

## Agilent 7000 Series Triple Quad GC/MS

## **Operation Manual**



## 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.

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## **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**

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

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

	To Install Your Hardware Library
	Insert Disk 1 into your DVD drive and follow the prompts.
	This can be installed by anyone who has authority to copy information onto the receiving computer.
	To Install Your Software Library
	Incart the memory stick into a UCD part and follow the promote
	insert the memory stick into a USB port and follow the prompts.

See the Agilent 7000 Series Triple Quadrupole GC/MS System Quick Start document (G7000-90042) for more details on how to install this information on your computer.



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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.

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 1Abbreviations

Table 1	Abbreviations	(continued)
		(00

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)
000	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 7890 Series gas chromatograph. The Triple Quad MS features:

- One split flow turbomolecular vacuum pump
- Rotary vane foreline pump
- Independently MS heated ion source, with self-cleaning option available
- Chemical and electron-ionization modes available (PCI/NCI/EI)
- Two independently MS heated hyperbolic quadrupole mass filters and interface tip seal
- Single hexapole collision cell
- High-energy dynode (HED) electron multiplier detector.
- Independently GC heated GC/MS interface
- · Independently GC controlled collision cell gas flows
- 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.

## 7000 CI MS System

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
- 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.





## 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
- Quadrupole
- Ion source

Always cool these areas of the system to room temperature before working on them. They will cool faster if you first set the temperature of the heated zone to room temperature. Turn the zone off after it has reached the setpoint. If you must perform maintenance on hot parts, use a wrench and wear gloves. Whenever possible, cool the part of the instrument that you will be maintaining before you begin working on it.

#### WARNING

Be careful when working behind the instrument. During 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.

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 2Hydrogen accumulation mechanisms

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 <i>not</i> 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.	

**Table 2**Hydrogen accumulation mechanisms (continued)

#### 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.

#### 1 Introduction

- 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 C

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.

#### 1 Introduction

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.



#### **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.

#### 1 Introduction

## **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 72.



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- To Install a Capillary Column in the GC/MS Interface using UltiMetal Plus Flexible Metal Ferrules 38

This chapter shows you how to select, install and condition a GC column, which must be performed before you operate your MS.



#### 2 Installing GC Columns

## 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 57 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 8 for EI mode and Table 11 for CI mode.

Feature	7000 Series
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 <sup>*</sup>	4
Maximum gas flow, mL/min <sup>†</sup>	6.5
Maximum column id	0.53 mm (30 m length)
Self-cleaning source He flow, mL/min	0.4

Table 3Gas flows

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

#### 2 Installing GC Columns

## To Prepare a Capillary Column for Installation

#### Materials needed

- Capillary column
- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Ferrules, Vespel
  - 0.27 mm id, for 0.10 mm id columns (5062-3518)
  - 0.37 mm id, for 0.20 mm id columns (5062-3516)
  - 0.40 mm id, for 0.25 mm id columns (5181-3323)
  - 0.5 mm id, for 0.32 mm id columns (5062-3514)
  - 0.8 mm id, for 0.53 mm id columns (5062-3512)
- Ferrules, UltiMetal Plus Flexible Metal
  - 0.1 0.25 mm id column (G3188-27501)
  - 0.32 mm id column (G3188-27502)
  - 0.45 0.53 mm id column(G3188-26503)
  - 0.25 0.32 mm id UltiMetal column (G3188-27505)
  - 0.53 mm id UltiMetal column (G3188-27506)
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Inlet column nut (5181-8830 for Agilent 7890, for use with vespel ferrules)
- MS Transferline nut (G2855-20530, for use with UltiMetal Plus Flexible Metal Ferrules)
- 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.

#### 2 Installing GC Columns

## 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

#### 2 Installing GC Columns

## **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 using Vespel Ferrules



This procedure is for the installation of a capillary column directly into the analyzer. There are two types of ferrules that can be used in the GC/MS interface: Graphite Vespel or UltiMetal Plus Flexible Metal. This procedure explains how to install a column using vespel ferrules.

## Agilent 7890 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.

#### 2 Installing GC Columns

#### 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 67) and open the front analyzer chamber (See "To Open the Front Analyzer Chamber" on page 106). 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

# To Install a Capillary Column in the GC/MS Interface using UltiMetal Plus Flexible Metal Ferrules



This procedure is for the installation of a capillary column directly into the analyzer using UltiMetal Plus Flexible Metal ferrules.

## Agilent 7890 GC

### **Materials** needed

- Column cutter, ceramic (5181-8836) or diamond (5183-4620)
- Ferrules, UltiMetal Plus Flexible Metal
  - 0.1 0.25 mm id column (G3188-27501)
  - 0.32 mm id column (G3188-27502)
  - 0.45 0.53 mm id column(G3188-26503)
  - 0.25 0.32 mm id UltiMetal column (G3188-27505)
  - 0.53 mm id UltiMetal column (G3188-27506)
- Flashlight
- Magnifying loupe
- Gloves, clean
  - Large (8650-0030)
  - Small (8650-0029)
- Internal nut (G2855-20530)
- 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 67) and open the front analyzer chamber (See "To Open the Front Analyzer Chamber" on page 106). 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** Tighten the nut in the clockwise direction to the lower range of degrees listed for your ferrule type (Table 4). Continue to tighten in 5 degree increments until you feel the ferrule grip the column.
- **10** Check the GC oven to be sure that the column does not touch the oven walls.
- **11** Check the nut's tightness after one or two heat cycles; retighten as appropriate.

### 2 Installing GC Columns

Ferrule part number	Degrees to tighten internal nut
• G3188-27501	50-100 degrees
• G3188-27502	30-70 degrees
• G3188-27503	20-50 degrees
• G3188-27504	60 degrees
• G3188-27505	40-90 degrees
• G3188-27506	20-50 degrees

### **Table 4**Degrees to tighten the internal nut for proper swaging



Figure 5 Installing a capillary column in the GC/MS interface



Agilent 7000 Triple Quad GC/MS Operation Manual

3

# **Operating in Electron Impact (EI) Mode**

Operating the MS from the Data System 42 Monitoring the MS from the LCP 42 LCP Menus 44 The ELGC/MS Interface 45 Before You Turn On the MS 47 Pumping Down 48 Controlling Temperatures 48 Controlling Column Flow 49 Controlling Collision Cell Flow 49 Venting the MS 50 To Set Monitors for MS Temperature and Vacuum Status 52 To Set the MS Analyzer Temperatures 54 To Set the GC/MS Interface Temperature from the MassHunter Workstation 56 To Calibrate the Column 57 To Configure CC Gas 59 To Set the Collision Cell Gas Flow Rates 60 To Autotune the MS for El Mode 61 To Open Left Side Panel for Access to the Analyzer Chambers 63 To Pump Down the MS 64 To Vent the MS 67 To Switch from the CI Source to the El Source 70 To Move or Store the MS 72 To Set the Interface Temperature from the GC 74 To Save a Method on the GC 75



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 a high sensitivity EI source (G2591C) that includes an extractor lens, which provides higher sensitivity when ionizing the sample.

# 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:



Press [Menu] until the desired menu appears.



Press [Item] until the desired menu item appears.

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



Use [Up] to increase the displayed value or to scroll up (such as in a message list).



Use [**Down**] to decrease the displayed value or to scroll down (such as in a message list).



Use [Yes/Select] to accept the current value.



Use [No/Cancel] to 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 5 through Table 7 list the menus and selections.

Table 5MS Parameters menu

Action	Description
High Vacuum Pressure	Displays the analyzer pressure.
lon Source Temp, <sup>o</sup> C	Displays the actual ion source temperature and the setpoint.
Quad 1 Temp, <sup>o</sup> C	Displays the actual front mass filter temperature and the setpoint.
Quad 2 Temp, <sup>o</sup> 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.

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 6Network 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 6) 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  $^{\circ}$ 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.

Figure 6 shows the EI GC/MS interface. If the Self-Cleaning Source has been installed, refer to the EI/CI GC/MS interface.

See also

"To Install a Capillary Column in the GC/MS Interface using Vespel Ferrules" on page 35 and "To Install a Capillary Column in the GC/MS Interface using UltiMetal Plus Flexible Metal Ferrules" on page 38

### WARNING

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





# **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 67.
- 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)
- Foreline pump pressure

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 57.

# **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 flow rates to the EPC 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 60.

### 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 Fast Vent feature loads a user-defined GC method that sets an oven temperature, turns off the transfer line heater and sets the column flow to a user predefined level.

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 El Mode**

The largest influences on operating pressure in EI mode are the carrier gas (column) and collision cell gas flows. Table 8 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%.

	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 <sup>-1</sup>	1.11 * 10 <sup>-4</sup>	8.82 * 10 <sup>-2</sup>	6.05 * 10 <sup>-7</sup>	1.36 * 10 <sup>-1</sup>	1.31 * 10 <sup>-4</sup>
0.7	1.61 * 10 <sup>-1</sup>	1.10 * 10 <sup>-4</sup>	9.92 * 10 <sup>-2</sup>	7.75 * 10 <sup>-7</sup>	1.39 * 10 <sup>-1</sup>	1.31 * 10 <sup>-4</sup>
1	1.66 * 10 <sup>-1</sup>	1.10 * 10 <sup>-4</sup>	1.00 * 10 <sup>-1</sup>	8.38 * 10 <sup>-7</sup>	1.44 * 10 <sup>-1</sup>	1.31 * 10 <sup>-4</sup>
1.2	1.69 * 10 <sup>-1</sup>	1.10 * 10 <sup>-4</sup>	1.05 * 10 <sup>-1</sup>	9.38 * 10 <sup>-7</sup>	1.47 * 10 <sup>-1</sup>	1.31 * 10 <sup>-4</sup>
2	1.80 * 10 <sup>-1</sup>	1.11 * 10 <sup>-4</sup>	1.22 * 10 <sup>-1</sup>	1.36 * 10 <sup>-6</sup>	1.60 * 10 <sup>-1</sup>	1.32 * 10 <sup>-4</sup>
3	1.95 * 10 <sup>-1</sup>	1.12 * 10 <sup>-4</sup>	1.41 * 10 <sup>-1</sup>	1.82 * 10 <sup>-6</sup>	1.75 * 10 <sup>-1</sup>	1.32 * 10 <sup>-4</sup>
4	2.10 * 10 <sup>-1</sup>	1.12 * 10 <sup>-4</sup>	1.57 * 10 <sup>-1</sup>	2.33 * 10 <sup>-6</sup>	1.90 * 10 <sup>-1</sup>	1.31 * 10 <sup>-4</sup>
6	2.37 * 10 <sup>-1</sup>	1.13 * 10 <sup>-4</sup>	1.89 * 10 <sup>-4</sup>	3.29 * 10 <sup>-6</sup>	2.18 * 10 <sup>-1</sup>	1.34 * 10 <sup>-1</sup>

 Table 8
 Influence of carrier and collision cell gas flows on ion vacuum gauge readings

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 7.

🖳 Select Monitors	Hanna .		- • ×
Available Monitors GC APC-1A Pressure GC APC-1B Pressure GC APC-1C Pressure	Add>	Selected Mo GC Oven Temperature GC Inlet-F Temperature GC Column-1 Row Calc.	nitors
GC APC28 Pressure GC APC28 Pressure GC APC2C Pressure GC APC3A Pressure GC APC3B Pressure GC APC3C Pressure GC Aux-1 Temperature	< Remove	GC Aux-2 Temperature MS Source Temp	
GC Colum-2 How Calc. GC Colum-3 How Calc. GC Colum-4 How Calc. GC Colum-5 How Calc. GC Colum-5 How Calc. GC Det-B Fuel How GC Det-B Makeup How GC Det-B Tempenture			
GC Det-B Uil Flow GC Det-F Hel Row GC Det-F Makeup Flow GC Det-F Temperature GC Det-F Uit Flow GC Intet-B Pressure GC Intet-B Temperature			
GC Inlet-B Total Row GC Inlet-F Pressure GC Inlet-F Total Row GC PCM-1A Pressure GC PCM-1B Pressure GC PCM-2B Pressure			
GC PCM-3A Pressure GC PCM-3B Pressure GC Valve 1 GC Valve 2 GC Valve 3 GC Valve 3 GC Valve 4 GC Valve 5			
GC Valve 6 GC Valve 7 GC Valve 8 MS Filament Current MS High Vac MS NIS1 Heater MS NIS2 Heater			
MS Fump I Current MS Pump 2 Current MS Rough Vac MS Turbo1 Speed MS Turbo2 Speed			
OK Cancel	Help		
OK Cancel	Help		

Figure 7 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 In the Available Monitors column, select Foreline Pressure and click the Add button to move the selection to the Selected Monitors column.
- 8 Select any other monitors you want and add them to the **Selected Monitors** column.
- **9** Click **OK**. The new monitors will be stacked on top of each other in the lower right corner of the **Instrument Control** window.
- 10 Select Window > Arrange Plots and Monitors, or click and drag each monitor to the desired position. See Figure 8 for one way of arranging the monitors.

2000GCMS /MassHunter - DEFAULT.M / default.sequence.xml			- C - X
Method Instrument Sequence Abort Window Help			
	- 🐝		
M Instrument Control			
Idle Sample Name: Data File: 70003CIMS021409_4.D	(10.00) Run Time		
Sequence	Method	Instrument	
		🕹 🕂 🗋 💓 🦃	) ( <b>)</b>
49 Oven Temperature	0.0 165 Column-1 Flow Calc.	1 18.0 Source Temp. Turbo 1 Speed	2 MS1 Quad Temp.
MS2 Quad Temp. Spectrum	TIC		

Figure 8 Arranging monitors

**11** 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 9 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 9 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 9 for recommended setpoint.
- 7 Select the **Files and Reports** tab then click the **Save** button to save the tune file with these changes.

#### Table 9 Recommended temperature settings

l operation
50 - 280 °C
50 °C
50 °C

iC-QQQ Tune	<b>X</b>
MassHunter\GCMS\2\7000\atunes.pci.tune.xml	
9- 9-	
8-	
4.3	
30	
25	
UZ U4 U5 U8 I IZ IA I5 IS Z ZZ Z4 Z5 ZS 3 3Z 3A 35 35 4 4Z 4A 45 4 5 9Z 2A 35 55 5 5Z 54 55 55 1 7Z 7A 75 7S 5 5Z 54 5	.6 8.8 9 9.2 9.4 9.6 9.8
tus	Off
iotune Advanced Autotune Manual Tune Additional Parameters Files and Reports	
ton Source MS1 Collision Cell MS2 Detector Acquisition Vacuum Control	MS Mode
© TTI Cutoff 50.00 amu Peak Width Unit ▼ 0.7 Parameter	MS1 SIM
SIM         Mass         267.00         amu         MS1 Mass Avis Gain         0.00           Scan         From         35.00         To         650.00         Sten         0.10	MS1 Scan
Profile     Mass (m/z)     Window     MS1 Mass Axis Offset     1.000     amu     Mass     Volume	MS1 Profile
▶         41         10         MS1 Width Gain         25.0         amu         (m/2)         Value           ★ <th>MS2 Stan</th>	MS2 Stan
267 10 MS1 Width Offset -0.500 amu	MS2 Profile
* MS1 Quad DC 5.0 V	© MRM
Post Hiter UL 5.0 V	
Misi uluad remp. 150 C Z	
From To Step Parameter Value < III	•
Ramp Parameter	
AS1 Quad DC   From 5 To 20 Step 0.2 Dwell Time 25 Get Defaults Do Ramp Accept	
Acquire Start Stop Capture MS On MS C	ff

Figure 9 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 $^\circ\mathrm{C}$ for the quadrupole or 350 $^\circ\mathrm{C}$ for the source.

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

You can also use the  $\ensuremath{\textbf{GC}}$  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 10). This example has the GC/MS interface temperature configured as Thermal Aux 2.

🖳 GC Edit Parameters						
10	-1 🔿	0 📌	0	hu 🗙 🥹	▲ 1,2	
Thermal Aux 2		► (Initial)	Rate °C/min	Value °C 280	Hold Time min	Run Time min 1 E e extended by GC run time,
Apply	ОК	Cance	il <u>H</u> elp			

Figure 10 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.

GC Conditions				
If measurement method, please	If measurement was made under conditions different from loaded method, please enter them below.			
Temperature:		70 °C		
Pressure into co	lumn:	1.1739 psi		
Pressure out of	column:	0 psi		
		Vacuum		
Gas type:		Helium		
Holdup Time of an Ur	retained Peak:	1.1 min		
	Current	Calculated		
▶ Length	28.136 m	29.51 m		
Diameter	320 μm	320 μm		
Holdup	1 min	1.1 min		
	ок	Cancel		

Figure 11 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 12.

Agilent 7890A	
Connection Configuration	
192.168.1.57	
Miscellaneous Columns Modules ALS	4
Front Inlet	
SS Inlet He 👻	
Back Inlet	
Volatiles Interface He	
QQQ Collision Cell EPC	
Collision Gas N2 V2	
Front Detector Ar	
FID	
Makeup N2 -	
Lit Offset 2.0 pA	
Aux EPC 4.5.6	
Aux EPC 4 N2 -	
Airy EPC 5 N2 -	
Aux EPC 6 N2 -	
UK Cancel <u>H</u> elp	
	h.

Figure 12 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 13.
- 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.

🖳 GC Edit	t Parameters		
×	0 1 0 0 🔊	P 🥘 💷 🗙 🕲 🔔 🎁	
1	Description 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 He Quench Gas	
	QQQ Collision Cell EPC           Back Volatiles Interface He           Aux EPC 4 N2           Aux EPC 5 N2           Aux EPC 6 N2	Image: On     2.25 mL/min       N2 Collision Gas     Reset to Defaults       Image: On     1.5 mL/min	E
	Δρρίγ ΟΚ	Cancel <u>H</u> elp	

Figure 13 Setting collision cell gas flow rate

# To Autotune the MS for El Mode

The MS can be tuned using the MassHunter Workstation software.

### Procedure

- 1 Set the system to the same conditions (column flow and MS analyzer temperatures) that will be used for data acquisition. Since the GC temperature varies throughout the run, select a temperature in the mid range.
- 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-000 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 **El source**.
- **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

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.



CAUTION

### 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 63.
- **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.

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**.

Elapsed Tin	ne: 4 secs		
	Actuals	Criteria	
Turbo 1 Speed	23.0	> 90	%
Turbo 1 Power	600.000		W
Rough Vac	2.00E+1		Tor
High Vac	2.10E+1		Tor
Source Heater	6		"C
MS1 Heater	7		'C
MS2 Heater	39		°C



### CAUTION

Within 10 to 15 minutes the turbo pump speed should be up to 80% (Figure 15). 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 61 or "To Perform a CI Autotune" on page 94.)

# To Vent the MS

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

#### Procedure



- 1 Click the **MS Tune** icon from the **Instrument Control** panel.
- 2 Select the Manual Tune tab.
- **3** Select the Vacuum Control tab.

### 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.

**4** Click the **Vent** button. If your system supports DCOMM, MassHunter will load the Fast Vent method to turn off the GC oven and interface heaters, the source and quad heaters, the turbo pump, and the MS.

If your system does not support DCOMM or if DCOMM has been disabled, you must perform the following steps when prompted:

**a** Set the GC/MS interface heater and the GC oven temperatures to ambient (room temperature).

### CAUTION

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

- **b** Turn off the MS by pressing the power switch. (See Figure 1.)
- **5** Unplug the MS power cord.

### **3** Operating in Electron Impact (EI) Mode

### WARNING

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

**6** Remove the analyzer window cover (See "To Open Left Side Panel for Access to the Analyzer Chambers" on page 63).



Figure 16 The MS vent valve knob

7 Turn the vent valve knob (Figure 16) 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 67. 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 63.
- **3** Open the front analyzer chamber. See "To Open the Front Analyzer Chamber" on page 106.
- **4** Remove the CI interface tip seal. See "To Install the CI Interface Tip Seal" on page 136.
- **5** Remove the CI ion source. See "To Remove the CI Ion Source" on page 123.
- 6 Install the EI ion source. See "To Install the EI Ion Source" on page 121.
- 7 Place the CI ion source 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.

- 8 If necessary, start the MassHunter Data Acquisition program and pump down the MS. (See "To Pump Down the MS" on page 64)
- **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.
- 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 67.)
- 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 63).
- 7 Finger-tighten the side plate thumbscrews for both analyzers. (See Figure 17).

### 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 17 Side plate thumbscrews

The MS can now be stored or moved. The f oreline 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.

#### **3** Operating in Electron Impact (EI) Mode

# 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.

#### Never exceed the maximum temperature of your column.

### CAUTION

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.

# Operating in Electron Impact (EI) Mode



Triple Quad GC/MS Operation Manual

Δ

# Operating in Chemical Ionization (CI) Mode

Setting Up Your MS to Operate in CI Mode 78 The CI GC/MS Interface 79 Operating the CI MS 81 High Vacuum Pressure in CI Mode 82 Other Reagent Gases 83 CI Autotune 85 The CI Flow Control Module 87 To Switch from the EI Source to the CI Source 89 To Operate the Reagent Gas Flow Control Module 91 To Set a Reagent Gas Flow 93 To Perform a CI Autotune 94

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/2 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 18) 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 using Vespel Ferrules" on page 35.

### CAUTION

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

# WARNING

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



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

Figure 18 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 10), and electron energy may need to be optimized for your specific analyte.

	lon 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

 Table 10
 Temperatures for CI operation

# 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 11 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 11). They were taken with the following set of conditions. Note that these are typical PCI temperatures:

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

Ta	ble	11	l Typica	ıl analyzer	vacuum	with	reagent	gast	t	ow
----	-----	----	----------	-------------	--------	------	---------	------	---	----

	Collision N2 =	cell gas flow on 1.5, He = 2.25	Collision cell gas flow off		
MFC (%)	Rough vac	High vac	Rough vac	High vac	
10	1.77 × 10 <sup>-1</sup>	7.15 × 10 <sup>-5</sup>	1.33 × 10 <sup>-1</sup>	2.56 × 10 <sup>-6</sup>	
15	1.86 × 10 <sup>-1</sup>	7.19 × 10 <sup>−5</sup>	1.43 × 10 <sup>-1</sup>	3.00 × 10 <sup>-6</sup>	
20	1.94 × 10 <sup>-1</sup>	7.23 × 10 <sup>−5</sup>	1.53 × 10 <sup>-1</sup>	3.45 × 10 <sup>-6</sup>	
25	2.02 × 10 <sup>-1</sup>	7.27 × 10 <sup>-5</sup>	1.63 × 10 <sup>-1</sup>	3.86 × 10 <sup>-6</sup>	
30	2.10 × 10 <sup>-1</sup>	7.31 × 10 <sup>-5</sup>	1.71 × 10 <sup>-1</sup>	4.30 × 10 <sup>-6</sup>	
35	2.18 × 10 <sup>-1</sup>	7.39 × 10 <sup>-5</sup>	1.80 × 10 <sup>-1</sup>	4.76 × 10 <sup>-6</sup>	
40	2.25 × 10 <sup>-1</sup>	7.43 × 10 <sup>-5</sup>	1.88 × 10 <sup>-1</sup>	5.18 × 10 <sup>-6</sup>	

# **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 the *Agilent 7000 Triple Quadrupole GC/MS Concepts Guide*. If you are not experienced with chemical ionization, we suggest reviewing that material before you proceed.

### **Isobutane CI**

Isobutane  $(C_4H_{10})$  is commonly used for chemical ionization when less fragmentation is desired in the chemical ionization spectrum. This is because the proton affinity of isobutane is higher than that of methane; hence less energy is transferred in the ionization reaction.

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

### Ammonia Cl

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  $\text{NH}_4^+$  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  $\text{NH}_4^+$ ,  $\text{NH}_4(\text{NH}_3)^+$ , and  $\text{NH}_4(\text{NH}_3)_2^+$ .

# 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 12). 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

CAUTION

After the source is changed from El to Cl 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.

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

#### Table 12CI tune default settings

Parameter Methane		ls	Isobutane		mmonia	
lon polarity	Positive	Negative	Positive	Negative	Positive	Negative
Emission	150 μA	50 μA	150 μA	50 µA	150 μA	50 μA
Electron energy	150 eV	150 eV	150 eV	150 eV	150 eV	150 eV
Filament	1	1	1	1	1	1
Repeller	3 V	3 V	3 V	3 V	3 V	3 V
lon focus	130 V	130 V	130 V	130 V	130 V	130 V

### 4 Operating in Chemical Ionization (CI) Mode

# Table 12 CI tune default settings (continued)

Parameter	Methane		Isobutane		Ammonia	
Entrance lens offset	20 V	20 V	20 V	20 V	20 V	20 V
EM volts	1200	1400	1200	1400	1200	1400
Shutoff valve	Open	Open	Open	Open	Open	Open
Gas select	А	А	В	В	В	В
Suggested flow	20%	40%	20%	40%	20%	40%
Source temp	250 °C	150 °C	250 °C	150 °C	250 °C	150 °C
Front quad temp	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
Interface temp	280 °C	280 °C	280 °C	280 °C	280 °C	280 °C
Autotune	Yes	Yes	No	Yes	No	Yes

N/A Not available

# **The CI Flow Control Module**

The CI reagent gas flow control module (Figure 19 and Table 13) 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 13).



Figure 19 Reagent gas flow control module schematic

# 4 Operating in Chemical Ionization (CI) Mode

Result	Gas A flow	Gas B flow	Purge with Gas A	Purge with Gas B	Pump out flow module	Standby, vented, or El mode
Gas A	Open	Closed	Open	Closed	Closed	Closed
Gas B	Closed	Open	Closed	Open	Closed	Closed
MFC	On $ ightarrow$ setpoint	$0n \rightarrow setpoint$	$0n \rightarrow 100\%$	$0n \rightarrow 100\%$	$0n \rightarrow 100\%$	0ff →0%
Shutoff valve	Open	Open	Open	Open	Open	Closed

 Table 13
 Flow control module state diagram

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

# To Switch from the El Source to the Cl Source

# CAUTION

Always verify GC/MS performance in El before switching to Cl operation.

#### Procedure

- 1 Vent the MS. See "To Vent the MS" on page 67.
- **2** Open the front analyzer chamber. See "To Open the Front Analyzer Chamber" on page 106.
- **3** Remove the EI ion source. See "To Remove the EI Ion Source" on page 109.

**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 134.
- **5** Install the interface tip seal. See "To Install the CI Interface Tip Seal" on page 136.
- **6** Close the analyzer.
- 7 Pump down the MS. See "To Pump Down the MS" on page 64. 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.

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

**Table 14**Default Tune Control Limits, used by CI autotune only

#### Notes for Table 14:

- 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  $\mu$ A.

# To Operate the Reagent Gas Flow Control Module

#### Procedure

 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.



Figure 20 CI flow control

#### 4 Operating in Chemical Ionization (CI) Mode

**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.

**Cl 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.

#### 4 Operating in Chemical Ionization (CI) Mode

# **To Perform a CI Autotune**

### CAUTION

Always verify MS performance in El 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 lon source tab. In the Cl Reagent Gas section, select Cl Gas A or Cl Gas B as the reagent gas and then enter a Cl 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.

# 4 Operating in Chemical Ionization (CI) Mode



Agilent 7000 Triple Quad GC/MS Operation Manual

5

# **General Maintenance**

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To Close the Rear Analyzer Chamber 155



# **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 15. 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.

Task	Every week	Every 6 months	Every year	As needed
Tune the MS				Х
Check the foreline pump oil level	Х			
Check the calibration vial(s)		Х		
Replace the foreline pump oil $\overset{{}_{\scriptstyle \ast}}{}$		Х		
Check the foreline pump				Х
Clean the ion source				Х
Check the carrier gas trap(s) on the GC and MS				Х
Replace the worn out parts				Х
Lubricate sideplate or vent valve O-rings $^{\dagger}$				х
Replace GC gas supplies				Х

#### Table 15Maintenance schedule

\* Or as needed.

\* Vacuum seals other than the side plate 0-ring and vent valve 0-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.

#### 5 General Maintenance

### **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 15 on page 98, 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 102 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 102) 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 102) before you open the analyzer chamber.

#### Procedure

- 1 Vent the MS. (See "Venting the MS" on page 50.)
- **2** Open the left side panel. (See "To Open Left Side Panel for Access to the Analyzer Chambers" on page 63.)

### 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 21) 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.

# 5 General Maintenance



Chamber open

Figure 21 The front analyzer chamber
# To Remove the El 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 67.)

# 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 106.)
- **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 22 and Table 16).

**4** Trace the wires for the ion source heater and temperature sensor to the feedthrough board. Disconnect them there (Figure 22).

Wire color	Connects to	Number of leads	
		Extractor	Drawout assembly
Blue	Entrance lens	1	1
Orange	lon 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
White beaded	Feedthrough board (bottom left)	2	2
White	Feedthrough board (bottom middle)	2	2

Table 16El lon source wires

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



Figure 22 Removing the El ion source

# To Disassemble the El 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 109.
- 2 Remove the filaments. See "To Remove a Filament" on page 138.
- **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 23.)
- **4** Disassemble the repeller assembly by removing the repeller nut, washers, ceramic insulators and repeller. (See Figure 23.)
- **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, extractor lens, extractor lens insulator, and entrance lens. (See Figure 23.)



Figure 23 Disassembling the El ion source

Table 17 Parts list for El ion so	urce (Figure 23)
-----------------------------------	------------------

ltem number	Item description
1	Setscrews
2	Screws
3	Source body
4	Extractor lens

ltem number	Item description
5	Extractor lens insulator
6	Filaments
7	Spring washer
8	Lens insulator
9	Entrance lens
10	lon focus lens
11	Repeller insulator
12	Repeller
13	Flat washer
14	Belleville spring washer
15	Repeller nut
16	Source heater block assembly
17	Insulator

### Table 17 Parts list for El ion source (Figure 23) (continued)

# To Clean the El 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 Ion Source" on page 112.
- 2 Collect the following parts to be cleaned if you are cleaning an EI ion source: (Figure 24)
  - Repeller
  - Source body
  - Extractor lens
  - Ion focus lens
  - Entrance lens

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

# CAUTION

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



Figure 24 El Source parts to be cleaned

### CAUTION

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

- **3** If the contamination is serious, such as an oil backflow into the analyzer, seriously consider replacing the contaminated parts.
- **4** Abrasively clean the surfaces that contact the sample or ion beam.

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

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

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

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

#### CAUTION

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

- 7 Ultrasonically clean the parts (each group separately) for 15 minutes in each of the following solvents:
  - Methylene chloride (reagent-grade)
  - Acetone (reagent-grade)
  - Methanol (reagent-grade)

# WARNING

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

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

# To Assemble the El 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

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.

- **5** Assemble the repeller assembly by attaching the repeller, repeller insulators, washers, and repeller nut onto the source heater assembly.
- **6** Attach the repeller assembly to the source body with two screws and washers.
- 7 Install the filaments. See "To Install a Filament" on page 140.



Figure 25 Assembling the El ion source

	Table 18	B Parts	list for El	ion source	(Figure 25
--	----------	---------	-------------	------------	------------

ltem number	Item description
1	Setscrews
2	Screws
3	Source body
4	Extractor lens
5	Extractor lens insulator
6	Filaments

ltem number	Item description
7	Spring washer
8	Lens insulator
9	Entrance lens
10	lon focus lens
11	Repeller insulator
12	Repeller
13	Flat washer
14	Belleville spring washer
15	Repeller nut
16	Source heater block assembly
17	Insulator

### Table 18 Parts list for El ion source (Figure 25) (continued)

# To Install the El 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 141.
- **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 145.
- **5** Pump down the MS. (See "To Pump Down the MS" on page 64.)
- **6** Tune the MS. (See "To Autotune the MS for EI Mode" on page 61.)



Figure 26 Installing the El 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 67.)

WARNINGThe analyzers, GC/MS interface, and other components in the analyzer chamber<br/>operate at very high temperatures. Do not touch any part until you are sure it is cool.CAUTIONAlways wear clean gloves to prevent contamination when working in the analyzer<br/>chamber.2Open the front analyzer chamber. (See "To Open the Front Analyzer<br/>Chamber" on page 106.)CAUTIONMake sure you use an antistatic wrist strap and take other antistatic precautions<br/>before touching analyzer components.CAUTIONWhen 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 19, Figure 36).
- **4** Trace the wires for the ion source heater and temperature sensor to the feedthrough board. Disconnect them there (Figure 34).

Wire color	Connects to	Number of loads
WITE COIDI	CONNECTS TO	Number of leaus
Blue	Entrance lens	1
Orange	lon focus	1
White	Filament 1 (top filament)	2
Red	Repeller	1
Black	Filament 2 (bottom filament)	2
White beaded	Feedthrough board (bottom left)	2
White	Feedthrough board (bottom middle)	2
Brown	Not used here	n/a

Table 19CI lon source wires

- **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 123.
- 2 Remove the filaments. See "To Remove a Filament" on page 138.
- **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.)





 Table 20
 Parts list for the Cl ion source (Figure 28)

Item number	Item description
1	Setscrew
2	Filament screw
3	CI interface tip seal
4	CI repeller insulator
5	CI lens insulator
6	CI drawout cylinder
7	CI drawout plate

ltem number	Item description
8	CI ion source heater block assembly
9	Entrance lens
10	Cl ion source body
11	Cl ion focus lens
12	CI repeller
13	CI filament
14	Dummy filament
14	Tool box

### Table 20 Parts list for the CI ion source (Figure 28) (continued)

# 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 126.
- 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.







CAUTION

#### 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.

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.





Table 21	Parts list for the CI ion source	(Figure 30	)
----------	----------------------------------	------------	---

ltem number	Item description
1	Setscrew
2	Filament screw
3	CI interface tip seal
4	CI repeller insulator
5	CI lens insulator
6	CI drawout cylinder
7	CI drawout plate
8	CI ion source heater block assembly

ltem number	Item description
9	Entrance lens
10	Cl ion source body
11	CI ion focus lens
12	Cl repeller
13	Cl filament
14	Dummy filament
14	Tool box

 Table 21
 Parts list for the CI ion source (Figure 30) (continued)

# 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 67.
- **2** Open the front analyzer chamber. See "To Open the Front Analyzer Chamber" on page 106.
- **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 145.)
- 7 Pump down the MS. (See "To Pump Down the MS" on page 64.)
- 8 Tune the MS. (See "To Perform a CI Autotune" on page 94.)

# 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. The CI tip seal can remain in place when operating with an EI source.

### CAUTION

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

#### Procedure

**1** Remove the 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 145.)



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 50.)

# 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 106.
- **3** Remove the ion source. See "To Remove the EI Ion Source" on page 109 or see "To Remove the CI Ion Source" on page 123.
- **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 (G7005-60061)
- Filament assembly, 2-pk, CI (G7005-60072)
- Gloves, clean, lint-free
  - Large (8650-0030)
  - Small (8650-0029)
- Tweezers (8710-2460)

#### Procedure

- 1 Remove the old filament. (See "To Remove a Filament" on page 138.)
- 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 121 or see "To Install the CI Ion Source" on page 134.)
- **6** Close the front analyzer chamber. (See "To Close the Front Analyzer Chamber" on page 145.)
- 7 Pump down the MS. (See "To Pump Down the MS" on page 64.)
- 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 22.

The wiring is described in Table 22 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.

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 El 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)	lon focus lens	
Blue (1)	Board, top right (ENT LENS)	Entrance lens	
White beaded (2)	lon source heater	Board, bottom left (HTR)	
White (2)	lon source sensor	Board, bottom (RTD)	
Green	Board, bottom left	lon source radiator	
Yellow	Board, bottom left	Front quad	

#### Table 22Analyzer wiring



Figure 34 Feedthrough board wiring



Figure 35 El Source wiring

#### FB = Feedthrough Board



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 64.)

## 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-10 and T-20

### 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 146.)
- **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

### 5 General Maintenance

## 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 102) before you open the analyzer chamber.

### Procedure

- 1 Vent the MS. (See "To Vent the MS" on page 67.)
- 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 146.)

## 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 21) 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



The 7000 series mass spectrometers are shipped with two different series of Triple Axis Detectors. The two series have multipliers that cannot be interchanged, therefore it is important to order the correct version of multiplier when it is being replaced. The replacement multiplier part number is stamped on the front face of the detector. Through MassHunter you can determine which series detector you have without having to directly check the detector. The detector series is displayed as either "Triple Axis Series 1" or "Triple Axis Series 2" in the detector tab of manual tune, in the detector section on the second page of the tune report and in the pump down window.

### **Materials** needed

- Electron multiplier horn (Series I HED TAD G3170-80103, Series II HED TAD G7000-80103 )
- 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 67.)

## 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 148.)

### 5 General Maintenance

- **3** Open the retaining clip (Figure 40 and 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 155.)



Figure 39 Replacing the horn (TAD Series 1)



**Figure 40** Replacing the horn (TAD Series 2)



Figure 41 Series 1 and Series 2 electron multiplier horns

## 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 64.)

## 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.

### 5 General Maintenance

- **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.



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