Agilent Technologies

# 6850 Series II GC User Information

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# Important Information
## About your 6850 Gas Chromatograph



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Part No. G2630-90329

First edition, March 2004

Replaces Part No. G2630-90327, G62630-90328

Printed in USA

## Important safety warnings

There are several important safety notices that you should always keep in mind when using the 6850 GC.

### Many internal parts of the GC carry dangerous voltages

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

- The wiring between the GC power cord and the AC power supply, the AC power supply itself, and the wiring from the AC power supply to the power switch.

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

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

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

**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 GC electronics

The printed circuit (PC) boards in the GC 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 operate at temperatures high enough to cause serious burns. These parts include but are not limited to:

- The inlet
- The oven and its contents
- The detector
- The column nuts attaching the column to an inlet or detector
- The valve box

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

**Warning** Be careful when working behind the instrument. During cool-down cycles, the GC emits hot exhaust which can cause burns.

### Insulation fibers can cause irritation

**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; wash your hands with mild soap and cold water after handling the insulation.

### Hydrogen

Hydrogen gas may be used as carrier gas, and/or as fuel for a detector. When mixed with air, hydrogen can form explosive mixtures.

**Warning** When using hydrogen (H2) as the carrier gas or fuel gas, be aware that hydrogen gas can flow into the oven and create an explosion hazard. Therefore, be sure that the supply is 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.

**Warning** 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.

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.

### Microcell electron capture detector (µECD)

This section describes the licensing information, handling precautions and safety requirements concerning the µECD.

The µECD contains a cell plated with $^{63}$Ni, a radioactive isotope. $^{63}$Ni releases β particles which collide with carrier gas molecules to produce low-energy electrons — each β particle produces approximately 100 electrons. The free electrons produce a small current — called the *reference* or *standing current* — which is collected and measured.

## The $^{63}$Ni isotope

The radioactive isotope used in the cell is $^{63}$Ni. It is plated onto the inner surface of the cell body and is solid at temperatures used in chromatography. Some other properties are listed in Table 1.

### Table 1. Properties of $^{63}$Ni

| | |
|---|---|
| Half-life: | 101.1 years |
| Emission: | 65.87 keV max., beta radiation |
| Melting point: | 1453 °C |
| Dimensions of the active part of the $\mu$ECD: | Inside diameter: 6 mm<br>Height: 4.2 mm |
| Total activity ($\mu$ECD cell): | 555 MBq (15 millicuries) maximum |

## µECD licenses

Customers in the United States can purchase a µECD under either a General License or a Specific License. Customers outside the United States should contact their local Agilent sales office for information.

The license details below reflect U.S. regulations.

### Specific license

Specific license µECDs require you to obtain a Materials License from the Nuclear Regulatory Commission (NRC) or local state agency, permitting you to possess the amount and kind of radioisotope used in the detector. You can typically ship, sell, or transfer the µECD to other Specific Licensees. If the license permits, you may also open the µECD for cleaning.

### General license

General license µECDs do not require a Materials License. You become a General Licensee automatically when you purchase a µECD directly from Agilent Technologies. Some states may require that you register the µECD with a state agency.

Certain restrictions apply to General Licenses:

- Owners may not open the µECD cell.
- Owners shall not modify the cell in any manner.
- Owners shall not use any solvent, including water, to internally clean the cell.

- Owners shall not interfere with or attempt to defeat the overheat circuitry that may be supplied with the µECD.
- Owners shall not transfer the µECD to another person or another location except as described in the applicable Regulations.
- Owners must perform a radioactive leak test at least every 6 months or as required by your local Agency.
- Owners must maintain records as required by your local Agency (the NRC or, in certain states, a state agency).
- Owners must notify the Agency in case of incidents or failures that might lead to a hazardous condition.

Additional information is available in the publication "Information for General Licensees," part no. 5961-5664.

### µECD warnings

Although beta particles at this energy level have little penetrating power —the surface layer of the skin or a few sheets of paper will stop most of them—they may be hazardous if the isotope is ingested or inhaled. For this reason the cell must be handled with care: radioactive leak tests must be performed at the required intervals, the inlet and outlet fittings must be capped when the detector is not in use, corrosive chemicals must not be introduced into the detector, and the effluent from the detector must be vented outside the laboratory environment.

**Warning** Materials that may react with the $^{63}$Ni source, either to form volatile products or to cause physical degradation of the plated film, must be avoided. These materials include oxidizing compounds, acids, wet halogens, wet nitric acid, ammonium hydroxide, hydrogen sulfide, PCBs, and carbon monoxide. This list is not exhaustive but indicates the kinds of compounds that may cause damage to 63Ni detectors.

**Warning** In the extremely unlikely event that both the oven and the detector heated zone should go into thermal runaway (maximum, uncontrolled heating in excess of 400 °C) at the same time, and that the detector remains exposed to this condition for more than 12 hours, take the following steps:

- After turning off the main power and allowing the instrument to cool, cap the detector inlet and exhaust vent openings. Wear disposable plastic gloves and observe normal laboratory safety precautions.
- Return the cell for exchange. Contact your local sales office for details.
- Include a letter stating the condition of abuse.

It is unlikely, even in this very unusual situation, that radioactive material will escape the cell. However, permanent damage to the $^{63}$Ni plating within the cell is possible, and therefore, the cell must be returned for exchange.

**Warning** Do not use solvents to clean the µECD.

You may not open the µECD cell unless authorized to do so by your local nuclear regulatory agency. Do not disturb the four socket-head bolts. These hold the cell halves together. Removing or disturbing them is a violation of the terms of the General License and could create a safety hazard.

### Safety precautions when handling µECDs

Always observe the following precautions:

- Never eat, drink, or smoke when handling µECDs.
- Always wear safety glasses when working with or near open µECDs.
- Wear protective clothing such as laboratory jackets, safety glasses, and gloves, and follow good laboratory practices. Wash hands thoroughly with a mild non-abrasive cleaner after handling µECDs.
- Cap the inlet and outlet fittings when the µECD is not in use.
- Connect the µECD exhaust vent to a fume hood or vent it to the outside.

Agilent Technologies recommends a vent line inside diameter of 6 mm (1/4-inch) or greater. With a line of this diameter, the length is not critical.

## Safety and regulatory certifications

The 6850 GC conforms to the following safety standards:

- Canadian Standards Association (CSA): C22.2 No. 1010.1
- CSA/Nationally Recognized Test Laboratory (NRTL): UL 61010A–1
- International Electrotechnical Commission (IEC): 61010–1
- EuroNorm (EN): 61010–1

The 6850 GC conforms to the following regulations on Electromagnetic Compatibility (EMC) and Radio Frequency Interference (RFI):

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

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

CE

The 6850 GC is designed and manufactured under a quality system registered to ISO 9001.

## Information

The Agilent Technologies 6850 Gas Chromatograph meets the following IEC (International Electro-technical Commission) classifications: Safety Class I, Transient Overvoltage Category II, Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and is designed for use indoors. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired. Whenever the safety protection of the 6850 Gas Chromatograph has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

Refer servicing to qualified service personnel. Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard.

The instrument weighs approximately 23 kg (50 lbs) and therefore should be moved in an upright position by two people supporting the bottom of the unit.

## Sound Emission Certification for Federal Republic of Germany

### Sound pressure

Sound pressure Lp < 48 dB(A) according to DIN-EN 27779.

When operating the 6850 with cryo valve option, the sound pressure 92.5 dB(A) during cryo valve operation for short burst pulses.

### Schalldruckpegel

Schalldruckpegel LP < 48 dB(A) nach DIN-EN 27779.

Bei Betrieb des 6850 mit Cryo Ventil Option treten beim Oeffnen des Ventils impulsfoermig Schalldrucke Lp bis ca. 92.5 dB(A) auf.

## Safety 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.

### In the manual

**Warning** A warning calls attention to a condition or possible situation that could cause injury to the user.

**Caution** A caution calls attention to a condition or possible situation that could damage or destroy the product or the user's work.

**On the instrument**

See accompanying instructions for more information.

Indicates a hot surface.

Indicates hazardous voltages.

Indicates earth (ground) terminal.

Indicates explosion hazard.

Indicates radioactivity hazard.

Indicates electrostatic discharge hazard.

Pinch hazard.

## Electromagnetic compatibility

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

1. This device may not cause harmful interference.
2. 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.
7. Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

# Miscellaneous

## Fuses and batteries

Table 2 and Table 3 list the fuses and batteries required for proper operation. These should only be accessed by Agilent service personnel.

**Table 2. AC Board Fuses**

| Fuse designation | Line voltage | Fuse rating and type |
|---|---|---|
| F1, F2 | All | 6 A, glass body, IEC Type F, non-time delay |
| F3, F4 | All, regular oven | 15 A, ceramic body, IEC Type F, non-time delay |
| | All, fast-heating oven | 20 A, ceramic body, IEC Type F, non-time delay |

**Table 3. Main Board Fuses**

| Fuse designation | Fuse rating and type |
|---|---|
| F1, F2, F3 | 5 A, 250 VAC, IEC 127 type f (non-time delay), ceramic body |

The battery on the main board is BT1, 3-volt lithium battery, Panasonic BR3032.

## Cleaning

To clean the exterior of the unit, disconnect the power and wipe down with a damp, lint-free cloth.

## Recycling the product

For recycling, contact your local Agilent sales office.

# Introduction

The 6850 Gas Chromatograph (GC) is a single-channel instrument with one inlet, a column oven, a detector, a simple keypad, and a display as shown in Figure 1.

To use it, you create a set of operating instructions, called a **method**, using the optional G2629A Control Module or a computer-based control device such as Agilent ChemStation or Agilent Cerity Networked Data System for Chemical QA/QC (Cerity Chemical). You can load and store these methods on your GC.

You can also:

- Use a sample introduction system, such as an automatic injector or gas or liquid sampling valve, to automate analyses.
- Use the Control Module to transfer methods from one 6850 GC to another, store methods on a PC flash memory card, diagnose problems in your instrument, and perform tests and checks.

## Display and keypad

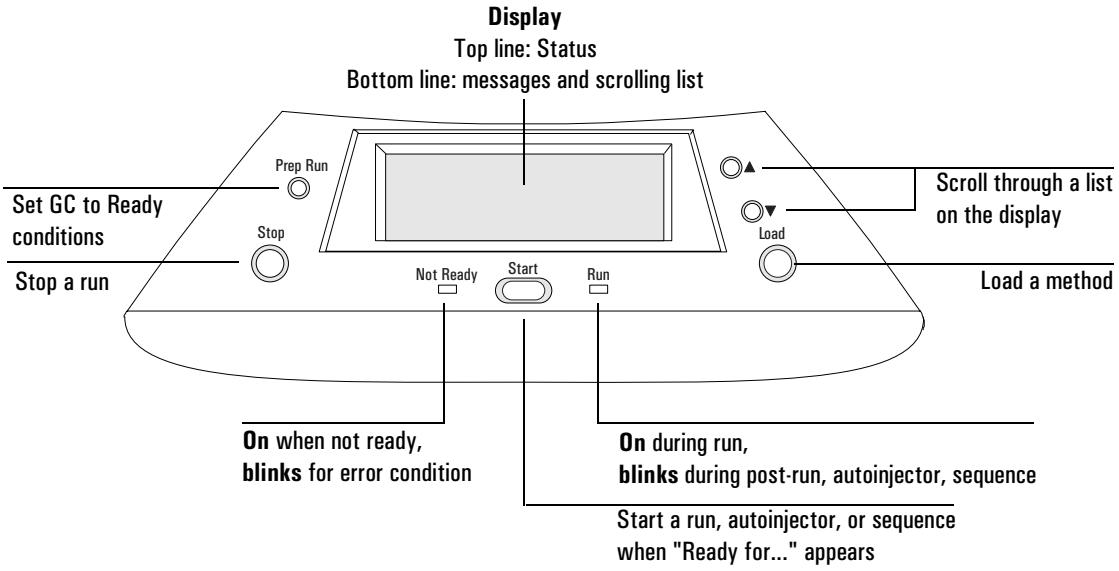



**Figure 1. Display and keypad**

Some keypad functions may be deactivated by the Control Module or ChemStation/Cerity Chemical, for example, to create a dedicated "run-only" unit. To learn about creating a "run-only" GC, refer to your ChemStation/Cerity Chemical or Control Module documentation.

Depending on the configuration set by the control module or data system, during a run the scrolling display can show:

- Oven temperature
- Inlet pressure
- Column flow rate
- Raw detector signal
- Messages
- Sequence information
- Run time

## Methods and memory

A **method** is a group of settings (times, temperatures, menus choices, on/off settings, etc.) that control how the GC analyzes an injected sample.

Three methods are loaded at the factory:

- An active method (defined shortly) with default setpoints.
- A checkout method for the detector.
- A `SERVICE` method used to prepare the GC for servicing. You can modify this method if needed. See your Control Module or GC ChemStation/Cerity Chemical documentation for details.

### GC long-term memory

Use a Control Module or ChemStation/Cerity Chemical to name and store methods in long-term memory. A backup battery prevents loss when power is turned off.

You can define and store up to six methods in the GC. 6850 Series GC methods that meet the 6850 instrument configuration can be used on the 6850. See your Agilent GC ChemStation/Cerity Chemical documentation for further details.

### The active method

The **active method** is the method the GC is currently using. To use a different method, you must **load** it. When a different method is loaded, the GC components immediately begin changing to the set of control values specified in the new method.

If you modify a method after loading it, the display will show a `+` symbol after its name. For example, `Default+` means you made changes to the `Default` method. Use the Control Module or ChemStation/Cerity Chemical to save the changes, if desired.

## Loading a method

To load an existing method:

1. Use the GC keypad scroll buttons to display the method you want to load.
2. Press `Load`.
3. The selected method loads and becomes the active method. The GC immediately begins changing to the new setpoints.

Allow a few seconds for the method to load completely. The Not Ready light comes on and remains on until the GC has stabilized. Stabilization time depends on how much the new and old setpoints differ.

## Run control features

### Gases

The GC automatically controls all gas pressures, adjusting for temperature and for any programs in your method.

Use a Control Module or ChemStation/Cerity Chemical to enter inlet, column, and detector gas stream settings as pressures or as flow rates. To use inlet and column flow rates, you **must** *define* the column by entering its dimensions (see your Control Module or ChemStation/Cerity Chemical documentation for details). We strongly recommend that you define your columns to take advantage of the GC's flow control capability.

### Column oven and column

Use a Control Module or ChemStation/Cerity Chemical to program up to six temperature ramps, separated by hold periods.

To control the behavior of the carrier gas in the column, specify constant or ramped flow, or constant or ramped pressure. The GC maintains this behavior for the entire run, even with temperature programming. The flow modes are available only if the column is defined.

### Automation

Use a Control Module or ChemStation/Cerity Chemical to:

- Set injector control parameters (sample size, syringe size, number of sample pumps, and other injection parameters)
- Build a **run table** to execute commands at specified times after injection
- Build a **clock table** to execute commands at specified times of day
- Create a **sequence** to analyze a set of samples (vials or stream selection valve positions) by the active method
- Inject using a gas or liquid sampling valve
- Control a multiposition stream selection valve

## Gas connections

All gas connections are made on the mainframe and lid back panels as shown in Figure 2.

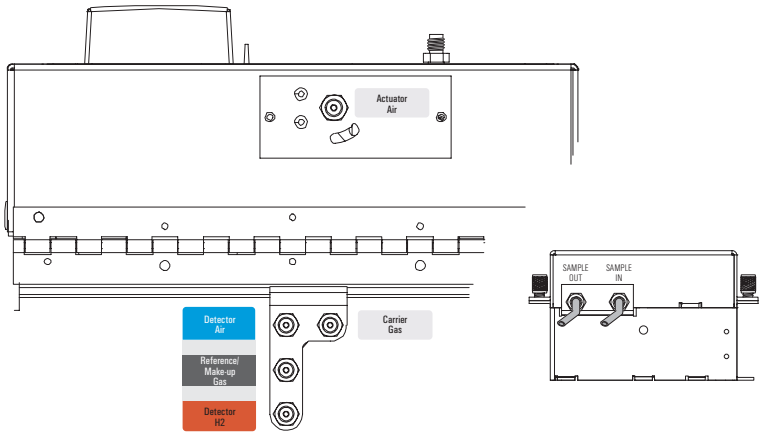



**Figure 2. Gas connections**

- **Actuator gas**—Valves or other devices may require a gas to drive them, usually compressed air. There is no high purity requirement for this gas. Since the supply pressure may experience surges as the device operates, the actuator gas must come from a different source than FID Air.
- **FID $H_2$**—The fuel gas for the flame ionization detector (FID)
- **Ref/makeup gas**—For an FID or a thermal conductivity detector (TCD), the makeup gas. For a TCD, this is also the reference gas. The reference/ makeup gas must be the same as the carrier gas, and can be taken from the same source.
- **FID air**—Supports combustion in the detector and must be separate from the actuator gas, if air is used there
- **Carrier gas**—The gas that moves the sample through the column

## Electrical connections

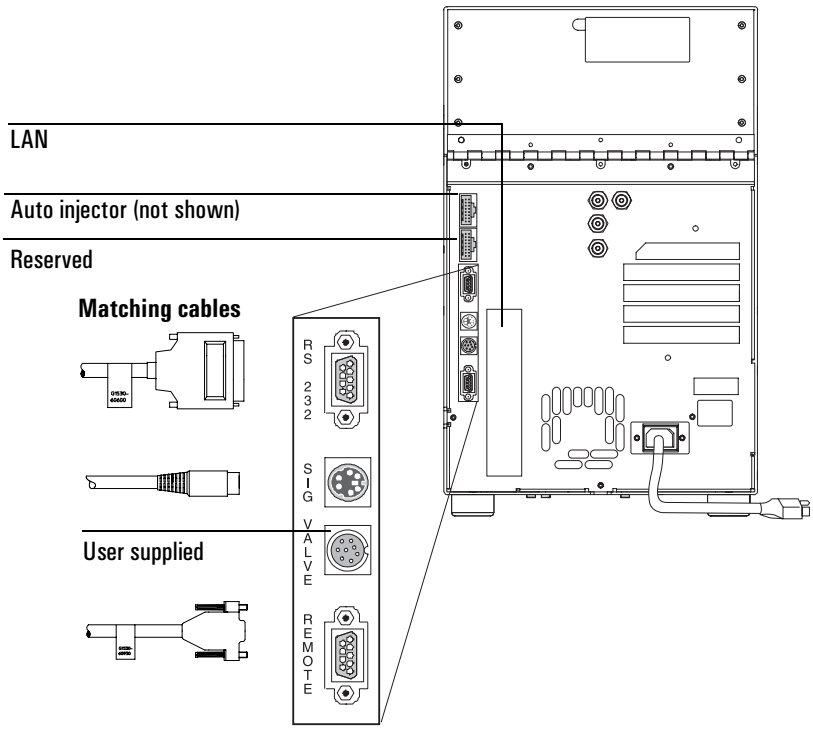



**Figure 3. Electrical connections**

### Auto injector

Controls an automatic injector.

### Reserved

For future development. Do not use.

### RS-232 connector

Use this connector for non-LAN external computers and data collection systems.

**Table 4. RS-232 Connector**

| Pin | Function | Input or output |
|---|---|---|
| 1 | No connection | |
| 2 | RXD | Input |
| 3 | TXD | Output |
| 4 | DTR | Output |
| 5 | GND | |
| 6 | DSR | Input |
| 7 | RTS | Output |
| 8 | CTS | Input |
| 9 | No connection | |

### SIG connector

The SIG connector is used for communication with an Agilent integration device, such as the 3395/96/97. It can also be configured to drive a strip chart recorder.

**Table 5. SIG Connector**

| Pin | Function |
|---|---|
| 1 | 1 mV COM |
| 2 | 1 V and 10 V COM |
| 3 | 1 mV |
| 4 | 1 V |
| 5 | Chassis GND |
| 6 | 10 V |

## VALVE connector

This connector provides the control relays and a BCD input for a stream selector Multi Valve.

**Table 6. VALVE Connector**

| Pin | Function | Maximum rating |
|---|---|---|
| 1 | Relay | 48 V AC/DC, 250 mA |
| 2 | Relay | 48 V AC/DC, 250 mA |
| 3 | LS digit 0 | |
| 4 | LS digit 1 | |
| 5 | LS digit 2 | |
| 6 | LS digit 3 | |
| 7 | MS digit 0 | |
| 8 | GND | |
| Shield | Chassis GND | |

## REMOTE connector

This connector provides Agilent APG remote start and stop capability.

**Table 7. REMOTE Connector**

| Pin | Function | Logic |
|---|---|---|
| 1 | Digital ground | |
| 2 | Prepare | LOW true |
| 3 | Start | LOW true (input) |
| 4 | Start relay | |
| 5 | Start relay | |
| 6 | No connection | |
| 7 | Ready | HIGH true (output) |
| 8 | Stop | LOW true |
| 9 | No connection | |

### LAN card

The LAN card in the GC conforms to industry standards and enables communication with the Cerity Chemical and ChemStation products as well as other external computers equipped with a LAN interface and suitable software. If you are using Cerity Chemical or ChemStation, the method of setting the IP address depends on the type of LAN card you have installed in your GC. See "Setting your IP address" on page 245 for more information.

## Data rates

Digital output to the ChemStation/Cerity Chemical is available at 11 speeds ranging from 0.1 Hz to 200 Hz, capable of handling peaks from 0.001 to 2 minutes wide. The data rate is selected from the ChemStation/Cerity Chemical and is described in the ChemStation/Cerity Chemical documentation.

## Keypad lockout

While the ChemStation/Cerity Chemical is controlling the GC, you cannot load methods from the GC keypad. The ChemStation/Cerity Chemical can also disable other keys and functions, for example, what information is available on the scrolling display.

# Columns and Traps

This section describes how to install and condition columns, traps, and related hardware in your GC in preparation for an analysis.

## Opening the oven

1. If an auto injector is present, lift it off its mounting post and place it on the bench top or on the (optional) bracket mounted on the side of the GC.
2. Press the buttons on each side of the GC just below the lid to release the latches (Figure 4).
3. Raise the lid.

To close the lid, press down on both top front corners to compress the insulation and engage the latches.

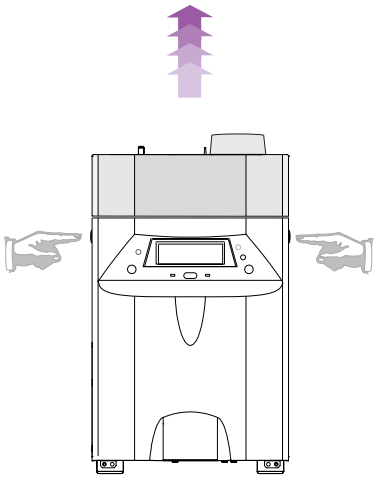


**Figure 4. Opening the oven**

## Capillary column hanger

Agilent capillary columns are wound on wire frames that slip over a hanger connected to the bottom of the oven lid (Figure 5).


**Figure 5. Capillary column hanger**

### Removing the capillary column hanger

If you are using a packed column, you will need to remove the column hanger.

1. Remove the two screws that secure the hanger to the top of the oven, then remove the hanger.
2. Reinstall the screws in the oven lid so you don't lose them. Save the hanger.

## Ferrules for capillary columns

Table 8 lists some of the ferrules used with capillary columns. See the Agilent catalog for consumables and supplies for a more complete listing.

### Graphite and graphitized-Vespel® ferrules

Select a ferrule that is sized for your column. Finger-tighten the connection and then use a wrench to make a 1/4 turn to produce a good seal. Loose ferrules may require so much force that the inlet fitting, the nut, or the ferrule may be damaged. With hard ferrules, it is best to start with an undersized hole and drill it to fit the column.

### Vespel ferrules

These ferrules can be more leak-tight than graphite but they have a lower temperature limit. Retighten after a few oven temperature cycles.

**Table 8. Hardware Used with Capillary Columns**

| Item, with ferrule ids | Typical use | Part no. |
|---|---|---|
| 1/4-in. graphitized Vespel ferrule, pkg of 10 | Inlet/detector liner/adapters | 5080–8774 |
| 1.0 mm graphite ferrule, pkg of 10 | 0.75 mm and 530 $\mu$m capillary columns | 5080–8773 |
| 0.5 mm graphite ferrule, pkg of 10 | 100 $\mu$m, 200 $\mu$m, 250 $\mu$m, and 320 $\mu$m capillary columns | 5080–8853 |
| Column nut | Connect column to inlet or detector | G2630–80720 |
| Column cutter | Cutting capillary columns | 5181–8836 |

## Ferrules for packed metal columns

Table 9 lists some of the nuts and ferrules used with packed metal columns. See the Agilent catalog for consumables and supplies for a more complete listing.

**Table 9. Nuts and Ferrules for Packed Metal Columns**

| Item* | Typical use | Part no. |
|---|---|---|
| 1/4-in. swage stainless steel, pkg of 20 (nut, front ferrule, back ferrule) | 1/4-in. | 5080-8753 |
| 1/8-in. swage stainless steel, pkg of 20 (nut, front ferrule, back ferrule) | 1/8-in. | 5080-8751 |
| 1/4-in. swage brass, pkg of 20 each (nut, front ferrule, back ferrule) | 1/4-in. | 5080-8752 |
| 1/8-in. swage brass, pkg of 20 each (nut, front ferrule, back ferrule) | 1/8-in. | 5080-8750 |
| 1/4-in. graphitized Vespel ferrule, pkg of 10 | Inlet/detector liner/adapters 1/4-in. columns | 5080-8774 |
| 1/8-in. graphitized Vespel ferrule, pkg of 10 | 1/8-in. columns | 0100-1107 |

* O-ring and ferrule ids

Ferrules that are prepared improperly cause leaks and contamination. Here are some hints to avoid problems.

### Graphite and graphitized-Vespel ferrules

Place these ferrules in a petri dish and bake in the GC oven at 250 to 300°C for 30 minutes before use to remove organic compounds absorbed by the graphite. Leave a petri dish of assorted ferrules in the GC oven to ensure a clean supply.

### Vespel ferrules

These ferrules can be more leak-tight than graphite, but have a lower temperature limit. They should be retightened after a few oven temperature cycles to ensure a good seal. Be sure to use the correct ferrule for the size column you are using.

## Ferrules and O-rings for glass packed columns

Table 10 lists some of the nuts and ferrules used with packed glass columns. See the Agilent catalog for consumables and supplies for a more complete listing.

Ferrules that are prepared improperly cause leaks and contamination. To avoid problems, place graphitized Vespel ferrules in a petri dish and bake in the GC oven at 250 to 300°C for 30 minutes before use to remove organic compounds absorbed by the graphite. Leave a petri dish of assorted ferrules in the GC oven to ensure a clean supply.

**Table 10. Glass Packed Columns Consumables**

| Item* | Typical use | Part no. |
|---|---|---|
| 1/4-in. graphitized Vespel ferrule, pkg of 10 | Inlet/Detector liners, 1/4-in. glass packed columns | 5080-8774 |
| Silicone O-ring, 6.0-mm | 1/4-in. glass packed columns | 0905-0322 |

* O-ring and ferrules ids

## Conditioning capillary columns

Conditioning involves establishing a flow of carrier gas through a column and then heating it for 1/2 hour to drive off contaminants.

**Warning** Do not use hydrogen as the carrier for conditioning! It vents into the oven and could create an explosion hazard.

1. Turn off the detectors. Shut off the detector support gases. Shut off hydrogen!
2. Install the proper liner in the inlet and attach the column in the normal manner.
3. Do not connect the column to the detector. Cap the detector fitting with a no-hole ferrule and column nut.
4. Select an appropriate column pressure from Table 11.

**Table 11. Recommended Gas Pressures for Conditioning Capillary Columns**

| | Recommended gas pressure, psi (kPa) | | | | |
|---|---|---|---|---|---|
| | Inside diameter | | | | |
| Length, m | 0.10 mm | 0.20 mm | 0.25 mm | 0.32 mm | 0.53 mm |
| 10 | 25 (170) | 6 (40) | 3.7 (26) | 2.3 (16) | 0.9 (6.4) |
| 15 | 39 (270) | 9 (61) | 5.6 (39) | 3.4 (24) | 1.4 (9.7) |
| 25 | 68 (470) | 15 (104) | 9.5 (65) | 5.7 (40) | 2.3 (16) |
| 30 | 83 (570) | 18 (126) | 12 (80) | 7 (48) | 2.8 (19) |
| 50 | | 32 (220) | 20 (135) | 12 (81) | 4.7 (32) |
| 60 | | 39 (267) | 24 (164) | 14 (98) | 5.6 (39) |

5. Enter the selected pressure. Let gas flow through the column at room temperature for 15 to 30 minutes to remove air.
6. Program the oven to heat from room temperature to the maximum temperature for the analysis. Do not exceed the temperature limit for the column. Increase the temperature at a rate of 10°C/min to 15°C/min and hold at the maximum temperature for 30 minutes.
7. If you will not be using the conditioned column immediately, remove it from the oven. Cap both ends to keep air, moisture, and other contaminants out.

## Conditioning packed columns

Conditioning involves establishing a flow of carrier gas through a column and then heating it overnight to drive off contaminants.

**Warning** Do not use hydrogen as the carrier for conditioning! It vents into the oven and could create an explosion hazard.

1. Turn off the detectors. Shut off the detector support gases. Shut off hydrogen!
2. Install the proper liner in the inlet and attach the column in the normal manner.
3. Do not connect the column to the detector. Cap the detector fitting with a no-hole ferrule and column nut.
4. Enter an appropriate column flow:
   - 20 to 30 mL/min for 2-mm id glass or 1/8-in. OD metal columns
   - 50 to 60 mL/min for 4-mm id glass or 1/4-in. OD metal columns
5. The conditioning temperature is never greater than the maximum temperature limit for the column; 30°C less than the maximum is usually sufficient. Slowly raise oven temperature to the conditioning temperature for the column.
6. Continue conditioning overnight at the final temperature. If you will not be using the conditioned column immediately, remove it from the oven. After removing the column, cap both ends to prevent air, moisture, or other contaminants from entering the column.

## Conditioning chemical traps

Preconditioned traps can be used immediately. All traps need periodic reconditioning, refilling, or replacement, usually after using one to four cylinders of gas or if gases of the highest purity were not used. Follow the manufacturer's instructions shipped with each trap. See Table 12.

**Table 12. Ordering Information for Agilent Traps**

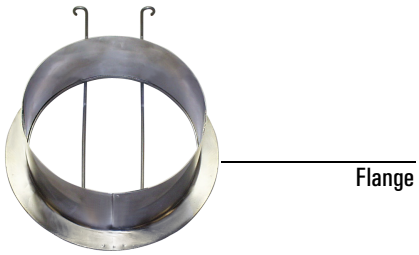
| Item | Part no. |
|---|---|
| Moisture trap (packed with Molecular Sieve 5A, 45/60 mesh) | 5060–9077 |
| Refillable Hydro-Moisture Trap 1/8-inch fitting | HMT200-2 |
| Absorbant Refill (1 pint) | HCRMS |
| Cap for ends of traps, 1/8-inch., 6 per package | 5180–4124 |
| Reducer trap fittings | 5062–3502 |

For the most complete listing of gas purification traps and systems, refer to the current Agilent catalog for consumables and supplies, or browse the Agilent online store at www.agilent.com/chem.

## Installing capillary columns

A correctly installed capillary column has the label to the front for easy reading, smooth connections with a minimum of slack to the inlet and detector, and no stray loops of tubing that might get caught in the oven or lid machinery.

1. Place the column on the hanger with the ends pointing up. If cryogenic oven cooling is to be used, be sure that the column hanger is the type with the flange.

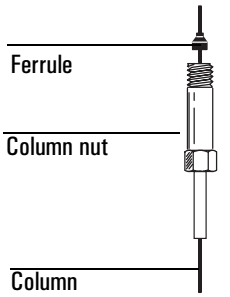



2. Connect the column to the detector as described on the next few pages. If necessary, unwrap one turn of the column.
3. Rotate the column clockwise on the hanger to remove slack on the detector side.
4. Place the column nut and ferrule on the inlet end. Pull excess column through the nut and ferrule to eliminate slack on the inlet side.
5. Cut off excess length and connect the column to the inlet.
6. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool and then retighten the fittings.

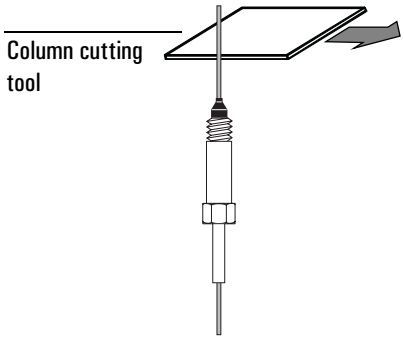
## Cutting column ends

**Warning** Wear safety glasses while handling, cutting, or installing glass or fused silica capillary columns. Use care in handling these columns to prevent puncture wounds.

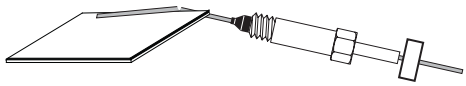
1. Place the appropriate nut and ferrule on the column.



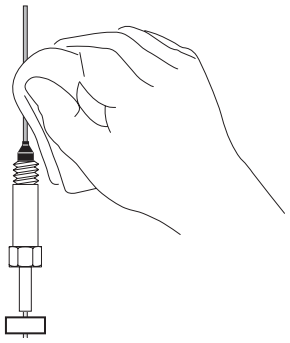

2. Score the column using a column cutting tool. The score must be square to ensure a clean break.




3. Support the column end against the column cutter opposite the scribe and break off the end. Inspect the end with a magnifying glass to make certain there are no burrs or jagged edges.



4. Wipe the column walls with a tissue dampened with isopropanol to remove fingerprints and dust.

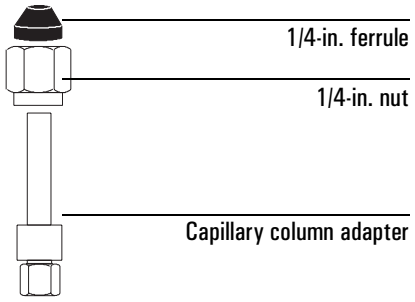


## Installing columns in the FID detector

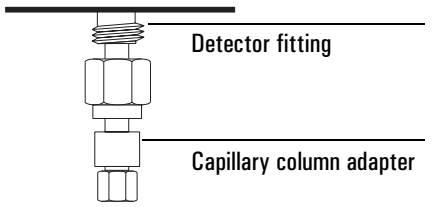
Be sure you have the correct jet installed in your detector before installing a column.

If the capillary column adapter is not installed on the detector, begin at step 1. If it is installed, begin at step 5.

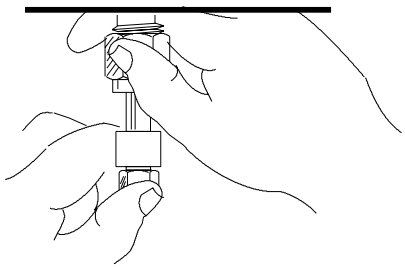
1. Assemble a brass nut and graphitized Vespel ferrule on the liner/adapter.



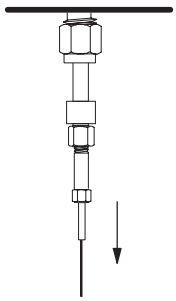

2. Insert the adapter straight into the detector base as far as possible.



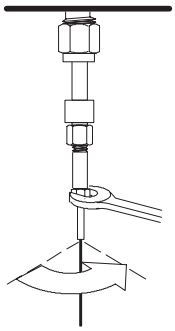
3. Hold the adapter in this position and tighten the nut finger tight. Tighten 1/4 turn more with a wrench.



4. Place the ferrule and column nut on the column end. Trim off a short piece to remove any ferrule fragments inside the column.
5. Gently insert the column into the detector until it bottoms; do not attempt to force it.
6. Tighten the column nut finger tight. Withdraw the column about 1 mm.

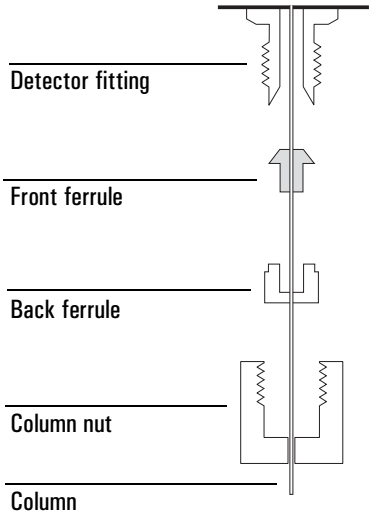


7. Use a wrench to tighten the nut an additional 1/4 turn.



## Installing capillary columns in the TCD detector

1. Assemble the ferrules and a 1/8-inch brass nut on the column as shown.




   See Table 13 for the proper ferrules. Trim off a short piece of column to remove any ferrule fragments inside the column.

2. Insert the column into the detector until it bottoms. Do not attempt to force it.
3. Slide the column nut and ferrule up the column to the detector and tighten the nut finger tight.
4. Pull the column out 1 mm. Use a wrench to tighten the nut an additional 1/4 turn. The column should not move.

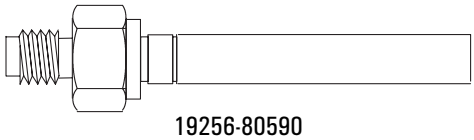
**Table 13. Ferrules for the TCD Detector**

| Column outside diameter | Back ferrule | Front ferrule |
|---|---|---|
| 0.8 mm | G1530–80400 | G1530–80410 |
| 0.53 mm | G1530–80400 | G1530–80420 |
| 0.45 mm | G1530–80400 | G1530–80430 |
| No-hole ferrule | G1530–80400 | G1530–80440 |

## Installing capillary columns in the FPD

The FPD uses an adaptable fitting that can use both packed and capillary columns. If your adaptable fitting does not have a capillary adapter installed, begin with step 1. If the capillary adapter is already installed in your adaptable fitting, begin with step 5.

The FPD uses a special adapter for capillary columns. The FPD Capillary Adapter, part number 19256-80570, allows fused silica columns as large as 530 µm id to be run right to the base of the FPD flame, minimizing sample tailing or loss of chemically active sites.



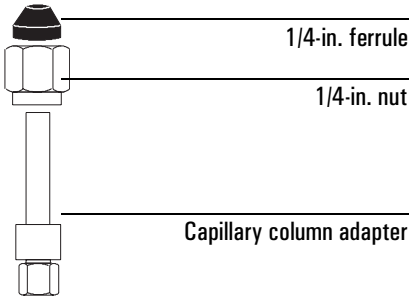
19256-80590
FPD 1/8-inch adapter

19256-80570
FPD Capillary Adapter

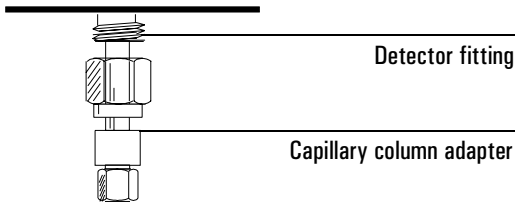
### Materials required

Column nut and ferrule
FPD Capillary column adapter
1/4-inch nut and ferrule
Column cutter
1/4-inch wrench
9/16-inch wrench
Metric ruler
Typewriter correction fluid

1. Assemble a brass nut and graphite/Vespel ferrule onto the adapter.



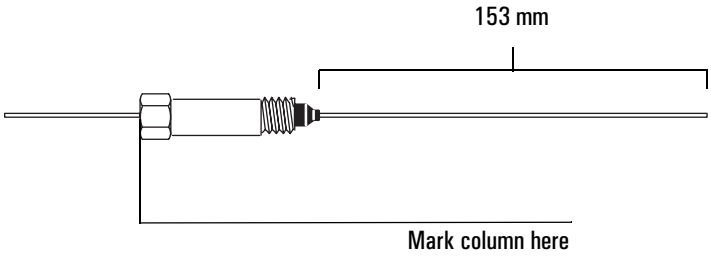
2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight. Use a wrench to tighten the nut an additional 1/4 turn.




3. Install a column nut (part no. 18740-20870) and graphite ferrule (1.0 mm id, part no. 5080-8773 or 0.5 mm id, part no. 5080-8853) on the column.
4. After installing the nut and ferrule, prepare a fresh column end by cutting off a short piece of the column. See page 28 for instructions.
5. Position the ferrule about 153 mm from the end of the column.

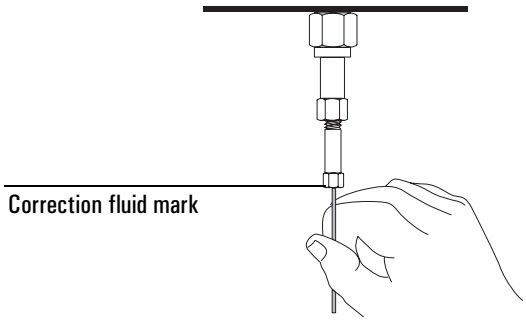
   Optimum height depends on sample type and gas flow rates. If it is too high, the column end will be exposed to the flame. If too low, the sample may be exposed to hot stainless steel, causing slight tailing.

   Mark the column at a point even with the bottom of the nut. Typewriter correction fluid works well.




6. Insert the column in the detector. Slide the nut and ferrule up the column to the detector base. Finger tighten the column nut until it starts to grab the column.

7. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut.




8. Tighten the column nut finger tight, then withdraw the column about 1 mm. Use a wrench to tighten the nut an additional 1/4 turn.
9. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool and then retighten the fittings.

## Installing capillary columns in a µECD

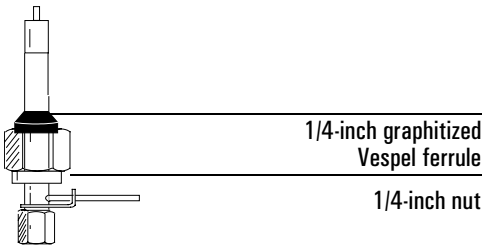
The detector is shipped with a capillary column adapter installed. If it has been removed, you must replace it before installing a capillary column.

The µECD requires the indented liner, which is necked down near one end and is clear.

### Materials required

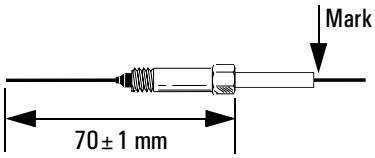
Capillary column adapter
Fused silica liner, indented
1/4-inch nut and 1/4-inch graphitized Vespel ferrule
Column nut and ferrule
Column cutter
1/4-inch and 9/16-inch wrenches

1. Install a 1/4-inch nut and graphitized-Vespel ferrule on the adapter.

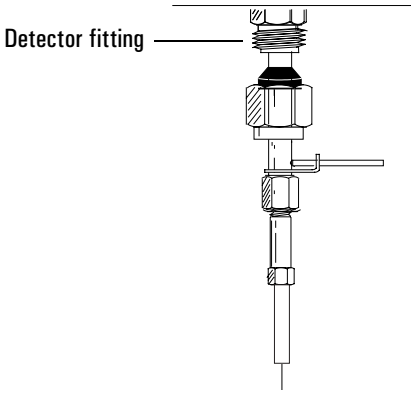


2. Prepare the column. See "Installing capillary columns" on page 27 for instructions.
3. **If the column id is 200 µm or more**, push the column into the adapter until it stops at the indentation. Pull it back 1 to 2 mm and tighten the column nut firmly.

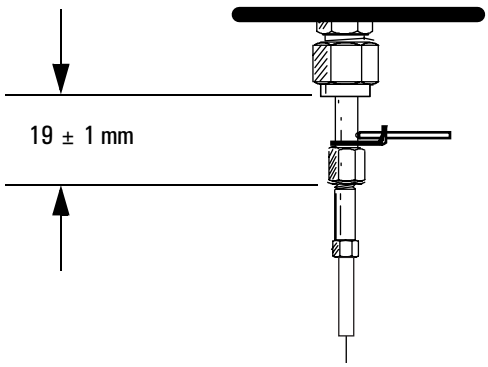
   **If the id is less than 200 µm**, mark the column with a septum 70±1 mm from the end. Insert column and nut into the adapter with the septum at the rear of the column nut, and tighten the column nut firmly.




4. Slowly install the adapter straight into the detector fitting. Make sure that the adapter is seated all the way into the detector fitting—jiggle it if necessary. Be careful not to break the column end.




   If the adapter is properly installed, the distance between the 1/4-inch nut and the bottom of the adapter will be 19 ± 1 mm. If it is 22 to 23 mm, reinstall the adapter into the detector fitting.



5. Slide the nut and ferrule up to the detector fitting and tighten the nut finger tight. Use a 9/16-inch wrench to tighten the nut an additional 1/4 turn.

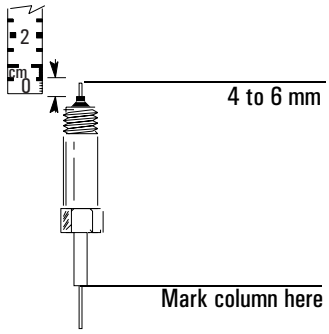
**Caution** Set the oven maximum temperature below the column limit to prevent accidental overheating of the column.

6. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

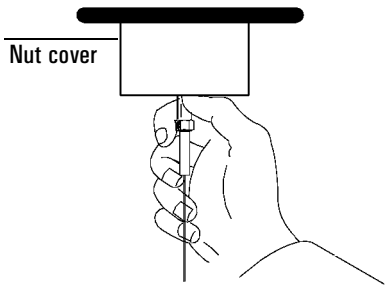
## Installing columns in the split/splitless inlet

Before installing the column, be sure you have the correct glass liner installed.

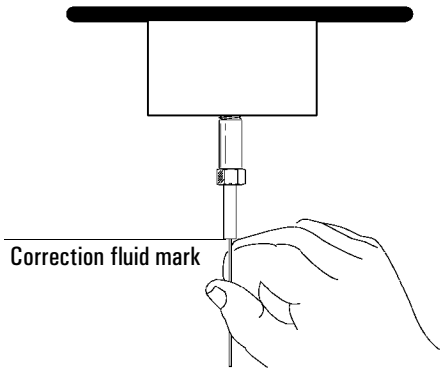
1. Place the ferrule and column nut on the column end. Trim off a short piece to remove any ferrule fragments inside the column.
2. Position the column so it extends 4 to 6 mm above the end of the ferrule. Mark the column with typewriter correction or other marking fluid at a point even with the bottom of the column nut.



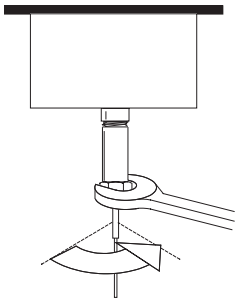

3. Insert the column in the inlet and slide the nut and ferrule up the column to the inlet base. Finger tighten the column nut until it starts to grip the column.



4. Adjust the column position so that the mark on the column is even with the bottom of the column nut.




5. Tighten the column nut an additional 1/4 to 1/2 turn beyond finger tight so that the column cannot be pulled from the fitting with gentle pressure.

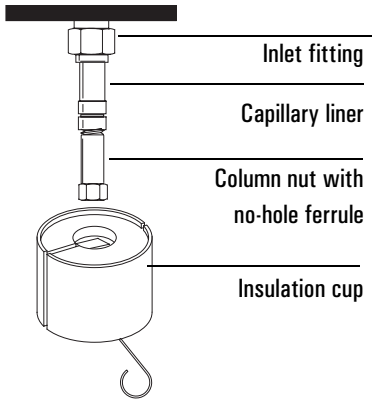



## Installing columns in the purged packed inlet

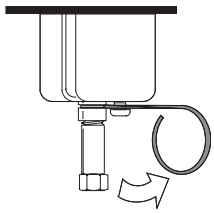
Agilent Technologies recommends that you avoid using capillary PLOT columns with a purged packed inlet with a gas sampling valve unless you plumb the valve directly to the column, bypassing the inlet.

Before installing a column in this inlet, be sure you have a capillary liner and glass insert installed. See "Installing a liner" on page 137 and "Installing a glass insert" on page 138. If your insulation cup is not installed, begin with Step 1. Otherwise, begin with Step 4.

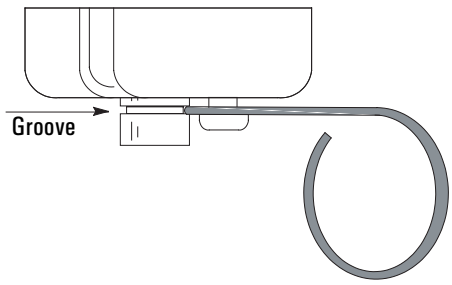
1. Install a plug (for example, a no-hole ferrule) in the inlet fitting.




2. Install the insulation cup, if needed. Push the cup spring to the right. Slide the cup over the inlet fitting so that the insulation at the top of the cup is flush against the oven roof.

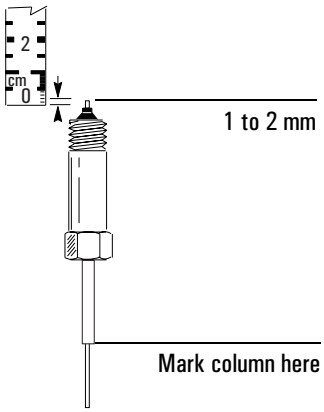


3. Place the spring into the groove in the inlet liner. Remove the column nut and put the no-hole ferrule aside.

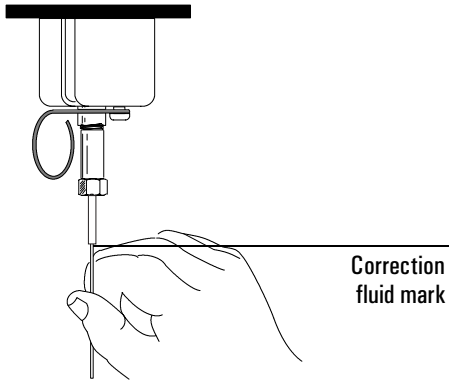



4. Place the ferrule and column nut on the column end. Trim off a short piece to remove any ferrule fragments inside the column.

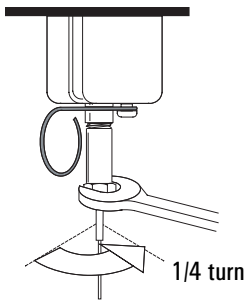
5. Position the column so it extends above the end of the column nut by 1 to 2 mm. Mark the column with typewriter correction or other marking fluid at a point even with the column nut.




6. Push the column up 1 cm and guide it into the inlet liner. Slide the nut and ferrule up the column to the inlet liner. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut. Finger tighten the column nut until it starts to grab the column.



7. Tighten the column nut an additional 1/4 to 1/2 turn so that the column cannot be pulled from the fitting when gentle pressure is applied.




8. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow these to cool, and then retighten the fittings.

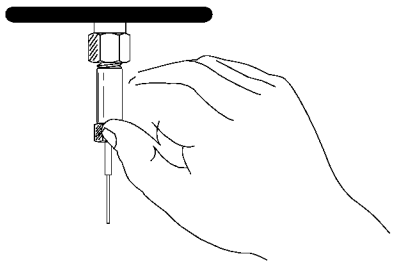
## Installing columns in the cool on-column inlet

Before installing the column, be certain you have the correct hardware installed for the column and type of injection you are doing. See "Maintaining a cool on-column inlet" on page 166 for detailed information.

### Materials required

Column nut and ferrule
Column cutter
1/4-inch wrench

1. Prepare the column. See page page 27 for instructions.
2. Gently insert the column into the inlet until it bottoms. Insert the column nut into the inlet fitting and tighten the nut finger tight.



3. Tighten an additional 1/4-turn with a wrench or until the column does not move.

4. If you are using an automatic injection system with 250 µm or 320 µm columns, verify the installation by pushing the syringe manually into the inlet.
5. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

### Installing columns in the PTV inlet

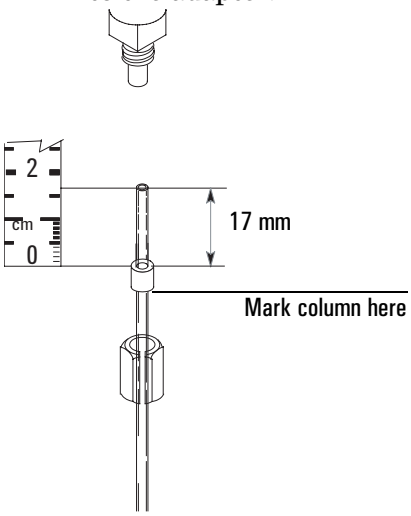
1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off.
2. Install the appropriate column adapter. See "Inlet adapters" on page 150.
3. Select the appropriate GRAPHPACK™-2M ferrule. These ferrules are sized to the column outer diameter.

**Table 14. Columns and Ferrules**

| Column id | GRAPHPACK ferrule hole id | Quantity | Part no. |
|---|---|---|---|
| 200 µm | 0.31 mm | 10 | 5182-9756 |
| 250 µm | 0.40 mm | 10 | 5182-9768 |
| 320 µm | 0.45 mm | 10 | 5182-9769 |
| 530 µm | 0.70 mm | 10 | 5182-9770 |

4. Place the appropriate GRAPHPACK ferrule onto the column inlet end and pull it at least 30 mm from the end.
5. With a glass knife or other fused silica cutter, remove approximately 10 mm from the column end to eliminate graphite contamination.

6. Position the ferrule so that it is 17 mm from the column end. Place a small mark (typewriter correction fluid is useful) at the back of the ferrule and, making sure that the column is correctly positioned, insert the column end into the adapter.




7. Screw the column nut on finger tight. Using a 5-mm wrench, tighten the column nut 1/8- to 1/4-turn. Be careful not to overtighten.
8. Check the connections for leaks. If there are any leaks at the column adapter, tighten it slightly more with the open end wrench provided.

## Installing packed metal columns

There are two sizes of packed metal columns, 1/4-in. and 1/8-in., in common use. This general procedure applies to both sizes of columns.

1. Install ferrules on your packed column (See "Installing ferrules on a metal column" on page 44).
2. If the capillary column hanger is installed, remove it. (See "Removing the capillary column hanger" on page 22.)
3. See Table 9 for fittings required. Install fittings as needed.

4. Install the adapter, if needed. See Table 15.

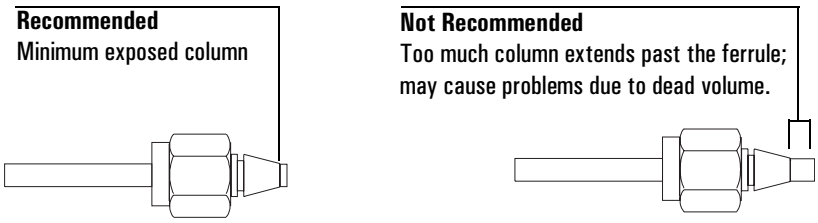
**Table 15. Fittings for 1/4-in. and 1/8-in. Packed Metal Columns**

| Inlet or detector | 1/4-in. Packed metal column | | 1/8-in. Packed metal column | |
|---|---|---|---|---|
| | **Where to install** | **Comments** | **Where to install** | **Comments** |
| Purged-packed inlet | 1/4-in. liner | See "Installing a liner" on page 137 | 1/8-in. liner | See "Installing a liner" on page 137 |
| FID | 1/4-in. adapter (part no. 19231-80530) | Remove or install adapter, as desired. | 1/8-in. adapter (part no. 19231-80520) | See "Installing an adapter in a detector fitting" on page 47 |
| TCD | 1/4-in. adapter (part no. G1532-20710) | See "Installing an adapter in a detector fitting" on page 47 | Detector fitting | Remove adapter, if necessary. |

5. Install the column. (See "Installing packed metal columns" on page 48.)
6. Establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.
7. Condition the column, if needed. (See "Conditioning packed columns" on page 26.)

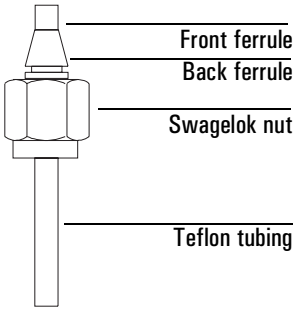
## Installing ferrules on a metal column

Before installing packed metal columns, a ferrule should be locked on the column end so that it is flush with the end of the column. This prevents problems caused by dead volume in the fitting.



Use the following instructions to install new SWAGELOK® nuts and ferrules onto 1/8-in. or 1/4-in. metal columns.

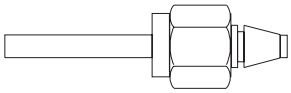
1. Install new SWAGELOK nut and ferrules on the column.




2. If needed, make a Teflon spacer sized for your column. (See "Make a spacer from Teflon tubing" on page 45.)
3. Install the Teflon tubing spacer in the male fitting. Fully insert the column with its nut and ferrules into the vise-held fitting. Tighten the nut finger tight.

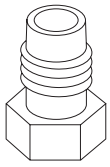
   Use a wrench to tighten the nut an additional 1-1/4 turn for 1/4-in. columns or 3/4 turn for 1/8-in. columns.\

   Unscrew the column nut from the vise-held fitting and remove the column. Ferrules should now be set in place on the column with the column correctly positioned.



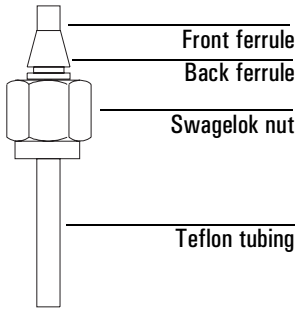
## Make a spacer from Teflon tubing

1. Secure a new male Swagelok fitting in a bench vise.

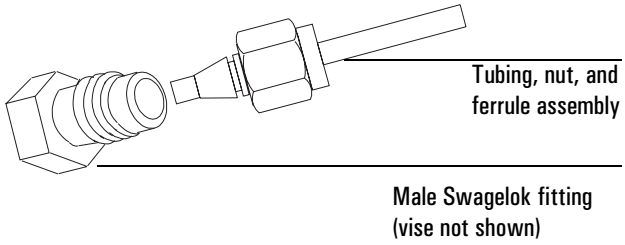


Male Swagelok fitting

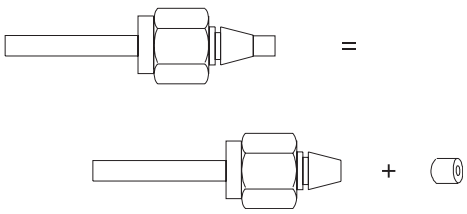
2. Slide a Swagelok nut, back ferrule, and front ferrule onto a piece of 1/4-in. or 1/8-in.Teflon tubing. (Select the tubing to match your column.) If the end of the tubing is not cut straight, use a razor or sharp knife to make a flat, smooth end.



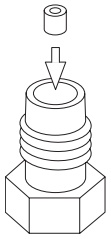

3. Insert the Teflon tubing, 1/4-in. or 1/8-in. ferrules, and matching nut into the vise-held Swagelok fitting. Tighten the nut 3/4 turn past finger tight to set the ferrules on the tubing.




4. Loosen the nut and remove the assembly from the male Swagelok fitting.
5. Cut off the end of the tubing extending beyond the ferrule with a razor or sharp knife. This piece of tubing is the spacer.



6. Insert the spacer into the vise-held Swagelok fitting.

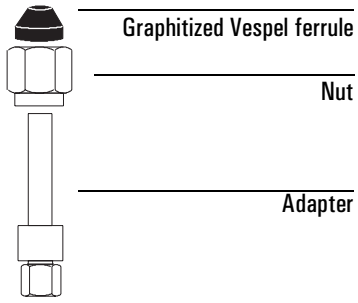


7. The male Swagelok fitting and spacer should be kept on hand to be used whenever new ferrules are being installed on a column.

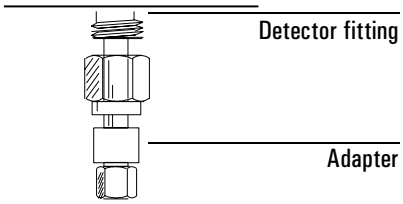
### Installing an adapter in a detector fitting

This is a general procedure for installing many types of adapters onto detector fittings. See Table 15 for adapter part numbers.

1. Assemble a brass nut and a graphitized Vespel ferrule onto the adapter.




2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight.
   - 1/4-in. column: tighten an additional 3/4 turn with a 9/16-in. wrench
   - 1/8-in. column: tighten an additional 1/4 turn with a 7/16-in. wrench

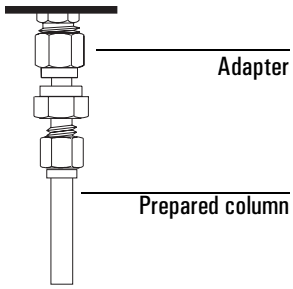



3. Install your column. See “Installing packed metal columns” on page 48.

## Installing packed metal columns

Before following this procedure, make sure an adapter or liner is installed (see Table 15 on page 44), if needed, and that your column is prepared (see "Installing ferrules on a metal column" on page 44).

1. Insert the column into the adapter, detector, or inlet liner until it bottoms. Tighten the nut finger tight.




2. If you are installing a column directly into the detector fitting:
   - 1/4-in. column: tighten an additional 3/4 turn with a 9/16-in. wrench
   - 1/8-in. column: tighten an additional 1/4 turn with a 7/16-in. wrench
3. If you are installing a column onto an adapter:

   Tighten the column nut using two wrenches in opposition, one on the column nut and the other on the liner or adapter body. This prevents the liner or adapter from rotating while you tighten the column nut.
   - 1/4-in. column: tighten an additional 3/4 turn with a 9/16-in. wrench
   - 1/8-in. column: tighten an additional 1/4 turn with a 7/16-in. wrench
4. Establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.
5. Condition the column, if needed. (See "Conditioning packed columns" on page 26.)

## Installing packed glass columns

Glass packed columns must be installed simultaneously at the inlet and the detector.

You can install glass packed columns directly in the purged-packed inlet and adaptable FID fittings. The TCD requires an adapter.

There are three types of glass packed columns available. You must make certain that your column is compatible with the inlet fitting and detector used. Table 16 summarizes the inlet and detector fittings required and the appropriate column configuration.

The general procedure is:

1. If the capillary column hanger is installed, remove it. (See "Removing the capillary column hanger" on page 22.)
2. See Table 16 for information on fittings and column configuration required.
3. Remove or install an adapter, if necessary. (See "Installing an adapter in a detector fitting" on page 47.)
4. Install the glass column. (See "Installing glass packed columns" on page 50.)
5. Condition the column, if needed. (See "Conditioning packed columns" on page 26.)

**Table 16. Fittings Required for Glass Packed Columns**

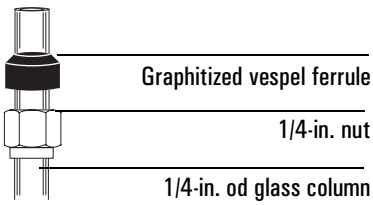
| Inlet or detector | Where to install | Comments |
|---|---|---|
| Purged packed inlet | Inlet fitting (no liner installed), **or** 1/4-in. liner. See "Installing a liner" on page 137 | Allow at least 50 mm of empty column to prevent an inserted syringe needle from contacting either the glass wool plug or column packing. |
| FID (adaptable) | Detector fitting | Remove adapter, if installed.<br>There must be at least 40 mm of empty column to prevent the bottom end of the jet from touching either column packing or the glass wool plug. |
| TCD | 1/4-in. adapter (part no. G1532-20710) | See "Installing an adapter in a detector fitting" on page 47 |

## Installing glass packed columns

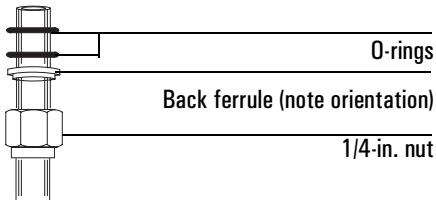
1. Assemble a brass nut and graphitized Vespel ferrule on each end of the column.

   Alternative method: Install a 1/4-in. nut, back ferrule, and two O-rings on each end of the column. An extra O-ring below the nut keeps the nut from dropping into the coiled portion of the column.

**Recommended**




**Alternative installation method**




2. Insert the column into the inlet until it bottoms. Insert the column into the detector fitting but do not force it. It may be necessary to start the long end of the column in the inlet at an angle.
3. Withdraw the column 1 to 2 mm from both the inlet and detector. Tighten both column nuts finger tight.

**Caution** Overtightening the column nut or forcing it to bottom in both the inlet and detector may shatter the column.

4. Tighten both column nuts 1/4 turn with a wrench. If you use graphitized Vespel ferrules, proceed to step 5. If you use O-rings, proceed to step 6.
5. Set flow through the column and raise the inlet, detector, and oven to operating temperature. Then set the oven to ambient and allow it to cool.
6. Use the wrench to tighten the nut an additional 1/2 turn. Tighten further as necessary to prevent leakage.
7. Condition your column, if needed. (See "Conditioning packed columns" on page 26.)

# Daily Operation

## To start up the GC

1. Open the oven lid and verify that the correct column is installed.
2. Turn the carrier and detector gases on at their sources.
3. If cryogenic cooling is used, turn the cryo coolant on.
4. If the oven has been cold for some time, the column fittings may have begun to leak. Check the fittings, tighten them if necessary, and close the oven.
5. Turn the GC power switch on. Wait for initialization to finish.
6. Load the correct method. Wait for the GC to become ready.
7. When doing trace analysis with temperature programming, make a blank run (no sample injected) to flush out materials from the carrier gas that have accumulated on the column.
8. Begin running analyses.

## To make a run

Load the desired method.

### Manual syringe injection

1. When the "Ready for manual inj" message appears, insert the syringe needle through the septum as far as it will go, press the plunger down quickly, press `Start`, and withdraw the syringe immediately.
2. If the Not Ready light stays on, examine the GC display for a message. It may be necessary to press the `Prep Run` key. If so, a message will say so. Press the key, wait for the Not Ready light to turn off, then make the injection and press `Start`.

### Auto injector without a sequence

1. Load the sample into a sample vial. Place the sample vial in position 1 of the injector turret. The Not Ready light can be on at this time. The "Ready for autoinject" message appears when the GC is ready, but it is not necessary to wait for this.

2. Press `Start`. When the GC is ready, the injector begins its cycle, makes 1 injection from the vial in position 1, and issues an internal `Start` command at the moment of injection.

### Auto injector with a sequence

1. Load the samples into sample vials. Place the vials in successive positions of the injector turret. The Not Ready light can be on at this time. The "Ready for sequence" message appears when the GC is ready, but it is not necessary to wait for it.
2. Set up the injector sequence. See the Control Module or ChemStation/ Cerity Chemical documentation for details.
3. Press `Start`. When the GC is ready, the injector performs the analyses as directed by the sequence.

Sometimes you will want to change the number of samples in the sequence. For example, the sequence is set up to run 10 samples but you only have 8 to do. To change the Last Vial parameter in the sequence from the GC panel:

1. Press `Load`. The Last Vial number is displayed.
2. Use the scroll keys (▲ and ▼) to change the number.
3. Press `Load`.

### Sample valve with a sequence

1. Set up the injector sequence. See the Control Module or ChemStation/ Cerity Chemical documentation for details.
2. The sample valve is in the Load state between runs. The Not Ready light may be on during this period. Either connect a sample stream to the valve for continuous flushing or use a gas syringe to flush the loop.
3. When the "Ready for sequence" message is displayed, press `Start`. The valve changes to the Inject state and, after `Inject min` elapses, returns to the Load state.

### Sample valve with a run table

1. Create a Run Table entry to turn the valve ON shortly (0.01 min) after the start of a run. Add a second entry to turn the valve OFF after enough time has elapsed to sweep the sample on to the column.
2. Either connect a sample stream to the valve for continuous flushing or use a gas syringe to flush the loop.
3. Press `Start`.

### Multi Valve with sample valve and a sequence

1. The sample valve is in the Load state between runs. The Not Ready light is on during this period. Connect the sample streams to the Multi Valve.
2. Set up the valve sequence. See the Control Module or ChemStation/Cerity Chemical documentation for details.
3. The "Ready for sequence" message appears when the GC is ready, but it is not necessary to wait for it. Press `Start`. The Multi Valve steps through the range of positions specified by the sequence, and the sample valve injects each one into the GC.

## To shut down the GC

### For a short period (including weekends)

1. Wait for the present run to finish.
2. If the active method has been modified since it was loaded, there will be a + symbol following its name. To save these changes, use a Control Module or ChemStation/Cerity Chemical.
3. Leave the main power switch on. This keeps the heated zones at their setpoint temperatures so that the GC will be ready for use when needed.
4. Turn off all gases, except the carrier, at their sources. Leave the carrier on to protect the column from atmospheric contamination.
5. If cryogenic cooling is used, you may want to turn the cyro coolant off.
6. This is an excellent time to replace the septum (see "Changing septa" on page 121).

### For a long period

1. Perform all the steps above.
2. Turn the carrier gas off at the source.
3. Turn the main power switch off.
4. Allow time for the oven to cool.
5. Remove the column from the oven. Cap both ends to keep contaminants out.

# Verifying Performance

This section contains Standard Operating Procedures along with typical examples of test sample chromatograms.

Note that injection volumes listed do not necessarily indicate the absolute volume injected. The volume reflects the graduation (plunger position) read from a standard 10 µL syringe. For a heated inlet, the actual sample volume injected may include an additional 0.4 to 0.7 µL, the volume of sample volatilized from inside the syringe needle.

Note that the following procedures and results are intended to provide evidence of a properly functioning inlet and/or detector system.

## Software required

The procedures in this section require use of a ChemStation. They are written for manual syringe injection.

If you are using Cerity Chemical, you can perform the chemical checkout for each detector by loading the specified method and running the checkout sample. Cerity Chemical cannot perform the noise test.

If a component is not yet supported on your data system version, you can enter the appropriate chemical checkout settings using a control module.

## TCD Checkout

### Scope

Use this procedure to verify proper TCD operation with the split/splitless or purged packed capillary column inlet.

## Parts and equipment required

**All inlet types**

6850 evaluation column, 30 m × 0.320 mm × 0.25 mm HP-1, part no. 19091Z–413Et

FID/TCD performance evaluation (checkout) sample, part no. 18710–60170

Chromatographic-grade helium as carrier, makeup, and reference

**Split/splitless and purged packed inlets**

10 µL syringe, part no. 9301–0810 or equivalent

O-ring, part no. 5180–4182 (split/splitless) or 5080–8898 (purged packed)

Septum, part no. 5181–1263

**Cool on-column inlet**

With septum nut:

- Needle, part no. 5182-0831, for manual syringe
- Septum nut, part no. 19245-80521

With duckbill septum

- Needle, fused silica, part no. 19091-63000
- Syringe, for fused silica needle, part no. 9301-0658
- Septum, part no. 19245-40050

**PTV inlet**

10 µL syringe, part no. 9301–0810 or equivalent

Inlet adapter, GRAPHPACK-2M, part no. 5182-9761

Silver seal, for GRAPHPACK-2M, part no. 5182-9763

Glass liner, multibaffle, part no. 5183-2037

Teflon ferrule, part no. 5182-9748 (septumless head)

Microseal replacement, part no. 5182-3444 (if installed)

GRAPHPACK 3D ferrule, part no. 5182-9749

### GC setup

**Warning** If the unit has been in operation, areas may be hot enough to cause serious burns. Switch off the heated zones and the oven and allow sufficient time for the unit to cool.

1. If you are using a split/splitless inlet, install a new septum, a new split liner, and a new O-ring seal in the inlet. See "To install a liner and O-ring" on page 120.

   If you are using a purged packed inlet, install a new liner and glass insert for the checkout column. Also install a new septum and O-ring. See "Changing septa" on page 121 and "Changing the O-ring" on page 142.

   If you are using a cool on-column inlet, install a new septum, then check for a proper fit of the needle in the column. See "Changing septa" on page 121 and "Cleaning the inlet" on page 131.

   If you are using a PTV inlet with the septum head, install a new septum or microseal, inlet adapter, silver seal, and glass liner. See "Changing septa" on page 121, "Replacing liners" on page 157, and "To replace the inlet adapter" on page 150.

   If you are using a PTV inlet with the septumless head, clean the head and replace the Teflon ferrule and glass liner. See "Cleaning the septumless head" on page 152, "Replacing the Teflon ferrule" on page 154, and "Replacing liners" on page 157.
2. Install the 6850 evaluation column.
3. If present, remove the protective cap from the TCD exhaust vent at the top of the detector and the caps on the inlet manifold vent(s).

### ChemStation setup

1. Bring up the proper on-line instrument for the 6850 to be tested. Make these settings:

| Path | Setting |
|---|---|
| View/Run Method/Control View/Instrument/ Edit Parameters/Options | Keep instrument keyboard locked after method is loaded? = No |
| View/Run Method/Control View/Instrument/ Edit Parameters/Signals | Assign Signal 1 to the detector |
| | Choose Save Data All |
| | Data rate = 5 Hz |

2. Enter the parameter values listed in Table 17.

3. Define the column.
4. Save the method. We suggest using the name `TCDCKO`.
5. Make sure the GC is turned on. Wait until it is ready.

**Table 17. TCD Checkout Conditions (Method TCDCKO)**

| | |
|---|---|
| **Column and sample** | |
| Type | HP-1, 30 m × 0.32 mm × 0.25 μm part number 19091Z–413E |
| Sample | FID/TCD checkout 18710-60170 |
| Injection volume | 1 μL |
| **Inlets** | |
| Temperature | 250°C Purged packed and split/splitless<br>Oven Track (cool on-column)<br>40°C PTV (see below) |
| Inlet pressure | 25 psi (constant pressure, helium) |
| **Split/splitless** | |
| Mode | Splitless |
| Purge flow | 60 mL/min |
| Purge time | 0.75 min |
| **PTV** | |
| Mode | Splitless |
| Inlet temperature | 40°C |
| Initial time | 0.1 min |
| Rate 1 | 720°C/min |
| Final temp 1 | 350°C |
| Final time 1 | 2 min |
| Rate 2 | 100°C/min |
| Final temp 2 | 250°C |
| Final time 2 | 0 min |
| Purge time | 0.75 min |
| Purge flow | 60 mL/min |
| **Detector** | |
| Temperature | 300°C |
| Reference flow (He) | 20 mL/min |
| Makeup flow (He) | 2 mL/min |
| Offset | Should be < 30 display counts |

**Table 17. TCD Checkout Conditions (Method TCDCKO) (Continued)**

| | |
|---|---|
| **Oven** | |
| Initial temp | 40°C |
| Initial time | 0 min |
| Rate 1 | 25°C/min |
| Final temp | 90°C |
| Final time | 0 min |
| Rate 2 | 15°C/min |
| Final temp | 170°C |
| Final time | 2 min |

## Offset checkout

Display the signal output to determine background offset. A stable offset at any value between 0.5 and 30 display units (inclusive) is acceptable.

- If the offset is < 0.5 display units, verify that the detector filament is on. If the offset is still < 0.5 display units, your detector requires service.
- If offset is > 30 display units, there may be chemical contamination contributing to the signal. Perform a thermal cleanout (see "Thermal cleaning (bakeout)" on page 185 for details). If repeated cleanings do not give an acceptable signal, check gas purity. Use higher purity gases and/or install traps.

## Noise, wander and drift

1. Make these settings on the ChemStation:

| Path | Setting |
|---|---|
| View | Full menu |
| View/Data Analysis/Report/Specify Report | Performance + noise, OK |
| | System Suitability, Edit Noise Ranges, enter several ranges that > 1.01 min plus one at 5 minutes, OK |
| View/Method/Save Method As | Method name |
| Instrument Menu/Edit Parameters | Oven, verify oven temp = 40°C |
| | Initial time = 12 min, Rate 1 = 0°C/min, OK |
| View/Run Control/Sample Information | Enter directory and file names |

2. Press `Start` to make a blank run and wait until it is finished.

3. Select `View/ Data Analysis`. Load the data file for the blank run just completed.
4. Select `Report/ Specify Report`. Choose printer and screen.
5. Select `Integration/Integrate`.
6. After integration is completed, Select `Report/ Print Report`. The report will list measurements for noise (peak to peak, % standard deviation, and ASTM) and for drift and wander.
   - ASTM noise should be < 0.057 display units (25 µV/display unit for TCD).
   - Wander or drift (for a 5-minute time window) should be < 0.14 display units.

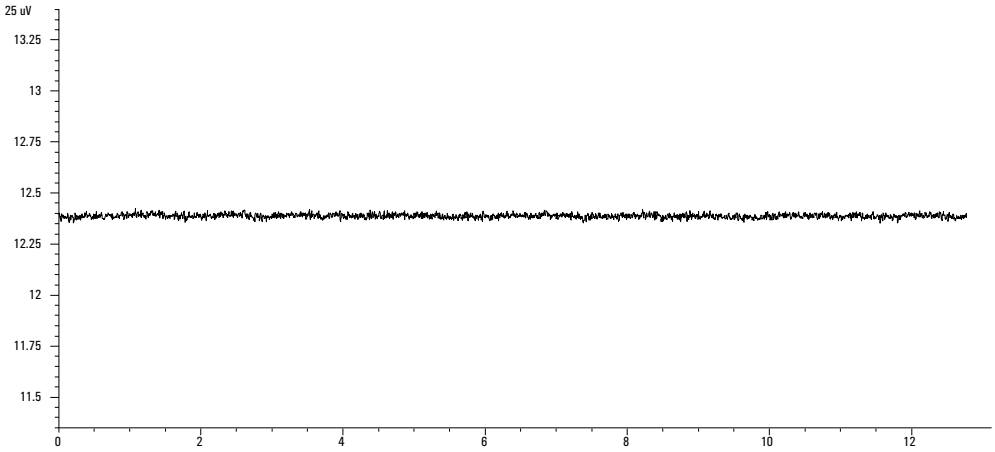



**Figure 6. Sample TCD noise, wander, and drift plot**

## Chemical checkout

1. Press `Prep Run` on the 6850 keypad to prepare the inlet for splitless injection. When the message "Ready for manual inj" appears, inject 1 µL of the checkout sample and press `Start` on the 6850.
2. The chromatogram should be similar to Figure 7. The acceptance criteria are:
   - Area counts for components labeled C14, C15, and C16 should each be > 73.
   - The area counts ratio calculated as C14/C16 should be 1.00 $\pm$ 0.10.
   - MDL (C16, pg/mL) = (1,272,000 × Noise) /(Area C16) ≤ 1,000.

3. If these criteria are not met, repeat the test. If after repeated testing the criteria cannot be met, consult your service and user documentation for additional information.

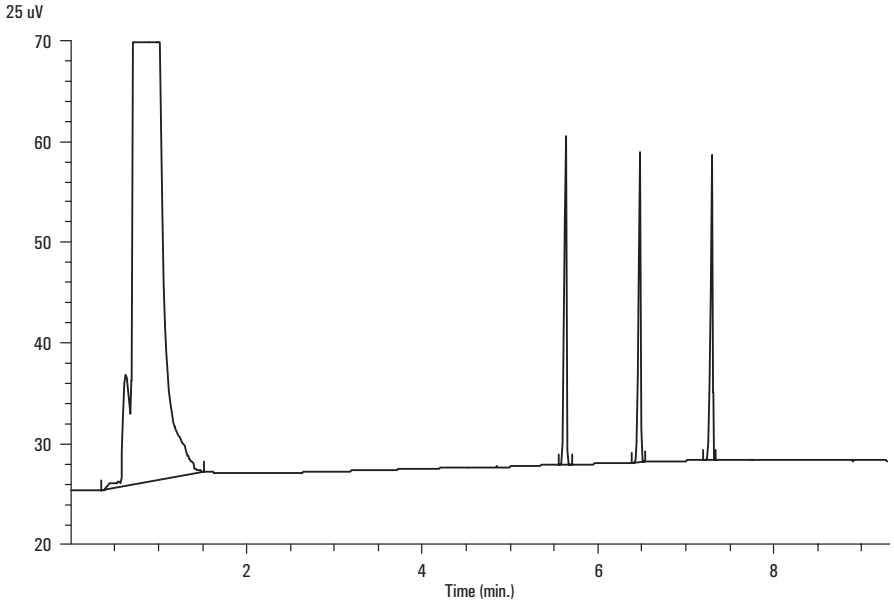



**Figure 7. Sample TCD checkout chromatogram**

# FID checkout

## Scope

Use the following procedure to verify proper FID operation.

## Parts and equipment required

**All inlet types**

6850 evaluation column, 30 m × 0.320 mm × 0.25 mm HP-1, part no. 19091Z–413E

FID/TCD performance evaluation (checkout) sample, part no. 18710–60170

Chromatographic-grade gases: helium, nitrogen, hydrogen, and air

**Split/splitless and purged packed inlets**

10 µL syringe, part no. 9301–0810 or equivalent

O-ring, part no. 5180–4182 (split/splitless) or 5080–8898 (purged packed)

Septum, part no. 5181–1263

**Cool on-column inlet**

With septum nut:

- Needle, part no. 5182-0831, for manual syringe
- Septum nut, part no. 19245-80521

With duckbill septum

- Needle, fused silica, part no. 19091-63000
- Syringe, for fused silica needle, part no. 9301-0658
- Septum, part no. 19245-40050

**PTV inlet**

10 µL syringe, part no. 9301–0810 or equivalent

Inlet adapter, GRAPHPACK-2M, part no. 5182-9761

Silver seal, for GRAPHPACK-2M, part no. 5182-9763

Glass liner, multibaffle, part no. 5183-2037

Teflon ferrule, part no. 5182-9748 (septumless head)

Microseal replacement, part no. 5182-3444 (if installed)

GRAPHPACK 3D ferrule, part no. 5182-9749

## GC setup

**Warning** If the unit has been in operation, areas may be hot enough to cause serious burns. Turn off the heated zones and the oven and allow sufficient time for the unit to cool.

1. If you are using a split/splitless inlet, install a new septum, a new splitless liner, and a new O-ring seal in the inlet. See "To install a liner and O-ring" on page 120.

   If you are using a purged packed inlet, install a new liner and glass insert for the checkout column. Also install a new septum and O-ring. See "Changing septa" on page 121 and "Changing the O-ring" on page 142.

   If you are using a cool on-column inlet, install a new septum, then check for a proper fit of the needle in the column. See "Changing septa" on page 121 and "Cleaning the inlet" on page 131.

   If you are using a PTV inlet with the septum head, install a new septum or microseal, inlet adapter, silver seal, and glass liner. See "Changing the septum" on page 156, "Replacing liners" on page 157, and "To replace the inlet adapter" on page 150.

   If you are using a PTV inlet with the septumless head, clean the head and replace the Teflon ferrule and glass liner. See "Cleaning the septumless head" on page 152, "Replacing the Teflon ferrule" on page 154, and "Replacing liners" on page 157.
2. Install a 0.28-mm (0.011-in.) jet and/or the capillary column adapter, if needed.
3. Install the 6850 evaluation column.
4. If present, remove the protective caps from the inlet manifold vents.

## ChemStation setup

1. Bring up the proper online instrument for the 6850 to be tested. Make these settings:

| Path | Setting |
|---|---|
| View/Run Method/Control View/Instrument/ Edit Parameters/Options | Keep instrument keyboard locked after method is loaded? = No |
| View/Run Method/Control View/Instrument/ Edit Parameters/Signals | Assign Signal 1 to the detector |
| | Choose Save Data All |
| | Data Rate = 20 Hz |

2. Enter the parameter values listed in Table 18.
3. Define the column.
4. Save the method. We suggest using the name `FIDCKO`.

**Table 18. FID Checkout Conditions (Method FIDCKO)**

| | |
|---|---|
| **Column and sample** | |
| Type | HP-1, 30 m × 0.32 mm × 0.25 μm part number 19091Z–413E |
| Sample | FID/TCD checkout 18710-60170 |
| Injection volume | 1 μL |
| **Inlets** | |
| Temperature | 250°C Purged packed and split/splitless<br>Oven Track (cool on-column)<br>40°C PTV (see below) |
| Inlet pressure | 25 psi (constant pressure, helium) |
| **Split/splitless** | |
| Mode | Splitless |
| Purge flow | 60 mL/min |
| Purge time | 0.75 min |
| **PTV** | |
| Mode | Splitless |
| Inlet temperature | 40°C |
| Initial time | 0.1 min |
| Rate 1 | 720°C/min |
| Final temp 1 | 350°C |
| Final time 1 | 2 min |
| Rate 2 | 100°C/min |
| Final temp 2 | 250°C |
| Final time 2 | 0 min |
| Purge time | 0.75 min |
| Purge flow | 60 mL/min |
| **Detector** | |
| Temperature | 300°C |
| $H_2$ flow | 30 mL/min |
| Air flow | 400 mL/min |
| Makeup flow ($N_2$) | 25 mL/min |
| Lit offset | Should be < 20 pA |

**Table 18. FID Checkout Conditions (Method FIDCKO) (Continued)**

| | |
|---|---|
| **Oven** | |
| Initial temp | 40°C |
| Initial time | 0 min |
| Rate 1 | 25°C/min |
| Final temp | 90°C |
| Final time | 0 min |
| Rate 2 | 15°C/min |
| Final temp | 170°C |
| Final time | 2 min |

## Background offset checks

1. If lit, turn the FID flame off.
2. Display the signal output to determine flame-off background offset. A stable value of < 2.0 display units is acceptable. If > 20 display units, clean the detector as described in "Maintaining a flame ionization detector (FID)" on page 187.
3. Ignite the FID flame. You should see an increase in detector output to a stable value.

   If not, hold a cool, shiny metal object, such as a chrome-plated wrench, just above the FID chimney. You will see water condensation if the flame is lit.

   If the flame has not ignited, recheck all flow rates and gas source pressures. If the flame still cannot be lit, see "The flame goes out or will not light" on page 188 for more information.
4. With the flame lit, observe the FID signal at the display. This is the flame-on background offset. A stable value between 5 and 20 display units is acceptable. If an air cleaning system is being used, the signal may stabilize below 5.

   If the value is > 20 display units, system bakeout is necessary. See "Thermal cleaning (bakeout)" on page 185 for details.

   If the value is < 5 display units, verify that the electrometer is on. If it was off, recheck the flame-off background offset. If it was already on, your detector requires service.

## Noise, wander and drift

1. Make these settings on the ChemStation:

| Path | Setting |
|---|---|
| View | Full Menu |
| View/Data Analysis/Report/Specify Report | Performance + noise, OK<br>System Suitability, Edit Noise Ranges, enter several ranges that > 1.01 min plus one at 5 min, OK |
| View/Method/Save Method As | Supply name |
| Instrument menu/Edit Parameters | Oven, verify oven temp = 40°C<br>Initial time = 12 min, Rate 1 = 0°C/min, OK |
| View/Run Control/Sample Information | Enter directory and file names, close window |

2. Press `Start` to make a blank run, then wait until it is finished (Figure 8).

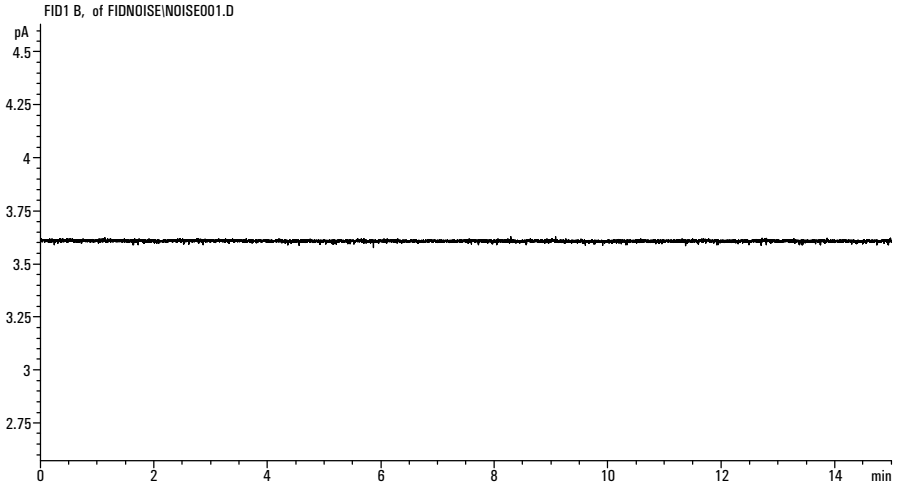



**Figure 8. Sample FID noise, wander, and drift plot**

3. Select `View/Data Analysis`. Load the data file for the blank run just completed.
4. Select `Report/ Specify Report`. Choose printer and screen.
5. Select `Integration/ Integrate`.
6. After integration is completed, Select `Report/Print Report`. The report will list measurements for noise (peak to peak, % standard deviation, and ASTM) and for drift and wander.
7. ASTM noise should be < 0.0382 pA.
8. Wander (for a 2-minute time window) should be < 0.0892 pA.
9. Drift (for a 5-minute time window) should be < 0.1911 pA.

## Chemical checkout

1. Press `Prep Run` to prepare the inlet for splitless injection. When "Ready for manual inj" appears, inject 1 µL of the checkout sample and press `Start` on the GC.
2. The chromatogram should be similar to Figure 9.
3. The acceptance criteria are:
   - Area counts for components labeled C14, C15, and C16 are all > 4,000.
   - The area counts ratio calculated as C14/C16 is 1.00 ± 0.05.
   - MDL (C16, pg/mL) = (436,000 × peak-to-peak noise)/(Area C16) ≤ 4.
4. If these criteria are not met, repeat the test. If after repeated testing the criteria cannot be met, consult your service and user documentation for additional information.

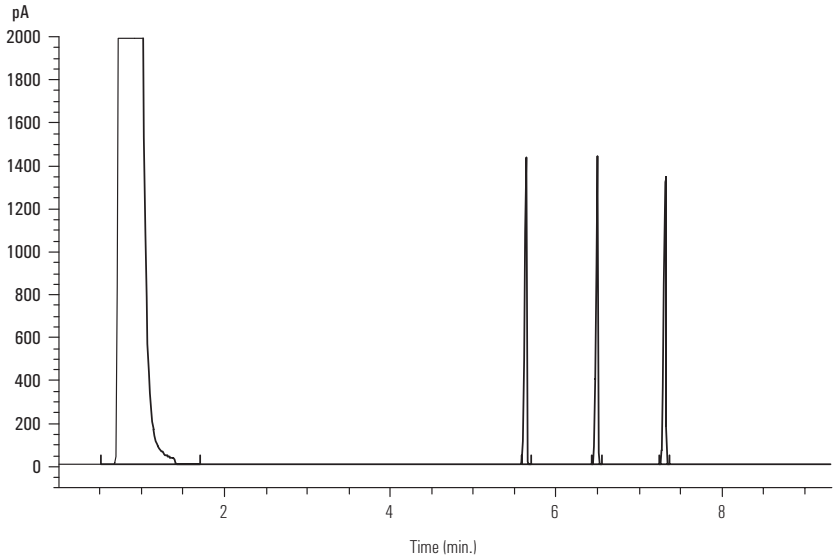



**Figure 9. Sample FID checkout chromatogram**

# μECD Checkout

## Scope

Use the following procedure to verify proper μECD operation.

## Parts and equipment required

**All inlet types**

6850 evaluation column, 30 m × 0.320 mm × 0.25 mm HP-1, part no. 19091Z–413E

μECD performance evaluation (checkout) sample, part no. 18713–60040

Chromatographic-grade helium

**Split/splitless and purged packed inlets**

10 μL syringe, part no. 9301–0810 or equivalent

O-ring, part no. 5180–4182 (split/splitless) or 5080–8898 (purged packed)

Septum, part no. 5181–1263

**Cool on-column inlet**

With septum nut:

- Needle, part no. 5182-0831, for manual syringe
- Septum nut, part no. 19245-80521

With duckbill septum

- Needle, fused silica, part no. 19091-63000
- Syringe, for fused silica needle, part no. 9301-0658
- Septum, part no. 19245-40050

**PTV inlet**

10 μL syringe, part no. 9301–0810 or equivalent

Inlet adapter, GRAPHPACK-2M, part no. 5182-9761

Silver seal, for GRAPHPACK-2M, part no. 5182-9763

Glass liner, multibaffle, part no. 5183-2037

Teflon ferrule, part no. 5182-9748 (septumless head)

Microseal replacement, part no. 5182-3444 (if installed)

GRAPHPACK 3D ferrule, part no. 5182-9749

### GC setup

**Warning** If the unit has been in operation, areas may be hot enough to cause serious burns. Turn off the heated zones and the oven and allow sufficient time for the unit to cool.

1. If you are using a split/splitless inlet, install a new septum, a new splitless liner, and a new O-ring seal in the inlet. See "To install a liner and O-ring" on page 120.

   If you are using a purged packed inlet, install a new liner and glass insert for the checkout column. Also install a new septum and O-ring. See "Changing septa" on page 139 and "Changing the O-ring" on page 142.

   If you are using a cool on-column inlet, install a new septum, then check for a proper fit of the needle in the column. See "Changing septa" on page 139 and "Cleaning the inlet" on page 131.

   If you are using a PTV (programmable temperature vaporization) inlet with the septum head, install a new septum or microseal, inlet adapter, silver seal, and glass liner. See "Changing the septum" on page 156, "Replacing liners" on page 157, and "To replace the inlet adapter" on page 150.

   If you are using a PTV inlet with the septumless head, clean the head and replace the Teflon ferrule and glass liner. See "Cleaning the septumless head" on page 152, "Replacing the Teflon ferrule" on page 154, and "Replacing liners" on page 157.
2. Install a new fused silica μECD liner (part no. G2397-20540) into the capillary column adapter, if needed.
3. Install the 6850 evaluation column.
4. If present, remove the protective caps from the inlet manifold vents.

## ChemStation setup

1. Bring up the proper on-line instrument for the 6850 to be tested. Make these settings:

| Path | Setting |
|---|---|
| View/Run Method/Control View/Instrument/ Edit Parameters/Options | Keep instrument keyboard locked after method is loaded? = No |
| View/Run Method/Control View/Instrument/ Edit Parameters/Signals | Assign Signal 1 to the detector |
| | Choose Save Data All |
| | Data Rate = 20 Hz |

2. Enter the parameter values listed in Table 19.
3. Define the column.
4. Save the method. We suggest using the name ECDCKO.

### Table 19. μECD Checkout Conditions (Method ECDCKO)

| | |
|---|---|
| **Column and sample** | |
| Type | HP-1, 30 m × 0.32 mm × 0.25 μm part number 19091Z–413E |
| Sample | μECD checkout 18713-60040 |
| Injection volume | 1 μL |
| **Inlets** | |
| Temperature | 200°C Purged packed<br>250°C Split/splitless<br>Oven Track (cool on-column)<br>80°C PTV (see below) |
| Inlet pressure | 25 psi (constant pressure, helium) |
| **Split/splitless** | |
| Mode | Splitless |
| Purge flow | 60 mL/min |
| Purge time | 0.75 min |
| **PTV** | |
| Mode | Splitless |
| Inlet temperature | 80°C |
| Initial time | 0.1 min |
| Rate 1 | 720°C/min |
| Final temp 1 | 350°C |
| Final time 1 | 2 min |

**Table 19. μECD Checkout Conditions (Method ECDCKO)**

| | |
|---|---|
| Rate 2 | 100°C/min |
| Final temp 2 | 250°C |
| Final time 2 | 0 min |
| Purge time | 0.75 min |
| Purge flow | 60 mL/min |
| **Detector** | |
| Temperature | 300°C |
| Anode purge flow ($N_2$) | 60 mL/min |
| Makeup flow ($N_2$) | 25 ± 2 mL/min |
| Offset | Should be < 1000 display counts |
| **Oven** | |
| Initial temp | 80°C |
| Initial time | 0 min |
| Rate 1 | 15°C/min |
| Final temp | 180°C |
| Final time | 10 min |

## Offset checkout

Display the signal output to determine background offset. A stable offset value less than 1000 display units (inclusive) is acceptable.

- If the offset is < 0.5 display units, verify that the electrometer is on. If the offset is still < 0.5 display units, your detector requires service.
- If offset is > 1000 display units, there may be chemical contamination contributing to the signal. Perform a thermal cleanout (see "Thermal cleaning" on page 207 for details). If bakeout does not give an acceptable signal, check gas purity. Use higher purity gases and/or install traps.

## Noise, wander and drift

1. Make these settings on the ChemStation:

| Path | Setting |
|---|---|
| View | Full Menu |
| View/Data Analysis/Report/Specify Report | Performance + noise, OK |
| | System Suitability, Edit Noise Ranges, enter several ranges that > 1.01 min plus one at 5 min, OK |
| View/Method/Save Method As | Supply name |
| Instrument menu/Edit Parameters | Oven, verify oven temp = 40°C |
| | Initial time = 12 min, Rate 1 = 0°C/min, OK |
| View/Run Control/Sample Information | Enter directory and file names, close window |

2. Press `Start` to make a blank run, then wait until it is finished.
3. Select `View/Data Analysis`. Load the data file for the blank run just completed.
4. Select `Report/ Specify Report`. Choose printer and screen.
5. Select `Integration/ Integrate`.
6. After integration is completed, Select `Report/Print Report`. The report will list measurements for noise (peak to peak, % standard deviation, and ASTM) and for drift and wander.
7. ASTM noise should be < 3.0 Hz.
8. Wander (for a 2-minute time window) should be < 5 Hz.
9. Drift (for a 5-minute time window) should be ≥ .|15| Hz/hour

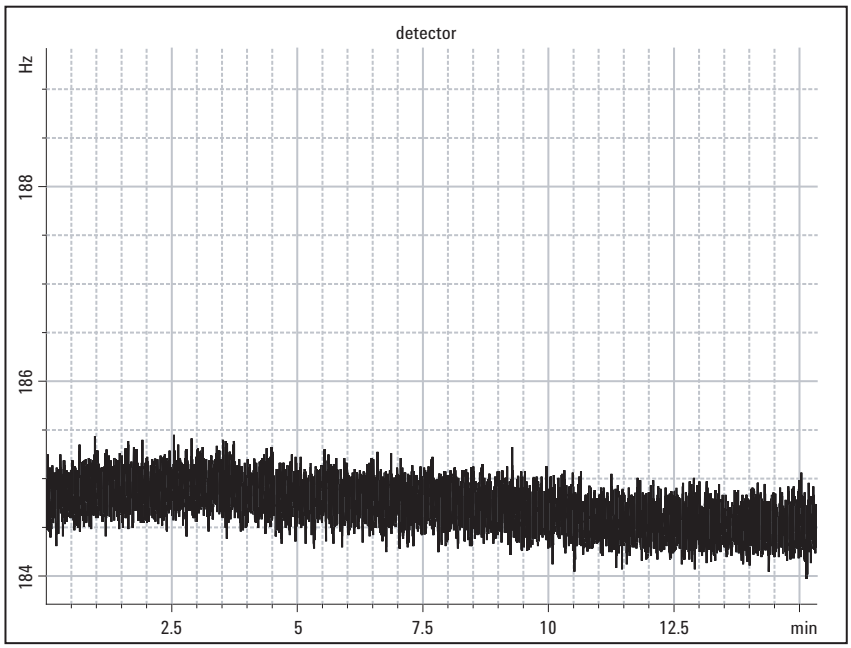
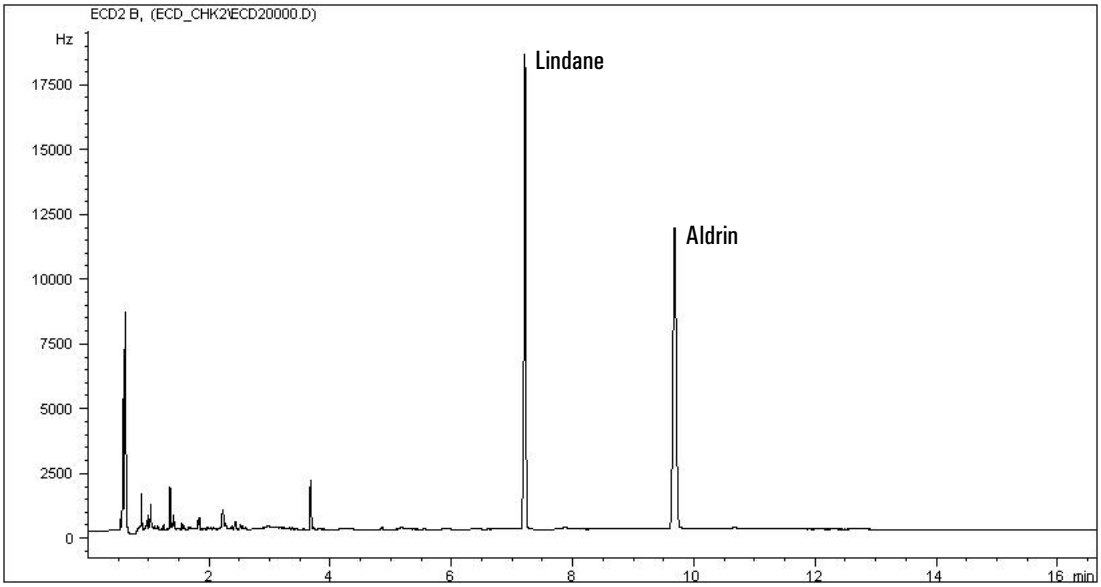



**Figure 10. Sample μECD noise, wander, and drift plot**

## Chemical checkout

1. Press `Prep Run` to prepare the inlet for splitless injection. When "Ready for manual inj" appears, inject 1 μL of the checkout sample and press `Start` on the GC.
2. The chromatogram should be similar to Figure 11.
3. The acceptance criteria is:
   - Peak response ≥ 7500 × Noise
4. If these criteria are not met, repeat the test. If after repeated testing the criteria cannot be met, consult your service and user documentation for additional information.




Your retention times will differ but peaks should resemble the example.

**Figure 11. Sample µECD checkout chromatogram**

# FPD Checkout

## Scope

Use the following procedure to verify proper FPD operation.

## Parts and equipment required

**All inlet types**

6850 evaluation column, 30 m × 0.320 mm × 0.25 mm HP-1, part no. 19091Z–413E

FPD performance evaluation (checkout) sample, part no. 8500-3697

Chromatographic-grade gases: helium, nitrogen, hyrogen, and air

**Split/splitless and purged packed inlets**

10 µL syringe, part no. 9301–0810 or equivalent

O-ring, part no. 5180–4182 (split/splitless) or 5080–8898 (purged packed)

Septum, part no. 5181–1263

**Cool on-column inlet**

With septum nut:

- Needle, part no. 5182-0831, for manual syringe
- Septum nut, part no. 19245-80521

With duckbill septum

- Needle, fused silica, part no. 19091-63000
- Syringe, for fused silica needle, part no. 9301-0658
- Septum, part no. 19245-40050

**PTV inlet**

10 µL syringe, part no. 9301–0810 or equivalent

Inlet adapter, GRAPHPACK-2M, part no. 5182-9761

Silver seal, for GRAPHPACK-2M, part no. 5182-9763

Glass liner, multibaffle, part no. 5183-2037

Teflon ferrule, part no. 5182-9748 (septumless head)

Microseal replacement, part no. 5182-3444 (if installed)

GRAPHPACK 3D ferrule, part no. 5182-9749

### GC setup

**Warning** If the unit has been in operation, areas may be hot enough to cause serious burns. Turn off the heated zones and the oven and allow sufficient time for the unit to cool.

1. If you are using a split/splitless inlet, install a new septum, a new splitless liner, and a new O-ring seal in the inlet. See "To install a liner and O-ring" on page 120.

   If you are using a purged packed inlet, install a new liner and glass insert for the checkout column. Also install a new septum and O-ring. See "Changing septa" on page 139 and "Changing the O-ring" on page 142.

   If you are using a cool on-column inlet, install a new septum, then check for a proper fit of the needle in the column. See "Changing septa" on page 139 and "Cleaning the inlet" on page 131.

   If you are using a PTV (programmable temperature vaporization) inlet with the septum head, install a new septum or microseal, inlet adapter, silver seal, and glass liner. See "Changing the septum" on page 156, "Replacing liners" on page 157, and "To replace the inlet adapter" on page 150.

   If you are using a PTV inlet with the septumless head, clean the head and replace the Teflon ferrule and glass liner. See "Cleaning the septumless head" on page 152, "Replacing the Teflon ferrule" on page 154, and "Replacing liners" on page 157.
2. Install the appropriate wavelength filter (S or P) for the test. See "Changing wavelength filters" on page 210.
3. Install the 6850 evaluation column.
4. If present, remove the protective caps from the inlet manifold vents.

## ChemStation setup

1. Bring up the proper online instrument for the 6850 to be tested. Make these settings:

| Path | Setting |
|---|---|
| View/Run Method/Control View/Instrument/ Edit Parameters/Options | Keep instrument keyboard locked after method is loaded? = No |
| View/Run Method/Control View/Instrument/ Edit Parameters/Signals | Assign Signal 1 to the detector |
| | Choose Save Data All |
| | Data Rate = 20 Hz |

2. Enter the parameter values listed in Table 20.
3. Define the column.
4. Save the method. We suggest using the name `FPDCKO`.

**Table 20. FPD Checkout Conditions (Method FPDCKO)**

| | |
|---|---|
| **Column and sample** | |
| Type | HP-1, 30 m × 0.32 mm × 0.25 μm part number 19091Z–413E |
| Sample | FPD checkout 8500-3697 |
| Injection volume | 1 μL |
| **Inlets** | |
| Temperature | 250°C Purged packed and split/splitless<br>Oven Track (cool on-column)<br>80°C PTV (see below) |
| Inlet pressure | 25 psi (constant pressure, helium) |
| **Split/splitless** | |
| Mode | Splitless |
| Purge flow | 60 mL/min |
| Purge time | 0.75 min |
| **PTV** | |
| Mode | Splitless |
| Inlet temperature | 80°C |
| Initial time | 0.1 min |
| Rate 1 | 720°C/min |
| Final temp 1 | 350°C |
| Final time 1 | 2 min |
| Rate 2 | 100°C/min |
| Final temp 2 | 250°C |

**Table 20. FPD Checkout Conditions (Method FPDCKO) (Continued)**

| | |
|---|---|
| Final time 2 | 0 min |
| Purge time | 0.75 min |
| Purge flow | 60 mL/min |
| **Detector** | |
| Temperature | 200°C |
| $H_2$ flow | 75 ±2 mL/min |
| Air flow | 100 ±2 mL/min |
| Makeup flow ($N_2$) | 60 ±2 mL/min |
| Offset, flow off (O-fa) | Should be < 40 display units |
| Offset, flame on (O+fb) | < [(O-fa) + 85 display units] |
| **Oven** | |
| Initial temp | 60°C |
| Initial time | 0 min |
| Rate 1 | 25°C/min |
| Final temp | 110°C |
| Final time | 0 min |
| Rate 2 | 10°C/min |
| Final temp | 170°C |
| Final time | 3 min |

## Background offset checks

1. If lit, turn the FPD flame off.
2. Display the signal output to determine flame-off background offset. A stable value of < 40 display units is acceptable.
3. Ignite the flame. You should see an increase in detector output to a stable value.

   If not, confirm ignition by holding a mirror or shiny surface near the aluminum exhaust tube, with the rubber drip tube removed, and observing condensation if the flame is lit.

   If the flame has not ignited, see "Flame ignition problems" on page 208.

4. With the flame lit, observe the FPD signal at the display. This is the flame-on background offset. A stable value < (Flame off + 85) display units is acceptable.

   If the value is < 5 display units, verify that the electrometer is on. If it was off, recheck the flame-off background offset. If it was already on, your detector requires service.

### Noise, wander and drift

1. Make these settings on the ChemStation:

| Path | Setting |
|---|---|
| View | Full Menu |
| View/Data Analysis/Report/Specify Report | Performance + noise, OK |
| | System Suitability, Edit Noise Ranges, enter several ranges that > 1.01 min plus one at 5 min, OK |
| View/Method/Save Method As | Supply name |
| Instrument menu/Edit Parameters | Oven, initial temp 60°C |
| | Initial time = 12 min, Rate 1 = 0°C/min, OK |
| View/Run Control/Sample Information | Enter directory and file names, close window |

2. Press `Start` to make a blank run, then wait until it is finished.
3. Select `View/Data Analysis`. Load the data file for the blank run just completed.
4. Select `Report/ Specify Report`. Choose printer and screen.
5. Select `Integration/ Integrate`.
6. After integration is completed, Select `Report/Print Report`. The report will list measurements for noise (peak to peak, % standard deviation, and ASTM) and for drift and wander.
7. Base signal should be:
   Flame off ≤ 40 display units
   Flame on ≤ Flame off + 85 display units
8. ASTM noise should be < 5 display units.
9. Wander (for a 2-minute time window) should be < 2.5 display units.

10. Drift (for a 5-minute time window) should be ≤ |5| display units/hour. See Figure 12.

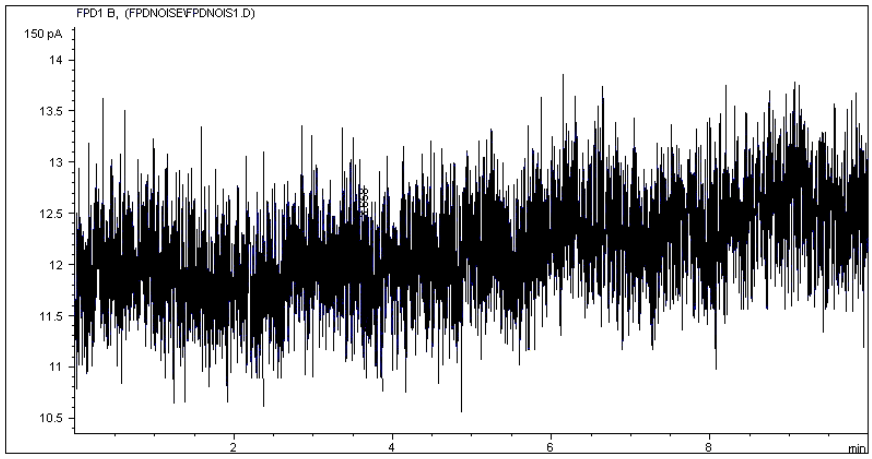
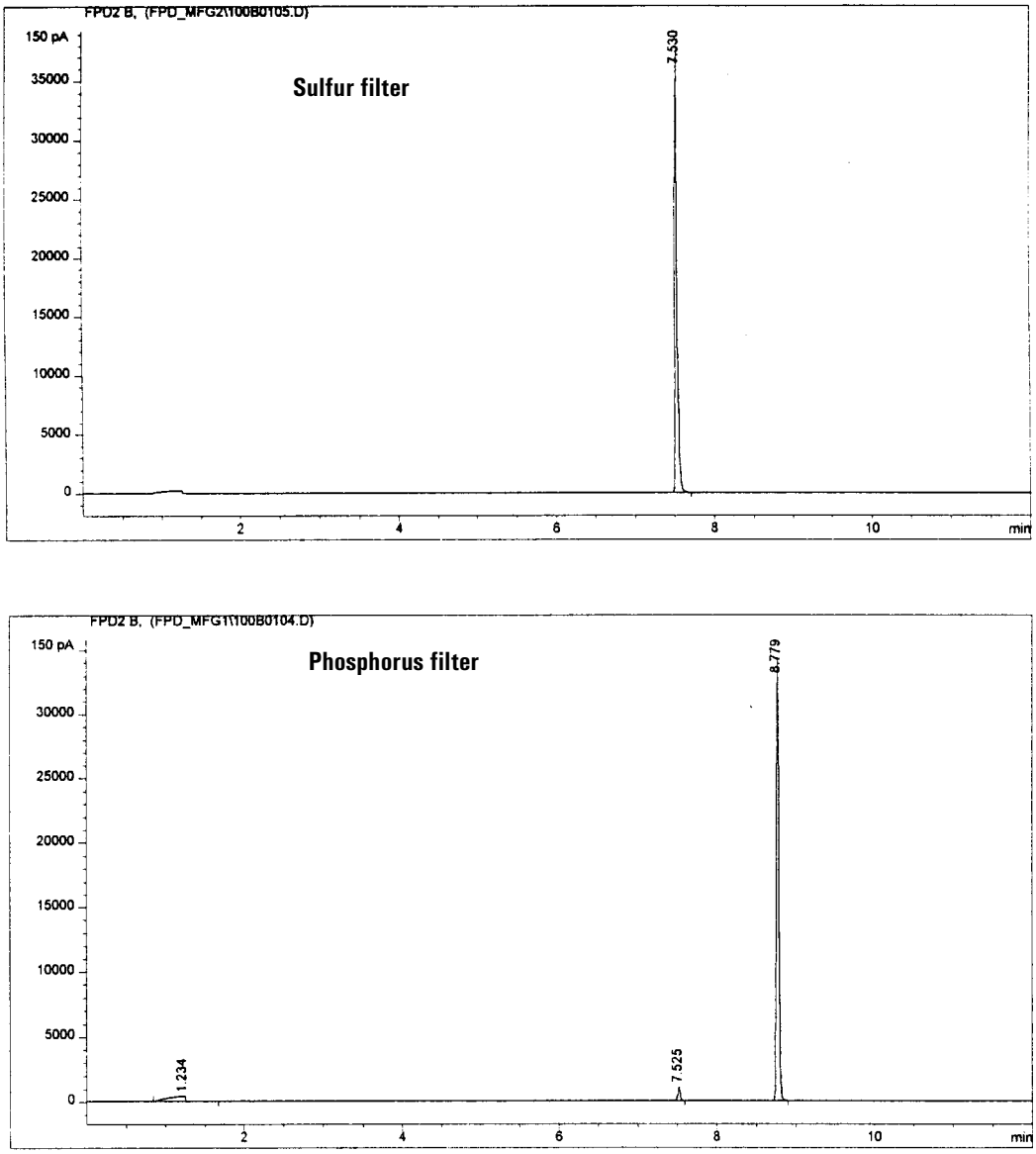



**Figure 12. Sample FPD noise, wander, and drift plot**

## Chemical checkout

1. Press `Prep Run` to prepare the inlet for splitless injection. When "Ready for manual inj" appears, inject 1 µL of the checkout sample and press `Start` on the GC.
2. The chromatogram should be similar to Figure 13.
3. The acceptance criteria are:
   - Sulfur peak response ≥ 24000 counts
   - Phosphorus peak response ≥ 24000 counts
4. If these criteria are not met, repeat the test. If after repeated testing the criteria cannot be met, consult your service and user documentation for additional information.




Your retention times will differ, but peaks should resemble this example.

**Figure 13. Typical FPD checkout chromatograms**

# Messages

The GC monitors the state of its detector, pneumatics, oven, PC boards, and other components. If a problem exists, the GC displays a message, beeps, or activates an LED and puts itself in a “safe state” if the problem could be dangerous to the user.

## Message types

**Bad main board and Fatal error**—These messages almost always indicate that the main board is malfunctioning and must be replaced. They usually appear when the instrument is first turned on. See Table 21 for a list of messages. With the few exceptions listed in Table 21, if you get a Bad main board or Fatal error message, you will need to contact your Agilent service representative to replace the board.

**Table 21. Bad Main Board and Fatal Error Messages**

| Message | Comments |
|---|---|
| **BAD MAIN BOARD** | |
| Main FPGA failure | Defective main board |
| Static RAM failure | Defective main board |
| Boot ROM checksum | Defective main board |
| Timeout $ # | Defective main board |
| **FATAL ERROR** | |
| Vect Offset $ # | Defective firmware or main board |
| Bus Error $ # | Defective firmware or main board |
| Addr Error$ # | Defective firmware or main board |
| Instruct $ # | Defective firmware or main board |
| Div by 0 $ # | Defective firmware or main board |
| Det interface board | The board is missing, defective, or installed incorrectly. The ribbon cable may be defective. |

**Not Ready**—Some component of the GC is not ready to begin a run. The Not Ready LED is on and there is a scrolling message in the top line of the display. See Table 22 on page 83.

**Method mismatch**—The active method has parameters that do not match the present GC configuration.

- If the mismatched parameter is set by the user, the method overwrites the present value. For example, if the gas type presently configured differs from the one in the method, the present gas type is overwritten with that of the method.
- If the mismatched parameter is hardware dependent, that part of the method is ignored and the present setpoints remain. For example, if the method is for a TCD detector but you have replaced it with an FID, the method TCD information is ignored and the present FID parameters remain.

**Warning**—A problem exists but the instrument will execute the run. The GC emits one beep and a scrolling message appears in the top line of the display. The GC can start the run and the warning disappears when a run starts. Warnings are not logged.

**Sequence warning**—This set of warnings is listed alphabetically since they are not numbered.

**Service warning**—These messages inform you when certain consumables, such as the septum, have exhausted their useful life.

**Shutdown**—There is a hardware problem that could compromise the safety of the user or damage the instrument. Before shutdown occurs, the GC emits a series of warning beeps (pneumatics shutdowns only). After a time specific for the component elapses, the component with the problem shuts down, the GC emits one beep, and a warning message appears. The GC is Not Ready. The error is not recorded in the run log.

**Fault**—Hardware problems exist that require user intervention. Depending on the type of error, the GC emits no beep or a single beep. The Not Ready LED lights and an error message appears. The error is recorded in the run log.

Two particular faults can shut down the entire GC: a pneumatics problem for a component configured for hydrogen gas and a thermal runaway condition for the GC oven. In these cases, the GC beeps continuously until you press the `Stop` key.

**Memory reset**—All GC methods, the Run Log, and the Log Book are stored in battery-backed GC memory. If the battery fails and power is turned off, this data is lost and the warning appears when the GC is turned on. If you update the GC firmware, this data is erased during the update and the warning appears after restart.

**Undefined errors**—Some error conditions that require service assistance do not have individual messages. They produce the Undefined error message.

This usually indicates either a defective main board or GC firmware that is obsolete for your present configuration. Check your instrument to determine the present status of the heated zones, inlet, gases, and detector.

## Not Ready messages

**Table 22. Not Ready Messages**

| Temperature messages | Comments |
|---|---|
| Aux, Detector, Inlet, or Oven temperature | Zone has not equilibrated at setpoint or oven is OFF, Turn oven ON and wait |
| Detector temp low | Temp or temp setpoint below minimum required to avoid damage, check setpoint. If setpoint OK, wait. |
| **Detector messages** | |
| Detector $H_2$, air, or makeup gas flow | Flow is not at setpoint, check gas supply. If OK, wait. |
| Detector igniting | Wait until ignition is done |
| Detector shutdown | Pneumatics failure or TCD filament failure |
| **Inlet messages** | |
| Inlet flow or pressure | Not at setpoint, check gas supply. If OK, wait. |
| Inlet purging | Press `Prep Run` to go ready |
| Inlet pulse inactive | Press `Prep Run` to go ready |
| Gas saver active | Press `Prep Run` to go ready |
| **Valve messages** | |
| Multiposition valve | Valve has not reached specified position, check BCD cable |
| Sampling valve | Valve is ON but Inject Time has not elapsed and not in run, or valve is OFF but Load Time has not ended |
| **Auxiliary EPC messages** | |
| Aux *N* pressure | Channel *N* not at setpoint, check gas supply. If OK, wait. |
| **Miscellaneous messages** | |
| Diagnostics mode | GC is not operating normally because of testing |
| Test in progress | Wait for test to end or terminate test |
| Host system | Host such as ChemStation or Cerity Chemical is not ready |
| External device | Device on REMOTE cable is not ready |
| Power on in progress | Wait |

# Warning messages

### WARNING 100 Oven sensor missing

Possible causes:
Sensor not connected
Sensor defective

The GC main board could not detect the oven temperature sensor. To prevent a thermal runaway, the oven is set to off and the instrument is not ready.

### WARNING 101 Excess heater power

Possible causes:
Heater wattage total > 210W
Defective main board

The total wattage for the detector, inlet, and auxiliary heated zones exceeds 210 Watts. This value is only checked at power on. The three heated zones are set to 'not installed.'

### WARNING 103 Signal 1 buffer full

Possible cause:
Data collection device is off-line and signal buffer is full

If your integrator or ChemStation goes off line during a run, signal data is stored in the GC's signal buffer. When the buffer is full, the warning appears and no new data is stored.

### WARNING 104 Signal 2 buffer full

Possible cause:
Control module is off-line and its signal buffer is full

If the control module goes off line during a run, signal data is stored in the GC's signal buffer. When the buffer is full, the warning appears and no new data is stored.

### WARNING 105 Analog out data loss

Possible causes:
Defective main board
Bad power supply (less likely)

The GC analog processors are not operating correctly. Some analog data is lost, but digital data may be uncorrupted.

### WARNING 106 Data loss

Possible causes:
Defective main board
Bad power supply (less likely)

The GC digital processors are not operating correctly. Both analog and digital data are lost.

### WARNING 107 Det config changed
### WARNING 109 Inlet config changed
### WARNING 111 Column config changed
### WARNING 113 Aux 3 config changed
### WARNING 114 Aux 4 config changed
### WARNING 115 Aux 5 config changed

Possible causes: There is a new detector/inlet/column/auxiliary EPC module in the GC
The detector/inlet/column/auxiliary EPC module in the GC does not match the method

The method was changed to default detector/inlet/column/auxiliary EPC module values because either a different one was installed, or a newly loaded method uses a different detector/inlet/column/auxiliary EPC module than is available.

If a new detector/inlet/column/auxiliary EPC module, the GC will enter default setpoints for the new type.

If the method uses a different detector/inlet/column/auxiliary EPC module, the method is unchanged and the warning message flashes.

### WARNING 116 Run log full

The Run log can hold 50 entries of information. When it reaches the maximum, this warning will appear.

### WARNING 117 Inlet calib deleted
### WARNING 119 Det calib deleted
### WARNING 121 Aux calib deleted

Possible cause: Power failure during recalibration

While entering a new flow module calibration, the GC crashed or encountered a power failure before the recalibration was complete. The warning appears when the instrument is turned on again.

The calibration for the module returned to the default. Recalibrate.

### WARNING 128 Inlet flow cal failed

Possible cause: Defective or leaking flow module

An attempted auto calibration of the flow sensor offset for the inlet exceeded the allowable calibration range. The previous calibration setpoints remain in memory.

### WARNING 130 Aux cryo disabled

Possible cause: Incorrectly installed inlet cryo valve or valve box

The inlet cryo valve is also being used by aux thermal zone 1. The valve is disabled.

### WARNING 146 Memory reset

Possible causes:
The main board battery failed
The GC firmware was updated

All GC methods, the run log, and the log book are stored in battery-backed GC memory. If the backup battery fails and power is turned off, this data is lost and the warning appears when the GC is turned on. If you update the GC firmware, this data is erased during the update, and the warning appears after restart.

### WARNING 901 GC firmware

Possible cause:
GC firmware revision is A.01.xx and a purged packed inlet is installed

A purged packed inlet is detected but the current GC firmware is out of date and may not support the new inlet. Contact Agilent Technologies to upgrade your GC firmware. Version A.02.00 or higher is required.

## Sequence warning messages

### Autoinject aborted

Possible cause:
The injector sequence was aborted during a run or injection. Other messages detail why.

### Bottle # out of range

Possible causes:
Incorrect last vial parameter
Wrong turret installed for your method
Turret was changed but not aligned

The GC failed to find the vial specified in the method because that vial number is not available in the turret installed. This message may appear if you changed the turret type but did not perform the alignment procedure.

### Incomplete injection

Possible causes:
Bent syringe needle
Defective plunger or syringe carriage

The injector syringe carriage did not reach the position at which the needle is fully inserted in the inlet. The injection could not be completed or some sample may have escaped.

### Inj door or mounting

Possible causes:
Injector door open
Injector not mounted properly on GC
Defective safety switch on injector

During an injection routine, the injector halts for safety reasons if either the injector door is open or if the injector is not properly mounted on the GC post.

### Injector comm error

Possible cause:
Defective injector board

During an injection in a sequence, the GC lost communication with the injector.

### Injector not aligned

Possible cause: Alignment procedure was not successfully completed

The turret in the 6850 automatic liquid sampler was changed or replaced, but the alignment procedure was not performed.

### Injector reset

Power to the injector was interrupted.

Possible causes:
Injector cable (most likely)
Defective injector power supply
GC main board or AC board

The sequence is aborted when the injector resets.

### Invalid sequence

Possible causes:
Sequence is set up for wrong injection device
Hardware required by sequence is not installed and configured
GC configuration was changed during sequence execution
Defective cabling or connection

The sequence type does not match the current GC configuration. The sequence loaded was set up for a valve or injector, and that valve or injector is no longer present or is not configured.

### No injector

Possible causes:
Injector cable defective or not connected properly
Defective GC or injector board

After the run began, the GC could no longer detect the injector. Either the injector cable is not securely connected to the GC or an electronics board failed.

### Plunger error

Possible causes:
Syringe plunger is sticking
Defective plunger solenoid
Defective plunger carrier encoder

The injector tracks plunger motion. The error occurs if the injector determines that the plunger is not moving as expected or if it cannot locate the plunger's position.

### Prerun > 10 min

Possible cause: GC Not Ready. Check for Not Ready and other messages for the cause.

During an injection, the GC became Not Ready and the sampler halted. When the GC did not become Ready within 10 minutes, the sampler reported an error and the sequence aborted.

### Sampler error

Possible causes: Defective sampler electronics

The sampler could not function properly for an undocumented reason. Record the code number shown in the Control Module error message. If the problem persists, contact Agilent Technologies for service and report the error number.

### Sequence aborted

The sequence was aborted during a run or injection. Other messages detail why.

### Seq method load err

Possible causes: Method corrupt or deleted
A method mismatch error occurred

Each method called in a sequence must be able to load without errors.

### Syringe error

Possible causes: Defective syringe carriage motor
Syringe not installed properly
Incorrect syringe type
Syringe carriage sensor defective

The syringe carriage could not complete its motions. First check the syringe. If the needle is bent, replace the syringe or straighten the needle. Make sure the syringe meets the dimensional specifications required by the injector. For other problems, see your injector documentation.

### Turret error

Possible causes: Something interfered with turret rotation.
Defective injector turret motor or encoder assembly
Turret type was changed without re-alignment
Turret is loose

The injector tracks turret motion, and determined that the turret was not moving as expected.

## Service warning messages

### WARNING 500 Change septum

The service limit (number of injections) for the septum has been exhausted. To maintain optimum performance, use your control module to go to Status/ Service/ Start Service when ready to replace the septum.

- The GC will still start a run when this warning is displayed.
- To adjust/turn off the service limit, go to Status/Service/Service Limits.

### WARNING 501 Change liner

The service limit (number of injections) for the liner has been exhausted. To maintain optimum performance, use your control module to go to Status/ Service/ Start Service when ready to replace the liner.

- The GC will still start a run when this warning is displayed.
- To adjust/turn off the service limit, go to Status/Service/Service Limits.

### WARNING 502 Service syringe

The service limit (number of injections) for the syringe has been exhausted. To maintain optimum performance, use your control module to go to Status/ Service/ Start Service when ready to service the syringe.

- The GC will still start a run when this warning is displayed.
- To adjust/turn off the service limit, go to Status/Service/Service Limits.

### WARNING 503 Service column

The service limit (number of injections) for the column has been exhausted. To maintain optimum performance, use your control module to go to Status/ Service/ Start Service when ready to service the column.

- The GC will still start a run when this warning is displayed.
- To adjust/turn off the service limit, go to Status/Service/Service Limits.

## Shutdown messages

### SHUTDOWN 1 Oven shut off

To clear the shutdown, turn the GC off and then on again or change the oven temperature.

Possible causes:

Check the oven flaps. If an oven flap is stuck open completely or partially, it is not operating correctly. During normal use, the flaps should be full open or full closed.
Oven lid down but not latched
Defective main board
Defective heater

The power required to keep the oven at setpoint exceeds the expected power for that temperature. The oven is shut off and the flaps should be opened half-way for cooling.

**SHUTDOWN 2** **Oven cryo shut off**

To clear the shutdown, turn the GC off, then on, or change the temperature setpoint.

Possible causes:
Cryo timeout: Check timeout setpoint.
Cryo fault: Check coolant supply. Defective cryo valve.

Cryo shutdowns conserve coolant when the GC is unable to start a run. The cryogenic cooling system may still be working properly. A cryo timeout occurs if your specified cryo timeout period expires before the oven reaches its temperature setpoint. A cryo fault occurs if cryo cooling has been on for over 16 minutes but the oven has not reached its temperature setpoint.

**SHUTDOWN 3** **Inlet pressure**
**SHUTDOWN 4** **Inlet flow**

Possible causes:
Source gas supply pressure too low
Large leak or broken column or column plugged
Gas saver flow rate set too low to get highest run pressure
Flow set too low for the column in use
Defective/Contaminated inlet flow module
The split ratio is too low

**Shutdown 3.** The inlet failed to reach its pressure setpoint within 5.5 minutes or cannot maintain its pressure setpoint. The inlet's flows go to purge mode and the instrument goes to a not ready state until the inlet reaches the pressure setpoint.

**Shutdown 4.** The inlet failed to reach its flow setpoint within 2 minutes or cannot maintain its flow setpoint. The GC will not be ready until the problem is fixed and the flow reaches its setpoint.

**SHUTDOWN 7** **Det $H_2$ gas pressure**
**SHUTDOWN 8** **Det air/ref pressure**
**SHUTDOWN 9** **Det makeup pressure**
**SHUTDOWN 13** **Aux 3 pressure**
**SHUTDOWN 14** **Aux 4 pressure**
**SHUTDOWN 15** **Aux 5 pressure**

To clear the shutdown, correct the problem so the detector or auxiliary flow module reaches the pressure setpoint.

Possible causes:
Source gas supply pressure too low
Leak in the system (including column)
Defective detector or auxiliary flow manifold

**Shutdown 7.** The detector's hydrogen gas was unable to reach or maintain the pressure setpoint in the allotted 2 minutes.

**Shutdown 8.** The detector's air/reference gas is unable to reach or maintain the pressure setpoint.

**Shutdown 9.** The detector's makeup gas is unable to reach or maintain the pressure setpoint.

**Shutdown 13–15.** The auxiliary EPC gas is unable to reach or maintain the pressure setpoint.

The detector gases are shut off and the instrument will be not ready until the problem is corrected and the detector reaches the pressure setpoint.

## SHUTDOWN 16 Valve 2 not switching

To clear the shutdown, enter a new setpoint.

Possible causes:
Valve not connected
Valve is stuck
Switching time is too short for the valve's speed
Valve sticking slightly or sample too viscous

The multiposition valve has tried to switch twice without success. The valve shuts down and becomes not ready (not at setpoint).

## SHUTDOWN 17 Valve 2 setpt error

To clear the shutdown, enter a new setpoint.

Possible causes:
Entered valve position is larger than valve supports.
Invert BCD setpoint is incorrect. With most valves, the invert should be On. If the BCD setpoint is already On, set it to Off.

The multiposition valve is switching to the wrong position or is unable to switch to the setpoint position. The valve shuts down and becomes not ready (not at setpoint).

## SHUTDOWN 18 Inlet cryo shut off

To clear the shutdown, turn the zone off, then on, or change the temperature setpoint.

Possible causes:
Cryo timeout: Check timeout setpoint.
Cryo fault: Check coolant supply. Defective cryo valve.

Cryo shutdowns conserve coolant when the GC is unable to start a run. The cryogenic cooling system may still be working properly. A cryo timeout occurs if your specified cryo timeout period expires before the oven reaches its temperature setpoint. A cryo fault occurs if cryo cooling has been on for over 16 minutes but the oven has not reached its temperature setpoint.

**SHUTDOWN 22** **Inlet heating slow**

To clear the shutdown, turn the inlet zone On, then Off or change the temperature.

Possible causes: Defective sensor
Defective heater

The inlet heater has been full on for over the allowed duration and the inlet is not at setpoint. The inlet thermal zone is shut down and the instrument becomes not ready.

## Fault messages

**FAULT 201** **Pneumatics control**

Possible cause: Defective main board

The main board is not functioning properly. Pneumatics are shut down.

**FAULT 202** **$H_2$ safety shutdown**

To clear the fault, turn the GC off, then on.

Possible causes: Hydrogen supply gas pressure low/no gas
Leak in system (supply, inlet, detector, column)
Broken column
Defective inlet flow manifold

The GC did not reach the hydrogen pressure setpoint before the time limit elapsed. Because hydrogen presents an explosion hazard, the entire GC has shut down. The instrument beeps continuously; press `Stop` to silence it. The oven is shut off with the flaps half open and the fan stays on for about 3 seconds. The column flow is turned off.

**FAULT 203** **Signal DSP faulty**
**FAULT 204** **Sig DSP ROM error**
**FAULT 205** **Sig DSP RAM error**
**FAULT 206** **Sig DSP registers**
**FAULT 207** **Sig DSP data corrupt**

Possible cause: Defective main board

The GC's signal processing electronics are not functioning correctly. This indicates a defective main board. The signal path will not function.

Turn the instrument off and then on at least one time. If the error still occurs, the main board is defective.

### FAULT 208 0-1mV signal path fail
### FAULT 210 Analog out path fail

Possible cause: Defective main board

**Fault 208.** The 0-1 mV out signal sent to a strip-chart recording device is not within the acceptable range. The GC will not be ready.

**Fault 210.** The 1 V out signal sent to an integrator is not within the acceptable range. The GC will not be ready.

Turn the instrument off and then on at least one time. If the error still occurs, the main board is defective.

### FAULT 212 Det electrometer

Possible causes:
Poor cable connection between detector and detector board
Defective electrometer
Defective detector board

The electrometer, inside the detector, measures and amplifies the signal from the detector. The signal is sent to the detector board, which converts it to digital form. If the electrometer is out of specification, the detector will never reach a ready state.

### FAULT 214 Detector flame out

Possible causes:
Hydrogen and/or air flow turned off
Hydrogen and/or air flow rates too low
Leak near the detector column fitting
Incorrect jet for the installed column
Change the Lit Offset to 2.0 (the default value)

The FID or FPD is not able to ignite or the flame is out (during a run). The detector will try to ignite the flame several times. If it fails, the hydrogen, air, and ignitor shut off, and the Fault message appears. The detector will be in a not ready state.

### FAULT 216 TCD filament open

Possible causes:
Wires from detector not connected properly to detector board
Damaged $\Delta$ PRT
Defective TCD cell

The TCD filament bridge voltage indicates that the filament resistance is too high. The filament may be broken or worn thin from use, or the wires from the TCD are not connected on the detector board, or the $\Delta$ PRT is shorted. The detector will not be ready until the condition is corrected.

### FAULT 218 TCD filament short

Possible causes:
Wires from detector not connected properly to detector board
Defective TCD cell

The resistance of the filament is too low. This could be caused by a worn or sagging filament, or if the TCD wires are not connected properly to the detector board or are touching each other, or if the Δ PRT wires are not properly connected to the detector board. The detector will not be ready until the condition is corrected.

### FAULT 220 Heater overcurrent

To clear the error message, turn off all the heated zones and turn the GC off and then on again.

Possible causes:
Shorted heater
Defective main board
Wrong heater (heater power > 70W)

A heater in one of the small heated zones tripped an over-current safety circuit. There is a short in one of the heaters, or the heated zone's electronics are defective. This error does not indicate a problem with the oven. Power to the small heated zones is disabled.

### FAULT 221 Thermal shutdown

To clear the fault, turn the GC off, then on.

Possible causes:
Defective main board
Shorted temperature sensor
Shorted heater
Wrong heater (heater power > 70W)

A thermal shutdown fault disables power to the oven and other heated zones (inlet, detector, aux). The GC will be not ready.

### FAULT 222 Oven temp too hot

Possible causes:
Defective oven temp sensor
Defective main board

The oven temperature reading indicates the oven is > 25 xb°C above its maximum allowable temperature. Oven and other heated zone power is shut down. The GC will be not ready.

To determine if the oven sensor is broken, test its resistance. The resistance will be 100 ohms if the sensor is working correctly; a much larger value indicates a faulty sensor.

### FAULT 223 Oven temp too cool

To clear the error message, turn off all the heated zones and turn the GC off and then on again.

Possible causes: Cryo valve stuck open
Shorted oven sensor

The oven temperature reading indicates the oven is less than its minimum allowable temperature. Power to the small heated zones is disabled.

### FAULT 224 Oven sensor shorted

Possible cause: Defective (shorted) oven sensor

The oven temperature reading is lower than expected, which indicates a shorted oven sensor. Power is turned off for all the heated zones.

### FAULT 225 Det temp too hot

To clear the message, turn off all the heated zones, then turn the instrument off and on again.

Possible causes: Defective sensor in the detector
Defective main board

The detector's temperature is higher than the maximum allowable temperature (detector type maximum temperature plus 25 xb°C). Power is disabled to all the heated zones.

### FAULT 226 Detector temp sensor

Possible cause: Defective sensor in the detector

The detector's temperature is lower than expected, which indicates a shorted sensor. Power is turned off for the detector.

### FAULT 229 Inlet temp too hot

To clear the message, turn off all the heated zones, then turn the instrument off and on again.

Possible causes: Defective inlet heater
Defective main board

The inlet's temperature reading is higher than the maximum allowed (inlet type maximum temp plus 25 xb°C). Power is disabled to the small heated zones and the oven.

### FAULT 230 Inlet temp sensor

Possible cause: Defective (shorted) inlet sensor

The inlet temperature reading is lower than expected, which indicates a shorted sensor. Power is turned off for the inlet.

### FAULT 233 Aux temp too hot

To clear the message, turn off all the heated zones, then turn the instrument off and on again.

Possible causes: Defective aux heater
Defective main board

The aux zone's temperature is higher than the maximum allowable temperature (aux type maximum temperature plus 25 xb°C). Power is disabled to the small heated zones and the oven.

### FAULT 234 Aux temp sensor

Possible cause: Defective aux temp sensor

The auxiliary thermal zone's temperature reading is lower than expected, which indicates a shorted sensor. Power will be turned off for the zone.

### FAULT 237 No line interrupt

Possible cause: Defective main board

The electronics on the main board are not functioning correctly. Power is shut off to the oven and other heated zones. The GC will not be ready.

### FAULT 238 Bad line interrupts

Possible causes: Excessive noise in the power supplied to the instrument
Defective main board

The main board detected an excessive amount of noise in the power supplied to the GC. Power is shut off to the oven and other heated zones. The GC will not be ready.

Turn the GC off and then on again. If the problem was caused by noise in the power supply, the GC will operate normally again. If the error persists, the main board is defective.

### FAULT 239 No mux ADC response
### FAULT 240 Mux ADC offset value

Possible cause: Defective main board

The multiplexer, which processes the GC's electronic signals, is not functioning. Most likely, the multiplexer's circuitry is defective. Power is shut off to the oven and other heated zones. The GC will not be ready.

### FAULT 241 Invalid line sense

Possible cause: Defective main board

The line sense circuitry on the main board is not measuring the line power correctly. Since it is indicating that the power supply is not within acceptable limits, the power to the heated zones is disabled.

**FAULT 242** **Aux3 faulty fact cal**
**FAULT 243** **Aux4 faulty fact cal**
**FAULT 244** **Aux5 faulty fact cal**

Possible cause: Defective auxiliary flow module

The auxiliary flow module's calibration is invalid.

**FAULT 245** **Detector module rev**
**FAULT 247** **Inlet module rev**
**FAULT 249** **Aux module revision**

Possible cause: Flow module version is obsolete and is not compatible with the GC

The flow module's EEPROM is not recognized by the GC. The module is unusable. The version of the module is not compatible with the version of the GC.

**FAULT 250** **Wrong det module**
**FAULT 252** **Wrong inlet module**
**FAULT 254** **Wrong aux module**

Possible causes: Wrong flow module installed for detector/inlet/aux
Defective detector/inlet/aux flow module
Flow module revision is obsolete and not recognized

The flow module installed for the detector/inlet/aux is unusable. The module is not recognized as a valid detector flow module.

**FAULT 255** **Det invalid type**
**FAULT 257** **Inlet invalid type**

Possible cause: The installed detector/inlet flow module is not supported by the GC

The detector/inlet flow module revision is not supported by the firmware installed in the GC.

**FAULT 259** **Det type mismatch**

Possible cause: GC detector type was changed but its board was not replaced or the reverse

The detector flow module type does not match the installed detector electronics board.

**FAULT 262** **Front RS232 failed**
**FAULT 263** **Back RS232 failed**
**FAULT 264** **Sampler RS232 failed**

Possible causes: Defective main board

The RS232 communications on the main board are faulty. Communication between the GC and the other device is halted.

**FAULT 265** **Inlet invalid PID**
**FAULT 267** **Detector invalid PID**
**FAULT 269** **Pneu aux invalid PID**

Possible cause: Defective flow module.

The GC main board has determined that the inlet/detector/aux flow module calibration setpoints are no longer valid. The module is unusable.

**FAULT 270** **Inlet bad checksum**
**FAULT 272** **Detector bad checksum**
**FAULT 274** **Pneu aux bad checksum**

Possible cause: Defective flow module

The inlet/detector/aux flow module's calibration setpoints are out of range and are not valid.

**FAULT 275** **Inlet bad fact cal**
**FAULT 277** **Det bad factory cal**
**FAULT 279** **Pneu aux factory cal**

Possible cause: Defective flow module

The inlet/detector/aux flow module's calibration is invalid.

**FAULT 280** **Inlet I/O failure**
**FAULT 282** **Det I/O failure**
**FAULT 284** **Pneu aux I/O failure**

Possible cause: Defective flow module.

The inlet/detector/aux flow module is malfunctioning. The GC main board could not read/write to the module EEPROM.

**FAULT 289** **Oven fan motor**

Possible causes: Oven fan motor not connected/missing
Oven fan motor defective
Defective main board

The controller for the primary oven fan motor detected a fault, and the oven is shut down for safety.

**FAULT 290** **Zones not updating**

Possible causes: Corrupted firmware
Defective main board

The program that controls one or more of the thermal zones has stopped, and the zones may be uncontrolled. To prevent a thermal runaway, all thermal zones are turned off.

Turn the GC off, then on. If the fault remains, update the GC firmware or replace the main board.

**FAULT 291** **Duct fan**
**FAULT 292** **Inlet fan**

Possible causes:
Fan not connected
Fan defective
Defective main board

During startup, the controller for the cooling fan detected a fault. The GC will not be ready until the problem is fixed and the GC is turned off, then on again.

# Routine Maintenance: General

The procedures described in this section can be performed by the user. They may involve exposure to heated surfaces, but not to hazardous voltages. The general warnings are repeated here for reference.

## General warnings

There are several important safety notices that you should always keep in mind when using the 6850 GC.

### Many internal parts of the GC carry dangerous voltages

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

- The wiring between the GC power cord and the AC power supply, the AC power supply itself, and the wiring from the AC power supply to the power switch.

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

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

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

**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 GC electronics

The printed circuit (PC) boards in the GC 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 operate at temperatures high enough to cause serious burns. These parts include but are not limited to:

- The inlet
- The oven and its contents
- The detector
- The column nuts attaching the column to an inlet or detector
- The valve box

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

**Warning** Be careful when working behind the instrument. During cool-down cycles, the GC emits hot exhaust which can cause burns.

### Insulation fibers can cause irritation

**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; wash your hands with mild soap and cold water after handling the insulation.

## Maintenance schedule

Periodically clean the surface of the instrument with a dampened lintless cloth. Do not get any water inside the instrument.

The frequency of maintenance depends upon:

- The level of usage
- The type of samples injected
- Whether injections are manual or automatic
- Whether the instrument is used for multiple applications or dedicated to one
- Other environmental factors, such as dirt, ambient temperature, etc.

**Table 23. Maintenance Schedule**

| Maintenance frequency | Items |
|---|---|
| Daily | Change septa, run a calibration sample, check the tightness of liner and column nuts.[1] |
| Weekly | Change glass liners and O-rings, if applicable. |
| Monthly | Clean the split/splitless inlet vent line trap.<br>Perform a leak check for hydrogen. Check all connections from the initial supply. At the GC, leak check the inlet and the column connections to the inlet and detector. |
| Quarterly | Renew gas cylinders.[2] |
| Semiannually | Clean detector.<br>If a $\mu$ECD is installed, perform a radioactivity wipe test. |
| Annually | Recondition or replace internal and external traps and chemical filters. |

[1] Very important for temperature programming using Vespel or Vespel/graphite ferrules

[2] With typical usage, A-size cylinders will supply four gas chromatographs for about 3 months. Replace the cylinder when its pressure drops below 500 psig.

### Early maintenance feedback

Four of the most frequently replaced or serviced parts on a GC are the inlet septum, the column, the inlet liner, and the syringe. Using Early Maintenance Feedback (EMF) and a G2629A Control Module, you can independently track usage of these items by number of injections. After your preset limit on the number of injections expires, you receive a warning message on the GC front display. This warning prompts you to replace or service the item, but readiness is not affected. You can still continue using the GC normally.

Always service the GC when not in a run or sequence. For details on how to set up and use EMF, see the G2629A Control Module user information.

## Swagelok connections

These connectors are widely used in gas chromatography. This procedure minimizes dead volume and internal stresses inside swaged fittings.

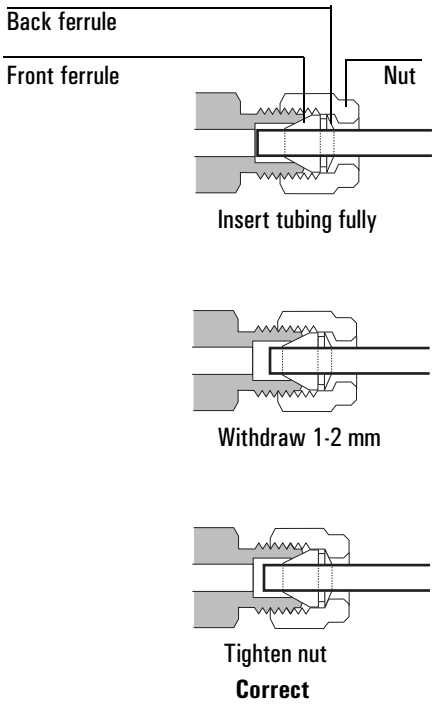



**Figure 14. Swagelok fittings**

1. Secure a new female swage-type fitting in a bench vise.
2. Slide a new nut, back ferrule, and front ferrule onto the tubing. Make sure their order and orientation is correct.

3. Install the assembly onto the vise-held female fitting and tighten the nut finger-tight.
4. Push the tube fully into the female fitting, then withdraw it approximately 1 to 2 mm.
5. While holding the tube in position, use a wrench to tighten the nut further: an additional 3/4 turn for 1/8-inch tubing, or an additional 1-1/4 turn for 1/4-inch tubing.

## Preparing to service the GC

If you can edit setpoints using either a Control Module or a ChemStation/Cerity Chemical, turn off the heated zones you will be working with and wait for them to cool. If you cannot edit setpoints, load the `SERVICE` method.

The factory provides a default `SERVICE` method, which:

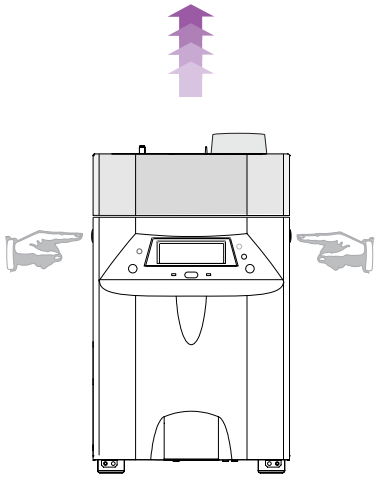
- Resets the oven setpoint to 35°C if greater than 35°C
- Turns inlet, detector, and auxiliary heaters off
- Turns cryogenic cooling off
- Turns the carrier gas off
- Turns the detector off

You can create a more elaborate SERVICE method if you wish using a Control Module or a ChemStation/Cerity Chemical. See their documentation for more detail.

## Opening the oven

1. If an auto injector is present, lift it off its mounting post and place it on the bench top or on the (optional) bracket that mounts on the side of the GC.

2. Press the buttons on each side of the GC just below the lid to release the latches.

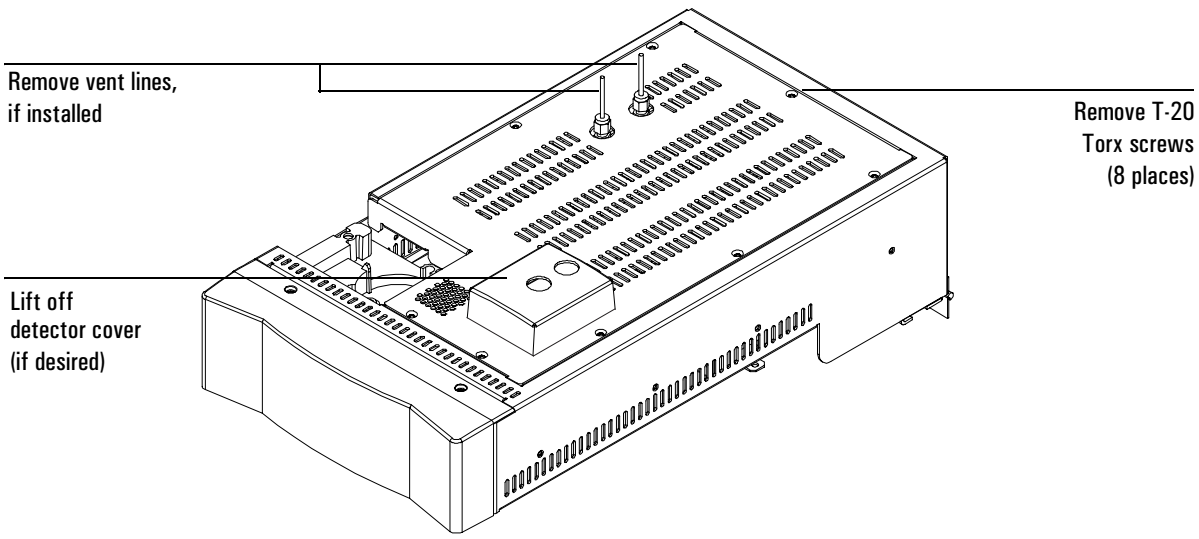


3. Raise the lid.
4. When closing the lid, press down on both top front corners to compress the insulation and engage the latches.

## Removing the lid top cover

To remove the lid top cover, refer to Figure 15.

- For FID, µECD, and TCD, top cover removal is as shown
- For FPD, remove the plastic vent tubing from the detector if it interferes with lid removal. Loosen the thumb screw at the front of the detector cover, and remove the remaining five lid mounting screws.




**Lid top cover for FID/TCD/$\mu$ECD, without valve box accessory, is shown**

**Figure 15. Removing the lid top cover**

## Maintaining a sampling valve (diaphragm type)

This section describes routine maintenance for diaphragm-type sampling valves.

### Replacing the gas sampling valve loop and diaphragm

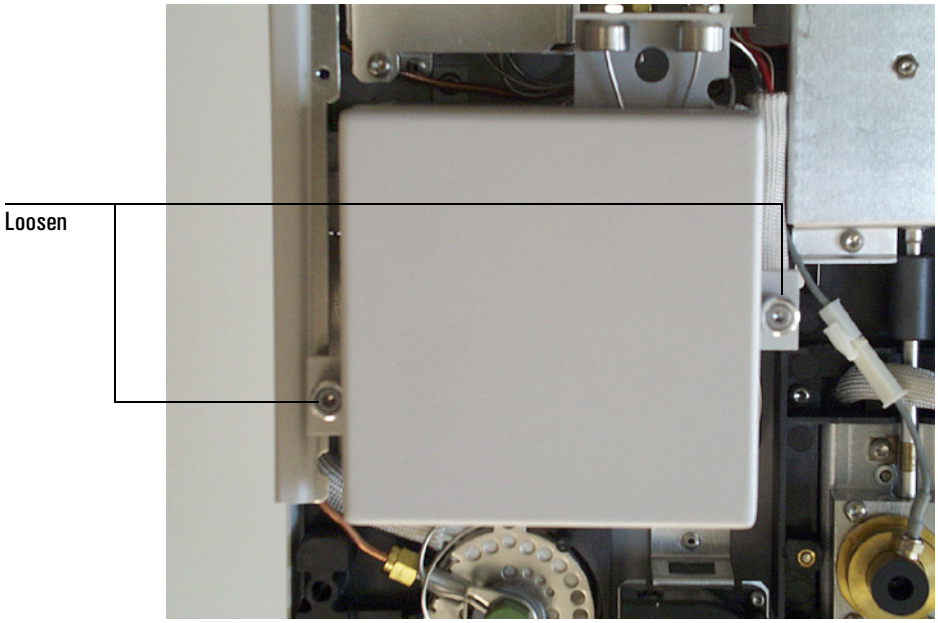
#### Tools and materials

- 9/64-inch hex key (Allen) wrench
- T–10 Torx driver

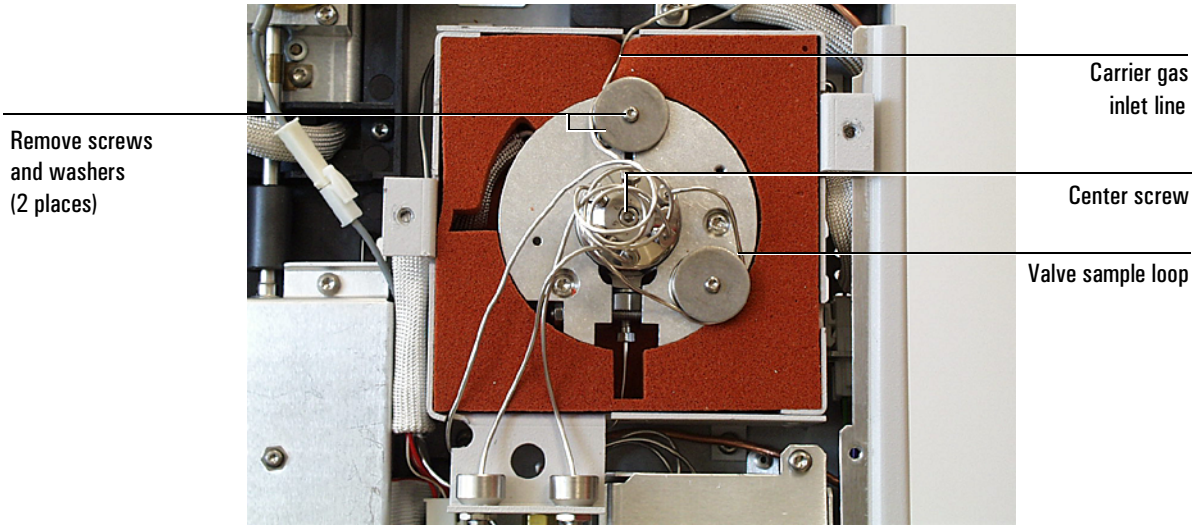
#### Procedure

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off.
2. Turn the carrier gas off at the source.
3. Remove the top cover.

4. Loosen the two valve box cover thumb screws and lift the cover off.

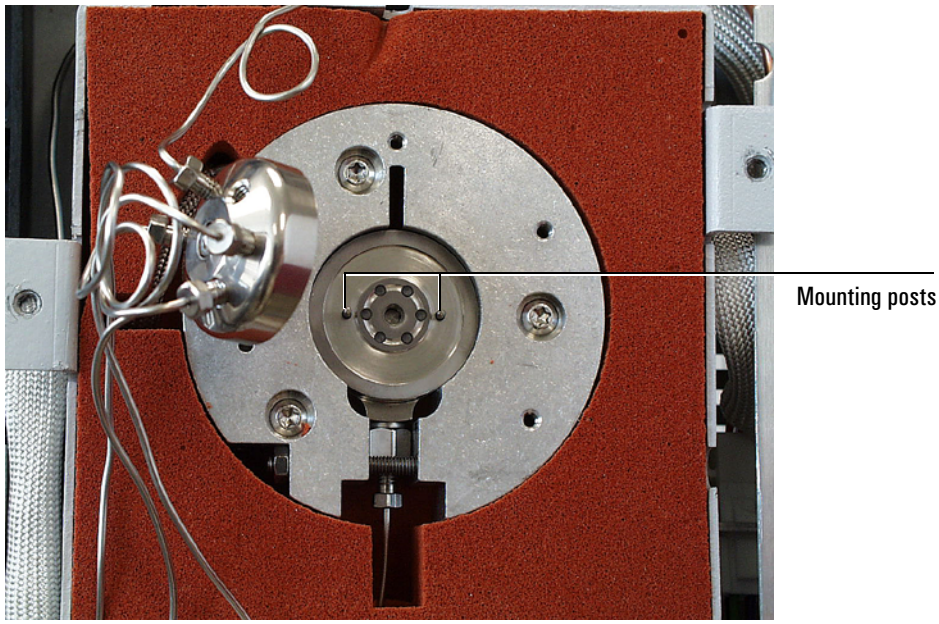



5. Remove the two screws and washers that hold down the valve sample loop and the carrier gas inlet line.




6. Loosen the sample loop's two 1/4-inch fittings on the valve head and lift the loop away from the instrument.

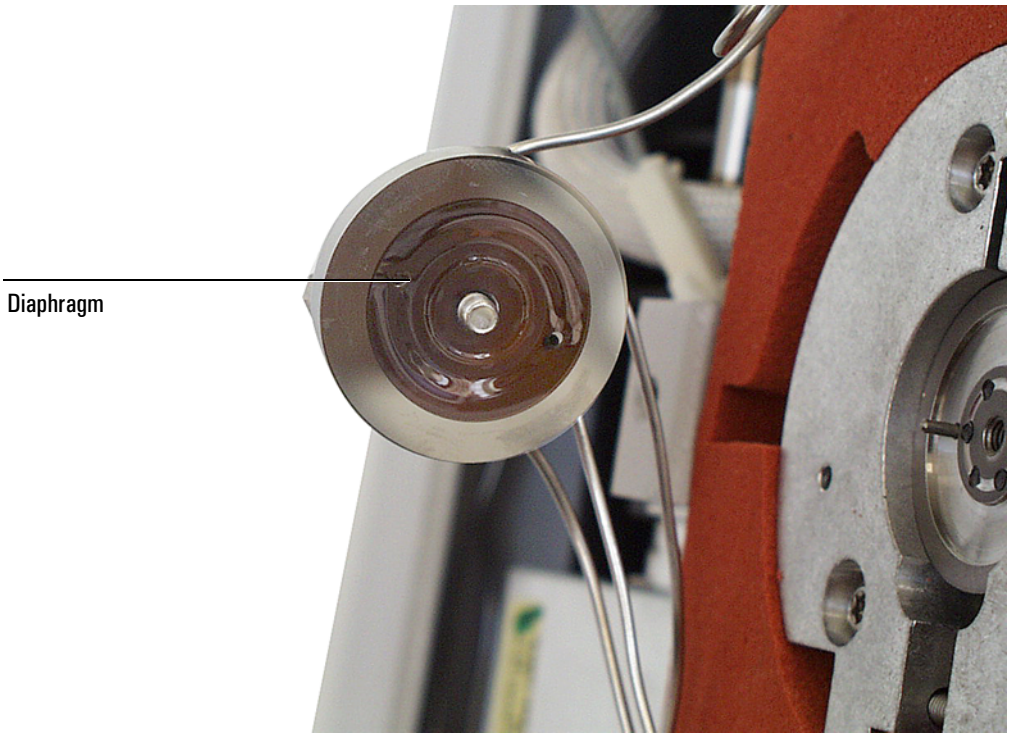
7. Unscrew the center screw in the valve head with the hex key wrench, then remove the valve head.




**Valve head removed**

**Caution** Do not scratch the surface of the valve head.

8. Peel off the diaphragm using forceps.




9. Align the new diaphragm over the mounting posts so that the valve diaphragm channel is oriented toward the shiny valve face.
10. Install the valve head using the center mounting screw.
11. Reinstall the carrier gas inlet line and sample loop.
12. Place the valve box over the assembly, and tighten the two thumb screws.
13. Check for leaks. See "Leak testing the split/splitless inlet" on page 128, or "Leak testing a purged packed inlet" on page 144, or refer to your G2629A Control Module documentation for an automated test.

### Replacing a liquid sampling valve diaphragm

See "Replacing the gas sampling valve loop and diaphragm" on page 106.

## Maintaining a sampling valve (rotary type)

The rotary sampling valve (gas or liquid) is actuated by a pneumatic solenoid controlled by the GC. The left (counterclockwise, CCW) and right (clockwise, CW) stops on general purpose valve bodies limit rotor rotation. The correct flow path results when the index pin is close to or against either stop of the index ring. See Figure 16. Grooves in the rotor surface form the paths between specific ports. The index pin prevents rotation beyond either stop of the index ring. Valve ports are connected by the grooves only when the index pin is close to or against either stop. Intermediate positions result in flow shutoff through the valve and possible damage if left in this position.

**Valve body**

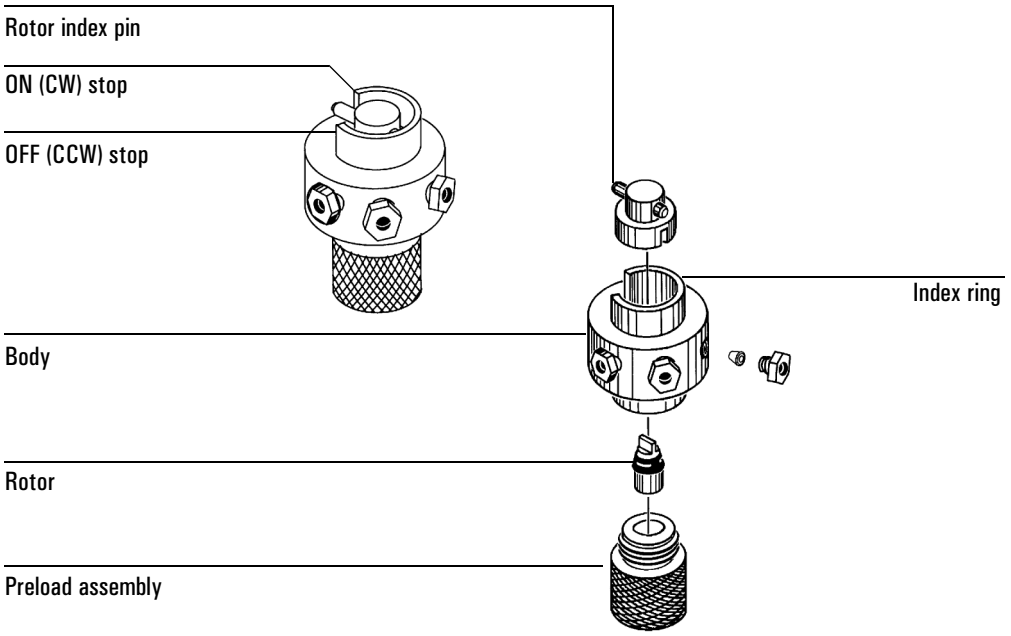



**Figure 16. A typical 6-port rotary valve**

### Aligning the rotor

Typically, the rotor index pin stops near, but does not contact, the CW and CCW stops. Occasionally, however, it may strike one of these stops when engaging or disengaging. Over time, the stop will wear down. This typically causes misalignment of the rotor with the valve ports, which causes flow problems such as low sample amounts, carryover, or restricted carrier gas flow. To correct this problem, realign the valve rotor.

1. Turn off the oven and valve box heater and allow the valve to cool to room temperature.

   Alternately, load the SERVICE method and wait until the GC becomes READY.

2. Using a control module or data system, set the valve to the OFF position.
3. Turn off the air supply to the valve actuator, then loosen the connection to the air supply tubing to relieve the back pressure at the actuator. When the pressure is released, disconnect the supply tube from the valve box. See Figure 17.

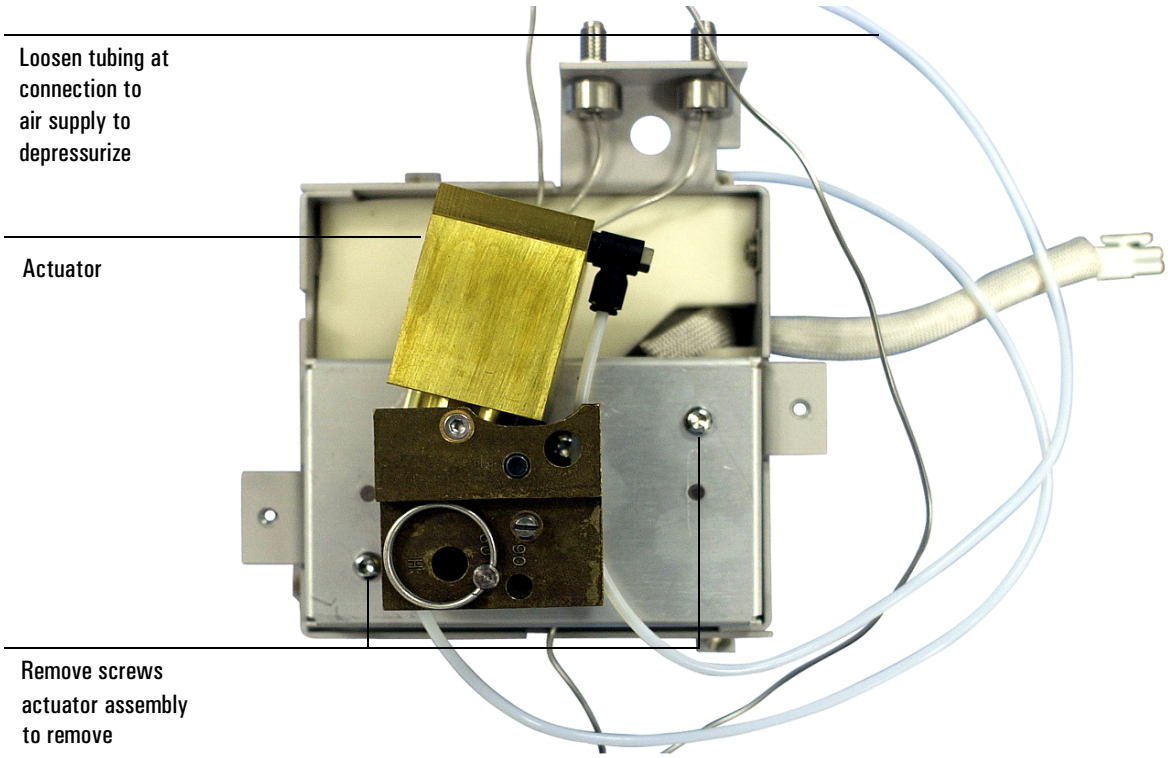



**Figure 17. Rotary sampling valve actuator air supply**

4. Loosen the adjustment set screw. See Figure 18.

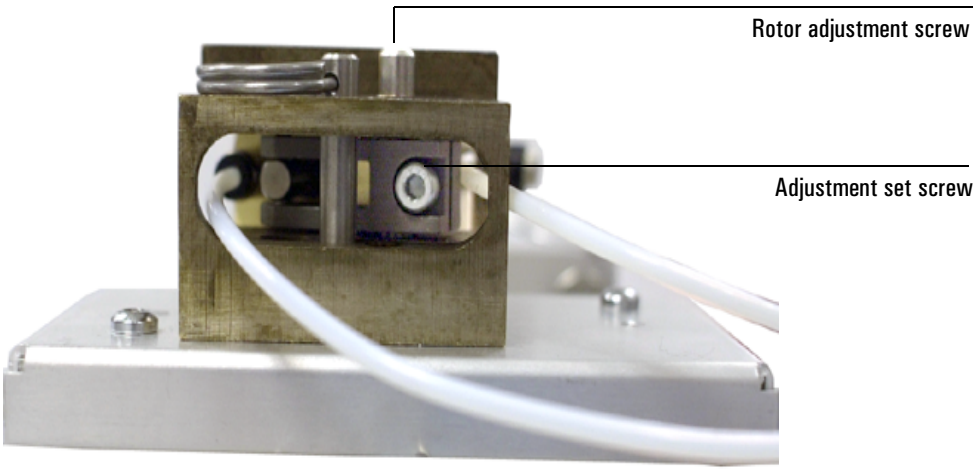



**Figure 18. The adjustment set screw**

5. Locate the rotor adjustment screw on top of the actuator. Using a flat-bladed screwdriver, rotate the valve rotor counter-clockwise until it stops, then back it off a small amount. This sets one end of the rotor's motion. See Figure 18.
6. Tighten the adjustment set screw.
7. Reconnect the air supply and turn it on.
8. Using a control module or data system, turn the valve ON.
9. Remove the two screws that secure the actuator assembly to the valve box, then remove the actuator assembly. Examine the rotor index pin. The rotor index pin should be fully clockwise without touching the stop.
10. Reassemble the valve box.

## Replacing a valve rotor

There are two types of rotors (see Figure 16) available for the 6850 rotary valves. Rotor type can be identified by color:

- An off-white rotor is made of a PTFE composite and may be used from room temperature to 200°C.
- A black rotor is made of polyimide and may be used from 100 to 350°C.

If experiencing leaks due to a worn rotor, or if analyzing a compound that requires a rotor change, replace the rotor as described below.

1. Turn off the oven and valve box heated zone and allow the valve to cool to room temperature.

   Alternately, load the SERVICE method and wait until the GC becomes READY.

2. Turn off the carrier gas and sample line flows and relieve any back pressure to the valve.
3. Remove the valve box cover.
4. Remove the two screws that secure the actuator assembly to the valve box, and lift the actuator off of the valve body. See Figure 19.

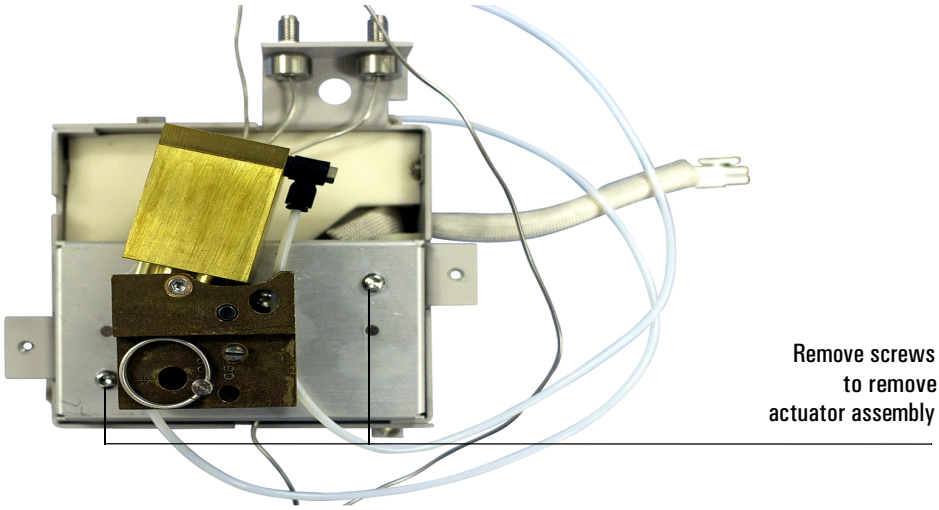



**Figure 19. Removing the valve actuator assembly from the valve box**

5. Lift the valve from the valve box, then unscrew the preload assembly from the valve body. See Figure 16 and Figure 20.

   To avoid potential leaks, try to perform this step without disconnecting the attached tubing.

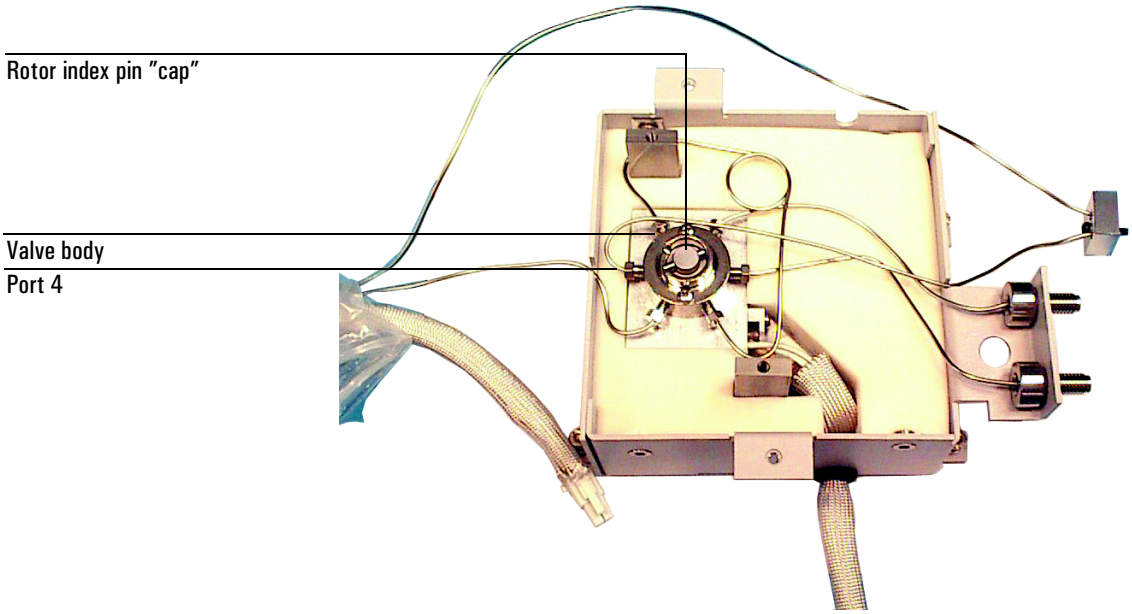



**Figure 20. Valve body in valve box**

6. Lift the index pin “cap” from the valve body, exposing the top of the valve rotor.
7. Press the rotor down and out of the valve body. Sometimes this requires significant force.

**Caution** The valve rotor sits on a highly polished conical surface. Do not scratch this surface.

8. Install the new rotor from the bottom so that the letter stamped in the top of the rotor is nearest port 4 (pointing towards the gap in the index ring). See Figure 21.

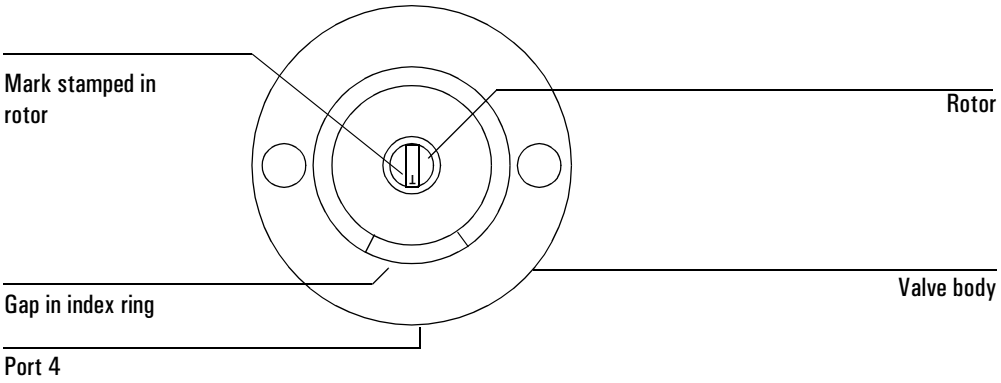



**Figure 21. Top of rotor aligned towards port 4**

9. Place the index pin cap over the valve, then screw the preload assembly onto the bottom of the valve until tight.
10. If disconnected, reconnect the plumbing and check for leaks. Figure 22 shows a typical gas sampling valve installation for reference.

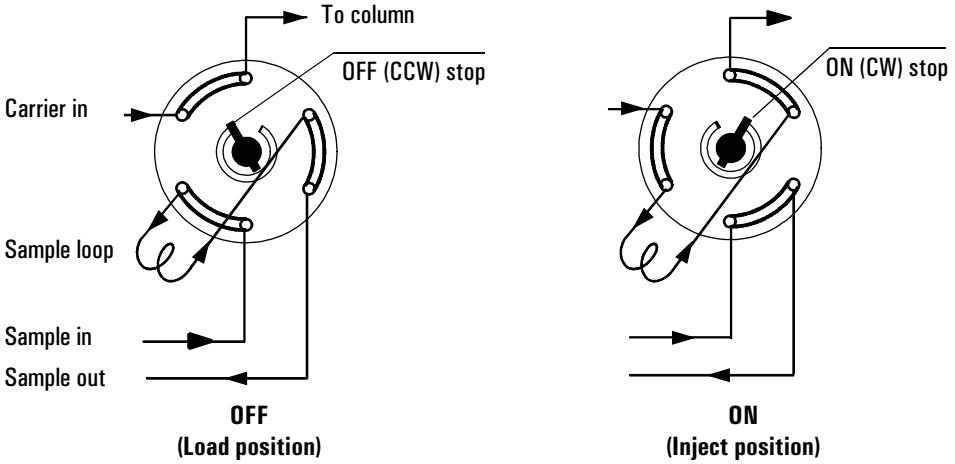



**Figure 22. Gas sampling valve plumbing**

11. Reinstall the actuator assembly.
12. Align the rotor as described in "Aligning the rotor" on page 110.

## Maintaining the auxiliary EPC module

The optional auxiliary EPC module provides three additional auxiliary pressure control channels. To work properly, there must be adequate flow resistance downstream of each channel's pressure sensor. The auxiliary EPC module provides this restriction for each channel via 3 frit-type restrictors located in the manifold block on the EPC module exterior.

Four frits are available. A colored dot distinguishes the different types from each other.

| Frit marking | Flow resistance | Part no. |
|---|---|---|
| Blue Dot | High | 19234-60660 |
| Red Dot | Medium | 19231-60770 |
| Brown Dot | Low | 19231-60610 |
| None (brass tube) | Zero | G1570-20540 |

Agilent ships the Red Dot frit in all three channels of a new auxiliary EPC module.

Select and install frits based on the gas type and flow requirements for the application. Figure 23 and Figure 24 show approximate pressure/flow relationships for the three Dot frits, assuming there is no significant additional resistance downstream of the frits. After changing the type of gas used with the auxiliary EPC module, check these tables to verify the installed frit types. Change the frits if needed.

If using a Zero resistance frit, the user **must** provide flow resistance downstream and generate the pressure/flow relationships.

**Warning** When hydrogen is used, dangerously high flows are possible if insufficient flow resistance is provided downstream of the supply tube. Always use either the High (Blue Dot) or Medium (Red Dot) frit with hydrogen.

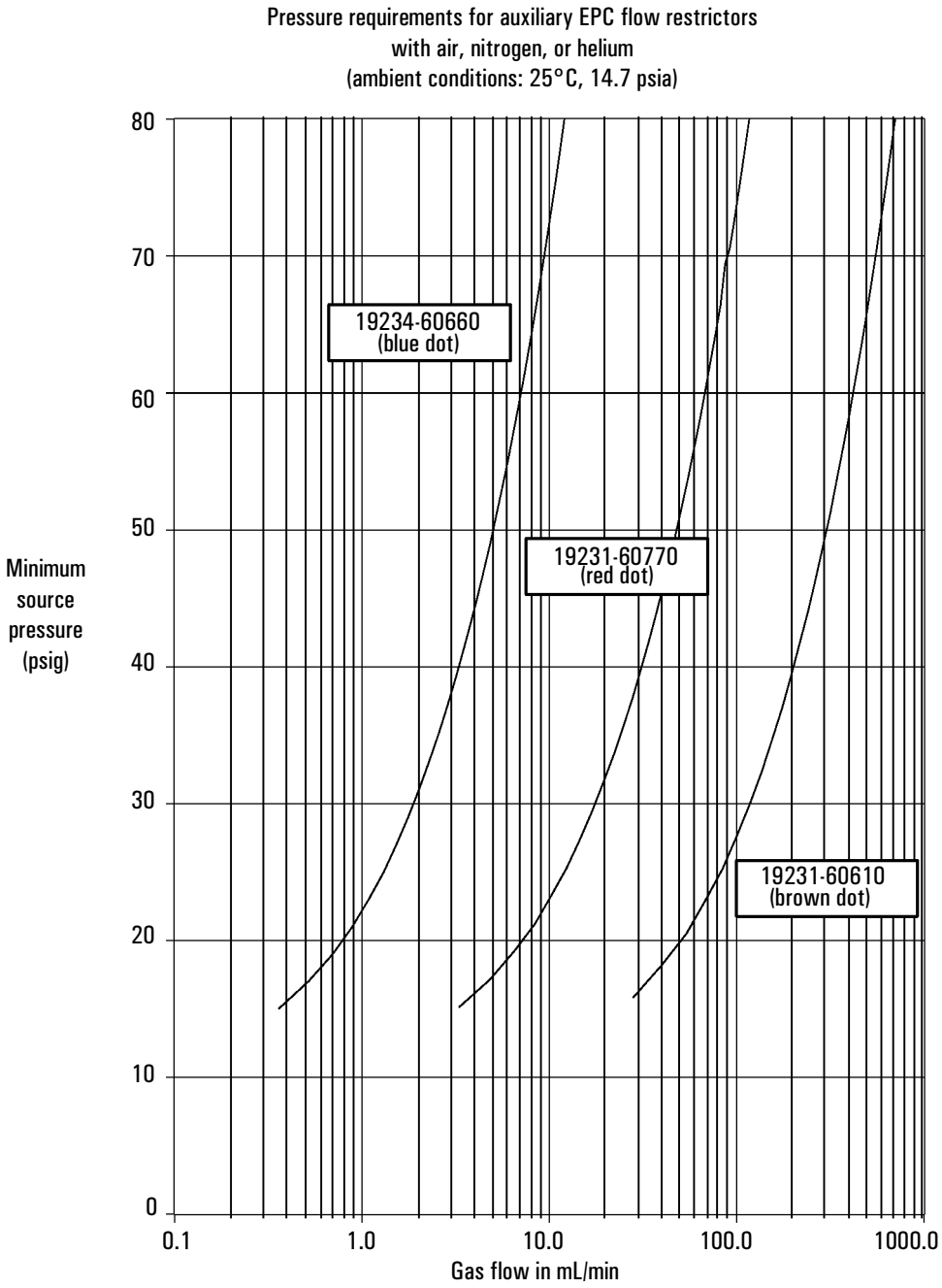



**Figure 23. Pressure requirements for auxiliary EPC flow restrictors with air, nitrogen, and helium**

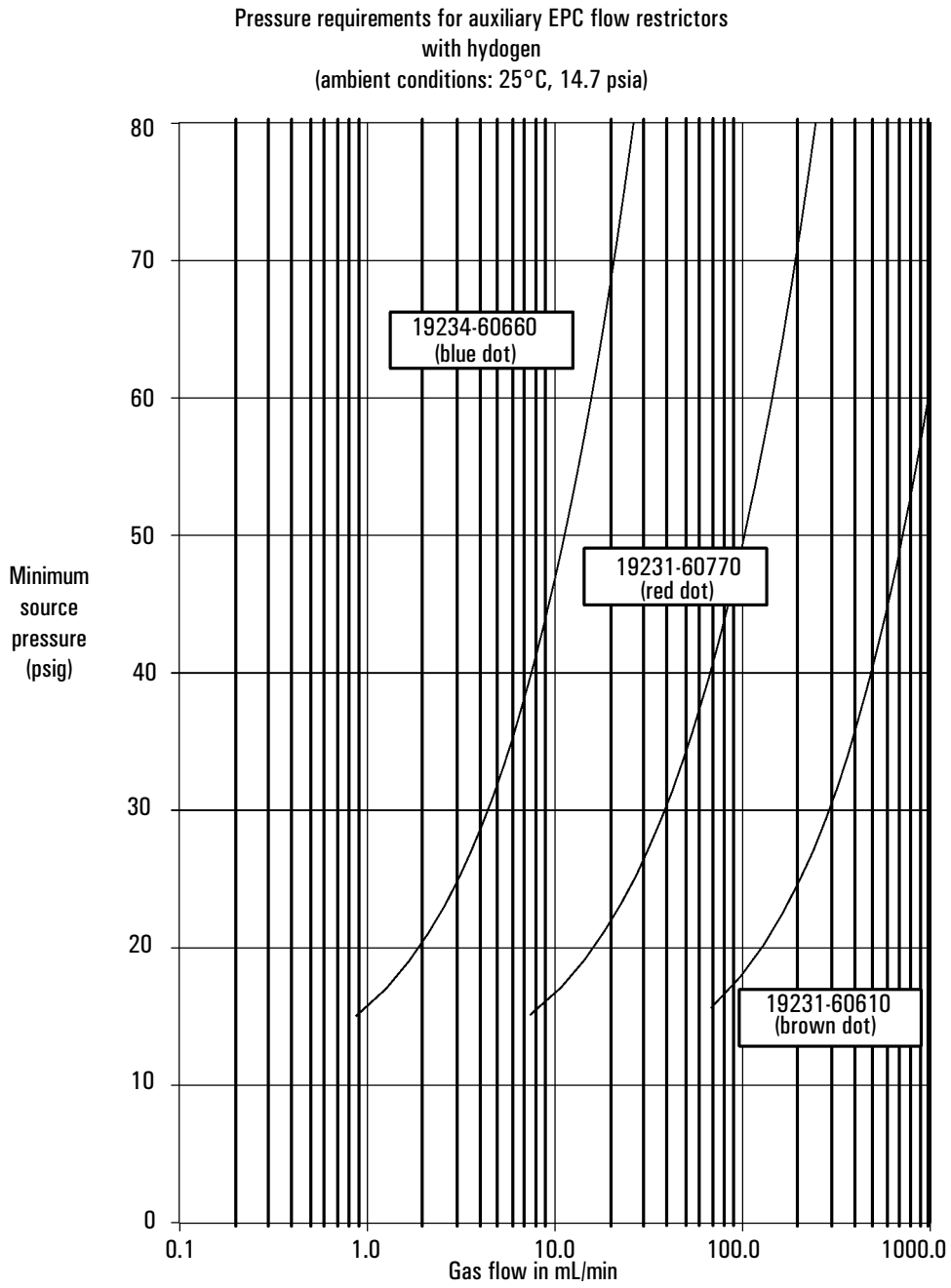



**Figure 24. Pressure requirements for auxiliary EPC flow restrictors with hydrogen**

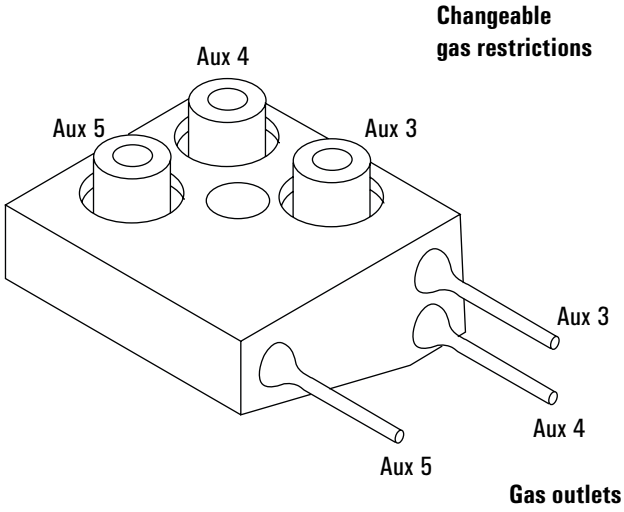
## Changing an auxiliary channel frit

**Warning** The oven exhaust can be hot enough to cause burns. The oven exhaust duct is near the auxiliary flow module. Turn off the oven and allow it to cool before beginning this procedure.

1. Load the SERVICE method or reset the oven temperature to OFF or turn the main power switch off.

**Warning** If using hydrogen gas, turn it off at its source before performing this procedure.

2. Turn any gases used by the auxiliary EPC module off at their sources.
3. Locate the block that connects the three gas outlet tubes for the auxiliary channels to the EPC module.
4. Remove the screw that holds the block to the EPC module. Pull the block free of the module and rotate it so that the frits are on top.




5. Pull the frit to be changed out of the block. Also remove the O-ring that seals it.
6. Place an O-ring on the new frit. Place the O-ring/frit combination in the block.
7. Reconnect the block to the pneumatics module. Tighten the screw firmly.

# Routine Maintenance: Inlets

The procedures described in this section can be performed by the user to maintain the inlets in the GC. They may involve exposure to heated surfaces, but not to hazardous voltages.

## Maintaining a split/splitless inlet

### To install a liner and O-ring

Choose liners according to the type of injection you are doing—split or splitless. Many liners are available and can be ordered from the Agilent catalog for consumables and supplies.

#### Tools and materials

- Liner, part no. 5183–4647 (split) or 5062–3587 (splitless)
- Septum wrench (part no. 19251–00100)
- Viton O-ring (part no. 5180–4182)

#### Procedure

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off.

**Warning** If the inlet cools to room temperature, the liner will probably stick and be broken. The rest of this procedure should be performed with the inlet hot. Use gloves and forceps to avoid burns

2. Turn the carrier gas off at the source.
3. Loosen the insert retainer nut, using a wrench, if needed.
4. Lift the top insert assembly straight up to avoid chipping or breaking the liner.
5. If a liner is present, remove it with tweezers or a similar tool. Be careful not to chip the liner.
6. Place a new Viton O-ring (see "O-Rings for the Split/Splitless Inlet" on page 121) on the new liner about 2 to 3 mm from its top end.
7. Press the liner straight down into the inlet, flush with the top of the weldment.

**Caution** Do not add an O-ring or other seal either at the bottom of the inlet or at the bottom of the liner; this will damage the inlet and shatter the liner.

8. Replace the insert retainer nut, tightening it to firm finger tightness. Do not overtighten.
9. Pressure check the inlet. Retighten if necessary.

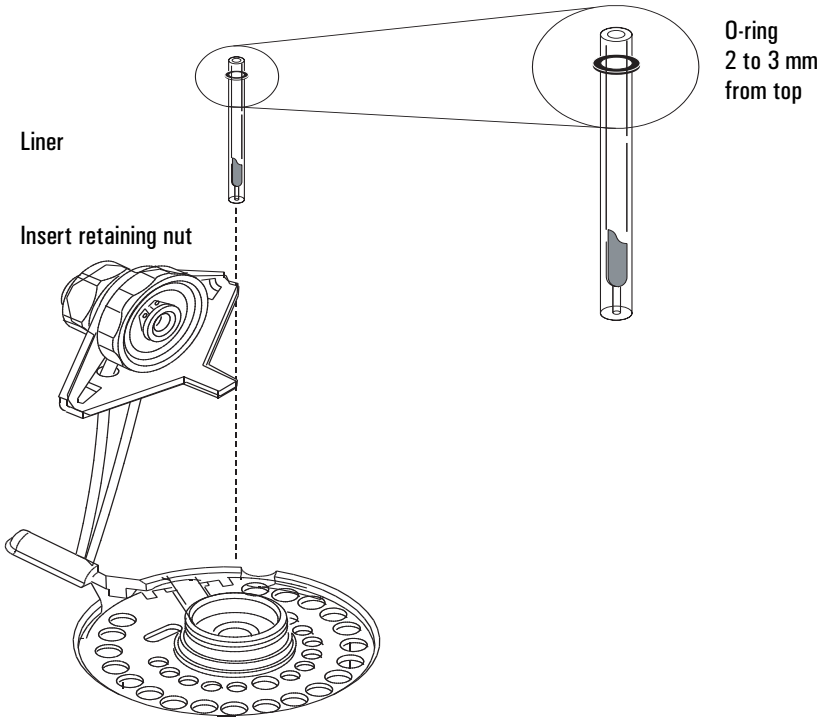



**Figure 25. Installing a liner**

**Table 24. O-Rings for the Split/Splitless Inlet**

| Description | Part no. |
|---|---|
| Viton O-ring for temperatures up to 350°C | 5181–4182 |
| Graphite O-ring for split liner (temperatures above 350°C) | 5180–4168 |
| Graphite O-ring for splitless liner (temperatures above 350°C) | 5180–4173 |

## Changing septa

If a septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Signal noise will increase.

Septum lifetime depends on injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is in steady use, daily septum replacement is recommended.

The type of septa you use depends on your chromatography needs. You can order septa directly from Agilent Technologies; see the Agilent catalog for consumables and supplies for ordering information.

**Table 25. Recommended Septa for the Split/Splitless Inlet**

| Description | Part no. |
|---|---|
| 11-mm septum, low-bleed red | 5181–1263 |
| 11-mm septum with partial through-hole, low-bleed red | 5181–3383 |
| 11-mm septum, low-bleed gray | 5080–8896 |
| Merlin microseal septum | 5181–8815 |
| 11-mm high-temperature silicon septum (350°C and higher) | 5182–0739 |

### Tools and materials

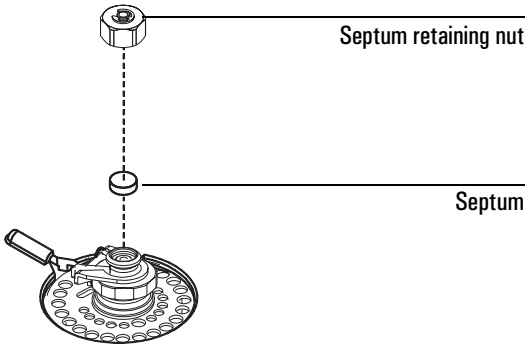
- New septum
- Septum nut wrench (part no. 19251–00100)
- 0- or 00-grade steel wool (optional)

### Procedure

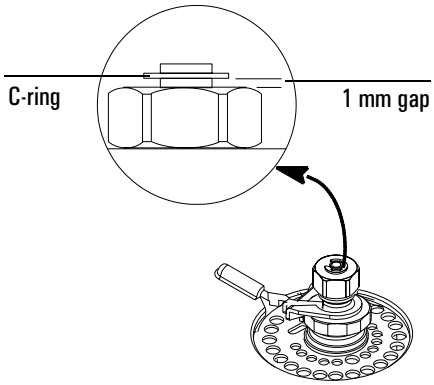
**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

1. Load the `SERVICE` method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.
2. Turn the inlet pressure off at the source.

3. Remove the septum retainer nut, using a wrench if the nut is hot or sticks. Remove the old septum. If the septum sticks, use a sharp tool to remove it. Take care to avoid gouging or scratching the metal.




4. If pieces of the septum stick, use a small piece of rolled-up steel wool and forceps or tweezers to scrub the residue from the retainer nut and septum holder. Use compressed air or nitrogen to blow away the debris.
5. Use forceps to insert a new septum. Press it into the fitting firmly.
6. Replace the septum retainer nut, tightening it until the C-ring is about 1 mm above the nut. Avoid overtightening.




7. Restore normal operating conditions.

## Replacing the inlet flow module

### Tools and materials

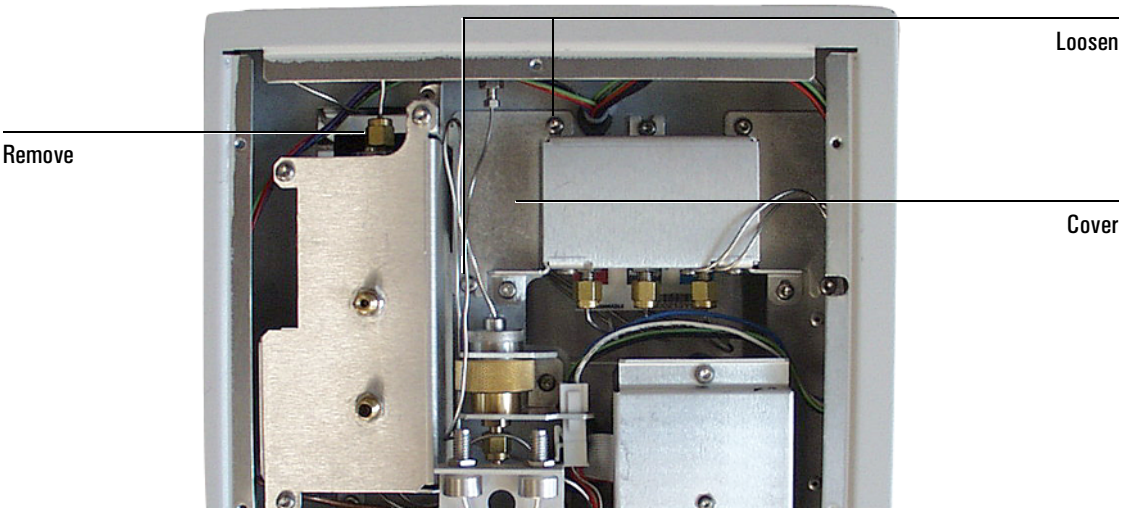
- T-10 and T-20 Torx drivers
- 7/16-inch wrench

### To remove the flow module

**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

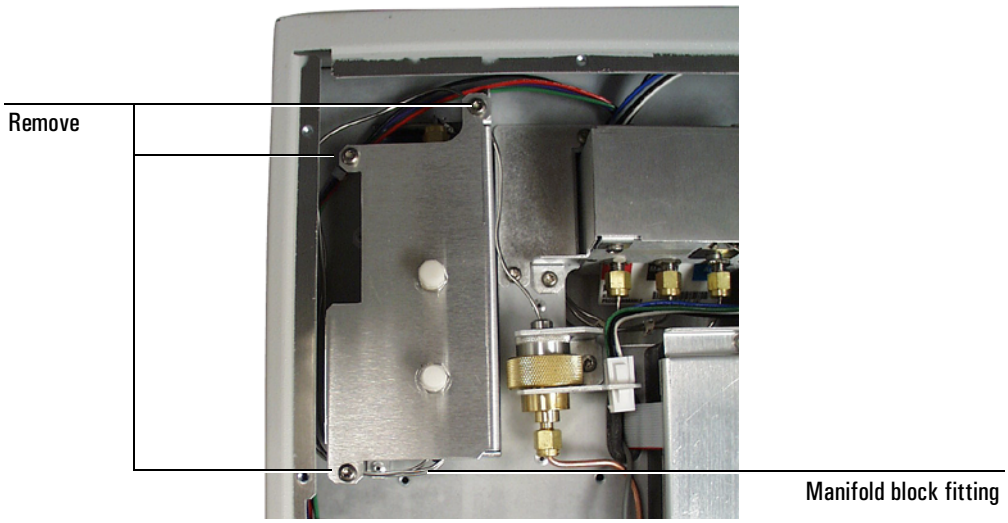
1. Load the `SERVICE` method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.

2. Turn the inlet pressure off at the source.
3. Remove the lid top cover.
4. Loosen the two T-20 Torx screws on the cover plate.




5. Slide the cover plate off to expose the ribbon cable.
6. Remove the gas inlet fitting from the back of the manifold.
7. Release the module ribbon cable connector from the jumper cable by pushing the two tabs down, and disconnect.

8. Remove the three screws that hold the inlet flow module in the lid.




9. If your 6850 has a valve box installed, remove the valve box cover.
10. Lift the inlet flow module out and remove the manifold block fitting from the front of the module.
11. Remove the inlet flow module from the instrument.

### To install the flow module

1. Use a lint free cloth to clean the surface of the manifold fitting O-rings so they will make a good compression seal.
2. Lower the inlet flow module into position and install the manifold block fitting and O-rings.

3. Reconnect the module ribbon cable to the jumper cable.
4. Secure the inlet flow module in place by tightening the three screws.
5. Slide the cover plate in place and tighten the two mounting screws.
6. Reconnect the gas inlet fitting.
7. Check for leaks. See "Leak testing the split/splitless inlet" on page 128 or refer to your G2629A Control Module documentation for an automatic test.

## Replacing the inlet base seal

Replace the inlet base seal whenever you loosen or remove the reducing nut. Chromatographic symptoms such as ghost peaks indicate that the inlet base seal is dirty and needs replacement.

You change the inlet base seal from inside the oven, so you must remove the column.

### Materials

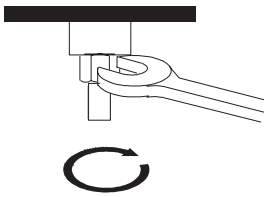
- T-20 Torx screwdriver
- 1/2-inch wrench
- A new washer (part no. 5061–5869)
- Gold-plated seal (part no. 18740–20885) or stainless steel seal (part no. 18740–20880)

### Procedure

**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

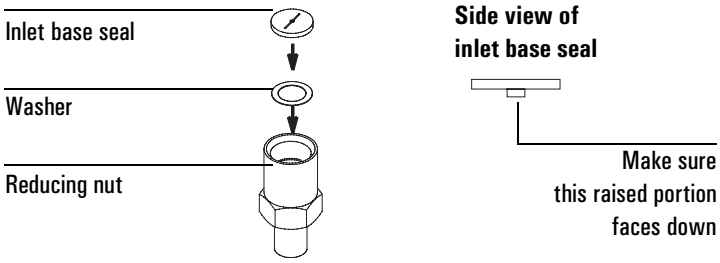
1. Load the `SERVICE` method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.
2. Turn the inlet pressure off at the source.

3. Remove the column from the inlet. Cap the end of the column to prevent contamination. Remove the insulation cup around the base of the inlet.
4. Use a wrench to loosen and remove the reducing nut. The washer and seal are inside the reducing nut. Remove them.



**Caution** Wear gloves to protect the inlet base seal and washer from contamination.

5. Place the washer in the reducing nut. Place the new inlet base seal on top of it.




6. Replace the reducing nut and tighten with a wrench. Replace the column and the insulation cup. After the column is installed, you can restore normal operating conditions.

## Leak testing the gas plumbing

Leaks in the gas plumbing can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the inlet flow manifold. If this portion of the system proves to be leak-free, refer to the next procedure to check the inlet and inlet manifold.

**Caution** Liquid leak detectors are not recommended, especially in areas where cleanliness is very important. If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

### Tools

Electronic leak detector capable of detecting your gas type or liquid leak detection fluid. If you use leak detection fluid, wipe off excess fluid when you have completed the test.

### Procedure

**Warning** To avoid a potential shock hazard when using liquid detection fluid, turn the GC off at the main power switch on the left side and disconnect the main power cord. Avoid spilling leak solution on electrical wiring, especially the detector and inlet heater leads.

1. Use the leak detector to check each connection you have made for leaks.
2. Tighten the leaky connections. Retest the connections; continue tightening until all connections are leak-free.

## Leak testing the split/splitless inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet. If the inlet is leaking, you should use an electronic leak detector to pinpoint the component that is leaking.

If you have access to a Control Module, you can perform this test automatically. See its documentation for details. The Control Module test program will prompt you to complete the hardware tasks outlined below.

### Tools

Septum nut wrench (part no. 19251–00100)

1/8-inch brass nut, 5180-4103

1/8-inch Vespel/graphite blank ferrule, 0100-1372

Column nut, 5181-8830

Vespel/graphite blank ferrule, 5020-8294

### Procedure

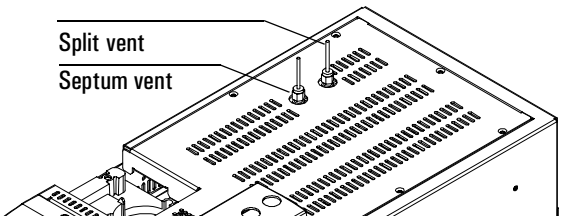
**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

This test requires either a Control Module or a ChemStation/Cerity Chemical to edit setpoints.

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off

2. Turn the inlet pressure off at the source.
3. Remove the column, if installed, and plug the column fitting with the column nut and a no-hole ferrule.
4. Replace the septum (see “Changing septa” on page 121), the liner O-ring (see “To install a liner and O-ring” on page 120), and the inlet base seal (see “Replacing the inlet base seal” on page 126) if their quality is not known.

5. Cap the septum purge fitting with a 1/8-inch Swagelok cap or with a capillary column nut, a solid piece of wire (such as a metal paper clip), and a 0.5 mm id graphite ferrule.




6. Set the oven and inlet to their normal operating temperatures.
7. Set the inlet to Split Mode.
8. Configure the column as 0 m length.
9. Make sure that the pressure at the gas supply is at least 35 psi. Enter a pressure setpoint between 20 and 25 psi, or enter your normal operating pressure if it is greater.
10. Set the total flow to 60 mL/min. Wait a few moments for the pressure and flow to equilibrate. If the GC cannot reach the setpoint, there is a large leak somewhere. See "Correcting leaks" on page 130.
11. Turn either the pressure or the flow `Off`. Because the septum purge and the column fittings are capped, gas is trapped in the system and the pressure should remain fairly constant.
12. Monitor pressure on the Control Module or ChemStation/Cerity Chemical for 10 minutes.
    - A pressure drop of 0.5 psi or less (approximately 0.05 psi/min) is acceptable
    - If the drop is greater than 0.5 psi, there is a leak that must be found and corrected. See "Correcting leaks" on page 130.
    - If the pressure rises, there is probably a leak in the in the flow manifold across the forward pressure proportional valve. Although a slight leak here does not cause a chromatographic problem, it may obscure other small leaks that let air get into the system. The forward valve can leak at 0.2 mL/min and be within specifications.
13. When the system is leak-free, restore normal connections and operating conditions.

### Correcting leaks

**Materials needed:**

- Electronic leak detector suitable for the gas type
- Tools to tighten parts of the inlet that leak (if leaks are detected)

1. Use an electronic leak detector to check all areas of the inlet that are potential sources of a leak.
2. Tighten loose connections to correct leaks, if necessary. You may need to repeat the leak test.

If the pressure drop is now less than 0.5 psi, you can consider the inlet system leak-free. If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak free, but the inlet system is still losing too much pressure, you may need to replace the inlet manifold.

### Potential leak points

Check the following areas (as applicable) when checking an inlet system for leaks.

- The plugged column connection
- The septum and/or septum nut
- The cooling tower assembly
- The ¼-inch ferrule (if a liner is used)
- The O-rings and connections
- The area where gas line(s) are plumbed to the inlet
- The capped purge vent
- The knurled nut
- The septum purge cap (septum head only)
- The connections for the carrier gas and septum purge (septum head only)
- The lower inlet seal at the bottom of the inlet
- The inlet bottom cap

## Checking the split vent line for restrictions

Over time, the split vent line (including the trap) can become obstructed with condensed vapors and other debris. If you have access to a Control Module, you can perform an automated test to check for split vent line restrictions that may cause problems in using the inlet. See the Control Module documentation for details. The Control Module test program will prompt you to complete hardware tasks similar to the ones described in "Leak testing the split/splitless inlet" on page 128.

## Cleaning the inlet

While the inlet will seldom require thorough cleaning, deposits from injected samples occasionally do build up inside the inlet. Before cleaning the inlet, replace dirty inlet liners and inserts with clean ones. If changing them does not correct the problems, then clean the inlet.

### Tools

Cleaning brushes—The FID cleaning kit contains appropriate brushes (part no. 9301–0985)

### Procedure

1. Complete the following preliminary steps:
   - Turn off flows to the inlet at the source
   - Turn off the GC (power switch on left side) and unplug it
   - Remove the inlet liner (see "To install a liner and O-ring" on page 120)
   - Remove the inlet base seal (see "Replacing the inlet base seal" on page 126)
2. Illuminate the inside of the inlet from below and look for signs of contamination or deposits. Insert the brush into the inlet. Scrub the interior walls of the inlet vigorously to remove all deposits.
3. Blow out loose particles and dry thoroughly with clean compressed air or nitrogen before reassembling.
4. Reassemble the inlet using a new inlet base seal. Restore to normal operating conditions.

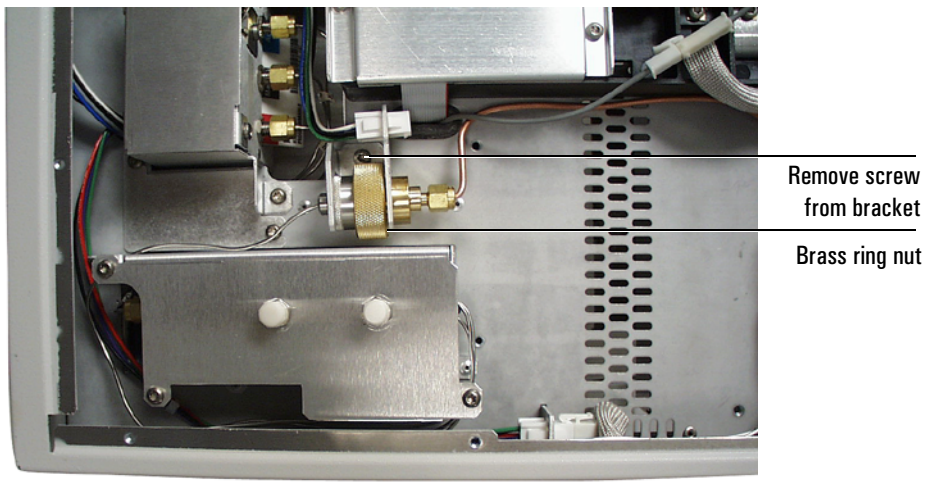
## Replacing the split vent trap filter cartridge

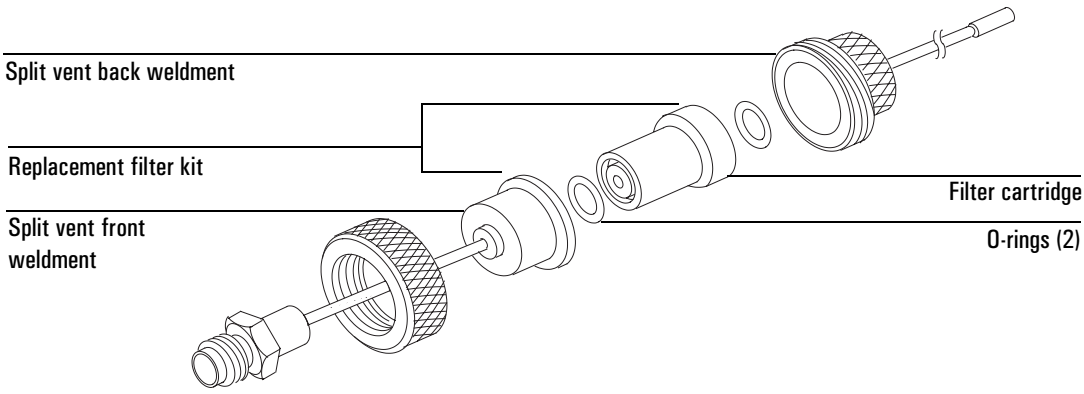
### Tools and materials

- T-20 Torx driver
- Replacement filter kit

**Warning** The split vent trap may contain residual amounts of any samples or other chemicals you have run through the GC. Follow appropriate safety procedures for handling these types of substances while replacing the trap filter cartridge.

1. Load the SERVICE method, or reset the inlet and oven temperatures to OFF, or turn off the main power switch. Let the heated zones cool to room temperature.
2. Remove the lid top cover.
3. Remove the screw that holds down the split vent trap clamp and remove the clamp.
4. Unscrew the brass ring nut and remove the cartridge assembly (the O-rings and the filter cartridge).






5. Replace the two O-rings and filter cartridge.
6. Check for leaks.

# Maintaining a purged packed inlet

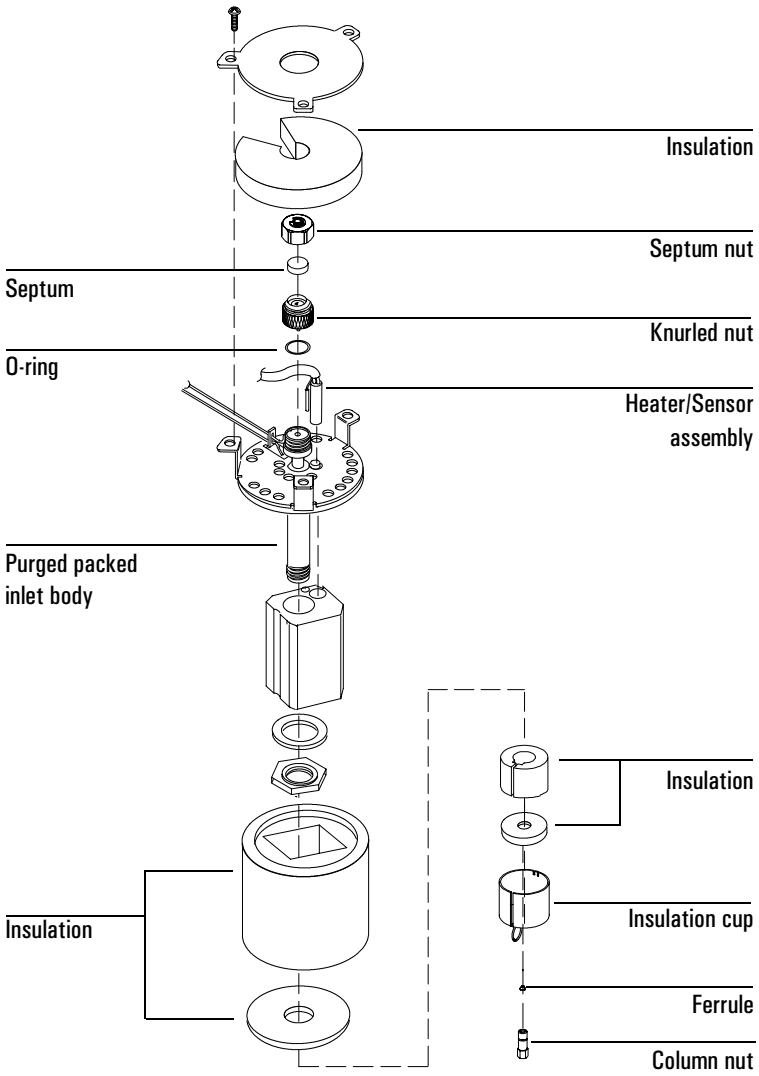



**Figure 26. The purged packed inlet**

## Liner and insert choices

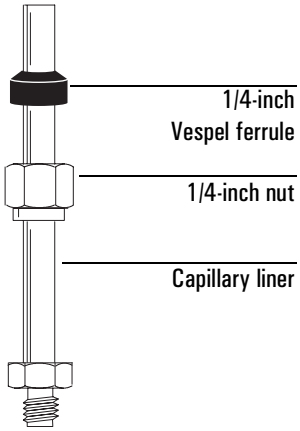
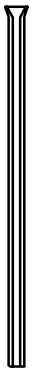
***Liners***. Your choice of liner depends on the type of column you are using. Liners are available for use with wide-bore capillary, 1/4-inch packed, or 1/8-inch packed columns. The liner functions as an adapter so that columns can be connected to the inlet. See "Installing a liner" on page 137.

***Inserts***. Glass inserts are often used with metal liners to reduce reactivity and trap nonvolatile residues. Always use them with capillary columns. Inserts are installed from the top of the inlet and should be installed before the column. See "Installing a glass insert" on page 138.

The purged packed inlet is shipped with a liner and insert for use with capillary columns; see Table 26. If you are using packed columns, consult Table 27.

Narrow-bore capillary columns are not recommended for use with this inlet.

**Table 26. Liner and Insert for Wide-Bore Capillary Columns**

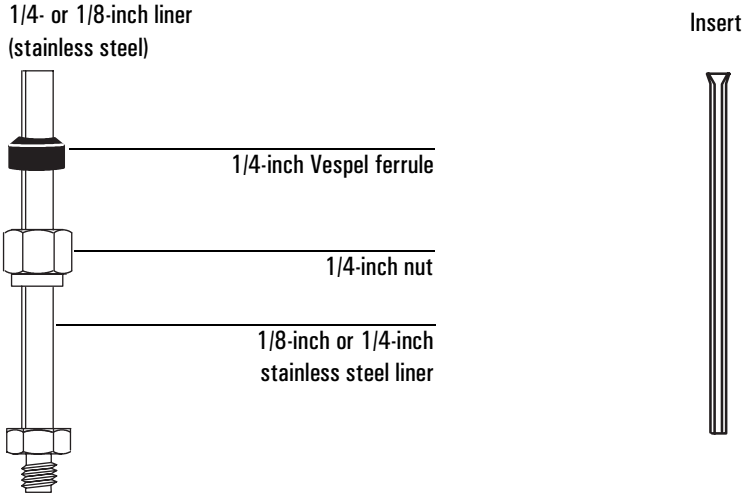
| Column type | Liner | Insert |
|---|---|---|
| 530 μm or 320 μm | 19244-80540   | 5080-8732 or 5181-3382*  |

*Deactivated

## Table 27. Liners and Inserts for Packed Columns

| Column type | Liner | Insert |
|---|---|---|
| 1/8-in. metal | 1/8-inch stainless steel<br>19243-80510 | None |
| | 19243-80530 | 5080-8732 or<br>5181-3382* |
| 1/4-inch metal | 1/4-inch stainless steel<br>19243-80520 | None |
| | 19243-80540 | 5080-8732 or<br>5181-3382* |
| 1/4-inch glass | No liner required. Column end functions as liner. Can also use 1/4-inch metal liner. | Not applicable |

*Deactivated



### Installing a liner

Use these instructions for installing all liner types. Graphitized Vespel ferrules are recommended because metal ferrules tend to lock permanently onto the liner. If a leak develops when using metal ferrules, you must replace the entire liner.

**Materials needed:**

- Liner, brass nut, and ferrule (see Table 26 or Table 27)
- Lint-free cloth
- Methanol
- 9/16-inch wrench

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.
2. Turn the inlet pressure off at the source.

**Warning** Be careful. The oven and inlet fittings may be hot enough to cause burns.

3. If a column is installed, open the GC lid and remove the column from the inlet.
4. If present, remove the old liner with a wrench.
5. Clean the end of the new liner with a lint-free cloth to remove contamination such as fingerprints. Use methanol as a solvent.
6. Place a brass nut and graphitized Vespel ferrule on the liner.
7. Locate the inlet base and insert the liner straight into the inlet base as far as possible.
8. Hold the liner in this position and tighten the nut finger tight.
9. Use a wrench to tighten the nut an additional 1/4 turn.
10. Install the column.

11. Establish a flow of carrier gas through the inlet, and heat the oven and inlet to operating temperatures. Allow these to cool, and then retighten the fittings.

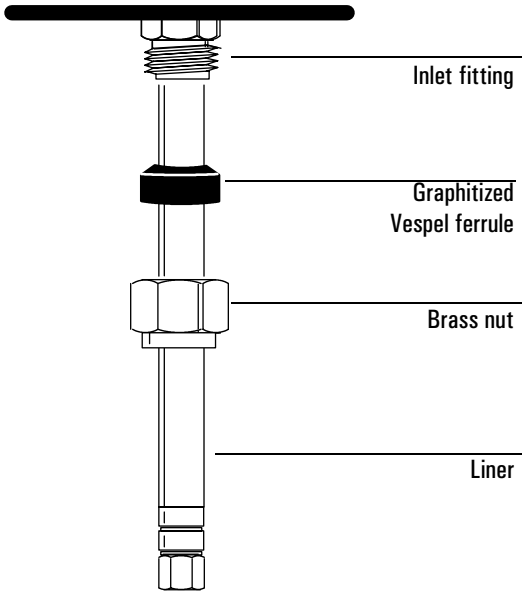



**Figure 27. Installing a liner**

## Installing a glass insert

**Materials needed:**

- Insert (see Table 26 or Table 27)
- Tweezers or hemostats
- Wire

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.
2. Turn the inlet pressure off at the source.

**Warning** Be careful. The inlet fittings may be hot enough to cause burns.

3. Remove the knurled nut at the top of the inlet. See Figure 28.

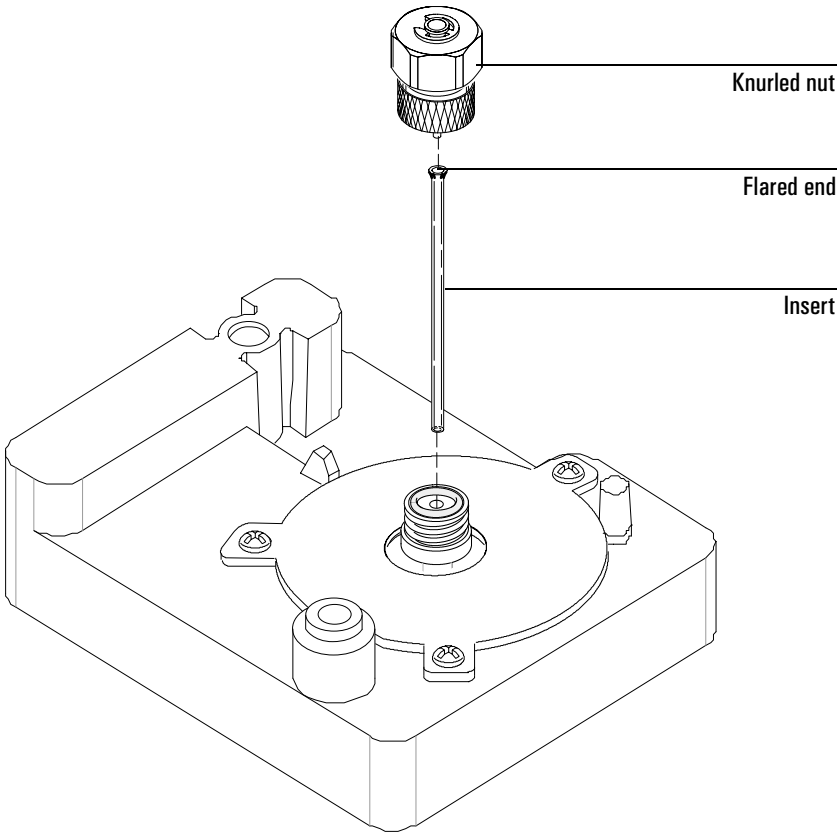



**Figure 28. Installing a glass insert in a purged packed inlet**

4. Carefully remove the old insert. A thin wire (such as a paper clip) may be helpful when lifting the insert from the inlet. See Figure 28.
5. Using tweezers or similar tool, grasp the top of the insert and install in the inlet with the flared end up.
6. If a capillary column is installed and the insert does not seat properly, you must remove the capillary column, install the insert, and replace the column.
7. Reinstall the knurled nut and tighten finger tight.

## Changing septa

If the septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Additionally, the detector signal will become increasingly noisy.

The useful lifetime of septa is determined by injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is used regularly, daily septum replacement is recommended.

The type of septa you use will depend on your chromatography needs. You can order septa directly from Agilent Technologies; see the Agilent catalog for consumables and supplies for ordering information.

**Table 28. Recommended Septa for the Purged Packed Inlet**

| Description | Part no. |
|---|---|
| 11-mm septum, low-bleed red | 5181-1263 |
| 11-mm septum with partial through-hole, low-bleed red | 5181-3383 |
| 11-mm septum, low-bleed gray | 5080-8896 |
| Merlin microseal septum | 5181-8815 |
| 11-mm high-temperature silicon septum (350°C and higher) | 5182-0739 |

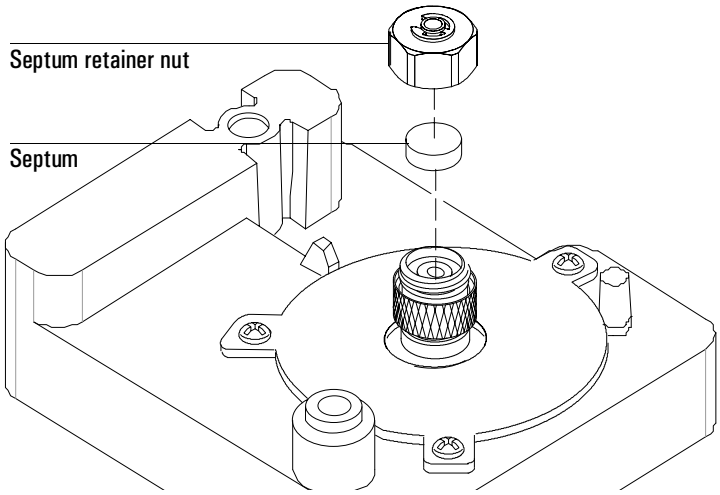
**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

**Caution** Column flow is interrupted while changing septa; since columns may be damaged at elevated temperatures without carrier flow, cool the oven to room temperature before proceeding.

**Materials needed:**

- Gloves (if the inlet is hot)
- New septum
- Septum nut wrench (part no. 19251-00100)
- A plastic or wood tool with a sharp tip to remove septum from inlet
- 0- or 00-grade steel wool (optional)
- Forceps or tweezers
- Compressed, filtered, dry air or nitrogen (optional)

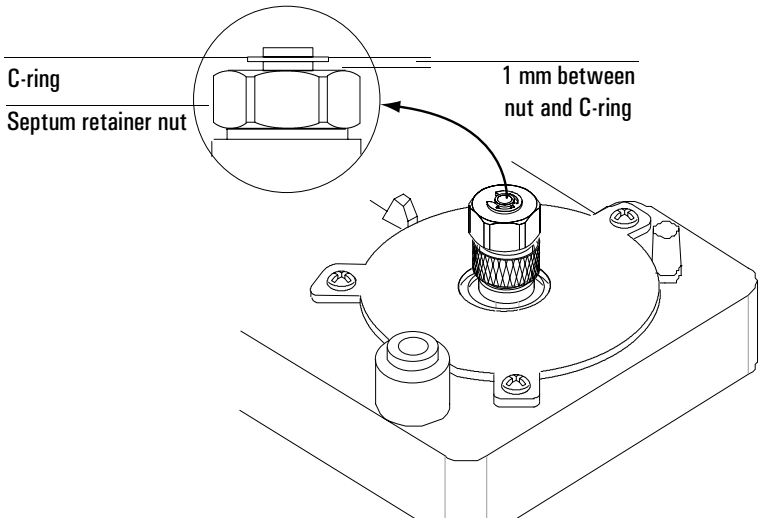
1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off
2. Turn the inlet pressure off at the source.
3. If the inlet is hot, wear gloves to protect your hands from burns. Remove the septum retainer nut, using the wrench to loosen or remove the nut if it is hot or sticks. Remove the old septum.




   If the septum sticks, use a sharp-tipped tool to remove it. Take care not to gouge the metal around the septum and make sure you remove all pieces of the old septum.
4. If pieces of the septum are sticking, grasp a small piece of steel wool with the forceps or tweezers and scrub the residue from the retainer nut and septum holder. Use compressed air or nitrogen to blow away the pieces of steel wool and septum.
5. Use the forceps to insert a new septum. Press it into the fitting firmly.

6. Replace the septum retainer nut, tightening it finger-tight until the C-ring is approximately 1 mm above the nut. Avoid overtightening

**Caution** Do not overtighten the nut. Over-tightening over-compresses the septum, which could cause premature failure and pieces of septum to contaminate the inlet.




7. Restore normal operating conditions.

### Changing the O-ring

You will need to change the O-ring periodically because it wears out and becomes a source of leaks in the inlet. To determine if the O-ring leaks, perform the leak test presented later in this chapter.

O-rings contain plasticizers that give them elasticity. The O-ring seals the top of the inlet and the inlet base. However, at high temperatures the plasticizers bake out, and the O-rings become hard and are unable to create a seal (this is referred to as “taking a set”). If you operate the inlet at high temperatures, you will probably need to replace the O-ring frequently.

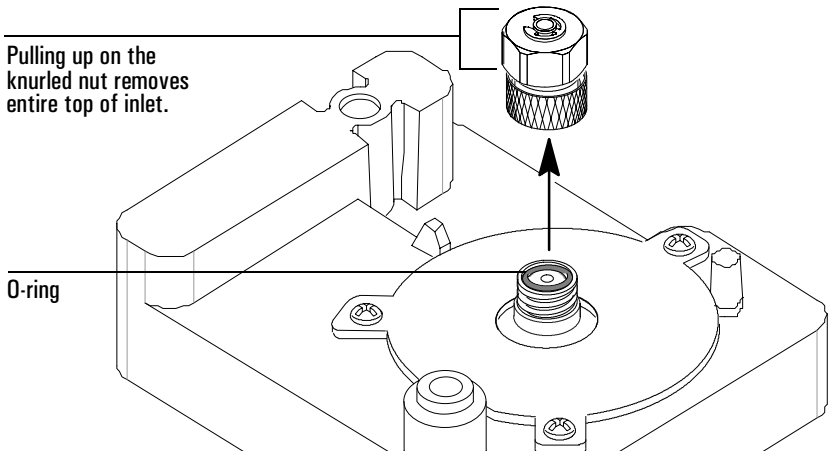
**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns. If the inlet is hot, be sure to wear gloves to protect your hands.

**Materials needed:**

- Gloves (if the inlet is hot)
- A new Viton O-ring (part no. 5080-8898)
- Septum nut wrench (part no. 19251-00100)
- Forceps or tweezers (optional)

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off
2. Turn the inlet pressure off at the source.
3. If the inlet is hot, use the septum nut wrench. Loosen the knurled nut completely. Pull up on the nut to remove the top portion of the inlet.

   The O-ring will be visible. Remove the old O-ring. You may need to use forceps to grab it. Using the tweezers, insert the new O-ring.




4. Replace the top portion of the inlet and tighten the knurled nut until you cannot tighten it further. Restore the GC to normal operating conditions.

### Leak testing the gas plumbing

Leaks in the gas plumbing system can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the inlet flow manifold. If this portion of the system proves to be leak-free, refer to the next procedure to check the inlet and inlet manifold.

**Caution** Liquid leak detectors are not recommended, especially in areas where cleanliness is very important. If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

**Tools:**

- Electronic leak detector or liquid leak detection fluid. If you use leak detection fluid, wipe off excess fluid when you have completed the test.
- Two 7/16-inch wrenches

**Procedure**

**Warning** To avoid a potential shock hazard when using liquid detection fluid, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads, especially the detector and inlet heater leads.

1. Using the leak detector, check each connection you have made for leaks.
2. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.

### Leak testing a purged packed inlet

This procedure allows you to determine if the inlet is leaking. It is recommended that you leak test the inlet at your normal operating temperature since the O-ring may leak if it is cooled to ambient.

If you have access to a Control Module, you can perform this test automatically. See its documentation for details. The Control Module test program will prompt you to complete the hardware tasks outlined below.

**Materials needed:**

- Gloves (if the inlet is hot)
- Septum nut wrench (part no. 19251-00100)
- Clock or watch
- 1/8-inch brass nut, 5180-4103
- 1/8-inch Vespel/graphite blank ferrule, 0100-1372

*If you are using capillary columns:*

- No-hole ferrule, 5020-8294
- Column nut, 5181-8830
- 7/16-inch wrench

*If you are using packed columns:*

- Solid Vespel plug, 0100-1372
- 9/16-inch wrench

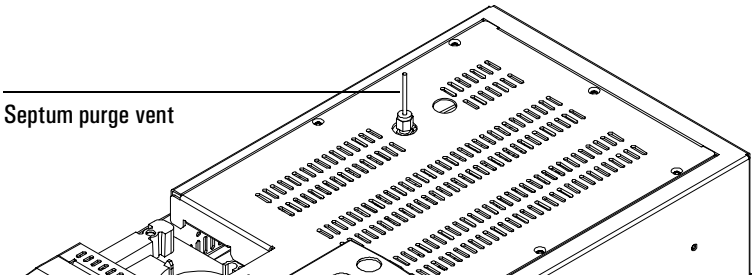
**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

This test requires either a Control Module or a ChemStation/Cerity Chemical to edit setpoints.

### Procedure

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off
2. Turn the inlet pressure off at the source.
3. Remove the column, if one is installed, and cap the column fitting. If you are using a capillary column, insert a no-hole ferrule in the column nut to create a plug. If you are using a packed column, use the Vespel plug.
4. Remove the old septum and replace it with a new one. For instructions on changing septa, see "Changing septa" on page 139.
5. Inspect the O-ring and replace it if it is hard and brittle or cracked. See "Changing the O-ring" on page 142 for instructions on changing the O-ring.

6. Cap the septum purge fitting with a 1/8-inch Swagelok cap or with a capillary column nut, a solid piece of wire (such as a metal paper clip), and a 0.5 mm id graphite ferrule.




7. Make sure that the pressure at the gas supply is at least 35 psi. Carrier source pressure should always be at least 10 psi greater than the desired inlet pressure.
8. Use a Control Module or ChemStation/Cerity Chemical to define a capillary column. Enter a length of 0 m and any diameter. The inlet will now be in pressure control mode.
9. Set the inlet and oven to normal operating temperature.
10. Set the inlet pressure to 25 psi. Wait a moment for the pressure to equilibrate. The pressure may exceed the setpoint briefly while it equilibrates.

    If the pressure cannot be reached, either there is a gross leak in the system or the source pressure is not high enough.
11. Turn the pressure off. Because the column and septum purge vent are capped, the pressure should remain fairly constant.
12. Monitor the pressure for 10 minutes.
    - If there is a pressure loss of less than 0.7 psi (0.07 psi/min or less), consider the system leak tight
    - If the pressure drop is much greater than 0.7 psi, there is a leak that must be found and corrected. See "Correcting leaks" on page 147.
13. When the system is leak free, restore the GC to your normal operating conditions. Remove the cap from the septum purge, install the column, define/undefine the correct column, and restore operating temperatures and pressures.

### Correcting leaks

**Materials needed:**

- Electronic leak detector suitable for the gas type
- Tools to tighten parts of the inlet that leak (if leaks are detected)

1. Use an electronic leak detector to check all areas of the inlet that are potential sources of a leak.
2. Tighten loose connections to correct leaks, if necessary. You may need to repeat the leak test.

If the pressure drop is now less than 0.7 psi, you can consider the inlet system leak-free. If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak free, but the inlet system is still losing too much pressure, you may need to replace the inlet manifold.

### Potential leak points

Check the following areas (as applicable) when checking an inlet system for leaks.

- The plugged column connection
- The septum and/or septum nut
- The cooling tower assembly
- The ¼-inch ferrule (if a liner is used)
- The O-rings and connections
- The area where gas line(s) are plumbed to the inlet
- The capped purge vent
- The knurled nut
- The septum purge cap (septum head only)
- The connections for the carrier gas and septum purge (septum head only)
- The lower inlet seal at the bottom of the inlet
- The inlet bottom cap

## Cleaning the inlet

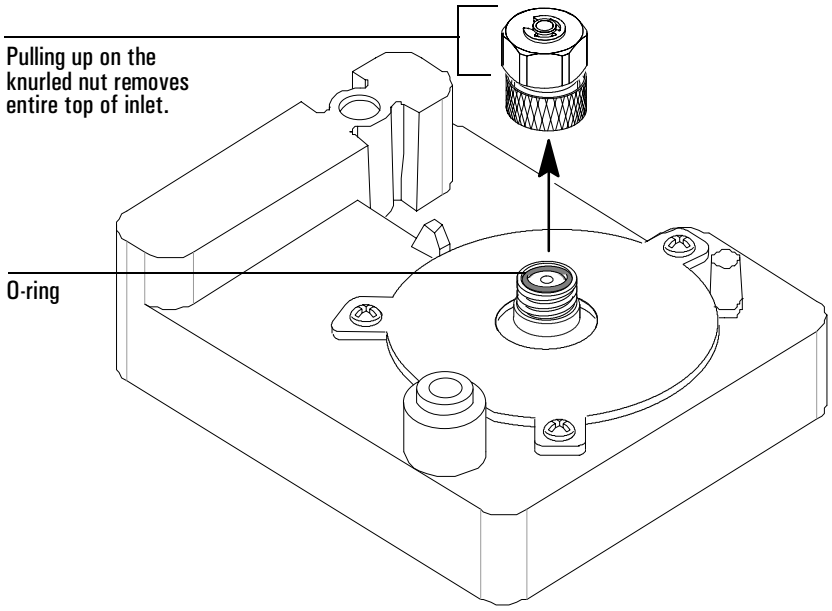
It is unlikely that the inlet will frequently require thorough cleaning as described below; however, deposits from injected samples occasionally build up inside the purged packed inlet. Before cleaning the inlet, replace the column liner and insert with clean ones. See "Installing a liner" on page 137 and "Installing a glass insert" on page 138. If the problem remains, then clean the inlet.

**Materials needed:**

- Cleaning brushes—The FID cleaning kit contains appropriate brushes (part no. 9301-0985)
- Solvent that will clean the type of deposits in your inlet
- Compressed, filtered, dry air or nitrogen

1. If you have entered parameters that you do not want to lose, store them as a method.
2. Turn off the GC and unplug it.
3. Turn the inlet pressure off at the source.
4. Let the heated zones (inlet, detector, oven, and valve box) cool to room temperature.
5. If the septum is worn out or dirty, replace it. See "Changing septa" on page 139 for instructions.
6. Remove the column and the column liner and insert. See "Columns and Traps" on page 21, "Installing a liner" on page 137, and "Installing a glass insert" on page 138 for details.

7. Loosen the knurled nut and pull it upward. The O-ring will be visible. Replace it if it is hard and brittle or cracked. See "Changing the O-ring" on page 142 for details.




8. Open the oven lid. Use a light source to illuminate the inside of the inlet from inside the oven while looking through the inlet from the top. If deposits are present, they should be visible.
9. Insert the brush into the inlet. Scrub the interior walls of the inlet vigorously to remove all deposits. You may need to wet the brush with solvent. Use a burst of compressed air or nitrogen to dry the inlet and remove loose contaminants.
10. Replace the top of the inlet and tighten the knurled nut. Replace the column. See "Columns and Traps" on page 21 for details.
11. Restore the GC to normal operating conditions.

## Replacing the inlet flow module

Replacing the purged packed inlet flow module is similar to replacing a split/splitless inlet flow module. See "Replacing the inlet flow module" on page 124.

# Maintaining a PTV

## Inlet adapters

The GRAPHPACK-2M connector (the inlet adapter) at the bottom of the inlet is sized to the column diameter. When a different diameter column is to be installed, the adapter must be changed.

The adapter number is stamped on the side of the adapters. Select the smallest hole diameter that will accept the column. See Table 29 for part numbers.

**Table 29. PTV Inlet Adapters**

| Column id | Inlet adapter number | Quantity | Part no. |
|---|---|---|---|
| 200 µm | 31 | 1 | 5182-9754 |
| 250 µm | 45 | 1 | 5182-9761 |
| 320 µm | 45 | 1 | 5182-9761 |
| 530 µm | 70 | 1 | 5182-9762 |

### To replace the inlet adapter

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off.
2. Unscrew the column nut from the adapter. Remove the nut and the column from the inlet.
3. With a 6-mm wrench, remove the inlet adapter, being careful not to lose the silver seal inside. Save the adapter for later use.

4. Select the appropriate inlet adapter for the column to be installed. Insert a new silver seal (part number 5182-9763, pkg of 5) into the adapter and screw the adapter onto the inlet finger tight. Use the 6-mm wrench to tighten the adapter an additional 1/16- to 1/8-turn.

   Do not overtighten the adapter. The inlet can be damaged if the adapter is forced. If the adapter leaks, check the silver seal and replace it if necessary.

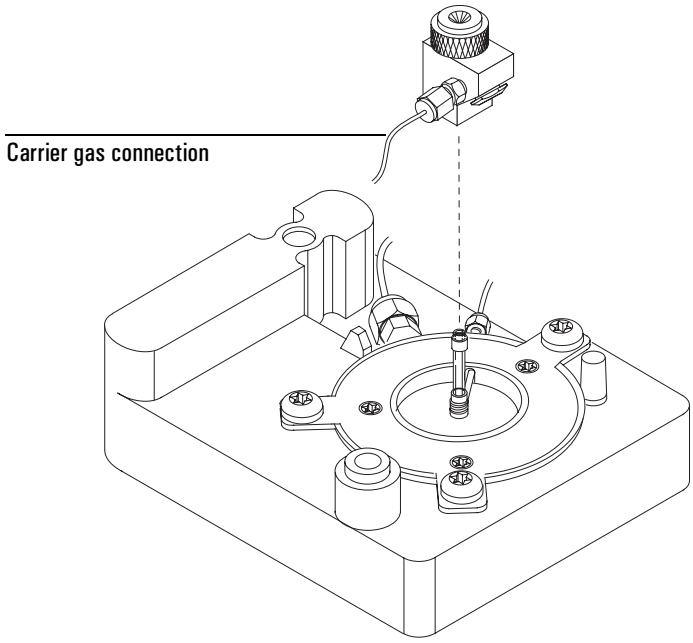
## The septumless head

This sampling head uses a check valve instead of a septum to seal the syringe entrance passage. It may be used with either automatic or manual injection. Syringes must have 23 gauge needles (see "Consumables and replaceable parts" on page 165).

### Removing the septumless head

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off.
2. Disconnect the carrier gas line.
3. Unscrew the septumless head counterclockwise from the inlet.

4. Screw the new head onto the inlet. Tighten it 1/8-turn past finger tight.



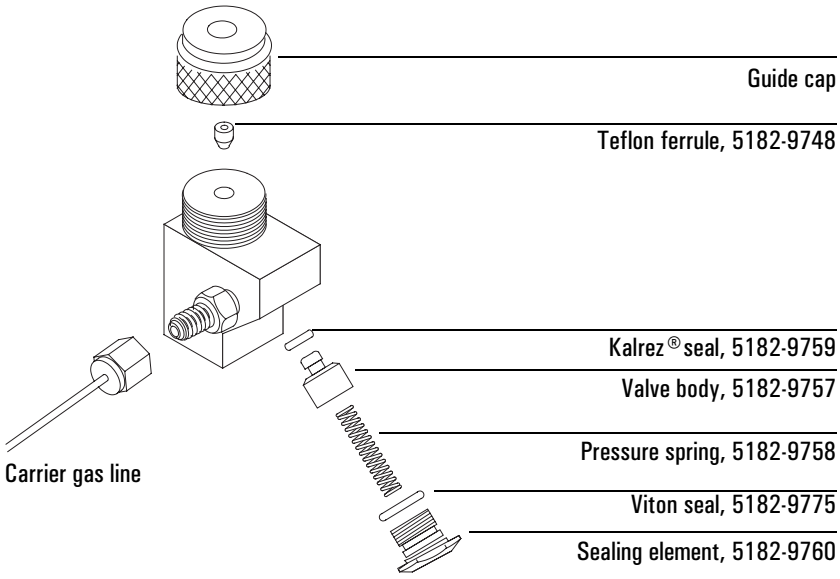

5. Reconnect the carrier gas line.
6. Check all connections on the sampling head for leaks. If necessary, tighten them again by hand.

### Cleaning the septumless head

Minor deposits from sample mixtures can collect in the head. Dust and abraded material particles can enter together with the syringe needle, eventually causing leaks. We recommend periodic cleaning.

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off.
2. Disconnect the carrier gas line and unscrew the head from the inlet.

3. Unscrew the sealing element from the head. Carefully remove the Viton seal and the pressure spring.



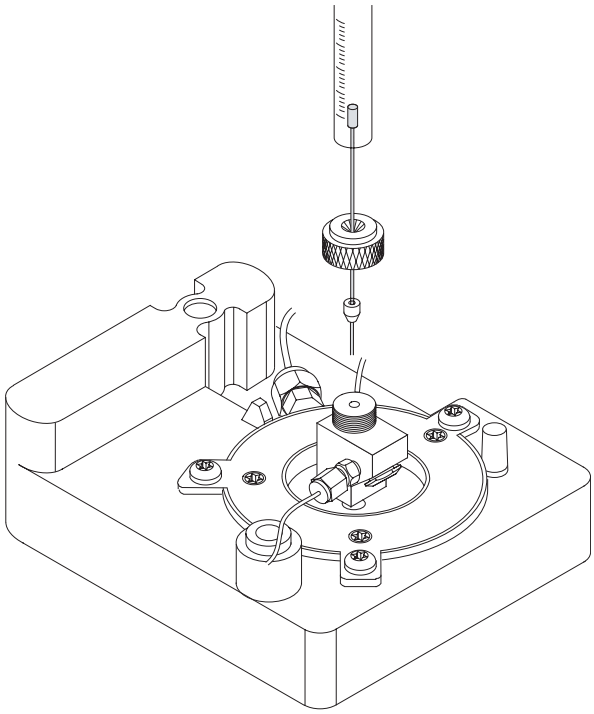

4. Unscrew the guide cap from the head and remove the Teflon ferrule.

**Caution** Do *not* use a sharp object to extract the valve body—this can leave scratches that cause leaks.

5. Insert a syringe with a 23 gauge needle carefully into the head to press the valve body with the Kalrez seal slightly out of the head. Carefully tap the head on a soft smooth surface so that the valve body falls out completely or slips so far out that you can grasp it with your fingers.
6. Remove the seal from the valve body.
7. Carefully clean all components in hexane.
8. Assemble the head in reverse order. Make sure that you work absolutely lint-free and that the seals and the pressure spring are not damaged.
9. Use this opportunity to check the Teflon ferrule. If it must be replaced, see page 154 for instructions.
10. Check the entire system again for leaks; if necessary, carefully retighten the guide cap slightly more with the syringe needle inserted and/or replace the Kalrez seal.
11. If the head leaks when a syringe is inserted, the Teflon ferrule is the problem. If the head leaks without a syringe inserted, the seals may need to be replaced.

### Replacing the Teflon ferrule

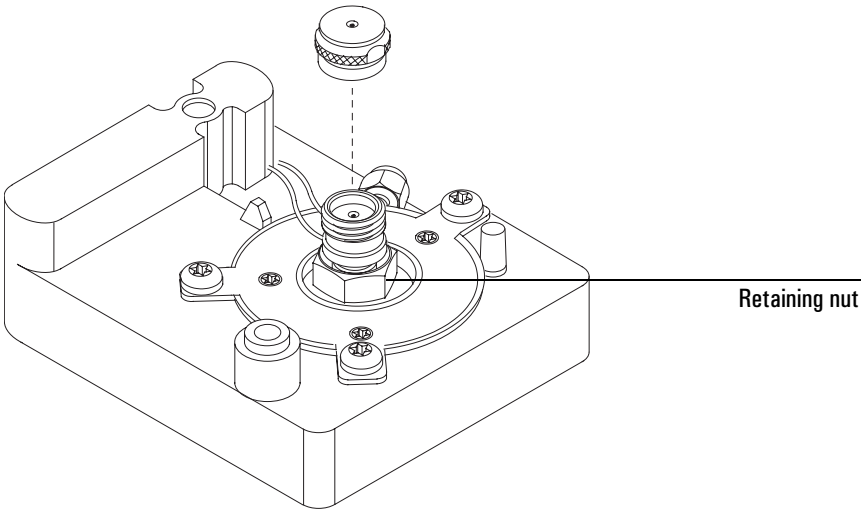
1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off.
2. Unscrew the guide cap from the septumless head and remove the Teflon ferrule.



3. Push the guide cap and the new Teflon ferrule over the syringe needle so that at least 10 mm of the needle tip is exposed.
4. Guide the end of the syringe needle into the septumless head until the ferrule meets the septumless head.
5. Tighten the guide cap until resistance is first felt.
6. Check for leaks when the syringe needle has been fully introduced.
7. If necessary, carefully tighten the guide cap until the inlet stops leaking.

## The septum head

The septum head uses either a regular septum or a Merlin Microseal to seal the syringe passage. A stream of gas sweeps the inner side of the septum and exits through the septum purge vent on the pneumatics module.




### Removing the septum head

The septum head connects to the inlet via a free-spinning retaining nut.

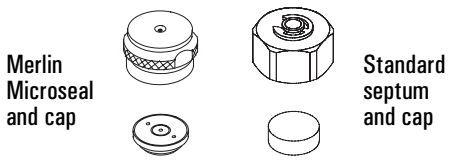
1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off.
2. Use a 5/8-inch wrench to loosen the retaining nut on the septum head.
3. Gently remove the septum head assembly from the inlet. Be careful not to overly bend the 1/16-inch lines. For best results, lift the head to clear the inlet and then push it to either side to allow access.
4. To reinstall the septum head, gently align the head with the inlet and manually engage the free-spinning nut to the inlet.
5. The nut should easily turn on to the inlet. If resistance is felt, unscrew the nut and retry. Excessive force can irreparably damage the inlet.
6. Tighten the retaining nut ½-turn past finger tight.
7. Check all connections for leaks. If necessary, the retaining nut can be tightened an additional ¼-turn to eliminate leaks.

### Changing the septum

Either a regular septum or a Merlin Microseal™ can be used with the septum head.

If the inlet temperature is set below 40°C, the Merlin Microseal may not seal effectively. For inlet temperatures below 40°C, use a regular septum for the inlet seal.

1. Before replacing the septum, cool the inlet to ambient temperature. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off.
2. Using the inlet tool or manually, unscrew the septum cap or Merlin cap counterclockwise. If the septum head begins to turn, support it manually while removing the cap.
3. Remove the septum or Merlin Microseal, taking care not to scratch the interior of the septum head.
4. Install a new septum or Merlin Microseal and the correct cap. When installing a Merlin Microseal, note that the side where the metal parts are visible goes down.




5. Check for leaks out of the cap and tighten the cap if necessary.

## Glass inlet liners

The liner is the chamber for sample deposition. Table 30 lists the liners available for the PTV inlet.

**Table 30. PTV Inlet liners**

| Type | Injection capacity | Inertness | Quantity | Part no. |
|---|---|---|---|---|
| Open baffled liner | Lowest capacity | Most inert | 10 | 5182-9751 |
| Liner packed with silanized glass wool | Higher capacity | Less inert | 10 | 5182-9752 |
| Unpacked liner, to be packed by the user | Depends on the packing | | 10 | 5182-9753 |

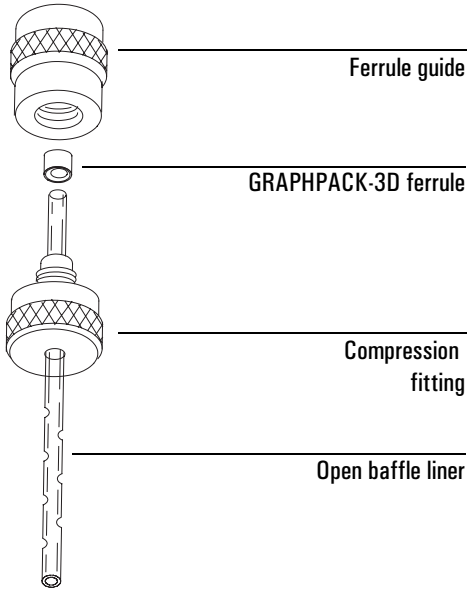
| Type | Injection capacity | Glass type | Glass wool packing* | Typical application | Part no. |
|---|---|---|---|---|---|
| Single baffle liner | 180 µL | Borosilicate deactivated | Yes | Large volume injection, not for extremely active compounds | 5183-2038 |
| Single baffle liner | 200 µL | Borosilicate deactivated | No | General purpose | 5183-2036 |
| Multi baffle liner | 150 µL | Borosilicate deactivated | No | Active compounds, drugs, pesticides | 5183-2037 |
| Fritted glass liner | 150 µL | Borosilicate deactivated | No | Large volume injection, all but the most active compounds | 5183-2041 |

*Silanized glass wool 10 gm (pesticide grade) part no. 5181-3317

### Replacing liners

1. Remove the head from the inlet. See "Removing the septumless head" or "Removing the septum head".
2. Grasp the liner by the GRAPHPACK ferrule. Remove the liner and ferrule.

3. Unscrew the assembly tool (part number G2617-80540) into two pieces, the ferrule guide and the compression fitting.




4. Slide the compression fitting onto the longer straight end of the new liner with the threads pointing toward the end of the liner.
5. Place a GRAPHPACK-3D ferrule on the same end of the liner with the recessed graphite end towards the compression fitting. Slide the ferrule on so that about 2 mm of liner is exposed beyond the ferrule.
6. Slide the compression fitting up to meet the ferrule. Screw the ferrule guide gently onto the compression fitting until it is finger tight.
7. Unscrew and remove the ferrule guide. Slide the compression fitting off the other end of the liner. The ferrule should now be set with 1 mm of liner exposed. Check that the graphite within the ferrule is flush with the top of the metal collar.
8. Insert the glass liner into the inlet from above until the unpacked side of the ferrule rests on the top of the inlet.
9. Replace the sampling head and reconnect the lines, if necessary.
10. Check all connections for leaks. If necessary, tighten them again by hand.

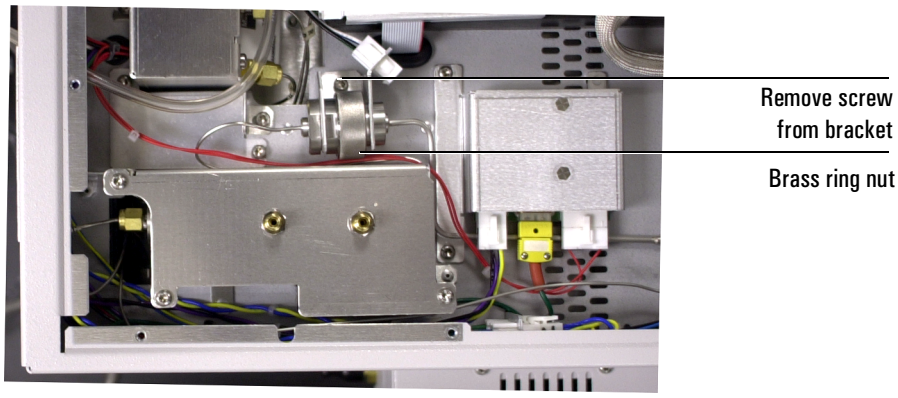
## Replacing the split vent trap filter cartridge

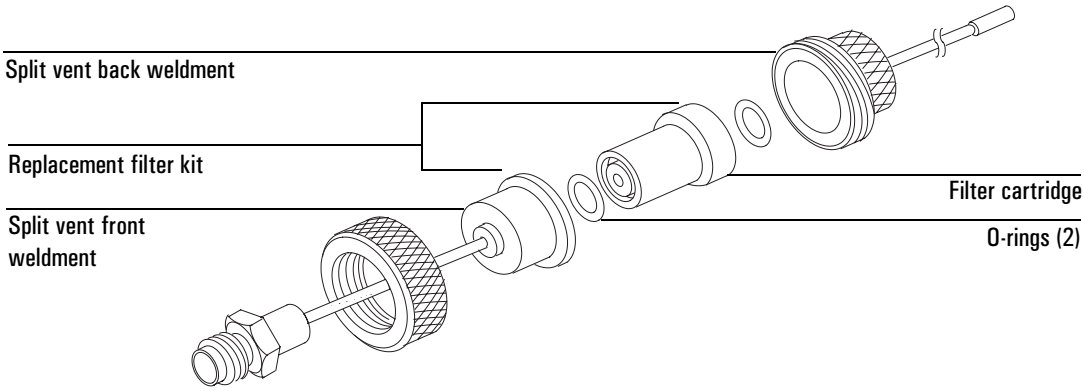
**Warning** The split vent trap may contain residual amounts of any samples or other chemicals you have run through the GC. Follow appropriate safety procedures for handling these types of substances while replacing the trap filter cartridge.

### Tools and materials

- T-20 Torx driver
- Replacement filter kit

1. Load the SERVICE method, or reset the inlet and oven temperatures to OFF, or turn off the main power switch. Let the heated zones cool to room temperature.
2. Remove the lid top cover.
3. Remove the screw that holds down the split vent trap clamp and remove the clamp.
4. Unscrew the brass ring nut and remove the cartridge assembly (the O-rings and the filter cartridge).



5. Replace the two O-rings and filter cartridge.
6. Check for leaks.

### Replacing the inlet flow module

Replacing the PTV inlet flow module is similar to replacing a split/splitless inlet flow module. See "Replacing the inlet flow module" on page 124.

### Leak testing the gas plumbing

Leaks in the gas plumbing can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the inlet flow manifold. If this portion of the system proves to be leak-free, refer to the next procedure to check the inlet and inlet manifold.

**Caution** Liquid leak detectors are not recommended, especially in areas where cleanliness is very important. If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

#### Tools

- Electronic leak detector capable of detecting your gas type or liquid leak detection fluid. If you use leak detection fluid, remove excess fluid when you have completed the test.
- Two 7/16-inch wrenches

#### Procedure

**Warning** To avoid a potential shock hazard when using liquid detection fluid, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads, especially the detector and inlet heater leads.

1. Using the leak detector, check each connection for leaks.
2. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.

### Leak testing the PTV inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet. If the inlet is leaking, you should use an electronic leak detector to pinpoint the component that is leaking.

If you have access to a Control Module, you can perform this test automatically. See its documentation for details. The Control Module test program will prompt you to complete the hardware tasks outlined below.

**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

**Materials needed:**

- 7/16-inch wrench
- Gloves (if the inlet is hot)
- Septum nut wrench (part no. 19251-00100)
- 9/16-inch wrench
- 1/8-inch no-hole (solid) Vespel type ferrules, 0100-1372
- 1/16-inch no-hole Vespel type ferrules, 5181-7458
- 1/8-inch Swagelok nut, 5180-4103
- Capillary column nut
- Septum
- Column nut, 5181-8830
- Vespel/graphite blank ferrule, 5020-8294

**Procedure**

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off
2. Turn the inlet pressure off at the source.
3. Remove the column, if one is installed, and plug the column fitting with the column nut and a no-hole ferrule.
4. Remove the old septum and replace it with a new one. For instructions, see “Changing the septum”.
5. Remove the column from the inlet fitting on the inside of the oven.
6. If a septum head is installed, and the quality of the septum (or Microseal) and GRAPHPACK-3D ferrule on the glass liner are unknown, replace them now.

7. Cap the inlet's column fitting and the septum purge vent (septum head only). Use solid (no hole) Vespel type ferrules 1/8-inch (part no. 0100-1372) and 1/16-inch (part no. 5181-7458) with a 1/8-inch Swagelok nut (part no. 5180-4103) and a capillary column nut.

   As alternate capping devices, a 1/8-inch Swagelok cap can be used for the septum purge vent. A capillary column nut with a solid piece of wire the size of a paper clip and a 0.5-mm id graphite ferrule may be used for the inlet column fitting.

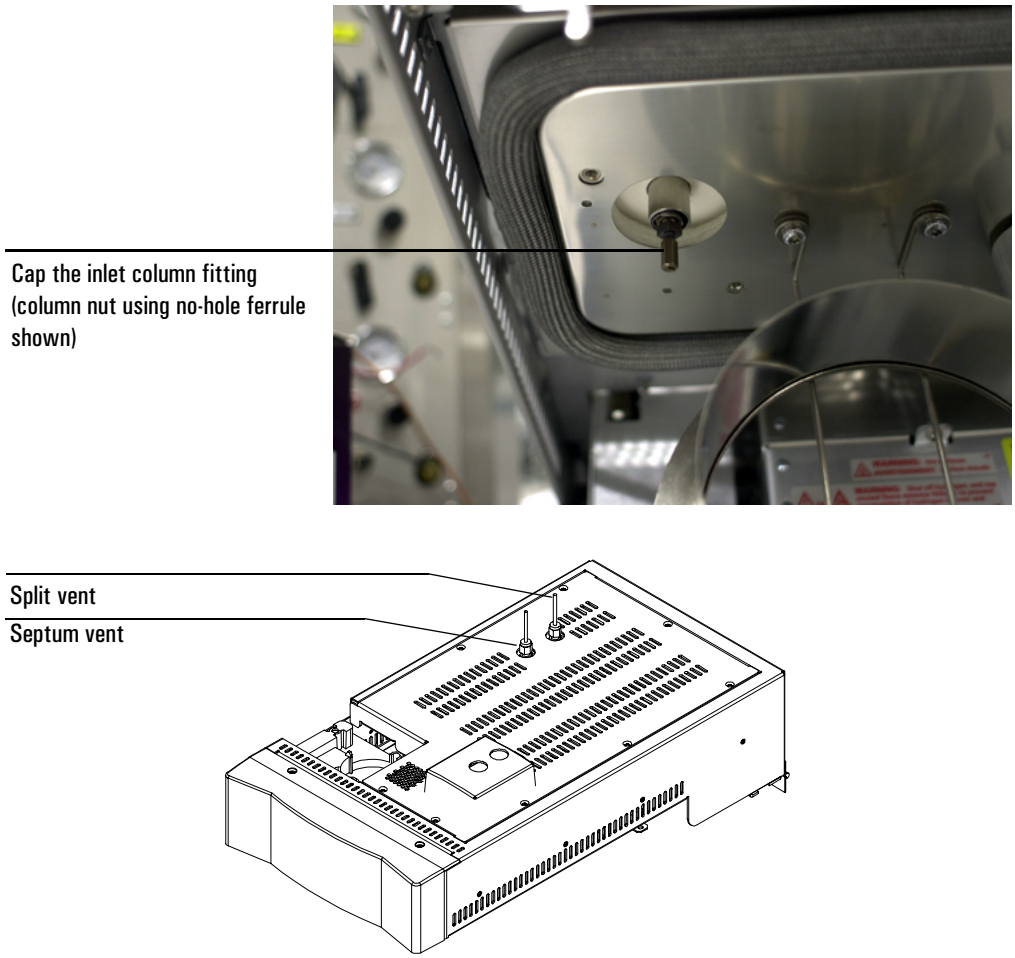



**Figure 29. Capping the bottom of the inlet and septum purge vent**

8. Make sure that the carrier gas source pressure is at least 35 psi. Carrier source pressure should always be at least 10 psi greater than the desired inlet pressure.

9. Configure the inlet for the test using a control module or data system.
   a. Set the inlet to “Split Mode.”
   b. Configure the column as 0 length.
   c. Set the inlet’s Total Flow to 60 mL/min.
   d. Set the pressure to 25 psi.
   e. Set the inlet temperature to its normal operating temperature.
10. Wait approximately 15 seconds for equilibration.

    If pressure cannot be achieved, either a very large leak is present in the system, or the supply pressure is not high enough.
11. Turn the inlet pressure “Off” using your control module or data system.

    Both the flow controller and the back pressure valves will close.
12. Note the “Actual” reading on the control module or data system and monitor the pressure for 10 minutes.
    - If there is less than 0.5 psi pressure loss, the system is leak tight.
    - If pressure loss is much greater than 0.5 psi, there is a leak that must be found and corrected. Note, however, that you may want to slightly decrease the leak test time based on the internal inlet volume which changes with the liner type used (smaller volumes = shorter acceptable leak test times). See “Correcting leaks” on page 163.
13. When the system is considered leak tight, the caps may be removed, the column reinstalled, its dimensions configured at keyboard, and the desired pressure and flow rate set.

### Correcting leaks

**Materials needed:**

- Electronic leak detector suitable for the gas type
- Tools to tighten parts of the inlet that leak (if leaks are detected)

1. Use an electronic leak detector to check all areas of the inlet that are potential sources of a leak.

2. Tighten loose connections to correct leaks, if necessary. You may need to repeat the leak test.

   If the pressure drop is now less than 0.5 psi, you can consider the inlet system leak-free. If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak free, but the inlet system is still losing too much pressure, you may need to replace the inlet manifold.

### Potential leak points

Check the following areas when checking an inlet system for leaks.

*In the oven*

- Make sure the bottom of the inlet is correctly capped.

*On the inlet*

- Septum (septum head only)
- Lower inlet seal at bottom of inlet
- Ferrule on inlet liner
- Connections for carrier gas, septum purge (septum head only)

*At EPC module*

- O-rings behind the block where the inlet's pneumatic lines enter the module
- Septum purge cap (septum head only)
- Chemical trap O-rings
- O-rings in manifold fittings

## Checking the split vent line for restrictions

Over time, the split vent line (including the trap) can become obstructed with condensed vapors and other debris. If you have access to a Control Module, you can perform an automated test to check for split vent line restrictions that may cause problems in using the inlet. See the Control Module documentation for details. The Control Module test program will prompt you to complete hardware tasks similar to the ones described in "Leak testing the PTV inlet" on page 160.

## Consumables and replaceable parts

Table 31 lists orderable PTV parts.

**Table 31. PTV Consumables and Replacement Parts**

| Description | Quantity | Part no. |
|---|---|---|
| Septumless head assembly | 1 | G2617-60507 |
| Service kit | 1 | 5182-9747 |
| Valve body | 1 | 5182-9757 |
| Pressure spring | 1 | 5182-9758 |
| Kalrez seal | 1 | 5182-9759 |
| Teflon guide | 1 | 5182-9748 |
| Sealing element | 1 | 5182-9760 |
| GRAPHPACK-3D ferrule for liners | 5 | 5182-9749 |
| Assembly tool for GRAPHPACK-3D ferrules | 1 | G2617-80540 |
| Single baffle liner | 1 | 5183-2038 |
| Single baffle liner | 1 | 5183-2036 |
| Multi baffle liner | 1 | 5183-2037 |
| Fritted glass liner | | 5183-2041 |
| GRAPHPACK-2M inlet adapter, 0.2 mm column id | 1 | 5182-9754 |
| GRAPHPACK-2M inlet adapter, 0.32/0.25 mm column id | 1 | 5182-9761 |
| GRAPHPACK-2M inlet adapter, 0.53 mm column id | 1 | 5182-9762 |
| Silver seal for GRAPHPACK-2M inlet adapter | 5 | 5182-9763 |
| Nut for GRAPHPACK inlet adapters | 5 | 5062-3525 |
| Ferrules for GRAPHPACK-2M inlet adapter, 0.2 mm column id | 10 | 5182-9756 |
| Ferrules for GRAPHPACK-2M inlet adapter, 0.25 mm column id | 10 | 5182-9768 |
| Ferrules for GRAPHPACK-2M inlet adapter, 0.32 mm column id | 10 | 5182-9769 |
| Ferrules for GRAPHPACK-2M inlet adapter, 0.53 mm column id | 10 | 5182-9770 |
| Syringes | | |
| 5 µL, 23 gauge fixed needle | 1 | 9301-0892 |
| 10 µL, 23 gauge fixed needle | 1 | 9301-0713 |
| 10 µL, Teflon-tipped plunger, 23 gauge fixed needle | 1 | 5181-8809 |
| 10 µL, Teflon-tipped plunger, 23 gauge removable needle | 1 | 5181-8813 |
| 25 µL, Teflon-tipped plunger, 23 gauge fixed needle | 1 | 5183-0316 |
| 25 µL, Teflon-tipped plunger, 23 gauge removable needle | 1 | 5183-0317 |
| 50 µL, Teflon-tipped plunger, 23 gauge fixed needle | 1 | 5183-0318 |
| 50 µL, Teflon-tipped plunger, 23 gauge removable needle | 1 | 5183-0319 |
| Septa and seals | | |
| Merlin Microseal starter kit (cap + 1 microseal) | 1 | 5182-3442 |
| Merlin Microseal replacement | 1 | 5182-3444 |
| 11-mm septa, red | 25 | 5181-1263 |

## Maintaining a cool on-column inlet

Maintaining the cool on-column inlet includes changing the insert, changing septa, cleaning inlet components, and checking and correcting leaks in the system.

The cool on-column inlet's hardware (see Figure 30) will vary depending on whether you will be making manual or automated injections, the type of needle you use, and the size of column you use.

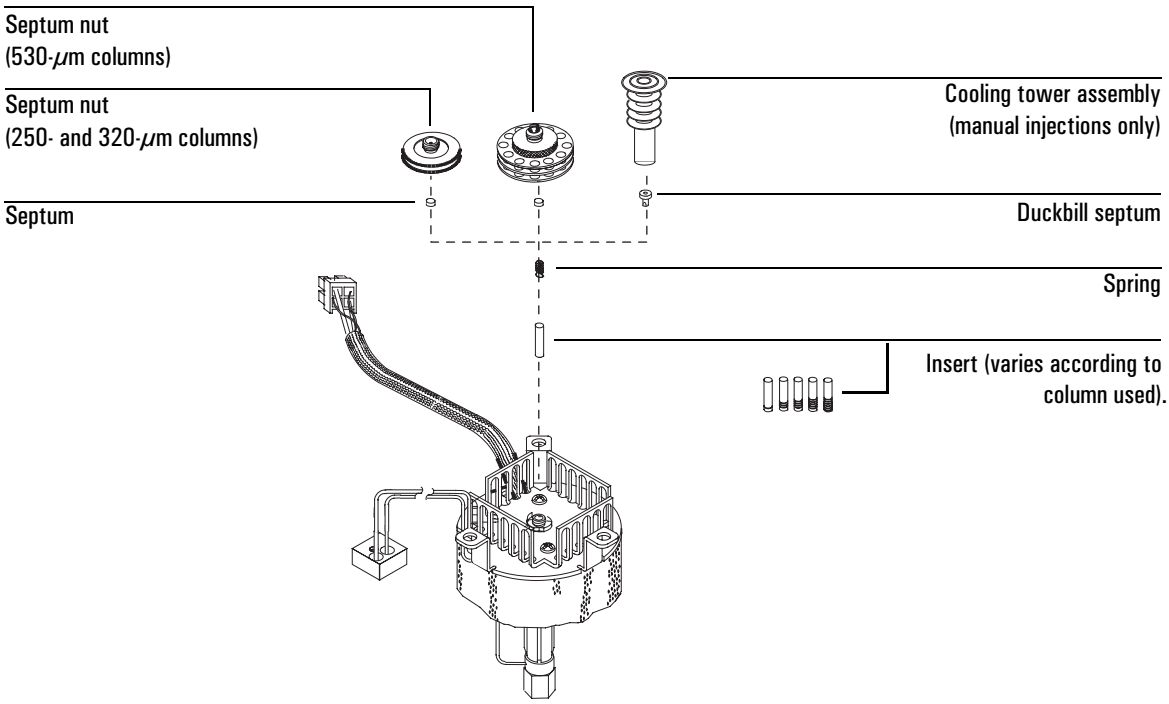



**Figure 30. The cool on-column inlet**

### Hardware

Because you are injecting sample directly into the column, most of the hardware required is determined by your column inside diameter. Injection technique, manual or automatic, must also be considered. Table 32 is a checklist for choosing hardware and shows where to find instructions for installing the hardware and injecting the sample.

Note that if you are performing automatic injections on a 250 µm/320 µm column using an autosampler, you must adapt it for on-column use. See "Adapting for cool on-column injection" on page 228 for details.

**Table 32. Hardware and Procedures Checklist**

| Automatic injection | Manual injection with septum nut | Manual injection with cooling tower |
|---|---|---|
| **Hardware** | | |
| See Table 33 for part numbers | See Table 33 for part numbers | See Table 34 for part numbers |
| ❒ Septum nut<br>❒ Insert<br>❒ Stainless steel needle | ❒ Septum nut<br>❒ Solid septum<br>❒ Insert<br>❒ Stainless steel needle | ❒ Cooling tower<br>❒ Duckbill septum<br>❒ Insert<br>❒ Fused silica needle (columns ≥200 µm)<br>or<br>❒ Stainless steel needle (columns ≥250 µm) |
| **Where to find instructions** | | |
| ❒ "Installing an insert" on page 173<br>❒ "Changing the septum nut or cooling tower and septum" on page 173 | ❒ "Installing an insert" on page 173<br>❒ "Changing the septum nut or cooling tower and septum" on page 173 | ❒ "Installing an insert" on page 173<br>❒ "Changing the septum nut or cooling tower and septum" on page 173 |
| ❒ "Check the needle-to-column size" on page 170 | ❒ "Manual injection with septum nut" on page 171 | ❒ "Manual injection with cooling tower" on page 171 (bottom) *and* "Replacing the fused silica syringe needle" on page 174 |

### Automatic or manual injection with septum nut

Choose a needle, septum nut, and insert based on your column inside diameter. See Table 33 to determine the correct hardware.

**Table 33. Automatic or Manual Injection with a Stainless Steel Needle**

| Column type and inside diameter | Needle part no.* | Septum nut part no. | Insert part no. |
|---|---|---|---|
| Fused silica: | | | |
| 530 μm id | 5182-0832** | G1545-80520  | 19245-20580 (no rings) |
| 320 μm id | 5182-0831 | 19245-80521  | 19245-20525 (5 rings) |
| 250 μm id | 5182-0833 | 19245-80521  | 19245-20515 (6 rings) |
| 200 μm id | Use cooling tower and duckbill septum | | 19245-20510 (1 ring) |
| Aluminum-clad, 530 μm id | 5182-0832 | G1545-80520 | 19245-20780 (4 rings) |
| Glass capillary | | | |
| 320 μm id | 5182-0831 | 19245-20670 | 19245-20550 (3 rings) |
| 250 μm id | 5182-0833 | 19245-20670 | 19245-20550 (3 rings) |

* Order removable needle syringe, part no. 5182-0836. If doing a manual injection, you must also order a plunger button, part no. 5181-8866.

** Many other needles can be used to inject onto a 530-μ column. Consult the Agilent catalog for consumables and supplies for details.

### Septa

Use a solid septum (5181-1261) for manual injection, or a through-hole septum (5181-1260) for auto injection.

## Manual injection with a cooling tower and duckbill septum

If you are doing this type of manual injection, use either fused silica or metal removable stainless steel needles. Use Table 34 to choose the correct insert, syringe, and other hardware.

**Table 34. Manual Injection Hardware—Cooling Tower and Duckbill Septum**

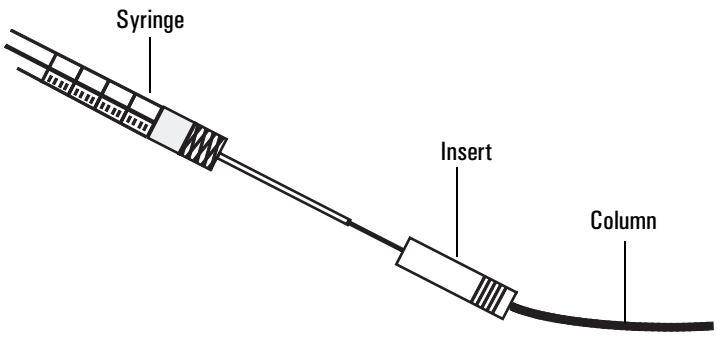
| Column type and inside diameter | Insert (part no.) |
|---|---|
| Fused silica | |
| 530 μm | 19245-20580 (no rings) |
| 320 μm | 19245-20525 (5 rings) |
| 250 μm | 19245-20515 (6 rings) |
| 200 μm | 19245-20510 (1 ring) |
| Aluminum-clad, 530 μm | 19245-20780 (4 rings) |
| Glass capillary | 19245-20550 (3 rings) |
| **Syringe and needle** | |
| **For fused silica needles** | |
| Fused silica needle syringe | 9301-0658 |
| Replacement needles, fused silica, 0.18 mm (6 pk) | 19091-63000 |
| Replacement Teflon® ferrule for syringe | 0100-1389 |
| **For stainless steel needles** | |
| Removable needle syringe, 10 μL | 5182-9633 |
| Replacement needles, 0.23 mm (3 pk) | 5182-9645 |
| **Inlet hardware** | |
| Cooling tower assembly | 19230-80625 |
| Duckbill septum (for columns > 200-μm id) | 19245-40050 |

### Check the needle-to-column size

**Caution** This applies to 250 µm and 320 µm columns only.

After selecting an insert and before installing a column, you need to check the needle-to-column size to make certain your needle fits in the column. You could bend the needle if you try to inject it into a smaller column. Use the insert that is the same size as your syringe needle to verify that the column you plan to use is the correct size.

1. Identify the correct insert.
2. Insert the column into one end of the insert as shown below.




3. Insert the syringe needle through the other end of the insert and into the column. If the needle cannot pass easily into the column, reverse the insert to try the needle and column in the other end.

If the needle still cannot pass into the column, you may have a column with an incorrect id. Check the column to make sure it is labeled correctly, and try a new column.

### Manual injection with septum nut

Before making your injection, make sure the correct septum nut and septum are installed.

1. Immerse the syringe needle in sample; pump the syringe plunger to expel air from the barrel and needle.
2. Draw the sample into the syringe.
3. Remove the needle from the sample and draw about 1 µL of air into the syringe.
4. Wipe the needle dry if it is wet.
5. Guide the needle straight into the septum nut, pierce the septum, and insert the needle fully into the inlet until it bottoms.
6. Start the run, depress the syringe plunger *as quickly as possible*, and withdraw the needle from the inlet.

These steps should be done smoothly, with minimal delay.

### Manual injection with cooling tower

When injecting with fused silica or metal removable stainless steel needles, be sure the cooling tower assembly and duckbill are installed on the inlet. Initial pressure must be set at less than 207 kPa (30 psi). Higher pressures will make needle insertion difficult.

1. Immerse the syringe needle in the sample and pump the syringe plunger to expel air from the barrel and needle.
2. Draw the sample into the syringe. Allow enough time for fluids to pass through the small bore of the needle.
3. Remove the needle from the sample and draw about 1 µL of air into the syringe. Wipe the needle with a tissue wetted with solvent.
4. Press down the top of the cooling tower with a pencil to open the duckbill.

**Warning** The cooling tower may be hot!

5. Hold down the cooling tower and guide the needle until it is fully inserted in the inlet. You may observe a drop in the pressure reading on the control table.

   If the needle does not go in all the way, try rotating the syringe and slightly releasing pressure on the cooling tower.

   If you still cannot get the needle in, the duckbill opening may be stuck. Try removing the duckbill, opening it manually, and reinstalling it.

6. Once the needle has entered the column, release the cooling tower and continue to insert the needle. Allow 1 to 2 seconds for back pressure on the duckbill to seal it around the inserted needle.
7. Start the GC, depress the syringe plunger as quickly as possible, and withdraw the needle from the inlet.

## Cool on-column inlet hardware problems

### The inlet cools very slowly

- The inlet fan is not running or is blowing upwards, out of the GC lid. Check the fan to make sure it is operating. If it is not, contact your Agilent service representative.

### The inlet is unable to reach a temperature setpoint

- Check the temperature equilibration time. If the equilibration time is too short, the inlet may oscillate. Increase the equilibration time.
- Check that cryogenic cooling is turned off. If you do not turn it off when not in use, both the inlet and the oven may be unable to reach their setpoints, particularly temperatures near room temperature. If you turn the cryogenic cooling off and the inlet still fails to reach the setpoint temperature, contact your Agilent service representative.

### The syringe needle bends during injections

- The needle may have been defective before the injection was made. Check each syringe before injection to make sure the needle is straight.
- Check that the needle support assembly is installed correctly.
- Check that the correct insert is installed and that it is installed correctly.
- Check the alignment of the inlet septum and the septum nut.
- The inlet septum hole may have closed. Replace the septum.

*If you are using the GC Automatic Liquid Sampler (GC ALS):*

See the GC ALS manual for additional information.

- The sampler vials may be over-crimped.
- Check the needle guide for signs of wear or damage. Replace the needle guide if necessary.
- Check the alignment of the inlet and the automatic sampler.

### Changing the septum nut or cooling tower and septum

If you need to change the insert, refer also to the next section, "Installing an insert".

1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure.

**Warning** Be careful! The inlet fittings may be hot enough to cause burns.

2. Locate the septum nut or cooling tower assembly at the top of the inlet and remove (see Figure 30). If you are using a cooling tower, grasp the three rings and unscrew. If you are using a septum nut, grasp the knurling and unscrew.

   There should be a small spring at the inlet base. If the spring is stuck to the septum nut, place it back in the inlet base.

3. If you are using a *septum nut*, remove the old septum with tweezers, hemostat, or septum remover. Use tweezers to install a new septum. Push the septum into the septum nut until properly seated.

   If you are using a *cooling tower assembly*, locate the duckbill septum and install in the inlet base so that the duckbill is inserted inside the coil spring.

4. Install the septum nut or cooling tower assembly and tighten firmly.
5. Before making an injection, check the alignment of the entire assembly.

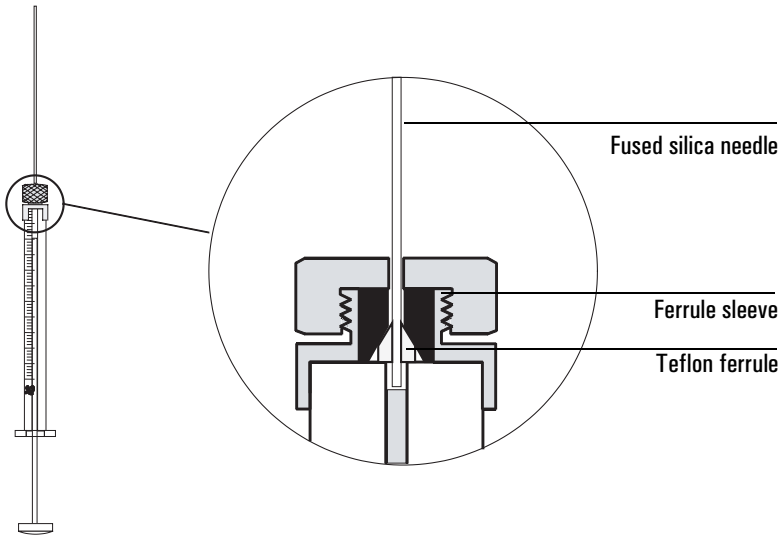
### Installing an insert

1. Choose an insert. See Table 33 or Table 34 for instructions on choosing an insert.
2. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure.
3. Remove the column, column nut, and ferrule.
4. Locate the septum nut or cooling tower assembly at the top of the inlet and remove it. If the septum remains in the septum nut, do not remove it unless you want to change it. If necessary, replace the existing septum or duckbill with a new one. See "Changing septa" on page 175 for detailed instructions. Set the inlet septum nut or cooling tower assembly aside.
5. Remove the spring from the inlet with an extraction wire, and set it aside. Be careful not to lose or damage it because you will use the spring to keep the new insert in position.

6. Remove the existing insert from the inlet by gently pushing it out from below with a wire or piece of column. Store the insert for possible later use.
7. Drop the new insert straight into the inlet from the top.
8. Replace the spring on top of the insert.
9. Reinstall the septum nut or duckbill septum and cooling tower assembly and tighten finger tight.
10. Reinstall the column, nut, and ferrule.

### Replacing the fused silica syringe needle

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off.
2. Hold the syringe vertically and insert the fused silica needle so it is visible *inside* the syringe barrel. If the fused silica needle cannot be inserted into the syringe barrel, the Teflon ferrule (part no. 0100-1389) may be blocked. You may need to replace the ferrule. Push the plunger down until it bottoms. The needle will now be flush with the end of the plunger.




3. When the needle is inserted, tighten the retaining nut to *firm* finger tightness. Pull the needle gently to be sure the Teflon ferrule has formed a tight seal with the needle. Tighten the retaining nut further, if necessary.

4. Loosen the retaining nut just enough so the needle is again free. Depress the syringe plunger slowly until it pushes the needle to the end of the barrel, then tighten the retaining nut to *firm* finger tightness.
5. Use a solvent to rinse the syringe and check for leaks or blocks.
6. Leaks (inability to eliminate air bubbles) *may* be fixed by further tightening the retaining nut. Blocks (or serious leaks) require repeating this procedure.

The Teflon ferrule may lose its seal in time. If so, first retighten the retaining nut and, if the seal still leaks, install a new Teflon ferrule and needle.

When not in use, loosen the retaining nut to avoid premature leaks.

If you are cutting replacement needles directly from fused silica column material:

1. Column material for making needles must have an outside diameter *smaller* than both the inside diameter of the on-column inlet (0.23 mm) and the inside diameter of the installed column.
2. Column material must be washed free of active stationary phase.
3. Score the column material about 1/4-inch from its end. Break off the end and discard. Then measure, score, and break off a 115±5 mm length to use as the syringe needle.

### Changing septa

If the septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Additionally, the detector signal will become increasingly noisy.

The useful lifetime of septa is determined by injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is used regularly, daily septum replacement is recommended.

The type of septa you use will depend on your chromatography needs. You can order septa directly from Agilent Technologies; see the Agilent catalog for consumables and supplies for ordering information.

**Caution** The procedure for changing septa differs depending on whether you cool on-column inlet has a cooling tower assembly or a septum nut. Make sure to follow the correct procedure for your inlet!

**Table 35. Recommended Septa for the Cool On-Column Inlet**

| Description | Part no. |
|---|---|
| Solid septum for manual and automatic injection (50 pk) | 5181-1261 |
| Through-hole septum for automatic injection (25 pk) | 5181-1260 |
| Solid septum, bleed and temperature optimized (50 pk) | 5182-0745 |
| Duckbill septum for manual injection only (must use cooling tower with the duckbill) (10 pk) | 19245-40050 |

**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

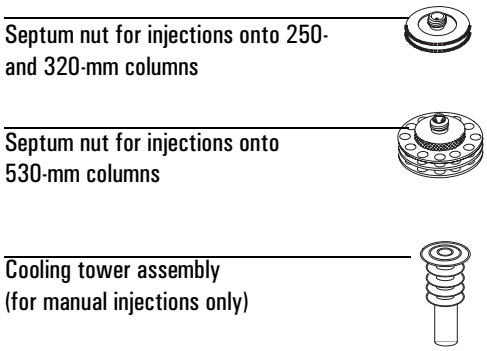
**Caution** Column flow is interrupted while changing septa; since columns may be damaged at elevated temperatures without carrier flow, cool the oven to room temperature before proceeding.

**Materials needed:**

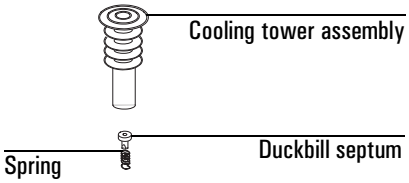
- New septum—see Table 35 for part numbers
- Forceps (or tweezers)
- A thin wire (0.2-inch diameter) for removing septum from inlet

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off.

Depending on your analysis and injection technique, the inlet will have one of the following septum nuts or a cooling tower assembly.

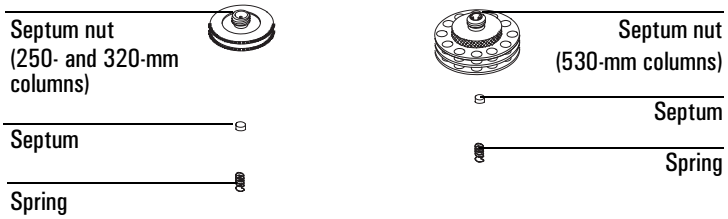



2. **If you have a cooling tower assembly installed:**
   Remove the assembly by grasping it and turning counterclockwise. The duckbill septum is underneath the cooling tower inside the spring. The spring and septum may pop out of the inlet when you remove the cooling tower.
   Be careful not to lose them. If they do not pop out, use a thin wire to remove them from the inlet.




   Insert the duckbill septum into the spring and place them in the inlet. Reattach the cooling tower assembly. Tighten it finger-tight.

3. **If you have a septum nut installed:**
   Remove the septum nut by grasping the knurling and turning counterclockwise. The septum is probably attached to the septum nut. The spring may also pop out when you remove the septum nut. Be careful not to lose it. If the septum is not attached, you may need to use tweezers to grasp and remove it.



Make sure the spring is in the inlet. Use the tweezers to place a new septum on the bottom of the septum nut, and then reattach the septum nut to the inlet. Tighten the nut firmly.

4. Restore normal GC operating conditions.

### Cleaning the inlet

Most laboratories have airborne lint and dust that accumulate on the cooling tower or septum nut and can enter the inlet or column on the syringe needle. Particulate matter in the inlet interferes with easy passage of the syringe needle. If dirt enters the column, it can alter the chromatography.

You can clean the needle guides, springs and inserts according to the following procedure.

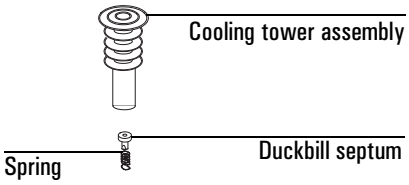
**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

**Materials needed:**

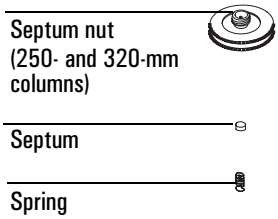
- 9/16-inch wrench
- Narrow wire (0.02-inch diameter) or piece of capillary column (250-µm diameter) for removing spring and insert
- Small ultrasonic cleaning bath with aqueous detergent
- Distilled water
- Methanol
- Compressed, filtered, dry air or nitrogen

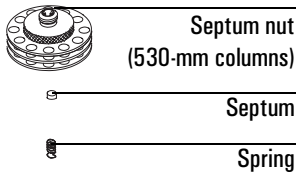
1. Load the SERVICE method or reset the inlet and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature. If you have entered parameters that you do not want to lose, store them as a method. If the detector is on, turn it off.
2. Turn off all flows to the inlet at the initial gas supply.
3. Turn off the GC and unplug it.
4. Remove the column. See "Installing columns in the cool on-column inlet" on page 41.

5. **If you have a cooling tower assembly installed:**
   Remove the assembly by grasping it and turning counterclockwise. The septum is underneath the cooling tower inside the spring. The spring and septum may pop out of the inlet when you remove the cooling tower. Be careful not to lose them. If they do not pop out, use a thin wire to remove them from the inlet.

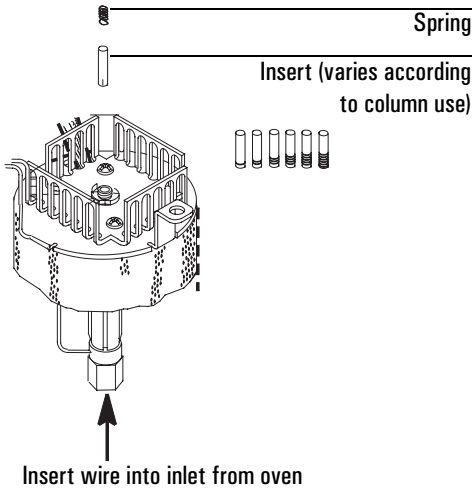



6. **If you have a septum nut installed:**
   Remove the septum nut by grasping the knurling and turning counter-clockwise. The septum is probably attached to the septum nut. The spring may also pop out when you remove the septum nut. Be careful not to lose it.




7. Insert the narrow wire (or a piece of capillary column) into the inlet through the oven, and push the insert and spring (if they did not come out previously) out through the top of the inlet.



8. Cleaning procedure:
   a. Fill the ultrasonic cleaning bath with aqueous detergent and place the spring and the insert into it. Sonicate for 1 minute.
   b. Drain the aqueous detergent and fill the bath with distilled water. Sonicate for 1 minute.
   c. Remove the parts from the bath and rinse them thoroughly with water and methanol.
   d. Dry the parts with a burst of compressed air or nitrogen.
9. Reinstall the insert. If you are using a septum nut, insert the spring and insert with the spring on top.
10. Attach a new septum to the bottom of the septum nut. If you are using the cooling tower assembly, insert a new duckbill septum into the spring, and place them in the inlet.
11. Attach the septum nut or the cooling tower and tighten finger-tight. Reinstall the column and restore normal operation conditions.

### Replacing the inlet flow module

Replacing the cool on-column inlet flow module is similar to replacing a split/splitless inlet flow module. See "Replacing the inlet flow module" on page 124.

### Leak testing the gas plumbing

Leaks in the gas plumbing system can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the inlet flow manifold. If this portion of the system proves to be leak-free, refer to the next procedure to leak-check the inlet and inlet manifold.

**Caution** Liquid leak detectors are not recommended, especially in areas where cleanliness is very important. If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

#### Tools

- Electronic leak detector capable of detecting your gas type or liquid leak detection fluid. If you use leak detection fluid, remove excess fluid when you have completed the test.
- Two 7/16-inch wrenches

### Procedure

**Warning** To avoid a potential shock hazard when using liquid detection fluid, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads, especially the detector and inlet heater leads.

1. Using the leak detector, check each connection you have made for leaks.
2. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.
3. Cap the septum purge vent with a 1/8-inch Swagelok cap.

## Leak testing the cool on-column inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet. If the inlet is leaking, use an electronic leak detector to pinpoint the leaking component.

If you have access to a Control Module, you can perform this test automatically. See its documentation for details. The Control Module test program will prompt you to complete the hardware tasks outlined below.

### Materials needed:

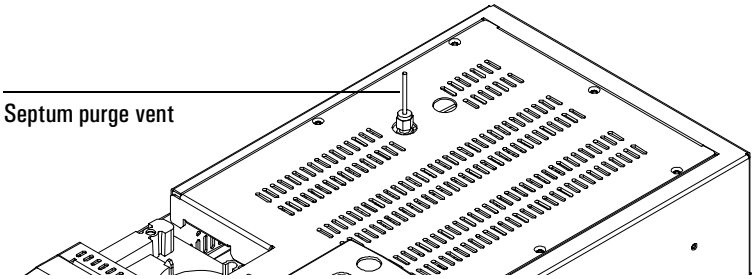
- 1/4-inch wrench
- Gloves (if the inlet is hot)
- 1/8-inch brass nut, 5180-4103
- 1/8-inch Vespel/graphite blank ferrule, 0100-1372
- Column nut, 5181-8830
- Vespel/graphite blank ferrule, 5020-8294

### Procedure

**Warning** Be careful! The oven and/or inlet may be hot enough to cause burns.

This test requires either a Control Module or a ChemStation/Cerity Chemical to edit setpoints.

1. Load the SERVICE method or reset the inlet and oven temperatures to OFF. Let the heated zones cool to room temperature. Also, make sure that:
   - If you have entered parameters that you do not want to lose, store them as a method
   - If the detector is on, turn it off
2. Turn the inlet pressure off at the source.
3. Remove the column, if one is installed, and plug the column fitting with the column nut with a no-hole ferrule installed.
4. Remove the old septum and replace it with a new one. For instructions, see page 175.
5. Make sure the carrier gas source pressure is at least 35 psi.
6. Cap the septum purge vent with a 1/8-inch Swagelok cap.




7. Using a control module or data system, set the oven temperature to its normal operating temperature.
8. Set the inlet to normal operating temperature.

   Enter a pressure to 25 psi, or enter your normal operating pressure if it is higher. Make sure that the pressure at the initial gas supply is at least 10 psi higher than the inlet pressure. If pressure cannot be achieved, either there is a large leak or the gas supply pressure is too low.
9. Wait a few minutes for the GC to equilibrate after the system has reached the pressure. The pressure may exceed the setpoint briefly during equilibration.
10. Turn either the pressure or the flow off. Because the column and septum purge fittings are capped, gas is trapped in the system and the pressure should remain fairly constant.
11. Monitor the pressure for 10 minutes.
    - A pressure drop of 1.0 psi (0.1 psi/min) or less is acceptable.

    If the pressure drop is much greater than 1.0 psi, go to the next section, "Correcting leaks" on page 183

12. When the system is leak-free, restore normal connections and operating conditions.

### Correcting leaks

**Materials needed:**

- Electronic leak detector suitable for the gas type
- Tools to tighten parts of the inlet that leak (if leaks are detected)

1. Use an electronic leak detector to check all areas of the inlet that are potential sources of a leak.
2. Tighten loose connections to correct leaks, if necessary. You may need to repeat the leak test.

If the pressure drop is now less than 0.3 psi, you can consider the inlet system leak-free. If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak free, but the inlet system is still losing too much pressure, you may need to replace the inlet manifold.

### Potential leak points

Check the following areas (as applicable) when checking an inlet system for leaks.

- The plugged column connection
- The septum and/or septum nut
- The cooling tower assembly
- The ¼-inch ferrule (if a liner is used)
- The O-rings and connections
- The area where gas line(s) are plumbed to the inlet
- The capped purge vent
- The knurled nut
- The septum purge cap (septum head only)
- The connections for the carrier gas and septum purge (septum head only)
- The lower inlet seal at the bottom of the inlet
- The inlet bottom cap

# Routine Maintenance: Detectors

The procedures described in this section can be performed by the user to maintain the detectors in the GC. They may involve exposure to heated surfaces, but not to hazardous voltages.

## Maintaining a thermal conductivity detector (TCD)

If the TCD is displaying problems such as a wandering baseline, increased noise level, or changes in response on a checkout chromatogram, it is probably contaminated with deposits from such things as column bleed or dirty samples.

The TCD is cleaned by a process known as thermal cleaning or bakeout. Perform bakeout only after you have confirmed that the carrier gas and the flow system components are leak and contaminant free.

### TCD plumbing

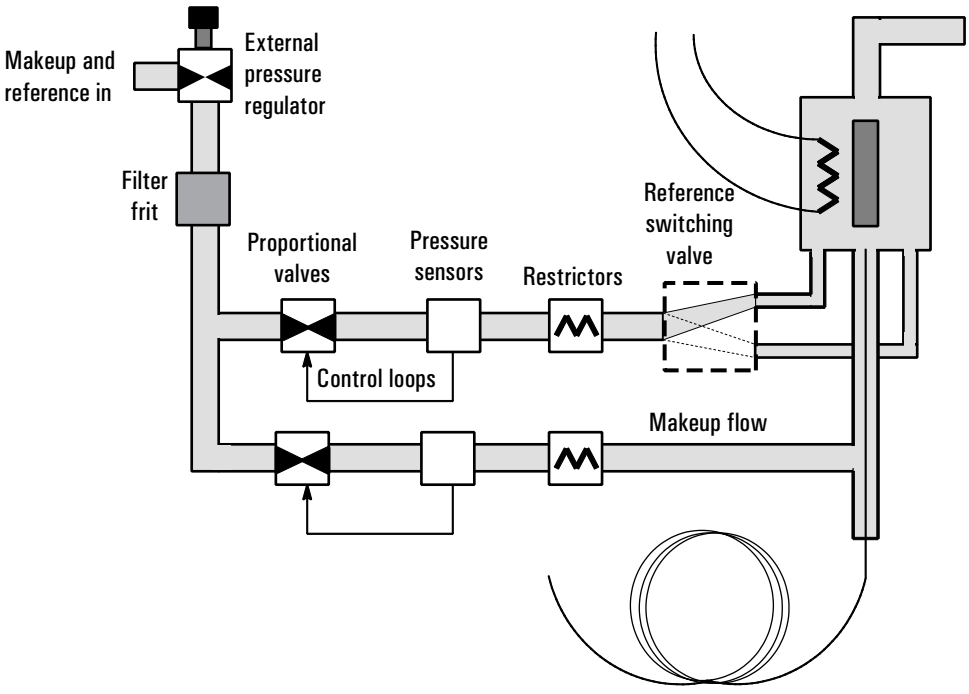



**Figure 31. Internal/External TCD plumbing**

## Thermal cleaning (bakeout)

**Caution** You must turn off the TCD filament and cap the detector column fitting to prevent irreparable damage to the filament caused by oxygen entering the detector.

1. Turn the detector off using a Control Module, ChemStation/Cerity Chemical, or other control device.
2. Remove the column from the detector and cap the detector column fitting.
3. Set the reference gas flow rate between 20 and 30 mL/min. Set the detector temperature to 375°C.
4. Allow thermal cleaning to continue for several hours, then cool the system to normal operating temperatures.

## Replacing the TCD flow module

### Tools and materials

- 7/16-inch wrench
- T-20 Torx driver

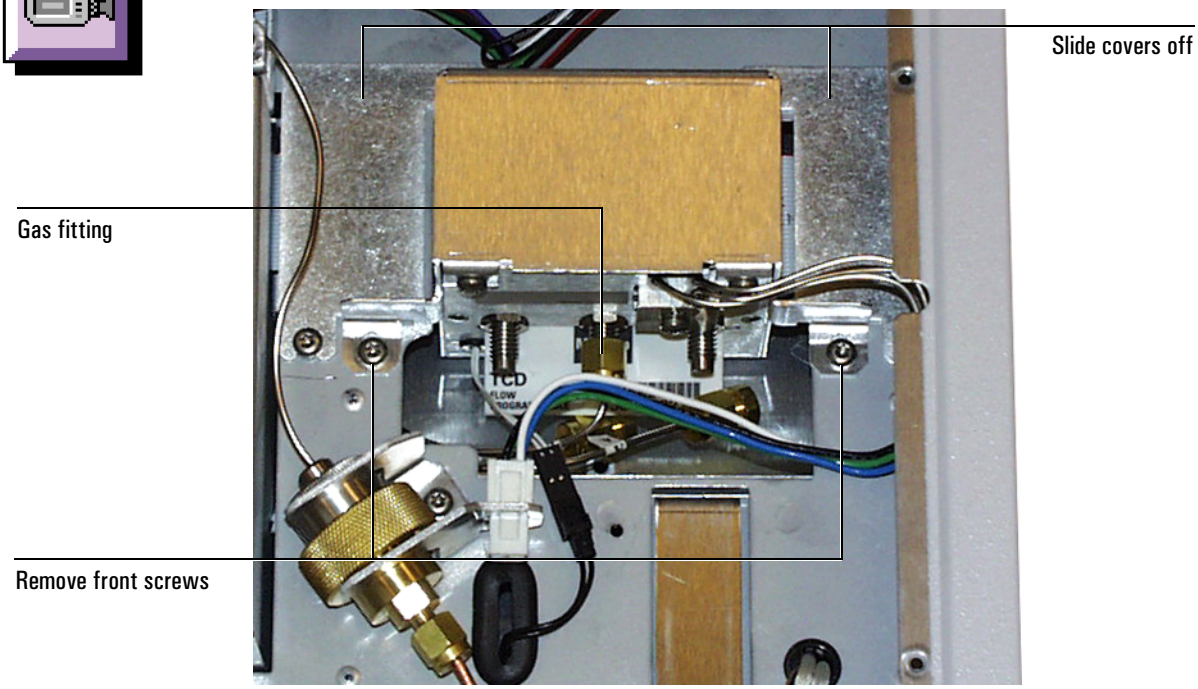
### To remove the flow module

**Warning** Be careful! The oven and/or detector may be hot enough to cause burns.

1. Load the `SERVICE` method or reset the detector and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.
2. Turn the detector pressure off at the source.
3. Turn off the instrument and remove the power cord.
4. Turn off all supply gases at their sources.
5. Disconnect all gases from the bulkhead connectors on the back of the instrument.
6. Remove the lid top cover.

**Caution** Use electrostatic discharge precautions during this procedure.

7. Remove the gas fitting from the flow module.

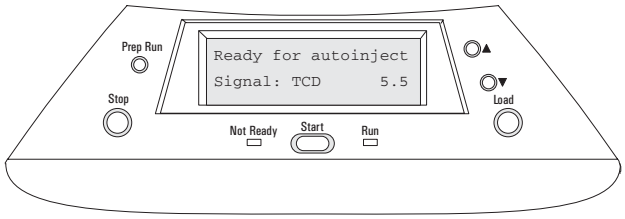



8. Unscrew the manifold block fitting from the flow module.
9. Loosen the two screws securing each of the connector covers, and slide the connector covers off.
10. Push the two tabs to release the ribbon cable connector and disconnect it.
11. Loosen the back screw, remove the two front screws, and pull out the flow module from the lid.
12. Disconnect the TCD switching valve connector at the jumper.

**To install the new TCD flow module**

1. Connect the TCD switching valve connector.
2. Place the module in the lid.
3. Connect the ribbon cable connector.
4. Fasten the flow module to the lid using the three T–20 Torx screws.
5. Slide the connector covers into position and tighten the mounting screws.
6. Install the manifold block fitting and reconnect the gas fittings.
7. Reconnect the supply gases and turn them on. Reconnect the power cord.
8. Turn the GC on.

9. Load a method that uses the TCD.
10. Check that the signal offset value is < 30 after the instrument equilibrates.




## Maintaining a flame ionization detector (FID)

**Warning** Flame ionization detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Detector fittings must have either a column or a cap connected at all times.

### FID gas flow paths

Figure 32 shows the pneumatics design for the FID and Table 36 shows the maximum gas flows.

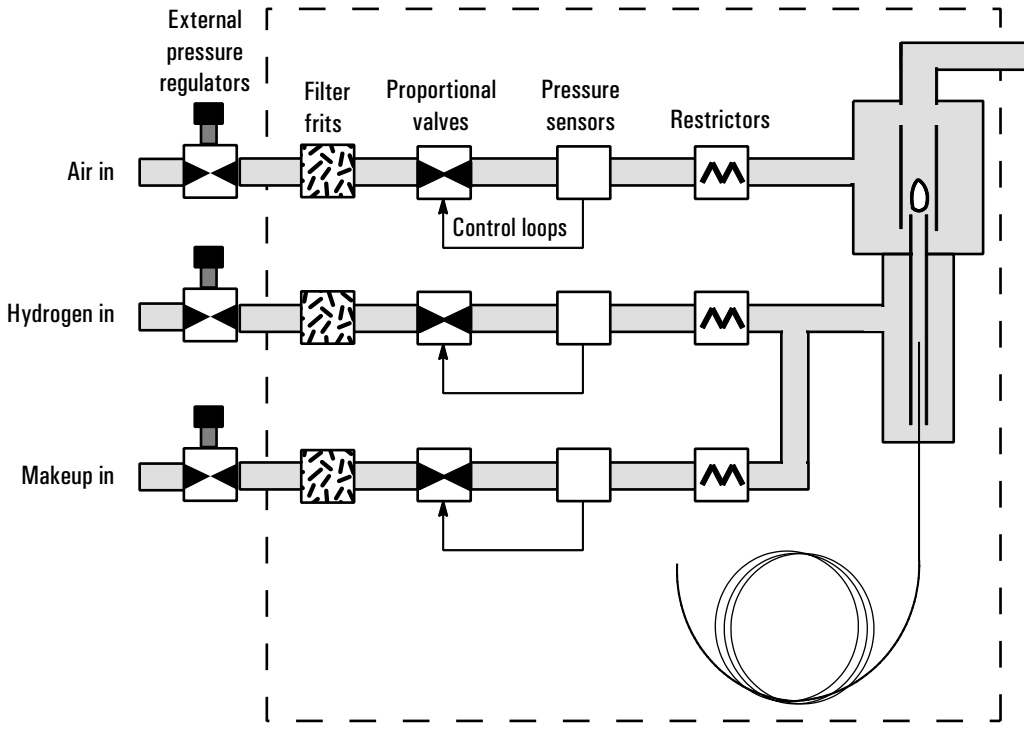



**Figure 32. FID gas flow paths**

**Table 36. Maximum Gas Flows**

| Gas | Maximum flow rate, mL/min |
|---|---|
| Hydrogen | 100 |
| Air | 800 |
| Nitrogen makeup | 100 |
| Helium makeup | 100 |
| Argon makeup | 100 |

## The flame goes out or will not light

- The column flow rate may be too high. Decrease the column flow rate or pressure. Switch to a more restrictive column, for example a longer column or one with a smaller id. If you must use a column with a large id, turn off the column flow long enough to allow the FID to light.
- Check that the gases are plumbed correctly, the external lines have been well purged and the system is leak-free.

- Check that the right type of jet is installed for the column you are using.
- Check that the jet is not damaged. Look for scores, galls, a crimped tube, or a plugged jet.
- Injecting large volumes of aromatic solvent can cause the flame to go out. Switch to a nonaromatic solvent.
- The lit offset value may be too low or too high. Adjust the value.

**Warning** Wear safety glasses for the next procedure. FID exhaust can be harmful.

- Check the ignitor by turning the flame off, then on again. If the ignitor is working properly, you will be able to see a glow from the detector chimney.

### Thermal cleaning (bakeout)

Elevated background signals (>20 display units with the flame on) may be due to contamination in the detector. If so, this bakeout procedure may reduce the background level.

1. Turn the detector off, remove the column from the GC, and cap the detector fitting.
2. Cap the inlet fitting.
3. Reset the detector temperature to 375°C.
4. Reset the oven temperature to 250°C.
5. Ignite the flame and monitor the detector signal; it should rise quickly to some higher value as the FID heats up, then decrease to some reasonably constant value.
6. Reinstall the column in the GC.
7. Restore the original setpoint temperatures and allow the unit at least 2 hours to stabilize. Observe the detector signal to determine if it is now within the acceptable range.
8. If repeated bakeouts do not give an acceptable signal level, the gases may be impure. Higher purity gases and/or traps may be necessary. If cleaning the gases fails to give an acceptable signal level, a thorough mechanical cleaning, as described in the following pages, may be necessary.

## Replacing the FID flow module

### Tools and materials

- 7/16-inch wrench
- T-20 Torx driver

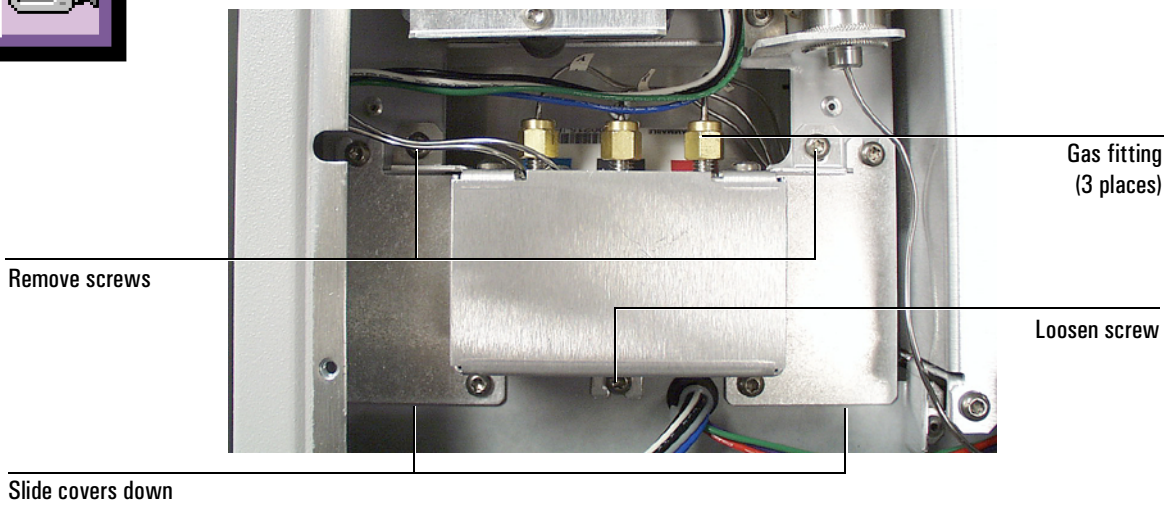
### To remove the flow module

**Warning** Be careful! The oven and/or detector may be hot enough to cause burns.

1. Load the `SERVICE` method or reset the detector and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.
2. Turn the detector pressure off at the source.
3. Turn off the instrument and remove the power cord.
4. Turn off all supply gases at their sources.
5. Disconnect all gases from the bulkhead connectors on the back of the instrument.
6. Remove the lid top cover.

**Caution** Use electrostatic discharge precautions during this procedure.

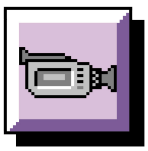
7. Loosen the two screws on each cover plate and slide the covers off. See the following figure.



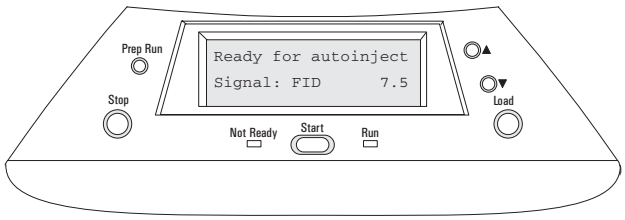

8. Remove the three gas fittings.

9. Unscrew the manifold block from the flow module.
10. Release the ribbon cable connector by pushing the two tabs and disconnect it.
11. Unscrew the two front mounting screws, loosen the back screw, and remove the flow module from the lid.

**To install the flow module**



1. Connect the ribbon cable to the flow module.
2. Attach the manifold block fitting.
3. Place the flow module into position and install it using the three mounting screws.
4. Connect the three gas fittings and tighten.
5. Install the cover plates and tighten the mounting screws.
6. Replace the lid top cover.
7. To check performance, load the method which ignites the flame. Check the offset. It must be between 5 picoamps and less than 20 picoamps after the instrument has equilibrated.




## Removing, inspecting, and replacing the jet

Jets require periodic cleaning or replacement. Even with normal use, deposits develop in the jet (usually white silica from column bleed or black, carbonaceous soot). These deposits reduce sensitivity and cause chromatographic noise and spikes. Although you can clean the jet, it is usually more practical to replace dirty jets with new ones. If you do clean the jet, be very careful not to damage it.

Your detector is shipped with a capillary column jet. If you are doing simulated distillation or high-temperature runs, you must change the jet.

**Table 37. Jets for the FID**

| Jet type | Part no. | Jet tip id |
|---|---|---|
| Capillary | 19244-80560 | 0.29 mm (0.011-inch) |
| Packed | 18710-20119 | 0.47 mm (0.018 inch) |
| Packed wide-bore *(use with high-bleed applications)* | 18789-80070 | 0.030 inch |
| High-temperature *(use with simulated distillation)* | 19244-80620 | 0.47 mm (0.018-inch) |

To change a jet, you must first remove the FID collector assembly. See "Removing the collector" on page 195.

This procedure is divided into three parts: removing and inspecting the jet, cleaning the jet (optional), and installing the jet.

### Tools

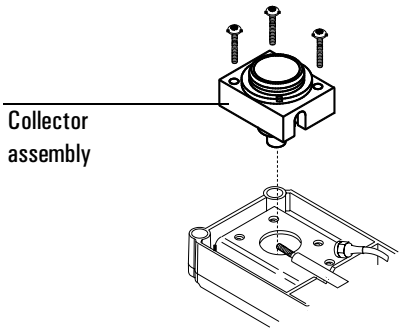
- T-20 Torx screwdriver
- 1/4-inch nut driver

### Procedure

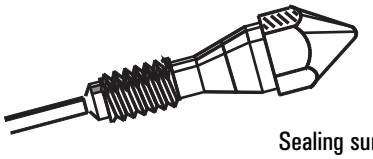
1. Load the `SERVICE` method, or reset the detector and oven temperatures to OFF, or turn the main power switch off. Allow the detector and oven to cool to room temperature.
2. Turn the inlet and detector gas pressures off at the sources.
3. When the oven is cool, remove the column and plug the detector to column fitting.
4. Confirm that the electrometer is turned off. If you are not sure, turn the GC main power off.

**Warning** High temperature.

5. Wear heat resistant gloves, if the detector is hot.
6. Remove the three screws holding the collector assembly in place. Lift off the assembly. The insulator can remain in the collector bottom.

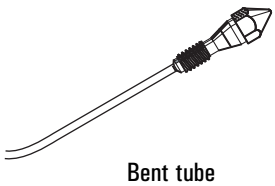



7. Using the nut driver, loosen the jet and pull it straight out. You may need to use the forceps to grasp the jet.
8. Inspect the jet sealing surface for scratches. You should see a ring around the sealing surface; other scratches are unacceptable.



Sealing surface

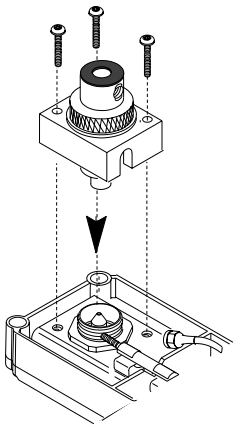
9. Inspect the jet tube to make sure it is not bent or crimped. Inspect the jet for contamination or pieces of broken column by holding it up to a light and looking through it. If no contamination is present, the tube will be clear.



Bent tube

**Caution** Do not over-tighten the jet! Over-tightening may permanently deform and damage the jet, the detector base, or both.

10. Insert the jet and tighten with the nut driver until it is snug. Re-install the collector assembly.



11. Install the column and restore normal operating conditions.

### Cleaning the jet

It is often more convenient to replace dirty jets with new ones than to clean them, especially jets that have been badly contaminated. See "Removing, inspecting, and replacing the jet" on page 191 for more information.

#### Tools

- Small ultrasonic cleaning bath
- Flame detector cleaning kit (part no. 9301–0985)

#### Procedure

1. Remove the jet from the detector. See "Removing, inspecting, and replacing the jet" on page 191.
2. Run a cleaning wire through the top of the jet. Run it back and forth a few times until it moves smoothly. Be careful not to scratch the jet.
3. Aqueous cleaning procedure:
    a. Fill the ultrasonic cleaning bath with aqueous detergent and place the jet in the bath. Clean for 5 minutes.
    b. Use a jet reamer to clean the inside of the jet.
    c. Ultrasonically clean again for 5 minutes.

**Caution** From this point on, handle the parts only with forceps or tweezers!

d. Remove the jet from the bath and rinse it thoroughly with hot tap water and then with a small amount of methanol.

e. Blow the jet dry with a burst of compressed air or nitrogen and then place the jet on a paper towel and allow it to air dry.

## Maintaining the collector

The collector requires occasional cleaning to remove deposits (usually consisting of white silica from column bleed, or black, carbonaceous soot). Deposits reduce sensitivity and cause chromatographic noise and spikes.

The cleaning procedure presented here suggests you use an ultrasonic bath to clean the collector and other parts of the detector. If your collector is not too dirty it may be sufficient to scrub it with a nylon brush and then use a burst of compressed air or nitrogen to blow stray particles away.

This procedure is divided into three steps: removing the collector, cleaning the collector, and reassembling the detector.

### Tools

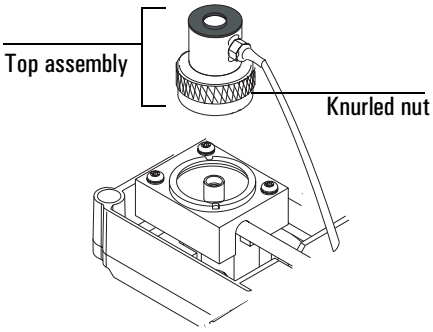
- T-20 Torx screwdriver
- 1/4-inch nut driver
- Small ultrasonic cleaning bath
- Flame detector cleaning kit (part no. 9301–0985)

### Removing the collector

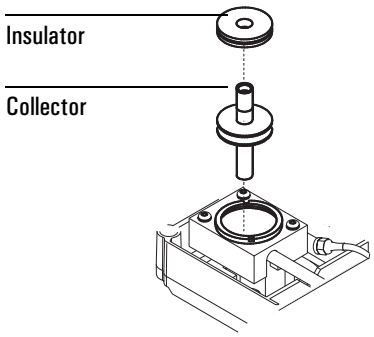
1. Load the `SERVICE` method or reset the inlet, detector, and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.

**Warning** Shock hazard. Turn off the electrometer or the GC main power.

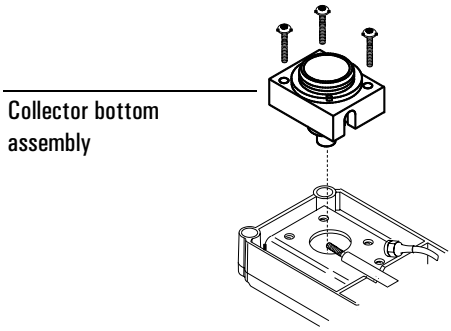
2. Confirm that the electrometer is turned off. If you are not sure, turn the GC main power switch off.
3. Turn the inlet and detector gas pressures off at the sources.
4. Loosen the knurled brass nut. Lift the top assembly straight up. The upper Teflon insulator might stick to the bottom of the assembly. Remove the insulator.




5. Lift out the collector. The upper insulator may be attached to the collector. You may need to use the tweezers to grasp the collector.




6. Remove the three screws that hold the collector bottom assembly in place. Lift off the assembly. Remove the lower insulator from the bottom assembly. You may need to use the forceps to grab it.




### Cleaning the collector

1. Fill the ultrasonic cleaning bath with aqueous detergent, and place the two insulators and the collector in the bath. Ultrasonically clean for 5 minutes.

2. Use the nylon brushes to clean each piece.
3. Replace the pieces in the bath and clean again for 5 minutes.

**Caution** From this point on, handle the parts only with forceps or tweezers!

4. Remove the pieces from the bath and rinse them thoroughly with hot tap water and then with a small amount of methanol.
5. Place the pieces on a paper towel and let them air dry.

### Reassembling the detector

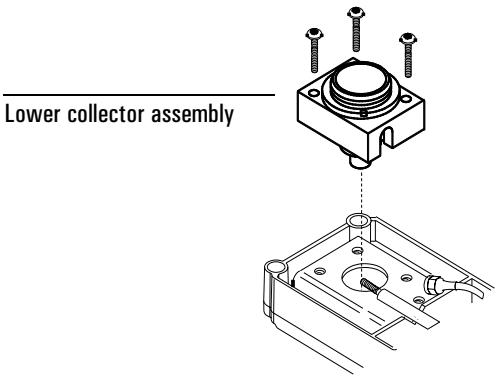
**Caution** Handle the clean collector and insulators only with forceps (or tweezers)!

#### Tools

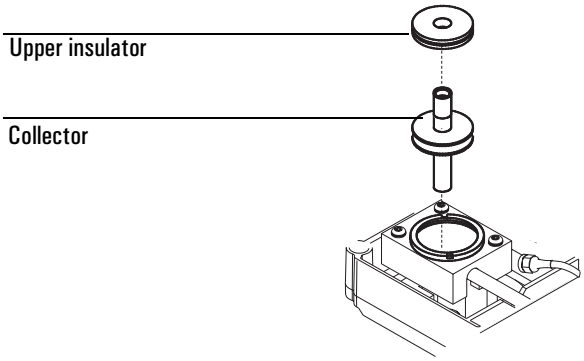
T-20 Torx screwdriver

**Warning** Shock hazard. Turn off the electrometer or the GC main power.

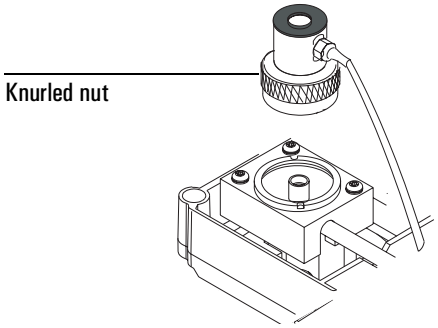
1. Confirm that the electrometer is turned off. If you are not sure, turn the GC main power switch off.
2. Insert the lower insulator into the lower collector assembly. Install the lower collector assembly and tighten the three screws.




3. Replace the collector and install the upper Teflon insulator.




4. Install the upper collector assembly and tighten the knurled nut finger-tight.




5. You can now restore normal operating conditions.

## Replacing the FID ignition wire

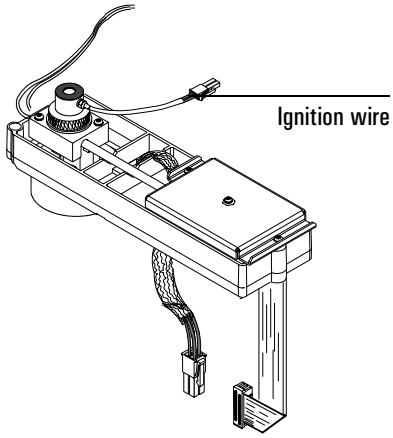
### Tools and materials

- T-20 Torx screwdriver
- ESD wrist strap
- New ignition wire assembly (part no. G1531–60680)

### Procedure

1. Load the `SERVICE` method or reset the detector and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.
2. Turn the inlet and detector gas pressures off at the sources.
3. Remove the lid top cover.

4. Disconnect the ignition assembly at the cable connection. Squeeze the lock and gently pull the connector free.




5. Unscrew the ignition wire from the detector. Do not lose the small copper washer underneath it.
6. Install the new ignition wire.
7. Restore normal operating conditions.

# Maintaining a Microcell Electron Capture Detector (µECD)

Figure 33 is an overview of the µECD and its electrometer.

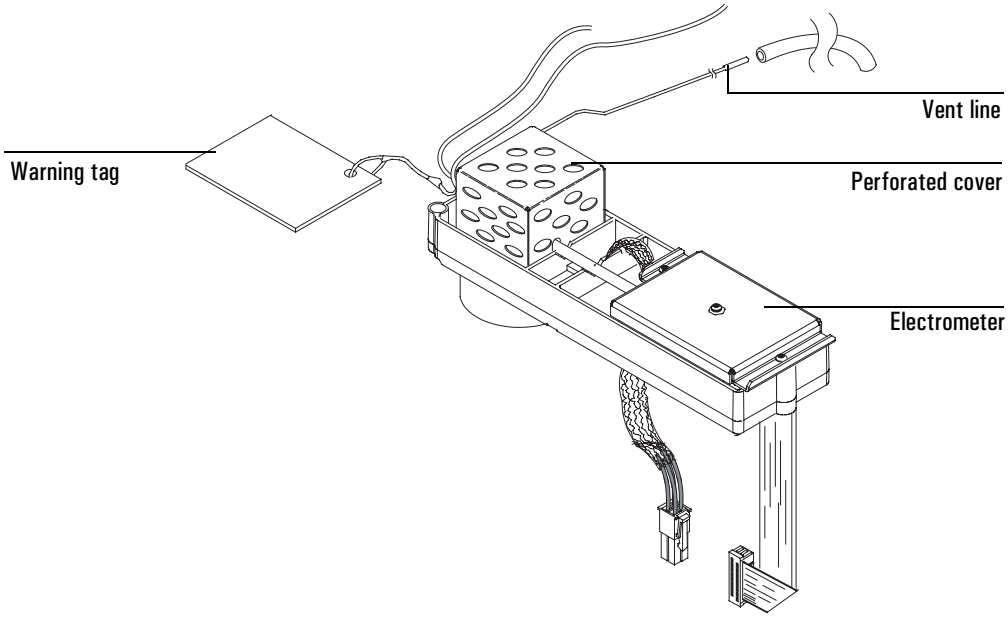



**Figure 33. The µECD**

## Replacing the µECD capillary column adapter liner

To replace the µECD capillary column adapter liner:

1. Remove the adapter cap.

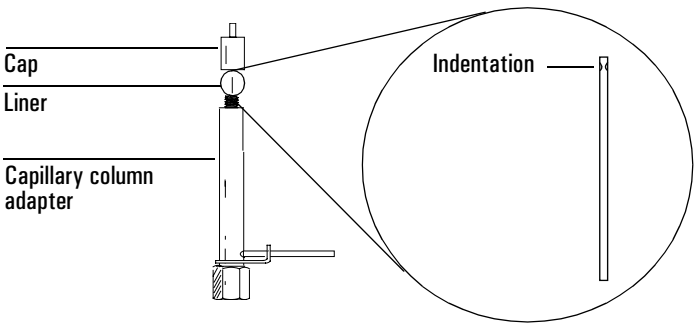



**Figure 34. The µECD adapter**

2. Check the liner. Replace it if it is broken and reinstall the cap. The indentation must be at the cap end of the adapter (See Figure 34).

## Correcting performance problems

Performance problems, such as an output reading that is too high or too low or unsatisfactory chromatographic results (for example, a noisy baseline), can be caused by leaks or deposits in the detector or other part of the chromatographic system. To determine the location of the problem, you need to perform a series of tests.

Before testing the detector, consider the nature of the problem. If you have recently made a change to the GC system and now see an elevated output level, there is a good chance that the change has either introduced contaminants or caused a leak in the system. For example, if you recently switched gas supplies, the new gas may contain impurities. Or if you recently installed a new column, there could be a leak at the detector fitting.

If the output value or noise level has been increasing gradually, the cause is probably a slow build-up of deposits. The detector may contain contaminants from column bleed or a trap may be saturated. If the change has been gradual and if you have not modified the GC system recently, you can probably start by checking for contamination. *Note: Contamination in this procedure refers to non-radioactive deposits from such things as column bleed or dirty samples!*

1. Make sure the detector is operating under normal conditions and that at least 2 hours have lapsed since the last run.

   Check the output value. If it differs considerably from the normal output level—either too high or too low—you should continue with this procedure to identify the cause of the abnormal reading.

2. Use an electronic leak detector to check for leaks at the inlet and detector and the column fittings. Correct leaks and then check the output level. If it is still abnormal, continue to step 3.

3. The detector itself is not a likely source of leaks, so you should leak test the inlet if the output reading is still abnormal. See the maintenance material for your inlet in:

   - "Maintaining a split/splitless inlet" on page 120
   - "Maintaining a purged packed inlet" on page 134
   - "Maintaining a PTV" on page 150
   - "Maintaining a cool on-column inlet" on page 166

   If the inlet is not leaking, go to step 4 to check for leaks in the detector.

   If the inlet is leaking, correct the leaks and check the output. If it is still abnormal, the detector also may be leaking. Go to step 4.

4. Follow the leak test for the detector later in this document.

   If the detector is not leaking, the cause of the problem is contamination. Go to step 5.

   If the detector is leaking, correct the leaks, and then recheck the output. If it is still abnormal, go to step 5.

5. Check for contamination:

   a. Remove the column and plug the detector connection with the cap (part no. 19234-20650) and cap nut (part no. 19234-20570).

   b. Run the detector at your normal operating conditions but with only makeup gas flowing through it. Monitor the output. If it is normal for your detector, then the contamination is from another part of the GC system. Go on to step 6.

   c. If the output is abnormal, then the detector is contaminated. Perform a thermal bake out to decontaminate the detector. The procedure is described later in "Thermal cleaning" on page 207.

6. One part at a time, check the rest of the GC system for contamination by making the following changes and monitoring the output:
   - Replace the column with an empty column and compare the output readings.
   - Switch to a different inlet (if possible), and compare the output.
   - Switch to a different source of gas and compare the output.
   - Replace the traps; compare the output.

## Replacing the μECD flow module

### Tools and materials

- 7/16-inch wrench
- T-20 Torx driver

### To remove the flow module

**Warning** Be careful! The oven and/or detector may be hot enough to cause burns.

1. Load the `SERVICE` method or reset the detector and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.
2. Turn the detector pressure off at the source.
3. Turn off the instrument and remove the power cord.
4. Turn off all supply gases at their sources.
5. Disconnect all gases from the bulkhead connectors on the back of the instrument.
6. Remove the lid top cover to expose the flow module.

**Caution** Use electrostatic discharge precautions during this procedure.

7. Remove the gas fitting from the flow module (Figure 35).

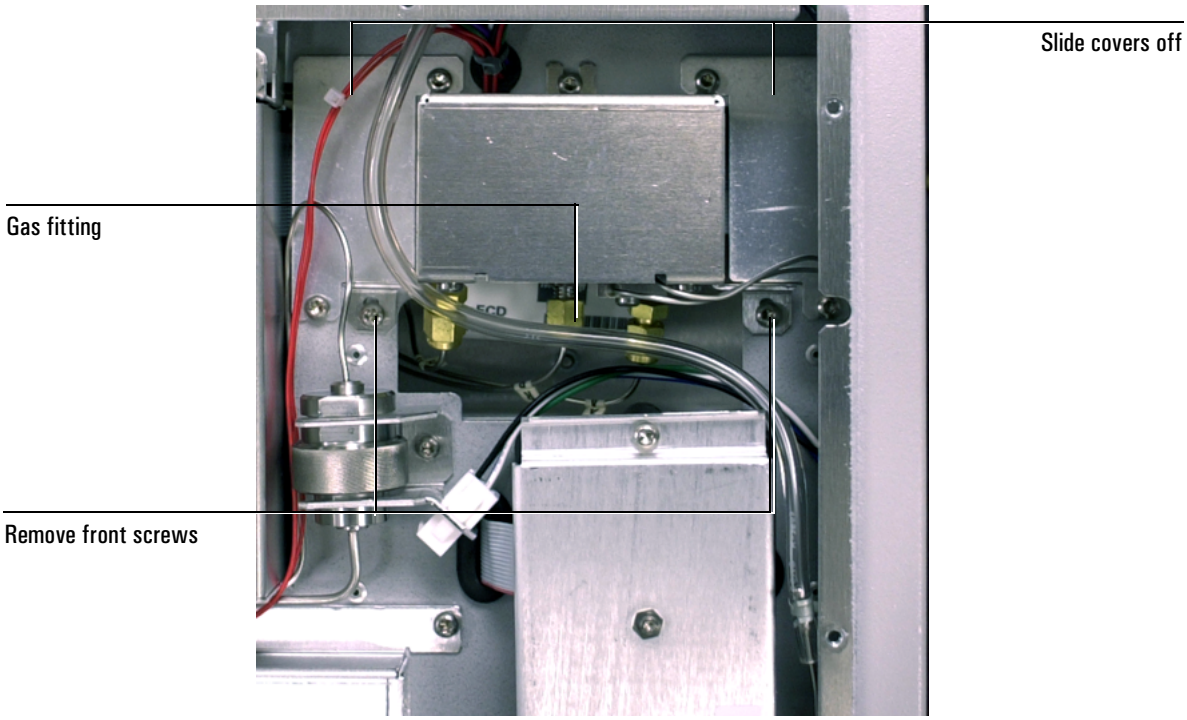



**Figure 35. Removing the flow module**

8. Unscrew the manifold block fitting from the flow module.
9. Loosen the two screws securing each of the connector covers, and slide the connector covers off.
10. Push the two tabs to release the ribbon cable connector and disconnect it.
11. Loosen the back screw, remove the two front screws, and pull out the flow module from the lid.

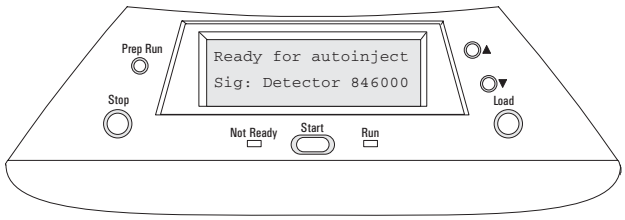
### To install the new µECD flow module

1. Place the module in the lid.
2. Connect the ribbon cable connector.
3. Fasten the flow module to the lid using the three T–20 Torx screws.
4. Slide the connector covers into position and tighten the mounting screws.
5. Install the manifold block fitting and reconnect the gas fittings.
6. Reconnect the supply gases and turn them on. Reconnect the power cord.
7. Turn the GC on.
8. Load a method that uses the µECD.

9. At first, the displayed signal will be very high–about 840,000. As clean gases purge contaminants from the system, the signal should drop to the baseline level seen before the manifold replacement. As a general guideline:

   - A new µECD should have a baseline offset < 200 Hz
   - Any µECD should have a baseline offset < 400 Hz
   - It may take up to 24 hours for a µECD to stabilize

   If a detector shows a baseline > 400 Hz after stabilization, there may be contamination (in the inlet, column, and connections as well as the detector) or a leak anywhere in the system, or the detector may require thermal cleaning, maintenance, or replacement. See the GC service manual for troubleshooting details.




## Checking for gas leaks

The detector is an unlikely leak source. If you suspect that there is a leak in your GC system and have checked the gas plumbing to the GC, the inlet, and the column inlet and detector connections without finding it, follow this procedure to test the detector.

The oven and inlet should be at their normal operating temperatures.

**Materials needed:**

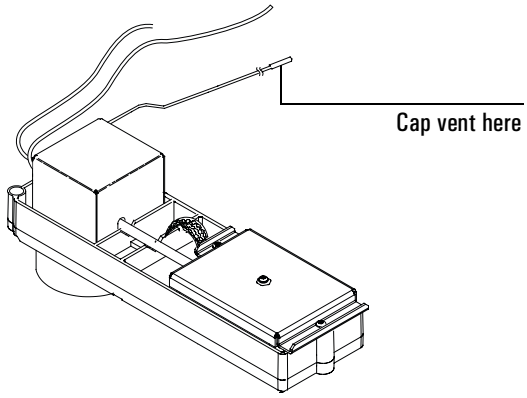
- A vent plug (part no. 5060-9055)
- An electronic leak detector capable of detecting your carrier gas

To perform this procedure, you will need a control module or data system.

1. Turn off the inlet pressure. Allow some time to purge the system of the gas.
2. Turn off the makeup gas flow.

   When there is no flow, the output will be at its maximum, which is approximately 840,000 for both argon/methane and nitrogen.

3. Cap the detector exhaust vent with the vent plug.




4. Set pressure at the inlet to 15 psi (103 kPa). Monitor the system pressure from the inlet. Allow time for the system to become fully pressurized (at least 1 minute). When the system is fully pressurized turn off the pressure or the gas.

   Monitor the pressure for 10 to 15 minutes. If the pressure stays stable or drops only by 0.2 or 0.3 psi/min, you can consider the detector leak-free.

   If pressure drops, you have a leak. Continue to step 5.

5. Use the electronic leak detector to check for leaks at the column fitting and plugged vent. If you find leaks, tighten the fittings, and repeat the leak test.

   If the other system components are leak-free, then the detector may be leaking. **The detector cannot be disassembled without special license from the Nuclear Regulatory Commission or Agreement State Licensing Agency (USA only).** Contact your Agilent service representative for more information.

## Thermal cleaning

If your baseline is noisy or the output value is abnormally high and you have determined that these problems are not being caused by leaks in the GC system, you may have contamination in the detector from column bleed. To remove contamination, you should perform a thermal cleaning (also called "bake-out") of the detector.

**Warning** Detector disassembly and/or cleaning procedures other than thermal should be performed only by personnel trained and licensed appropriately to handle radioactive materials. Trace amounts of radioactive $^{63}$Ni may be removed during other procedures, causing possible hazardous exposure to β- and x-radiation.

**Warning** To prevent possible hazardous contamination of the area with radioactive material, the detector exhaust vent always must be connected to a fume hood, or otherwise vented in compliance with the latest revision of Title 10, CFR, Part 20, or with state regulations with which the nuclear regulatory commission has entered into an agreement (USA only). For other countries, consult with the appropriate agency for equivalent requirements.

**Materials needed:**

- Cap for the detector connection (part no. 19234-20650)
- The nut to connect the cap (part no. 19234-20570)

This procedure requires a control module or data system to edit Go set points.

1. With the detector and oven at normal operating temperatures, press ▲ or ▼ to scroll to the detector output, or view the output on the control module or data system. Note its value for later comparison.
2. Turn off the anode purge and the makeup gas flow.
3. Cool the oven and detector to a safe handling temperature.
4. Remove the column from the detector. Make sure to cap the unconnected end. Install the detector cap and nut into the column detector fitting to plug the connection.
5. Make the following settings:

| | |
|---|---|
| Detector temperature | 350 to 375°C |
| Makeup gas flow | 60 mL/min |
| Oven temperature | 250°C |

6. Allow thermal cleaning to continue for several hours and then cool the system to normal operating temperatures.
7. Check the µECD output value. It should be lower than the first reading. If it is not, contact your Agilent service representative.

### Performing a wipe test (radioactivity leak test)

Electron capture detectors must be tested for radioactive leakage at least every 6 months or as required by your local Agency. Records of tests and results must be maintained for possible inspection by the Nuclear Regulatory Commission and/or responsible state agency. More frequent tests may be conducted when necessary.

The procedure used is the **wipe test**. A wipe test kit is supplied with each new detector. Refer to the information card supplied in the wipe test kit for instructions on performing the test.

## Maintaining the flame photometric detector (FPD)

**Caution** Do not store the Flame Photometric Detector (FPD) at temperatures above 50°C, since this may damage the photomultiplier tube (PMT).

### Flame ignition problems

If the FPD flame will not light or stay lit, check/do the following:

1. Be sure there is a problem. Confirm ignition by holding a mirror or shiny surface near the aluminum exhaust tube, with the rubber drip tube removed, and observing condensation if the flame is lit.
2. Using a control module or data system, check the `Lit offset`. If it is zero, autoignition is turned off. If it is too large, the GC will not know that the flame is lit and will shut the detector down.
3. Increase the air supply pressure to the GC. This makes the flame easier to light but does not affect the air flow rate setpoint.
4. If the flame does not light at all, check the glow plug circuit. Use the GC display or a control module to display detector output. Observe the actual signal output, which will momentarily go to greater than 65500 counts when the flame lights. If the display does not change, check the lead connection on the glow plug. If the glow plug has failed, replace it with part no. 0854-0141.
5. The flame is easier to light at higher detector temperatures.

6. Under some operating conditions, the flame may be more easily lit with the rubber drip tube removed. After lighting the flame, reinstall the drip tube.
7. If the flame still will not light, there could be a large leak in the system. This results in measured flow rates being different from actual flow rates, causing non-ideal ignition conditions. Thoroughly leak check the whole system.
8. If the analysis permits, substitute nitrogen for helium as carrier and makeup.
9. Increase hydrogen and air flow rates until ignition occurs, then reduce them toward the recommended values. Experiment for the best values.

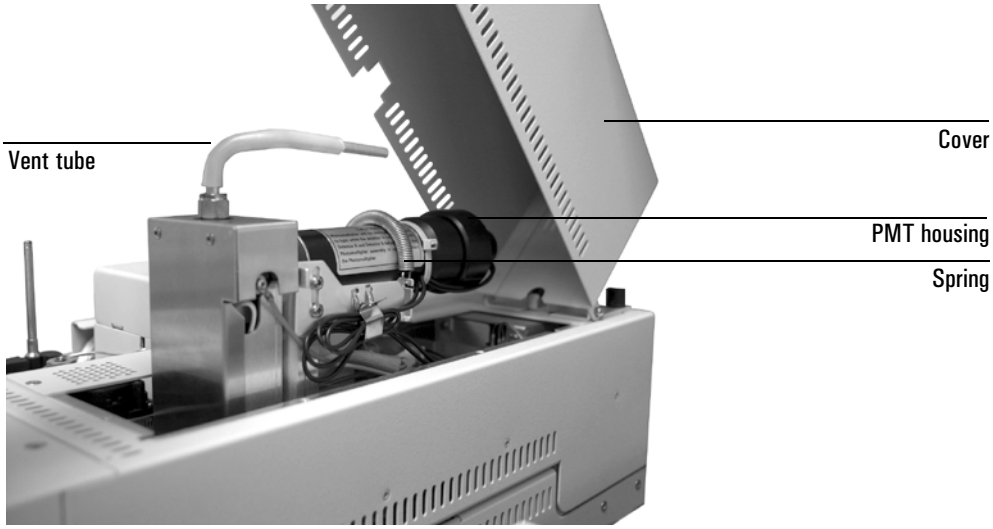
## Changing wavelength filters

Install the correct optical filter, depending on the choice of Sulfur or Phosphorus mode. For Sulfur Mode, use the 393 nanometer filter (part no. 19256-80000). For Phosphorus Mode, use the 525 nanometer filter (part no. 19256-80010).

To change the filter:

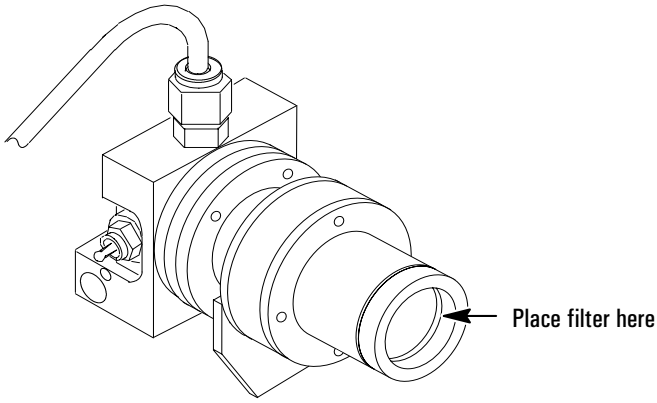
**Warning** Turn off the main power switch located on the left side of the GC. If the photomultiplier tube is exposed to room light with the power on, it will be destroyed.

1. Loosen the thumbscrew on the detector cover and lift the cover.




2. Release the retaining spring around the photomultiplier housing.
3. Pull the photomultiplier housing off the detector body. A twisting motion helps.
4. Remove the old filter. Use tissue to avoid fingerprints.

5. Place the new filter in the recess so that the silvered side faces the flame.




6. Push the PMT housing as far onto the detector body as it will go.
7. Install the retaining spring around the housing.
8. Restore power.

## Replacing the FPD flow module

### Tools and materials

- 7/16-inch wrench
- T-20 Torx driver

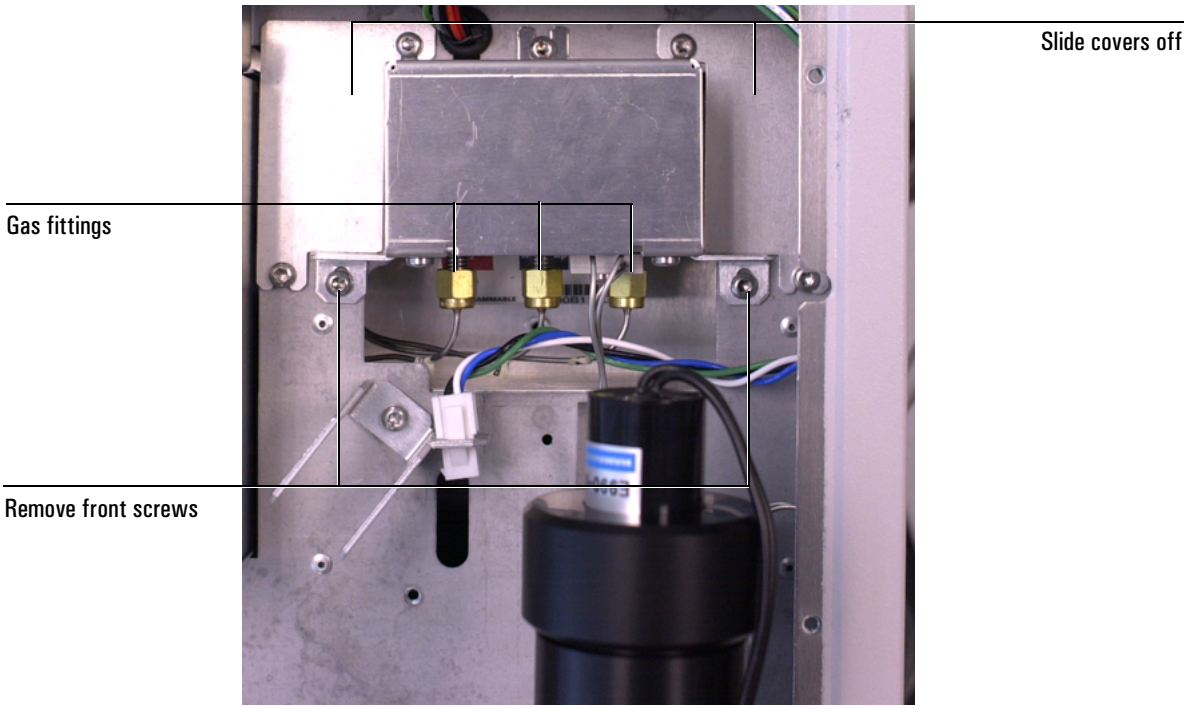
### To remove the flow module

**Warning** Be careful! The oven and/or detector may be hot enough to cause burns.

1. Load the `SERVICE` method or reset the detector and oven temperatures to OFF or turn the main power switch off. Let the heated zones cool to room temperature.
2. Turn off all supply gases at their sources.
3. Turn off the instrument and remove the power cord.
4. Disconnect all gases from the bulkhead connectors on the back of the instrument.
5. Remove the lid top cover.

**Caution** Use electrostatic discharge precautions during this procedure.

6. Remove the gas fittings from the flow module.

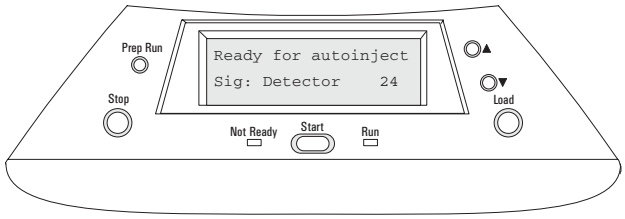



7. Unscrew the manifold block fitting from the flow module.
8. Loosen the two screws securing each of the connector covers, and slide the connector covers off.
9. Push the two tabs to release the ribbon cable connector and disconnect it.
10. Loosen the back screw, remove the two front screws, and pull out the flow module from the lid.

### To install the new FPD flow module

1. Place the module in the lid.
2. Connect the ribbon cable connector.
3. Fasten the flow module to the lid using the three T–20 Torx screws.
4. Slide the connector covers into position and tighten the mounting screws.
5. Install the manifold block fitting and reconnect the gas fittings.
6. Reconnect the supply gases and turn them on. Reconnect the power cord.
7. Turn the GC on.
8. Load a method that uses the FPD.

9. Check that the signal offset value is < 40pA after the instrument equilibrates.




## Leak testing

Turn off all supply gases. Cap the detector exhaust tube with a 1/4-inch Swagelok plug (part no. 0100-0196) and a 40% graphitized Vespel ferrule (part no. 0100-1061).

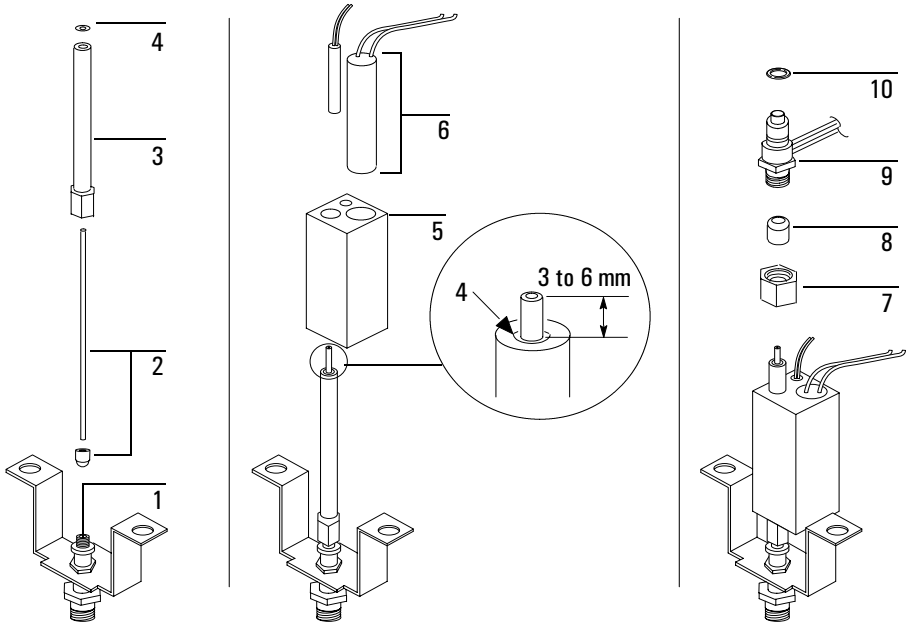
**Caution** When testing the flow system under pressure, do not exceed 210 kPa (30 psig). Higher pressures may damage the detector block window or seals.

Turn one of the gases on for a few seconds and then turn it off. Monitor the detector flow using a control module or data system. The indicated flow (which is actually measured as a pressure) should remain constant or drop slowly. If not, there is a leak in the system. Begin checking possible leak sources and monitor the flow number to determine when the leak has been eliminated.

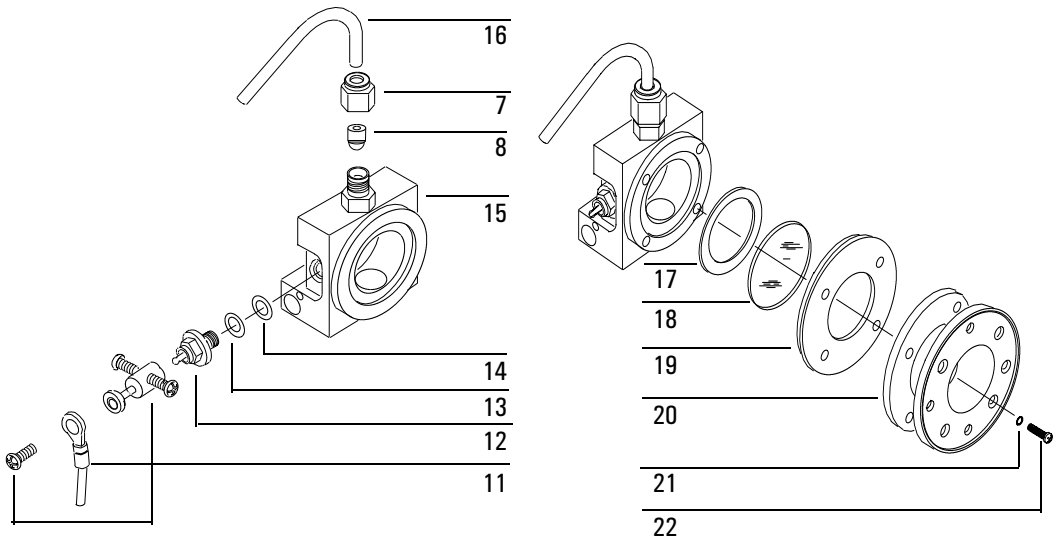
Possible leak sources, in order of decreasing probability, are:

- Septum
- Column fittings
- Supply line swage-type plumbing connections
- Detector block O-ring or Vespel seals
- Other system plumbing

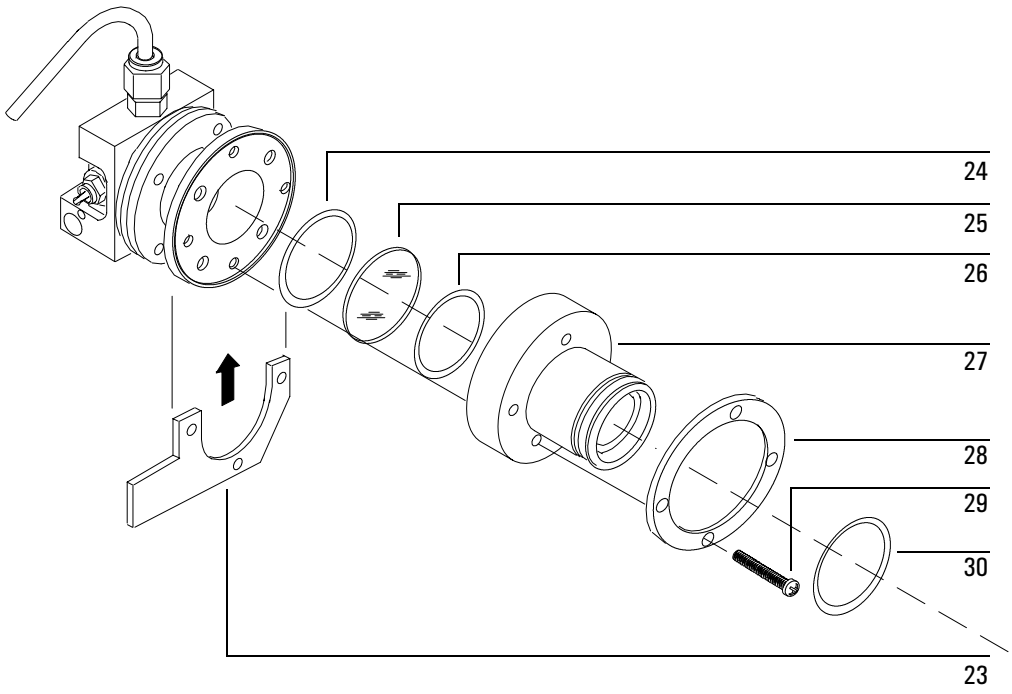
## Parts identification




| Item | Description | Part no. |
|---|---|---|
| 1 | Base assembly weldment | |
| 2 | Gigabore liner/ferrule assembly | 19256-60590 |
| 3 | Transfer tube | 19256-80550 |
| 4 | O-ring, Kalrez, transfer tube | 0905-1101 |
| 5 | Lower heater block | |
| 6 | Heater/sensor assembly | |
| 7 | Nut, brass, 1/4-inch | 0100-0056 |
| 8 | Ferrule, Vespel, 1/4-inch id | 5080-8774 |
| 9 | Jet cartridge | G1535-80500 |
| 10 | O-ring, Kalrez, jet cartridge | 0905-1103 |




| Item | Description | Part no. |
|---|---|---|
| 7 | Nut, brass, 1/4-inch | 0100-0056 |
| 8 | Ferrule, Vespel, 1/4-inch id | 5080-8774 |
| 11 | Ignitor cable assembly | G1535-60600 |
| 12 | Glow plug | 0854-0141 |
| 13 | Spacer, ignitor | 19256-20590 |
| 14 | O-ring, Kalrez, ignitor | 0905-1102 |
| 15 | Weldment, block | |
| 16 | Exit tube assembly, aluminum<br>Exit tube assembly, stainless steel | 19256-20700<br>19256-20705 |
| 17 | Gasket, head shield | 19256-80040 |
| 18 | Window, first heat shield | 19256-80030 |
| 19 | Disk, heat shield | 19256-20580 |
| 20 | Coupling, stainless steel | 19256-20550 |
| 21 | Lockwasher (4 required) | 2190-0108 |
| 22 | Screw, M3 x 12 (4 required) | 0515-0911 |




| Item | Description | Part no. |
|---|---|---|
| 23 | Clamp | 19256-00090 |
| 24 | O-ring, silicone, 0.926-inch id (orange) | 0905-0955 |
| 25 | Window, second heat shield | 19256-80060 |
| 26 | O-ring, silicone, 1.05-inch id (orange) | 0905-1104 |
| 27 | Flange adapter | |
| 28 | Flange ring | 19256-00200 |
| 29 | Screw, M3 x 25 (4 required) | 0515-0065 |
| 30 | O-ring, Viton, 1.239-inch id (brown) | 0905-1100 |
| Filters (not shown) | | |
| | Sulfur mode | 19256-80000 |
| | Phosphorus mode | 19256-80010 |

# Automatic Liquid Sampler and Injector

Refer to the instructions below to use and maintain your 6850 Automatic Liquid Sampler (ALS) or Automatic Injector, if installed.

## Pre-run checklist

Use this checklist to make sure the sampler is ready before you begin.

- ❒ Sample vials are half full
- ❒ Cap is centered, no wrinkles, septum is flat
- ❒ Sample vial positions match the run parameters
- ❒ 4.5 mL of fresh solvent in each solvent bottle
- ❒ Waste bottles are empty
- ❒ There is enough solvent or waste capacity for your sample vials
- ❒ Syringe is new or clean
- ❒ Syringe design and size are correct
- ❒ Plunger is secure in plunger carrier loop
- ❒ Needle is aligned with septum retainer nut
- ❒ Syringe is rinsed with solvent
- ❒ GC inlet liner is clean and deactivated
- ❒ GC inlet liner or insert is correct type for injection technique
- ❒ GC inlet septum type is correct
- ❒ GC inlet septum is less than 200 injections old
- ❒ Correct septum nut is installed in GC inlet

## Running a sample

When running a sample, keep your hands away from the syringe needle. The needle is sharp and may contain hazardous chemicals.

To operate your automatic liquid sampler or injector:

1. Install a clean syringe. See “Installing a syringe” on page 222.
2. Fill the sample vials. See “Preparing sample vials” on page 218

3. Fill the solvent and waste bottles. See "Preparing the solvent and waste bottles" on page 220.
4. Load the solvent bottles, waste bottles, and sample vials into the turret.
   - 6850 Automatic Liquid Sampler—See "The sample turret" on page 229.
   - 6850 Automatic Injector—See "Loading the turret" on page 236.
5. Load (or program) the GC sequence.
6. Run the GC sequence.

When the GC becomes ready, the sampler or injector begins the injections.

## Preparing sample vials

### Selecting sample vials

The sampler and injector turrets use clear or amber glass sample vials with crimp-caps or screw-caps. Use amber glass vials for light-sensitive samples. Refer to your Agilent catalog for consumables and supplies for acceptable vial types. Incompatible sample vials cause turret errors.

Figure 36 shows the critical dimensions for sample vials and microvial inserts to be used with the sampler and injector. These dimensions do not make up a complete set of specifications.

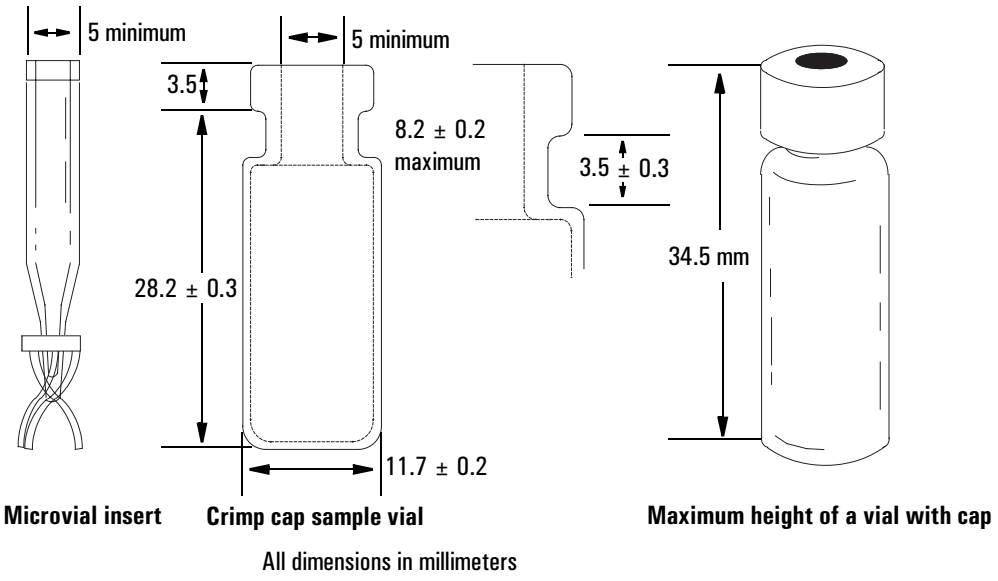



**Figure 36. Dimensions for sample vials and microvial inserts**

## Labeling sample vials

Vials are available with a write-on spot for easy marking. If you choose to make and apply your own labels, Agilent Technologies recommends the positioning and maximum label thickness shown in Figure 37.

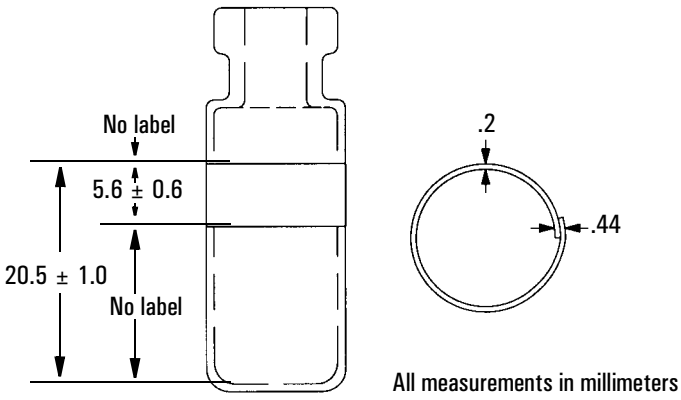



**Figure 37. Label specifications**

## Sample vial septa

There are two types of septa used with crimp caps and screw-on caps, each with different resealing characteristics and different resistance to solvents.

One type is natural rubber formulation coated with Teflon on the sample side. This septum is suitable for samples with a pH range of 4.0 to 7.5. It is less resistant to solvents after puncture and is more easily cored than silicone rubber. Coring may deposit septum pieces in the vial and affect your chromatograms.

Another is high-quality, low-extractable silicone rubber septa, coated with Teflon on one or both sides. This is more resistant to solvents after puncture and to coring by the needle.

Refer to your Agilent catalog for consumables and supplies for more information.

Figure 38 shows the diameter for vial cap apertures.

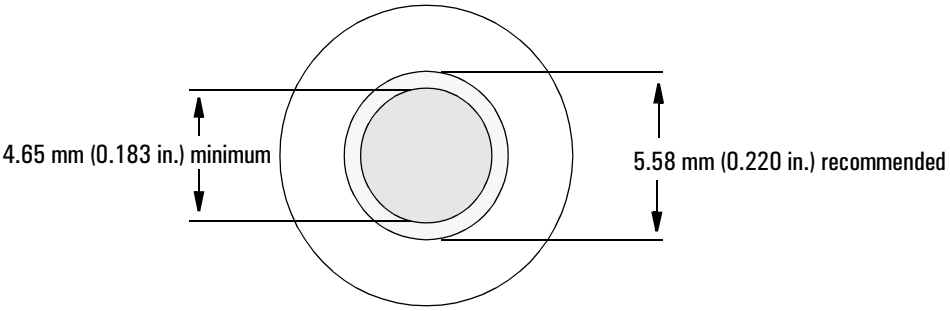



**Figure 38. Vial cap aperture specifications**

### Filling sample vials

Recommended fill volumes are:

- 1 mL for the 2-mL vial
- 50 µL for the 100-µL vial

Refer to the *Sampling Techniques Handbook* on the CD-ROM for more information on sample vial volumes and how it can effect chromatographic results.

## Preparing the solvent and waste bottles

Solvent bottles hold solvent for rinsing the syringe between injections. The liquid sampler or injector discards the solvent washes and sample washes into waste bottles. The number of samples that can be analyzed may be limited by the wash or waste bottle capacity shown in Figure 39 and Figure 40.

Rinse and fill each solvent bottle with 4 to 4.5 mL of fresh solvent. The liquid level will be near the shoulder of the bottle. Good laboratory practice dictates using no more than 2.0 mL of the 4.5 mL solvent for syringe washes. The needle tip draws solvent at a height of 18.5 mm from the bottom of the vial. See Figure 39.

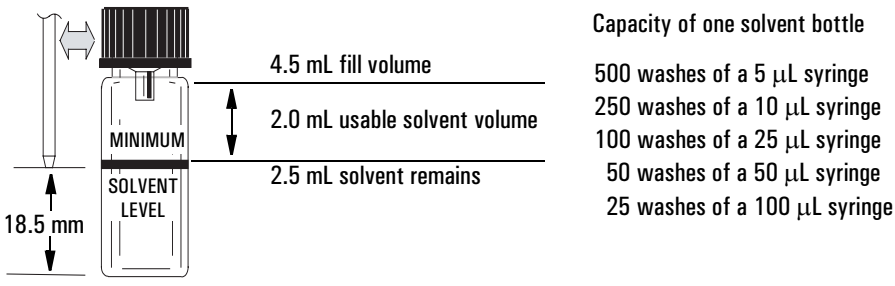



**Figure 39. Position of syringe tip when withdrawing solvent**

Empty and rinse each waste bottle after each multiple vial run. The syringe can discard about 4 mL of waste into a waste bottle. See Figure 40.

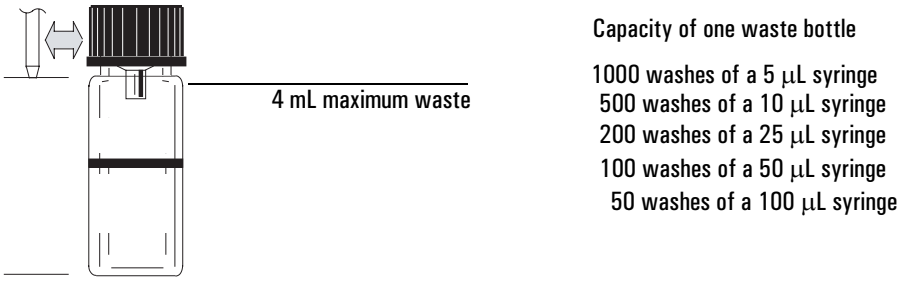



**Figure 40. Position of syringe tip when discarding waste**

## Syringes

### Inspecting a syringe

Before installing a syringe:

Roll the syringe on the edge of a clean flat surface. If the tip of the needle moves in a circle, straighten the shaft by bending it slightly near where it connects to the syringe barrel and check it again. See Figure 41.

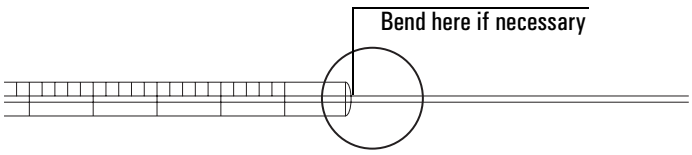



**Figure 41. Syringe needle inspection**

Check for a rough needle. The needle surface may have closely spaced concentric ridges that act like a miniature file and abrade pieces of the septum into the inlet or vial. The ridges are easy to see under 10X magnification.

If there are ridges, polish the needle by pulling it through a folded piece of fine emery paper between your finger and thumb until the ridges are gone. Be careful not to modify the tip of the syringe.

Check for a sticky plunger. Slide the plunger of the syringe up and down a few times. It should move smoothly—without sticking or binding. If it is sticky, remove the plunger, and clean it with solvent. For more information, see your *Sampling Techniques Handbook*.

### Installing a syringe

To install a syringe:

1. Disconnect the sampler or injector cable from the GC and lay the sampler or injector on its back on a flat surface.
2. Open the door.
3. Slide the syringe carriage up (or down) until the syringe is accessible. See Figure 42.
4. Pass the syringe needle through the hole in the needle support foot.
5. Align the syringe barrel with the flange guide and syringe clip and press the syringe into place, keeping the needle in the hole of the needle support foot.
6. Close the syringe latch by swinging it clockwise.

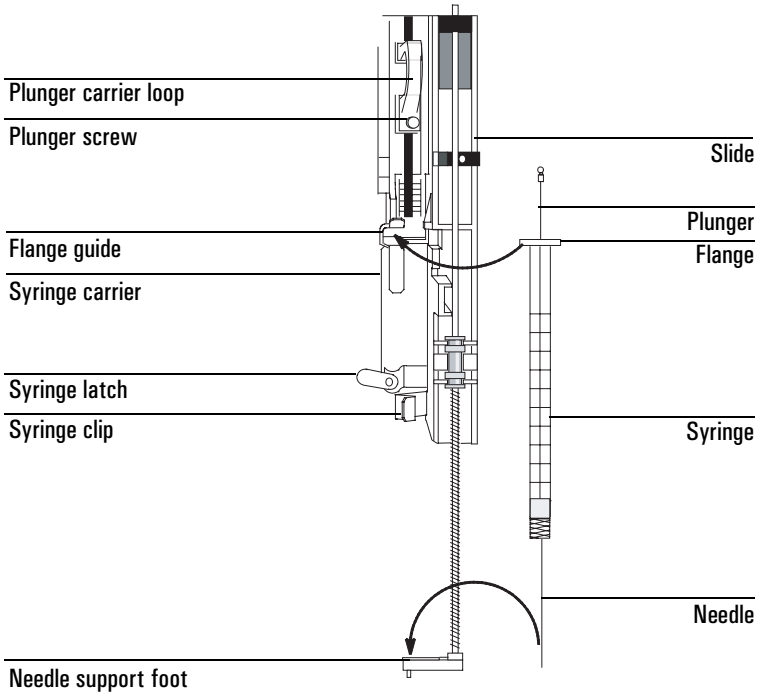



**Figure 42. Installing the syringe**

7. Move the plunger carrier loop down and tighten the plunger screw.

8. Move the plunger carrier loop up and down. If the syringe plunger does not move along with the carrier, repeat the previous steps. Be sure the plunger screw is tight.
9. Check that the needle is aligned with the needle guide in the foot by moving the slide up and down. The needle should slide smoothly in the needle guide. See Figure 43.

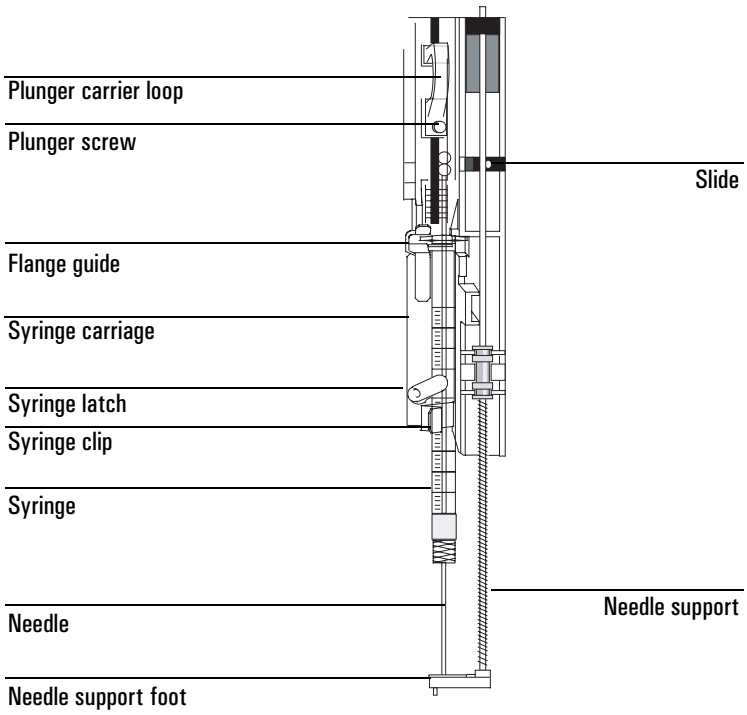



**Figure 43. Syringe carriage and needle support with needle installed**

**Caution** Do not operate the injector or sampler without a syringe in place because the syringe latch may interfere with the motor if it is allowed to swing freely.

10. Pull down the syringe carriage until the needle tip is near the top of the inlet septum nut.

    The needle should be centered exactly over the hole in the septum retainer nut. Make sure the needle will hit the septum without rubbing on the nut.
11. If the needle is not centered over the septum retainer nut, check that the syringe is installed correctly in the syringe carrier, the syringe needle is straight, and the needle support assembly is properly installed.

### Removing a syringe

1. Disconnect the injector or sampler cable from the GC and lay the injector or sampler on its back on a flat surface.
2. Loosen the plunger screw and raise the plunger carrier loop off the syringe.
3. Open the syringe latch.

**Caution** Be careful not to bend the syringe needle. Only pull the syringe out of the carriage until clear. The needle bends easily when still seated in the needle support.

4. Carefully lift the syringe flange out of the flange guide until clear, then lift the syringe needle out of the needle support.

### Replacing the needle support assembly

To *remove* the needle support assembly (Figure 44):

1. Remove all vials from the turret, disconnect the injector or sampler cable from the GC and lay the injector or liquid sampler on its back on a flat surface.
2. Open the door.
3. Remove the syringe.

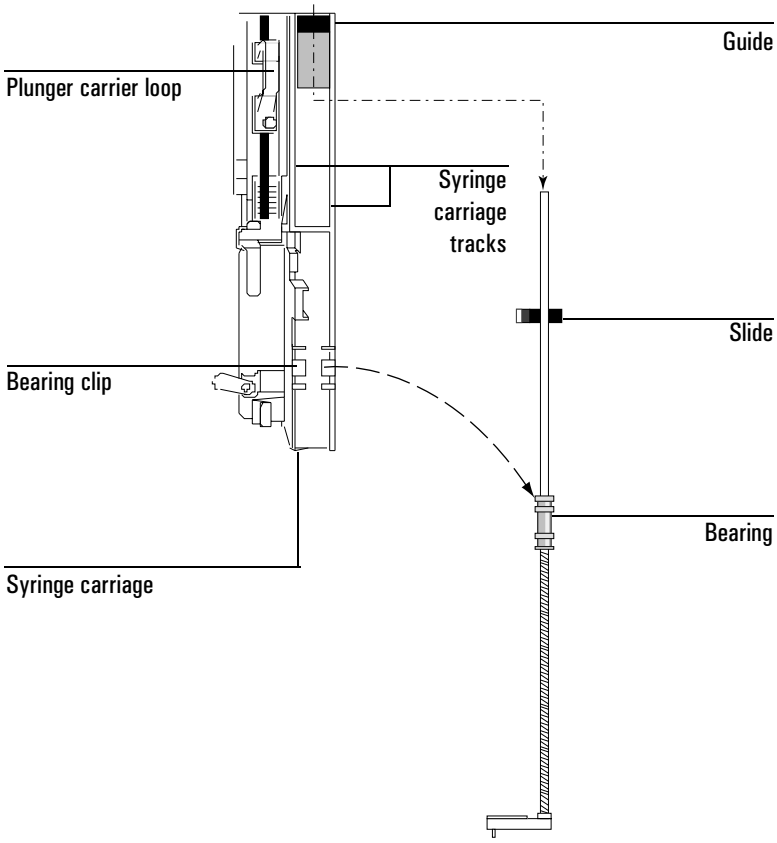



**Figure 44. Removing the needle support assembly**

4. With your finger under the shaft near the bearing on the needle support assembly, pull gently to release the bearing from the bearing clip in the syringe carriage.
5. Carefully use the bearing to pull the rod down until you can lift the assembly out of the syringe carriage.

**Caution** Be careful not to pull the assembly *by its metal shaft*. The shaft is easily bent.

6. To *install* the needle support assembly, select the proper type using Figure 45. Hold it in your right hand and insert the upper end of the rod into the plastic guide to the right of the plunger carrier loop.

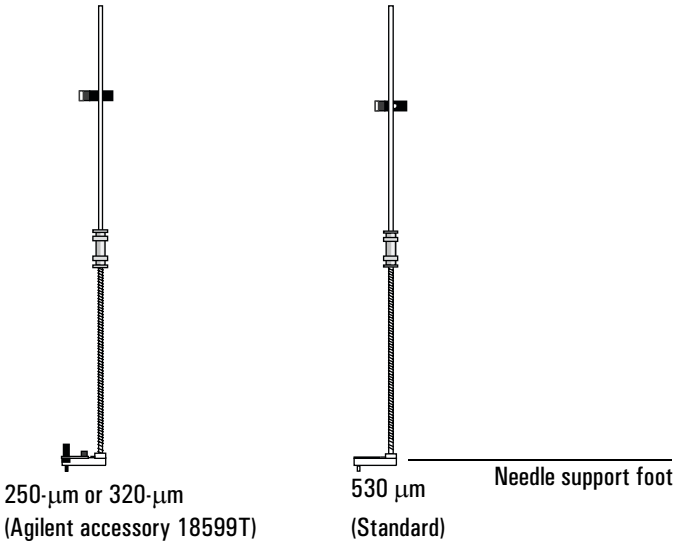



**Figure 45. Needle support assembly**

7. Turn the needle support assembly so that the flat surface of the slide glides up and down the syringe carriage tracks as shown in Figure 46.

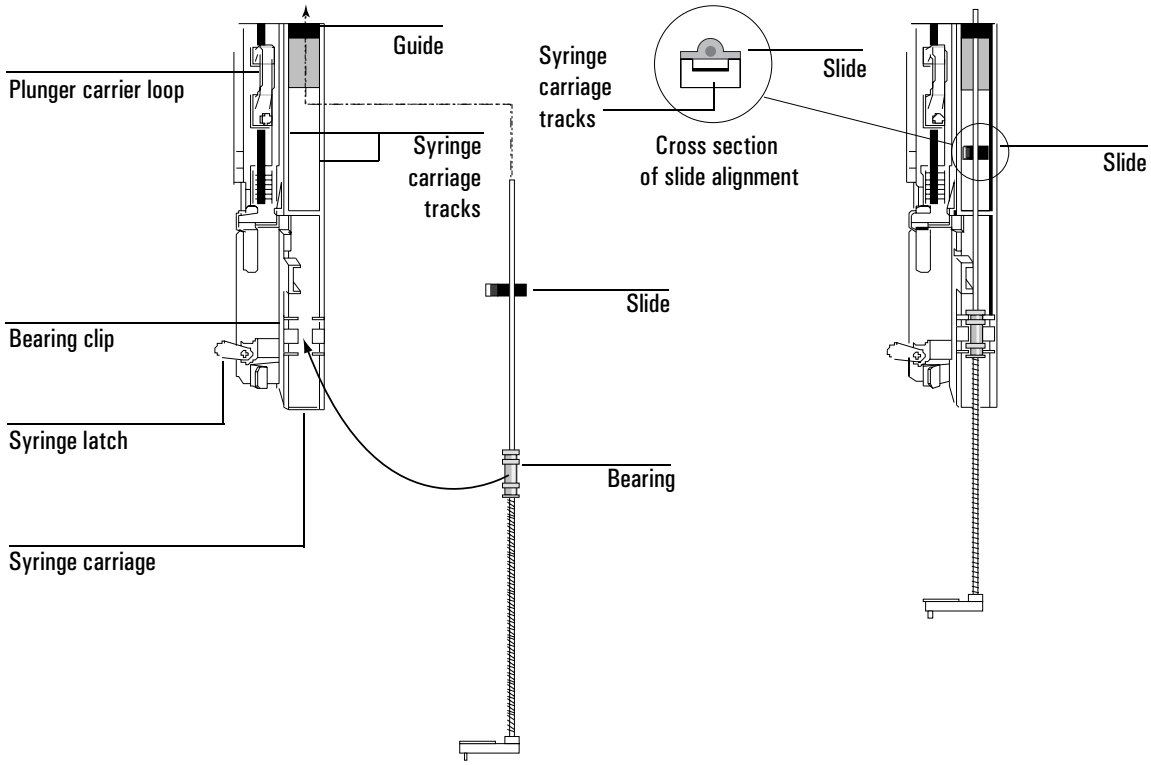



**Figure 46. Installing the needle support assembly**

8. Align the bearing on the needle support assembly with the plastic bearing clip to the right of the syringe latch and push gently on the bearing until the assembly snaps into place.
9. Place the injector or liquid sampler back on the GC or on a parking post.

**Caution** Be careful not to bend the needle during installation.

**Caution** Do not operate the injector or sampler without a syringe in place because the free swinging syringe latch may interfere with the motor.

10. Install the syringe. See "Installing a syringe" on page 222 for details.
11. Move the slide up and down to make sure the needle is aligned with the needle guide in the support foot. The needle should slide smoothly in the needle guide.

# Cool on-column injection

## Technique

If using a cool on-column inlet, the automatic liquid sampler or injector performs injections directly onto 250-µm, 320-µm, and 530-µm columns.

When performing on-column injections, the injector:

- Slows the carriage speed so the overall injection time increases to 500 milliseconds.
- Lowers the tip of the syringe needle an additional 19 mm onto the column.

## Adapting for cool on-column injection

To adapt the injector and GC for cool on-column use:

1. Select the on-column syringe needed for the column size. See your Agilent catalog for consumables and supplies and "Maintaining a cool on-column inlet" on page 166 for a list of parts.
2. Prepare the GC inlet. See "Maintaining a cool on-column inlet" on page 166.
   - Check your needle to column size.
   - Verify that the insert matches the needle size.
   - If necessary, replace the septum.
3. Verify that the correct needle support assembly is installed in the injector. Replace it if necessary. See "Replacing the needle support assembly" on page 224.
4. If necessary, reinstall the injector onto the GC.
5. Install the syringe. See "Installing a syringe" on page 222.
6. Rotate the turret clockwise until it stops, then verify the installation by manually sliding the syringe carriage down until the needle enters the inlet.

# The 6850 automatic liquid sampler

## The sample turret

The sample turret contains positions for 27 2-mL vials. The optional 4-mL sample vial turret has a capacity of 22 vials. See Figure 47. When used with Agilent Cerity Chemical or ChemStation control software, the sample vials can be analyzed in random order. If controlled using a G2629A Control Module, you must load your sample vials in the order you want them run.

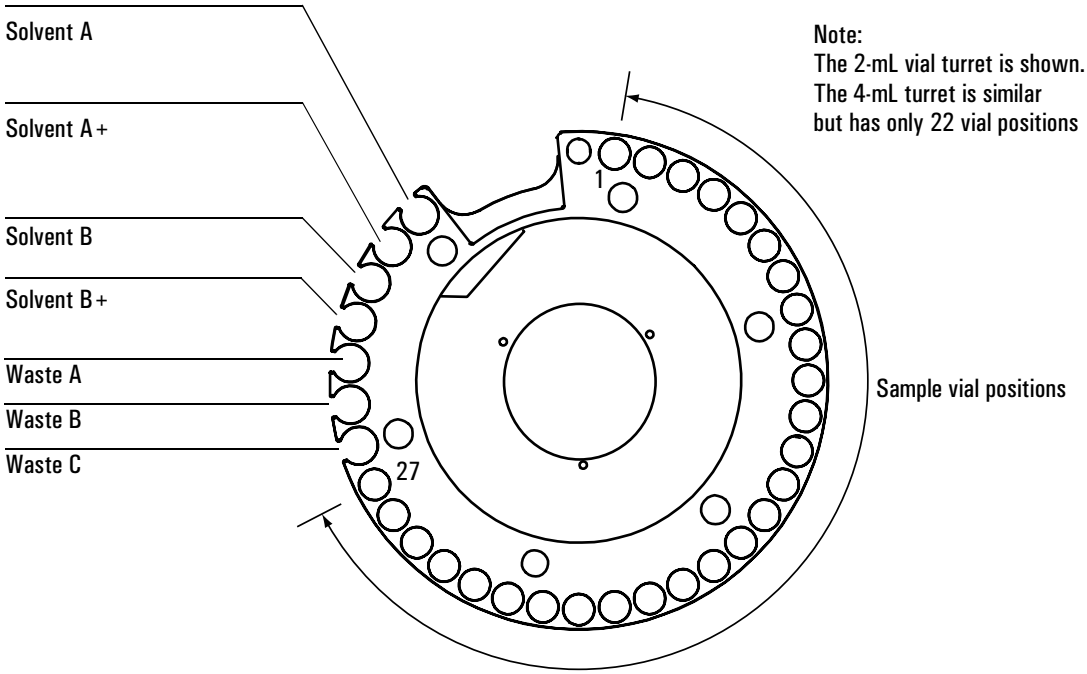



**Figure 47. The 6850 Automatic Liquid Sampler turret (2-mL version shown)**

## Solvent and waste bottle usage

When a syringe is washed (both pre- and post-injection washes), it is filled to 80% of its full volume and then emptied into a waste bottle. Sufficient solvent must be available for the washes and waste bottles must be present to receive the used solvent.

With either turret type, you can use one, two or four solvent bottles for pre- and post-injection rinses. The choice depends on whether you want to use different solvents for the two kinds of wash and on the amount of solvent needed for the samples you intend to run.

| Bottle | Use |
|---|---|
| Solvent A | Can be the only bottle if solvent usage is less than 2 mL. Either Solvent A or Solvent B must be present. |
| Solvent A+ | Additional solvent A when usage exceeds 2 mL. |
| Solvent B | Can be the only bottle if solvent usage is less than 2 mL. Either Solvent A or Solvent B must be present |
| Solvent B+ | Additional solvent B when usage exceeds 2 mL. |
| Waste A | Empty. Receives waste from Solvent A and A+ washes. Required if Solvent A is used. |
| Waste B | Empty. Receives waste from Solvent B and B+ washes. Required if Solvent B is used. |
| Waste C | Empty. Receives waste from sample washes. Always required. |

## How many bottles do I need?

| Bottles | When to use |
|---|---|
| 1 | Your solvent need is less than 2 mL<br>**and**<br>You want to use the same solvent for both pre- and post-injection washes |
| 2 | Your solvent need is between 2 mL and 4 mL<br>**or**<br>You want to use different solvents for the pre- and post-injection washes |
| 4 | Your solvent need exceeds 4 mL |

## What is my solvent need?

See your *6850 GC User Information* CD-ROM for information on how to estimate the number of samples you can run using 2 mL of a solvent. It is in the *Injection Techniques* section.

Good laboratory practice suggests that to reduce the possibility of contamination, only half the solvent in the 4-mL bottle be used. The injector will not access the last 2 mL in the bottle.

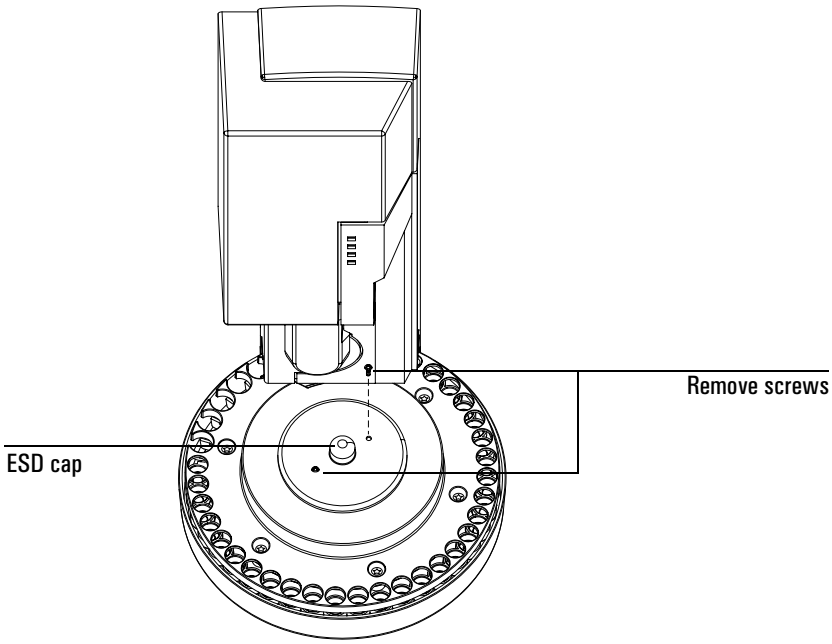
Solvent levels should always be maintained above the "min solvent level" marked on the solvent bottles.

### Filling the turret for use

1. Load all samples to be run into the turret. Make sure that you place them in the turret positions that correspond to the sequence (Control Module or ChemStation/Cerity Chemical control) or Work List (Cerity control). The vial positions are labeled.
2. Load clean, empty waste bottles into the appropriate waste locations. Always load a bottle in the Waste C location.
3. Load your solvent bottles into the solvent locations as needed.

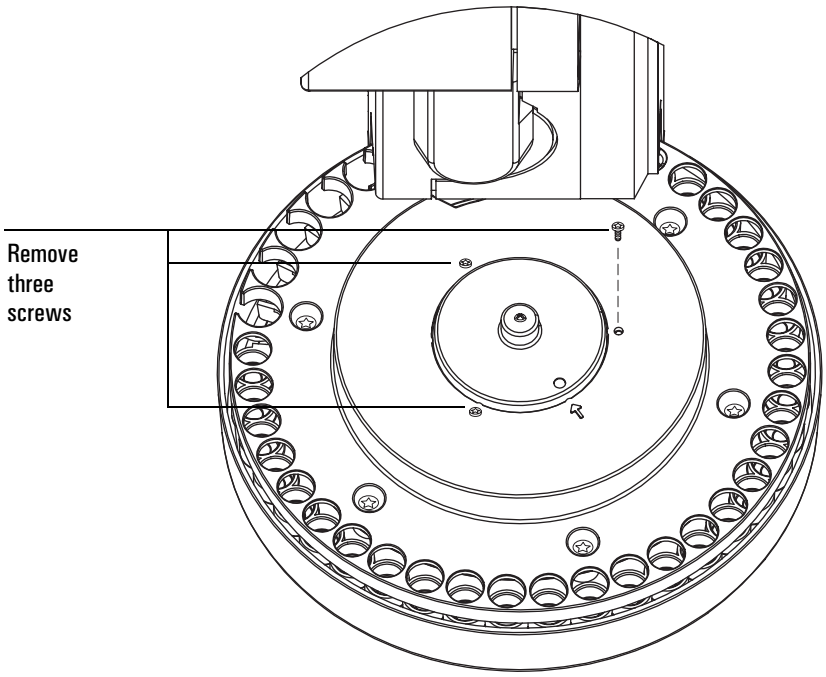
### Removing the turret

1. Remove all vials from the turret.
2. Remove the syringe and the needle support foot.
3. Remove the two plastic screws in the ESD cap, and remove the cap from the motor hub.

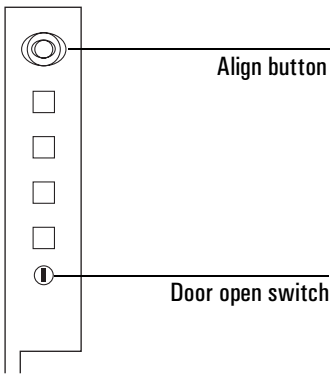



**Caution** Do not loosen the central mounting screw. This screw secures the motor hub in place.

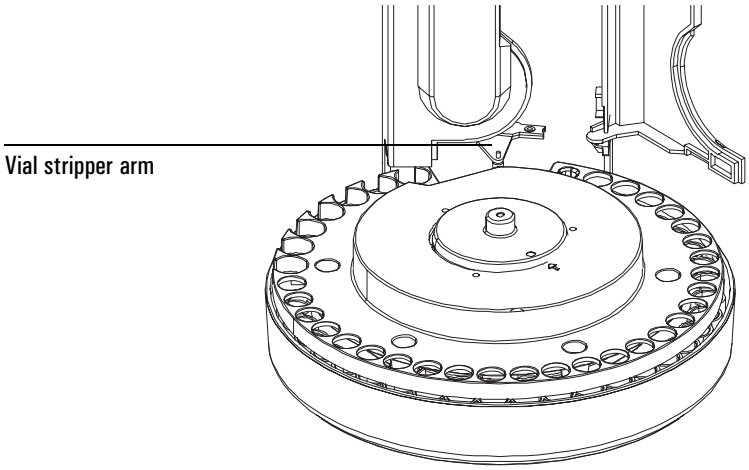
4. Remove the three mounting screws in the turret.




5. With the syringe door open, hold down the door open switch and press the Align button.




The tray will rotate to the position shown here. Note the position of the roller on the vial stripper arm relative to the turret.




6. To remove the turret, lift the turret straight up to free it from the motor hub.
7. Tilt the near edge of the turret up and pull it out slowly up and toward you until it clears the roller on the vial stripper arm. If you move it too far toward you it may get caught on the motor hub.
8. Rotate clockwise to clear the stripper, then simultaneously raise and rotate counterclockwise to clear the injector body. Remove the turret.

   Note that the bottom of the turret can easily get caught on the motor hub. If you have the vials removed, you can look through the empty vial positions and see if the inside edge of the turret is caught on the hub.

### Installing the turret

This is the reverse of the removal process just described.

1. Orient the turret so that the cutout in the turret faces the injector.
2. Place the turret over the hub so that the metal lip on the turret bottom is past the hub, then tilt the far end down. Slip the edge of the turret between the hub and the edge of the injector body. Gently push it inwards and down till it rests on the hub.
3. Rotate the turret on the hub until the arrow marked on the turret aligns with the circular depression on the hub. The turret will now seat flat against the hub.

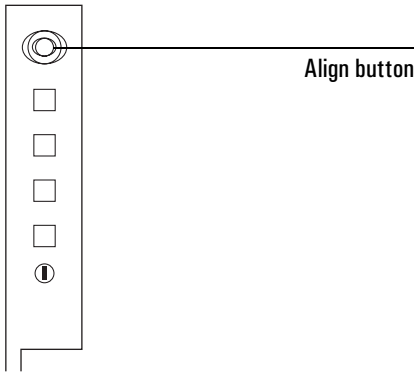
4. Install one mounting screw, then rotate the turret. The outer edge should remain level. Install the remaining screws.
5. Reinstall the ESD cap.
6. Align the turret. See "Align the turret" on page 234.

### Align the turret

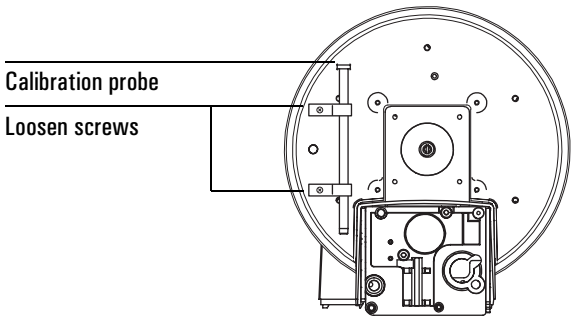
The alignment procedure makes sure that your 6850 ALS is ready for operation using the installed turret.

Always perform an alignment after changing turret types (from the 2-mL vial turret to the 4-mL vial turret, and vice-versa).

1. Remove all vials from the turret. Open the injector door and remove the syringe.
2. Remove the needle support assembly. See "Replacing the needle support assembly" on page 224.
3. Push the align button. The injector and turret will perform a series of checks.



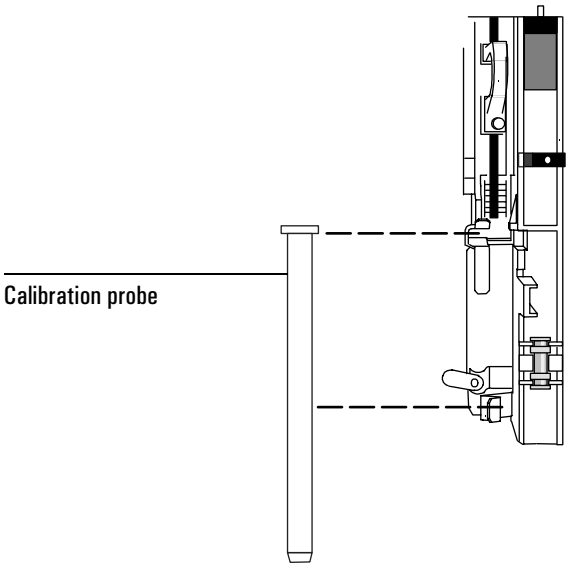

4. Loosen the screws in the clamps that hold the calibration probe to the bottom of the turret and remove the probe. Do not remove the clamps.




5. When the 6850 display reads

   ```
   Install calib probe
   press align button
   ```

   install the calibration probe into the syringe carrier. The probe installs just like a syringe. Press the Align button.




6. When alignment is complete, the green light will be on if the injector door is closed, or the red light will be on if it is open. Remove the calibration probe and secure it in its clamps below the turret.

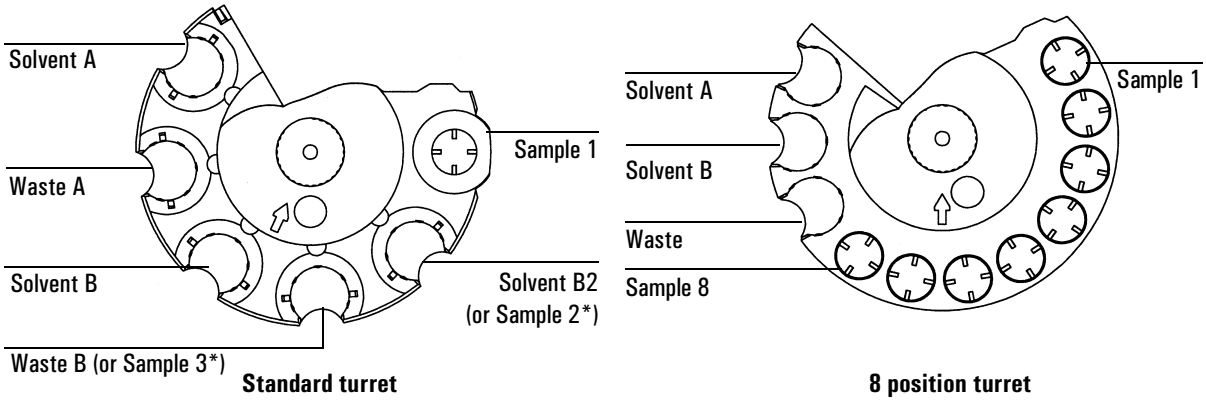
   If the 6850 ALS fails alignment, refer to the section on the injector and automatic sampler in your 6850 GC User Information CD-ROM for details about the indicator lights, likely causes and resolution.

7. Install the needle support assembly.

# The 6850 automatic injector

## Loading the turret

You can place one vial in the standard injector turret, or you can place up to three vials in it by installing optional inserts (part no. 07673-40150). If you use the eight sample position turret, you can place up to eight sample vials in it. See Figure 48.




*Requires optional insert (part no. 07673-40150) to hold sample vial.

**Figure 48. Vial placement in the turret**

## Changing the turret

Your injector comes with two types of turrets. You may use either turret.

If you need to remove or replace your turret, use the following instructions for proper replacement.

1. Disconnect the injector cable from the GC.
2. Open the injector door. Unscrew and remove the knurled nut from the top of the turret. See Figure 49.

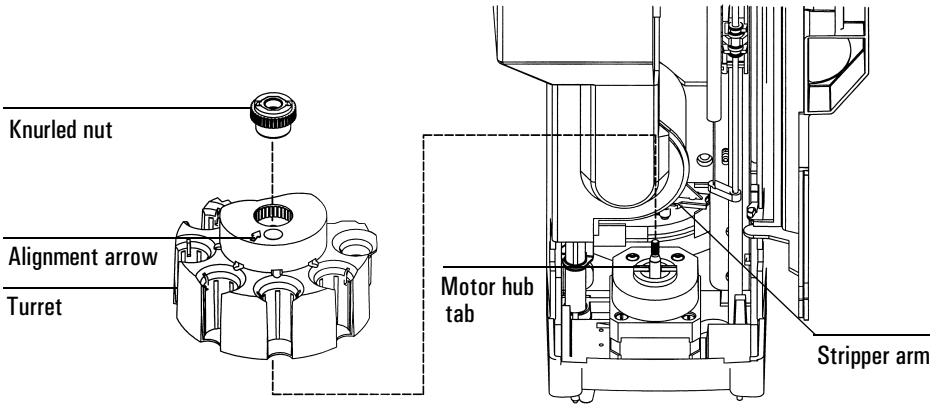



**Figure 49. Turret removal**

3. Rotate the turret so that the open section faces the back of the tower and the arrow on top of the turret points directly back (see Figure 49). Push the stripper arm toward the back, then lift the turret up so that it clears the center shaft.
4. To install the replacement turret, align the tab on the motor hub with the slot in the underside of the turret.
5. Push the stripper arm to the back of the tower, insert the turret with the open section facing the back of the tower and the arrow pointing back, and install the turret with the tab seated in the slot. Seat the turret on the hub, rotating it gently if necessary until it drops completely into position. The top of the threaded shaft should be almost level with the top of the turret.
6. Replace the knurled nut and tighten it firmly (finger-tight).
7. Close the injector door.
8. Plug the injector cable into the GC and check for the Ready light.

For increased accuracy of the needle sampling depth, perform the turret alignment procedure each time the turret is changed. See "Aligning the turret" on page 237.

## Aligning the turret

If you changed turrets in the injector and desire increased needle sampling depth accuracy, or if the Align Mode light is on, perform this alignment procedure. When the Align Mode light is on, the injector will not operate until this procedure has been performed.

1. When the injector is not operating, open the injector tower door.
2. Slide the syringe carriage up until it stops.
3. Remove the syringe. See "Removing a syringe" on page 224.
4. Carefully remove the needle support assembly from the injector. See "Replacing the needle support assembly" on page 224 for details.
5. Use a pen to press the recessed alignment button above the indicator lights, then close the door. See Figure 50.

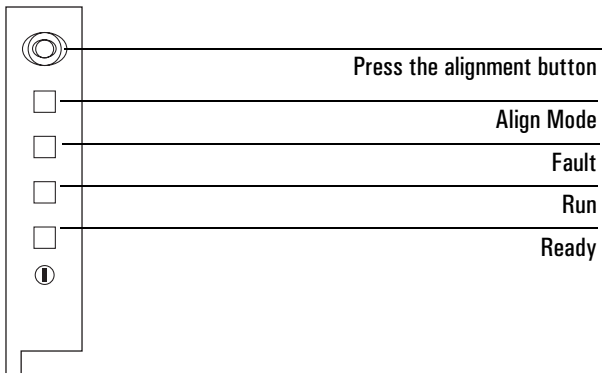



**Figure 50. Aligning the turret**

6. The injector goes through the following steps:
   - The turret rotates to verify that the needle support assembly was removed, then rotates to determine what type of turret is installed
   - The syringe carriage moves all the way down, then moves back up again and clears the turret
   - The syringe carriage steps down until it touches the turret. This sets its position relative to the turret.
   - The plunger moves to calibrate stops
7. When the alignment is complete, the Ready light turns on and the sampler returns to the ready state. Install the needle support assembly and syringe.

   If the injector fails alignment, see "Faults" on page 239.

# Faults

Four lights on the injector indicate its status (Figure 51).

During normal operation, the Ready light is on. If the injector is busy, the Run light is on.

If another combination of lights are on, an error has occurred.

Use the following instructions to try to solve the problem before obtaining Agilent service.

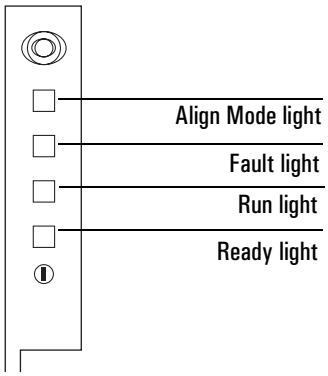



**Figure 51. Injector status lights**

## No light is on

### Probable causes

- The line voltage to the GC is off.
- The injector cable or connection to the GC is bad.
- Your GC requires service.

### Suggested actions

1. Verify the injector is properly connected to the GC.
2. Check the power source for your GC.
3. Obtain Agilent service.

## The Fault light is on

### Probable cause

The injector door is open.

### Suggested actions

1. Ensure that the injector door is closed.
2. If the fault light stays on, obtain Agilent service.

## The Fault and the Run lights are on

### Probable causes

- The injector is mounted incorrectly on the mounting post
- Incorrect mounting post

### Suggested actions

1. Ensure that the injector is mounted properly. For more information, see "Installing a sampler or injector on the 6850 GC" on page 242.
2. Ensure the correct mounting post is installed.
3. If the fault lights stay on, obtain Agilent service.

## Align Mode light is on

### Probable causes

- The turret is not properly installed
- The type of turret was changed while the power was on
- The system was not initialized
- There is an injector memory error

### Suggested action

1. Verify the turret is properly installed. See "Changing the turret" on page 236.
2. Perform the alignment procedure to initialize the system. See "Aligning the turret" on page 237.

## Align Mode light is on, all others are flashing (6850 Automatic Liquid Sampler only)

### Probable cause

Turret alignment is in progress.

### Suggested action

Install the calibration probe and press the Align button to complete the alignment.

## All lights are on

### Probable cause

- The cables connections are loose
- There is a board failure
- There is a firmware revision conflict

### Suggested action

1. Check all cable connections.
2. Turn the instrument off, then on again.
3. If the lights remain on, obtain Agilent service.

## Installing a sampler or injector on the 6850 GC

Use Figure 52 to install the mounting post and the injector.

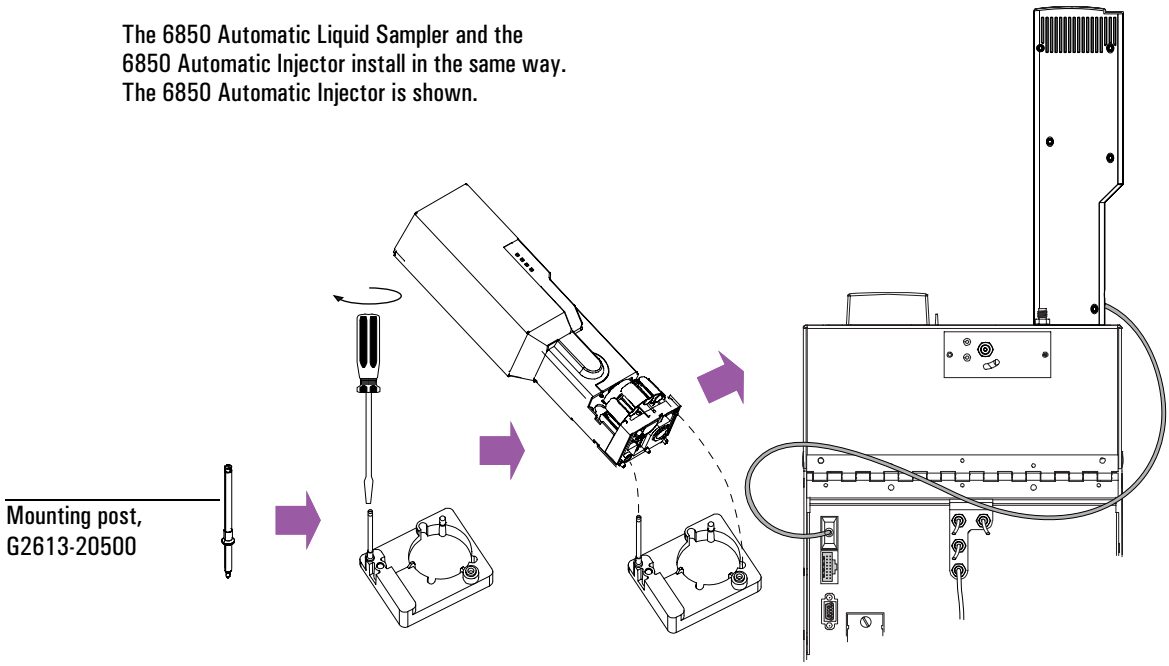



**Figure 52. Installing the mounting post and mounting the injector**

## Installing the injector mounting bracket and post

Use Figure 53 to install the injector mounting bracket.

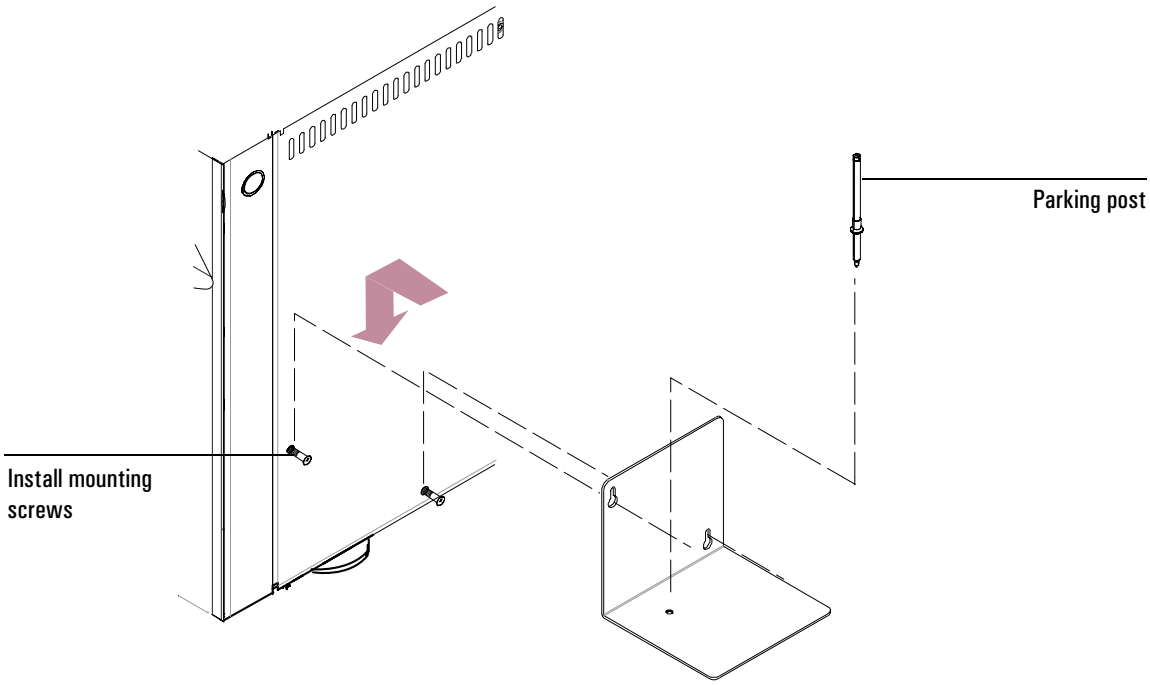



**Figure 53. Installing the injector mounting bracket on the side of the GC**

# Installation

## Installation checklist

The table below summarizes the installation steps for your 6850 GC. See also your Agilent 6850 installation poster.

| Step | | |
|---|---|---|
| 1. | Remove protective caps from TCD and vents. | ❒ |
| 2. | Connect all cables. | ❒ |
| 3. | Remove plugs and install column (label to front). | ❒ |
| 4. | If available, install cryo cooling coil. Bend bracket to secure coil. | ❒ |
| 5. | If available, install your sampler and cable. | ❒ |
| 6. | Remove caps and connect your gases. If valve installed, connect sample lines. | ❒ |
| 7. | Set gas source pressures and check for leaks. | ❒ |
| 8. | Turn on the GC. If available, connect cryo filter and coolant.* | ❒ |
| 9. | Load CHECKOUT method and wait until “Ready...” | ❒ |
| 10. | For manual injection, prepare the checkout sample and start. | ❒ |
| 11. | If available, prepare checkout sample and start. | ❒ |
| 12. | Verify chromatographic peaks. | ❒ |
| 13. | If available, plug in the control module. | ❒ |

* See “Prepare the cryogenic oven cooling system” on page 247 for more details.

## Setting your IP address

Each of the three LAN cards that could be installed in your 6850 GC provide different methods of obtaining the GC's IP address. Table 38 lists the types of LAN cards and their available control modes.

**Table 38. Available Control Modes**

| LAN card | Control modes | GC firmware |
|---|---|---|
| J2552B | BootP[a] | A.03.XX or greater |
| J4100A[b] | BootP[a], GC front panel | A.03.XX or greater |
| Lantronics | DHCP, GC front panel, Control Module | A.03.XX or greater |

a Refer to your Cerity Chemical or ChemStation software CD for a copy of BootP server software
b Local addressing requires LAN card firmware revision K.08.04 or greater

### To determine which LAN card is installed

If you are not sure which LAN card is installed in your GC:

1. Turn the GC off.
2. Press and hold `LOAD` and turn the GC on. Continue to hold `LOAD` until five dots appear in the display.
3. When the GC finishes initializing, you will see one of the following:

```
DHCP MODE:
DISABLED
```

**Lantronics card installed**

or

```
IP ADDRESS
XXX.XX.XX.XX
```

**Card J4100A installed**

If the GC does not display either of these two screens, your GC uses card J2552B or card J4100A and firmware that does not support local IP addressing. If this is the case, BootP must be used to obtain the IP address.

### To set the IP address locally

**Caution** Be sure the IP address you enter is correct and is not in use by another device (such as a printer).

1. Turn the GC off.
2. Press and hold `LOAD` and turn the GC on. Continue to hold `LOAD` until five dots appear in the display.

3. When the GC finishes initializing, you will see one of the following:

| | | |
|---|---|---|
| `DHCP MODE:`<br>`DISABLED` | or | `IP ADDRESS`<br>`XXX.XX.XX.XX` |
| Lantronics card installed | | Card J4100A installed |

   If the `DHCP MODE` is displayed, be sure it is set to `DISABLED`. If not, change the mode to `DISABLED` by pressing ▲ or ▼. Press `LOAD` to continue to the `IP ADDRESS`.

4. The display will now read:

```
IP ADDRESS
XXX.XX.XX.XX
```

5. Press `LOAD` to adjust the `IP ADDRESS` values. Press ▲ or ▼ to change values and `LOAD` to move from one value to the next.

6. When `IP ADDRESS` is completed, the display reads:

```
DEFAULT GATEWAY
XXX.XXX.XXX.XXX
```

   Change the `DEFAULT GATEWAY` as you did the `IP ADDRESS`.

7. Change the `SUBNET MASK` value in the same manner.

8. Cycle the GC power for new settings to take effect.

### To use DHCP to set the IP address

1. Turn the GC off.

2. Press and hold `LOAD` and turn the GC on. Continue to hold `LOAD` until five dots appear in the display.

3. When the GC starts, you will see:

```
DHCP MODE:
DISABLED
```

4. Press ▲ or ▼ to change to `ENABLED`. Press `LOAD` again. The display will now show the GC's `MAC ADDRESS`. The `MAC ADDRESS` cannot be altered. Record the `MAC ADDRESS` if needed.

5. Cycle the power to activate `DHCP MODE`.

## Safely route vent tubing

The FPD provides a plastic vent tube that exits the back of the detector cover. During normal operation, this detector produces water vapor. Vent the tubing appropriately.

The µECD provides a vent tube that exits the back of the lid. Connect this tube to a fume hood or other location as directed by your radiation safety officer or environmental health and safety organization. See "µECD warnings" on page 7 for more information.

## Prepare the cryogenic oven cooling system

**Warning** Pressurized liquid $CO_2$ is a hazardous material. Take precautions to protect personnel from high pressures and low temperatures. $CO_2$ in high concentrations is toxic to humans; take precautions to prevent hazardous concentrations. Consult your local supplier for recommended safety precautions and delivery system design.

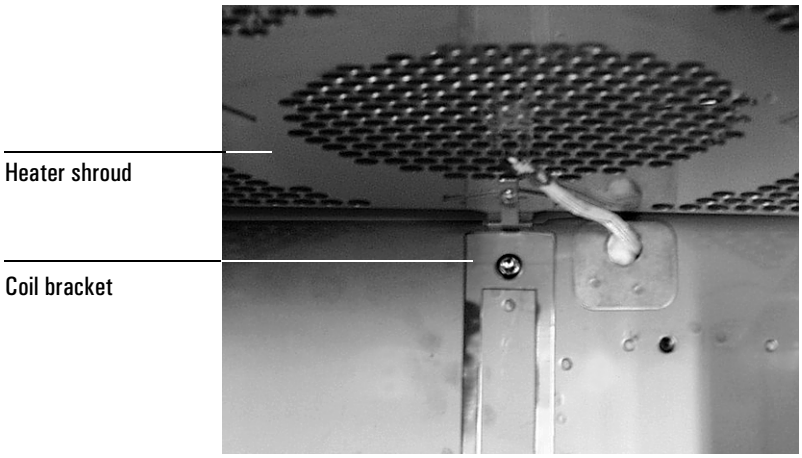
**Warning** Do not use copper tubing or thin-wall stainless steel tubing with liquid $CO_2$. Both harden at stress points and may explode.

**Caution** Liquid $CO_2$ should not be used as a coolant for temperatures below –40°C because the expanding liquid may form solid $CO_2$—dry ice—in the GC oven. If dry ice builds up in the oven, it can seriously damage the GC.

If you ordered $CO_2$ cryogenic oven cooling with the GC, the cooling coil for the oven and the filter on the cryogenic chassis are shipped uninstalled because of the possibility of damage if shipped installed.

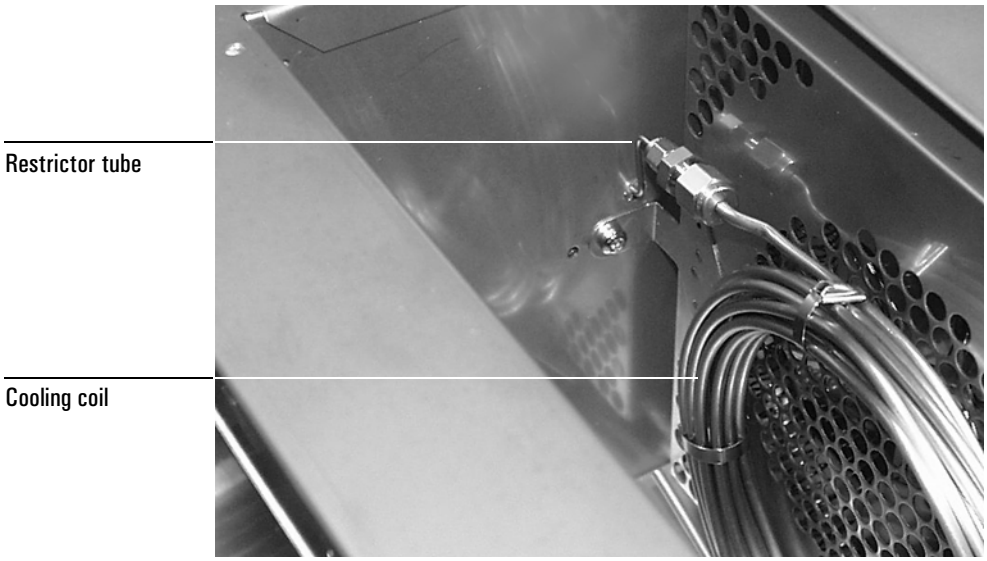
## Install the cooling coil

1. Inside the oven, locate the coil bracket at the bottom center of the oven. It is secured by the same screw that holds the bottom of the heater shroud.




2. Place the cooling coil in the oven with the fitting at the upper left. The bottom center of the coil should rest on the coil bracket.

.




3. Remove the stainless steel plug from the fitting on the end of the restrictor tube. Leave the rest of the fitting in place.
4. Connect the fittings on the restrictor tube and the coil. Tighten the 1/16-inch nut finger tight.

5. Press the coil against the oven floor and bend the three bracket legs around it.


6. Check that:
   - The restrictor tube enters the oven and turns upward
   - The restrictor tube is connected to the coil and the fittings are finger tight
   - The coil is near to, but makes minimal contact with, the heater shroud
7. Tighten the nut on the restrictor tube 1/2 turn past finger tight. Use two wrenches.

## Install the coolant filter

Some GC owners have had problems with particles in the $CO_2$ supply that clog the cryo valve. The filter and connector prevent this.

1. Locate the arrows (flow direction) on the body of the filter. Add the connector to the exit end of the filter. Assemble the parts as shown. Tighten the nut.

2. Locate the cryo chassis on the left side of the GC.

   The cryo chassis contains one or two fittings for coolant, depending upon the cryo options purchased. See Figure 54.

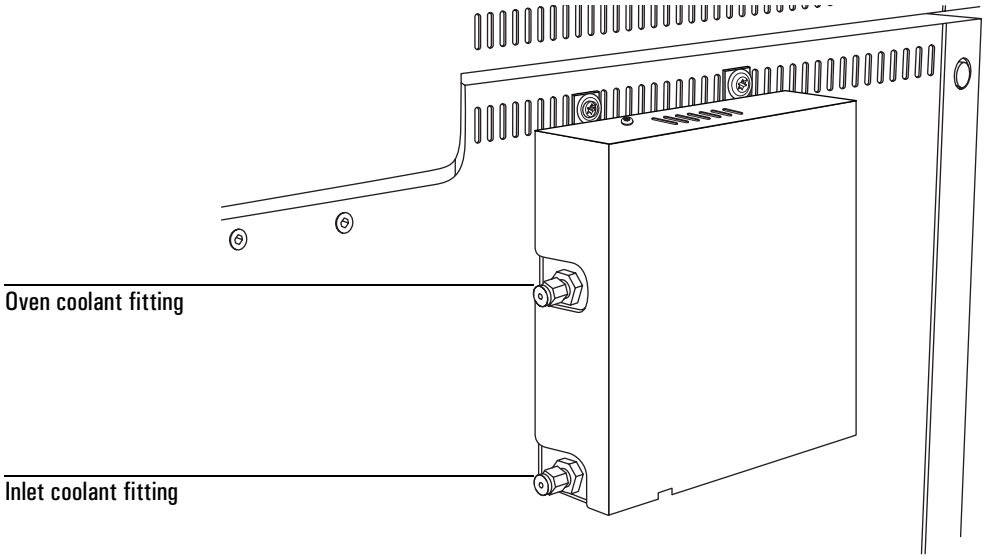



**Figure 54. 6850 Cryo coolant fittings**

3. If using inlet and oven cooling, install a “Tee” (part 0100-0542) at the coolant fittings as shown in Figure 55. Agilent recommends that you use spectral-link tubing (part SL-8).

**Warning** The high pressure $LCO_2$ requires thick-walled, high pressure stainless steel tubing to avoid ruptures.

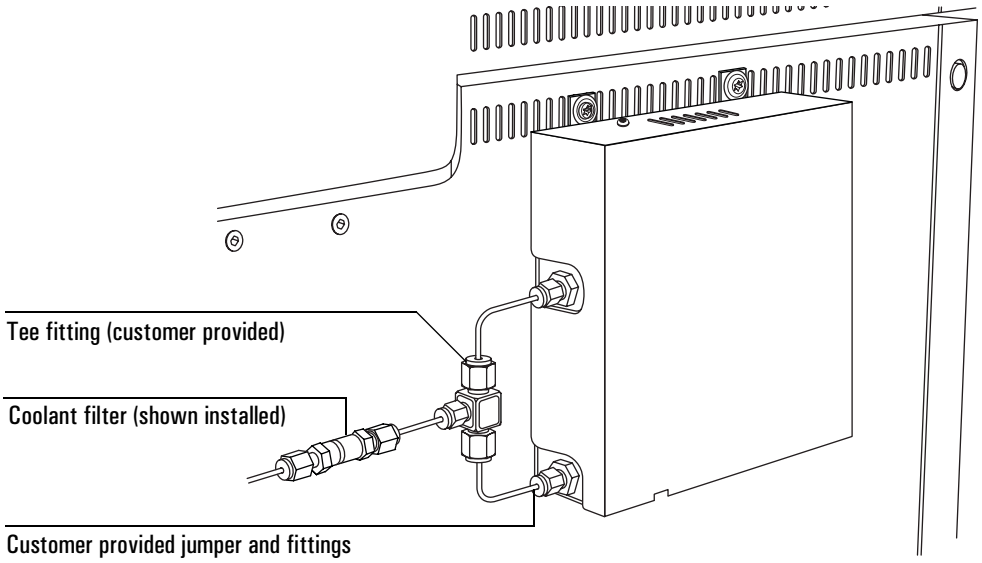


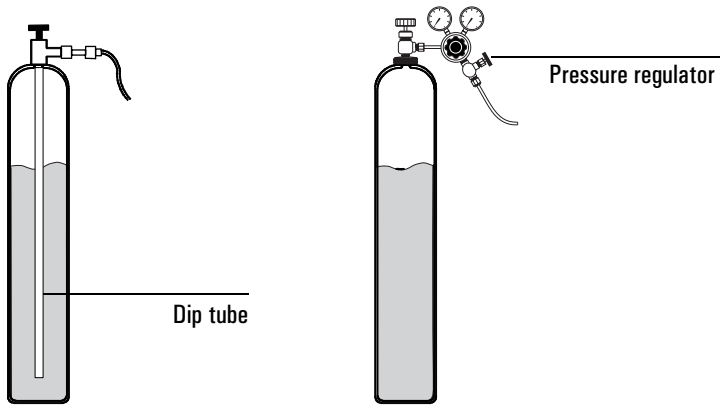

**Figure 55. Cryo coolant fittings**

4. Remove the nut and ferrules from the coolant inlet fitting. Use these parts to attach he filter/connector assembly to the inlet fitting or Tee fitting. See Figure 55.

## Connect the cryogenic coolant supply

Liquid $CO_2$ is available in high-pressure tanks containing 50 pounds of liquid. The tank must have an internal dip tube or eductor tube to deliver liquid $CO_2$ instead of gas. Do not use a padded tank (one to which another gas is added to increase the delivery pressure).

**Caution** Do not install a pressure regulator on the $CO_2$ tank, as vaporization and cooling would occur in the regulator instead of the oven.




**Warning** All fittings and tubing should be stainless steel. The pressure inside a liquid $CO_2$ tank can be as high as 1000 psi.

1. Connect a CGA 320 to 1/4-inch male NPT fitting to the tank followed by a Swagelok part no. SS-200-7-4 1/4-inch female NPT to 1/8-inch tube fitting.
2. Connect the tank to the cryogenic filter inlet with 1/8-inch diameter heavy-wall stainless steel tubing. The tubing may be up to 50 feet long.

   Allow some slack in the line to make movement of the tank or GC easier. Coil and fasten the ends of the tubing to keep it from “whipping” if it breaks.

## Configuration

In order to properly control instrument gas flows and temperatures, and to provide proper functionality for valves, the instrument maintains a list of manually-configured settings. These include carrier and detector gas types, valve types (if installed), and the time and date. Agilent sets these parameters at the factory. In general, if using the same gases as used for the detector checkout method, no further configuration is needed.

Additional configuration steps may be required whenever:

- Changing carrier or detector makeup gases
- After installing new hardware
- After modifying hardware
- Setting the GC time and date

To re-configure the GC, use a control module. Refer to the *6850 Series Control Module User Information* manual included on the 6850 Series II User Information CD-ROM.

# Site Prep Summary

## Summary of GC benchtop and lab requirements

### Table 39. Physical Dimensions

| Height | Width | Depth | Weight |
|---|---|---|---|
| 50 cm (19.7-inch)<br>61 cm (23-inch), FPD | 29 cm (11.4 inch), standard<br>34 cm (13.4 inch), cryo cooling<br>37 cm (14.6 inch), 6850 ALS | 54 cm (21.3-inch)<br>64 cm (25.2-inch), auxiliary EPC | < 23 kg (51 lb.) |

### Table 40. Power Requirements and Heat Output

| Line voltage (±10%) | Current | Maximum power consumption | Line frequency | Heat output |
|---|---|---|---|---|
| **Regular oven** | | | | |
| 100 V* (Japan) | 15 A | 1440 W | 47.5 – 63 Hz | ≤ 4800 BTU/hour |
| 120 V* (USA) | 15 A | 1440 W | 47.5 – 63 Hz | ≤ 4800 BTU/hour |
| 230 V* (Europe) | 12 A maximum<br>8 A minimum | 2000 W | 47.5 – 63 Hz | ≤ 4800 BTU/hour |
| **Fast-heating oven** | | | | |
| 120 V* (USA) | 20 A | 2400 W | 47.5 – 63 Hz | ≤ 4800 BTU/hour |
| 200 V* (Japan) | 12 A | 2400 W | 47.5 – 63 Hz | ≤ 4800 BTU/hour |
| 230 V* (Europe) | 11 A | 2400 W | 47.5 – 63 Hz | ≤ 4800 BTU/hour |

*Requires an isolated ground and dedicated outlet.

### Table 41. Temperature and Humidity Ranges

| | Temperature range | Humidity range | Altitude range |
|---|---|---|---|
| Operating | 15–35°C | 5–95% | |
| Maximum | 5–40°C | 5–95% | ≤ 2000 m |

## Table 42. Gas Recommendations for Capillary Columns

| Detector | Carrier gas | Preferred makeup gas | Second choice/ comments | Detector, anode purge, or reference gas |
|---|---|---|---|---|
| Microcell electron capture | Hydrogen | Argon/Methane | Nitrogen | Anode purge must be the same as makeup |
| | Helium | Argon/Methane | Nitrogen | |
| | Nitrogen | Nitrogen | Argon/Methane | |
| | Argon/ Methane | Argon/Methane | Nitrogen | |
| Flame ionization | Hydrogen | Nitrogen | Helium | Must use hydrogen and air as detector gases |
| Flame photometric | Hydrogen | Nitrogen | | Hydrogen and air for detector |
| | Helium | Nitrogen | | |
| | Nitrogen | Nitrogen | | |
| | Argon | Nitrogen | | |
| Thermal conductivity | Hydrogen | Must be same as carrier and reference gas | Must be same as carrier and reference gas | Reference gas must be same as carrier and makeup |
| | Helium | | | |
| | Nitrogen | | | |

## Table 43. Gas Recommendations for Packed Columns

| Detector | Carrier gas | Comments about carrier gas | Detector, or reference gas |
|---|---|---|---|
| Microcell electron capture | Nitrogen | Maximum sensitivity | Nitrogen |
| | Argon/ Methane | Maximum dynamic range | Argon/Methane |
| Flame ionization | Nitrogen | Maximum sensitivity | Nitrogen |
| | Helium | maximum dynamic range | |
| Flame photometric | Hydrogen | | Hydrogen and air for detector |
| | Helium | | |
| | Nitrogen | | |
| | Argon | | |
| Thermal conductivity | Helium | General use | Reference gas must be same as carrier gas |
| | Hydrogen | Maximum sensitivity[1] | |
| | Nitrogen | Hydrogen detection[2] | |
| | Argon | Maximum hydrogen sensitivity[2] | |

[1] Slightly greater sensitivity then helium. Incompatible with some compounds.

[2] For analysis of hydrogen or helium. Greatly reduces sensitivity for other compounds.

## Table 44. Gas Purity Recommendations

| Gas | Purity when used as carrier or capillary makeup gas | Purity when used as detector support gas |
|---|---|---|
| Helium | 99.9995% | 99.9995% |
| Nitrogen | 99.9995% | 99.9995% |
| Hydrogen | 99.9995% | 99.9995% |
| Argon/Methane | 99.9995% | 99.9995% |
| Air (dry) | (Not used) | Zero-grade or better |

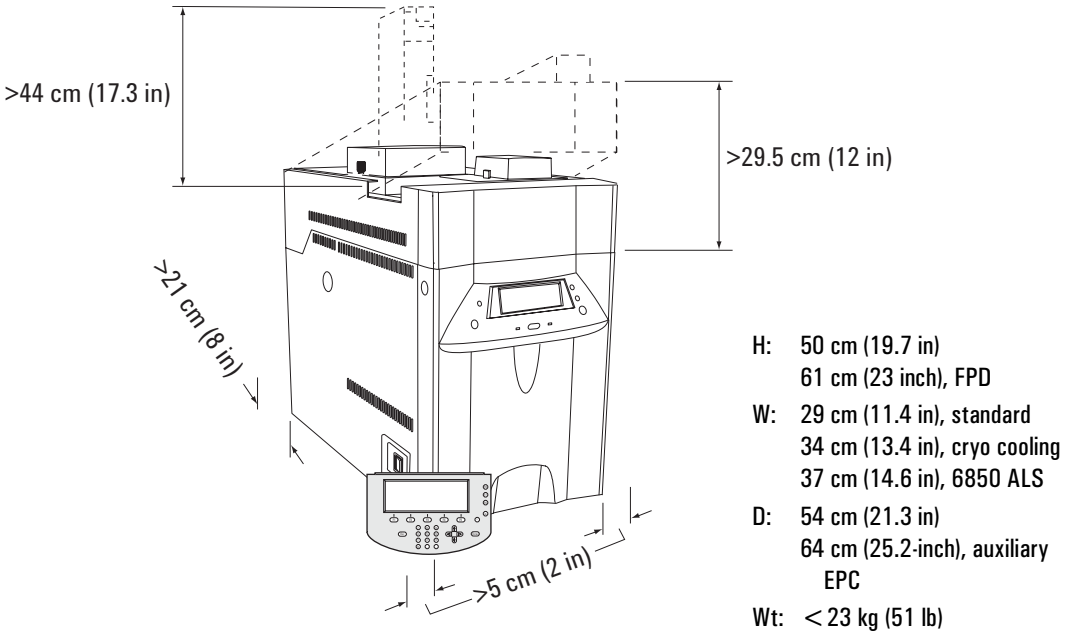



**Figure 56. 6850 GC bench space requirements**