION

Electrostatic discharges to analyzer components are conducted to the quad driver board, where they can damage sensitive components. Wear a grounded antistatic wrist strap and take other antistatic precautions (see “Electrostatic discharge” on page 98) before you open the analyzer chamber.

Procedure

1. Vent the MS. (See “Venting the MS” on page 48.)
2. Open the left side panel. (See “To Open Left Side Panel for Access to the Analyzer Chambers” on page 61.)

WARNING

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

CAUTION

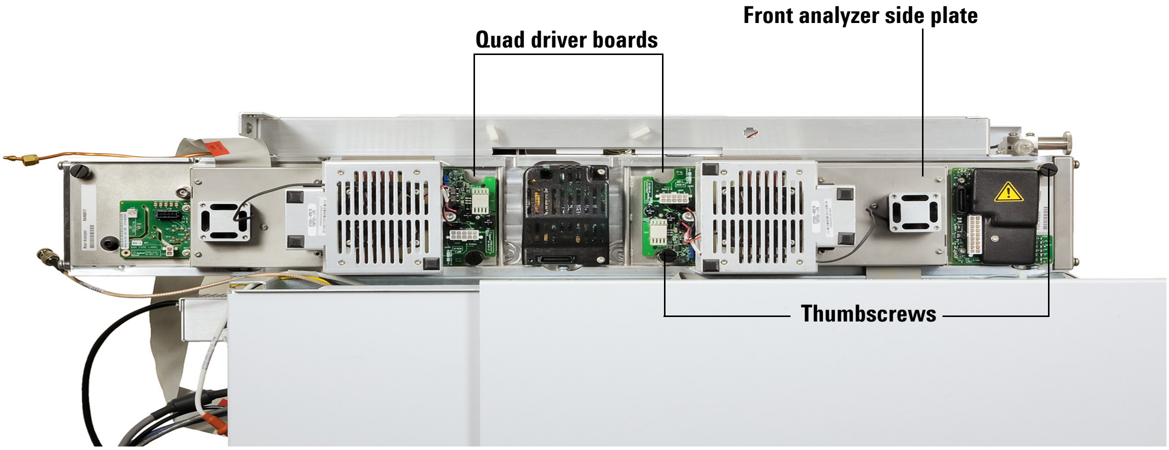
Always wear clean gloves to prevent contamination when working in the analyzer chamber.

3. Loosen the front analyzer side plate thumbscrews (Figure 19) if they are fastened.

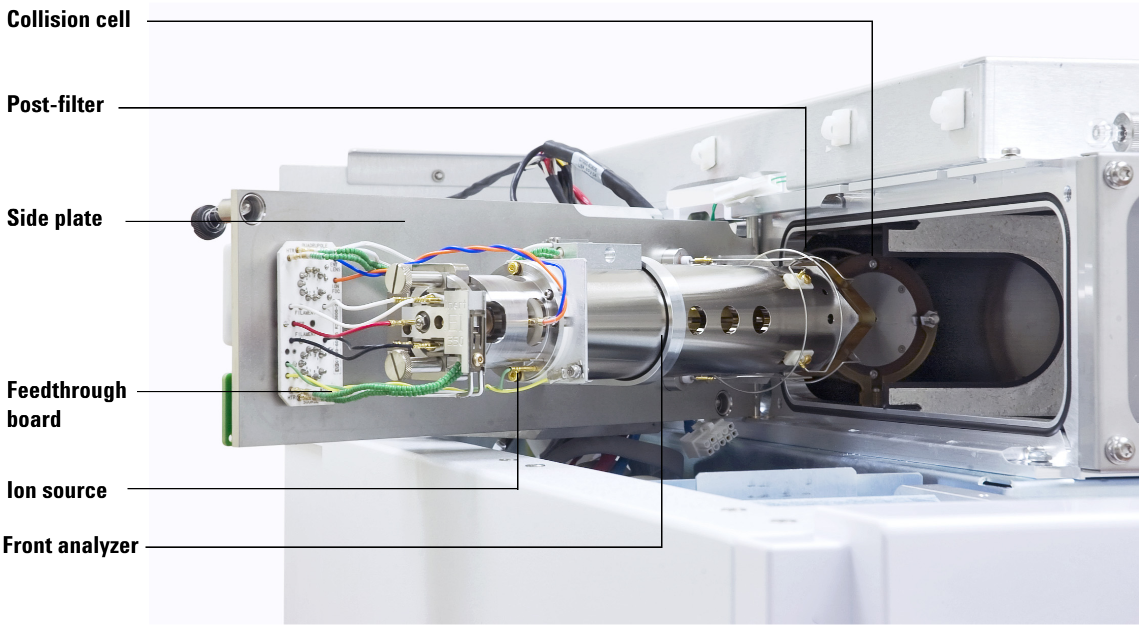
The bottom thumbscrew on the front analyzer side plate should be unfastened during normal use. It is only fastened during shipping. The top thumbscrew on the front side plate should only be fastened if hydrogen or other flammable or toxic substances are used for carrier gas, or during CI operation.

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

4 *Gently* swing the side plate out.



Chamber closed (cables removed for clarity)



Chamber open

Figure 19 The front analyzer chamber

To Remove the EI Ion Source

Materials needed

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

Procedure

1 Vent the MS. (See “To Vent the MS” on page 65.)

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

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

2 Open the front analyzer chamber. (See “To Open the Front Analyzer Chamber” on page 102.)

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

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

3 Disconnect the wires from the ion source. Do not bend the wires any more than necessary (Figure 20 and Table 15).

4 Trace the wires for the ion source heater and temperature sensor to the feedthrough board. Disconnect them there (Figure 20).

Table 15 EI Ion source wires

Wire color	Connects to	Number of leads	
		Extractor	**Drawout assembly**
Blue	Entrance lens	1	1
Orange	Ion focus	1	1
Brown	Extractor lens	1	n/a
White	Filament 1 (top filament)	2	2
Red	Repeller	1	1
Black	Filament 2 (bottom filament)	2	2
Green beaded	Feedthrough board (bottom left)	2	2
White	Feedthrough board (bottom middle)	2	2

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

6 Pull the ion source out of the source radiator.

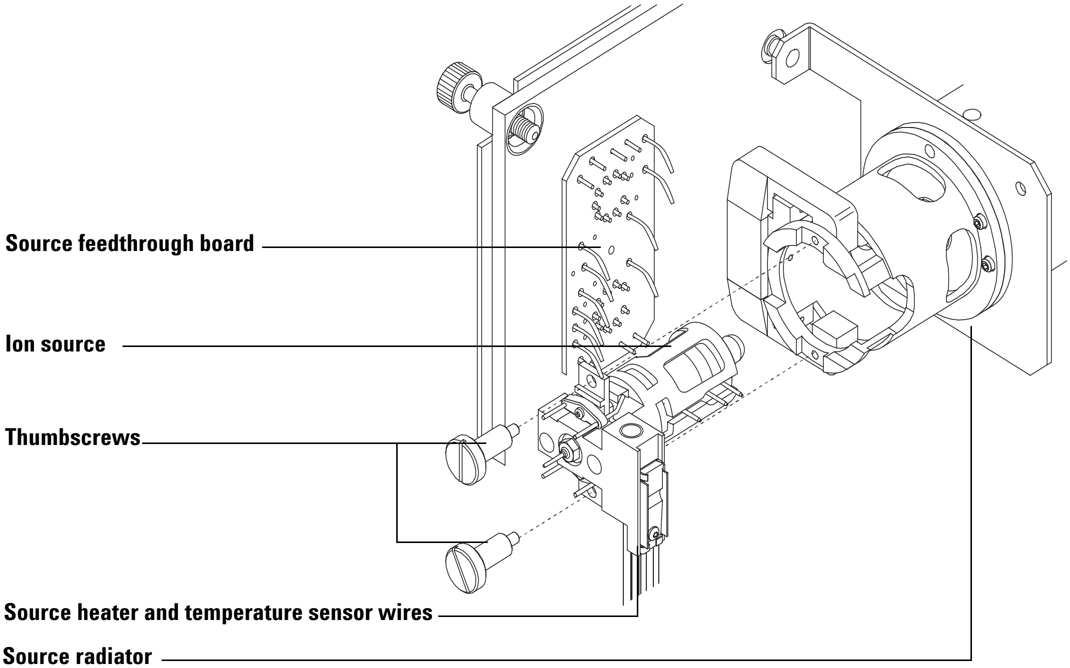


Figure 20 Removing the EI ion source

To Disassemble the Standard 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 Remove the EI Ion Source" on page 105.
2. Remove the filaments. See "To Remove a Filament" on page 134.
3. Separate the source heater assembly from the source body by removing the two screws. The source heater assembly includes the source heater, repeller, and related parts. (See Figure 21.)
4. Disassemble the repeller assembly by removing the repeller nut, washers, ceramic insulators and repeller. (See Figure 21.)
5. Unscrew the interface socket from the source body. A 10 mm open-end wrench fits the flat sides on the interface socket.
6. Remove the setscrew securing the lenses to the source body.
7. Pull the lenses out of the source body and separate the lens insulator, ion focus lens, drawout plate, drawout cylinder, and entrance lens. (See Figure 21.)

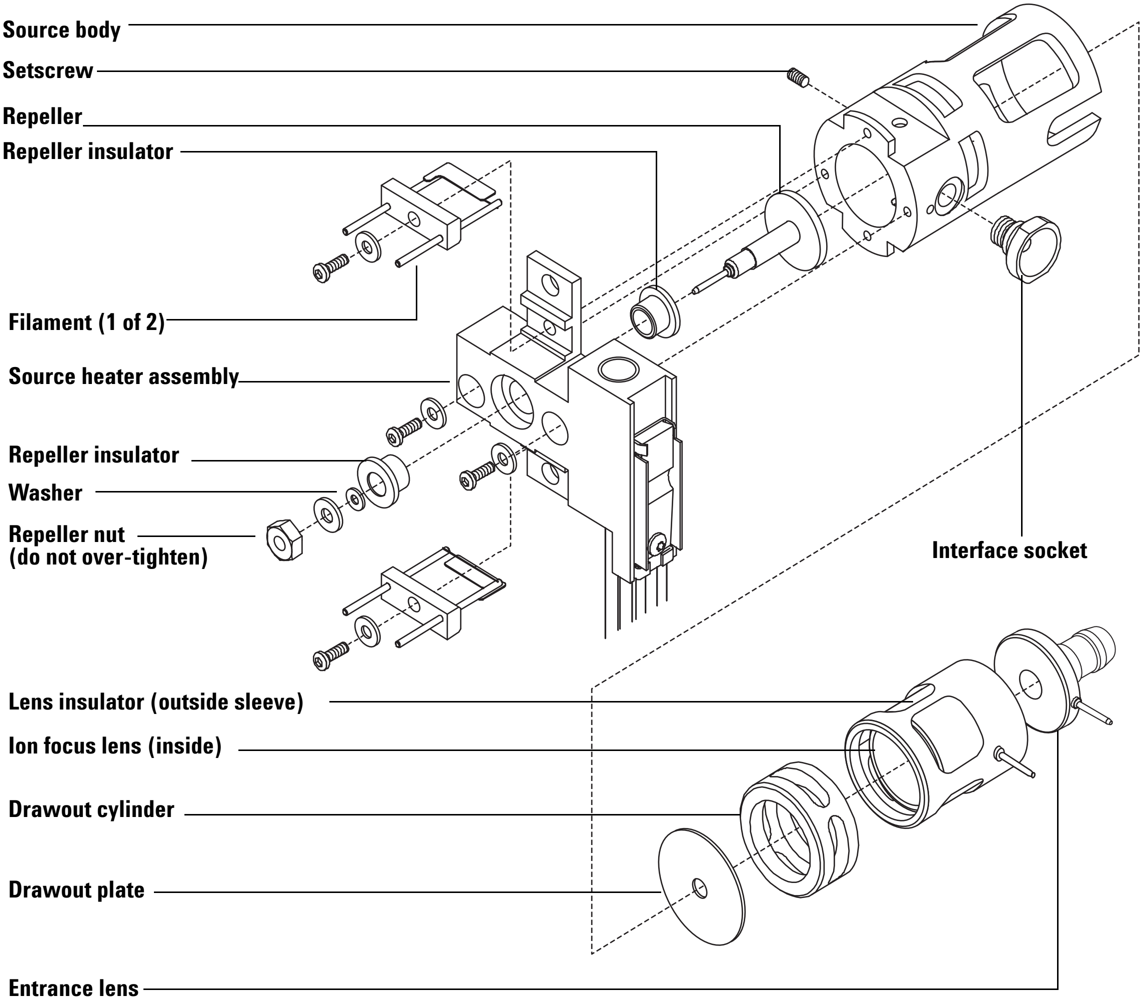


Figure 21 Disassembling the standard EI ion source

To Disassemble the EI Extractor 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 Remove the EI Ion Source” on page 105.
2. Remove the filaments. See “To Remove a Filament” on page 134.
3. Separate the source heater assembly from the source body by removing the two screws. The source heater assembly includes the source heater, repeller, and related parts. (See Figure 22.)
4. Disassemble the repeller assembly by removing the repeller nut, washers, ceramic insulators and repeller. (See Figure 22.)
5. Unscrew the interface socket from the source body. A 10 mm open-end wrench fits the flat sides on the interface socket.
6. Remove the setscrew securing the lenses to the source body.
7. Pull the lenses out of the source body and separate the lens insulator, ion focus lens, extractor lens, extractor lens insulator, and entrance lens. (See Figure 22.)

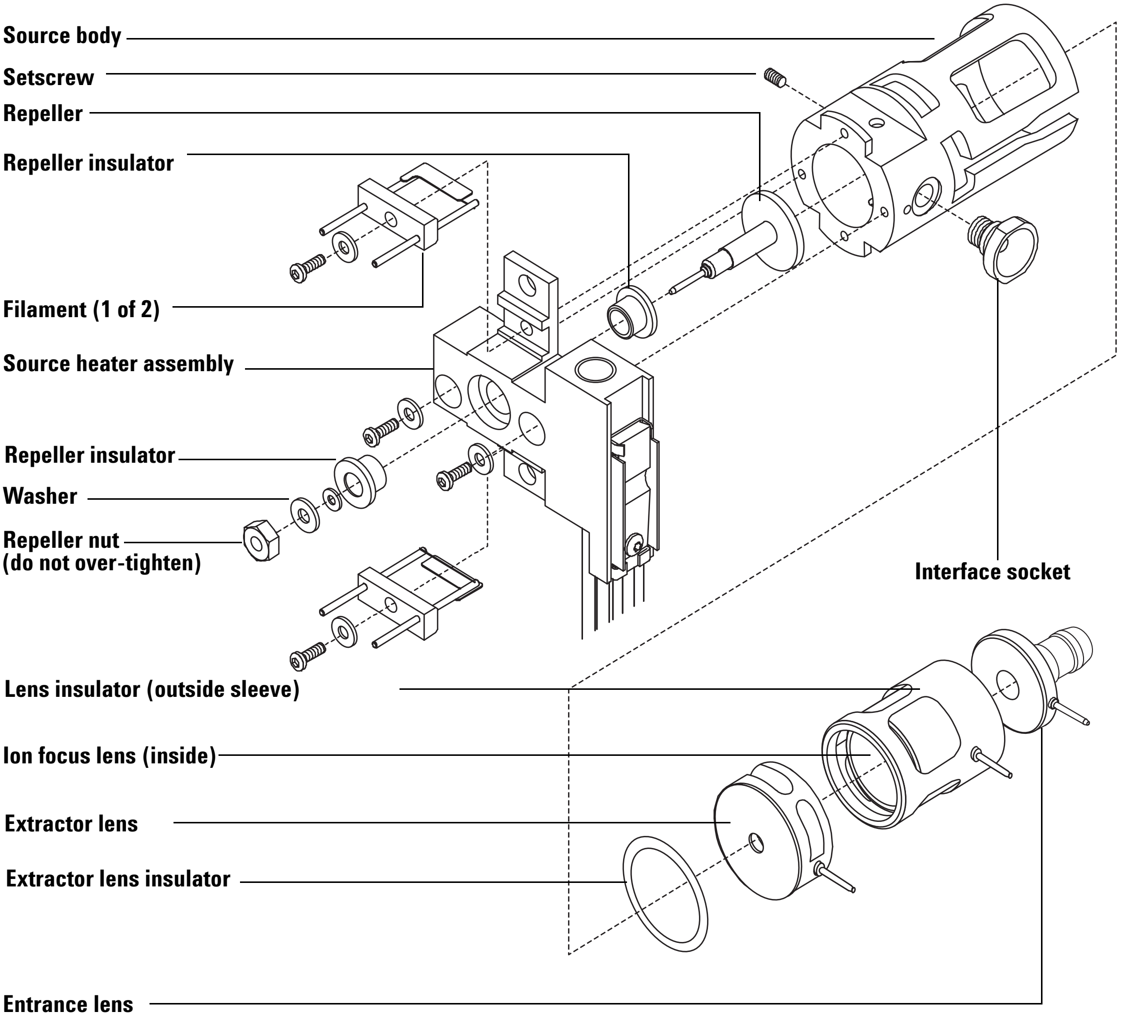


Figure 22 Disassembling the EI extractor ion source

To Clean the EI Ion Source

Materials needed

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

Preparation

1. Disassemble the ion source. See “To Disassemble the EI Extractor Ion Source” on page 110 or “To Disassemble the Standard EI Ion Source” on page 108.
2. Collect the following parts to be cleaned if you are cleaning a high sensitivity extractor EI ion source: (Figure 23)
    - Repeller
    - Source body
    - Extractor lens
    - Ion focus lens
    - Entrance lens

3 Collect the following parts to be cleaned if you are cleaning a standard EI ion source. (Figure 24)

- Repeller
- Source body
- Drawout plate
- Drawout cylinder
- Ion focus lens
- Entrance lens

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

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

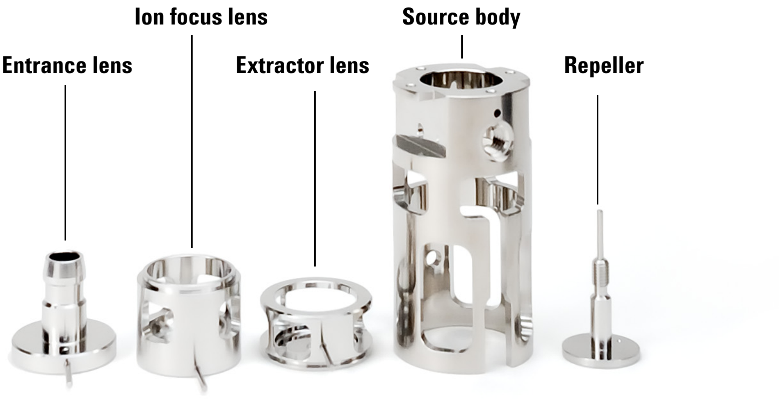


Figure 23 Extractor EI Source parts to be cleaned

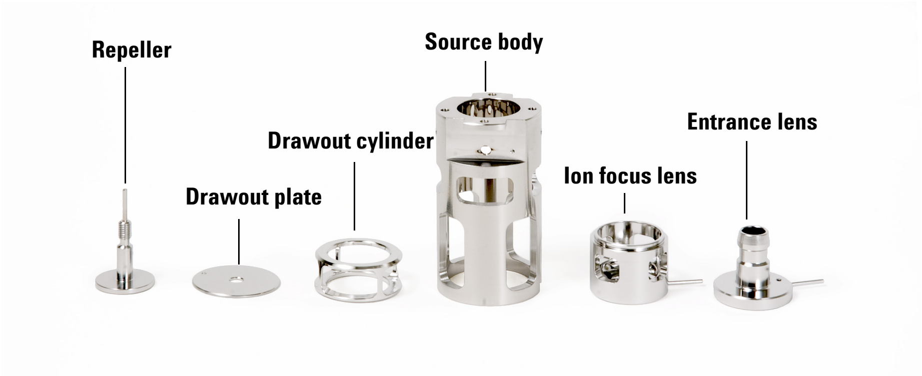


Figure 24 Standard EI ion source parts to be cleaned

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

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

5 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.

6 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.

7 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.

8. 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.**

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

To Assemble the Standard 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 ion focus lens, entrance lens, and lens insulator (Figure 21).
2. Slide the drawout plate and drawout cylinder into the source body (Figure 21).
3. Slide the parts assembled in step 1 into the source body.
4. Install the setscrew that holds the lenses in place.

CAUTION When installing the interface socket, do not overtighten. Overtightening could strip the threads.

5. Reinstall the interface socket.

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.

6. Assemble the repeller assembly by attaching the repeller, repeller insulators, washers, and repeller nut onto the source heater assembly.
7. Attach the repeller assembly to the source body with two screws and washers.
8. Install the filaments. See “To Install a Filament” on page 136.

To Assemble the EI Extractor 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 ion focus lens, entrance lens, and lens insulator (Figure 25).
2. Attach the extractor lens insulator to the extractor lens, and slide them into the source body (Figure 25).
3. Slide the parts assembled in step 1 into the source body.
4. Install the setscrew that holds the lenses in place.

CAUTION When installing the interface socket, do not overtighten. Overtightening could strip the threads.

5. Install the interface socket.

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.

6. Assemble the repeller assembly by attaching the repeller, repeller insulators, washers, and repeller nut onto the source heater assembly.
7. Attach the repeller assembly to the source body with two screws and washers.
8. Install the filaments. See "To Install a Filament" on page 136.

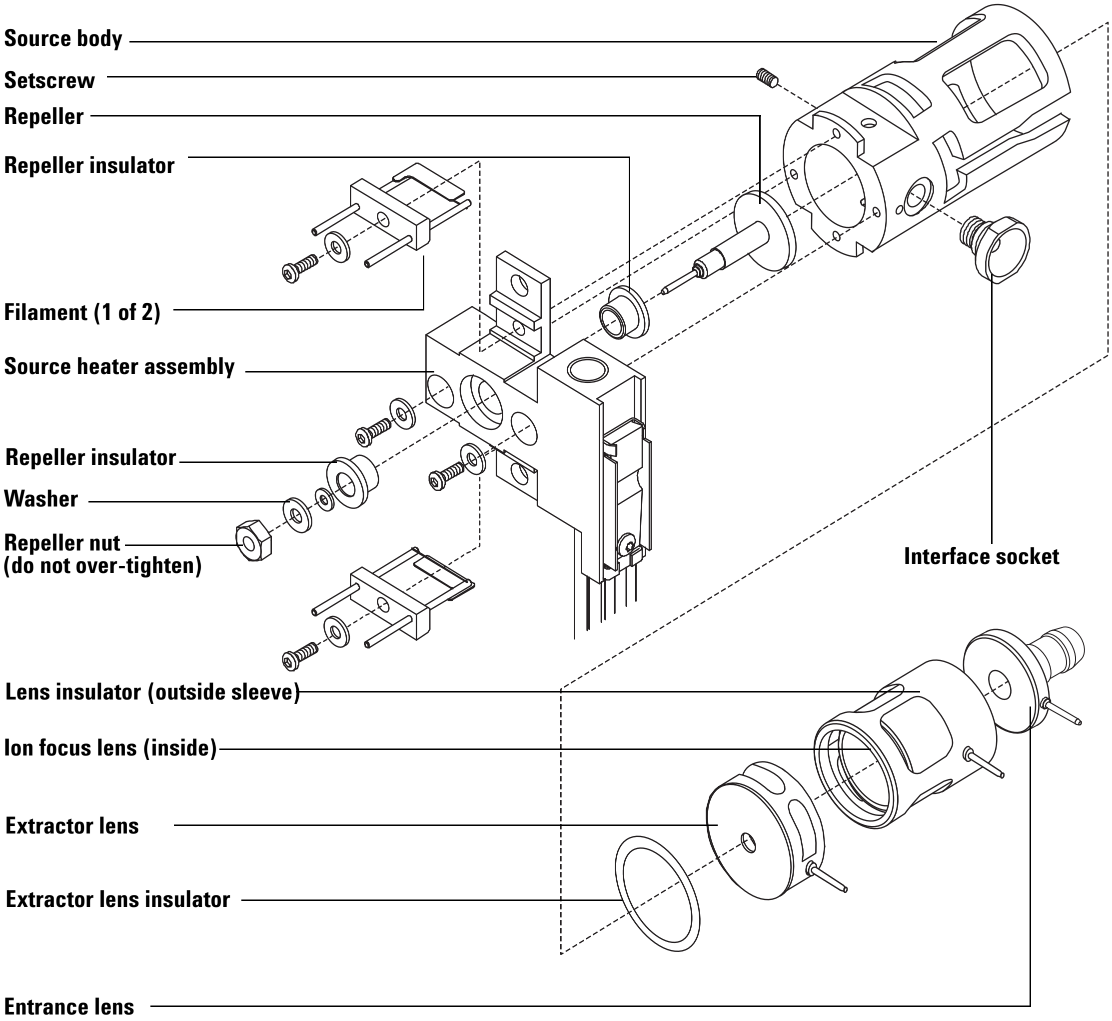


Figure 25 Assembling the EI extractor ion source

To Install the EI Ion Source

Materials needed

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

Procedure

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

---

1. Slide the ion source into the source radiator (Figure 26).
2. Connect the ion source wires as shown in "To Attach Wiring from the Ion Source to the Side Board" on page 137.
3. Install and hand-tighten the source thumbscrews. Do not overtighten the thumbscrews.
4. Close the front analyzer chamber. See "To Close the Front Analyzer Chamber" on page 141.
5. Pump down the MS. (See "To Pump Down the MS" on page 62.)
6. Tune the MS. (See "To Autotune the MS for EI Mode" on page 59.)

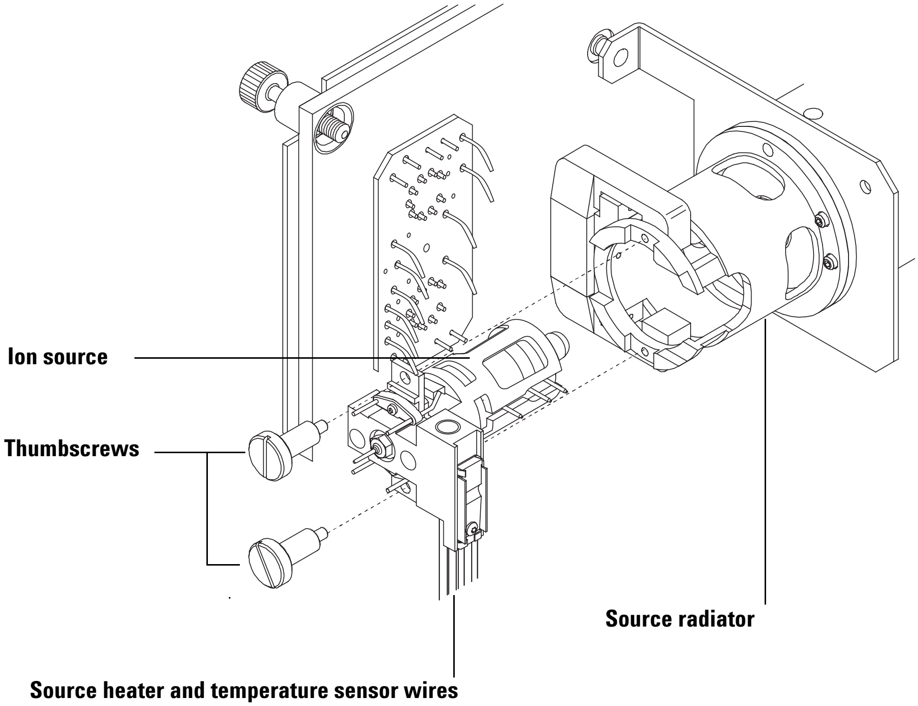


Figure 26 Installing the EI ion source

To Remove the CI Ion Source

Materials needed

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

Procedure

1 Vent the MS. (See “To Vent the MS” on page 65.)

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

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

2 Open the front analyzer chamber. (See “To Open the Front Analyzer Chamber” on page 102.)

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

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

3 Disconnect the wires from the ion source. Do not bend the wires any more than necessary (Table 16, Figure 36).

4 Trace the wires for the ion source heater and temperature sensor to the feedthrough board. Disconnect them there (Figure 34).

Table 16 CI Ion source wires

Wire color	Connects to	Number of leads
Blue	Entrance lens	1
Orange	Ion focus	1
White	Filament 1 (top filament)	2
Red	Repeller	1
Black	Filament 2 (bottom filament)	2
Green beaded	Feedthrough board (bottom left)	2
White	Feedthrough board (bottom middle)	2
Brown	Not used here	n/a

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

6 Pull the ion source out of the source radiator.

FB = Feedthrough Board

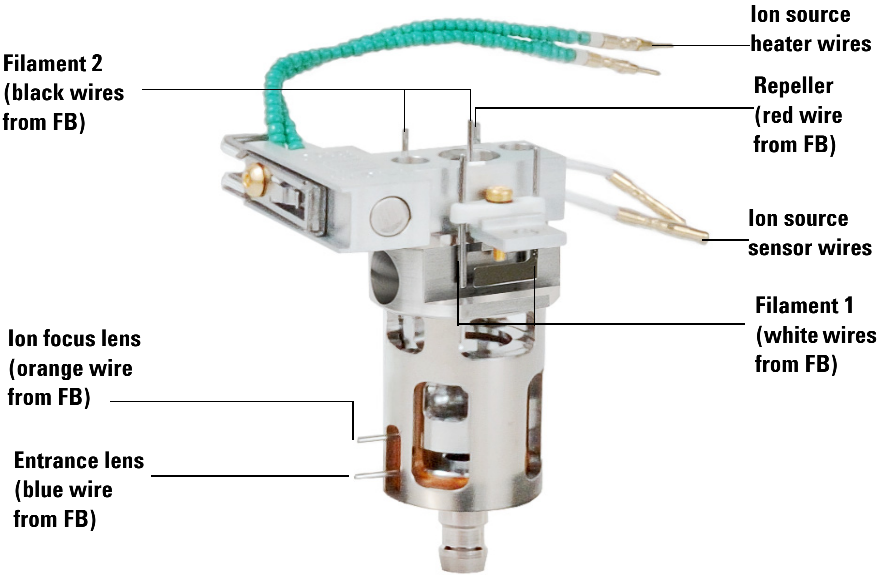


Figure 27 CI Ion Source Wiring

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 Remove the CI Ion Source" on page 121.
2. Remove the filaments. See "To Remove a Filament" on page 134.
3. Separate the source heater assembly from the source body. The source heater assembly includes the source heater, repeller, and related parts. (See Figure 28.)
4. Disassemble the repeller assembly by removing the ceramic insulator from the repeller. (See Figure 28.)
5. Remove the setscrew securing the lenses to the source body.
6. Pull the lenses out of the source body and separate the lens insulator, ion focus lens, drawout cylinder, drawout lens, and entrance lens. (See Figure 28.)

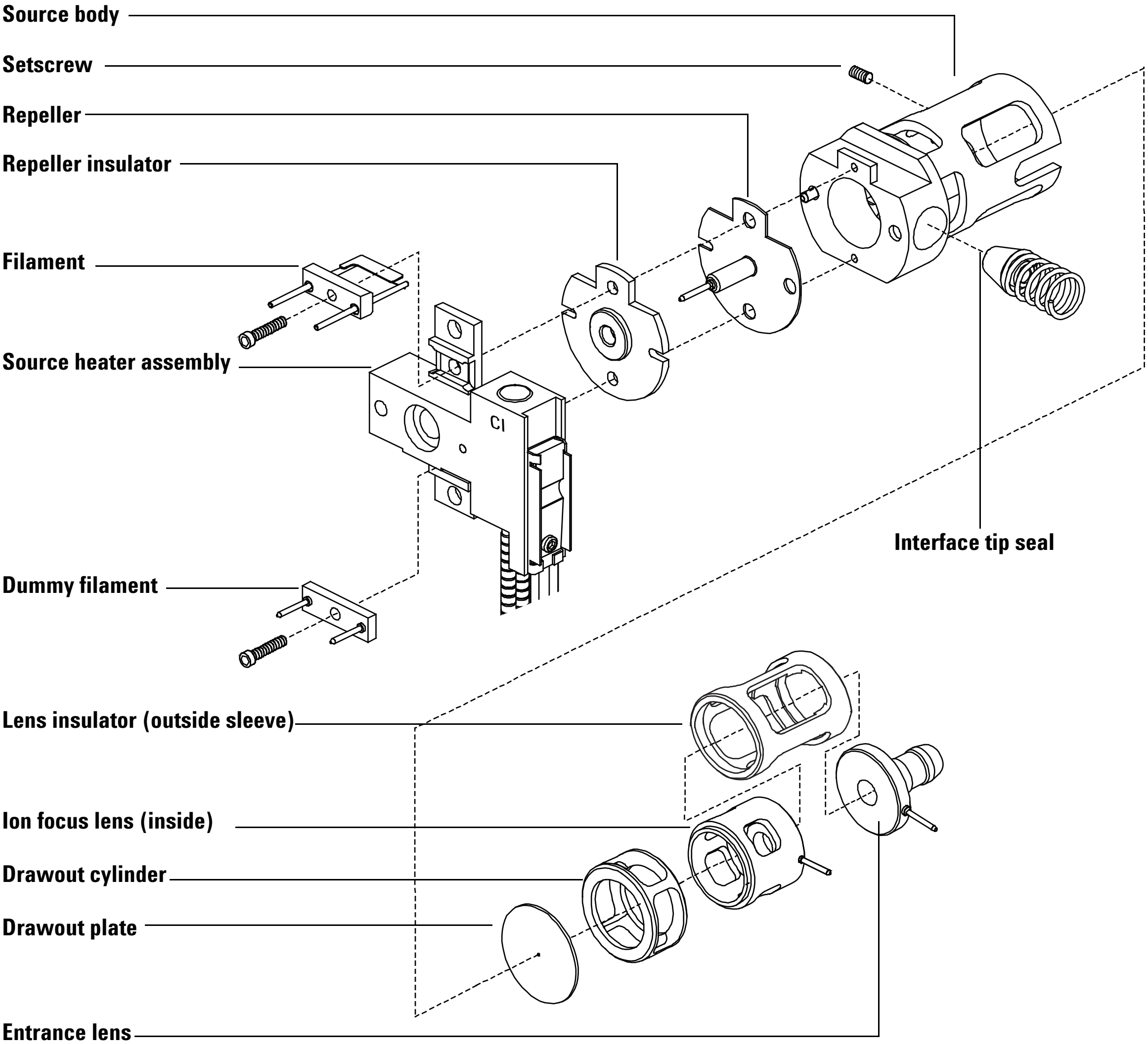


Figure 28 Disassembling the CI ion source

To Clean the CI 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 7000 Series Triple Quad GCMC Troubleshooting and Maintenance Procedure 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 CI ion source. See "To Disassemble the CI Ion Source" on page 124.
2. Collect the following parts to be cleaned: (Figure 29)
   - Repeller
   - Source body
   - Drawout plate
   - Drawout cylinder
   - Ion focus lens
   - Entrance lens

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

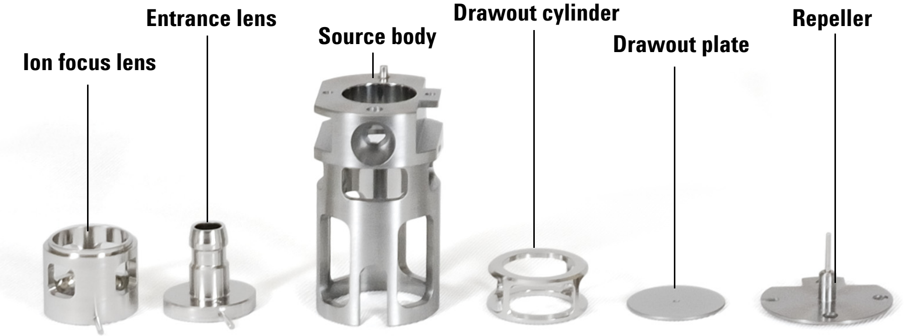


Figure 29 CI Ion Source Parts to be Cleaned

Procedure

Cleaning the CI ion source is very similar to cleaning the EI ion source. Use the cleaning procedure in "To Clean 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 and the ion exit hole in the drawout plate.
- Do not use halogenated solvents. Use hexane for the final rinse.

CAUTION Do not use halogenated solvents to clean 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. Assemble the ion focus lens, entrance lens, and lens insulator (Figure 30).
2. Slide the drawout plate and the drawout cylinder into the source body (Figure 30).
3. Slide the parts assembled in step 1 into the source body.
4. Install the setscrew that holds the lenses in place.
5. Attach the ceramic disk to the repeller and place on top of the source body.

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

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

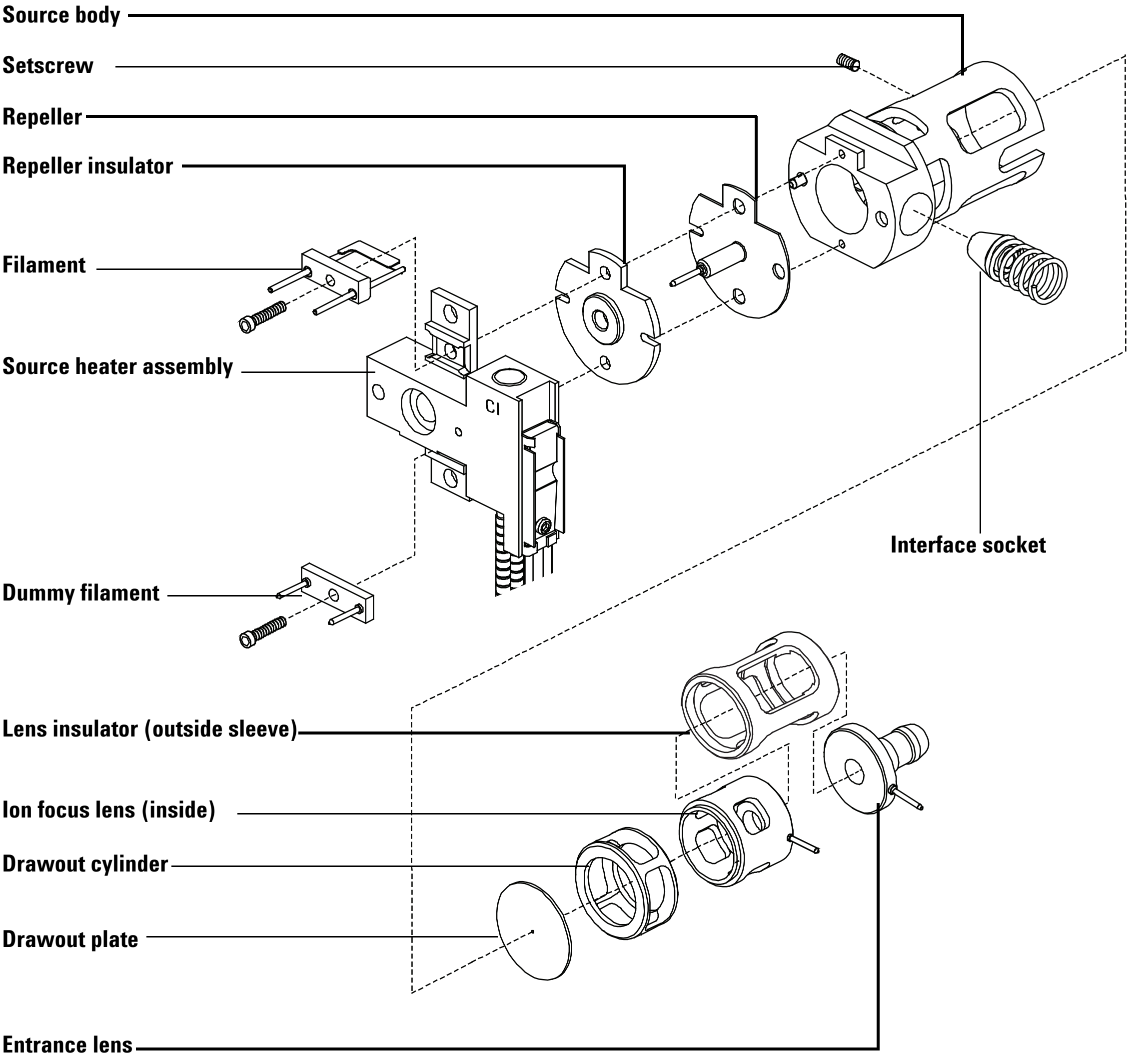


Figure 30 Assembling the CI ion source

To Install the CI Ion Source

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

Procedure

1. Vent the MS. See page 65.
2. Open the front analyzer chamber. See “To Open the Front Analyzer Chamber” on page 102.
3. Slide the CI ion source into the radiator.
4. Install the thumbscrews (Figure 31).
5. Connect the wiring as described in “To Attach Wiring from the Ion Source to the Side Board” .

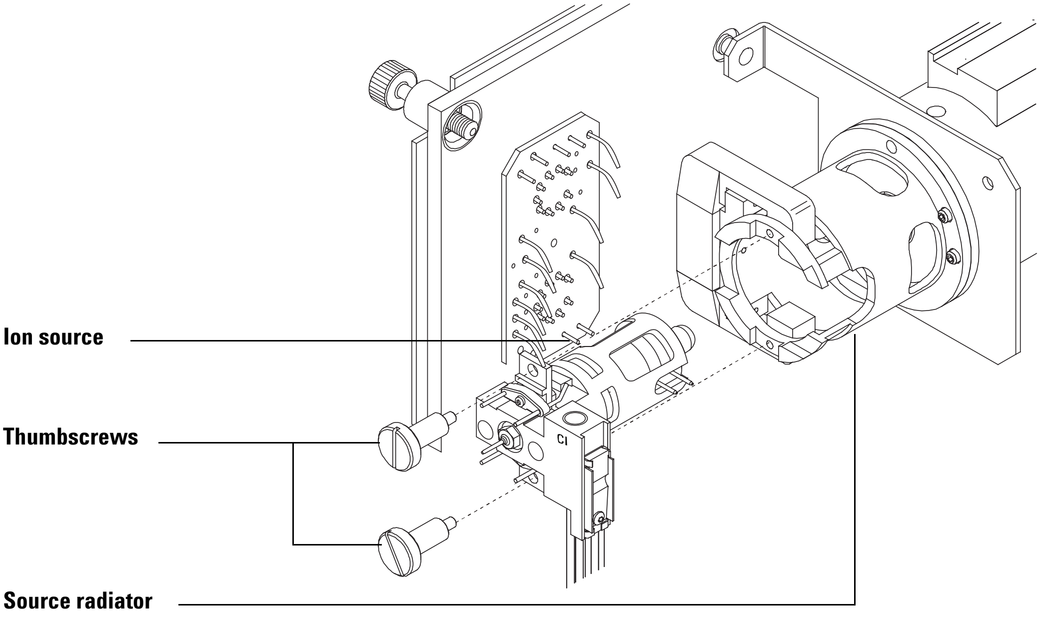


Figure 31 Installing the CI ion source

6 Close the front analyzer chamber. (See “To Close the Front Analyzer Chamber” on page 141.)

7 Pump down the MS. (See “To Pump Down the MS” on page 62.)

8 Tune the MS. (See “To Perform a CI Autotune” on page 90.)

To Install the CI Interface Tip Seal

Materials needed

- Interface tip seal (G1099-60412)

  The interface tip seal must be in place for CI operation. It is necessary to achieve adequate ion source pressure for CI.

CAUTION

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

Procedure

1. Remove the seal from the ion source storage box.
2. Verify that the CI ion source is installed.
3. Place the seal over the end of the interface. To remove the seal, reverse the above steps.
4. *Gently* check the alignment of the analyzer and the interface.

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

CAUTION

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

---

5. You can align the front analyzer chamber and interface by wiggling the side plate on its hinge. If the analyzer still will not close, contact your Agilent Technologies service representative.
6. Close the front analyzer chamber. (See “To Close the Front Analyzer Chamber” on page 141.)

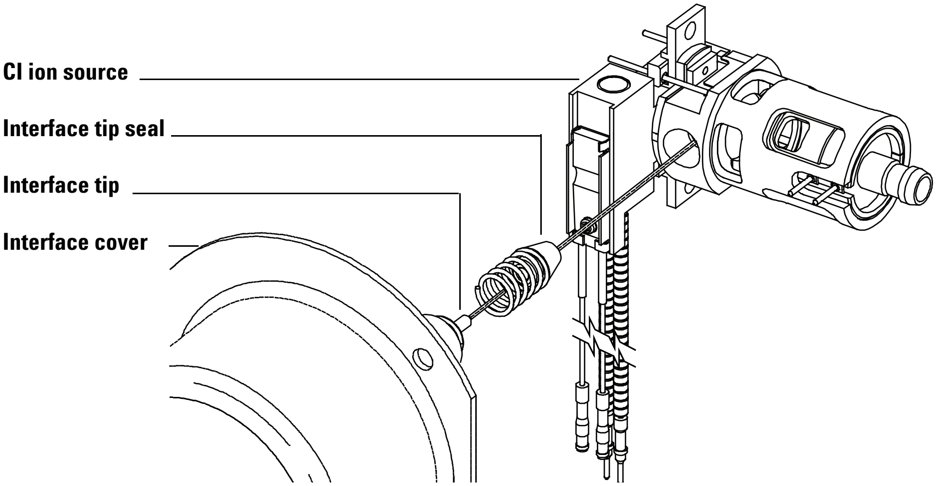


Figure 32 CI Ion Source Tip Seal

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

1 Vent the MS. (See “Venting the MS” on page 48.)

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

2 Open the front analyzer chamber. See “To Open the Front Analyzer Chamber” on page 102.

3 Remove the ion source. See “To Remove the EI Ion Source” on page 105 or see “To Remove the CI Ion Source” on page 121.

4 Remove the screw holding the filament to the ion source body. (See Figure 33.)

5 Slide the filament off the ion source assembly. (See Figure 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.**

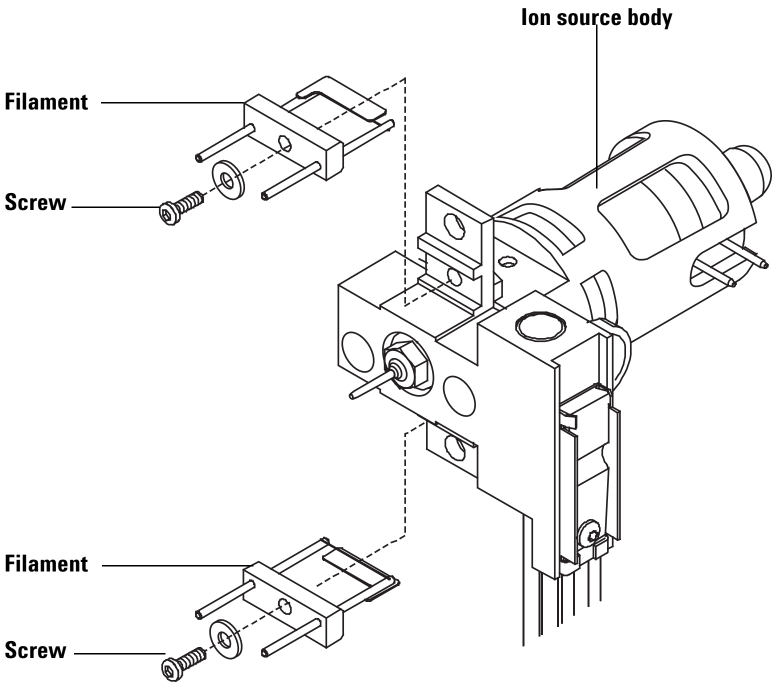


Figure 33 Changing the filament

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 134.)
2. Place the new filament into its position on the ion source body. (See Figure 33.)
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 Install the EI Ion Source" on page 119 or see "To Install the CI Ion Source" on page 130.)
6. Close the front analyzer chamber. (See "To Close the Front Analyzer Chamber" on page 141.)
7. Pump down the MS. (See "To Pump Down the MS" on page 62.)
8. Autotune the MS.

To Attach Wiring from the Ion Source to the Side Board

Materials needed

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

Procedure

1 Attach the internal front analyzer electrical leads to the pins specified in Table 17.

The wiring is described in Table 17 and illustrated in Figure 34, Figure 35, and Figure 36. The term “Board” in the table refers to the feedthrough board located next to the ion source.

Table 17 Analyzer wiring

Wire description	Wired	Attach to connector
Green beaded (2)	Quad (1) heater	Board, top left (HTR)
White with braided cover (2)	Quad (1) sensor	Board, top (RTD)
Brown/black	Board, center left	Extractor lens (high sensitivity EI ion source only)
White (2)	Board, center (FILAMENT-1)	Filament 1 (top)
Red (1)	Board, center left (REP)	Repeller
Black (2)	Board, center (FILAMENT-2)	Filament 2 (bottom)
Orange (1)	Board, top right (ION FOC)	Ion focus lens
Blue (1)	Board, top right (ENT LENS)	Entrance lens
Green beaded (2)	Ion source heater	Board, bottom left (HTR)
White (2)	Ion source sensor	Board, bottom (RTD)
Green	Board, bottom left	Ion source radiator
Yellow	Board, bottom left	Front quad

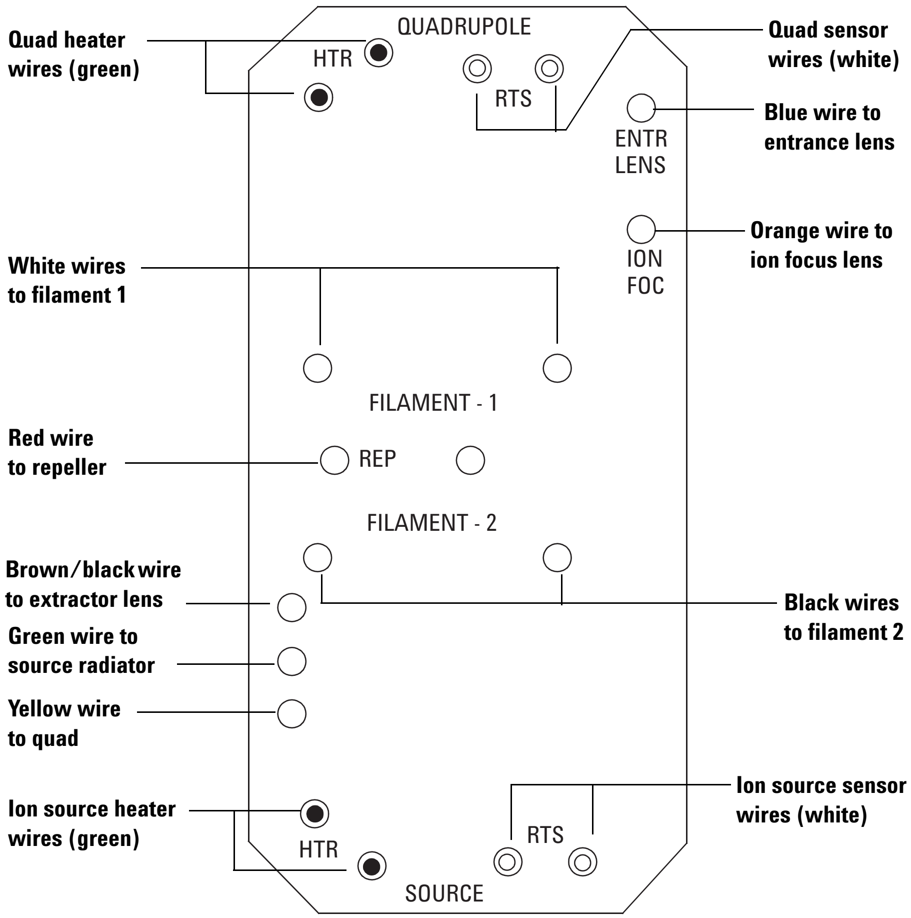


Figure 34 Feedthrough board wiring

FB = Feedthrough Board

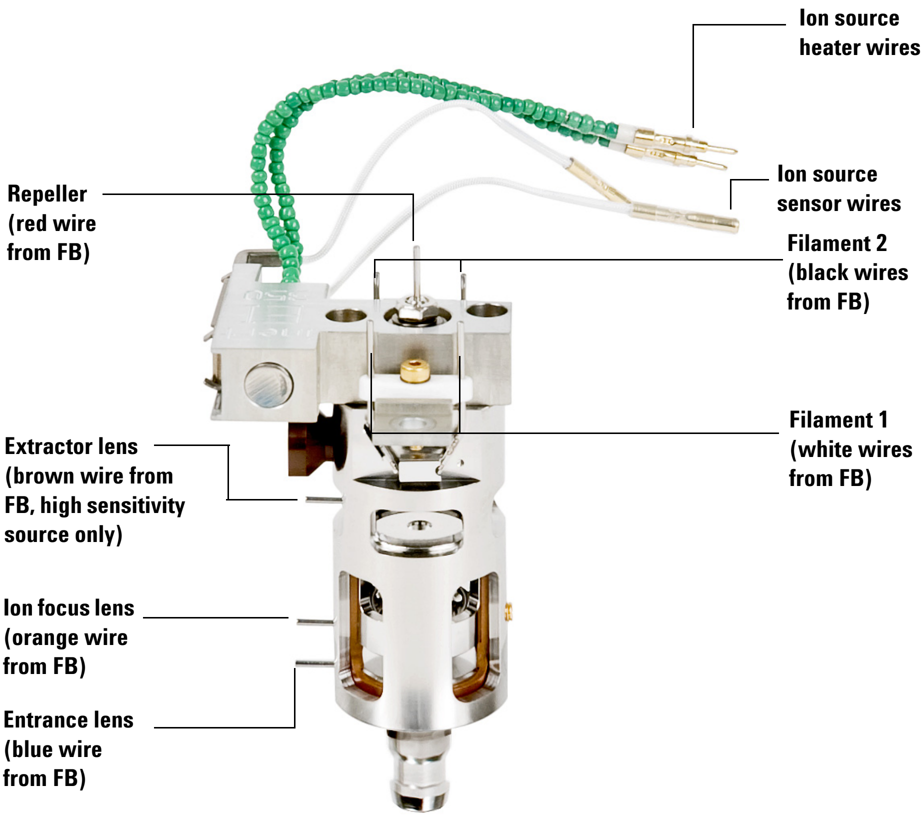


Figure 35 EI Source wiring - High sensitivity source shown

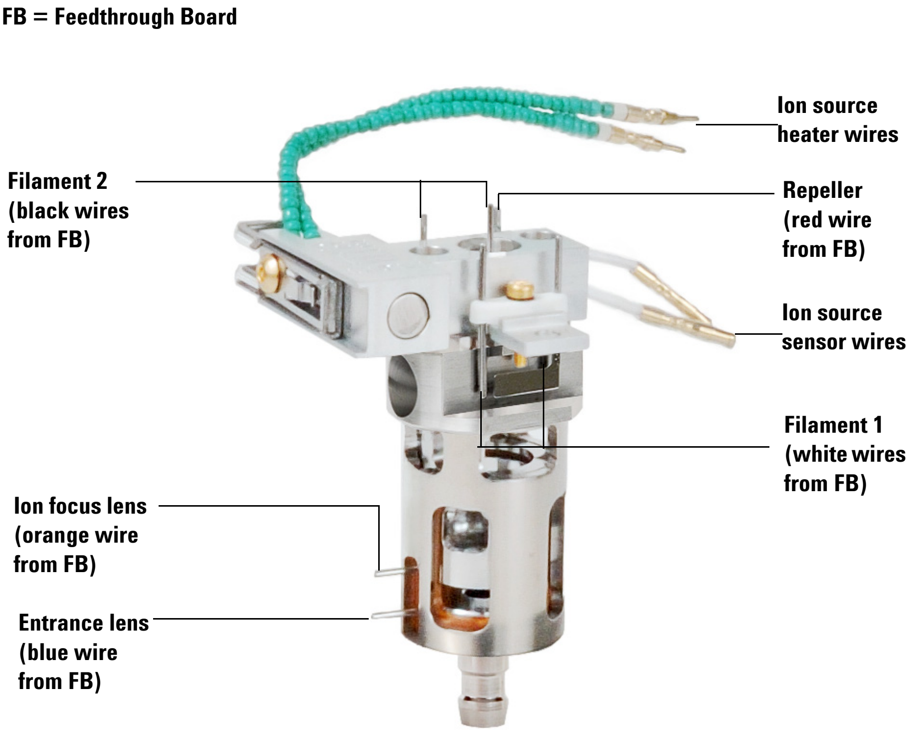


Figure 36 CI Source wiring

To Close the Front Analyzer Chamber

Procedure

1 Check the side plate O-ring.

Make sure the O-ring has a *very* light coat of Apiezon L high vacuum grease. If the O-ring is very dry, it may not seal well. If the O-ring looks shiny, it has too much grease on it. (Refer to the *7000 Series MS Troubleshooting and Maintenance Manual* for lubricating instructions.)

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

---

2 Swing the front analyzer side plate closed.

The post-filter on the exit side of the quad helps to position the collision cell when the analyzer door is closed. When closing, the door should give minimal resistance while the quad is reseating the collision cell. The analyzer should slide into place with minimal pressure.

3 Make sure the rear analyzer door is closed.

4 Make sure the vent valve is closed.

5 If hydrogen or other flammable or toxic substance is used for carrier gas, *gently* hand-tighten the top thumbscrew on the front analyzer side plate.

6 Pump down the MS. (See “To Pump Down the MS” on page 62.)

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

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

---

7 Once the MS has pumped down, close the left analyzer cover and replace the window cover.

8 Tune the MS.

To Remove Left Rear Cover for Access to the Rear Analyzer Chamber

The rear cover should be removed to open the rear analyzer sideplate. This is necessary when replacing the electron multiplier horn. If you need to access the rear analyzer chamber, follow these procedures to remove the rear cover (Figure 37).

Materials needed

- Screwdriver, Torx T-15 (8710-1622)

Procedure

1. Open the left side panel according to the procedure. (See “To Remove Left Rear Cover for Access to the Rear Analyzer Chamber” on page 142.)
2. Remove the top screw from the rear cover.
3. Lift the bottom flap of the cover out of the groove in the back of the MS to free the cover.

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

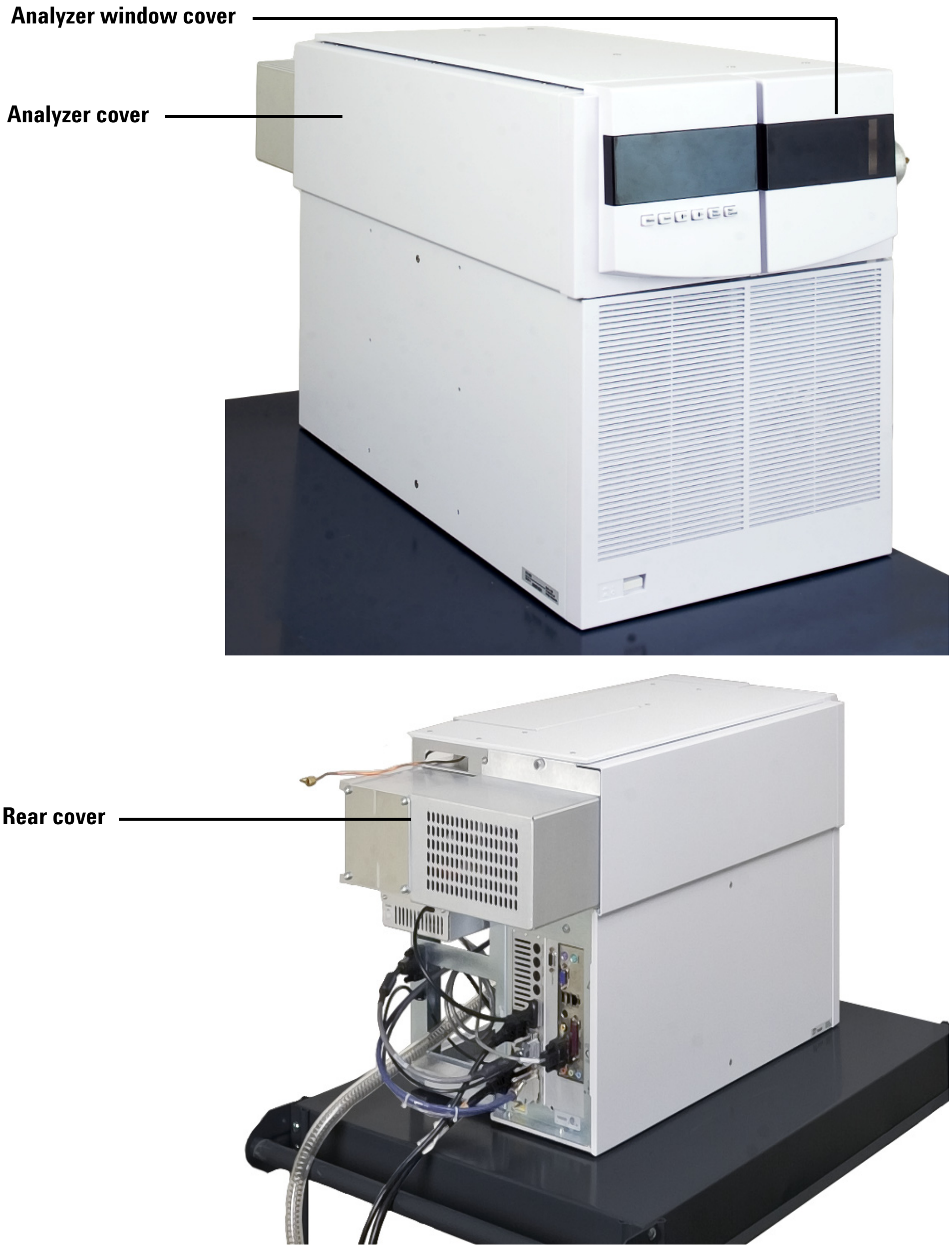


Figure 37 Removing covers

To Open the Rear Analyzer Chamber

The rear analyzer chamber should only be opened to change the electron multiplier horn.

Materials needed

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

CAUTION Electrostatic discharges to analyzer components are conducted to the quad driver board where they can damage sensitive components. Wear a grounded antistatic wrist strap and take other antistatic precautions (see "Electrostatic discharge" on page 98) before you open the analyzer chamber.

Procedure

1. Vent the MS. (See "To Vent the MS" on page 65.)
2. Remove the analyzer window cover and open left side panel. Remove rear cover. (See "To Remove Left Rear Cover for Access to the Rear Analyzer Chamber" on page 142.)

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

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

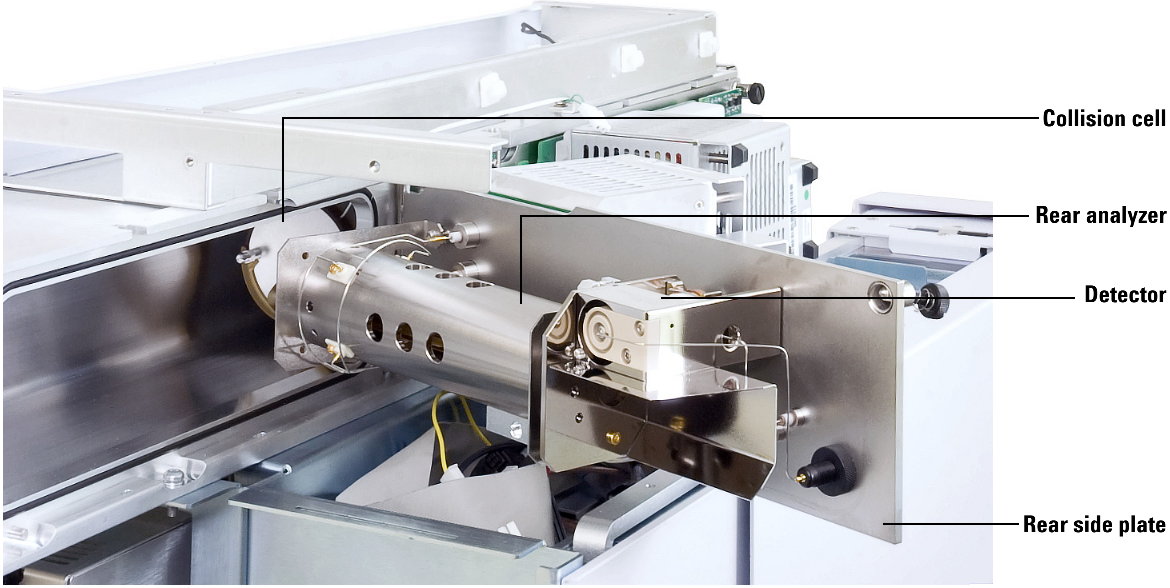
3 Loosen the rear analyzer side plate thumbscrews (Figure 19) if they are fastened.

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

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

4 *Gently* swing the side plate out.

Chamber closed (cables removed for clarity)



Chamber open

Figure 38 The rear analyzer chamber

To Replace the Electron Multiplier Horn

Materials needed

- Electron multiplier horn (G3170-80103)
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Pliers, long-nose (8710-1094)

Procedure

1 Vent the MS. (See“To Vent the MS” on page 65.)

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

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

2 Open the rear analyzer chamber. (See “To Open the Rear Analyzer Chamber” on page 144.)

3 Open the retaining clip (Figure 39). Lift the arm of the clip up and swing the clip away from the electron multiplier horn.

4 Slide the red signal wire from the connector in the sideplate.

5 Remove the electron multiplier horn.

6 Hold the new horn with red signal wire end down, and reconnect the red signal wire to the connector in the sideplate.

7 Slide the electron multiplier horn into position.

8 Close the retaining clip.

9 Close the rear analyzer chamber. (See “To Close the Rear Analyzer Chamber” on page 149.)

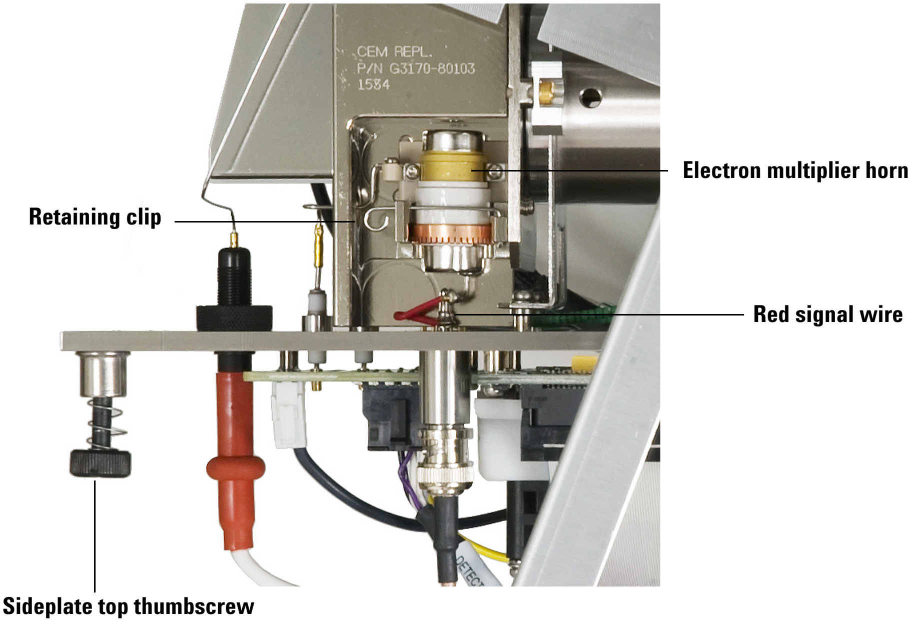


Figure 39 Replacing the electron multiplier horn

To Close the Rear Analyzer Chamber

Materials needed

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

Procedure

1 Check the side plate O-ring.

Make sure the O-ring has a very light coat of Apiezon L high vacuum grease. If the O-ring is very dry, it may not seal well. If the O-ring looks shiny, it has too much grease on it. (Refer to the *7000 Series MS Troubleshooting and Maintenance Manual* for lubrication instructions.)

2 Close the rear analyzer side plate. The pre-filter on the entrance side of the quad helps to position the collision cell when the analyzer door is closed. When closing, the door should give minimal resistance while the quad is reseating the collision cell. The analyzer should slide into place with minimal pressure.

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

3 Make sure the front analyzer door is closed.

4 Make sure the vent valve is closed.

5 Pump down the MS. (See “To Pump Down the MS” on page 62.)

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

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

6. If hydrogen or any other flammable material is used for the carrier gas, gently hand-tighten the top thumbscrew on the rear analyzer side plate.
7. Once the MS has pumped down, close the left analyzer cover, replace the rear cover, and replace the analyzer window cover.
8. Tune the MS.

A
Chemical Ionization Theory

Chemical Ionization Overview 152
Positive CI Theory 154
Negative CI Theory 161

Chemical Ionization Overview

Chemical ionization (CI) is a technique for creating ions used in mass spectrometric analyses. There are significant differences between CI and electron ionization (EI). This section describes the most common chemical ionization mechanisms.

In EI, relatively high-energy electrons (70 eV) collide with molecules of the sample to be analyzed. These collisions produce (primarily) positive ions. Upon ionization, the molecules of a given substance fragment in fairly predictable patterns. EI is a direct process; energy is transferred by collision from electrons to the sample molecules.

For CI, in addition to the sample and carrier gas, large amounts of reagent gas are introduced into the ionization chamber. Since there is so much more reagent gas than sample, most of the emitted electrons collide with reagent gas molecules, forming reagent ions. These reagent-gas ions react with each other in primary and secondary reaction processes that establish an equilibrium. They also react in various ways with sample molecules to form sample ions. CI ion formation involves much lower energy and is much more "gentle" than electron ionization. Since CI results in much less fragmentation, CI spectra usually show high abundance of the molecular ion. For this reason, CI is often used to determine the molecular weights of sample compounds.

Methane is the most common CI reagent gas. It yields certain characteristic ionization patterns. Other reagent gases yield different patterns and may result in better sensitivity for some samples. Common alternative reagent gases are isobutane and ammonia. Carbon dioxide is often used in negative CI. Less common reagent gases are carbon dioxide, hydrogen, Freon, trimethylsilane, nitric oxide, and methylamine. Different ionization reactions occur with each reagent gas.

WARNING **Ammonia is toxic and corrosive. Use of ammonia requires special maintenance and safety precautions.**

Water contamination in reagent gases will decrease CI sensitivity dramatically. A large peak at *m/z* 19 (H_30^+) in positive CI is a diagnostic symptom of water contamination. In high enough concentrations, especially when combined with calibrant, water contamination will result in a heavily contaminated ion source. Water contamination is most common immediately after new reagent gas tubing or reagent gas cylinders are connected. This contamination will often decrease if the reagent gas is allowed to flow for a few hours, purging the system.

References on chemical ionization

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W. B. Knighton, L. J. Sears, E. P. Grimsrud, “High Pressure Electron Capture Mass Spectrometry”, *Mass Spectrometry Reviews* (1996), **14**, 327-343.

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Positive CI Theory

Positive CI (PCI) occurs with the same analyzer voltage polarities as EI. For PCI, the reagent gas is ionized by collision with emitted electrons. The reagent gas ions react chemically with sample molecules (as proton donors) to form sample ions. PCI ion formation is more “gentle” than electron ionization, producing less fragmentation. This reaction usually yields high abundance of the molecular ion and is therefore often used for determining molecular weights of samples.

The most common reagent gas is methane. Methane PCI produces ions with almost any sample molecule. Other reagent gases, such as isobutane or ammonia, are more selective and cause even less fragmentation. Because of the high background from the reagent gas ions, PCI is not especially sensitive and detection limits are generally high.

There are four fundamental ionization processes that take place during positive chemical ionization at ion source pressures in the 0.8 to 2.0 Torr range. These are:

- Proton transfer
- Hydride abstraction
- Addition
- Charge exchange

Depending on the reagent gas used, one or more of these four processes can be used to explain the ionization products observed in the resulting mass spectra.

EI, methane PCI, and ammonia PCI spectra of methyl stearate are shown in Figure 40. The simple fragmentation pattern, large abundance of the $[MH]^+$ ion, and the presence of the two adduct ions are characteristic of positive chemical ionization using methane as a reagent gas.

The presence of air or water in the system, especially in the presence of PFDTD calibrant, quickly contaminates the ion source.

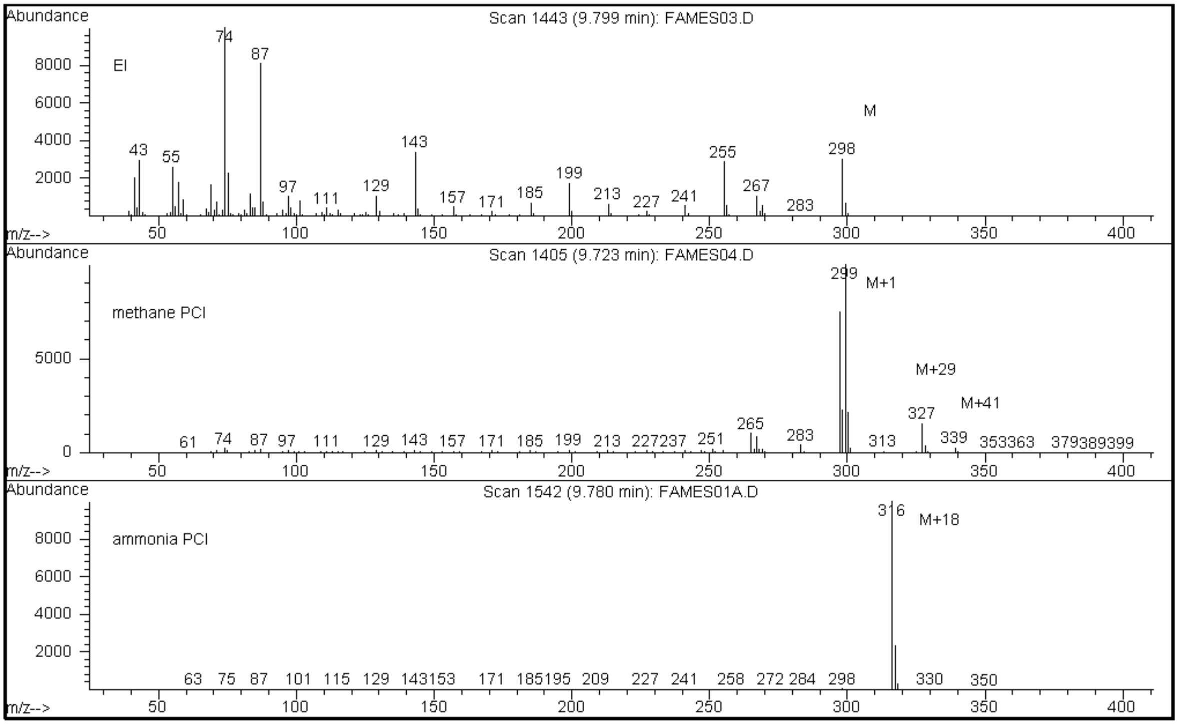

Figure 40 Methyl stearate (MW = 298): EI, methane PCI, and ammonia PCI

Proton transfer

Proton transfer can be expressed as

$$BH^+ + M \rightarrow MH^+ + B$$

where the reagent gas B has undergone ionization resulting in protonation. If the proton affinity of the analyte (sample) M is greater than that of the reagent gas, then the protonated reagent gas will transfer its proton to the analyte forming a positively charged analyte ion.

The most frequently used example is the proton transfer from CH_5^+ to the molecular analyte, which results in the protonated molecular ion MH^+.

The relative proton affinities of the reagent gas and the analyte govern the proton transfer reaction. If the analyte has a greater proton affinity than the reagent gas, then proton transfer can take place. Methane (CH_4) is the most common reagent gas because its proton affinity is very low.

Proton affinities can be defined according to the reaction:

$$B + H^+ \rightarrow BH^+$$

where the proton affinities are expressed in kcal/mole. Methane's proton affinity is 127 kcal/mole. Tables 18 and 19 list the proton affinities of several possible reagent gases and of several small organic compounds with various functional groups.

The mass spectrum generated by a proton-transfer reaction depends on several criteria. If the difference in proton affinities is large (as with methane), substantial excess energy may be present in the protonated molecular ion. This can result in subsequent fragmentation. For this reason, isobutane with a proton affinity of 195 kcal/mole may be preferred to methane for some analyses. Ammonia has a proton affinity of 207 kcal/mole, making it less likely to protonate most analytes. Proton-transfer chemical ionization is usually considered to be “soft” ionization, but the degree of the softness depends on the proton affinities of both the analyte and the reagent gas, as well as on other factors including ion source temperature.

Table 18 Reagent gas proton affinities

Species	Proton affinity kcal/mole	Reactant ion formed
H_2	100	H_3^+ (*m/z* 3)
CH_4	127	CH_5^+ (*m/z* 17)
C_2H_4	160	$C_2H_5^+$ (*m/z* 29)
H_2O	165	H_3O^+ (*m/z* 19)
H_2S	170	H_3S^+ (*m/z* 35)
CH_3OH	182	$CH_3OH_2^+$ (*m/z* 33)
t-C_4H_{10}	195	t-$C_4H_9^+$ (*m/z* 57)
NH_3	207	NH_4^+ (*m/z* 18)

Table 19 Proton affinities of selected organic compounds for PCI

Molecule	Proton affinity (kcal/mole)	Molecule	Proton affinity (kcal/mole)
Acetaldehyde	185	Methyl amine	211
Acetic acid	188	Methyl chloride	165
Acetone	202	Methyl cyanide	186
Benzene	178	Methyl sulfide	185
2-Butanol	197	Methyl cyclopropane	180
Cyclopropane	179	Nitroethane	185
Dimethyl ether	190	Nitromethane	180
Ethane	121	n-Propyl acetate	207
Ethyl formate	198	Propylene	179
Formic acid	175	Toluene	187
Hydrobromic acid	140	*trans*-2-Butene	180
Hydrochloric acid	141	Trifluoroacetic acid	167

Table 19 Proton affinities of selected organic compounds for PCI (continued)

Molecule	Proton affinity (kcal/mole)	Molecule	Proton affinity (kcal/mole)
Isopropyl alcohol	190	Xylene	187
Methanol	182		

Hydride abstraction

In the formation of reagent ions, various reactant ions can be formed that have high hydride-ion (H^-) affinities. If the hydride-ion affinity of a reactant ion is higher than the hydride-ion affinity of the ion formed by the analyte's loss of H^-, then the thermodynamics are favorable for this chemical ionization process. Examples include the hydride abstraction of alkanes in methane chemical ionization. In methane CI, both CH_5^+ and $C_2H_5^+$ are capable of hydride abstraction. These species have large hydride-ion affinities, which results in the loss of H^- for long-chain alkanes, according to the general reaction

$$R^+ + M \rightarrow [M{-}H]^+ + RH$$

For methane, R^+ is CH_5^+ and $C_2H_5^+$, and M is a long-chain alkane. In the case of CH_5^+, the reaction proceeds to form $[M{-}H]^+ + CH_4 + H_2$. The spectra resulting from hydride abstraction will show an M−1 *m/z* peak resulting from the loss of H^-. This reaction is exothermic so fragmentation of the $[M{-}H]^+$ ion is often observed.

Often, both hydride-abstraction and proton-transfer ionization can be evident in the sample spectrum. One example is the methane CI spectrum of long-chain methyl esters, where both hydride abstraction from the hydrocarbon chain and proton transfer to the ester function occur. In the methane PCI spectrum of methyl stearate, for example, the MH^+ peak at *m/z* 299 is created by proton transfer and the $[M{-}1]^+$ peak at *m/z* 297 is created by hydride abstraction.

Addition

For many analytes, proton-transfer and hydride-abstraction chemical ionization reactions are not thermodynamically favorable. In these cases, reagent gas ions are often reactive enough to combine with the analyte molecules by condensation or association (addition reactions). The resulting ions are called adduct ions. Adduct ions are observed in methane chemical ionization by the presence of $[M{+}C_2H_5]^+$ and $[M{+}C_3H_5]^+$ ions, which result in M+29 and M+41 *m/z* mass peaks.

Addition reactions are particularly important in ammonia CI. Because the NH_3 has a high proton affinity, few organic compounds will undergo proton transfer with ammonia reagent gas. In ammonia CI, a series of ion-molecule reactions takes place, resulting in the formation of NH_4^+, $[NH_4NH_3]^+$, and $[NH_4(NH_3)_2]^+$. In particular, the ammonium ion, NH_4^+, will give rise to an intense $[M+NH_4]^+$ ion observed at M+18 *m/z*, either through condensation or association. If this resulting ion is unstable, subsequent fragmentation may be observed. The neutral loss of H_2O or NH_3, observed as a subsequent loss of 18 or 17 *m/z*, respectively, is also common.

Charge exchange

Charge-exchange ionization can be described by the reaction:

$$X^{+\bullet} + M \rightarrow M^{+\bullet} + X$$

where X^+ is the ionized reagent gas and M is the analyte of interest. Examples of reagent gases used for charge exchange ionization include the noble gases (helium, neon, argon, krypton, xenon, and radon), nitrogen, carbon dioxide, carbon monoxide, hydrogen, and other gases that do not react “chemically” with the analyte. Each of these reagent gases, once ionized, has a recombination energy expressed as:

$$X^{+\bullet} + e^- \rightarrow X$$

or simply the recombination of the ionized reagent with an electron to form a neutral species. If this energy is greater than the energy required to remove an electron from the analyte, then the first reaction above is exothermic and thermodynamically allowed.

Charge-exchange chemical ionization is not widely used for general analytical applications. It can, however, be used in some cases when other chemical ionization processes are not thermodynamically favored.

Negative CI Theory

Negative chemical ionization (NCI) is performed with analyzer voltage polarities reversed to select negative ions. There are several chemical mechanisms for NCI. Not all mechanisms provide the dramatic increases in sensitivity often associated with NCI. The four most common mechanisms (reactions) are:

- Electron capture
- Dissociative electron capture
- Ion pair formation
- Ion-molecule reactions

In all of the cases except the ion-molecule reactions, the reagent gas serves a function different from the function it serves in PCI. In NCI, the reagent gas is often referred to as the buffer gas. When the reagent gas is bombarded with high energy electrons from the filament, the following reaction occurs:

$$\text{Reagent gas} + e^{-}_{\ (230\text{eV})} \rightarrow \text{Reagent ions} + e^{-}_{\ (\text{thermal})}$$

If the reagent gas is methane (Figure 41), the reaction is:

$$CH_4 + e^{-}_{\ (230\text{eV})} \rightarrow CH_4^{+} + 2e^{-}_{(\text{thermal})}$$

The thermal electrons have lower energy levels than the electrons from the filament. It is these thermal electrons that react with the sample molecules.

There are no negative reagent gas ions formed. This prevents the kind of background that is seen in PCI mode and is the reason for the much lower detection limits of NCI. The products of NCI can only be detected when the MS is operating in negative ion mode. This operating mode reverses the polarity of all the analyzer voltages.

Carbon dioxide is often used as a buffer gas in NCI. It has obvious cost, availability, and safety advantages over other gases.

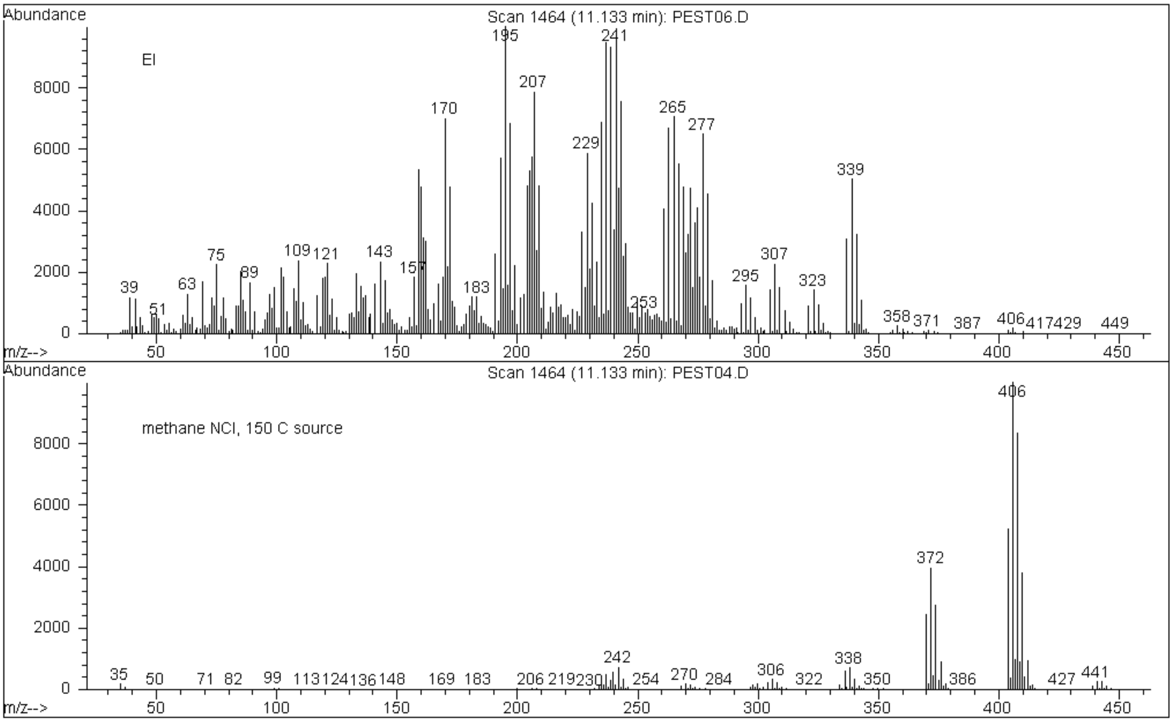

Figure 41 Endosulfan I (MW = 404): EI and methane NCI

Electron capture

Electron capture is the primary mechanism of interest in NCI. Electron capture (often referred to as high-pressure electron capture mass spectrometry or HPECMS) provides the high sensitivity for which NCI is known. For some samples under ideal conditions, electron capture can provide sensitivity as much as 10 to 1000 times higher than positive ionization.

Note that all the reactions associated with positive CI will also occur in NCI mode, usually with contaminants. The positive ions formed do not leave the ion source because of the reversed lens voltages, and their presence can quench the electron capture reaction.

The electron capture reaction is described by:

$$MX + e^{-}_{(thermal)} \rightarrow MX^{-\bullet}$$

where MX is the sample molecule and the electron is a thermal (slow) electron generated by the interaction between high energy electrons and the reagent gas.

In some cases, the $MX^{-\bullet}$ radical anion is not stable. In those cases the reverse reaction can occur:

$$MX^{-\bullet} \rightarrow MX + e^{-}$$

The reverse reaction is sometimes called autodetachment. This reverse reaction generally occurs very quickly. Thus, there is little time for the unstable anion to be stabilized through collisions or other reactions.

Electron capture is most favorable for molecules that have hetero-atoms. For example: nitrogen, oxygen, phosphorus, sulfur, silicon, and especially the halogens: fluorine, chlorine, bromine, and iodine.

The presence of oxygen, water, or almost any other contaminant interferes with the electron-attachment reaction. Contaminants cause the negative ion to be formed by the slower ion-molecule reaction. This generally results in less sensitivity. All potential contamination sources, especially oxygen (air) and water sources, must be minimized.

Dissociative electron capture

Dissociative electron capture is also known as dissociative resonance capture. It is a process similar to electron capture. The difference is that during the reaction, the sample molecule fragments or dissociates. The result is typically an anion and a neutral radical. Dissociative electron capture is illustrated by the reaction equation:

$$MX + e^{-}_{(thermal)} \rightarrow M^{\bullet} + X^{-}$$

This reaction does not yield the same sensitivity as electron capture, and the mass spectra generated typically have lower abundance of the molecular ion.

As with electron capture, the products of dissociative electron capture are not always stable. The reverse reaction sometimes occurs. This reverse reaction is sometimes called an associative detachment reaction. The equation for the reverse reaction is:

$$M^{\bullet} + X^{-} \rightarrow MX + e^{-}$$

Ion pair formation

Ion pair formation is superficially similar to dissociative electron capture. The ion pair formation reaction is represented by the equation:

$$MX + e^{-}_{(thermal)} \rightarrow M^{+} + X^{-} + e^{-}$$

As with dissociative electron capture, the sample molecule fragments. Unlike dissociative electron capture however, the electron is not captured by the fragments. Instead, the sample molecule fragments in such a way that the electrons are distributed unevenly and positive and negative ions are generated.

Ion-molecule reactions

Ion-molecule reactions occur when oxygen, water, and other contaminants are present in the CI ion source. Ion-molecule reactions are two to four times slower than electron-attachment reactions and do not provide the high sensitivity associated with electron capture reactions. Ion-molecule reactions can be described by the general equation:

$$M + X^- \rightarrow MX^-$$

where X^- is most often a halogen or hydroxyl group that was created by ionization of contaminants by electrons from the filament. Ion-molecule reactions compete with electron capture reactions. The more ion-molecule reactions that occur, the fewer electron capture reactions occur.

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