

# **Clarus 600 GC Hardware Guide**

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## Release History

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Any comments about the documentation for this product should be addressed to:

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# Introduction **1**

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*Introduction*

## ***About This Manual***

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This hardware guide is divided into following chapters:

### ***Chapter 1 Introduction***

This chapter contains a brief introduction on the instrument, the conventions and warnings used in the manual.

### ***Chapter 2 Safety Practices***

Important safety information for the Clarus GC is provided in this chapter.

### ***Chapter 3 System Description***

This chapter contains information on the components of the instrument, how it works and instrument specifications.

### ***Chapter 4 Before You Install a Column***

This chapter contains general information regarding column installation and the basic procedures you should know in order to install a column.

### ***Chapter 5 Installing a Packed Column***

This chapter contains procedures for connecting a packed column to the packed column injector and setting the carrier gas flow using PPC and/or manual pneumatics.

### ***Chapter 6 Installing a Capillary Column***

This chapter contains procedures for connecting a capillary column to a Capillary Injector (CAP); a Programmed Split/Splitless Capillary Injector (PSS); and a Programmed On-Column Capillary Injector (POC). It also describes how to set the gas flows using PPC and/or manual pneumatics.

## *Introduction*

### ***Chapter 7 PreVent***

PreVent™ is an enhanced capillary inlet system for the Clarus GC that uses columns with an inside diameter (i.d.) between 0.25 mm and 0.53 mm.

### ***Chapter 8 PPC Fundamentals***

This chapter describes the fundamentals of Programmable Pneumatic Control (PPC™) and how to use PPC to control your injectors.

### ***Chapter 9 Maintenance***

Maintenance and cleaning procedures for the various components of your instrument are provided.

### ***Chapter 10 Troubleshooting***

Performance checks, troubleshooting information, software and system error messages along with practical hints for running for the instrument are provided.

### ***Appendix I: U.S. Nuclear Regulations***

Regulations from the Nuclear Regulatory Commission.

### ***Appendix II: Ionization Potential***

Ionization potentials for representative compounds.



## ***Conventions Used in this Manual***

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Normal text is used to provide information and instructions.

**Bold** text refers to button or tab page that is displayed on the touch screen.

All eight digit numbers are PerkinElmer part numbers unless stated otherwise.

### ***Notes, cautions and warnings***

Three terms, in the following standard formats, are also used to highlight special circumstances and warnings.

**NOTE:** *A note indicates additional, significant information that is provided with some procedures.*

#### **CAUTION**

*We use the term CAUTION to inform you about situations that could result in **serious damage to the instrument** or other equipment. Details about these circumstances are in a box like this one.*



#### **WARNING**

*We use the term WARNING to inform you about situations that could result in **personal injury** to yourself or other persons. Details about these circumstances are in a box like this one.*

## ***Customer Service***

PerkinElmer, Inc.  
710 Bridgeport Avenue  
Shelton, CT 06484-4794 U.S.A

Tel: 1 (800) 762-4000 or (203) 762-4000  
Internet: <http://www.perkinelmer.com>

## ***Electromagnetic Compatibility (EMC)***

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### ***United States (FCC)***

This product is classified as a digital device used exclusively as industrial, commercial, or medical test equipment. It is exempt from the technical standards specified in Part 15 of the FCC Rules and Regulations, based on Section 15.103 (c).

### ***European Union***

All information concerning EMC standards will be in the Declaration of Conformity and these standards will change as the European Union adds new requirements.

#### ***European Union Industrial Environment***

The 220/230/240 V/50 Hz. Clarus GC has been manufactured for use in the European Union and is intended for the industrial environment. The instrument is to be connected to a mains power network supplied from a high or medium-voltage transformer dedicated for the supply of an installation feeding a manufacturing or similar plant.

Industrial environments are characterized by the existence of one or more of the following conditions:

- industrial, scientific and medical (ISM) apparatus are present
- heavy inductive or capacitive loads are frequently switched
- currents and associated magnetic fields are high

These are the major contributors to the industrial electromagnetic environment and as such distinguish the industrial from other environments. The instrument is not intended for connection to a public mains network supplying residential, commercial and light-industrial locations.

### ***Susceptibility to RF Interference***

With the exception of the Flame Ionization Detector (FID), an RF field strength of 10 V/m between 80 MHz. and 1000 MHz. with 80% modulation at 1 kHz. may cause a deflection on the chromatographic detector baseline that exceeds its

normal pattern. This implies that if a transmitting device, such as a walkie-talkie carried by a security guard, is use near the detector, a spike or peak on the chromatographic baseline may occur. If you are concerned that such an event may occur, PerkinElmer recommends that walkie-talkie restriction notices be posted in the vicinity. Cell phones, beepers, and other similar devices operate in a much higher frequency range and do not cause interference.

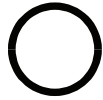
## **Warning Signs on the Instrument**



*Alternating current.*



*Protective conductor terminal.*



*Off position of the main power source.*



*On position of the main power source.*



*Warning, hot surface*

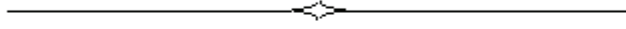


*Warning, risk of electric shock.*



*Warning, Risk of Danger.  
Consult the manual to determine the nature of the potential hazard and any actions which have to be taken. Also, consult the manual for allowable gases and maximum permitted pressures.*

*Introduction*



# **Safety Practices 2**

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*Safety Practices*



## **Chapter Overview**

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This chapter describes the general safety practices and precautions that must be observed when operating the Clarus 600 GC.

This advice is intended to supplement, not supersede, the normal safety codes in the user's country. It is also a supplement to the PerkinElmer standard Safety and Health Policy. The information provided does not cover every safety procedure that should be practiced. Ultimately, maintenance of a safe laboratory environment is the responsibility of the analyst and the analyst's organization.

Please consult all manuals supplied with the Clarus GC and accessories before you start working with the instrument. Carefully read the safety information in this chapter and in the other manuals supplied. When setting up the instrument or performing analyses or maintenance procedures, strictly follow the instructions provided. The Clarus GC should be used in accordance with the instructions provided in this manual. If used otherwise, the protection provided by the instrument may be impaired.

### **Generic Warnings**

Before installing or operating the Clarus GC, read the following information concerning hazards and potential hazards. You should ensure that anyone involved with installation and/or operation of the Clarus GC is knowledgeable in both general safety practices for the laboratory and safety practices for the Clarus GC. Get advice from your safety engineer, industrial hygienist, environmental engineer, or safety manager before you install or use this instrument.

### **Heated Zones**

Heated zones should be treated with caution, for example, injector caps and detectors. Avoid physical contact with the injector caps. The detector cover may get hot, especially if flame ionization detectors are operated at high temperatures. As a general rule, allow heated zones to cool before attempting to work in the oven, injector, or detector areas.

**CAUTION**

***THERMAL RUNAWAY PROTECTION:*** *The Clarus GC software shuts down the instrument if any heated zone exceeds 470 °C. Should this occur, the following error message is displayed:*

**INSTRUMENT SHUTDOWN**

*xxx THERM RUNAWAY where xxx is the heated zone*

*Call your PerkinElmer Representative.*

*Instrument shutdown also occurs if there is a PRT (Platinum Resistance Thermometer) or MPU (Micro Processor Unit) failure. In these cases the following error message is displayed:*

**INSTRUMENT SHUTDOWN**

*xxx PRT ERROR where xxx is the failed zone.*

*Call your PerkinElmer Representative.*

## Precautions

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*Be sure that all instrument operators read and understand the precautions listed below. It is advisable to post a copy of the precautions near or on the instrument shelf.*

**CAUTION**

*The protection provided by this equipment may be impaired if the equipment is used in a manner not specified by PerkinElmer.*

The following precautions must be observed when using the Clarus GC:

- This equipment requires no specified inspection or preventive maintenance to ensure the continuous functioning of its safety features.
- Be sure that the power line voltage of the Clarus GC corresponds to the voltage used in your laboratory.
- Never remove the side panels of the Clarus GC without shutting down the instrument and disconnecting the instrument power cord from line power.
- Do not immerse the purge gas exit line in a liquid, as the liquid may be drawn back into the sample holder.
- Only high quality purge gases should be used with the Clarus GC. Minimum purity of 99.995% is recommended. A high quality filter-dryer accessory is recommended for the removal of any moisture from the purge gases.

## **Environmental Conditions**

### **Operating Conditions**

**CAUTION**      *The Clarus GC is designed for indoor use only.*

**CAUTION**      *Do not operate in a Cold Room or a refrigerated area. The Clarus GC operates most efficiently under the following conditions:*

- *Ambient temperature is 10 °C to 35 °C (50 °F to 95 °F). The GC will operate safely between 5 °C and 40 °C (41 °F and 104 °F).*
- *Ambient relative humidity is 20% to 80% non-condensing.*
- *Operating altitude is in the range of 0 to 2 000 m.*



**WARNING**

*The Clarus GC is not designed for operation in an explosive environment.*

### **Pollution Degree**

The Clarus GC will operate safely in environments that contain nonconductive foreign matter up to Pollution Degree 2 in IEC 1010-1.

### **Cleaning the Instrument**

Exterior surfaces may be cleaned with a soft cloth, dampened with a mild detergent and water solution. Do not use abrasive cleaners or solvents.

### **Cleaning and Decontamination**

Before using any cleaning or decontamination methods except those specified by PerkinElmer, users should check with PerkinElmer that the proposed method will not damage the equipment.”

### **Clarus GC Touch Screen**

For optimum performance, the Clarus GC’s touch screen may require periodic re-calibration. The interval between re-calibration may be affected by exposure to combined heat and humidity conditions (ambient conditions between 30 °C / 50% RH and 35 °C / 80% RH).

### **Storage Conditions**

The Clarus GC may be stored under the following conditions:

- ambient temperature is -20 °C to +60 °C (-4 to 140 °F)
- ambient relative humidity is 20 to 80%, non-condensing
- altitude is in the range 0 to 12 000 m.

### **General Laboratory Safety**

Your laboratory should have all equipment ordinarily required for the safety of individuals working with chemicals (fire extinguishers, first-aid equipment, safety shower and eye-wash fountain, spill cleanup equipment, etc.).

## ***Electrical Safety***

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The Clarus GC contains high voltage. To prevent the risk of shock, unplug the line cord from the AC outlet and wait at least one minute before opening or removing any instrument panels.

The instrument has been designed to protect the operator from potential electrical hazards. This section describes some recommended electrical safety practices.

**CAUTION**

*This unit contains protective circuitry. Contact PerkinElmer Service before performing any AC line tests.*



**WARNING**

**Safety Interlocks**

*PerkinElmer provides a number of safety interlocks on its instruments that monitor such things as doors, gas pressures, temperatures of various components. Do not attempt to defeat these interlocks; you may compromise your own or someone else's safety.*



**WARNING**

*Connect the GC to an AC line power outlet that has a protective ground connection. To ensure satisfactory and safe operation of the GC, it is essential that the protective ground conductor (the green/yellow lead) of the line power cord is connected to a true electrical ground. Any interruption of the protective ground conductor, inside or outside the GC, or disconnection of the protective ground terminal may impair the protection provided by the GC.*



**WARNING**

*Do not operate the GC with any covers or parts removed.*



**WARNING**

*To avoid electrical shock, disconnect the power cord from the AC outlet before servicing. Servicing on the GC is to be performed only by a PerkinElmer service representative or similarly trained and authorized person.*



**WARNING**

*Do not attempt to make adjustments, replacements or repairs to this GC except as described in the user documentation.*

**CAUTION**

*To ensure adequate cooling of the instrument electronics, do not obstruct the gap at the base of the GC, and leave at least a 6-inch clearance between instruments.*

Ensure that the power cord is correctly wired and that the ground leads of all electrical units (for example, recorders, integrators) are connected together via the circuit ground to earth. Use only three-prong outlets with common earth ground connections.

Servicing of incoming AC line components in your laboratory should be performed only by a licensed electrician.



**WARNING**

*Lethal voltages are present at certain areas within the instrument. Installation and internal maintenance of the instrument should only be performed by a PerkinElmer service engineer or similarly authorized and trained person. When the instrument is connected to line power, opening the instrument covers is likely to expose live parts. Even when the power switch is off, high voltages can still be present. Capacitors inside the instrument may still be charged even if the instrument has been disconnected from all voltage sources.*

The instrument must be correctly connected to a suitable electrical supply. The supply must have a correctly installed protective conductor (earth ground) and must be installed or checked by a qualified electrician before connecting the instrument.



**WARNING**

*Any interruption of the protective conductor (earth ground) inside or outside the instrument or disconnection of the protective conductor terminal is likely to make the instrument dangerous. Intentional interruption is prohibited.*

When working with the instrument:

- Disconnect the instrument from all voltage sources before opening it for any adjustment, replacement, maintenance, or repair. If afterwards, the opened instrument must be operated for further adjustment, maintenance, or repair, this must only be done by a PerkinElmer Service engineer.
- Whenever it is possible that the instrument is no longer electrically safe for use, make the instrument inoperative and secure it against any unauthorized or unintentional operation. The electrical safety of the instrument is likely to be impaired if, for example, the instrument shows visible damage, has been subjected to prolonged storage under unfavorable conditions, or has been subjected to severe stress during transportation.

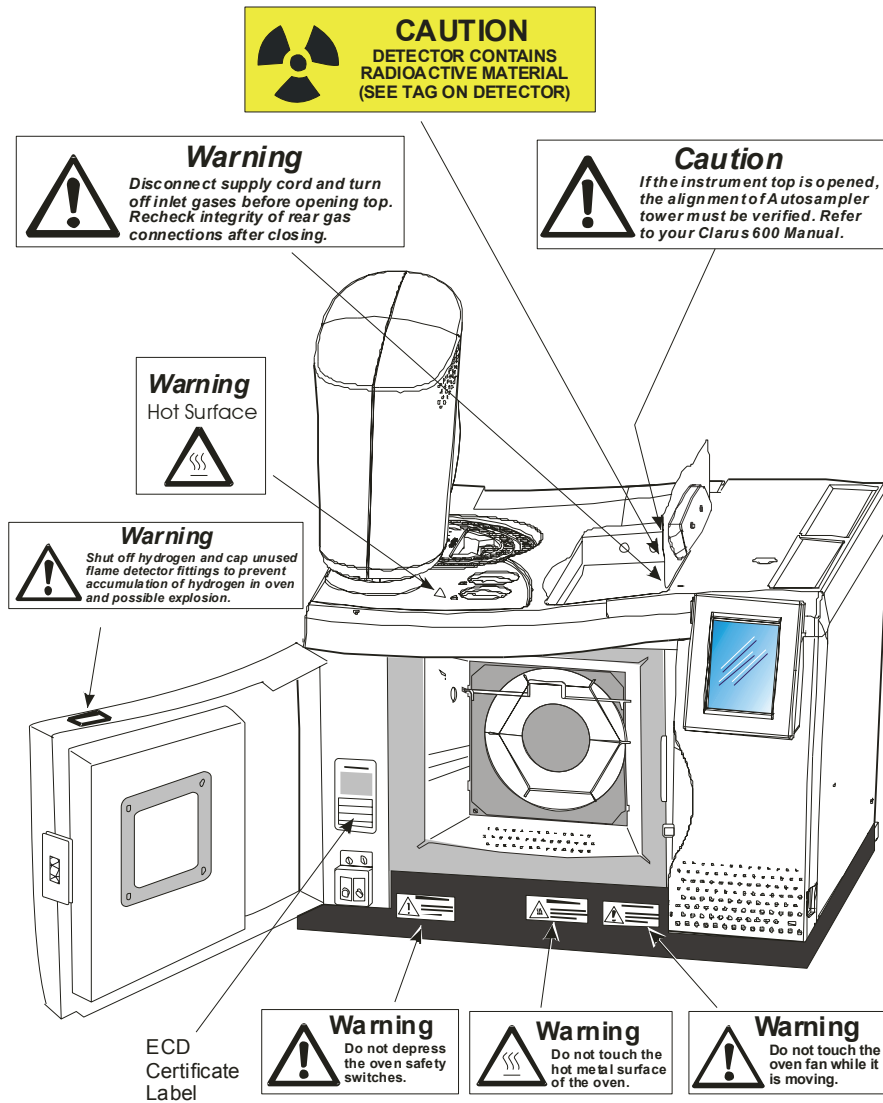


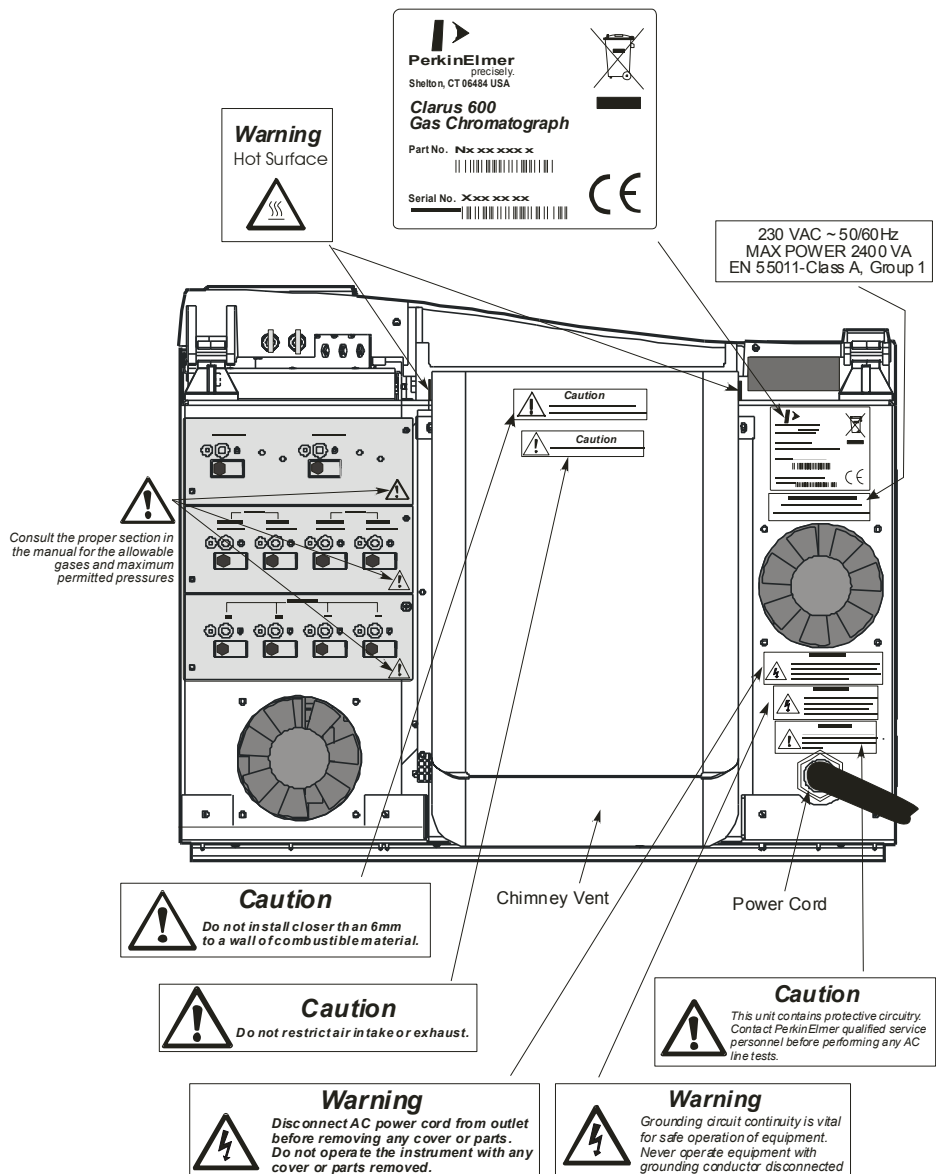
## ***Moving the Clarus GC***

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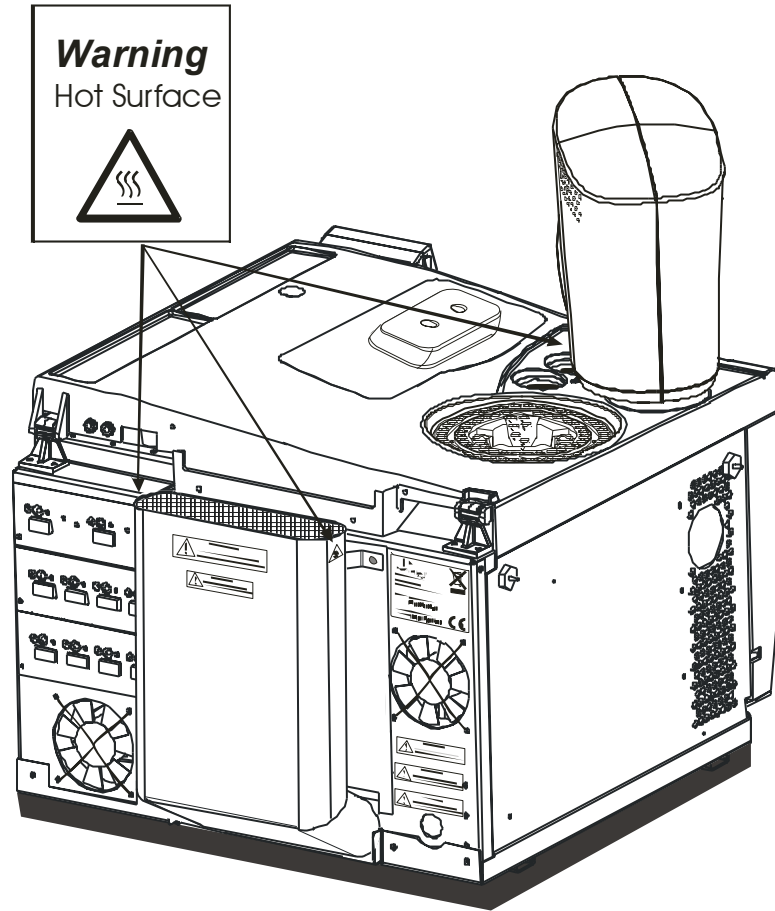
The Clarus GC weighs 69 kg (152 lb). Improper lifting can cause injury to the back. If the instrument must be moved, we recommend that at least two people carefully lift the instrument in order to move it.

## Labels on the Clarus GC





*Safety Practices*



## ***ECD Radioactive Hazards***

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**WARNING**

*To assure that removable radioactive contamination on the external parts of the ECD remains at a safe level, the United States Nuclear Regulatory Commission requires that:*

- *The ECD must be wipe tested at least once every six months.*
- *A record of the results must be maintained for NRC inspection.*

### ***United States Government Regulations for ECDs***

**NOTE:** *To repair an Electron Capture Detector cell requires a specific license issued by the U.S. Nuclear Regulatory Commission (NRC) and/or in some states by the equivalent state agency. For further information on obtaining a license, contact the Customer Service Department at PerkinElmer, Shelton, Connecticut, or the NRC Material Branch, Office of Nuclear Materials, Safety and Safeguards, Washington, DC 20555.*

All USNRC regulations can be obtained through the internet at [www.nrc.gov/reading-rm/](http://www.nrc.gov/reading-rm/)

**NOTE:** *These instructions are for ECD cell purchasers who are not specifically licensed to handle radioactive materials.*

The Clarus GC Electron Capture Detector model (Part No. N610-0063) contains a maximum of 15 mCi of Nickel 63 (Ni 63), a radioactive material. Your possession and use of this detector is governed by 10 C.F.R. Section 31.5 which is reproduced in Appendix I. Under the provisions of that regulation you are deemed a "General Licensee."

Your possession and use of the detector cell may also be regulated by the state where you are located. The requirements of state regulatory agencies are substantially similar to those contained in NRC regulation 10 C.F.R. Section 31.5, but they may differ in some respects. It is suggested that you procure a copy of the regulations of your particular state. (Supplement 2 in Appendix I contains a list of the "Agreement States" which have been granted authority by the U.S.

## *Safety Practices*

Nuclear Regulatory Commission to regulate the possession and use of radioactive material.)

It is required that you be familiar with regulation 10 C.F.R. Section 31.5 (Appendix 1 in the Hardware Guide 0993-6781). Following are summaries of its requirements.

### ***Labels***

Do not remove any of the labels attached to the ECD cell or any of the labels attached to your Clarus Gas Chromatograph that refer to the ECD cell. Follow all instructions and abide by all precautions provided by the labels and in user instruction manuals referred to by the labels.

### ***Leak Testing***

You are obligated under U.S. federal and state regulations to make certain that the ECD cell is wipe-tested for leakage of radioactive materials at intervals of no longer than six months, and that the analysis of these wipe tests is conducted by a person specifically licensed to do so, either by the U.S. Nuclear Regulatory Commission or by an Agreement State. The analyses can be performed by the firm listed below:

National Leak Test Center  
P.O. Box 486  
North Tonawanda, New York 14120

### ***ECD Cell Failure or Damage***

If a leak test detects more than 0.005  $\mu\text{Ci}$  (microcurie) of removable radioactive material on the surface of an ECD cell, or if the cell itself is damaged in such a way as to indicate that it may no longer adequately shield the radioactive material inside, you must immediately suspend operation of your chromatograph until the cell has been repaired or disposed of by ***a person specifically licensed to do so***. Any such incident must be reported by you to the Regional Office, Inspection and Enforcement, U.S. Nuclear Regulatory Commission.

## **Reporting Radiation Incidents, Theft or Loss**

Please read Regulation 10 C.F.R. Section 20.2201 and 20.2202. These describe your duties should the radioactive material (Ni 63) in the ECD cell be lost, stolen, or released, or should any person be exposed to radiation.

## **Other ECD Requirements**

Regulation 10 C.F.R. Section 31.5 (see Appendix I) does not permit you to abandon the ECD cell or export it. It may not be transferred except to a person specifically licensed to receive it. Within thirty days of such a transfer, you must report to the Director of Nuclear Material Safety and Safeguards, U.S. Nuclear Regulatory Commission, Washington, D.C. 20555, the name and address of the transferee. However, no report is needed to transfer a defective ECD cell to PerkinElmer in order to obtain a replacement.

You may transfer the ECD cell to another general licensee, like yourself, only when it remains at the same location to which it was shipped by PerkinElmer. Give the transferee a copy of these instructions and the regulations in Appendix I, and report to the commission as required in Regulation C.F.R. Section 31.5.



**WARNING**

***NEVER DISMANTLE THE ECD CELL!!***

*You can remove the ECD cell from the GC for repair.*

## **United Kingdom Regulations**

In the U.K., registration is required under the Radioactive Substances Act of 1960, for anyone keeping or using radioactive materials. Application should be made to any one of the following governing bodies:

**ENGLAND**    Department of the Environment  
                  Queen Anne's Chambers

*Safety Practices*

Tothill Street  
London, SW1H 9J4

**SCOTLAND** Scottish Development Department  
21 Hill Street  
Edinburgh, EH2 3J4

**WALES** Welsh Office  
Cathay's Park  
Cardiff, CF1 3NG

**NORTHERN  
IRELAND** Ministry of Development  
Parliament Building  
Storemont  
Belfast, Northern Ireland



## ***Safe Handling of Gases***

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When using hydrogen, either as the combustion gas for a flame ionization detector or as a carrier gas, special care must be taken to avoid buildup of explosive hydrogen/air mixtures. Ensure that all hydrogen line couplings are leak-free and do not allow hydrogen to vent within the oven.

### ***Ventilation***

Adequate ventilation must be provided, particularly if a liquid nitrogen or carbon dioxide subambient accessory is in constant use. When analyzing hazardous compounds, such as pesticides, it may be necessary to arrange for venting of detector effluent into a fume hood.

### ***Using Hydrogen***



**WARNING**

*Flame Ionization Detectors (FID) and Flame Photometric Detectors (FPD) use hydrogen as fuel. If the hydrogen is turned on without a column attached to the injector and detector fittings inside the oven, hydrogen could diffuse into the oven creating the possibility of an explosion. To avoid possible injury, **DO NOT TURN ON THE HYDROGEN UNLESS A COLUMN IS ATTACHED AND ALL JOINTS HAVE BEEN LEAK TESTED.***

*Before disconnecting a column, make certain that the hydrogen has been turned **OFF**.*

*If two FIDs or FPDs are installed and only one has a column attached to it, make certain that you cap off the unused detector inlet fitting with a 1/8-inch stainless steel plug (Part No. N930-0061).*



**Contact the gas supplier for a material safety data sheet (MSDS) containing detailed information on the potential hazards associated with the gas. Carefully use, store, and handle compressed gases in cylinders. Gas cylinders can be hazardous if they are mishandled.**

**NOTE:** *The permanent installation of gas supplies is the responsibility of the user and should conform to local safety and building codes.*

If liquid nitrogen is used, the gas cylinder must be fitted with an over-pressure regulator which will vent the cylinder as necessary to prevent it from becoming a safety hazard.

Consult the following references for more detailed information and additional guidelines about gas cylinders.

- Compressed Gas Association (USA), "Safe Handling of Compressed Gases in Containers," pamphlet no. P-1, 1984.
- Compressed Gas Association (USA), "The Inert Gases – Argon, Nitrogen and Helium," pamphlet no. P-9, 1992.

### **Identification of Gas Cylinders**

- Legibly mark cylinders to identify their contents. Use the chemical name or commercially accepted name for the gas.

### **Storing Gas Cylinders**

Review the following precautions with the customer to ensure the safe use and storage of gas cylinders.

- Cylinders should be stored in accordance with the regulations and standards applicable to the customer's locality, state, and country.

- When cylinders are stored indoors in storage rooms, the storage room should be well ventilated and dry. Ensure that the ventilation is adequate to prevent the formation of dangerous accumulations of gas. This is particularly important in small or confined areas.
- Do not store cylinders near elevators, gangways, or in locations where heavy moving objects may strike or fall against them.
- Use and store cylinders away from exits and exit routes.
- Locate cylinders away from heat sources, including heat lamps. Compressed gas cylinders should not be subjected to temperatures above 52 °C (126 °F).
- It is recommended that gas cylinders be stored and placed outside the laboratory and connected to the instrument through copper lines.

### ***Handling of Gas Cylinders***

- Do not allow ignition sources in the storage area and keep cylinders away from readily ignitable substances such as gasoline or waste, or combustibles in bulk, including oil.
- Store cylinders standing upright, fastened securely to an immovable bulkhead or permanent wall.
- When storing cylinders outdoors, they should be stored above ground on a suitable floor and protected against temperature extremes (including the direct rays of the sun).
- Arrange gas hoses where they will not be damaged or stepped on and where things will not be dropped on them.
- Take care not to kink or stress the gas lines. For safety, cylinders should be firmly clamped in position.

### *Safety Practices*

- If it becomes necessary to move the cylinders, do so with a suitable hand truck after insuring that the container cap is secured and the cylinder is properly fastened to the hand truck.
- Use only regulators, tubing and hose connectors approved by an appropriate regulatory agency.
- Do not refill cylinders.
- Check the condition of pipes, hoses and connectors regularly. Perform gas leak tests at all joints and seals of the gas system regularly, using an approved gas leak detection system.
- When the equipment is turned off, close all gas cylinder valves tightly at the cylinder. Bleed the remainder of the line before turning the exhaust vent off.

## **Hazardous Chemicals**

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Before using samples, you should be thoroughly familiar with all hazards and safe handling practices. Observe the manufacturer's recommendations for use, storage and disposal. These recommendations are normally provided in the Material Safety Data Sheets (MSDS) supplied with the solvents.

Be aware that the chemicals that you use in conjunction with the GC may be hazardous. **DO NOT** store, handle, or work with any chemicals or hazardous materials unless you have received appropriate safety training and have read and understood all related Material Safety Data Sheets (MSDS). MSDS's provide information on physical characteristics, precautions, first aid, spill clean up and disposal procedures. Familiarize yourself with the information and precautions contained in these documents before attempting to store, use or dispose of the reagents. Comply with all federal, state, and local laws related to chemical storage, handling, and disposal.

You must work under a suitable hood when handling and mixing certain chemicals. The room in which you work must have proper ventilation and a waste collection system. Always wear appropriate safety attire (full-length laboratory coat, protective glasses, gloves, etc.), as indicated on Material Safety Data Sheets.



*Some chemicals used with this GC may be hazardous or may become hazardous after completion of an analysis. The responsible body (for example, the Lab Manager) must take the necessary precautions to ensure that the GC operators and the surrounding workplace are not exposed to hazardous levels of toxic substances (chemical or biological) as defined in the applicable Material Safety Data Sheets (MSDS) or OSHA, ACGIH, or COSHH documents. Venting for fumes and disposal of waste must be in accordance with all national, state and local health and safety regulations and laws.*

## **Definitions in Warning for Hazardous Chemicals**

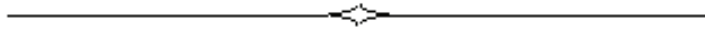
**Responsible body.** “Individual or group responsible for the use and maintenance of equipment, and for ensuring that operators are adequately trained.” [per IEC 61010-1].

**Operator.** “Person operating equipment for its intended purpose.”  
[per IEC 61010-1].

**OSHA:** Occupational Safety and Health Administration (United States)

**ACGIH:** American Conference of Governmental Industrial Hygienists

**COSHH:** Control of Substances Hazardous to Health (United Kingdom)



# **System Description 3**

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*System Description*



The Clarus 600 Gas Chromatograph is a programmable pneumatic control (PPC™), dual-channel, temperature-programmable stand-alone gas chromatograph (GC). It is available in many configurations, such as with or without, an autosampler, and a variety of injector/detector combinations to provide you with total GC flexibility. The Clarus GC is microprocessor controlled. You use the touch screen to operate this instrument.



**Figure 3-1. The Clarus 600 GC.**

## **Overview of the Clarus 600 GC**

---

Your Clarus GC may have none, one, or two of the following detectors installed:

- Flame Ionization (FID)
- Electron Capture (ECD)
- Thermal Conductivity (TCD)
- Photoionization (PID)
- Nitrogen Phosphorus (NPD)
- Flame Photometric (FPD)

The FID, ECD, TCD, NPD, or FPD may be installed in either the front or the rear detector position. But only one PID can be installed and it must be installed only in the rear position (Channel 2).

Each installed detector has one analog output which may be attached to either an integrator or recorder. Signals may be routed under instrument control.

Either none, one, or two packed column injectors; none, one, or two capillary column injectors; or one of each injector type may be installed. Capillary column injectors consist of the conventional split/splitless injector (CAP), a temperature-programmed split/splitless injector (PSS), and a temperature programmed on-column injector (POC).

Up to two gas sampling valves may be installed.

In programmable pneumatic control (PPC) the carrier gas and detector gases are monitored and controlled by the microprocessor, thereby producing a fully automated system that is capable of managing all pneumatic functions within the gas chromatograph.

The Clarus GC can store up to five GC methods. Methods can be generated, copied, deleted, edited, set up, and printed.

The autosampler can run up to 15 injections per vial from as many as 82 vials and one priority vial using one or two autosampler programs. In the latter case, a different GC method can be used by each program if desired. The autosampler operates in one of two program modes, single or multi.

Real-time digital readouts are provided to simplify setting carrier gas pressures and flows, if flow readout or PPC is installed. An additional digital readout can be displayed to show the detector output (in mV).

## About the Touch Screen

---

You control the Clarus GC by touch screens. The touch screens are your link to the software. The screens are designed to provide the easiest access to the most frequently-used features. The touch screen allows quick access to settings and functions, such as the Oven program and Ignite button.

The System Status screen provides access to all heated zones. The icons represent configured components. These icons will guide you through the application.

The System Status screen provides you with one-click access to the oven settings. For detailed information on the touch screen see the *Clarus 600 User Guide (P/N 0993- 6780)*.

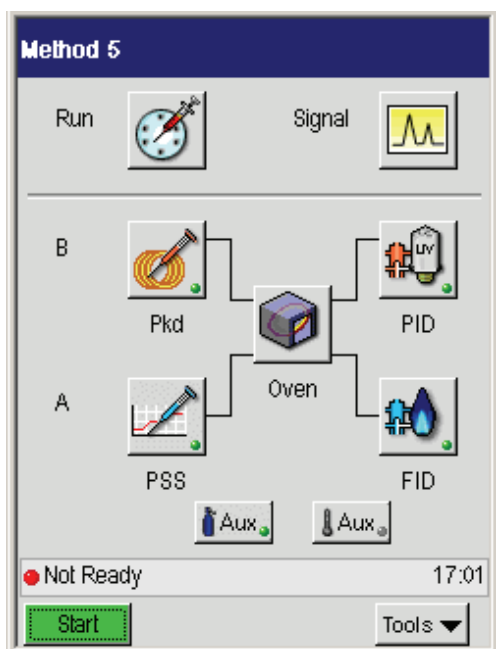


Figure 3-3. The Clarus GC System Status screen.

# **Before You Install a Column 4**

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*Before You Install a Column*



**WARNING**

*At the moment you turn on the GC, the oven, injector(s), and detector(s) begin to heat up rapidly. To avoid injury while installing a column, all heaters should be turned off and their respective zones allowed to cool before touching the injector septum caps or any of the fittings inside the oven.*


This chapter contains general column installation information, and the following procedures:

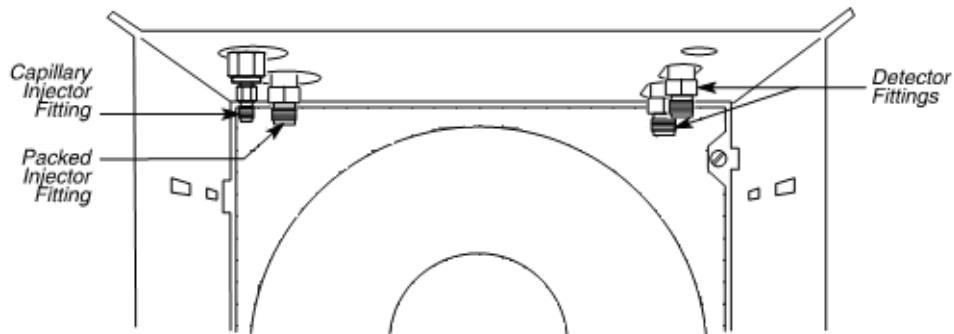
- Installing your column
- Protecting your column

## **Column Installation Information**

### ***Injector and Detector Fittings***

Columns are installed inside the oven. The injector fittings are on the left side and the detector fittings are on the right side of the oven ceiling. Figure 4-1 shows a capillary injector fitting in the front position and a packed injector fitting in the rear position.

	<p><i>Before installing a column, make certain the oven is OFF (by opening the oven door), the oven fan has stopped, and the oven is cool.</i></p>
<p><b>WARNING</b></p>	



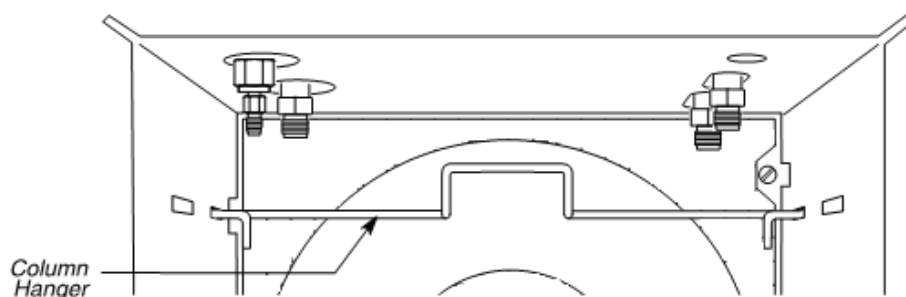
**Figure 4-1. Injector and detector fittings location in the oven.**



## Capillary Column Hangers

Capillary columns are supported on column hangers. The left and right sides of the oven walls each have two rectangular slots into which column hangers are inserted. The two rear slots are used to install a column hanger in the rear position. The two front slots are used to install a column in the front position.

To install a column hanger, simply insert one end into the left slot and the other end into the right slot. If you are installing two capillary columns, install the rear hanger and the rear column before installing the front hanger and the front column.



**Figure 4-2. A column hanger installed in the rear oven position.**

## **Protecting Your Column**

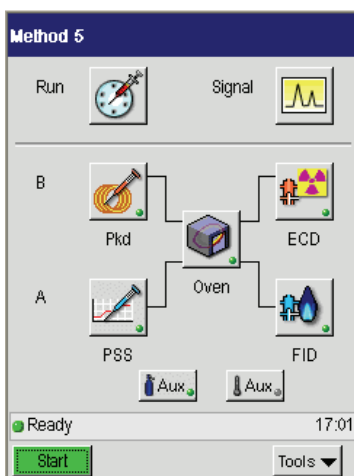
The Clarus 600 GC provides a means for protecting your column(s) from overheating. It does this by not allowing the oven to heat up beyond the **Oven Maximum Temperature Limit (OMTL)**, a value that you set in the active method.

You should set the OMTL equal to or less than the maximum permissible operating temperature recommended in the specifications for your column. If you are installing two columns, use the lower of the two permissible maximum operating temperatures.

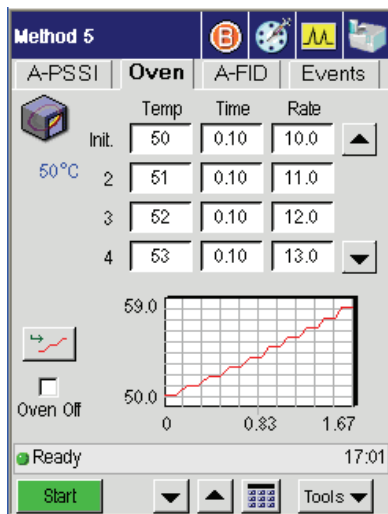
To protect the column, the OMTL value (that you enter) works in conjunction with the Oven Temperature specified in the Active Method. Should you (or someone else) attempt to set an Oven Temperature in the Active Method to a value greater than the OMTL, the system displays an error message and will not allow you to continue until an appropriate new oven temperature is set.

### **To set the oven temperature:**

1. Go to the System Status screen and touch the **Oven** icon:



2. Touch the field you need to update. It will appear black and then using the plus and minus buttons input the correct oven temperatures and time.



For information on turning the heaters and detectors on and off see the *Clarus 600 Users Guide (P/N 0993-6780)* chapter on the Active Method.

*Before You Install a Column*



# Installing a Packed Column **5**

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This chapter describes procedures for installing a packed column and setting the carrier gas flow using programmed pressure control (PPC). After installing a packed column as described in this chapter, you should set up the detector(s) to be used with this injector.

## **Summary**

---

The following steps summarize how to install a *new* packed column and get it ready for use:

- Step 1: Turn off the heaters.
- Step 2: Set the carrier gas flow.
- Step 3: Attach one end of the column to the packed injector.
- Step 4: Leak test.
- Step 5: Condition the column
- Step 6: Attach the other end of the column to the detector.
- Step 7: Leak test the column/detector connection.

**NOTE:** *A new packed column should not be used until it is conditioned overnight with carrier gas flowing through the column. **Do not connect** the column to the detector during the conditioning process.*



## ***Materials and Tools Required***

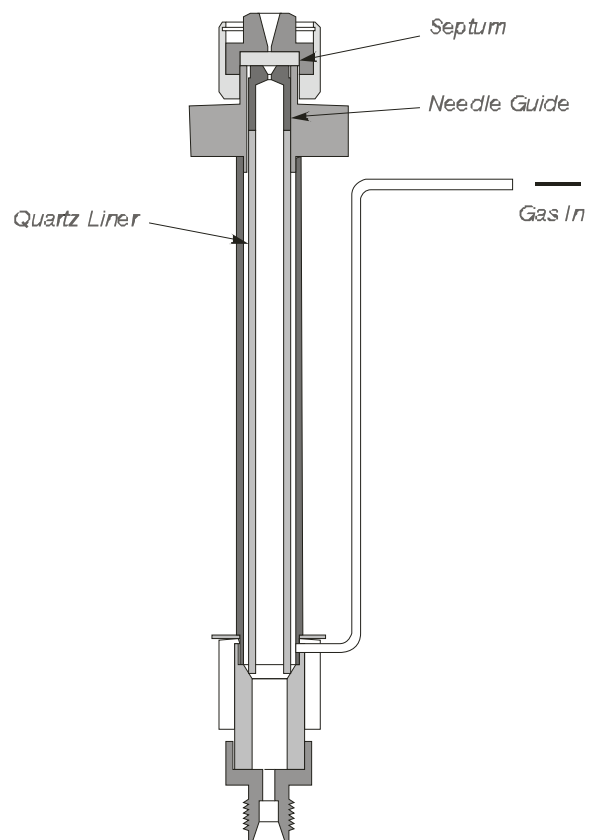
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- Two 7/16-inch open end wrenches when a normal 1/8-inch column is being installed. Two 9/16-inch open end wrenches will be required in addition if a 1/4-inch column is being installed.
- Two 1/8-inch to 1/4-inch adapters (P/N 0008-0100) if a 1/4-inch column is being installed.
- A flow meter or the optional flow readout.
- A packed column of your choice.
- A leak test solution or an electronic leak tester.

## ***Packed Column Injector Overview***

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The packed column injector consists of a septum cap, needle guide, quartz injector liner, and the injector body. This injector is used with 1/8-inch or 1/4-inch glass or metal packed columns. In addition, by installing the 530 Micron Wide-Bore Adapter Kit (P/N N612-0001) you can convert the injector to accept wide-bore capillary columns.



**Figure 5-1. Packed Column Injector.**

## About the Wide-Bore Adapter

If you are operating in the off-column mode at above optimum flow rates (>10 mL/min), you may not need to install the wide-bore quartz injector liner. Depending on your sample or solvent, the solvent profile (tail) may be acceptable for your application with the standard liner (the illustration at the left in Figure 5-2) and the addition of the adapter fitting (P/N N610-0083). However, if the solvent profile is not acceptable, install the wide-bore quartz injector liner.

The off-column or on-column flash vaporization mode of operation is determined by the position of the hourglass portion of the wide-bore quartz injector liner in the packed column injector. When installed correctly, this liner produces improved solvent profiles, especially at optimum flow rates. For complete installation instructions, refer to the *Installation Instructions: 530 Micron Wide-Bore Adapter Kit for the AutoSystem GC and Clarus GC* (P/N 0993-8661).

Insert the wide-bore quartz injector liner (P/N N612-1003) into the packed column injector with the hourglass portion in the correct position for your desired mode of operation. Figure 5-2 shows a cross section of a packed column injector containing a standard liner and a cross section of a packed column injector containing a wide-bore quartz injector liner installed in the off-column position and the on-column position.

### ***For off-column flash vaporization (hourglass end first):***

To avoid contaminating the quartz wool, wear vinyl, powder-free disposable gloves (the same type used to perform maintenance on TurboMass). Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the liner. Then using a 1/16-inch rod (P/N N610-T100), push the quartz wool into the liner. Loosely pack some quartz wool in the top portion of the liner to wipe the syringe needle upon injection. Insert the wide-bore quartz injector liner into the packed column injector with the hourglass end first.

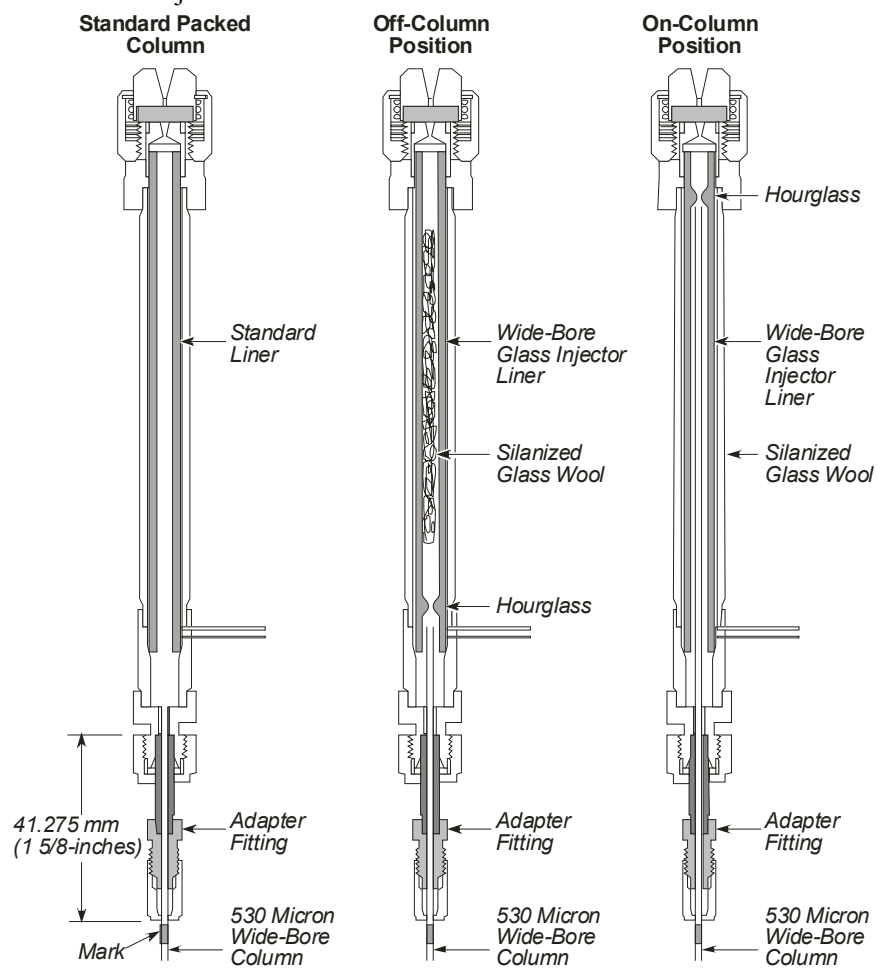
**Or**

For on-column flash vaporization (hourglass end last):

Insert the wide-bore quartz injector liner into the packed column injector with the hourglass end of the liner last. Do not pack the wide-bore quartz injector liner with silanized quartz wool. You must use a 0.47-mm O.D. syringe in this mode.

## Installing a Packed Column

If you are using the autosampler, install a 0.47-mm O.D. syringe and use the "SLOW" injection mode. If the Clarus GC software is Rev. 1.6 or lower, use the "ON-COL" injection mode.



**Figure 5-2. Cross sections of three packed injector configurations with a wide-bore column.**

## Step 1: Turn off the Heaters



**WARNING**

*The moment the GC is turned on, the oven, injector(s), and detector(s) begin to heat up rapidly. To avoid burns and injury while installing a column, all heaters should be turned off and their respective zones allowed to cool before touching the injector septum caps or any of the fittings inside the oven.*

**NOTE:** See the Clarus 600 GC Users Guide for detailed procedures for turning heaters off and on.

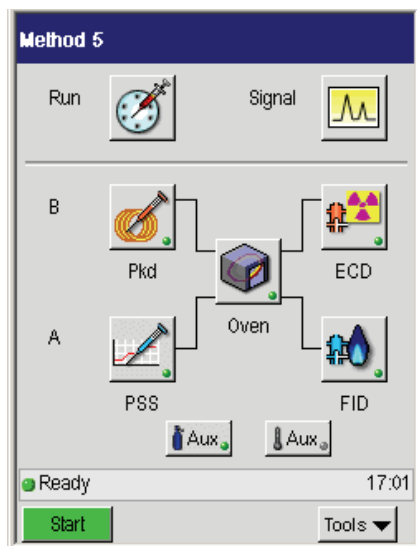
**NOTE:** It is recommended that you remove the injector liner shipped with the packed injector and pack it with a small amount of silanized glass wool before performing analyses. Please refer to the Maintenance chapter in this manual.

## Step 2: Set the Carrier Gas Flow

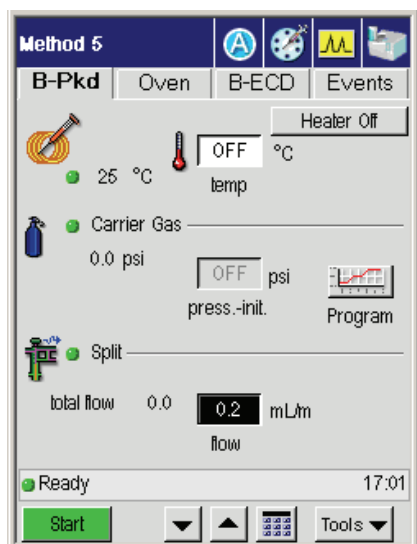
To set the carrier gas using PPC pneumatics modules:

1. Turn on the carrier gas at the tank.
2. Adjust the line pressure between 60 to 90 psig.
3. From the System Status screen select either **A** or **B** injector (the example below shows the **B** injector ready for setting the carrier gas).




## Installing a Packed Column

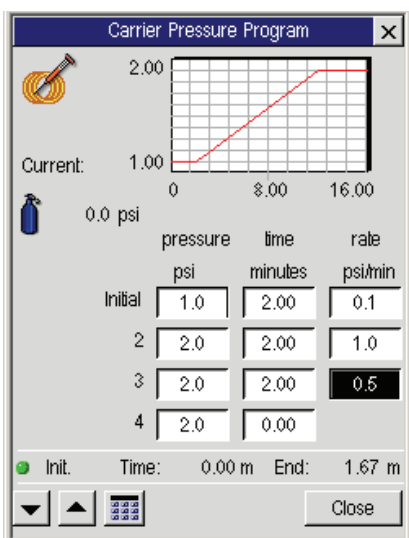


4. Touch the Pkd button  and the following screen appears:



5. Touch the Program button in the **Carrier Gas** field.

6. Touch a field to select it and using the up and down arrow buttons or the keypad    button to enter the desired values.

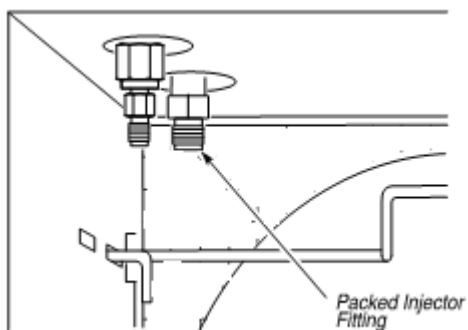


## Installing a Packed Column

### Setting the Carrier Gas Flow Using a Soap Bubble or Electronic Flowmeter

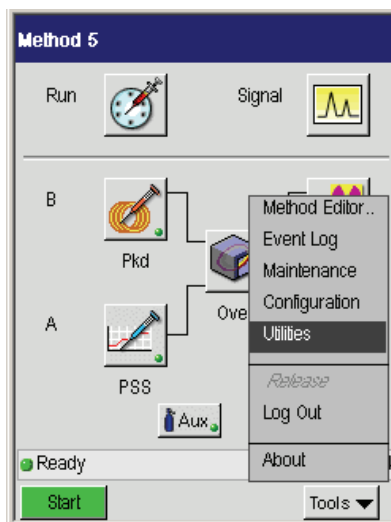
The procedure below assumes that you know how to measure carrier gas flow using a soap bubble or electronic flowmeter and the built-in stopwatch. If you need instructions, please read “Using Tools,” in the *Clarus 600 GC Users Guide* (P/N 0993-6780) before proceeding.

1. Locate the packed injector fitting inside the oven.



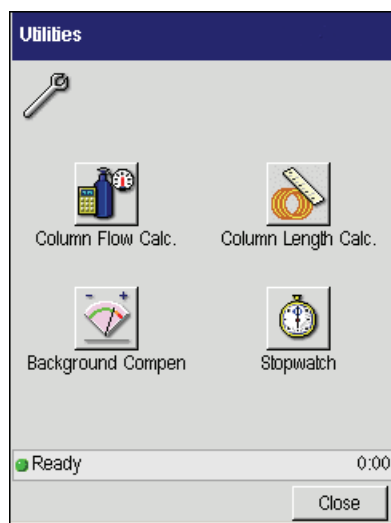
**Figure 5-4. A packed injector fitting.**


2. Attach a soap bubble flowmeter to the packed injector fitting.
3. Turn on the carrier gas at the tank and adjust the line pressure to 90 psig.
4. From the System Status Screen touch the **Tools** button and then select **Utilities** from the drop down menu.

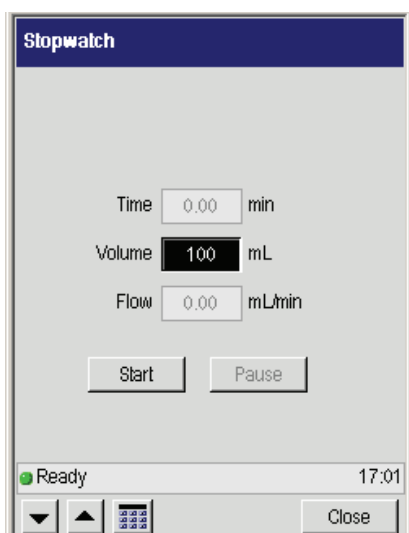




5. In the **Utilities** window select the **Stopwatch** button.



6. Using the up and down arrow buttons or the keypad  button enter the appropriate values and press **Start**.



### *Installing a Packed Column*

7. Start the carrier gas flowing by turning the flow controller knob counterclockwise and measure the flow.

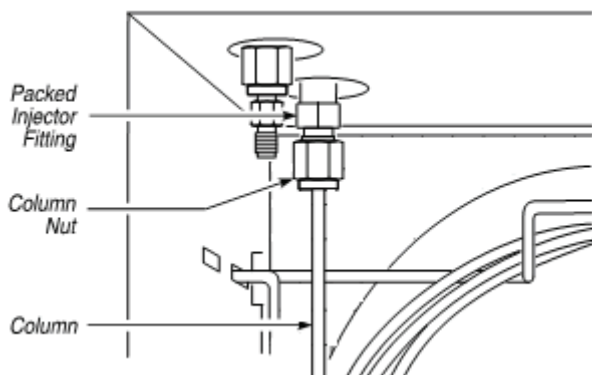
**NOTE:** *For best accuracy, use a soap bubble flowmeter volume that gives a reading of at least 30 seconds.*

8. Adjust the flow to the desired set point by repeatedly measuring the flow and turning the flow controller knob counterclockwise to increase the flow, clockwise to decrease the flow, until the desired flow is obtained.
9. Disconnect the soap bubble flow meter before proceeding to the next step.

### **Step 3: Connect One End of the Column to the Packed Injector**

**NOTE:** *If you are installing a 1/4-inch column, attach a 1/8-inch to 1/4-inch adapter to the packed injector fitting before continuing. Finger tighten the adapter, then while holding the packed injector fitting steady with a 7/16-inch wrench, tighten the adapter with a 9/16-inch wrench.*

1. Insert one end of the column into the packed injector fitting until it bottoms, then finger tighten the column nut onto the packed injector fitting (see below).



**Figure 5-3. Packed column connected to a packed injector fitting.**

2. While holding the packed injector fitting with one 7/16-inch wrench, tighten the column nut an additional 1/8 to 1/4 turn with the other wrench.

**NOTE:** *Do not overtighten column nuts. Overtightening causes permanent damage to the fittings.*

### **Step 4: Leak Test**

Test the connection to the packed injector fitting for leaks using a 50/50 mixture of isopropanol/water or an electronic leak detector. To avoid contaminating the system, DO NOT use a soap solution for leak testing. Tighten all leaking connections.

### **Step 5: Condition the Column**

This section contains a suggested temperature program for conditioning a column. The program starts off by holding the oven temperature at a medium value for 10 minutes, gradually increasing the oven temperature at a fixed rate (5 °C/min) to the column operating temperature, then holding that temperature overnight with the carrier gas flowing.

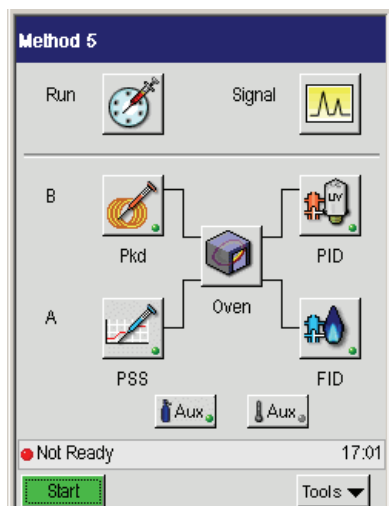
**CAUTION**


*The temperatures shown in the following examples should only be used as guidelines. Please refer to the column manufacturer's operating instructions for specific temperature recommendations.*

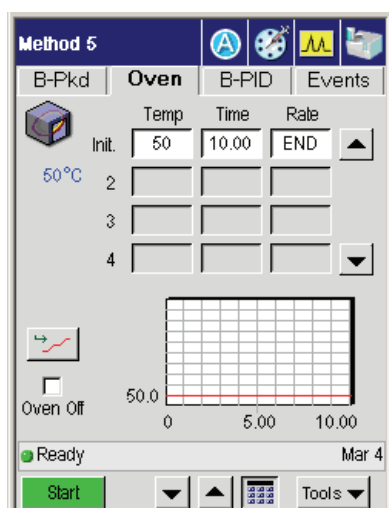
To condition the column:

1. Close the oven door, go to the System Status screen, and touch the oven icon.




## Installing a Packed Column

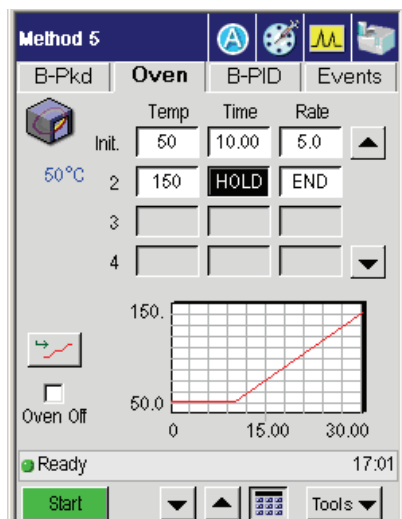


2. Select the **Init Temp** field. Then using the up and down arrow or keypad  buttons enter the **initial (Init)** oven temperature to a set point of **50**.



3. Enter an **Init Time** of **10**.
4. To add another program step, enter an **Init Rate** of **5** (°C/min).

- Using the up and down arrow or keypad    buttons enter for **Temp 2**, a set point **25 °C to 50 °C** above your planned analytical operating temperature. For example, enter a set point of **150**.





- Set **Time 2** to **999**. This displays HOLD in the field.

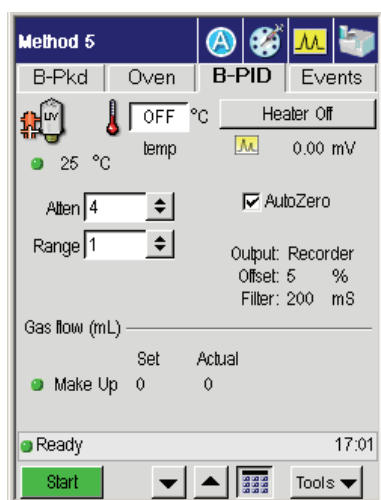
**CAUTION** *To avoid damaging the column, do not enter a temperature higher than the maximum operating temperature specified by the column manufacturer.*

- Select the B injector by clicking the **B-Pkd** tab.

## Installing a Packed Column



8. In the above screen touch the **temp** field, then using the up and down arrow or keypad   buttons set an Injector **Temp** about **50 °C** higher than the **Oven Temp 2** setting. See the chapter “Active Method” in the *Clarus 600 GC User’s Guide* for information on setting Temp 2.
9. Select the detector **B-PID** tab, then turn the Detector Temperature off and run the system overnight.

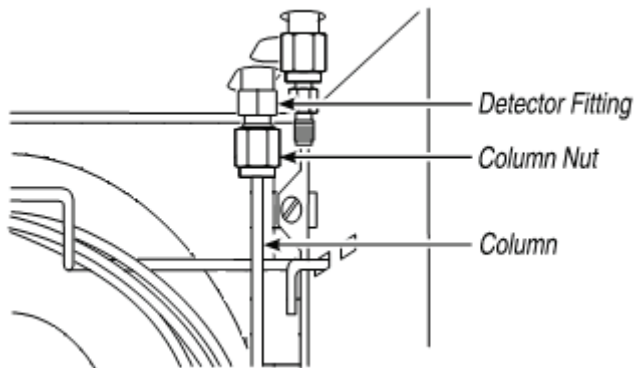


10. The next morning go to the System Status screen and select the **Oven** button.
11. Reset the **Oven** temperature set point to that specified for **Temp 1** at the beginning of the temperature program.
12. Open the oven door and allow the oven to cool until the oven fan goes off. This occurs when the oven cools down to 40 °C.

**NOTE:** *Condition a new column before using it in an analysis. Once it is conditioned, you will not need to recondition it.*

### **Step 6: Attach the Other End of the Column to the Detector**

1. Insert the free end of the column into the detector fitting, then finger tighten the column nut onto the detector fitting.



**Figure 5-5. Packed column attached to the rear detector fitting.**

2. While holding the detector fitting with one of the 7/16-inch wrenches, tighten the column nut an additional 1/8 to 1/4 turn with the other wrench.

## Installing a Packed Column

### CAUTION

*Make certain that no part of the column touches the bottom or sides of the oven once it is installed.*

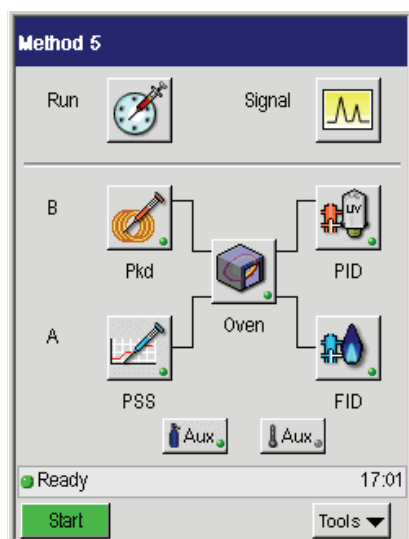
**NOTE:** *If you are installing a 1/4-inch column, attach a 1/8-inch to 1/4-inch adapter to the detector fitting before continuing. Finger-tighten the adapter, then while holding the detector fitting steady with a 7/16-inch wrench, tighten the adapter with a 9/16-inch wrench.*

### Step 7: Leak Test the Column/Detector Connection

The following procedures describe leak testing the column to detector connections.

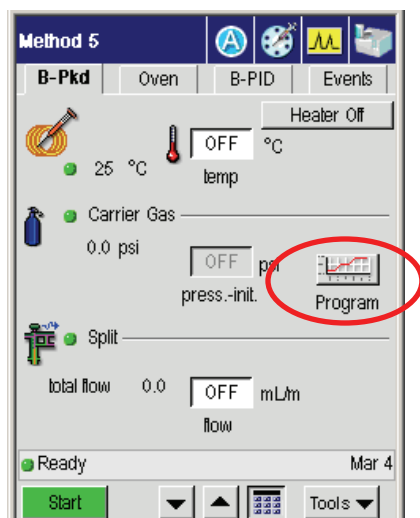
1. From the System Status screen select either A or B injector (the example here shows the B packed injector ready for setting the carrier gas).




In this example, touch the **Channel B** packed injector button

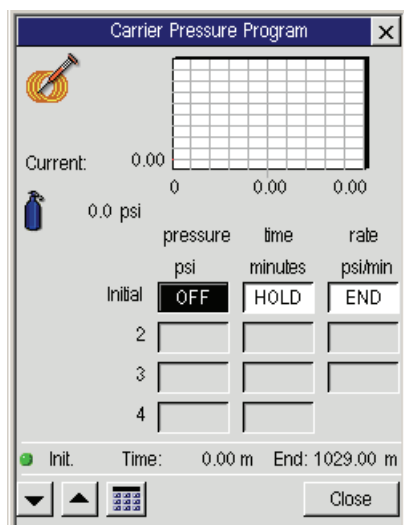




- In the following window touch the **Program** button in the **Carrier Gas** field.



- Using the up and down arrow or keypad    buttons enter pressure 0.1 to 100 psig for minimum and maximum.



### *Installing a Packed Column*

The Clarus 600 GC monitors the column head pressure with a carrier-gas mass-flow controller in use. After selecting the carrier-gas **Flow** mode in configuration, you enter the minimum and maximum values. If the inlet pressure drops below or rises above the set limits for more than one consecutive minute, an error message will appear: **Carrier** (with specific carrier number) **unable to maintain pressure**.

**NOTE:** *Refer to the Clarus 600 GC User's Guide (0993-6780) for minimal and maximum values.*

You should correct the leak; the most common area of a leak is the injector septum. Clear the message by touching the **OK** button before continuing to use the GC. This will stop an autosampler program from continuing.

If you do not make any entries, leak checking will not be done. If you are temperature programming, then the maximum value you enter must be above the column head pressure at the highest oven temperature. You can just enter a value for the minimum and leave the maximum blank. See the *Clarus 600 User's Guide (0995-6780)* for more information.



# Installing a **6** Capillary Column

---



This chapter describes how to install a capillary column in the following three injectors:

- Capillary Split/Splitless (CAP)
- Programmed Split/Splitless (PSS)
- Programmed On-Column (POC)

The information in this chapter is presented as one sequential procedure (Steps A through I) for all three injectors (CAP, PSS, and POC) with the following procedural steps common to all three injectors:

- Setting carrier gas flow using PPC modules

**NOTE:** *For a thorough understanding of PPC, refer to Chapter 8, “PPC Fundamentals.”*

- Leak testing
- Conditioning the column
- Attaching the column to the detector and leak checking

**NOTE:** *If you are analyzing reactive compounds, appropriately deactivate injector liners and wool for your sample type.*

**CAUTION**

*All three capillary injectors; CAP, PSS, and POC use a 1/16-inch fitting for the column connection. This fitting is fragile. To preserve the integrity of the fitting, carefully connect the column nut to prevent cross-threading the fitting and/or overtightening the nut on the fitting. You can also preserve the integrity of the fitting by allowing the injector to cool before connecting a nut.*

## **Summary**

---

The following steps summarize how to install a capillary column and get it ready for use:

- A.** Turn the heaters off.
- B.** Connect the column to the CAP, PSS, or POC injector.
  - 1. Split/Splitless (CAP) injector
  - 2. Programmed Split/Splitless (PSS) injector
  - 3. Programmed On-Column (POC) injector
- C.** Set the carrier gas. (Set the pressure for the CAP and the PSS, or the flow for the POC, using the optional flow readout or a flowmeter.)
  - Setting carrier gas for PPC module
- D.** Leak test all new connections.
- E.** Condition the column (to the manufacturers specifications) and the mechanical joint between the column and pre-column.
- F.** Connect the column to the detector.
- G.** Leak test all new connections.

## ***Materials and Tools Required***

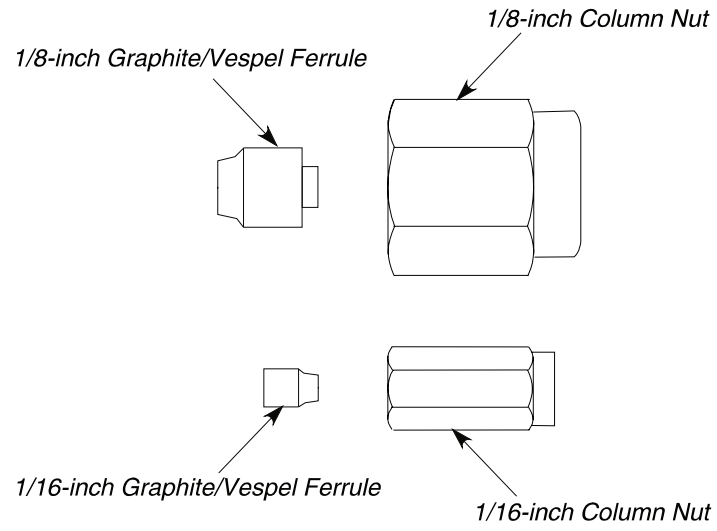
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- 1/8-inch x 1.0-mm graphite ferrules (P/N 0990-3394) for 0.53-mm i.d. columns
- 1/16-inch x 0.8-mm graphite ferrules (P/N 0992-0141)<sup>1</sup> for 0.53-mm i.d. columns
- Two 7/16-inch open end wrenches
- Two 1/4-inch open end wrenches
- One 1/8-inch graphite ferrule (P/N 0990-3981)<sup>1</sup> for 0.32/0.25-mm i.d. columns
- One 1/8-inch column nut (P/N 0990-3453)
- One 1/16-inch graphite ferrule (P/N 0990-3700)<sup>1</sup> for 0.32/0.25-mm i.d. columns
- One 1/16-inch column nut (P/N 0990-3392)<sup>1</sup>
- One screwdriver (P/N 0990-7273)<sup>1</sup>
- Deactivated 0.53-mm i.d. fused silica (P/N N610-1724)
- Fused-silica universal connector (P/N N930-2149)
- Capillary column of your choice
- White-out or felt-tip marker
- Scribe for cutting columns (P/N N930-1376)  
(Pointed scribes are ***not*** recommended.)
- Leak-test solution or electronic leak tester

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<sup>1</sup> Shipped in the Clarus 600 GC Shipping Kit.

*Installing a Capillary Column*



**Figure 6-1. Examples of required fittings.**



## **Step A: Turn the Heaters Off:**

**CAUTION**

*The moment the GC is turned on, the oven, injector(s), and detector(s) begin to heat up rapidly. To avoid injury while installing a column, all heaters should be turned off and their respective zones allowed too cool before touching the injector septum caps or any of the fittings inside the oven.*

**CAUTION**

*Before you install a column, follow the detailed procedures for turning heaters off and on, in Chapter 4 of this manual “Before You Install a Column.” If you have not read this chapter, please do so before proceeding*

## ***Step B: Connect the Column to the Injector:***

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This step contains a separate procedure that describes how to connect a column to each of the following three Clarus 600 GC injectors:

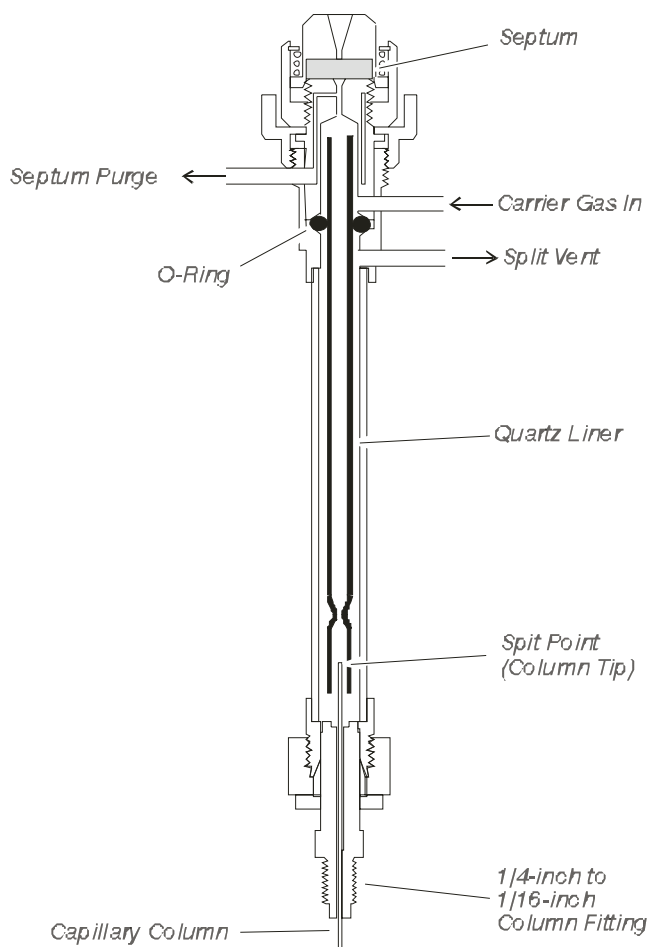
- Step B1:** Connect the Column to the Split/Splitless (CAP) Injector.
- Step B2:** Connect the Column to the Programmed Split/Splitless (PSS) Injector.
- Step B3:** Connect the Column to the Programmed On-Column (POC) Injector.

Proceed to the page that contains the procedure for your injector.

### ***Step B1: Connect the Column to the Split/Splitless (CAP) Injector***

#### ***Overview***

The Split/Splitless injector (CAP) consists of a septum purge assembly and the injector body. Carrier gas enters the injector body at the point just above the O-ring and flows through the quartz liner past the column tip.



**Figure 6-2. Cutaway view of the Split/Splitless injector (CAP).**

### ***About the Injector Liners***

The CAP injector uses the following two quartz liners:

- Narrow-bore (2-mm i.d.) liner (P/N N612-1002).
- Wide-bore (4-mm i.d.) liner (P/N N612-1001).

### *Installing a Capillary Column*

The narrow-bore liner is generally used for splitless injections and the wide-bore liner is generally used for split injections. Due to its small internal volume (0.3 mL), the amount of sample injected into the narrow-bore liner should be limited to about 0.5  $\mu\text{L}$ . This prevents the solvent expansion upon injection from overfilling the liner with vapor.

To wipe the syringe needle, we recommend packing a small amount of quartz wool in the *top portion* of all liner types or injection modes (for example, split or splitless). Each liner should be packed with the quartz wool as described later in this chapter.

### ***Splitless Injections***

In the splitless injection mode, the narrow-bore quartz liner is typically used without quartz wool. The narrow-bore decreases the sample residence time in the liner, making it useful for trace analysis with smaller sample volumes (0.5  $\mu\text{L}$  or less). By closing the split vent, most of the sample mixture enters the column. Then, opening the split vent clears the inlet of residual solvent.

For splitless injection volumes over 0.5  $\mu\text{L}$ , the wide-bore liner with an internal volume of 1.25 mL should be used. However, the amount of sample should be limited to a maximum of 2  $\mu\text{L}$  for hydrocarbon solvents and less than that for high-expansion solvents such as water or  $\text{CH}_2\text{Cl}_2$ . Refer to Table 6-1 for examples of gas volumes formed upon sample injection for selected solvents.

If the wide-bore liner is used for splitless injection, the splitless sampling time (vent-on time) should be at least one minute or more. Also, lower initial oven temperatures may be required to produce good solute resolution in the first few minutes after the solvent peak. The wide-bore liner should be used with columns having an i.d. of 0.32 mm or greater.

**Table 6-1. Gas Volumes Formed Upon Sample Injection  
(Injector 250 °C, Inlet Pressure 10 psig)**

Solvent	Volume Injected (µL)	Gas Volume Generated (µL)
Methylene Chloride	1	401
	2	802
Methanol	1	632
	2	1264
Water	1	1420
	2	2840

***Split Injections***

In the split injection mode, the wide-bore quartz liner is packed with quartz wool to ensure thorough mixing of the sample and carrier gas before they encounter the column tip. The split vent is open at the time of injection so that a fraction of the sample mixture enters the column while the remainder is routed out through the split vent.

*The PPC* pneumatics consists of PPC modules that regulate the inlet flow and pressure of the gases using software controlled by an Clarus GC method.

**CAUTION**

*The CAP injector is shipped with the wide-bore liner installed without quartz wool packing. Before using the injector, remove the liner and pack it with quartz wool. If you are using the injector in the splitless mode, you may want to install the narrow-bore liner.*

***Connecting a Column to the Cap Injector***

The following five steps describe how to connect a column to the CAP injector:

- Step 1. Remove the CAP injector liner.
- Step 2. Select an appropriate CAP injector liner.

## Installing a Capillary Column

Step 3. Pack the CAP injector liner with quartz wool.

Step 4. Reinstall the liner in the CAP injector.

Step 5. Connect a column to the CAP injector.

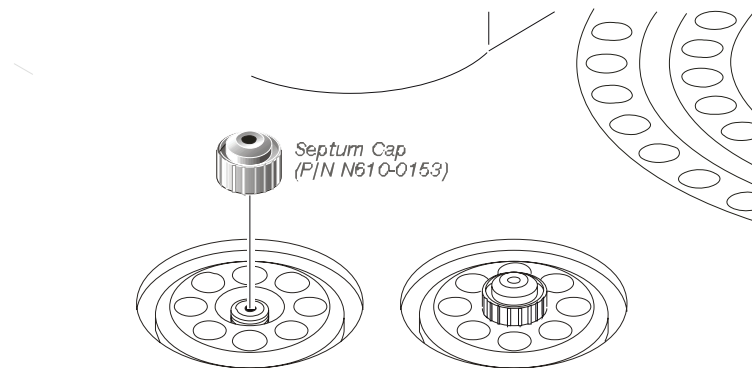
### **Step 1. Remove the CAP Injector Liner.**

To remove a CAP injector liner:

1. Ensure that the injector heater has been turned off.

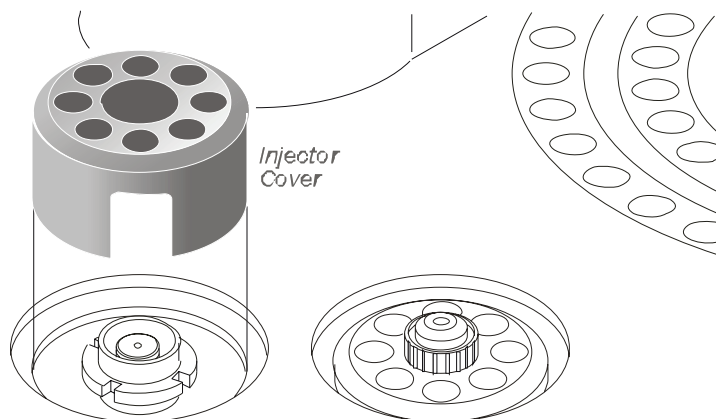
Allow the injector to cool until it is slightly warm to the touch. **Cooling the injector to too-low a temperature (less than 80 °C) will make it difficult to remove the injector liner.**

2. Remove the septum cap.



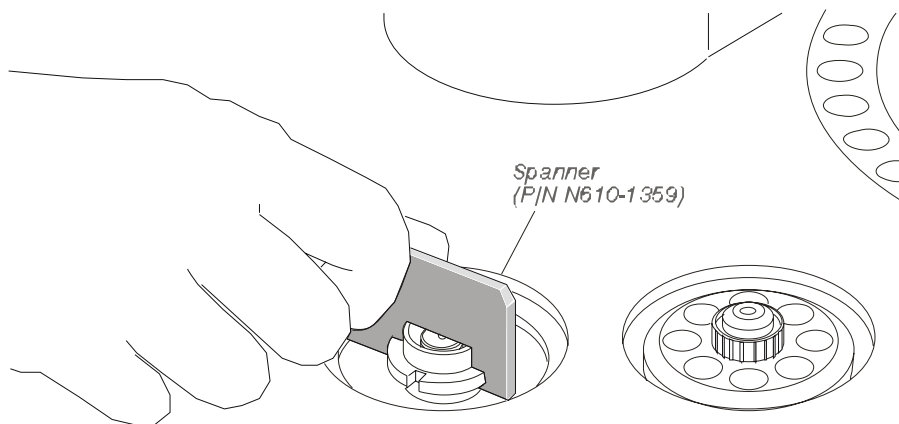
**Figure 6-3. Removing the septum cap.**

3. Remove the injector cover.



**Figure 6-4. Removing the injector cover.**

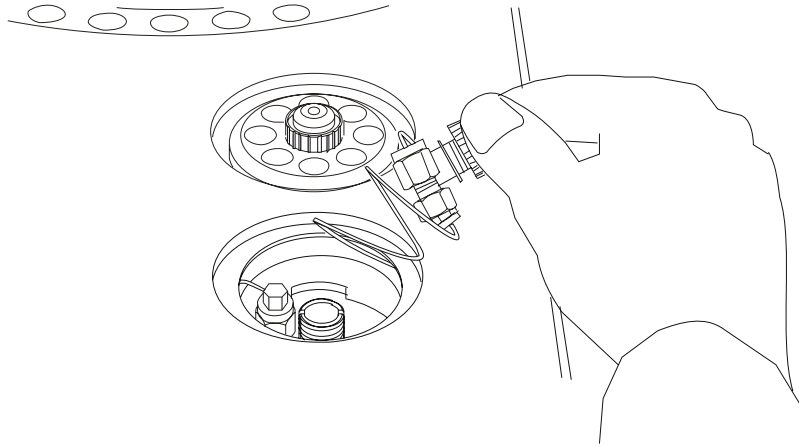
4. Loosen the threaded collar by using the spanner (P/N N610-1359) provided, then remove the threaded collar.



**Figure 6-5. Loosening the threaded collar.**

5. Replace the septum cap on the injector.
6. Pull the septum cap upwards to remove the septum purge assembly.

## Installing a Capillary Column

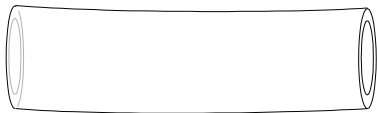


**Figure 6-6. Removing the septum purge assembly.**

The carrier gas inlet line is coiled to allow you to pull the septum purge assembly over to the side and gain access to the liner.

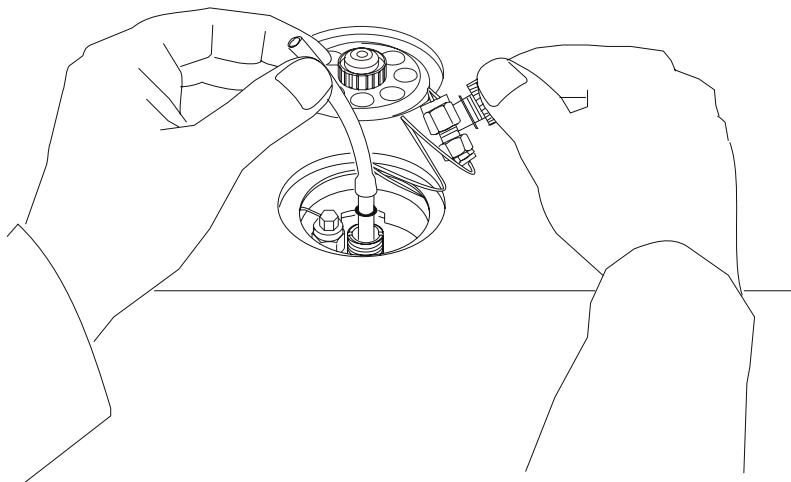
7. Ensure that the CAP injector liner is cool, then twist the CAP injector liner-removal tool (P/N 0250-6534, see Figure 6-8) onto the injector liner. Remove the injector liner by lifting it up and out of the injector.

**The CAP liner must be cool (no hotter than 100 °C) or the liner-removal tool will melt!** The end of the CAP liner-removal tool may flare out with use. If this happens, cut off the flared end with a razor blade or scissors.



**Figure 6-7. CAP injector liner-removal tool (P/N 0250-6534).**





**Figure 6-8. Removing a capillary injector liner.**

**CAUTION**

*If the O-ring adheres to the injector body, use a small screwdriver to loosen the O-ring so that you can remove the liner and O-ring. **Be careful not to scratch the barrel where the O-ring seals.** Discard this O-ring and install a new O-ring.*

**NOTE:** *If the liner breaks inside the CAP injector, it can be removed by first removing the column. Then using a 9/16-inch wrench, remove the 1/4-inch injector fitting inside the oven. The liner should fall out. If the liner is stuck, you can push it out from the top or bottom.*

**Step 2. Select an Appropriate CAP Injector Liner.**

Select the appropriate injector liner for your application. The following two injector liners are available for the CAP injector:

- 4-mm i.d. and 6-mm o.d. CAP injector wide-bore liner (P/N N612-1001)
- 2-mm i.d. and 6-mm o.d. CAP injector narrow-bore liner (P/N N612-1002)

The narrow-bore liner is generally used for a splitless injection, and the wide-bore liner is generally used for a split injection. Due to the small internal volume

### *Installing a Capillary Column*

(0.3 mL) of the narrow-bore liner, you can prevent overfilling the liner with vapor (caused by solvent expansion upon injection) by limiting the amount of sample injected to 0.5  $\mu$ L.

The wide-bore liner is used for splitless injection volumes over 0.5  $\mu$ l since its internal volume is 1.25 mL. The sample size should be limited to a maximum of 2  $\mu$ L for hydrocarbon solvents and less than that for high-expansion solvents, such as water or CH<sub>2</sub>Cl<sub>2</sub>. Refer to Table 6-1.

If the wide-bore liner is used for splitless injection, the splitless sampling time (vent-on time) should be more than one minute. Also, lower initial oven temperatures may be required to give good resolution in the first few minutes after the solvent peak. The wide-bore liner should be used with columns having an i.d. of 0.32 mm or greater.

#### **Step 3. Pack the CAP Injector Liner with Quartz Wool.**

To wipe the syringe needle, we recommend packing a small amount of quartz wool in the top portion of the liner regardless of the liner type or injector mode used (for example, split or splitless). This packing assures that reproducible volumes are injected because it wipes the syringe needle every time the needle is inserted.

Remove the liner and replace the quartz wool packing on a regular basis, particularly if your samples contain nonvolatile components that could build up on the wool. This could cause adsorption of peaks of interest, tailing, and loss of sensitivity. Remove the wool with a small hook on the end of a thin wire, or blow it out using compressed air.

**NOTE:** *To avoid contaminating the quartz wool when packing the injection liner, wear vinyl, powder-free, disposable.*

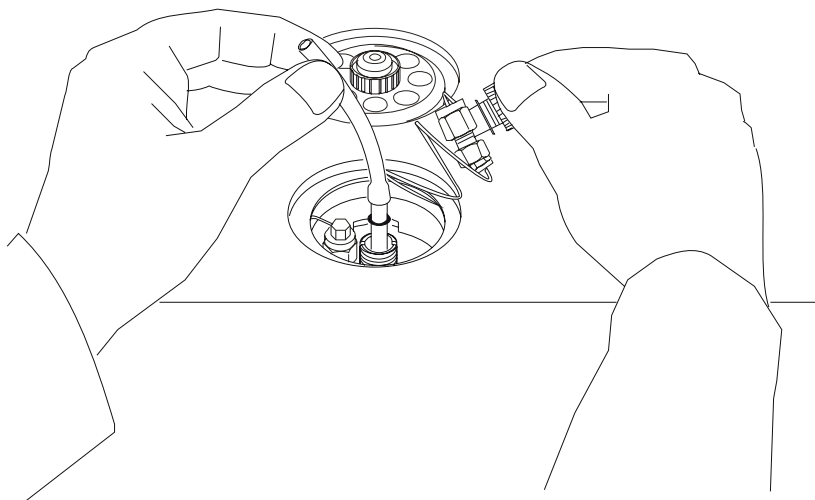
#### **Packing CAP Injector Liner for Split Operation**

Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the liner. Then using a 1/16-inch rod (P/N N610-T100), push the

quartz wool into the liner. Pack the wool tightly<sup>2</sup> from the dimple upwards (about one inch [2.5 cm]). Loosely pack quartz wool in the top portion of the liner to wipe the syringe needle upon injection.

### Packing a CAP Injector Liner for Splitless Operation

Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the liner. Then using a 1/16-inch rod (P/N N610-T100), push the quartz wool into the liner. Pack a one-inch piece (2.5 cm) of quartz wool *loosely* below the top ground portion of the liner (see Figure 6-11). The sample is then injected into the wool, thereby preventing the delivery of sample beyond the column. The wool also wipes the syringe needle upon injection.



**Figure 6-9. CAP injector liners packed with quartz wool.**

### **Step 4. Reinstall the Liner in the CAP Injector.**

To reinstall the liner:

1. Install a new O-ring near the ground portion of the liner.
2. Insert the liner in the injector body.

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<sup>2</sup> The recovery of high molecular weight components (e.g., C<sub>40</sub>) may be improved if the liner is loosely packed.

### Installing a Capillary Column

3. Place the septum purge assembly over the liner.
4. Press the septum purge assembly down to correctly position the liner in the injector.
5. Replace the threaded collar and tighten the assembly using the spanner (P/N N610-1359).

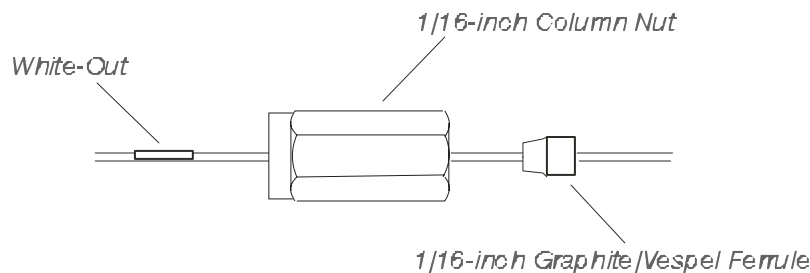
#### Step 5. Connect a Column to the CAP Injector.

#### CAUTION

*This injector terminates in a 1/16-inch fitting. This fitting is fragile. To preserve the integrity of the fitting, carefully connect the nut to prevent cross-threading the fitting and/or overtightening the nut on the fitting. You can also preserve the integrity of the fitting by allowing the injector to cool before connecting a nut.*

To connect a column:

1. Insert a 1/16-inch column nut (P/N 0990-3392) and 1/16-inch graphite ferrule (0.8 mm i.d., P/N 0992-0141 or 0.5 mm i.d., P/N 0990-3700) over one end of the column as shown below:

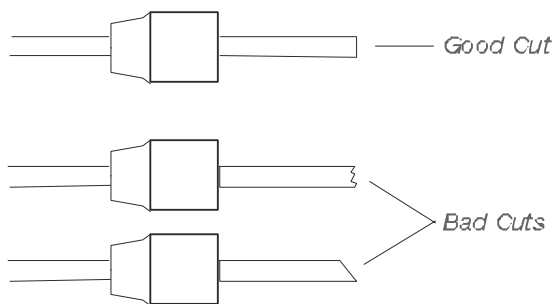


**Figure 6-10.** Narrow-bore capillary column, nut, and ferrule on the injector end of a column.

**NOTE:** *Verify that the tapered end of the ferrule is facing towards the nut as shown above.*

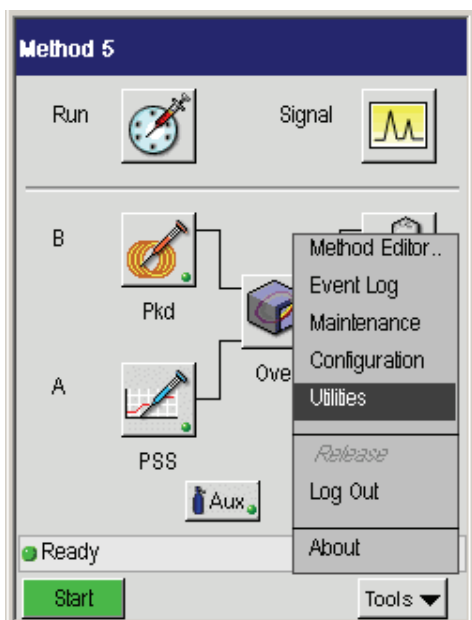
2. Cut off about 1 cm (3/8 inch) from the column end using a wafer scribe (P/N N930-1376, pkg. of 10 scribes). Break off the tubing at the score mark

so that the break is clean and square. Examine the cut with a magnifying glass and compare it to the examples shown in the following figure:



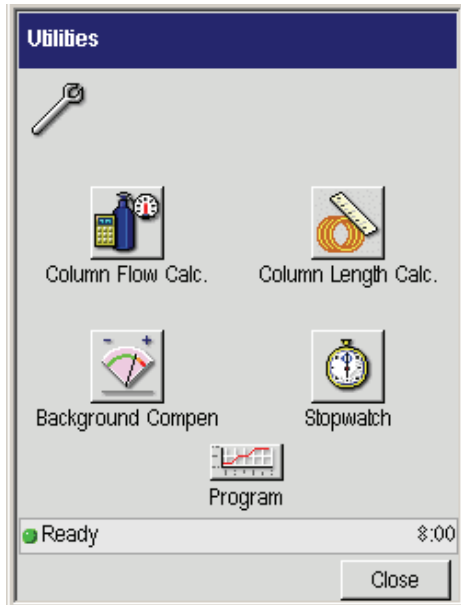
**Figure 6-11. Example of a good column cut and bad cuts.**

- To automatically calculate the position of the column nut on the column so that the back of the nut is the correct distance from the end of the column, go to the system Status Screen and touch the **Tools** button.



- Touch **Utilities** from the drop down menu. The **Utilities** screen appears.

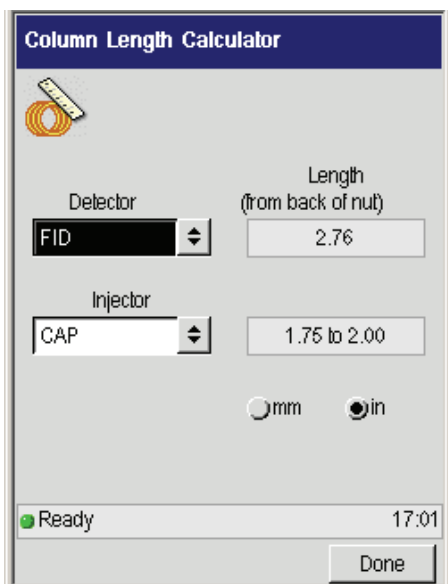
## Installing a Capillary Column



5. In the **Utilities** screen touch the **Column Length Calc** button



6. Using the drop down menu touch the type of **Detector** and **Injector** you are using. Select whether you want the calculation in **mm** or **inches**. The proper distance will be calculated automatically.

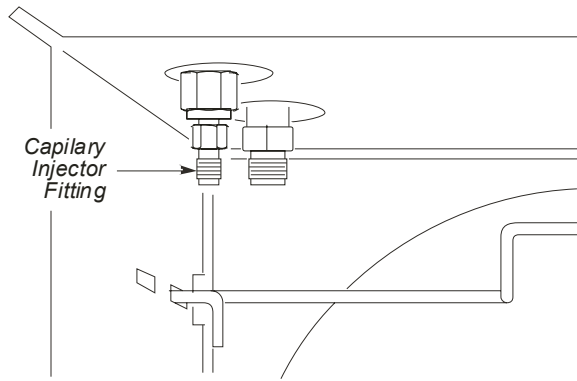


7. Using correction fluid (white-out) or a felt-tipped pen, make a mark on the column just beyond the back edge of the column nut (see Figure 6-10).

**CAUTION** *To avoid contaminating the system, make certain that the nut and ferrule do not contact the mark on the column.*

8. Locate the capillary injector fitting inside the GC oven.

## Installing a Capillary Column



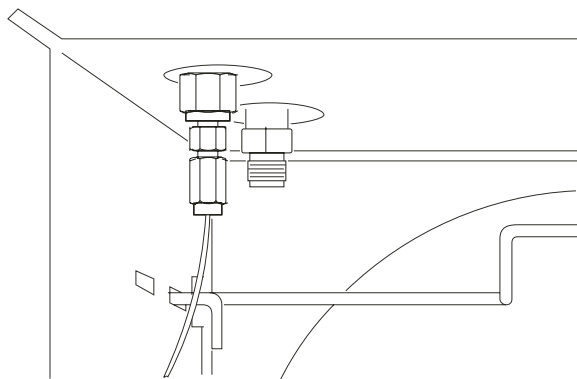
**Figure 6-12. Capillary injector fitting inside the oven.**

9. Insert the column into the capillary injector fitting. Then hand-tighten the column nut onto the capillary injector fitting. Insert the column into the capillary injector fitting until the mark is aligned with the back of the nut.
10. Using two 1/4-inch wrenches, tighten the column nut only until the column cannot be pulled out of the fitting.

**CAUTION**

*Do not overtighten column nuts. Overtightening can cause damage to the ferrule and/or column.*





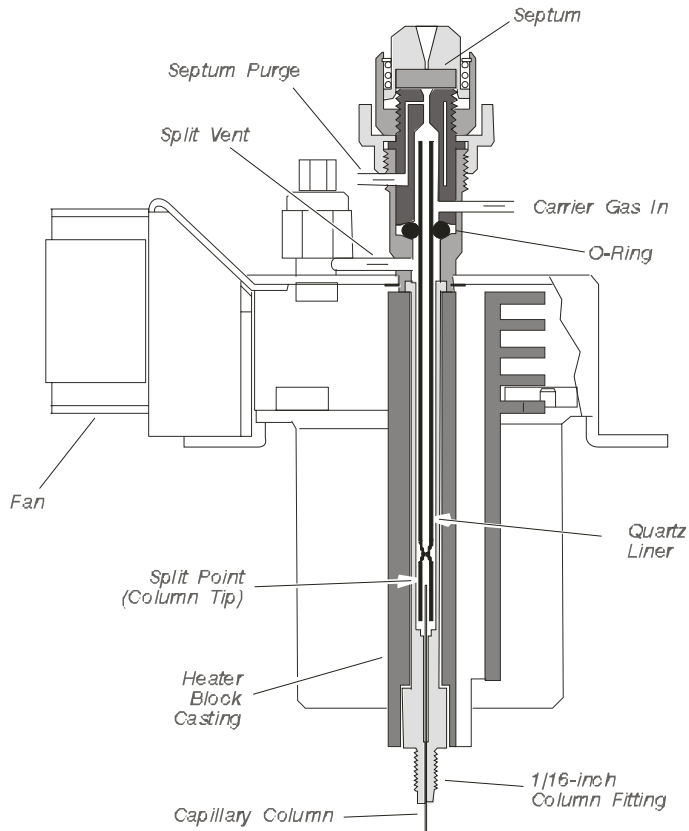
**Figure 6-13. Capillary column attached to capillary injector fitting.**

## **Step B2 Connect the Column to the Programmed Split/Splitless (PSS) Injector:**

### **Overview**

The Programmed Split/Splitless injector (PSS) consists of a septum purge assembly and the injector body. Carrier gas enters the injector at a point just above the O-ring and flows through the quartz liner past the column tip.

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**Figure 6-14. Cutaway view of the Programmed Split/Splitless injector (PSS).**

### **About the Injector Liners**

The PSS injector uses the following three quartz liners:

- 2.0-mm i.d. (wide-bore) liner (P/N N612-1004)
- 1-mm i.d. (narrow-bore) liner (P/N N612-1006)
- hourglass liner (N610-1539)

In general, for split or splitless injections, use the 2-mm or 1-mm i.d. liner and operate the PSS in the inlet-programmed mode. For on-column operation, use the

hourglass liner and the oven program mode. The 2-mm i.d. liner should be packed with quartz wool as described in this chapter and used for either split or splitless operation. The 1-mm i.d. liner may give better resolution of the early-eluting peaks in the split or splitless mode and it is better for labile compounds; however, it should be used for those samples with early-eluting peaks for which additional solute trapping focusing cannot be obtained by lowering the initial oven temperature or by using a column with a thicker stationary phase film.

To wipe the syringe needle, in all liner types (wide-bore or narrow-bore) or injection modes (split or splitless), we recommend packing a small amount of quartz wool in the top portion of the liner.

The sample is injected into the PSS injector at a "cool" temperature. The injector temperature is then programmed to increase. This is useful for samples that are thermally labile and/or have a wide molecular weight range. The PSS injector can also be used in a programmed on-column mode by replacing the quartz liner with the hourglass liner and closing the split vent flow.

**CAUTION**

*The PSS can be used in the "hot" split or splitless mode. This, however, is not recommended for use with the 1 mm i.d. liner; it could cause solvent flashback in the injector. This mode should be used with caution depending upon the solvent and temperatures you choose. See Table 6-1.*

**CAUTION**

*When using the PSS in the on-column mode with the autosampler, you must use a special syringe that has a needle o.d. of 0.47 mm (P/N N610-1253 or N610-1380). Refer the Active Method Chapter in the Clarus 500 Users Guide, "Controlling the Autosampler," for more detail. You must use only the "Norm" injection speed with this syringe in the on-column mode. The "Fast" injection speed will bend this thin needle; the "Slow" injection speed may produce double peaks due to the momentary stoppage of column flow during the longer injection. You can achieve better precision in the on-column mode when sample volumes of 1.0  $\mu$ L or greater are injected.*

### ***Split Injections***

In the split mode, the wide-bore quartz liner is packed with quartz wool to insure thorough mixing of the sample and carrier gas before they reach the column tip. The split vent is open at the time of injection so that a fraction of the sample mixture enters the column while the remainder is routed out through the split vent.

### ***Splitless Injections***

In the splitless mode, the narrow-bore quartz liner is typically used without tightly packed quartz wool for mixing. Instead, a small amount of quartz wool in the center is recommended. This is useful for trace analysis with smaller sample volumes (less than 1  $\mu\text{L}$ ). By closing the split vent, most of the sample mixture enters the column, then the split vent is opened to clear the injector inlet of residual solvent.

### ***Solvent Purge Injections***

In applications where the sample is in a trace concentration and has a high molecular weight, this sample can be injected into the PSS with the vent open and the starting temperature near the boiling point of the solvent. In this way, only the solvent is purged out through the vent, then the vent is closed and the injector temperature is programmed up to elute the peaks of interest onto the column. This technique allows larger quantities of sample to enter the column without large solvent effects.

### ***About the Pneumatics Control***

The pneumatics consists of PPC modules that regulate the inlet flow and pressure of the gases using software controlled by a Clarus 600 GC method.

#### **CAUTION**

*The PSS injector is shipped with the wide-bore liner installed but without the quartz wool packing. Before using the injector, remove the liner and pack it with quartz wool.*

Connect a column to the programmed split/splitless injector in either one of the following two modes:

- Split/Splitless Mode
- On-Column Mode using the hourglass liner

### ***Connect the Column to the PSS Injector***

The following five steps summarize how to connect a column to the PSS injector in the split/splitless mode:

- Step 1. Remove the PSS injector liner.
- Step 2. Select an appropriate PSS injector liner.
- Step 3. Pack the PSS injector liner with quartz wool.
- Step 4. Reinstall the liner in the PSS injector.
- Step 5. Connect a column to the PSS injector.

#### ***Step 1. Remove the PSS Injector Liner.***

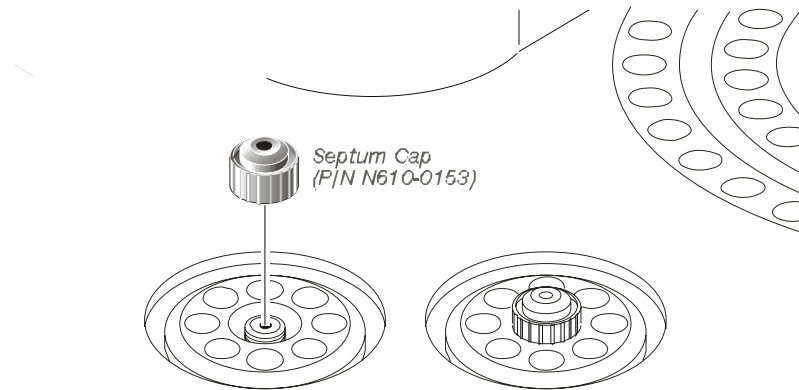
To remove the PSS injector liner:

1. Ensure that the injector heater has been turned off.

Allow the injector to cool until it is slightly warm to the touch. **Cooling the injector to too low a temperature (less than 80 °C) will make it difficult to remove the injector liner.**

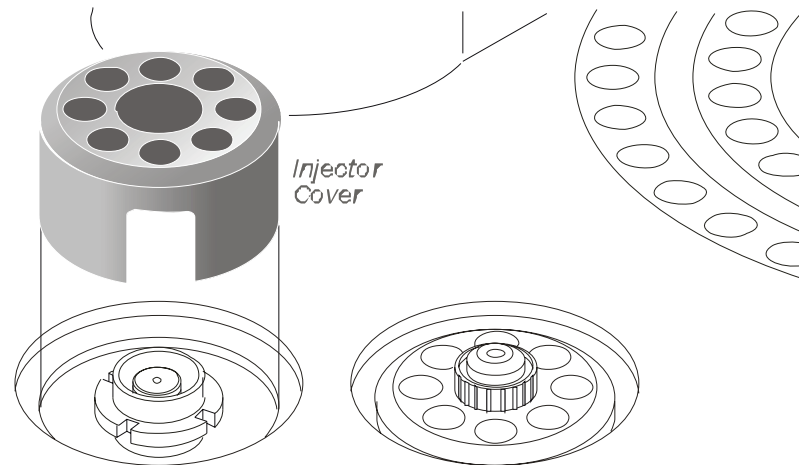
2. Remove the septum cap.

## Installing a Capillary Column



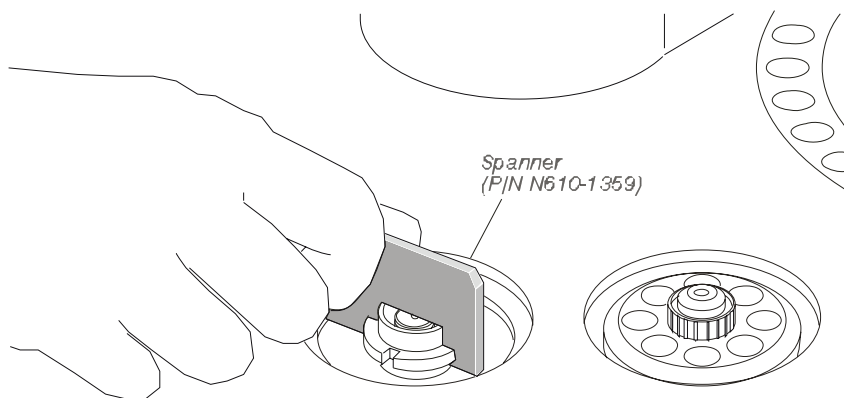
**Figure 6-15. Removing the septum cap.**

3. Remove the injector cover.



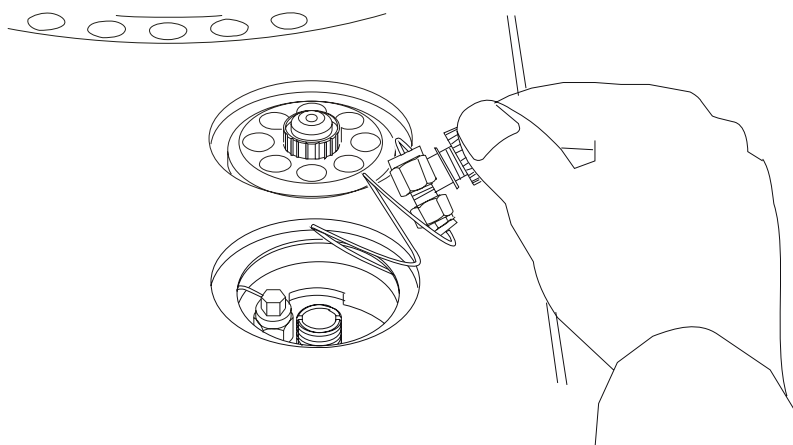
**Figure 6-16. Removing the injector cover.**

4. Loosen the threaded collar by using the spanner (P/N N610-1359) provided, then remove the threaded collar.



**Figure 6-17. Loosening the threaded collar.**

5. Replace the septum cap on the injector.
6. Pull the septum cap upwards to remove the septum purge assembly.



**Figure 6-18. Removing the septum purge assembly.**

The carrier gas inlet line is coiled to allow you to pull the septum purge assembly over to the side and gain access to the liner.

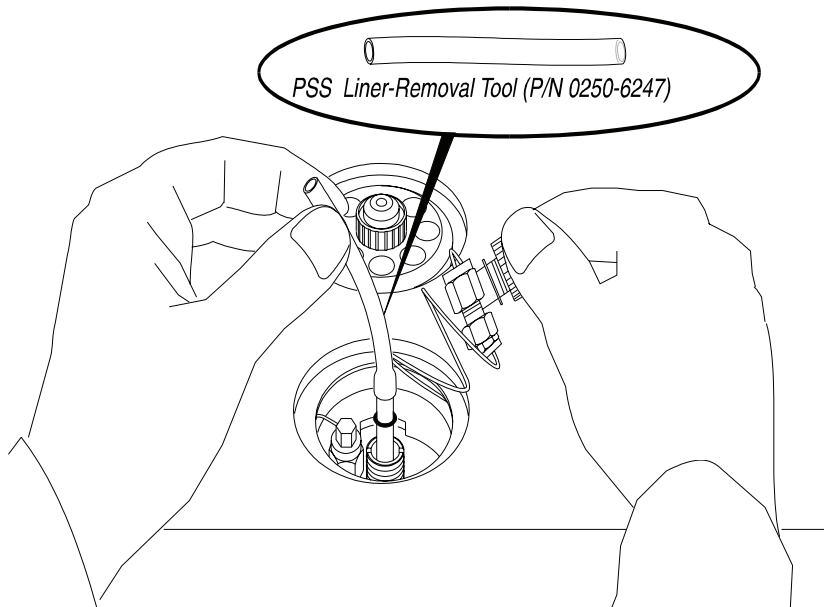
## Installing a Capillary Column

7. Ensure that the PSS injector liner is cool, then insert the end of the PSS liner-removal tool (P/N 0250-6247) over the end of the injector liner that is shipped with PSS injector. Remove the liner by lifting it up and out of the injector.

**The PSS liner must be cool (no hotter than 100 °C) or the liner-removal tool will melt!** The end of the PSS liner-removal tool may flare out with use. If this happens, cut off the flared end with a razor blade or scissors.

### CAUTION

*The PSS injector does not have a removable fitting at the bottom of the assembly. Be very careful when removing this liner to prevent breaking it. As the injector cools, the O-ring adheres to the metal base of the injector body. Use a small screwdriver to loosen the O-ring, then remove the liner and O-ring. Be careful not to scratch the barrel where the O-ring seals. Replace the O-ring with a new O-ring when you replace the liner.*



**Figure 6-19. Removing a PSS injector liner.**



**Step 2. Select an Appropriate PSS Injector Liner.**

The PSS injector uses the following three liners:

- 2-mm i.d. PSS injector wide-bore liner (P/N N612-1004)
- 1-mm i.d. PSS injector narrow-bore liner (P/N N612-1006)
- PSS injector on-column (hourglass) liner (P/N N610-1539)

The PSS injector is operated in the inlet-programmed mode for split or splitless injection with either the 2-mm or 1-mm i.d. liner. If used in the “hot” split or splitless mode, the 2-mm i.d. liner should be used. It is recommended that you do not inject more than approximately 0.5  $\mu\text{L}$  in the “Hot” mode. Refer to Table 6-1. For PSS on-column operation, use the hourglass liner and the oven-program mode. The 2-mm i.d. PSS liner that is used for either split or splitless operation should be packed with quartz wool as described in this chapter. The 1-mm i.d. PSS liner may give better early-eluting peak resolution in the split or splitless mode. This liner should be used for samples with early-eluting peaks that cannot be resolved by additional solute trapping/focusing (by lowering the initial oven temperature or by using a column with a thicker stationary-phase film).

**Step 3. Pack the PSS Injector Liner with Quartz Wool.****CAUTION**

*Never pack the hourglass liner with wool.*

To wipe the syringe needle, we recommend packing a small amount of quartz wool in the top portion of the liner regardless of the liner type (wide-bore or narrow-bore but never the hourglass) or injector mode (split or splitless). Quartz wool assures that reproducible volumes are injected by wiping the syringe needle every time it is inserted. Remove the liner and replace the quartz wool on a regular basis, particularly if your samples contain nonvolatile components that could build up on the wool. This could cause adsorption of peaks of interest,

## Installing a Capillary Column

tailing, and loss of sensitivity. Remove the wool with a small hook on the end of a thin wire, or blow it out using compressed air.

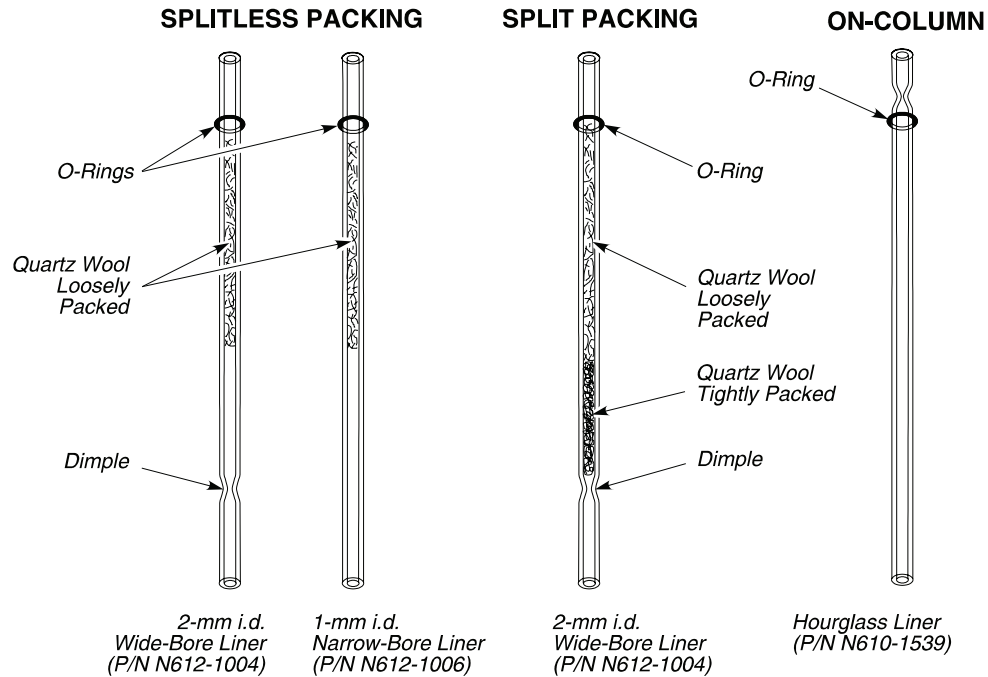


Figure 6-20. PSS liners packed with quartz wool.

## Packing a PSS Injector Liner for the Splitless Mode

### CAUTION

*To prevent contaminating the quartz wool when packing the injection liner, wear vinyl, powder-free, disposable gloves (for example, the same type of gloves used to perform maintenance on TurboMass).*

Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the liner. Then using a 1/16-inch o.d. rod (P/N N610-T100), push the quartz wool into the liner. Pack a 2.5-cm (one-inch)

piece of quartz wool *loosely* below the top ground portion of the liner (see Figure 6-21). The sample is then injected into the wool, thereby preventing the delivery of sample beyond the column. The wool also wipes the syringe needle upon injection.

**NOTE:** *The narrow-bore liner is more difficult to pack because of its small i.d. However, there is a dimple in the middle of the liner to hold the wool in place. Do not pack the wool too tightly!*

When the narrow-bore liner is installed, use a 5-cm needle-length syringe when making manual injections.

#### **Packing PSS Injector Liner for the Split Mode**

##### **CAUTION**

*To prevent contaminating the quartz wool when packing the injection liner, wear vinyl, powder-free, disposable gloves.*

Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the liner. Then using a 1/16-inch o.d. rod (P/N N610-T100), push the quartz wool into the liner. Pack the wool tightly<sup>3</sup> from the dimple upwards (about one inch [2.5 cm]). Loosely pack quartz wool in the top portion of the liner to wipe the syringe needle upon injection.

**NOTE:** *As you can see in Figure 6-20, each liner has an O-ring installed on the part furthest away from the dimple on the PSS injector liner. If the O-ring has adhered to the liner, you may not be able to easily remove the liner. Use a small screwdriver to dislodge the O-ring before removing the liner and O-ring. Be careful not to scratch the barrel where the O-ring seals.*

#### **Step 4: Reinstall the Liner in the PSS Injector.**

To reinstall the liner:

---

<sup>3</sup> The recovery of high molecular weight components (e.g., C<sub>40</sub>) may be improved if the liner is loosely packed.

### *Installing a Capillary Column*

1. Install a new O-ring on the top portion of the liner.
2. Insert the liner in the injector body.
3. Place the septum purge assembly over the liner.
4. Press the septum purge assembly down to correctly position the liner in the injector.

Make sure that you ***tightly secure*** the septum purge assembly to the injector base with the spanner.

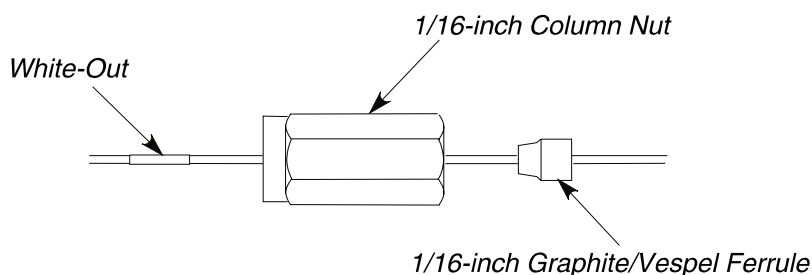
### **Step 5. Connect a Column to the PSS Injector.**

To connect a column:

1. Insert a 1/16-inch column nut (P/N 0990-3392) and 1/16-inch graphite ferrule (0.8 mm i.d., P/N 0992-0141 or 0.5 mm i.d., P/N 0990-3700) over one end of the column as shown below:

#### **CAUTION**

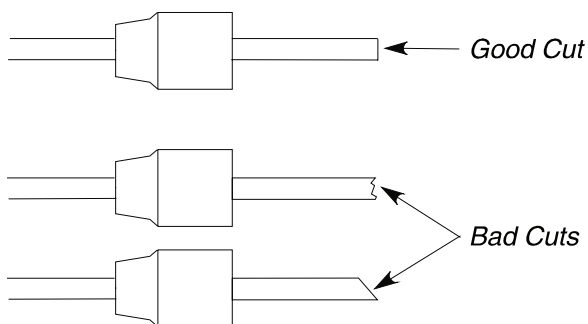
*This injector terminates in a 1/16-inch fitting. This fitting is fragile. To preserve the integrity of the fitting, carefully connect the nut to prevent cross-threading the fitting and/or overtightening the nut on the fitting. You can also preserve the integrity of the fitting by allowing the injector to cool before connecting a nut.*



**Figure 6-21. Column, nut, and ferrule on the injector end of a narrow-bore capillary column.**

**NOTE:** Make certain that the tapered end of the ferrule (in Figure 6-21) is facing towards the nut.

2. Cut off about 1 cm (3/8 inch) from the column end using a wafer scribe (P/N N930-1376, pkg. of 10 scribes). Break off the tubing at the score mark so that the break is clean and square. Examine the cut with a magnifying glass and compare it to Figure 6-22.

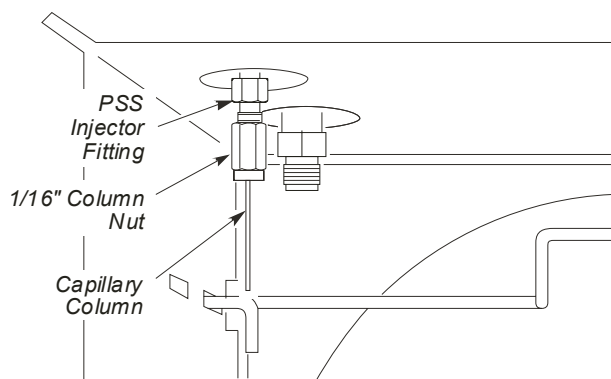


**Figure 6-22. Example of a good cut and bad cuts.**

3. Position the column nut on the column so that the back of the nut is 3.8 cm to 4.4 cm (1 1/2 to 1 3/4 inches) from the end of the column.
4. Using typewriter "white-out" or a felt tipped pen, make a mark on the column just beyond the back edge of the column nut (see Figure 6-23).

**CAUTION**

*To avoid contaminating the system, make certain that the nut and ferrule do not come into contact with the mark on the column.*



**Figure 6-23.** Capillary column attached to PSS injector fitting.

***Connect the Column to the PSS Injector in the On-Column Mode Using the Hourglass Liner***

The on-column mode is not recommended for use with columns having an internal diameter of 0.1 mm. This is due to the flow differences between the pre-column (0.53 mm i.d.) and the column.

The following three steps summarize how to connect a column to the PSS injector in the on-column mode:

- Step 1. Remove the PSS split/splitless injector liner.
- Step 2. Install the hourglass liner in the PSS injector.
- Step 3. Connect a column to the PSS injector.

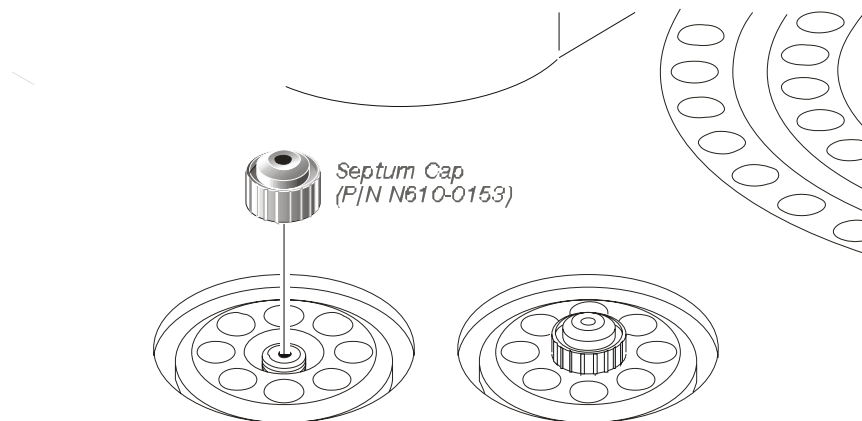
**Step 1. Remove the PSS Split/Splitless Injector Liner.**

To remove the PSS injector liner:

1. Ensure that the injector heater has been turned off.

Allow the injector to cool until it is slightly warm to the touch. **Cooling the injector to too low a temperature (less than 80 °C) will make it difficult to remove the injector liner.**

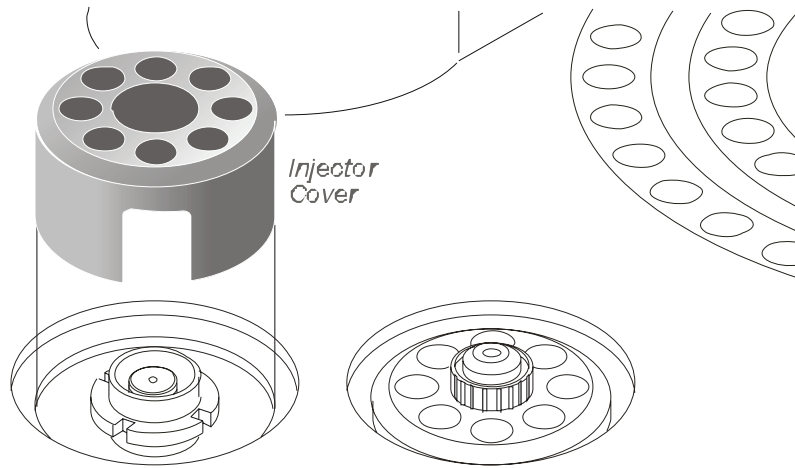
2. Remove the septum cap.



**Figure 6-24. Removing the septum cap.**

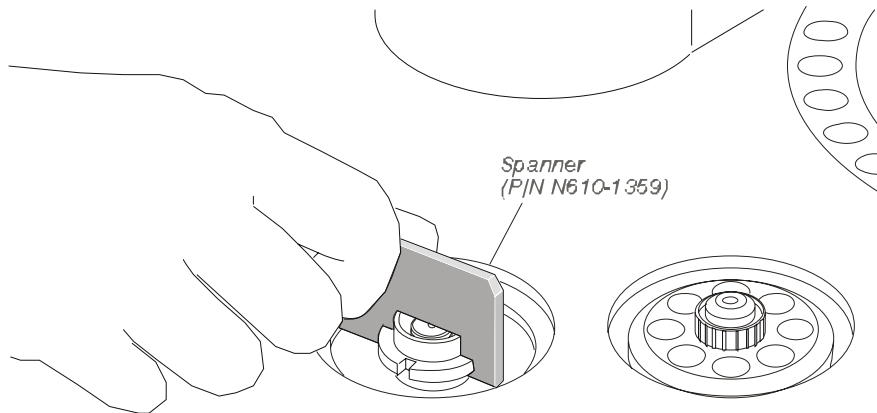
3. Remove the injector cover.

## Installing a Capillary Column



**Figure 6-25. Removing the injector cover.**

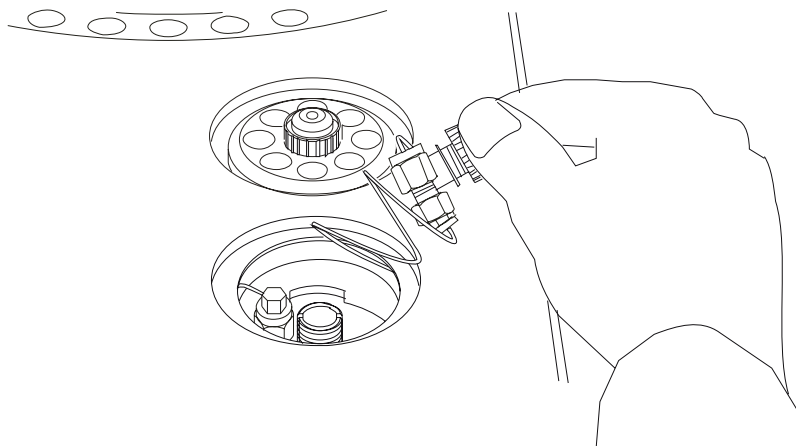
4. Loosen the threaded collar using the spanner (P/N N610-1359) provided, then remove the threaded collar.



**Figure 6-26. Loosening the threaded collar.**

5. Replace the septum cap on the injector.
6. Pull the septum cap upwards to remove the septum purge assembly.





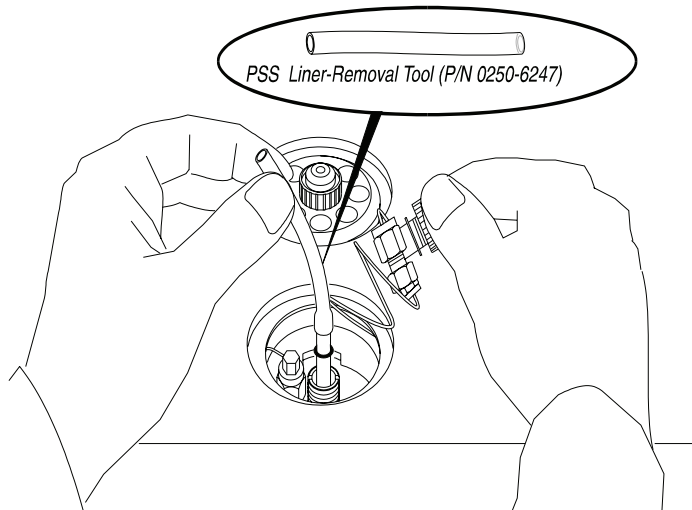
**Figure 6-27. Removing the septum purge assembly.**

The carrier gas inlet line is coiled to allow you to pull the septum purge assembly over to the side and gain access to the liner.

7. Ensure that the liner is cool, then insert the end of the PSS liner-removal tool (P/N 0250-6247) over the end of the wide-bore or narrow-bore PSS injector liner. Remove the liner by lifting it up. Gently probe the O-ring if it sticks to the injector body.

**The PSS liner must be cool (no hotter than 100 °C) or the tubing will melt!** The end of the PSS liner-removal tool may flare out with use. If this happens, cut off the flared end with a razor blade or scissors.

## Installing a Capillary Column



**Figure 6-28. Removing an injector liner.**

### **Step 2. Install the Hourglass Liner in the PSS Injector.**

To install the hourglass liner:

1. Replace the existing liner with the hourglass liner. Install the liner with the hourglass end closest to the septum and use a new O-ring.



**Figure 6-29. Hourglass liner (P/N N610-1539).**

2. Replace the septum purge assembly removed previously. **DO NOT** secure it with the collar at this time.

We recommend the following procedure to connect a column and pre-column to the universal connector. You may find that changing the order of the steps is more convenient for you. However, the critical concerns are:

- Making straight, clean, even cuts on the column/pre-column.
- Wetting the ends of the column/pre-column.
- Wetting the fused-silica universal connector/hourglass needle guide.
- GENTLY twisting the column/pre-column into the hourglass fused-silica universal connector.
- Conditioning the mechanical joint (this is critical).

In the on-column mode, you will install a column (0.53 mm i.d.) or a deactivated fused silica pre-column (0.53 mm i.d.) in the injector body. This will enable the sample to be deposited directly into the column or the pre-column.

### **Step 3. Connect a Column to the PSS Injector**

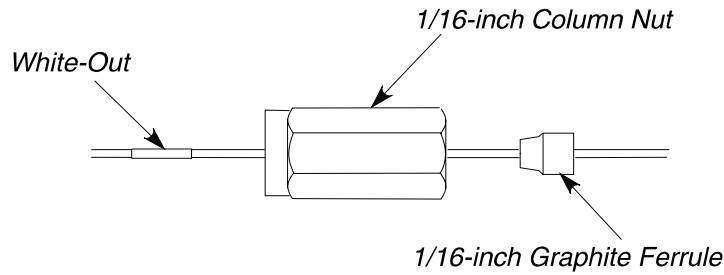
To connect a column to the PSS in the on-column mode using the hourglass liner:

1. Insert a 1/16-inch column nut (P/N 0990-3392) and 1/16-inch graphite ferrule (P/N 0992-0141) over the other end of the 0.53-mm i.d. column or pre-column as shown below:

#### **CAUTION**

*If you are using a 0.53-mm i.d. column, insert it directly into the injector. If you are using a column with an i.d. less than 0.53 mm, insert a one-meter piece of deactivated 0.53-mm i.d. fused silica (pre-column) into the injector. This pre-column will also serve as a retention gap. In addition, the long length of this tubing will enable you to change columns and still have enough of the pre-column to make numerous new column connections. Connect a column to the end of this pre-column (in the oven) with a universal connector (P/N N930-2149).*

### Installing a Capillary Column



**Figure 6-30.** Column, Nut, and Ferrule on the injector end of a narrow-bore capillary column.

**CAUTION**

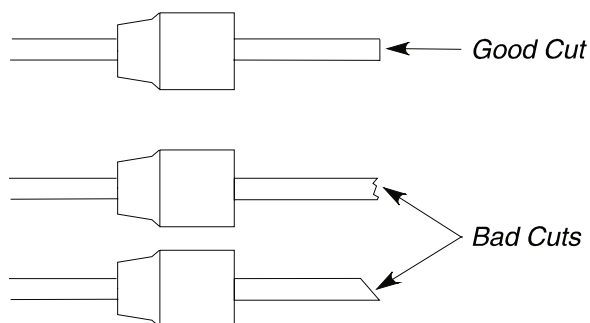
*Ensure that the tapered end of the ferrule is facing towards the nut as shown above.*

*We have found that graphite/Vespel ferrules used to connect the column to the injector fitting loosen after the injector temperature cycles several times. Use graphite ferrules to eliminate this problem.*

2. Cut off about 1 cm (3/8 inch) from the column end using a wafer scribe (P/N N930-1376, pkg. of 10 scribes) or other column cutting tool. Break off the tubing at the score mark so that the break is clean and square. If you are using a 0.53-mm i.d. pre-column, make a clean square cut on both ends. Examine the cut with a magnifying glass and compare it to the following figure:

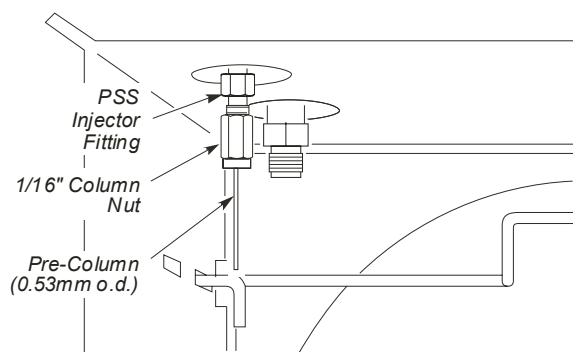
**CAUTION**

*A clean, square cut is especially critical to produce a leak-free seal in the fused-silica universal connector.*



**Figure 6-31. Example of a good cut and bad cuts.**

3. Locate the PSS injector fitting inside the oven.



**Figure 6-32. PSS injector fitting in the column oven.**

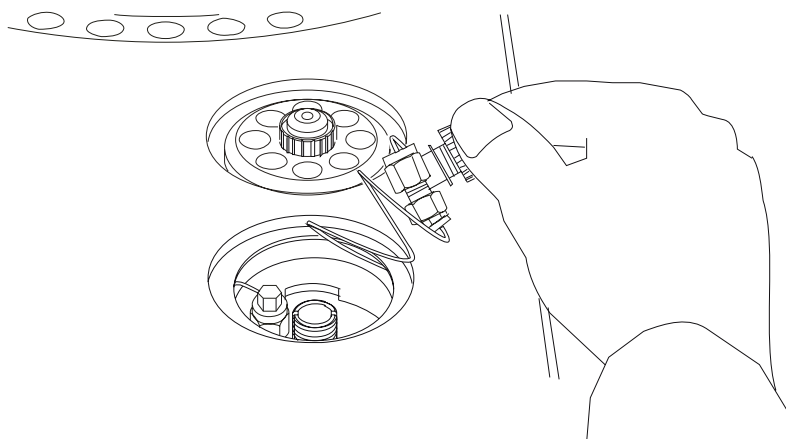
4. Insert the 0.53-mm i.d. column or pre-column into the PSS injector fitting. Tighten the column nut so the column/pre-column can still be moved but with some resistance.
5. Push the column/pre-column up into the injector until it can not go any further.

You may notice the septum purge assembly move upward.

6. Mark the column with typewriter "white out" just below the column nut.

## Installing a Capillary Column

7. Push the septum purge assembly down into position, place the threaded collar on the assembly, and tighten the threaded collar with the spanner provided.



**Figure 6-33. Replacing the septum purge assembly.**

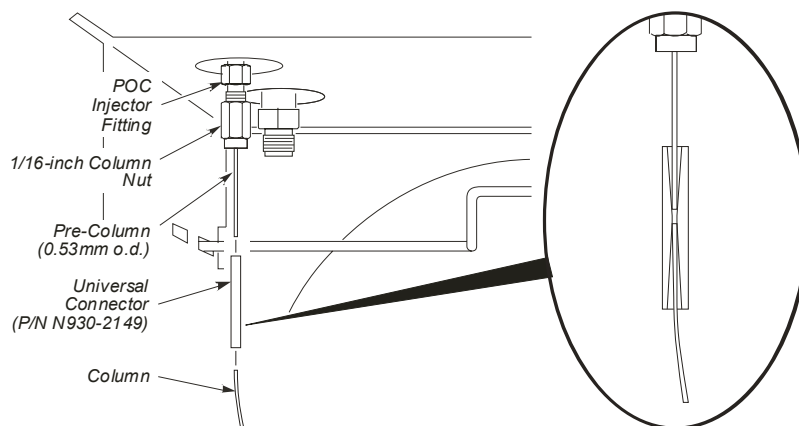
8. Using a 1/4-inch wrench, tighten the fitting until the column cannot be pulled out of the fitting.

**CAUTION**

*Do not overtighten column nuts. Overtightening can cause damage to the ferrule and/or column.*

9. Replace the septum cap but do not tighten it. Manually insert the 0.47-mm o.d. syringe needle into the 0.53-mm i.d. column/pre-column. With the syringe still inserted in the injector, tighten the septum cap completely. This aligns the column and septum cap to ensure a smooth movement of the syringe through the septum cap and into the 0.53-mm i.d. column/pre-column.

10. If your column i.d. is smaller than 0.53 mm, connect your column to the 0.53-mm i.d. pre-column as follows:
  - a) Make a clean, even cut on the end of your column and examine it with a magnifying glass.
  - b) Drop some solvent (for example, methanol) into both sides of the fused-silica universal connector (P/N N930-2149, package of 5).
  - c) Wet a tissue with solvent and wipe the end of the column.
  - d) Insert the column end into the universal connector and *gently* twist it. The column should seal in the universal connector.
11. Wet a tissue with solvent (for example, methanol) and wipe the end of the pre-column.
12. Carefully push the universal connector, with the attached column, up into the end of the pre-column. *Gently* twist the universal connector to seal the connection between pre-column and universal connector. This connection is called the mechanical joint.



**Figure 6-34.** Connecting a column and universal connector to the pre-column to create the mechanical joint.

**CAUTION**

*Typically the split vent is closed in this mode of operation.*

*Condition the mechanical joint between the pre-column and column prior to making any analytical runs. Condition the joint by slowly temperature programming the oven up to 200 °C and holding it at 200 °C for one hour. Do not exceed the recommended temperature of your column. This can be incorporated when you condition the column, see Step E in this Chapter.*

*If you replace the column, do not reuse the universal connector. Cut the column and pre-column from the universal connector. Replace the universal connector with a new one. The pre-column ought to be long enough so that you can reuse it.*

*If you replace the pre-column, it will also require some conditioning. If the entire installation is new, the column conditioning procedure will condition the pre-column, the column, and the mechanical joint. If you are just replacing the pre-column, you can condition it and the mechanical joint by the above procedure. If a high background persists with a fresh pre-column, condition the pre-column by only increasing the injector temperature for one hour.*

**CAUTION**

*When using the PSS injector in the on-column mode or with the autosampler, you must use a special syringe that has a needle o.d. of 0.47 mm (P/N N610-1253 or N610-1380). Refer to Chapter 9 for more details. You must use only the "Norm" injection speed with this syringe when you are in the on-column mode. The "Fast" injection speed will bend this thin needle and the "Slow" injection speed may produce peak break up or distorted peaks. You can achieve better precision in the on-column mode when sample volumes of 1.0 µL or greater are injected.*



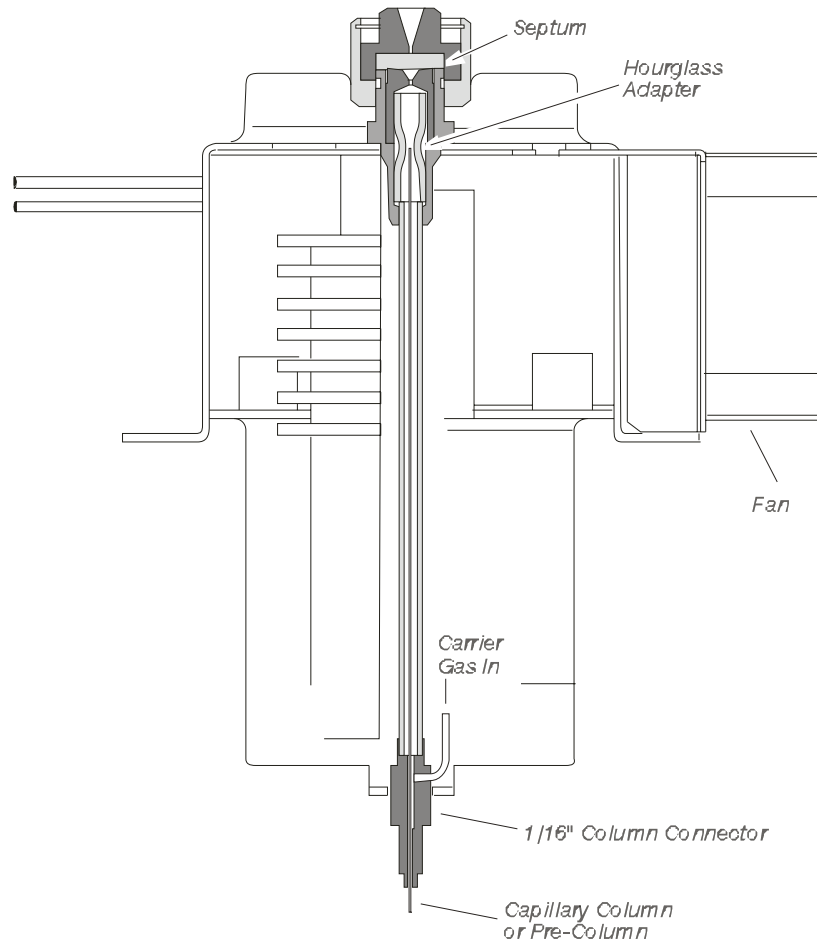
## **Step B3 Connect the Column to the Programmed On-Column Injector (POC):**

### **Overview**

The Programmed On-Column injector (POC) consists of an hourglass adapter, a deactivated 0.53-mm i.d. fused-silica pre-column, and a fused-silica universal connector. The sample is injected into the POC at a "cool" temperature; the injector temperature is then programmed to increase. This is helpful for samples that are thermally labile and/or of a wide molecular weight range. This injector is used only for trace analysis or diluted solutions.

<p><b>CAUTION</b>      <i>Only use a syringe that has a 0.47-mm o.d. needle (P/N N610-1380) with this injector.</i></p>
---

## Installing a Capillary Column



**Figure 6-35. Cutaway view of a Programmed On-Column injector (POC).**

### ***About the Pneumatics Control***

The POC pneumatics consist of a PPC (programmable pneumatics controlled) modules that regulate the inlet flow and pressure of the gases using software controlled by a Clarus 600 GC method.

**CAUTION**

*This injector terminates in a 1/16-inch fitting. When connecting a nut to this fitting, take special care not to crossthread the fitting or overtighten the nut..*

On-column injection is not recommended for columns with an internal diameter of 0.1 mm. This is because of flow differences between the pre-column (0.53 mm) and the column.

**CAUTION**

*If you have ordered the POC without the flow readout option, measure the carrier gas flow before connecting the column to the POC. Proceed to: C2 Setting the Carrier Gas for manual pneumatics and follow the procedure "Set the Carrier Gas Flow for a POC injector using the Soap Bubble Flowmeter" in this Chapter now!*

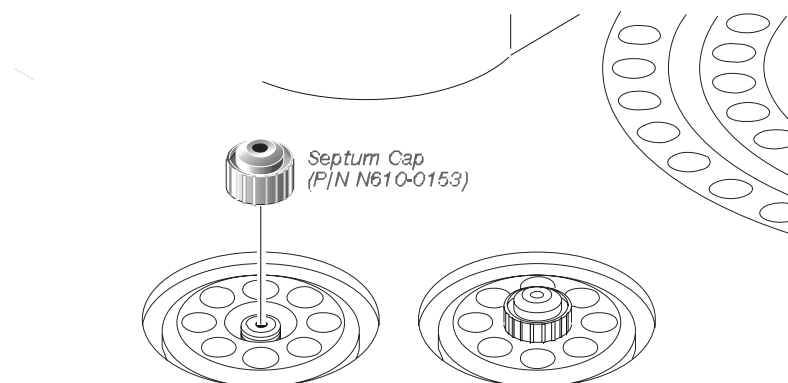
We recommend that you use the following procedure to connect a column and pre-column with the universal connector. You may find that changing the order of the steps is more convenient for you. However, the critical concerns are:

- Making straight, clean, even cuts on the column/pre-column.
- Wetting the ends of the column/pre-column with solvent.
- Wetting the universal connector/hourglass needle guide with solvent.
- GENTLY twisting the column/pre-column into the hourglass universal connector.
- Conditioning the mechanical joint (this is critical).

## Installing a Capillary Column

To connect a column to the programmed on-column injector (POC):

1. Remove the septum cap.



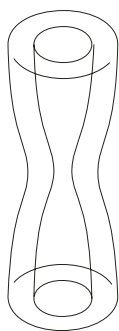
**Figure 6-36. Removing a septum cap.**

2. Remove the septum shield (P/N N610-1702) with the large end of the liner-removal tool (P/N N610-0102).



**Figure 6-37. Liner-removal tool (P/N N610-0102).**

3. Remove the hourglass needle guide (P/N N610-1703) with a pair of small pliers or tweezers.

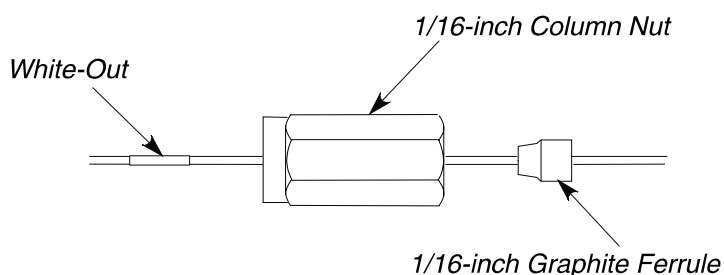


**Figure 6-38. Hourglass needle guide.**

**CAUTION**

*If you are using a 0.53-mm i.d. column, insert it directly into the injector. If you are using a column with an i.d. of less than 0.53 mm, insert a one-meter piece of deactivated 0.53-mm i.d. fused silica into the injector to act as a pre-column. This pre-column will also serve as a retention gap. In addition, the long length of this tubing will enable you to change columns and still have enough of the pre-column to make numerous new column connections. Connect a column to the end of this pre-column (in the oven) with a universal connector (P/N N930-2149).*

4. Insert the column nut (P/N 0990-3392) and ferrule (P/N 0992-0141) on the end of the column/pre-column.



**Figure 6-39. Column, nut, and ferrule on the injector end of a narrow-bore capillary column.**

**CAUTION**

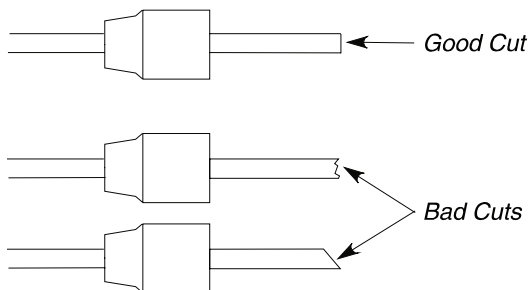
*We have found that graphite/Vespel ferrules used to connect the column to the injector fitting loosen after the injector temperature cycles several times. Use graphite ferrules to eliminate this problem. Make certain that the tapered end of the ferrule is facing towards the nut as shown above.*

## Installing a Capillary Column

5. Make a clean, square cut on the end of the 0.53-mm column or on both ends of the 0.53-mm pre-column. Wipe the end with a tissue soaked in methanol to remove fragments of polyimide or silica.

### CAUTION

*A clean, square cut is especially critical to obtain a leak-free seal in the hourglass needle guide and fused-silica universal connector.*



**Figure 6-40.** Example of a clean column cut and bad cuts.

6. Locate the injector fitting in the oven. Then insert the column/pre-column until it protrudes out the septum end of the injector.
7. Thread the nut onto the injector fitting. Then tighten it so that the column/pre-column can still be moved, but you feel some resistance.
8. Push the column/pre-column into the hourglass needle guide and twist it slightly.
9. Place the metal septum shield over the hourglass needle guide and push it into the injector until it is completely seated. Replace the septum cap.
10. Tighten the column nut inside the oven until the column cannot be pulled out of the fitting.

**CAUTION**

*Do not overtighten column nuts. Overtightening can cause damage to the ferrule and/or column.*

You may want to mark the column, just behind the nut, with typewriter "white-out." The mark will act as a guide when you test to see if the fitting is tight enough.

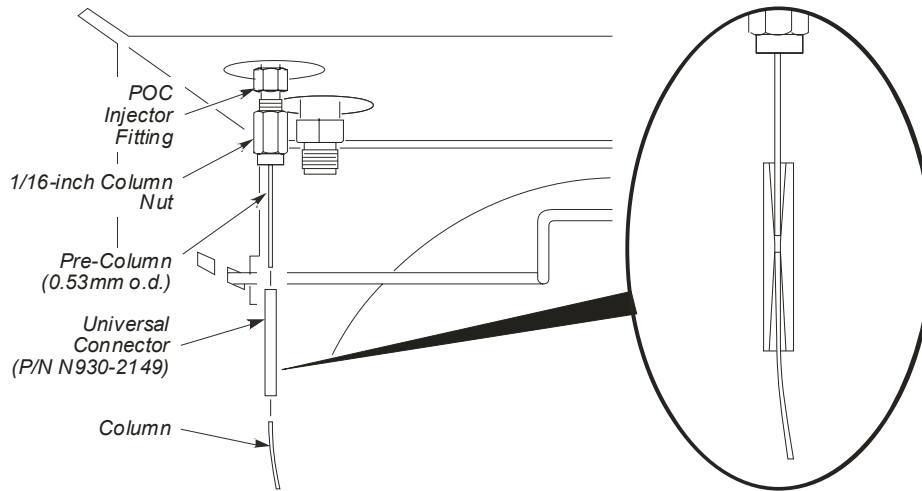
**CAUTION**

*To avoid contaminating the system, make certain that the nut and ferrule do not contact the mark on the column.*

11. Loosen the septum cap and manually insert the 0.47-mm o.d. syringe needle into the column/pre-column. With the syringe in the injector, tighten the septum cap completely. This ensures smooth movement of the syringe through the septum cap and into the column/pre-column.
12. If your column i.d. is smaller than 0.53 mm, connect your column to the 0.53-mm i.d. pre-column as follows:
  - a) Make a clean, even cut on the end of your column and examine it with a magnifying glass.
  - b) Drop some solvent (for example, methanol) into both sides of the fused-silica universal connector (P/N N930-2149, package of 5).
  - c) Wet a tissue with solvent and wipe the end of the column.
  - d) Insert the column end into the universal connector and **gently** twist it. The column should seal in the universal connector.
13. Wet a tissue with solvent (for example, methanol) and wipe the end of the pre-column.

## Installing a Capillary Column

14. Carefully push the universal connector, with the attached column, up into the end of the pre-column. **Gently** twist the universal connector to seal the connection between the pre-column and universal connector. This connection is called the mechanical joint.



**Figure 6-41.** Connecting the column and universal connector to the pre-column to create the mechanical joint.



**CAUTION**

*Condition the mechanical joint between the pre-column and column prior to making any analytical runs. Condition the joint by slowly temperature programming the oven up to 200 °C and holding it at 200 °C for one hour. Do not exceed the recommended temperature of your column. This can be incorporated when you condition the column, see Step E in this Chapter.*

*If you replace the column, do not reuse the universal connector. Cut the column and pre-column from the universal connector. Replace the universal connector with a new one. The pre-column ought to be long enough so that you can reuse it.*

*If you replace the pre-column, it will also require some conditioning. If the entire installation is new, the column conditioning procedure will condition the pre-column, the column, and the mechanical joint. If you are just replacing the pre-column, you can condition it and the mechanical joint by the above procedure. If a high background persists with a fresh pre-column, condition the pre-column by only increasing the injector temperature for one hour*

**CAUTION**

*When using the POC with the autosampler, you must use a special syringe that has a needle o.d. of 0.47 mm (P/N N610-1253 or N610-1380). Refer to Chapter 9 for more detail. You must only use the "Norm" injection speed with this syringe in the on-column mode. The "Fast" injection speed will bend this thin needle and the "Slow" injection speed may produce peak break up or distorted peaks. You can achieve better precision in the on-column mode when injecting sample volumes of 1.0 mL or greater.*

## **Step C: Set the Carrier Gas**

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This step describes how to set the carrier gas for PPC modules

### **Setting the Carrier Gas for PPC Modules**

The following procedure describes how to:

- Set the Carrier Gas Pressure for the Split/Splitless Injector (CAP) and Programmed Split/Splitless Injector (PSS)
- Set the Carrier Gas Flow for the Programmed On-Column Injector (POC)

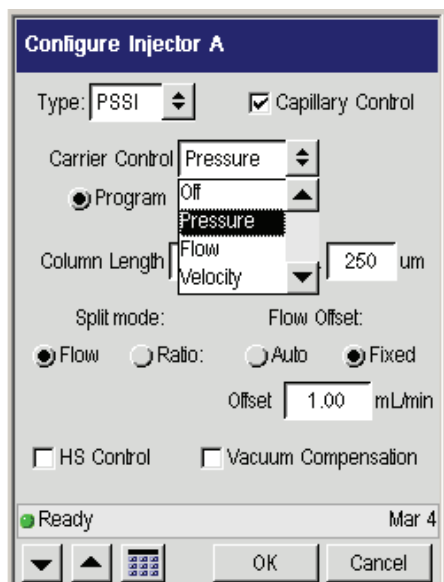
#### **Setting the Carrier Gas Pressure for the Split/Splitless Injector (CAP) and Programmed Split/Splitless Injector (PSS)**


**NOTE:** *The line pressure should be at least 15 psi greater than the highest carrier gas pressure setting, up to a maximum of 100 psig.*

To set the carrier gas pressure:

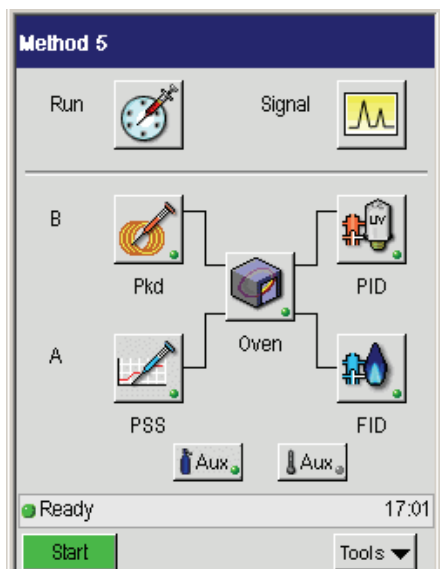
1. Turn on the carrier gas at the tank. Then adjust the line pressure to 60 - 90 psig.
2. Since a capillary injector is in position A, and Capillary Control is selected the **Carrier Control** drop down menu in the **Configuration** screen allows you to select **Pressure**, **Flow**, or **Velocity**.

The following screen show **Pressure** selected.

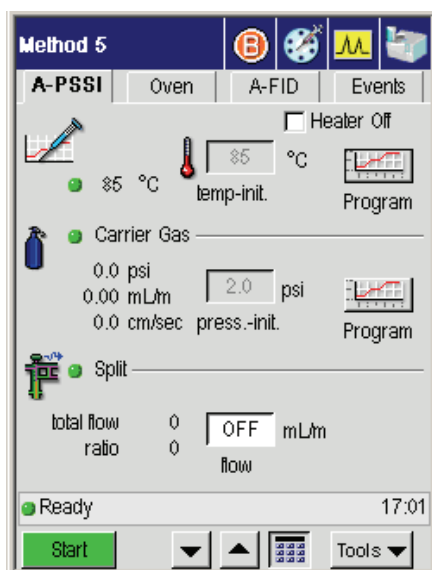





- From the System Status screen, select either the A or B injector (the example here shows the A - PSS injector ready for setting the carrier gas). In this example, touch the Channel A PSS injector button .

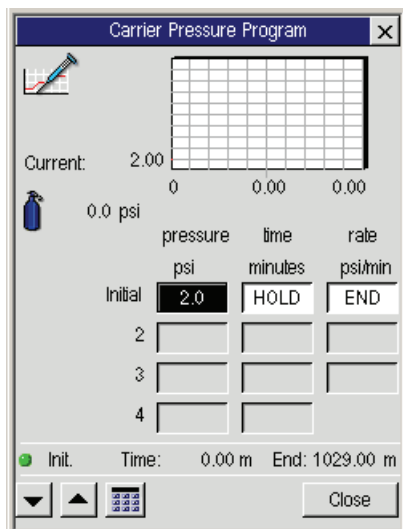
## Installing a Capillary Column






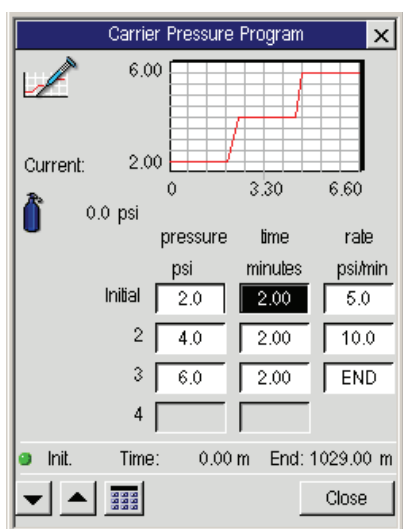
4. In the following window touch the **Program** button in the **Carrier Gas** field.



5. Using the up and down arrow or keypad    buttons enter an Initial pressure (0.1 to 75 psig). The following screen shows **2.0 psi**.



6. Using the up and down arrow or keypad    buttons enter the appropriate values. Once entered select **Close** to exit.



**NOTE:** The pressure readout display is factory configured to display the actual pressure as psig.

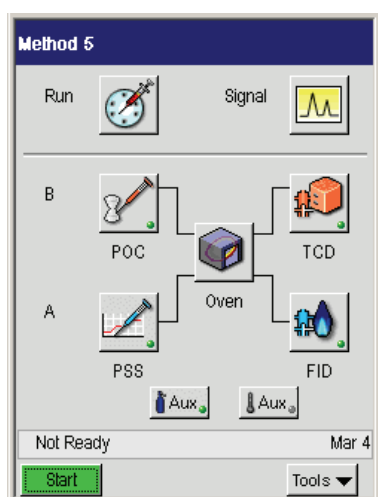
## Installing a Capillary Column

7. Enter a pressure set point. (See Suggested Capillary Column Pressures on Table 6-5.).

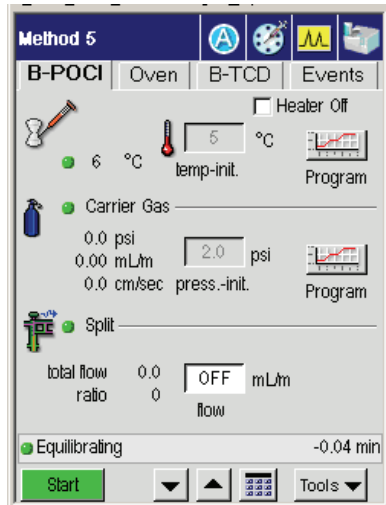
### **Setting the Carrier Gas Flow for the Programmed On-Column Injector (POC)**

To set the carrier gas flow:


1. Turn on the carrier gas at the tank. Adjust the line pressure to 60 - 90 psig.
2. From the System Status screen select either **A** or **B** injector (the example here shows the **B** injector ready for setting the carrier gas).

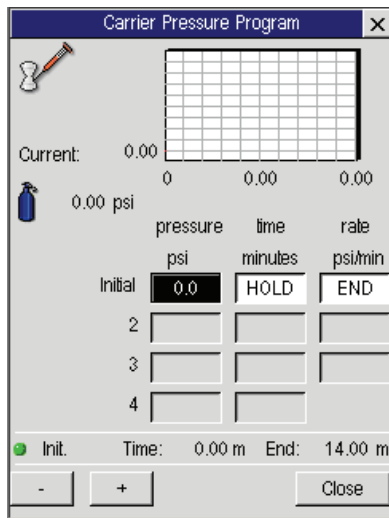


3. In the following window touch the **Program** button in the **Carrier Gas** field.



The Carrier Pressure Program screen appears.

- Using the up and down arrow or keypad  buttons enter a pressure (0.1 to 100 psig).



**NOTE:** If you wish to generate a flow program method, refer to the *Clarus 600 User's Guide* (P/N 0993-6780).

## Suggested Capillary Column Pressures:

Table 6-2. Calculated Pressure Drops (psig) for 10m Column<sup>4</sup>

$\bar{u}$ <sup>5</sup>	Column I.D. (μm)		
	320	250	100
10	1.0	2.4	10.0
20	2.1	4.9	21.2
30	3.1	7.3	31.8
40	4.1	9.8	42.3
60	6.2	14.6	63.5
80	8.3	19.5	84.7

Table 6-3. Calculated Pressure Drops (psig) for 25m Columns<sup>4</sup>

$\bar{u}$ <sup>5</sup>	Column I.D. (μm)		
	320	250	100
10	2.6	6.1	26.5
20	5.2	12.2	52.9
30	7.8	18.3	79.4
40	10.3	24.4	-
60	15.5	36.6	-
80	20.7	48.8	-

<sup>4</sup> In psig, using helium as a carrier gas at 100 °C.

<sup>5</sup> Average linear velocity (cm/sec).



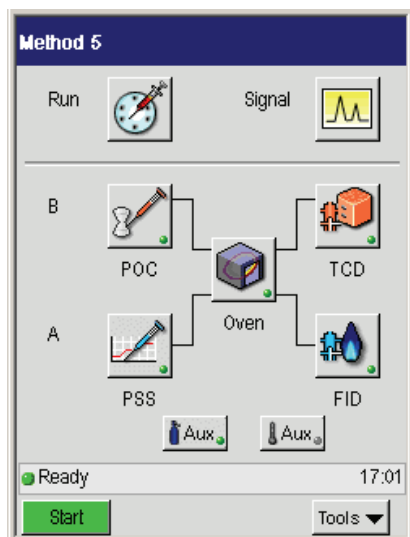
**Table 6-4. Calculated Pressure Drops (psig) for 50m Columns<sup>4</sup>**

$\bar{u}^5$	Column I.D. ( $\mu\text{m}$ )		
	320	250	100
10	5.2	12.2	52.9
20	10.3	24.4	-
30	15.5	36.6	-
40	20.7	48.8	-
60	31.0	73.2	-
80	41.3	-	-

**Setting the Carrier Gas Flow for the Programmed On-Column Injector (POC) Using the Optional Flow Readout**

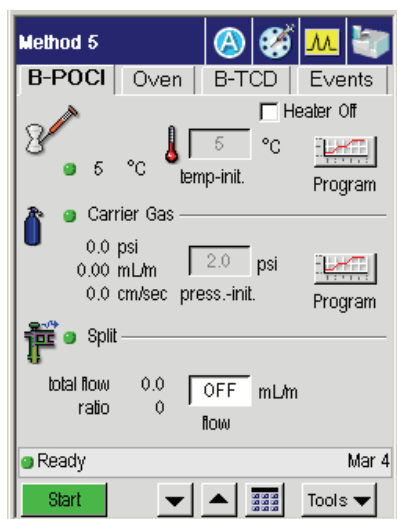
To set the carrier gas flow with the optional flow readout:




1. Turn on the carrier gas at the tank. Adjust the line pressure to 90 psig.
2. From the System Status screen select either **A** or **B** injector (the example here shows the B injector ready for setting the carrier gas).

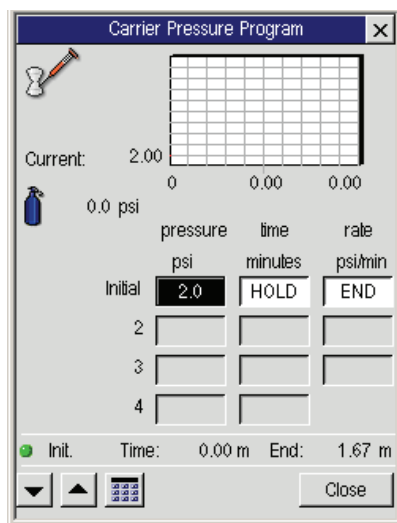


## Installing a Capillary Column

3. In the following window touch the **Program** button in the **Carrier Gas** field.



4. Using the up and down arrow or keypad    buttons enter the pressure (0.1 to 100 psig) values.



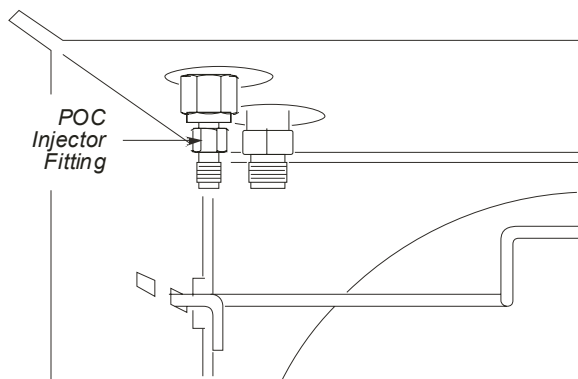
4. Type the desired flow set-point value.

**Set the Carrier Gas Flow for the Programmed On-Column Injector (POC) Using the Soap Bubble Flowmeter**

To perform this procedure, you must know how to measure carrier gas flow using a soap bubble flowmeter and the Clarus 500 GC built-in stopwatch. If you are not familiar with this measurement, please read "Using the Built-in Stopwatch" in Chapter 4 before you install a column.

To set the carrier gas flow using the soap bubble flowmeter:

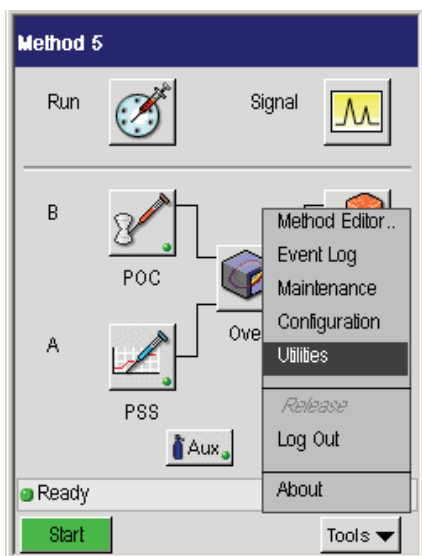
1. Locate the POC injector fitting inside the column oven. The POC injector fitting is shown below:



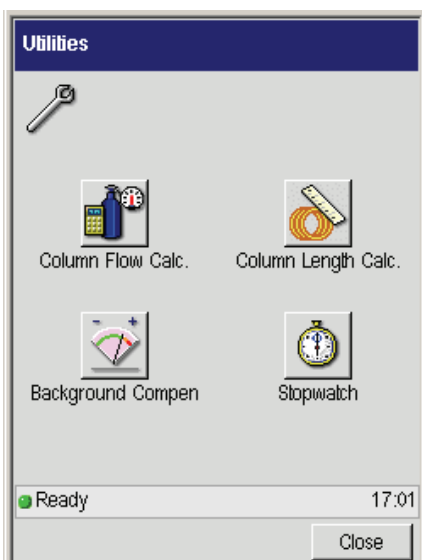
**Figure 6-44. POC injector fitting in the column oven.**




2. Attach a soap bubble flowmeter to the POC injector fitting.
3. Turn on the carrier gas at the tank. Adjust the line pressure to 90 psig.
4. From the System Status Screen select the Tools button and then select Utilities.

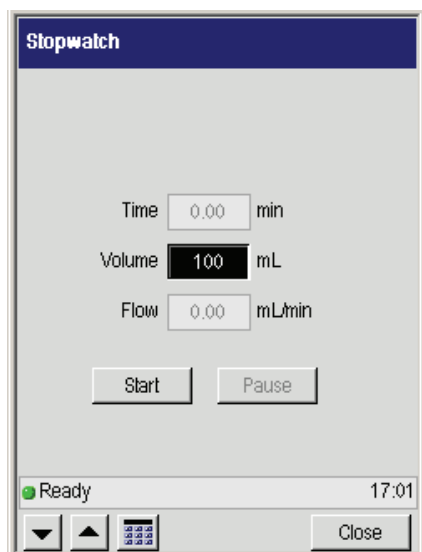
## Installing a Capillary Column



5. In the Utilities window select the Stopwatch icon.



6. Using the up and down arrow or keypad    buttons enter the appropriate values and press **Start**.



7. Start the carrier gas flowing by turning the flow controller knob counterclockwise and measure the flow.

**NOTE:** For best accuracy, use a soap bubble flowmeter volume that gives a reading of at least 30 seconds.

8. Adjust the flow to the desired set point by repeatedly measuring the flow and turning the flow controller knob counterclockwise to increase the flow, clockwise to decrease the flow, until the desired flow is obtained.
9. Disconnect the soap bubble flowmeter before proceeding to the next step to Step D “Leak Test all New Connections.”

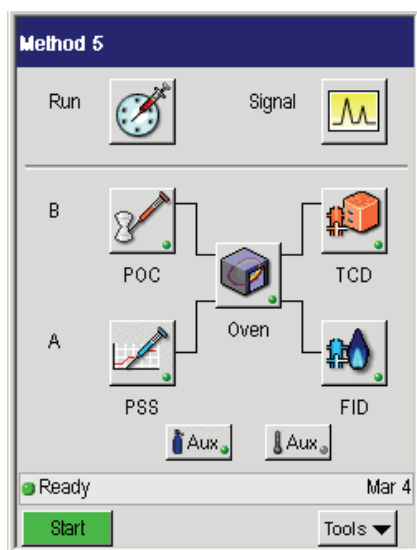
**Table 6-5. Suggested Capillary Column Flow Rates Using Helium As Carrier**

Column i.d. (µm)	250	320	530
Flow mL/min	0.6 - 0.8	1.0 - 1.5	2.5 - 4.0

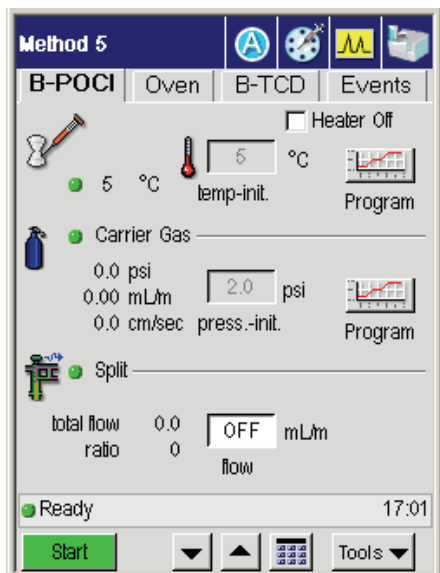
## **Step D Leak Test All New Connections:**

### **PPC Pneumatics (POC Injector Only)**

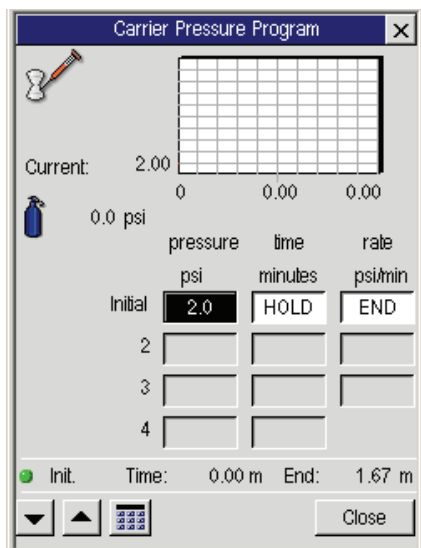
1. In the following example select the **POC** from the System Status screen.



2. In the next window select the Carrier Gas **Program** button.



3. In the **Program** screen enter a minimum value between 0.1 and 100 psig. Enter a maximum value between 0.1 and 100 psig.



### *Installing a Capillary Column*

The Clarus 600 GC monitors the inlet column head pressure with a carrier-gas mass-flow controller in use. You enter the minimum and maximum values after selecting the carrier-gas **Flow** mode in configuration. If the inlet column head pressure drops below or rises above the set limits for more than one minute, a warning message appears on the display. The following error message will appear: **Carrier** (with specific carrier number) **unable to maintain pressure**.

You should correct the leak; the most common area would be the injector septum. Then clear the message by touching **OK** before continuing to use the GC. This will stop an autosampler program from continuing. If you do not make any entries, leak checking will not be done. If you are temperature programming, then the value you enter for the maximum must be above the column head pressure at the highest oven temperature. You can just enter a value for the minimum and leave the maximum off (zero).



## **Step E: Condition the Column and the Mechanical Joint Between the Pre-column and Column:**

---

This section contains a suggested temperature program for conditioning a column. The program starts off by holding the oven temperature at a medium value for 10 minutes, gradually increasing the oven temperature at a fixed rate (5 °C/min) to the column operating temperature, then holding that temperature overnight with the carrier gas flowing.

**CAUTION**

*The temperatures shown in the examples which follow should be used as guidelines. Please refer to the column manufacturer's operating instructions for specific temperature recommendations.*

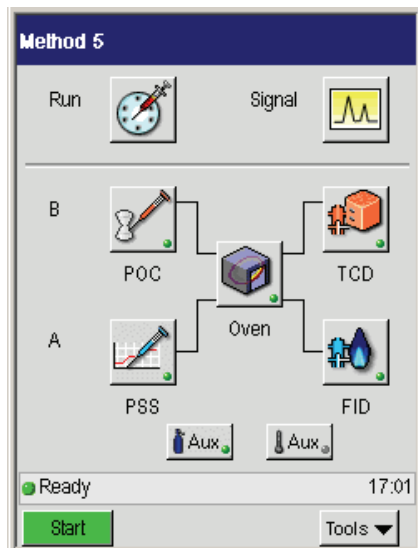
**CAUTION**

*To keep the injector clean, open the split vent to direct more gas through the injector.*

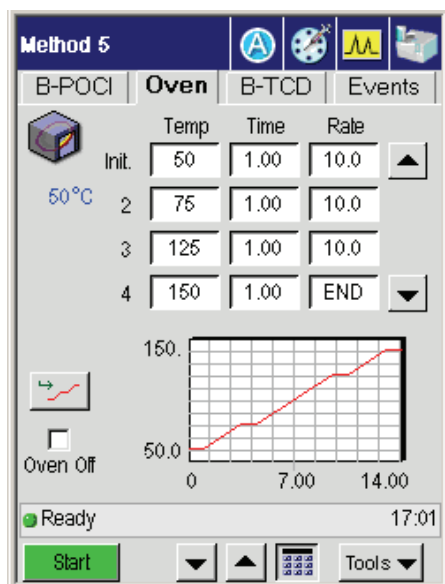
## Installing a Capillary Column

To condition the column:

1. Close the oven door and from the System Status screen touch the **Oven** button.



2. In the oven screen enter an oven temperature set point of 50 °C and enter a (Hold) time of 10.



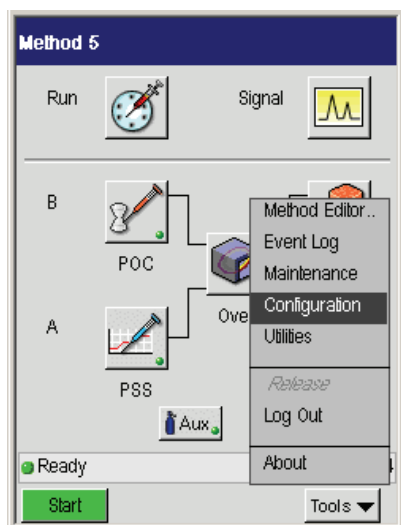
- To add another program step, enter a **Rate** of 5(°C/min).
- For **Temp** 2, enter a set point 25 to 50 °C above your planned analytical operating temperature.

**CAUTION**

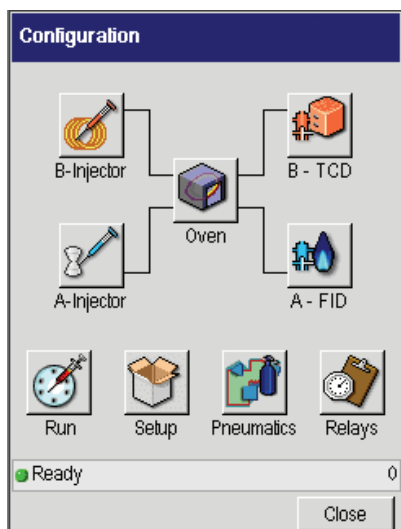
*To avoid damaging the column, do not enter a temperature higher than the maximum recommended temperature specified by the column manufacturer.*

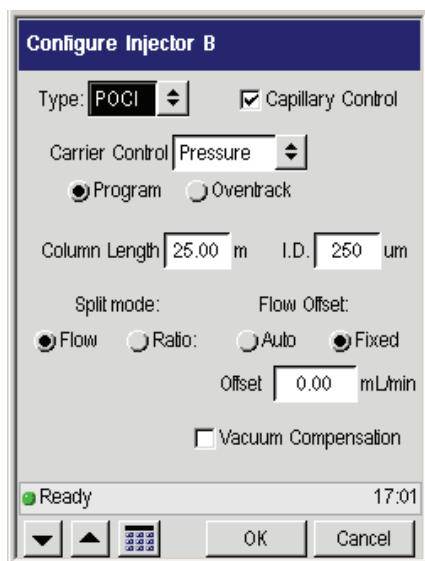
- Configure the injector for the **Oven** mode. From the System Status screen touch the **Tools** button and select the **Configuration** menu.

## Installing a Capillary Column

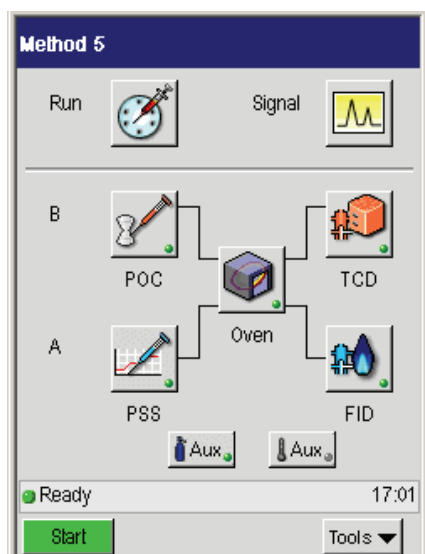


6. In the **Configuration** menu select the **Injector** button. In the following example **Injector B** has been selected to be configured.



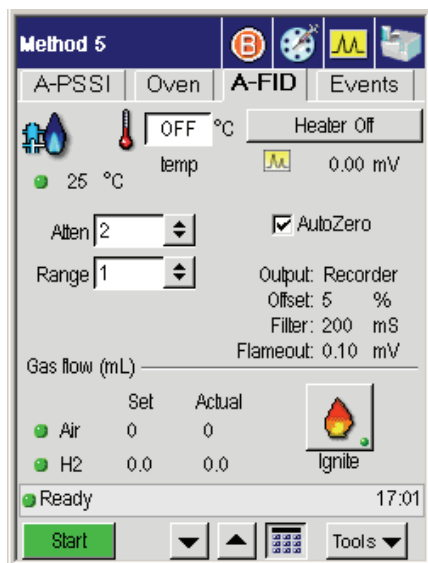


7. Select the **Program** button.
8. Touch the **OK** button to close this screen then touch the **Close** button to close the **Configuration** screen.



## Installing a Capillary Column

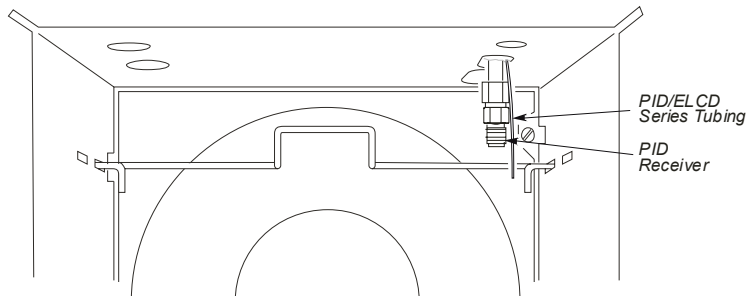
9. Touch the a detector button (for example FID) and turn the Detector Temperature off. Allow the system to run overnight.



10. In the morning, reset the oven the oven temperature set point to the initial **Temp** at the beginning of the temperature program. See step 4 earlier in this procedure.
11. Open the oven door. Allow the oven to cool until the oven fan turns off. This occurs when the oven cools down to 40 °C.

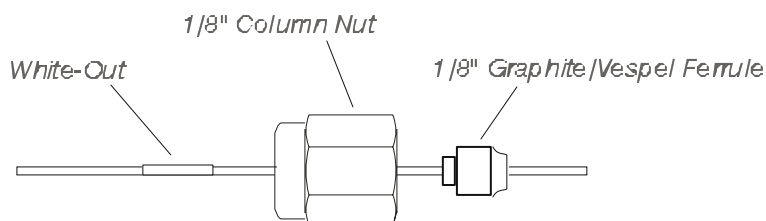
## **Step F: Connect the Column to the Detector:**

**NOTE:** Before connecting a wide-bore column (0.53-mm i.d.) to a Photoionization Detector (PID), remove the 1/8-inch diameter receiver from the PID base.



**Figure 6-45. PID receiver viewed inside the column oven**

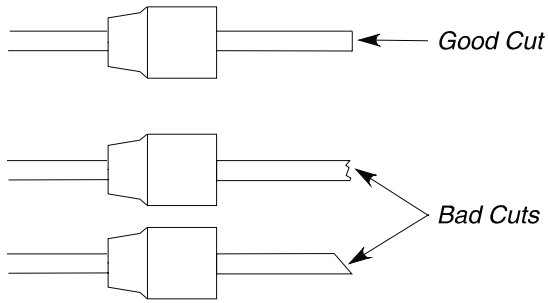
1. Place the column over the hanger so that no part of the column touches the bottom or sides of the oven.
2. Insert a 1/8-inch column nut and graphite ferrule over the free end of the column as shown below:



**Figure 6-46. Nut and ferrule on the detector end of a narrow-bore capillary column.**

3. Cut about 1 cm (3/8 inch) from the column end using a wafer scribe (P/N N930-1376, pkg. of 10 scribes) or other column cutting tool. Break off the tubing at the score mark so that the break is clean and square. Examine the cut with a magnifying glass and compare it to the following figure:

### Installing a Capillary Column



**Figure 6-47. Example of a good column cut and bad cuts.**

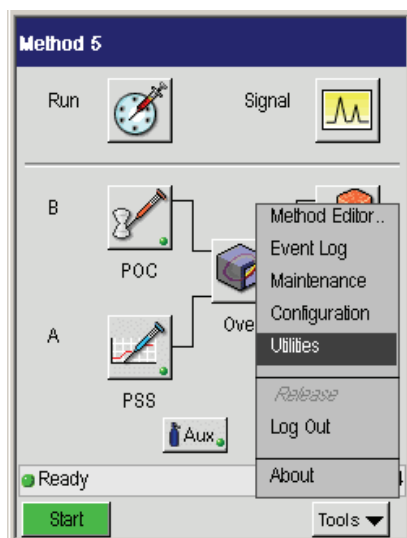
4. Mark the column the following distances from the end using correction fluid (white-out) or a felt-tipped pen:

Column Diameter	Detector to which you are attaching the column	Distance from back of nut
<input type="checkbox"/> 0.53 mm i.d.	FID	2.75 inches (70 mm)
<input type="checkbox"/> 0.53 mm i.d.	ECD <sup>6</sup>	2.5 inches (64 mm)
<input type="checkbox"/> 0.53 mm i.d.	TCD	4 inches (103 mm)
<input type="checkbox"/> 0.53 mm i.d.	NPD	3 inches (77 mm)
0.53 mm i.d.	FPD	2.5 inches (64 mm)
<input type="checkbox"/> 0.32 mm i.d.	FPD	4 inches (103 mm)
0.53 mm i.d.	PID	4 inches (103 mm)
<input type="checkbox"/> 0.32 mm i.d.	PID	5.25 inches (135 mm)
<input type="checkbox"/> 0.53 mm i.d.	EICD	3.5 inches (90 mm)

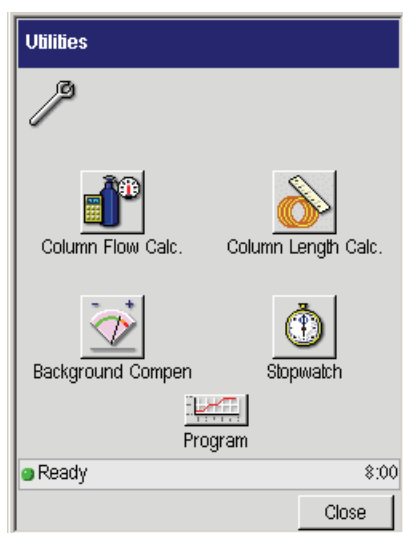
5. From the System Status screen select the **Tools menu**. From the tools menu select **Utilities**.

<sup>6</sup> A glass-lined receiver (P/N N600-0968) is available to reduce high background readings. For more details, see the Important on page 8-22.



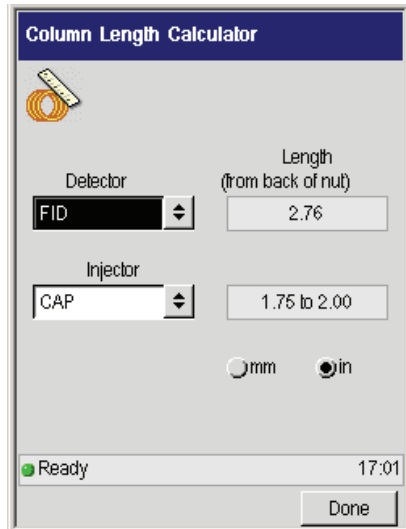


6. In the Utilities screen select the **Column Length Calc** icon.



7. Using the drop down menus you will be able to select the proper detector and injector for the calculator. The using the plus and minus buttons input the length from the back of the nut and the Column Length Calculator will automatically calculate the column length in millimeters or inches.

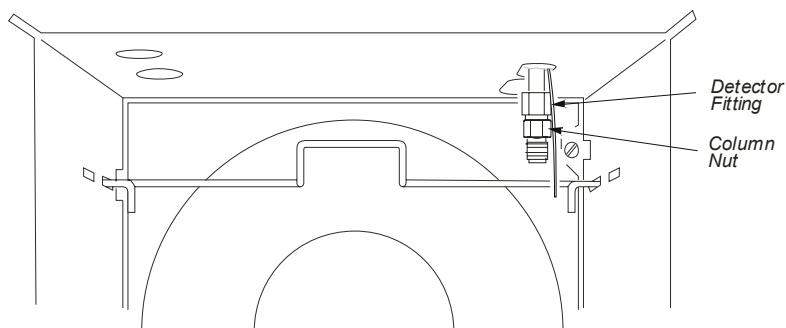
## Installing a Capillary Column



### CAUTION

*To avoid contaminating the system, make certain that the nut and ferrule do not contact the mark on the column.*

8. Locate the detector fitting protruding from the right side of the oven roof.
9. Insert the column into the detector fitting, keeping the mark just behind the column nut.
10. While holding the column in position, hand-tighten the column nut.
11. Hold the detector fitting steady with one of the 7/16-inch wrenches as you gradually tighten the column nut with the other wrench. Tighten the nut only until you cannot pull the column out of the nut. **DO NOT OVERTIGHTEN THE NUT!**



**Figure 6-48. Capillary column connected to the detector fitting.**

**CAUTION**

*Make certain that no part of the column touches the walls or bottom of the oven.*

**CAUTION**

*Do not overtighten column nuts. Overtightening can cause damage to the ferrule and/or column.*

**Step G: Leak Test All New Connections:**

Test the detector connection for leaks using a 50/50 mixture of isopropanol/water or an electronic leak detector. To avoid contaminating the system, **DO NOT** use a soap solution for leak testing. Tighten all leaking connections.

## **Step H: Set up the Split Mode for a CAP or PSS Injector:**

---

The Split Mode is used to analyze concentrated samples. In this mode only part of the sample enters the column; the remainder is split and vented through a charcoal filter to atmosphere. This step describes how to set the split mode for PPC modules.

**NOTE:** *In the Split Mode, the split vent is always open.*

**NOTE:** *The injector is shipped with an unpacked wide-bore injector liner installed. Remove the liner and pack it with quartz wool before running your analysis. Refer to Step B, Connect the Column to the Injector, in this chapter*

The following procedure assumes that the carrier gas pressure has been set (see Step C in this chapter).

For information on Setting the Split Mode Using PPC, Setting up the Splitless Mode for a CAP or PSS Injector, Setting the Splitless Mode Using PPC Modules, see the *Clarus 600 GC Users Guide* (P/N 0993-6780) Using the Active Method chapter.

## ***PSS and POC Operating Hints:***

---

The Programmed Split/Splitless (PSS) and Programmed On-Column (POC) injectors can be operated in either the oven-programming mode or the inlet-programming mode. For specific instructions, refer to Chapter 9 of this manual.

**CAUTION**

*If you have the subambient option, the POC and PSS injectors are linked to the oven subambient option; therefore, you cannot operate the injectors below the oven subambient temperature.*

### ***Oven Programming Mode***

This is the default mode for both the POC and the PSS injectors. This mode is the easiest to operate since only the oven temperature program needs to be entered into the method. In this mode, the injector will follow the oven temperature profile plus five degrees Celsius. In this mode, the sample is introduced into the injector when the temperature of the inlet is at the boiling point of the solvent. Then start the injector and oven temperature program.

If the initial temperature of the oven is above the boiling point of the solvent you are using, then it would be better to modify the oven program to start at a lower temperature or to configure the injector for the Inlet mode and set a temperature for the injector separate from the oven temperature.

### ***Inlet Programming Mode***

This mode permits the use of independent injector temperatures and rates that you define in the method. The injector will be programmed for injector temperature 1, injector time 1, injector rate 1, injector temperature 2, injector time 2, etc. You can program up to three temperatures and two ramps for each PSS or POC configured in the inlet mode. It is important to set the initial injector temperature to approximately the boiling point of the solvent you are using.

**CAUTION**

*The PSS can be used in the "hot" split or splitless mode. This, however, is not recommended for use with the 1 mm i.d. liner; it could cause solvent flashback in the injector. This mode should be used with caution depending upon the solvent and temperatures you choose. Always use the 2-mm i.d. injector liner. See Table 6-1 at the beginning of this chapter.*

**CAUTION**

*When using the PSS in the on-column mode, or the POC with the autosampler, you must use a special syringe that has a needle o.d. of 0.47 mm (P/N N610-1253 or N610-1380). You must only use the "Norm" injection speed with this syringe in the on-column mode. The "Fast" injection speed will bend this thin needle and the "Slow" injection speed may produce peak break up or distorted peaks. You can achieve better precision in the on-column mode when sample volumes of 1.0  $\mu\text{L}$  or greater are injected.*

If a column is used extensively at high temperatures (350 °C or greater), the polyimide may become very brittle. This brittleness will cause the column to fracture when you try to seal it in the universal adapter. If you wish to continue using a brittle column, use a low dead-volume union instead of the universal adapter.

### **Solvent Purge Mode**

The PSS injector can be operated in the *solvent purge mode* without any modification. This technique is an enhanced mode of the splitless injection and may use either the 0.5- $\mu\text{L}$  or 5.0- $\mu\text{L}$  syringe. To obtain better detection limits, you may also use a 50- $\mu\text{L}$  syringe to make larger volume injections. In the solvent purge mode, the split vent is open at the time of injection and the PSS injector temperature is held below the boiling point of the solvent. Once most of the solvent has vented, the split vent closes and the PSS injector is heated so that it can transfer the analytes onto the column.

You must select the starting temperature of the PSS injector based on the solvent you are using. In addition, to eliminate most of the solvent without losing any of the compounds of interest, you must experimentally determine the minimum split flow and vent time.

The initial value of the split vent can be ON or OFF, depending upon your solvent volatility. When you make your injection, the split vent OPENS (turns ON) and stays open for the time you have selected. After that time, the split vent CLOSES (turns OFF via a timed event) and the PSS injector heats to the selected temperature. To help maintain good peak shape and quantitative recovery, we recommend holding the Clarus GC oven temperature at its starting value until the PSS injector temperature reaches its final temperature and all of the sample has been transferred onto the column. To minimize tailing of the residual solvent and ensure that the system is completely cleaned for the next injection, start the oven temperature program and re-open the split vent.

The charcoal trap's capacity is too small to handle the large volumes of solvent and it will saturate quickly. When making large volume injections, we recommend disconnecting the charcoal trap from the PSS injector. For details, refer to “Replacing the Charcoal Trap on the Split/Splitless, CAP, and PSS Injectors” in Chapter 9, Maintenance. To retain the vented solvent, we strongly recommend a larger capacity charcoal trap after (downstream from) the vent on the AutoSystem.

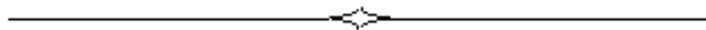
**NOTE:** *When using the PSS injector in the split mode, the charcoal trap is required to maintain the split ratio throughout the injection.*

## **Calculating a Capillary Column Split Ratio**

The following procedure and examples show how to calculate the capillary column split ratio using PPC.

1. Enter the column length, I.D., and vacuum compensation values.
2. Measure the unretained peak time and correct the column length, see Chapter 8.
3. Read the column and split flows from the screen on the Clarus 600 GC.
4. Calculate the Split Ratio:

$$\text{Split Rate} = \frac{\text{Flow rate from the split vent in mL/min} + \text{Flow rate of the column}}{\text{Flow rate of the column}}$$





**Prevent 7**

---

*Prevent*

**7-2**

## Overview

---

PreVent™ is an enhanced capillary inlet system for the Clarus 600 GC that uses columns with an inside diameter (i.d.) between 0.25 mm and 0.53 mm. You can use the pre-column venting technique to:

- Manage difficult samples
- Protect the column and gas chromatograph
- Lower detection limits
- Increase analytical throughput

PreVent is used in the following five Modes of Operation:

1. Column Isolation
2. Solvent Purge (PSS injector only)
3. Large Volume Injection (PSS injector only)
4. Sample Residue Purge (PSS injector only)
5. Time Saver

This chapter describes how to install the PreVent adapter and restrictor on an injector or detector. Based on your mode of operation, the PreVent adapter is installed on either the injector or detector (including TurboMass MS). Turn to the procedure in this chapter that describes how to install PreVent:

Installing PreVent on an Injector

Installing PreVent on a Detector

Installing PreVent on a TurboMass MS Detector

## *Prevent*

**NOTE:** *The instructions in this chapter assume that the PreVent option has been installed in the front injector position (Channel A). The mid-point pressure for Channel A is controlled by valve 3, and the mid-point pressure for Channel B is controlled by valve 4.*

**NOTE:** *The PreVent option contains a bleed flow of nominally 15 mL/min of carrier gas that constantly flows and vents to atmosphere when you set the pressure on the PreVent mid-point module. If your carrier gas is hydrogen, you should connect tubing to the barbed fitting on the bleed controller (on the underside of the GC top cover), and direct it to a venting hood.*

## ***Installing PreVent on an Injector***

---

The following procedure describes how to install the PreVent adapter on a capillary inlet system (CAP and/or PSS) and how to make it ready for an analysis.

### ***Summary***

The following steps summarize how to install an enhanced capillary inlet system to an injector and get it ready for use:

- A. Install the restrictor in the PreVent adapter.
- B. Connect the PreVent adapter to the injector.
- C. Connect the column to the PreVent adapter.
- D. Leak-check the system.
- E. Connect the column to the detector.
- F. Set the initial pressures.
- G. Condition the system.

### ***A. Install the Restrictor in the PreVent Adapter***

The following restrictors are available for the injector:

- 0.06 mm i.d. restrictor (P/N N610-3080)
- 0.06 mm i.d. to 0.250 mm i.d. restrictor (P/N N610-0342)
  1. Select the appropriate restrictor for your analysis and cut it to the proper length. Refer to the following table:

**Table 7-1 Recommended Restrictors**

<b>Injector</b>	<b>Injection Mode</b>	<b>Restrictor</b>	<b>Part Number</b>	<b>Length (mm)</b>
Split/Splitless	Split	0.06 mm i.d.	N610-3080	108
Split/Splitless	Splitless	0.06 mm i.d.	N610-3080	108
PSS	Split	0.06 mm i.d. to 0.250 mm i.d. <sup>1</sup> .	N610-0342	104
PSS	Splitless	0.06 mm i.d. <b>or</b>	N610-3080	104
		0.06 mm i.d. to 0.250 mm i.d.	N610-0342	

Thread the selected restrictor first through a 0.5 mm graphite ferrule (P/N 0990-3700) and then through the PreVent adapter (P/N N610-0328) until 5 mm protrudes through the column connection on the bottom. When using the 0.06 mm i.d. to 250 mm i.d. restrictor, the narrow end should protrude through the bottom.

**CAUTION**

*To prevent contamination problems and the need for extensive conditioning procedures, ensure that all PreVent components are thoroughly clean. Baking them in an oven prior to assembly is recommended or wiping with a tissue dampened with alcohol. Always wear clean gloves when assembling the PreVent components.*

<sup>1</sup> This restrictor is pre-cut to the correct length for PSS.

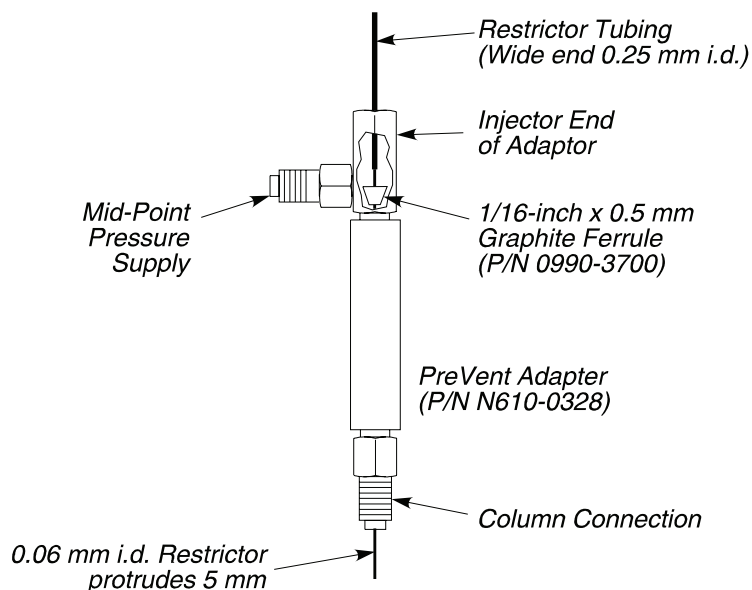


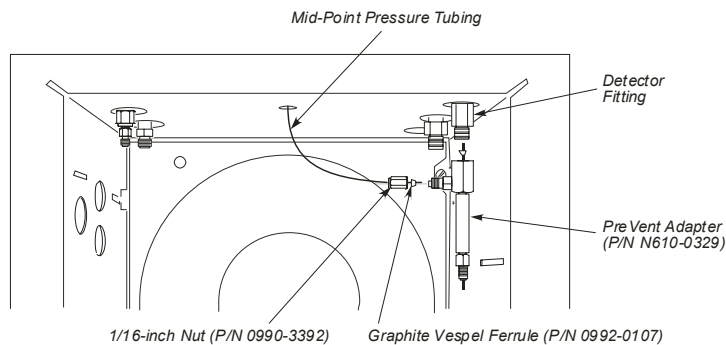
Figure 7-1. Installing the restrictor in the PreVent adapter.

## B. Connect the PreVent Adapter to the Injector

1. Verify that the injector liner is correct for your application. If necessary, pack the injector liner with quartz wool. For details, refer to packing a PSS or split/splitless injector liner in Chapter 6.
2. Carefully connect the PreVent adapter by screwing the injector end of the adapter into the capillary injector fitting until it is finger tight.
3. Using a 1/4-inch wrench, tighten the PreVent adapter until the graphite ferrule just starts to tighten on the restrictor.
4. Locate the stainless steel tubing (that supplies the mid-point pressure) inside the oven.
5. Insert a 1/16-inch nut (P/N 0990-3392) and a graphite/Vespel ferrule (P/N 0992-0107) over the end of this tubing.

## Prevent

6. Measure and mark the tubing 1/2-inch from the back of the nut.
7. Insert the tubing into side arm on the PreVent adapter, keeping the mark just behind the nut. Then tighten the nut and ferrule fingertight.
8. Make a leak-free seal by tightening the PreVent adapter, on the capillary injector fitting, another 1/4 to 1/2 turn.



**Figure 7-2. Capillary injector fitting inside the oven.**

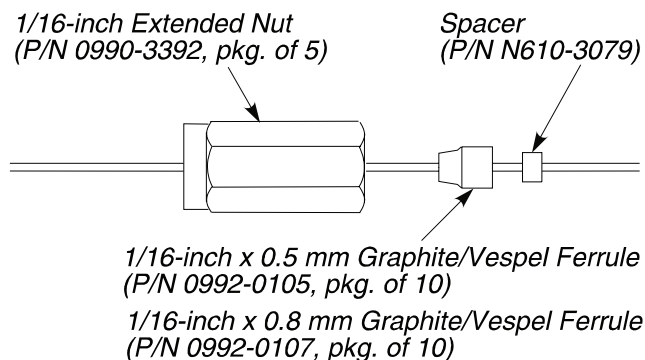
### **C. Connect the Column to the PreVent Adapter**

1. Place the column on the hanger so that no part of the column touches the bottom or sides of the oven.
2. Insert a 1/16-inch extended nut (P/N 0990-3392, pkg. of 5), a graphite/Vespel ferrule (1/16-inch x 0.5 mm P/N 0992-0105, pkg. of 10 for 0.25/0.32 i.d. column, or a 1/16-inch x 0.8 mm P/N 0992-0107, pkg. of 10 for a 0.53 mm i.d. column), and a spacer (P/N N610-3079), over one end of the column.

#### **CAUTION**

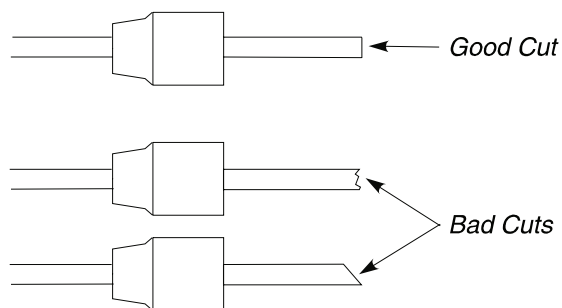
*Do not use a graphite ferrule since it will extrude up into the body of the PreVent adapter.*





**Figure 7-3. Extended nut, graphite/Vespel ferrule, and spacer.**

3. Cut about 1 cm (3/8 inch) from the end of the column tubing using a wafer scribe (P/N N930-1386, pkg. of 10 scribes) or other column cutting tool. Break off the tubing at the score mark making sure that the break is clean and square. Examine the cut with a magnifying glass and compare it to the following figure:



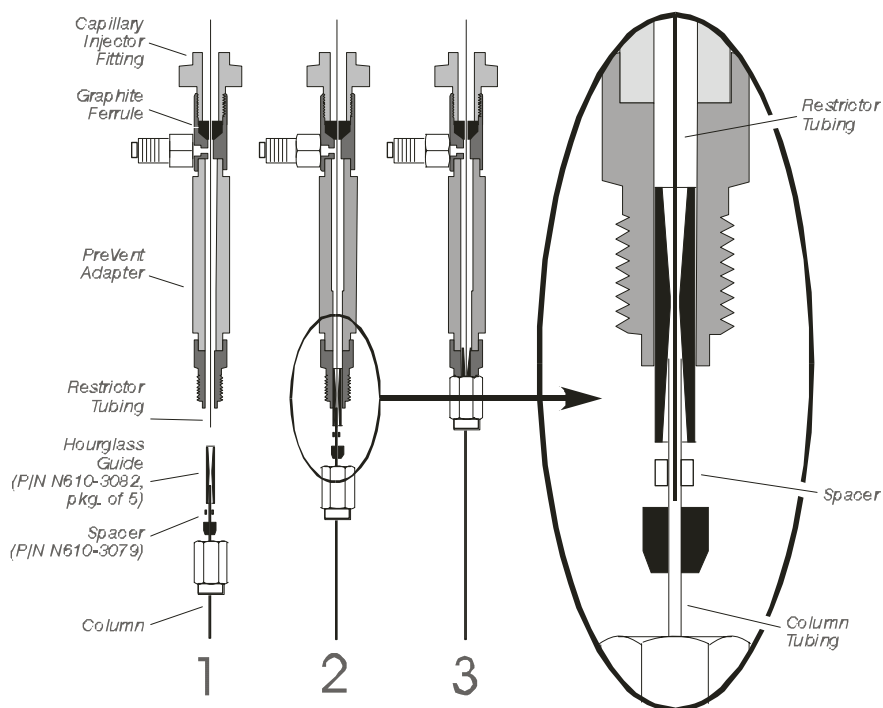
**Figure 7-4. Example of a good column cut and bad cuts.**

4. Insert the column end (as shown in Figure 7-5, Step 1) into the hourglass guide (P/N N610-3082).
5. Align the end of the restrictor tubing that protrudes from the PreVent adapter with the open end of the hourglass guide. Then, carefully push the hourglass guide, with the attached column, up into the PreVent adapter as shown in Figure 7-5, Step 2. Notice that the restrictor tubing fits inside the column.

## PreVent

**NOTE:** *Slightly twist the column as you push the hourglass guide into the PreVent adapter.*

6. Loosely connect the column nut to the PreVent adapter.
7. Carefully push the column up and into the PreVent adapter until it bottoms in the adapter. Then pull the column back 3 mm as shown in the following figure, Step 3.
8. Use two 1/4-inch wrenches to tighten the nut. **DO NOT OVERTIGHTEN THE NUT!**

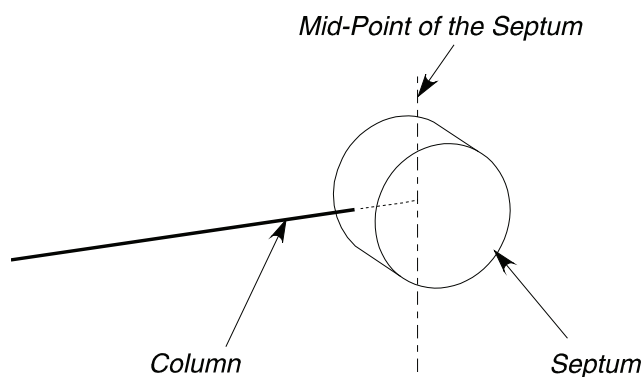


**Figure 7-5. Connecting a column to the PreVent injector adapter.**




### **D. Leak-Check the System**

1. Locate the detector end of the column.

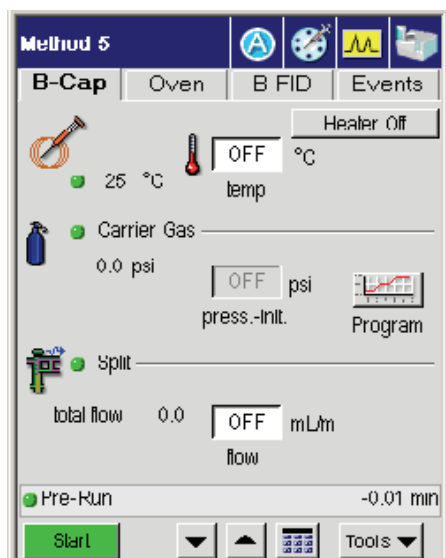
2. Seal the column end by firmly inserting the detector end of the column into the side of a septum to about the mid-point as shown in the following figure.



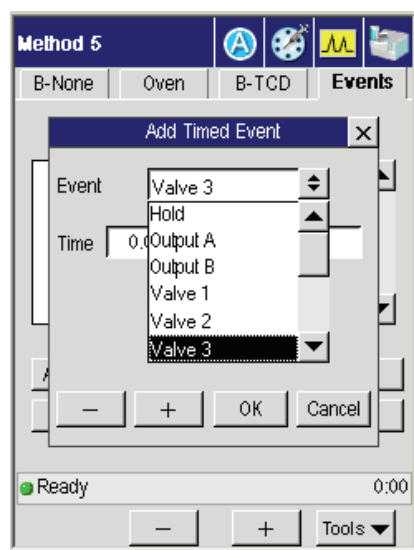
**Figure 7-6. Column inserted into a septum.**

3. Turn on the GC. When the GC is turned on, it is set to the initial default settings.
4. From the System Status screen touch the injector to get to the following screen. Touch the numeric field until it turns black and use the up and down arrow or keypad    buttons to get the proper split flow of 1.5 and a ratio of 25.

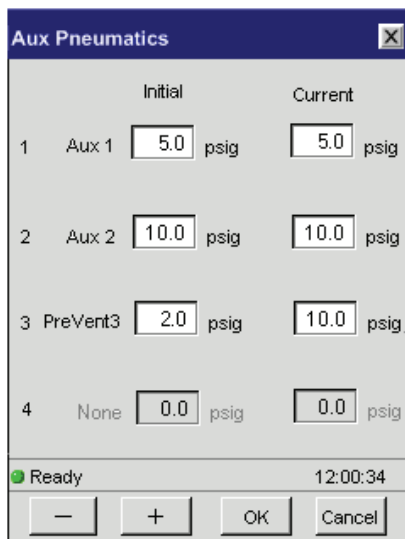
Prevent



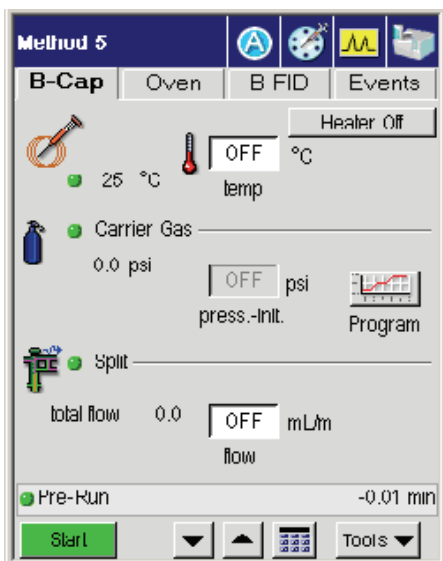
5. From the Events tab turn off the mid-point carrier gas supply by closing **Valve 3**.



6. Allow the system to equilibrate for several minutes, then view the Aux 1 pressure, view the Aux Gas button and view the current psig.



- Go back to the inject screen to view the pressure read out on the carrier gas which should be at 0 psig.



- Set the injector pressure to 75 psig.

## Prevent

9. After approximately 5 minutes, if no leaks exist, the **Aux 1** reading should be stable at approximately 75 psi. If it is low, then leak-check the fitting connections.
10. If you suspect a leak, test the connections for leaks using a 50/50 mixture of isopropanol/water or an electronic leak detector. To prevent contaminating the system, DO NOT use a soap solution for leak testing. Tighten all leaking connections.

If you detect a leak, check and tighten the fittings on the mid-point pressure tee and/or the connections on the rear of the Clarus 600 GC.

**CAUTION**      *Do not overtighten the column nut. If this fitting has a persistent leak, replace the graphite/Vespel ferrule.*

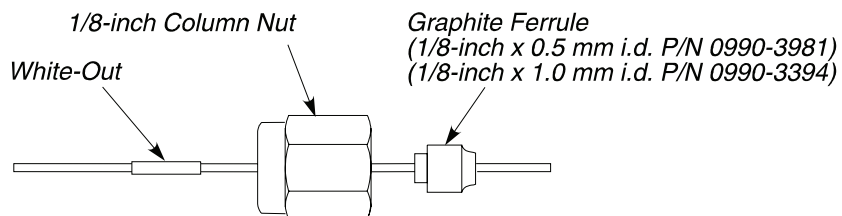
11. Turn off the two gas pressures and remove the septum from the end of the column.

## **E. Connect the Column to the Detector**

**CAUTION**      *Make certain that no part of the column touches the walls or bottom of the oven.*

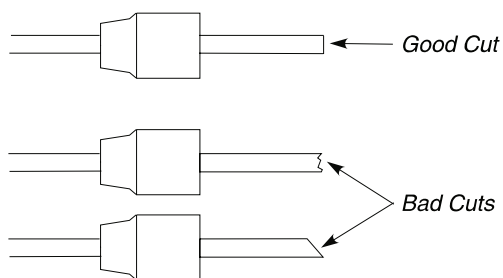
**NOTE:** *To connect a column to the TurboMass, refer to the instructions in the TurboMass Software Guide (Part No. 0993-6691).*

1. Insert a 1/8-inch column nut and graphite ferrule over the free end of the column as shown in the following figure.



**Figure 7-7. Nut and ferrule on the detector end of a narrow-bore capillary column.**

2. Cut about 1 cm (3/8 inch) from the column end using a wafer scribe (P/N N930-1376, pkg. of 10 scribes) or other column cutting tool. Break off the tubing at the score mark so that the break is clean and square. Examine the cut with a magnifying glass and compare it to the following figure:

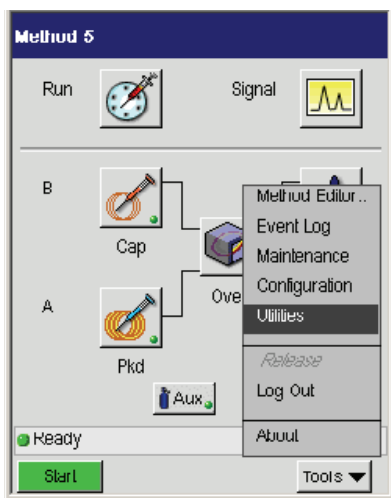


**Figure 7-8. Example of a good cut and bad cuts.**

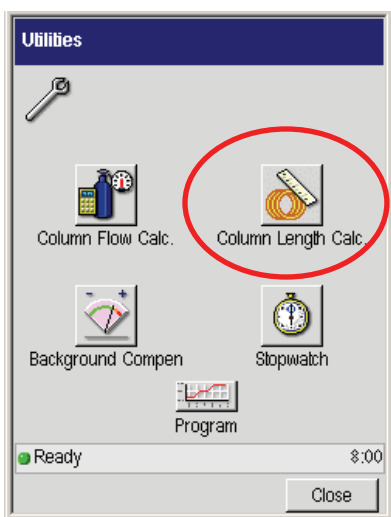
3. Mark the column using typewriter "white-out" or a felt-tipped pen. The distances are calculated using **Column Length Calc** procedure that follows.

Prevent

4. From the System Status screen select the **Tools** menu. From the tools menu select **Utilities**.



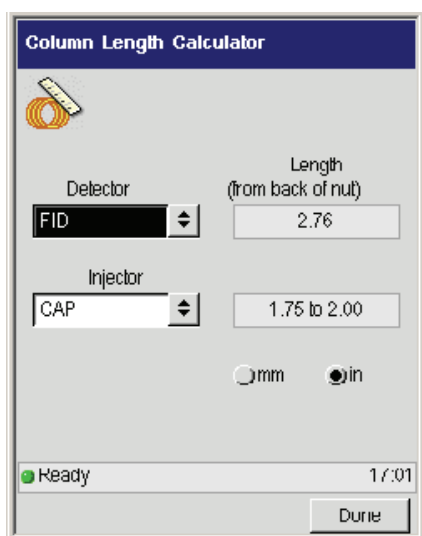
5. In the Utilities screen select the **Column Length Calc** icon.



6. Using the drop down menus you will be able to select the proper detector and injector for the calculator. The using the plus and minus buttons input the



length from the back of the nut and the Column Length Calculator will automatically calculate the column length in millimeters or inches.

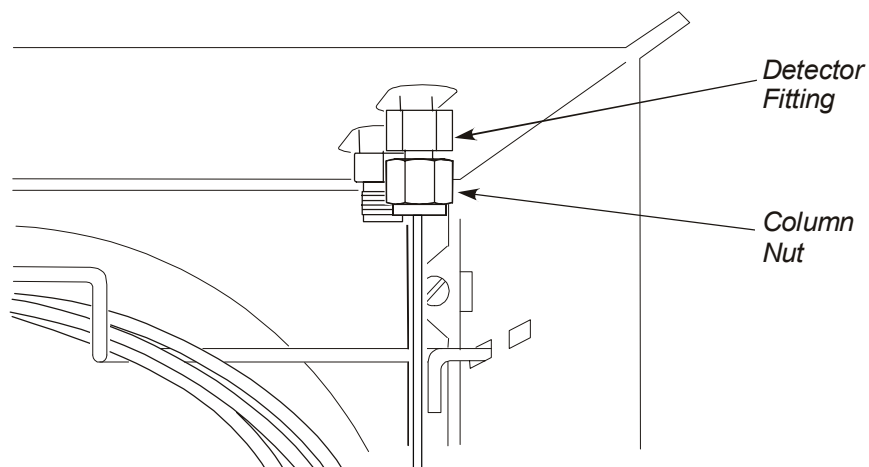


**CAUTION**

*To prevent contaminating the system, ensure that the nut and ferrule do not contact the mark on the column.*

7. Locate the detector fitting that protrudes through the right side of the oven roof.
8. Insert the column into the detector fitting, keeping the mark just behind the column nut.
9. While holding the column in position, hand-tighten the column nut.
10. Holding the detector fitting steady with one of the 7/16-inch wrenches, gradually tighten the column nut with the other wrench only until you cannot pull the column out of the nut. **DO NOT OVERTIGHTEN THE NUT!**

Prevent



**Figure 7-9. Capillary column connected to the detector fitting.**

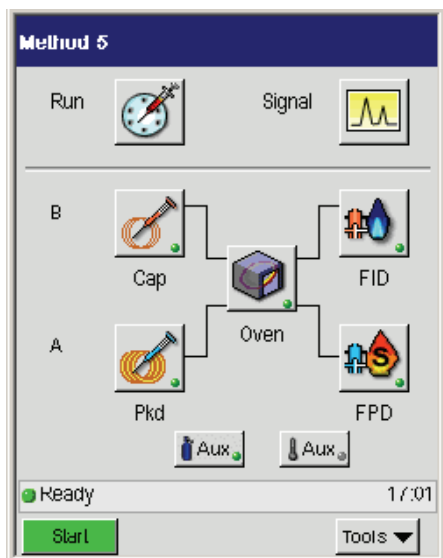
**CAUTION**

*Do not overtighten column nuts. Overtightening can cause damage to the ferrule and/or column.*

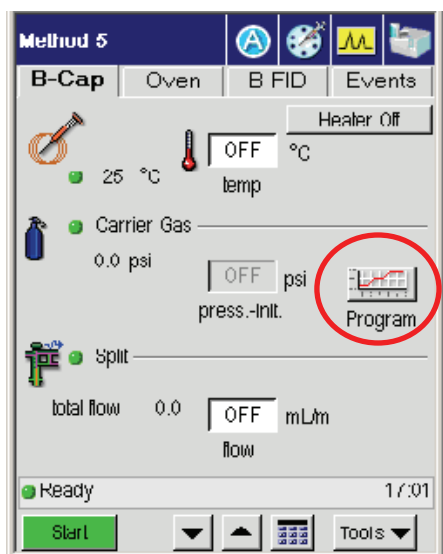
**F. Set the Initial Pressures**

To set the mid-point pressure:




1. From the System Status screen select either A or B injector (the example here shows the B injector ready for setting the carrier gas).

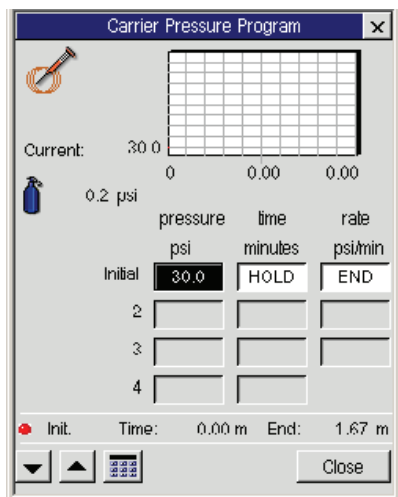


2. In the following window touch the **Program** graph screen under the Carrier Gas field.

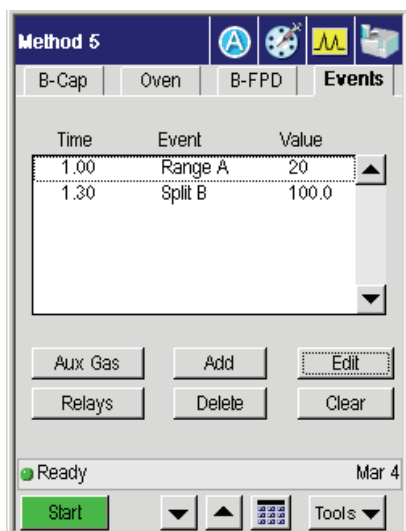


*Prevent*

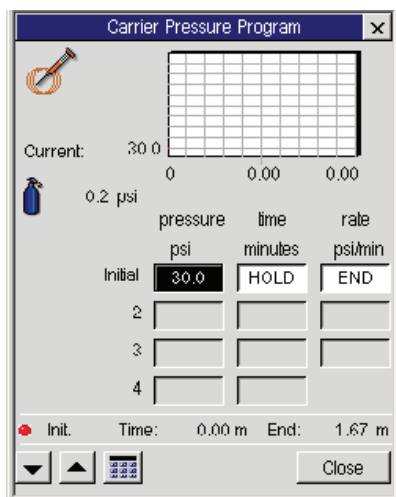
- Using the up and down arrow or keypad    buttons, enter the initial value of 30 psig for the Carrier gas. Press **Close** to go back to the previous screen (as seen in step 2).



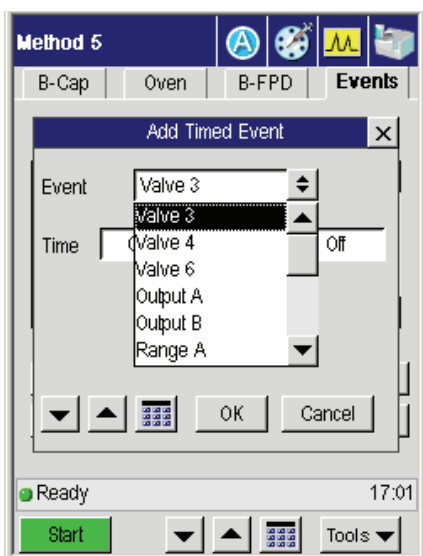
- In the Events tab touch the **Split** event and then press **Edit**. Set Split 1 to 100 mL/min.



- From the oven tab set the oven temperature to its highest programmed temperature.

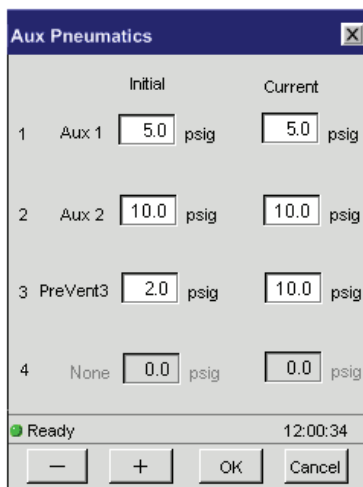


- From the Events tab turn off the mid-point carrier gas supply by closing **Valve 3**.



## Prevent

7. Allow the system to equilibrate for several minutes, then view the Aux 1 pressure, view the Aux Gas button and view the current psig.



**Note:** *If the Aux 1 pressure is very low, then the graphite ferrule that holds the restrictor inside the PreVent adapter may be leaking. Remedy this by tightening the adapter on the injector and observe if there is an improvement.*

8. Adjust the pressure until the **Aux** pressure is maintained at about 1.5 psig below the required column inlet pressure. Wait a few minutes for the system to stabilize to ensure that no further changes are required.
9. Open **Valve 3** and set the **Aux 1** pressure to the required column inlet pressure; for example, 1.5 psig higher than the Aux 1 reading established in step 9 above. This ensures a positive gas flow through the Aux 1 supply to the mid-point tee.

### CAUTION

*Valve 3 MUST remain open (ON) during PreVent operation.*

Condition the system to ready it for an analysis.

## **G. Condition the System**

1. Write down the pressures established for Press 1 and Aux 1 in step **F**.
2. Set **Press 1** to 2 psig, **Split 1** to 100 mL/min, and **Aux 1** to 80 psig.
  - If you are using a PSS, set its temperature to the maximum programmed temperature.
  - Set the oven to the maximum programmed temperature.
  - Allow the system to bake at this temperature for several hours — overnight if possible.
  - Shorter conditioning times may be possible, but that depends on the condition of the PreVent components and condition of the column.
3. Cool the oven (and if you are using a PSS injector, cool the injector).
4. Reset the initial settings of **Press 1** and **Aux 1**.

The system is now ready to run an analysis.

## ***Installing PreVent on a Detector***

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This section describes how to install PreVent on a detector (except for TurboMass MS). The next section describes how to install PreVent on TurboMass MS.

### ***Summary***

The following steps summarize how to install a PreVent adapter to a detector and make it ready for an analysis:

- A. Install the restrictor in the PreVent adapter.
- B. Connect the PreVent adapter to the detector.
- C. Connect the column to the PreVent adapter to the detector.
- D. Leak-check the system.
- E. Connect the column to the injector.
- F. Set the initial pressures.
- G. Condition the system.

### ***A. Install the Restrictor in the PreVent Adapter***

1. Cut the 0.075 mm i.d. restrictor (P/N N610-3081) to the proper length for your detector. Refer to the following table:



**Table 7-1. Recommended Restrictor Lengths for Detectors**

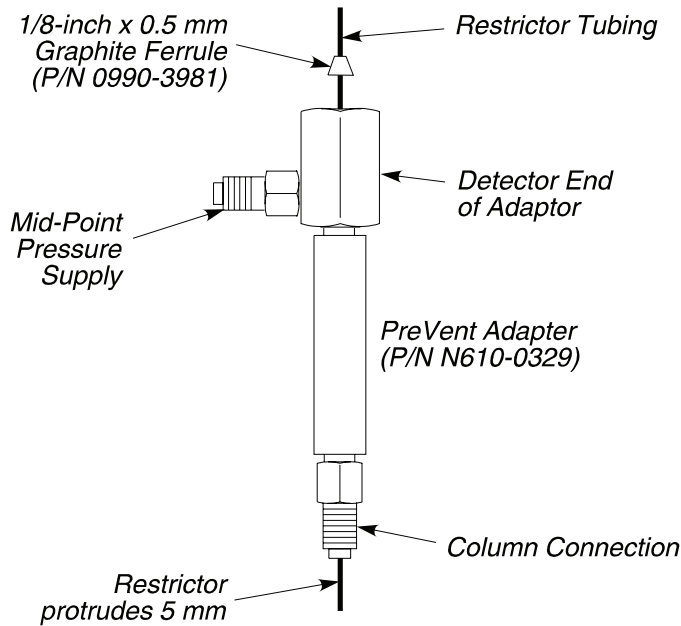
Detector to which you are attaching the adapter	Restrictor Length
PID	195 mm (7.66 inches)
TCD	163 mm (6.40 inches)
FID	137 mm (5.38 inches)
ECD <sup>2</sup>	124 mm (4.87 inches)
EICD	150 mm (5.89 inches)
NPD	137 mm (5.38 inches)
FPD	163 mm (6.40 inches)

2. Insert a 1/8-inch graphite ferrule over one end of the restrictor tubing.
3. Insert the end of the restrictor tubing into the top of the PreVent detector adapter (P/N N610-0329).
4. Feed the restrictor tubing through the PreVent adapter until 5 mm protrudes through the column connection on the bottom.

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<sup>2</sup> A glass-lined receiver (P/N N600-0968) is available to reduce high background readings.

## Prevent



**Figure 7-10. Installing the restrictor in the PreVent detector adapter.**

### **B. Connect the PreVent Adapter to the Detector**

1. Carefully connect the PreVent adapter by screwing the detector end of the adapter into the detector fitting until it is finger tight.
2. Using a 7/16-inch wrench, tighten the PreVent adapter until the graphite ferrule just starts to tighten on the restrictor.
3. Locate the stainless steel tubing (that supplies the mid-point pressure) inside the oven.
4. Insert a 1/16-inch nut (P/N 0990-3392) and a graphite/Vespel ferrule (P/N 0992-0107) over the end of this tubing.
5. Measure and mark the tubing 1/2-inch from the back of the nut.
6. Insert the tubing into side arm on the PreVent adapter, keeping the mark just behind the nut. Then tighten the nut and ferrule fingertight.

7. Make a leak-free seal by tightening the PreVent adapter on the detector fitting another 1/4 to 1/2 turn.

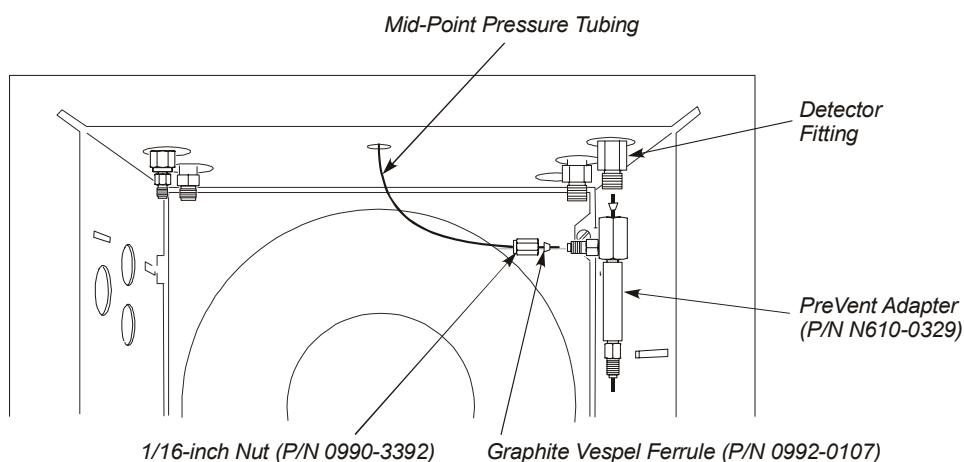


Figure 7-11. Location of the detector fitting.

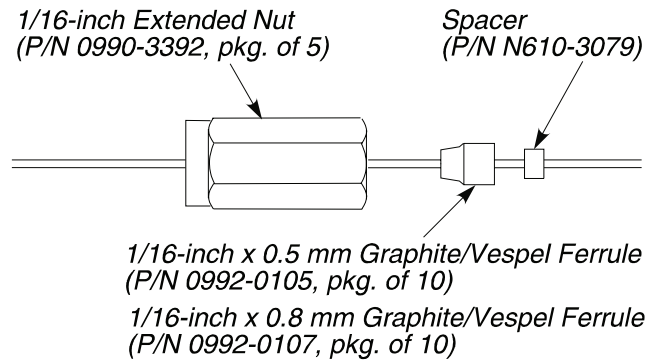
### C. Connect the Column to the PreVent Adapter

1. Place the column on the hanger so that no part of the column touches the bottom or sides of the oven.
2. Insert a 1/16-inch extended nut (P/N 0990-3392, pkg. of 5), a graphite/Vespel ferrule (1/16-inch x 0.5 mm P/N 0992-0105, pkg. of 10 for 0.25/0.32 i.d. column or a 1/16-inch x 0.8 mm P/N 0992-0107, pkg. of 10 for a 0.53 mm i.d. column), and a spacer (P/N N610-3079) over one end of the column.

#### CAUTION

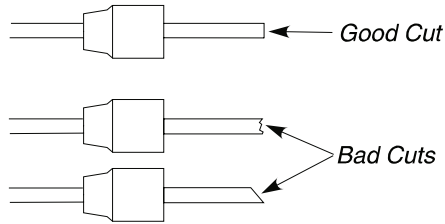
*Do not use a graphite ferrule since it will extrude up into the body of the PreVent adapter.*

*Prevent*



**Figure 7-12. Extended nut, graphite/Vespel ferrule, and spacer.**

3. Cut about 1 cm (3/8 inch) from the end of the column tubing using a wafer scribe (P/N N930-1386, pkg. of 10 scribes) or other column cutting tool. Break off the tubing at the score mark making sure that the break is clean and square. Examine the cut with a magnifying glass and compare it to the following figure:

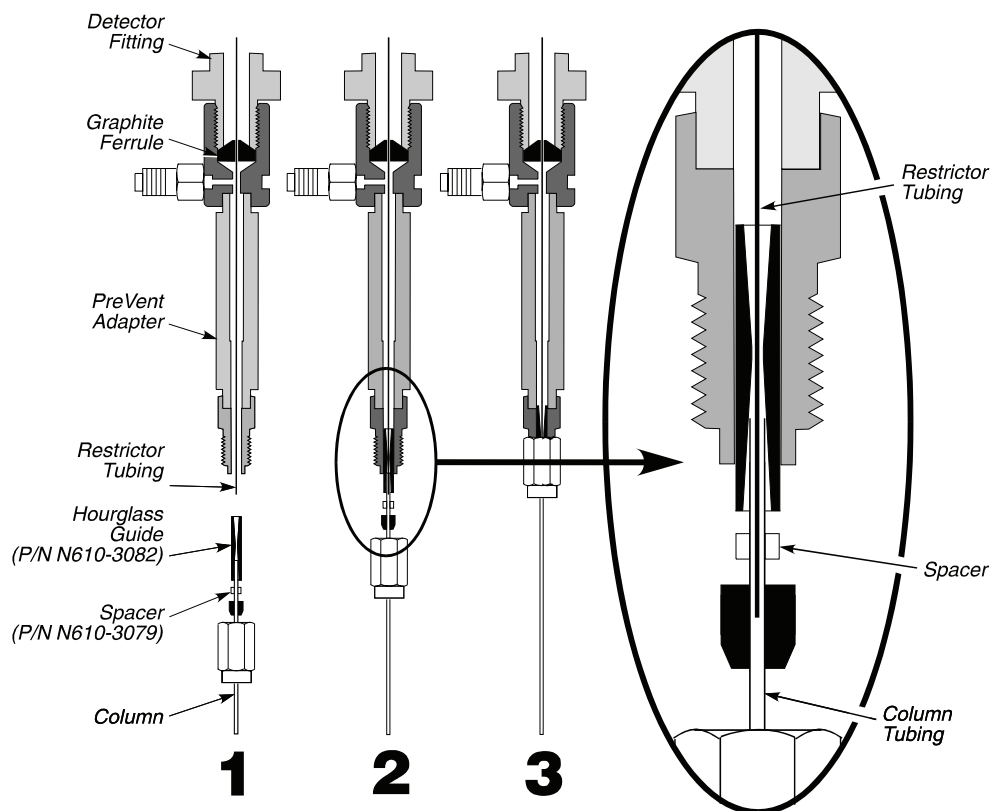


**Figure 7-13. Example of a good tubing cut and bad cuts.**

4. Insert the column end into the hourglass guide (P/N N610-3082).
5. Align the end of the restrictor tubing that protrudes from the PreVent adapter with the open end of the hourglass guide. Then carefully push the hourglass guide, with the attached column, up into the PreVent adapter as shown in the following figure. Notice that the restrictor tubing fits inside the column.

**NOTE:** Slightly twist the column as you push the hourglass guide into the PreVent adapter.

6. Loosely tighten the column nut.
7. Carefully push the column up into the PreVent adapter until it bottoms in the detector. Then pull the column back 3 mm.
8. Use two 1/4-inch wrenches to tighten the nut. **DO NOT OVERTIGHTEN THE NUT!**

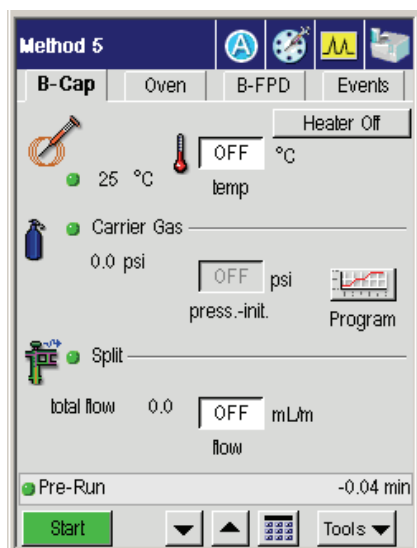


**Figure 7-14. Connecting a column to the PreVent detector adapter.**

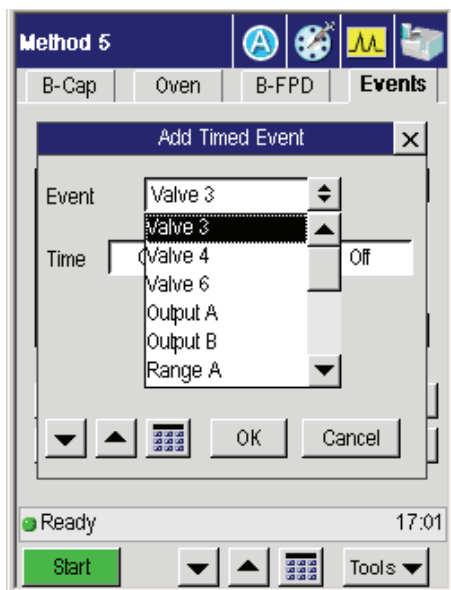
Prevent

## D. Leak-Check the System

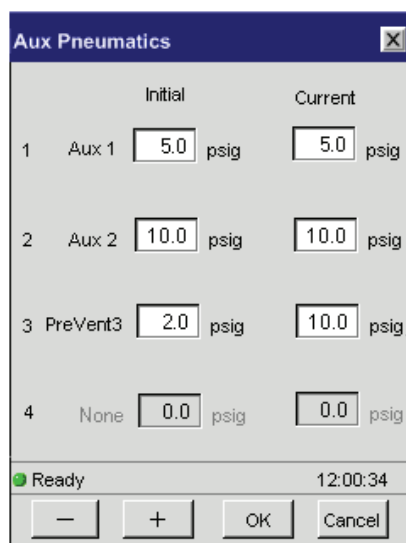
1. Turn on the GC. When the Clarus 600 GC is turned on, it is set to the initial default settings.
2. From the System Status screen touch the injector to get to the following screen. Turn the detector temperature off.



3. From the Events tab turn off the mid-point carrier gas supply by opening **Valve 3**.

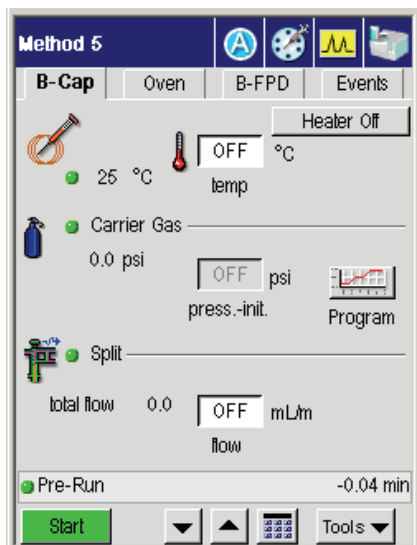


4. Allow the system to equilibrate for several minutes, then view the Aux 1 pressure, view the Aux Gas button and view the current psig.



## Prevent

- Go back to the inject screen to view the pressure read out on the carrier gas which should be at 0 psig.



- After approximately 5 minutes, if no leaks exist, the **Aux 1** reading should be stable at approximately 75 psi. If it is low, then leak-check the fitting connections.
- If you suspect a leak, test the connections for leaks using a 50/50 mixture of isopropanol/water or an electronic leak detector. To prevent contaminating the system, DO NOT use a soap solution for leak testing. Tighten all leaking connections.

If you detect a leak, check and tighten the fittings on the mid-point pressure tee and/or the connections on the rear of the GC.

**CAUTION** *Do not overtighten the column nut. If this fitting has a persistent leak, replace the graphite/Vespel ferrule.*

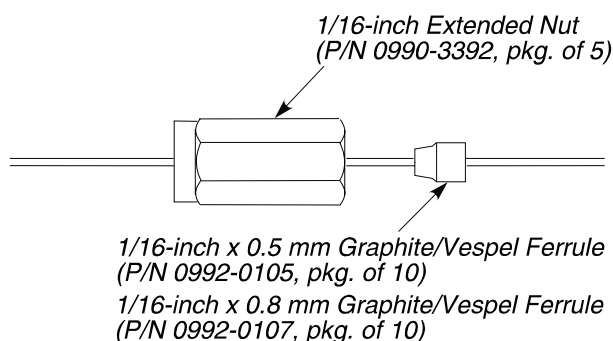


## E. Connect the Column to the Injector

**CAUTION**

*Make certain that no part of the column touches the walls or bottom of the oven.*

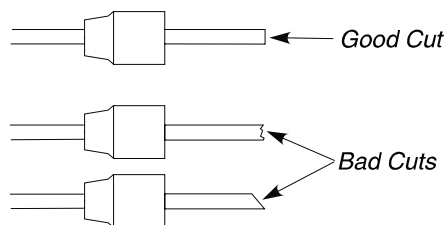
1. Insert a 1/16-inch extended nut (P/N 0990-3392, pkg. of 5) and graphite/Vespel ferrule (1/16-inch x 0.5 mm P/N 0992-0105, pkg. of 10 for 0.25/0.32 i.d. column or a 1/16-inch x 0.8 mm P/N 0992-0107, pkg. of 10 for a 0.53 mm i.d. column) over one end of the column.



**Figure 7-15. Extended nut and graphite/Vespel ferrule.**

2. Cut about 1 cm (3/8 inch) from the end of the column tubing using a wafer scribe (P/N N930-1386, pkg. of 10 scribes) or other column cutting tool. Break off the tubing at the score mark making sure that the break is clean and square. Examine the cut with a magnifying glass and compare it to the following figure:

*Prevent*



**Figure 7-16. Example of a good column cut and bad cuts.**

3. Position the column nut on the column so that the back of the nut is 4.4 cm to 5.1 cm (1 3/4 inches to 2 inches) from the end of the column for the CAP injector.

**or**

Position the column nut on the column so that the back of the nut is 1 1/2 inches to 1 3/4 inches from the end of the column for the PSS injector.

4. Using typewriter "white out" or a felt-tipped pen, make a mark on the column just beyond the back edge of the column nut (see Figure 6-10).

**CAUTION**

*To avoid contaminating the system, make certain that the nut and ferrule do not contact the mark on the column.*

5. Locate the capillary injector fitting inside the oven.

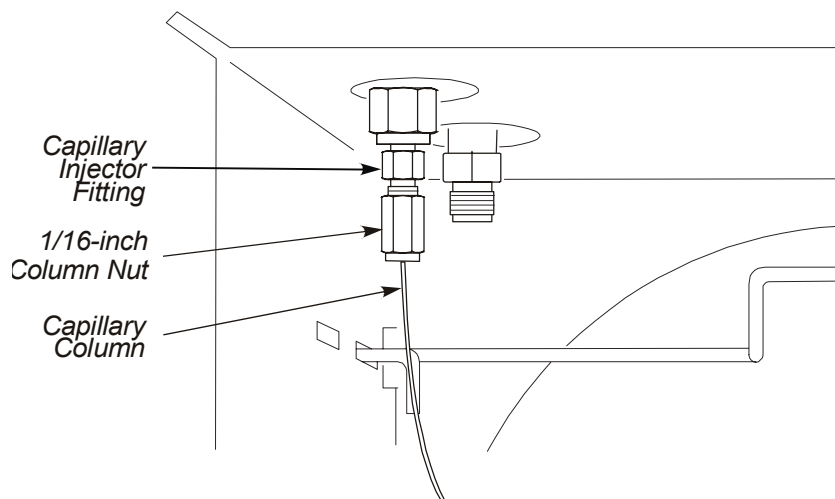
Refer to the following figures to connect the column to the Cap or PSS injector.

**CAUTION**

*The injector terminates in a 1/16-inch fitting. This fitting is fragile. To preserve the integrity of the fitting, carefully connect the nut to prevent cross-threading the fitting and/or overtightening the nut on the fitting. You can also preserve the integrity of the fitting by allowing the injector to cool before connecting a nut.*

**CAUTION**

*Do not overtighten column nuts. Overtightening can cause damage to the ferrule and/or column.*



**Figure 7-17. Capillary column attached to a CAP injector fitting.**

Prevent

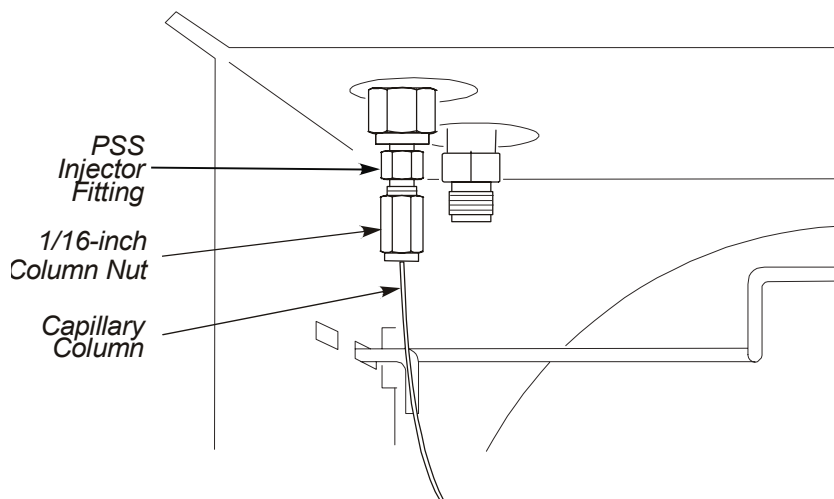
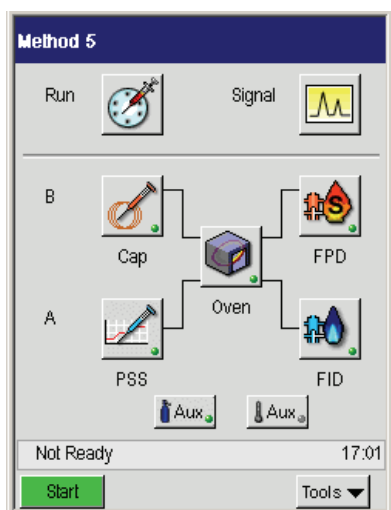


Figure 7-18. Capillary column attached to PSS injector fitting.

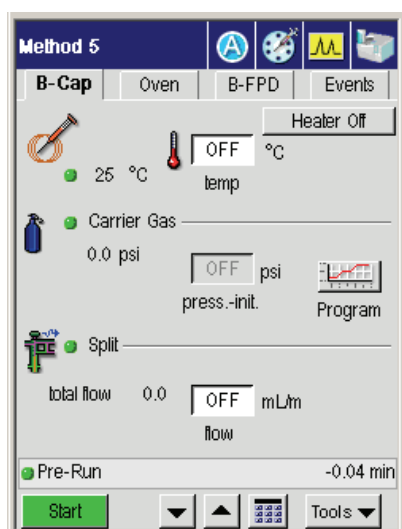
## F. Set the Initial Pressures




To set the mid-point pressure:

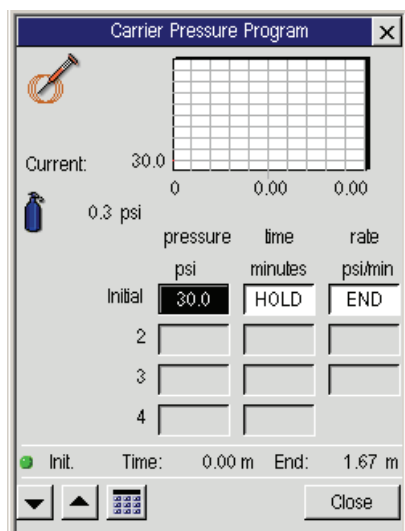
1. From the System Status screen select either A or B injector (the example here shows the B cap injector ready for setting the carrier gas).



- In the following window touch the Program Graph screen under the Carrier Gas field.

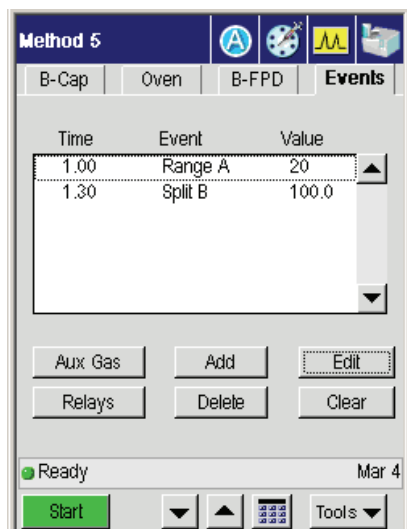


- Using the up and down arrow or keypad    buttons enter the **initial** value of 30 psig for the Carrier gas.

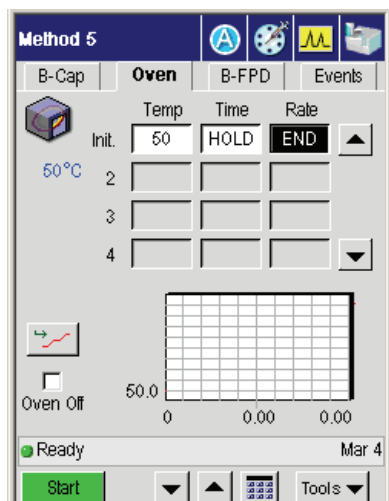


*Prevent*

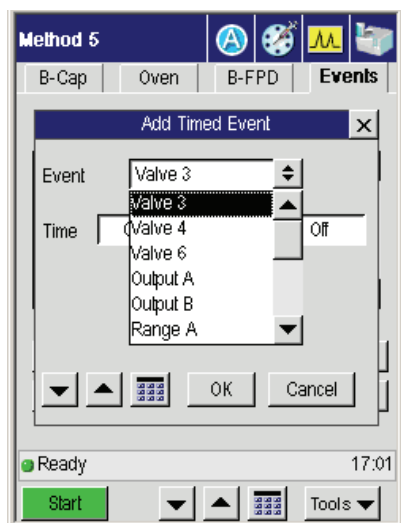
4. Press **Close** to go back to the previous screen (as seen in step 2).
5. In the **Events** tab touch the **Split** event and then press **Edit**. Set Split B to 100 mL/min.



6. From the **Oven** tab set the oven temperature to its highest programmed temperature.



- From the Events tab turn off the mid-point carrier gas supply by closing **Valve 3**.



- Allow the system to equilibrate for several minutes, then view the **Aux 1** pressure, view the Aux Gas button and view the current psig.

## Prevent

		Initial	Current
1	Aux 1	5.0 psig	5.0 psig
2	Aux 2	10.0 psig	10.0 psig
3	PreVent3	2.0 psig	10.0 psig
4	None	0.0 psig	0.0 psig

Ready 12:00:34

[-] [+] [OK] [Cancel]

**Note:** *If the Aux 1 pressure is very low, then the graphite ferrule that holds the restrictor inside the PreVent adapter may be leaking. Remedy this by tightening the adapter on the injector and observe if there is an improvement.*

- Adjust the pressure until the **Aux** pressure is maintained at about 1.5 psig below the required column inlet pressure. Wait a few minutes for the system to stabilize to ensure that no further changes are required.
- Open **Valve 3** and set the **Aux 1** pressure to the required column inlet pressure; for example, 1.5 psig higher than the Aux 1 reading established in step 9 above. This ensures a positive gas flow through the Aux 1 supply to the mid-point tee.

### CAUTION

*Valve 3 MUST remain open (ON) during PreVent operation.*

Condition the system to ready it for an analysis.



## **G. Condition the System**

1. Write down the pressures established in step **F** for **Press 1** and **Aux 1**.
2. Set **Press 1** to 2 psig, **Split 1** to 100 mL/min, and **Aux 1** to 80 psig.
3. If you are using a PSS, set its temperature to the maximum programmed temperature. Set the oven to the maximum programmed temperature.
4. Allow the system to bake for several hours — overnight if possible.

Shorter conditioning times may be possible, but that depends on the condition of the PreVent components and condition of the column. Cool the oven (and if you are using a PSS injector, cool the injector.)

5. Reset the initial settings of **Press 1** and **Aux 1**.

The system is now ready to use for an analysis.

## **Installing PreVent on a Clarus MS Detector**

This procedure describes how to install PreVent on a Clarus MS detector.

### **Summary**

The following steps summarize how to install a PreVent injector adapter to a Clarus MS detector and make it ready for an analysis:

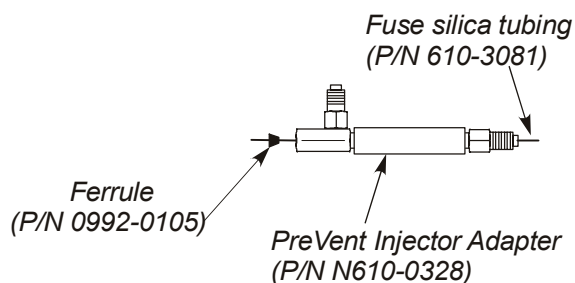
- A. Prepare the Clarus MS
- C. Install the restrictor in the Clarus MS Transfer Line.
- D. Install the PreVent injector adapter.
- E. Connect the column to the PreVent injector adapter.
- F. Connect the Transfer Line to the Clarus MS
- G. Leak-check the system.
- H. Connect the column to the injector.
- I. Set the initial pressures.
- J. Condition the system

### **A. Prepare the Clarus 600 MS**

1. Install the Clarus 600 MS and Clarus 600 GC according to the instructions given in the Clarus 600 Mass Spectrometer and Clarus 600 GC installation manuals.
2. If a column is installed, cool the Gas Chromatograph oven, the Clarus MS ionizer and the Clarus MS transfer line to ambient temperature. Vent the detector and remove the column from the transfer line.

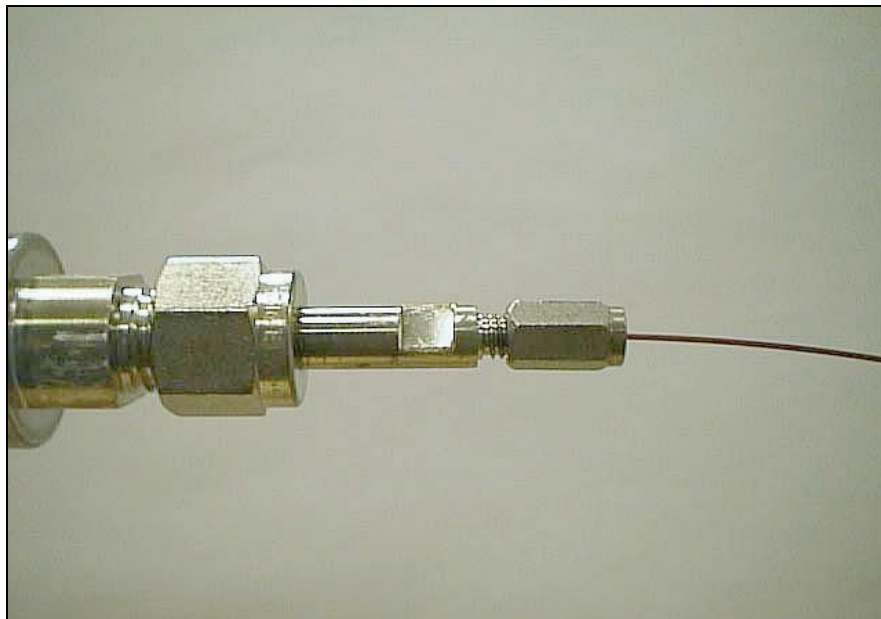
## **B. Install the PreVent Injector Adapter in the TMMS Transfer Line**

1. Cut a 40cm length of the 0.075mm i.d. fused silica tubing (P/N 610-3081). Make sure that the two ends are cut clean and square. Wipe the outside of the tubing with a tissue dampened with methanol.
2. Feed the fused silica tubing through the PreVent *injector* adapter (P/N N610-0328) and a graphitized Vespule ferrule (P/N 0992-0105) as shown in the following figure.



**Figure 7-19. Feeding the fused silica tubing through the injector adapter.**

3. Introduce the fused silica tubing into the Clarus MS transfer line until it reaches a stop. Loosely screw the PreVent adapter onto the 1/16" threaded union at the end of the Clarus MS transfer line as shown in the following figure.

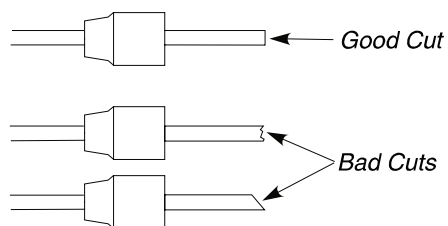


**Figure 7-20. Normal connection of column to Clarus MS transfer line.**

4. Withdraw the fused silica tubing by about 5mm and using two 1/4 inch wrenches tighten up the PreVent adapter on the union at the end of the Clarus MS transfer line.
5. Cut the fused silica tubing so that about 7mm is exposed beyond the PreVent adapter as shown in the following figure. The cut should be clean and square.

**CAUTION**

*Take care when working near the exposed end of the fused silica tubing. It can be easily broken off.*



**Figure 7-21. Example of a good column cut and bad cuts.**

Refer to the following figure as you perform the following procedure.

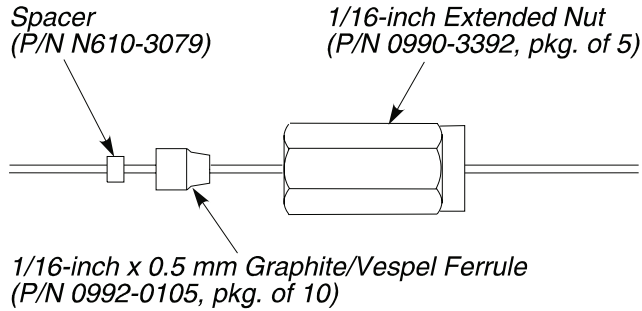
6. Locate the stainless steel tubing (that supplies the mid-point pressure) inside the oven.
7. Insert a 1/16-inch nut (P/N 0990-3392) and a graphite/Vespel ferrule (P/N 0992-0107) over the end of this tubing.
8. Measure and mark the tubing 1/2-inch from the back of the nut.
9. Insert the tubing into side arm on the PreVent adapter, keeping the mark just behind the nut. Then tighten the nut and ferrule fingertight.
10. Make a leak-free seal by using a two 1/4-inch wrenches to tighten the PreVent adapter on to the union another 1/4 to 1/2 turn.

### **C. Connect the Column to the PreVent Injector Adapter**

1. Place the column on the hanger so that no part of the column touches the bottom or sides of the oven.
2. Insert a 1/16-inch extended nut (P/N 0990-3392, pkg. of 5) a graphite/Vespel ferrule (P/N 0992-0105, pkg. of 10), and a spacer (P/N N610-3079) over one end of the column.

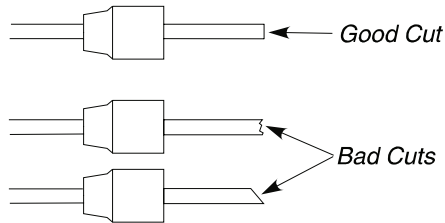
**CAUTION**

*Do not use a graphite ferrule since it will extrude up into the body of the PreVent adapter.*



**Figure 7-22. Extended nut, graphite/Vespel ferrule, and spacer.**

3. Cut about 1 cm (3/8 inch) from the end of the column tubing using a wafer scribe (P/N N930-1386, pkg. of 10 scribes) or other column cutting tool. Break off the tubing at the score mark making sure that the break is clean and square. Examine the cut with a magnifying glass and compare it to the following figure:



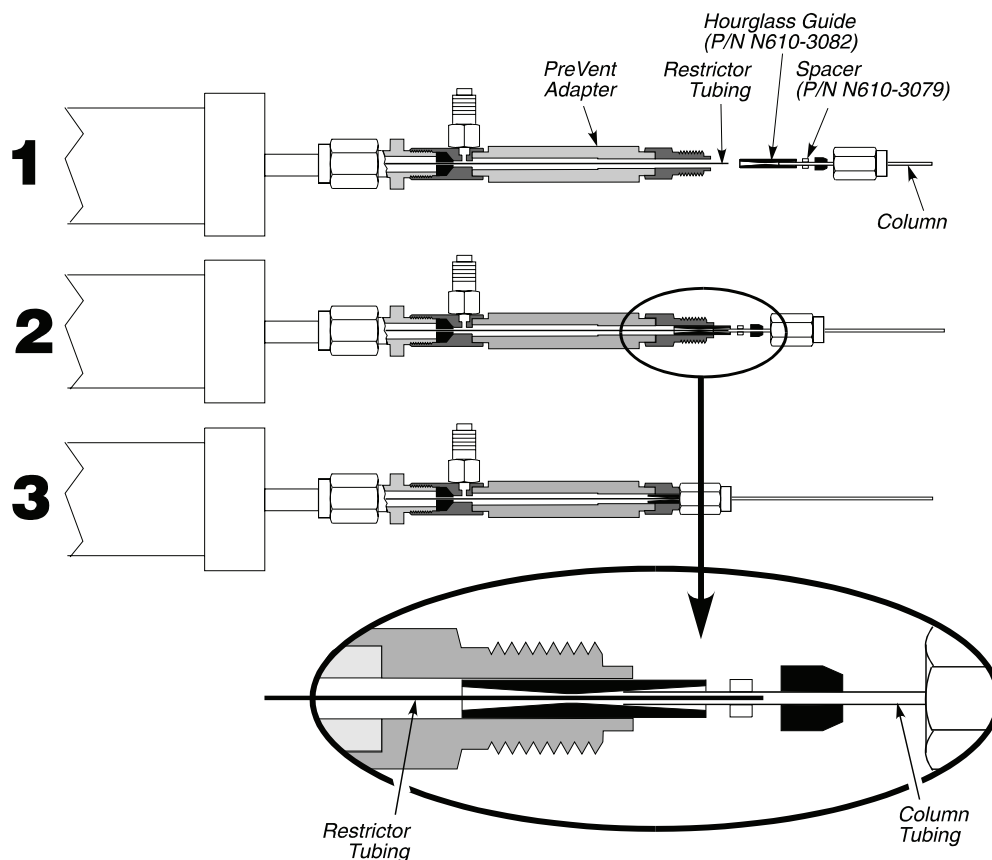
**Figure 7-23. Example of a good tubing cut and bad cuts.**

4. Insert the column end into the hourglass guide (P/N N610-3082).

5. Align the end of the restrictor tubing that protrudes from the PreVent adapter with the open end of the hourglass guide. Then carefully push the hourglass guide, with the attached column, into the PreVent adapter as shown in the following figure, step 2. Notice that the restrictor tubing fits inside the column.

**NOTE:** *Slightly twist the column as you push the hourglass guide into the PreVent adapter.*

6. Loosely connect the column nut to the PreVent adapter.
7. Carefully push the column up and into the PreVent adapter until it bottoms in the adapter. Then pull the column back 3 mm.
8. Use two 1/4-inch wrenches to tighten the nut. **DO NOT OVERTIGHTEN THE NUT!**



**Figure 7-24. Connecting a column to the PreVent adapter in a Clarus MS system.**

#### **D. Connect the Transfer Line to the Clarus MS**

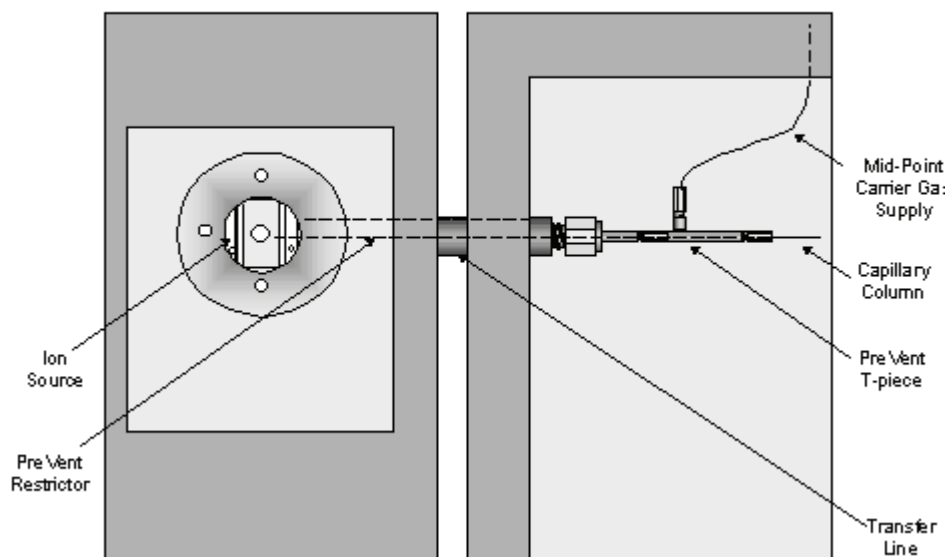
Install the PreVent device into the Clarus MS as shown in Figure 7-25.

Take the PreVent 'injector' T-piece and screw it onto the 1/16" fitting at the end of the standard Clarus MS transfer line. Be sure the fused silica restrictor is inserted through it, through the heated transfer line and directly into the Clarus MS ion source.



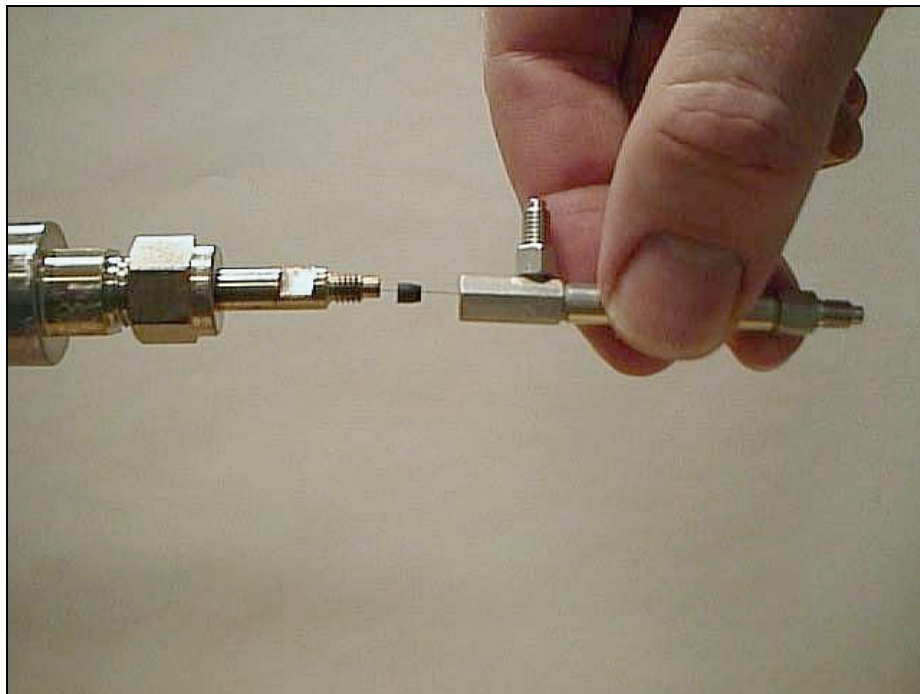
Ensure that the end of the restrictor is exposed beyond the T-piece to enable the column to be threaded over it with the aid of an hourglass guide. Figures 7-26 through 7-28 show details of the installation.

Figure 7-30 shows a detail of the column-restrictor interface. Because the restrictor is inserted directly *into* the end of the capillary column, there are no issues with active sites or dead volumes. Therefore an optimum chromatographic performance is assured.

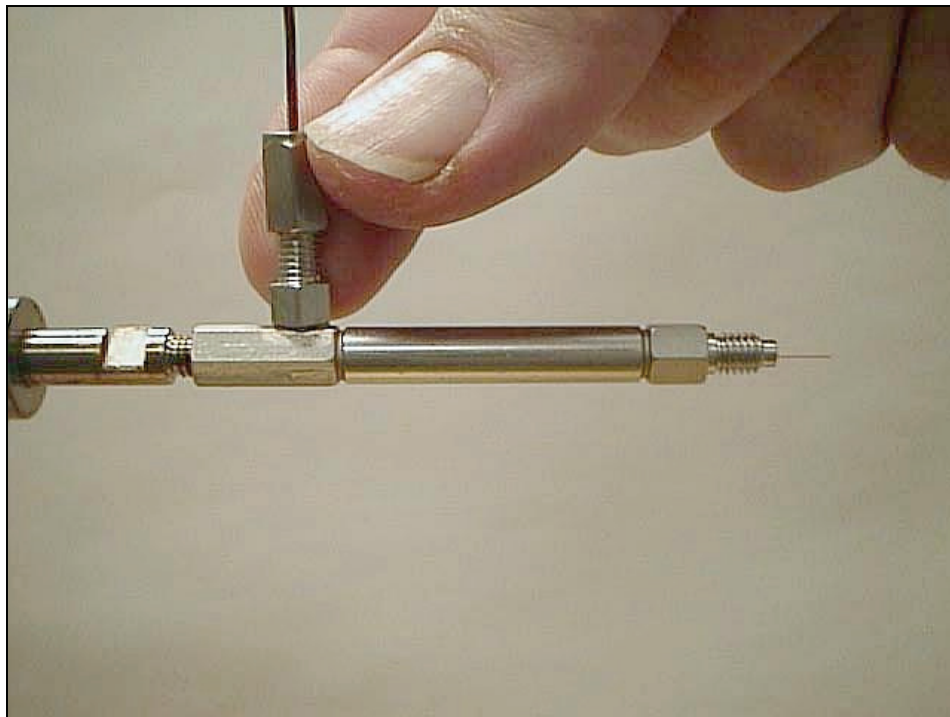


**Figure 7-25. Diagram showing PreVent device installed into the Clarus MS detector (The Clarus 600 MS is the figure on the left and the Clarus 600 GC is the figure on the right).**

*Prevent*

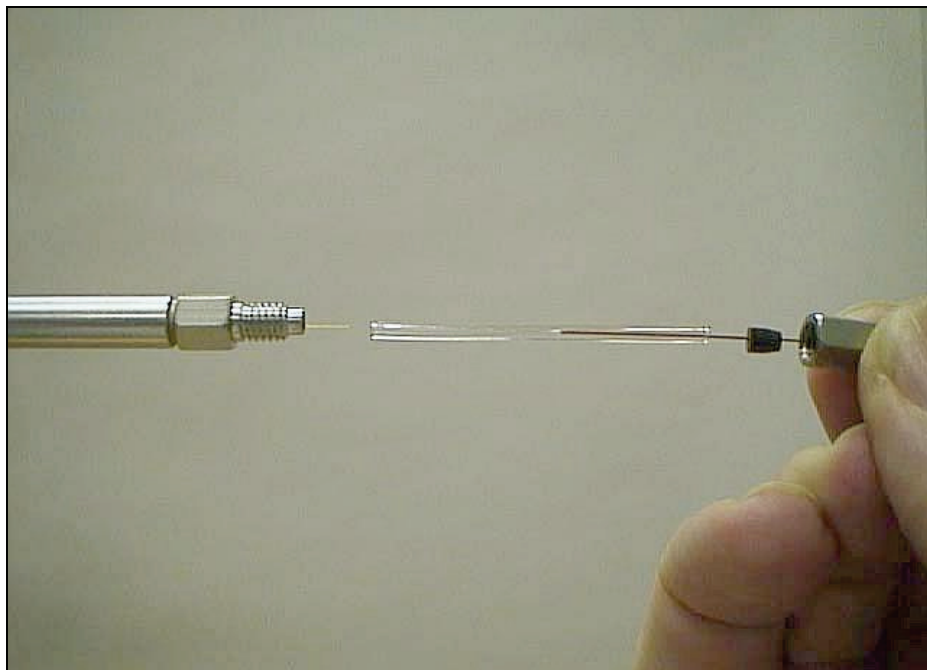


**Figure 7-26. Column is detached and PreVent adapter is attached to Clarus MS transfer line with fused silica restrictor pushed through into ionizer.**

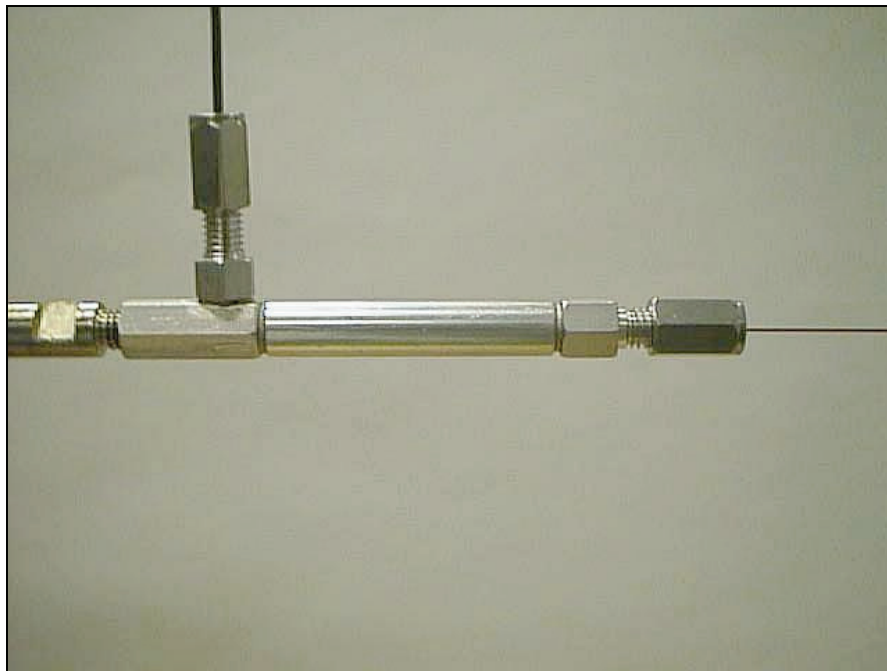


**Figure 7-27. Restrictor is sealed within adapter with end protruding. Midpoint gas supply is connected to port on adapter.**

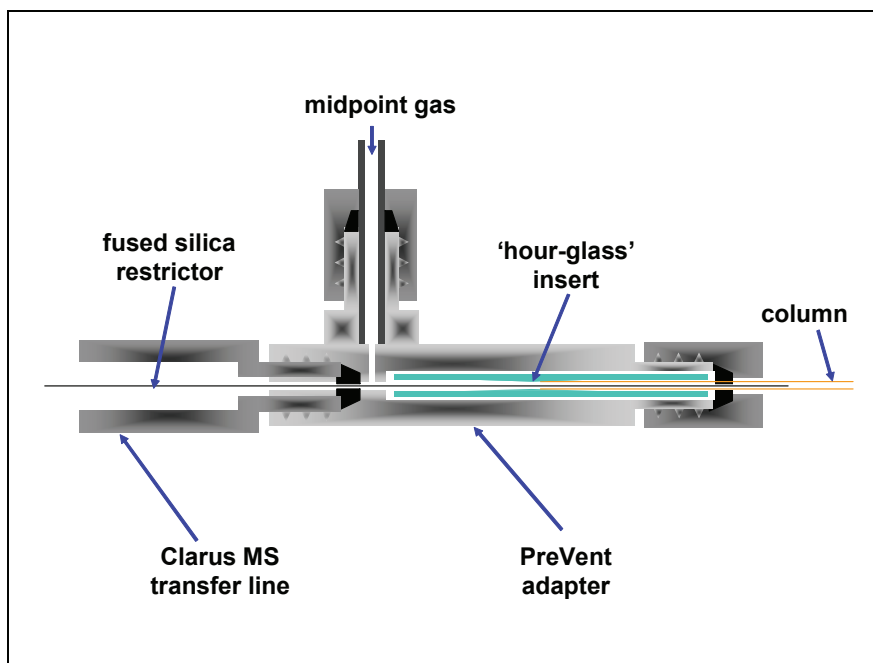
Prevent



**Figure 7-28. Hourglass guides restrictor *into* capillary column**



**Figure 7-29. Installation completed.**



**Figure 7-30. Detail of column-restrictor interface at end of Clarus MS transfer line.**

- Once installed, this PreVent configuration will allow the capillary column to be back-flushed and so offers the following benefits:
- Allows injector maintenance without having to cool or vent the vacuum on the mass spectrometer.
- Allows a degraded section of column to be removed from its inlet without the need to cool or pump-down the detector.
- Facilitates easier column replacement.
- Reduces risk of air entering the detector in the event of a leak or column breakage.
- Prevents column bleed from entering the detector while conditioning or when the system is idle.

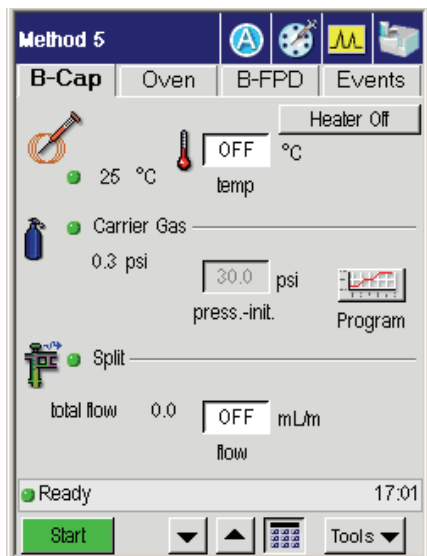
- Allows the injector liner and quartz wool packing to be deactivated *in-situ*.
- Reduces analysis times by eliminating the need for extensive temperature programming to elute unwanted less volatile sample residue from the column.
- Increases the life of a column by eliminating temperature programming completely.
- Removes the potential of contaminants before they reach the detector.

It will also have the added benefit of maintaining a constant flow of carrier gas into the MS detector in both forward flow and backflush modes during temperature programming.

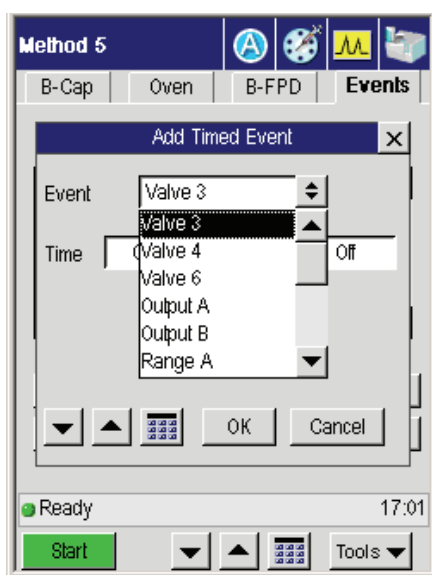
### ***E. Leak-Check the System***

- 1 Turn on the GC. When the Clarus 600 GC is turned on, it is set to the initial default settings.
- 2 From the System Status screen touch the injector to get to the following screen. Touch the numeric field until it turns black and use the plus minus buttons to get the proper split flow of 100 and a ratio of 100.

Prevent

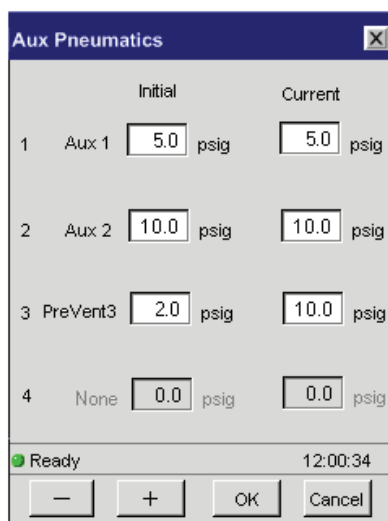


3. Select **Valve 3** (or valve 4 if *PreVent* is installed in channel 2) from the Event drop down menu.

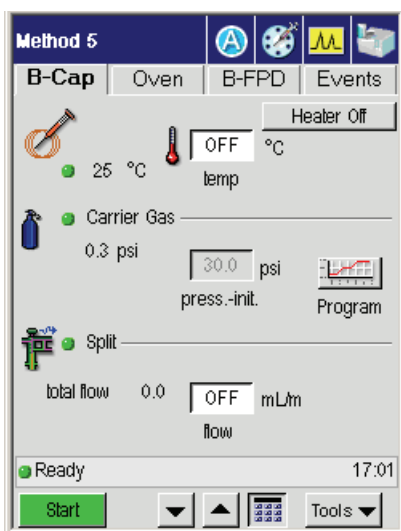




- Allow the system to equilibrate for several minutes, then view the Aux 1 pressure, view the Aux Gas button and view the current psig.



- Go back to the inject screen to view the pressure read out on the carrier gas which should be at 0 psig.



*Prevent*

6. Set the injector pressure to 75 psig.
7. After approximately 5 minutes, if no leaks exist, the **Aux 1** reading should be stable at approximately 75 psi. If it is low, then leak-check the fitting connections.
8. If you suspect a leak, test the connections for leaks using a 50/50 mixture of isopropanol/water or an electronic leak detector. To prevent contaminating the system, DO NOT use a soap solution for leak testing. Tighten all leaking connections.

If you detect a leak, check and tighten the fittings on the mid-point pressure tee and/or the connections on the rear of the GC.

**CAUTION**

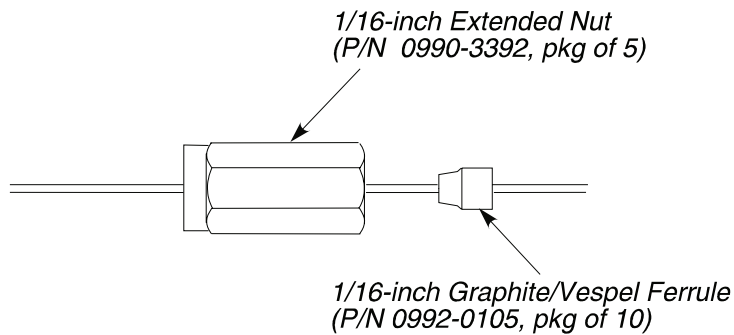
*Do not overtighten the column nut. If this fitting has a persistent leak, replace the graphite/Vespel ferrule.*

**F. Connect the Column to the Injector**

**CAUTION**

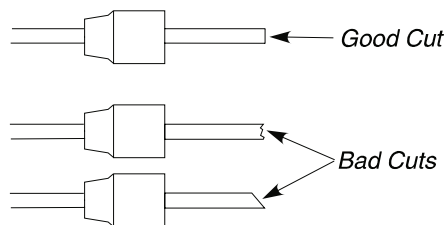
*Make certain that no part of the column touches the walls or bottom of the oven.*

1. Insert a 1/16-inch extended nut (P/N 0990-3392, pkg of 5) and graphite/Vespel ferrule (P/N 0992-0105, pkg of 10) over one end of the column.



**Figure 7-31. Extended nut and graphite/Vespel ferrule.**

2. Cut about 1 cm (3/8 inch) from the end of the column tubing using a wafer scribe (P/N N930-1386, package of 10 scribes) or other column cutting tool. Break off the tubing at the score mark making sure that the break is clean and square. Examine the cut with a magnifying glass and compare it to the following figure:



**Figure 7-32. Example of a good column cut and bad cuts.**

3. Position the column nut on the column so that the back of the nut is 4.4 cm to 5.1 cm (1 3/4 inches to 2 inches) from the end of the column for the CAP injector.

**or**

Position the column nut on the column so that the back of the nut is 1 1/2 inches to 1 3/4 inches from the end of the column for the PSS injector.

4. Using typewriter "white out" or a felt-tipped pen, make a mark on the column just beyond the back edge of the column nut.

Prevent

**CAUTION**

*To avoid contaminating the system, make certain that the nut and ferrule do not contact the mark on the column.*

5. Locate the capillary injector fitting inside the oven. Refer to the following figures to connect the column to the Cap or PSS injector.

**CAUTION**

*The injector terminates in a 1/16-inch fitting. This fitting is fragile. To preserve the integrity of the fitting, carefully connect the nut to prevent cross-threading the fitting and/or overtightening the nut on the fitting. You can also preserve the integrity of the fitting by allowing the injector to cool before connecting a nut.*

**CAUTION**

*Do not overtighten column nuts. Overtightening can cause damage to the ferrule and/or column.*

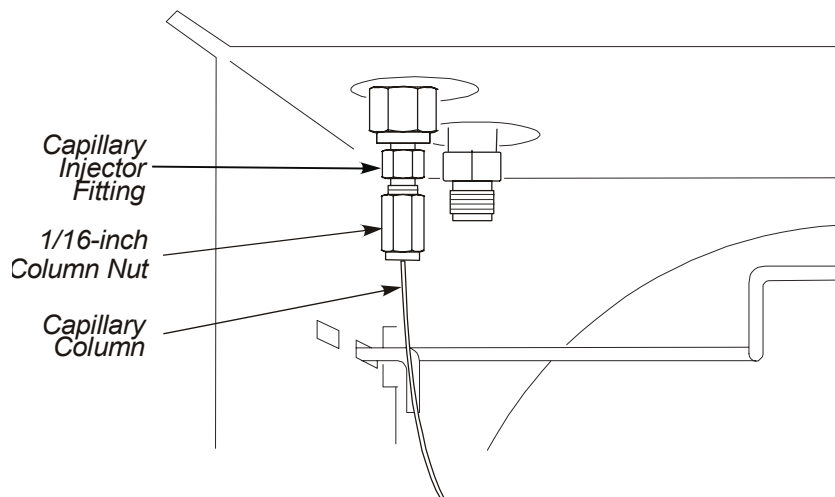


Figure 7-33. Capillary column attached to a CAP injector fitting.

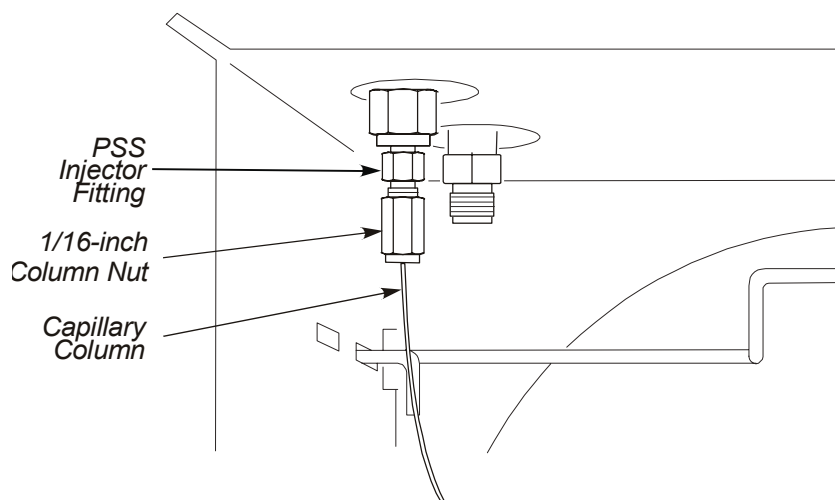
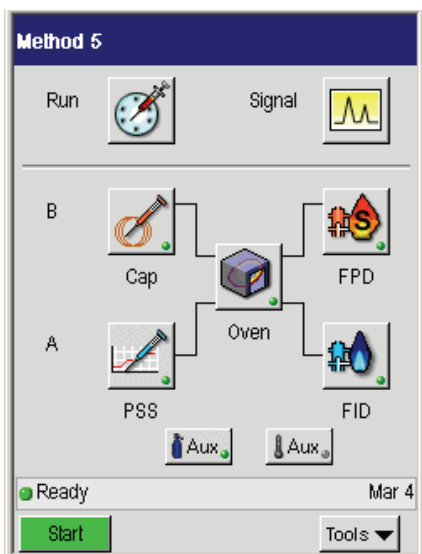


Figure 7-34. Capillary column attached to PSS injector fitting.

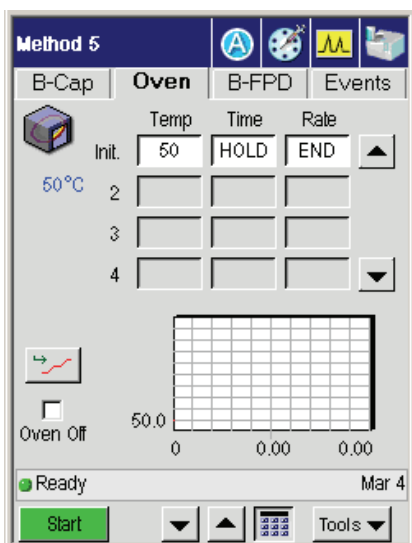
## G. Set the Initial Pressures

1. From the System Status screen select the **oven** icon.

Prevent

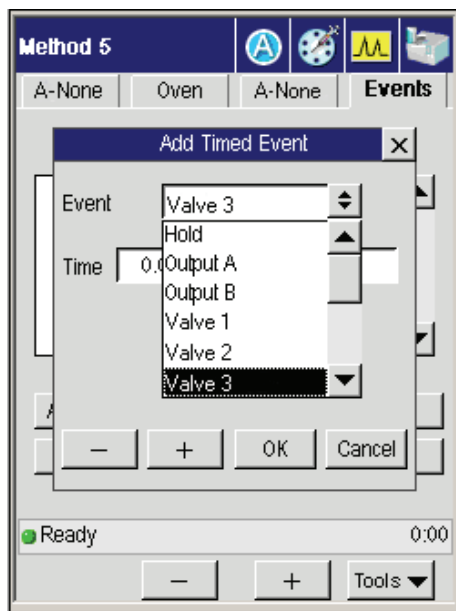


2. Set the oven to the lowest programmed temperature.



3. Set the Clarus MS transfer line to its operating temperature.
4. From the Events tab turn off the mid-point carrier gas supply by closing **Valve 3**.

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5. Start the Clarus MS vacuum
6. See the gas pressure to 30 psi.

## Prevent

		Initial		Current	
1	Aux 1	5.0	psig	5.0	psig
2	Aux 2	10.0	psig	10.0	psig
3	PreVent3	2.0	psig	10.0	psig
4	None	0.0	psig	0.0	psig

Ready 12:00:34

[-] [+] [OK] [Cancel]

**Note:** *If the aux 1 pressure is very low, then the graphite ferrule that holds the restrictor inside the PreVent adapter may be leaking. Remedy this by tightening the adapter on the injector and observe if there is an improvement.*

7. Adjust the pressure until the **Aux** pressure is maintained at about 1.5 psig below the required column inlet pressure. Wait a few minutes for the system to stabilize to ensure that no further changes are required.
8. Open **Valve 3** and set the **Aux 1** pressure to the required column inlet pressure; for example, 1.5 psig higher than the Aux 1 reading established in step 9 above. This ensures a positive gas flow through the Aux 1 supply to the mid-point tee.

### CAUTION

*Valve 3 MUST remain open (ON) during PreVent operation.*

Condition the system to ready it for an analysis.



## **H. Condition the System**

1. Write down the pressures established for **Press 1** and **Aux 1** in step **I**.
2. Set **Press 1** to 2 psig, **Split 1** to 100 mL/min, and **Aux 1** to 80 psig.
3. Set oven to maximum programmed temperature.

If you are using a PSS, set its temperature to the maximum programmed temperature.

4. Allow the system to bake for several hours — overnight if possible.

Shorter conditioning times may be possible, but that depends on the condition of the PreVent components and condition of the column.

5. Cool the oven (and if you are using a PSS injector, cool the injector.)
6. Reset the initial settings of **Press 1** and **Aux 1**.

The system is now ready to use for an analysis.

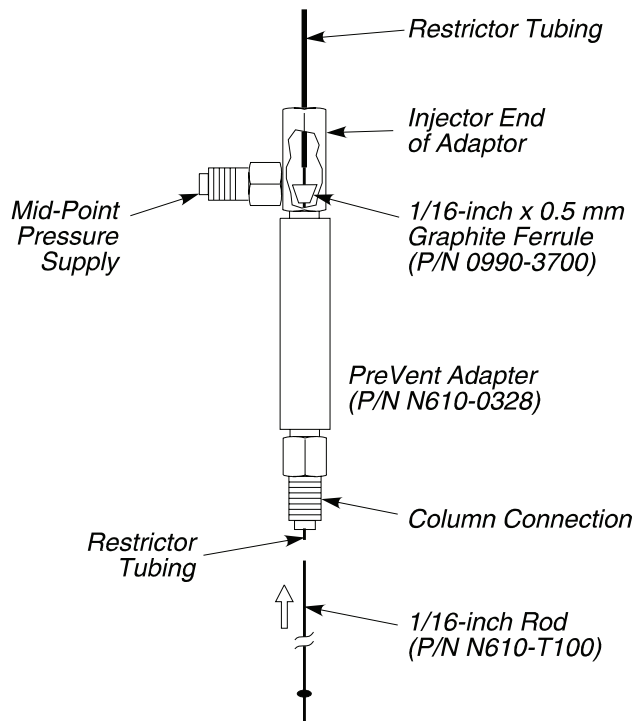
## **I. Replacing the Restrictor**

To replace a restrictor in the PreVent injector or detector adapter:

1. Turn off the GC oven and detector and wait until they are cool to the touch.
2. Turn off all gases.
3. Using a 1/4-inch wrench, remove the 1/16-inch column nut and the column from the PreVent adapter.
4. Using two 1/4-inch wrenches, remove the nut and mid-point pressure line from the side arm of the PreVent adapter.
5. Using the appropriate wrenches, remove the PreVent adapter from either the injector or detector.

## Prevent

- Use two 1/4-inch wrenches to remove the PreVent adapter from the injector fitting.
  - Use two 7/16-inch wrenches to remove the PreVent adapter from the detector fitting.
6. Insert a 1/16-inch rod (P/N N610-T100) in the column connection end of the PreVent adapter and push the restrictor tubing and ferrule of the injector end.
  7. Install a new restrictor by referring to the appropriate procedure in this chapter.



**Figure 7-35. Removing a restrictor from a PreVent adapter.**

## ***PreVent Operating Techniques***

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### ***Summary of the PreVent Techniques***

Table 7-3 summarizes the various PreVent techniques and the hardware configurations that support them.

- Column Isolation
- Solvent Purge
- Large Volume Injection
- Sample Residue Purge
- Time Saver

**Table 7-3. Summary of PreVent Techniques and Supporting Configurations**

<b>Technique</b>	<b>Split/Splitless</b>	<b>PSS</b>	<b>Restrictor in Injector</b>	<b>Restrictor in Detector</b>
Column Isolation	Yes	Yes	Yes	Yes*
Solvent Purge	No	Yes	Yes	Yes
Large Volume Injection	No	Yes	Yes	Yes
Sample Residue Purge	No	Yes	Yes**	Yes
Time Saver	Yes	Yes	No	Yes
* Cannot be used while chromatography is in progress				
** Not recommended				

Table 7-3 demonstrates the advantage of the PSS injector over the conventional Split/Splitless injector in that all the PreVent techniques are supported by this one injector. Several of these techniques are possible with a single restrictor configuration. If a different configuration is required, changing the restrictor type is a simple operation requiring only a few minutes to perform.

## Column Isolation Technique

This technique uses the Injector Restrictor Configuration to fully isolate the chromatographic column from actions occurring within the injector. Figures 7-36 and 7-37 illustrate the principle.

For sample introduction, the inlet pressure (P1) is set up to be higher than the mid-point pressure (P2). P1 and P2 are set as described in “Installing PreVent on the Injector” in this chapter. P2 represents the pressure at the column inlet. Carrier gas will now flow from the injector, through the restrictor and into the column, carrying the vaporized sample with it. The sample may be injected manually using a standard syringe or by autosampler. The injection temperature and split vent operation are set as for normal split or splitless injection techniques. The pressures are not adjusted until the injector needs to be isolated. To isolate the column, Press 1 (P1) is simply reduced to below that of Aux 1 (P2) causing the carrier gas to flow backwards from the mid-point ‘T’, through the restrictor, into the injector and out through the split vent. It is important to ensure that the split vent is open (for example, 50 mL/min) during this step or the technique will not work.

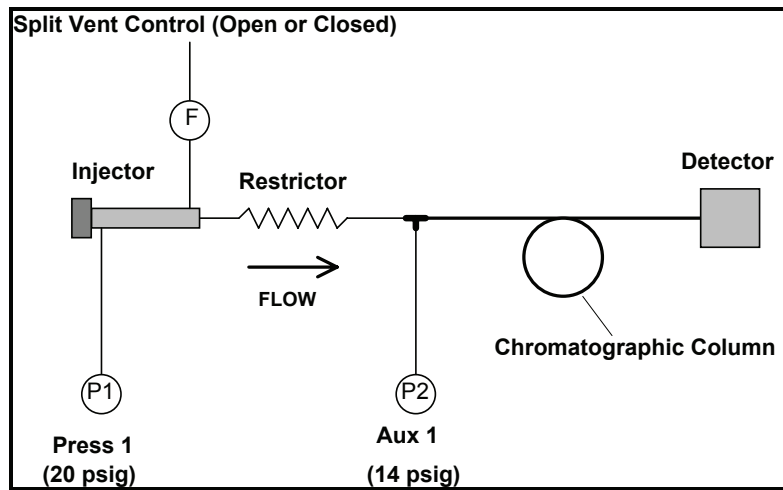
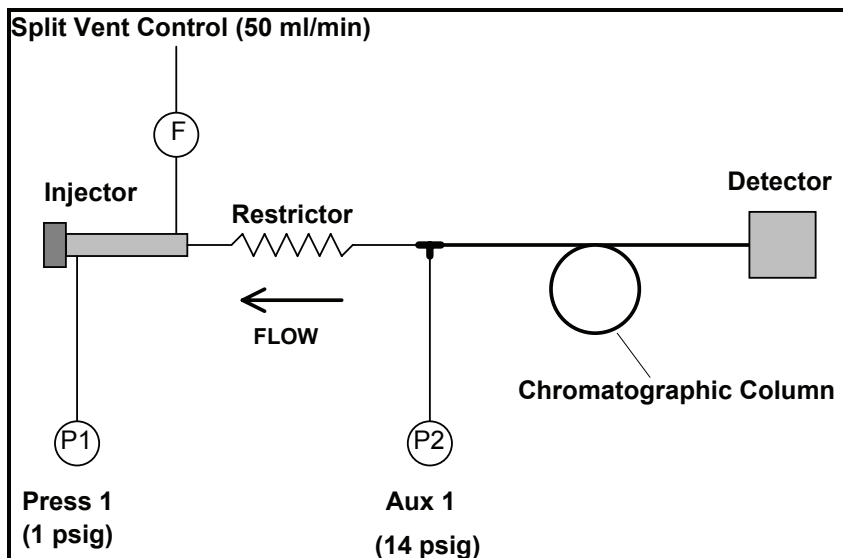


Figure 7-36. Normal operation.



**Figure 7-37. Column Isolation Mode.**

P1 should not be turned off completely, but to a level (for example, 1.0 psig) that maintains forward flow of carrier gas through the liner to prevent the build-up of contamination within the liner while the restrictor is being backflushed and also to maintain a purge flow out of the injector inlet while it is being serviced with the septum cap removed. The reduction in P1 is not normally included as a timed event in the GC method as this will not be routinely adjusted during each run.

The Column Isolation Mode can be used to support the following actions:

- Septum exchange and conditioning even during chromatography
- Liner exchange and conditioning even during chromatography
- *In situ* chemical deactivation of the liner even during chromatography
- Column and detector protection while the instrument is idle

Column Isolation Mode is also possible with the restrictor fitted to the detector as shown in Figures 7-38 and 7-39. In this instance, however, chromatography is

Prevent

not possible during column isolation although it now would be possible to condition a column without the effluent reaching the detector.

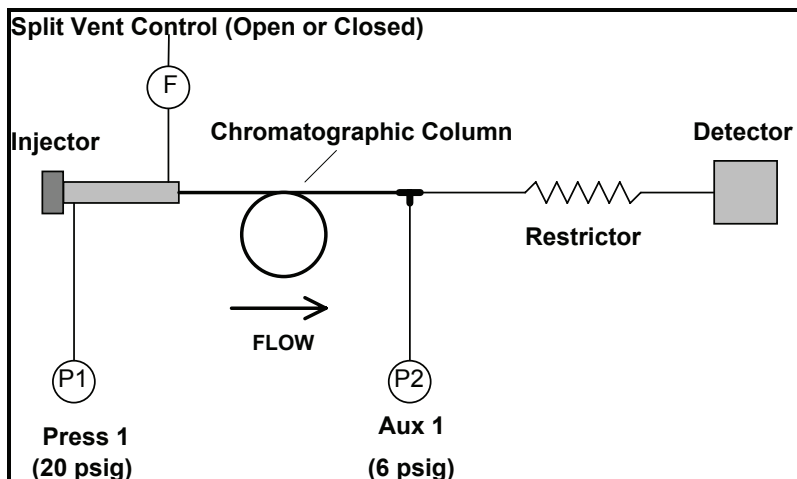


Figure 7-38. Normal operation.

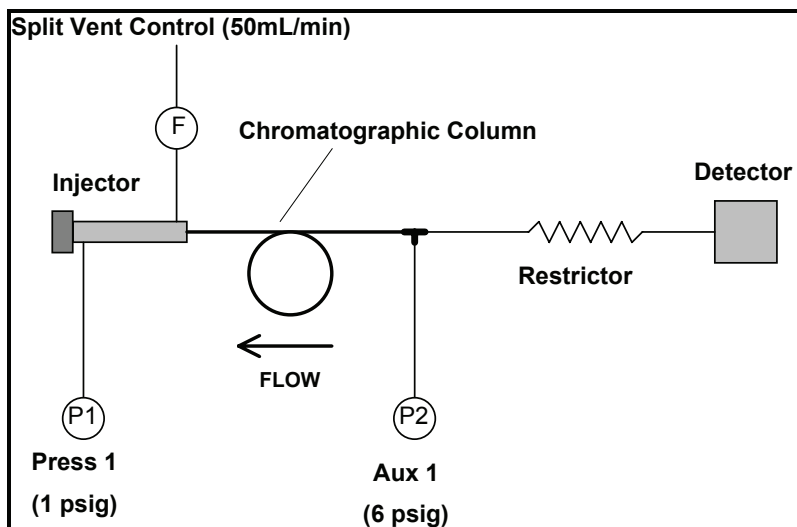
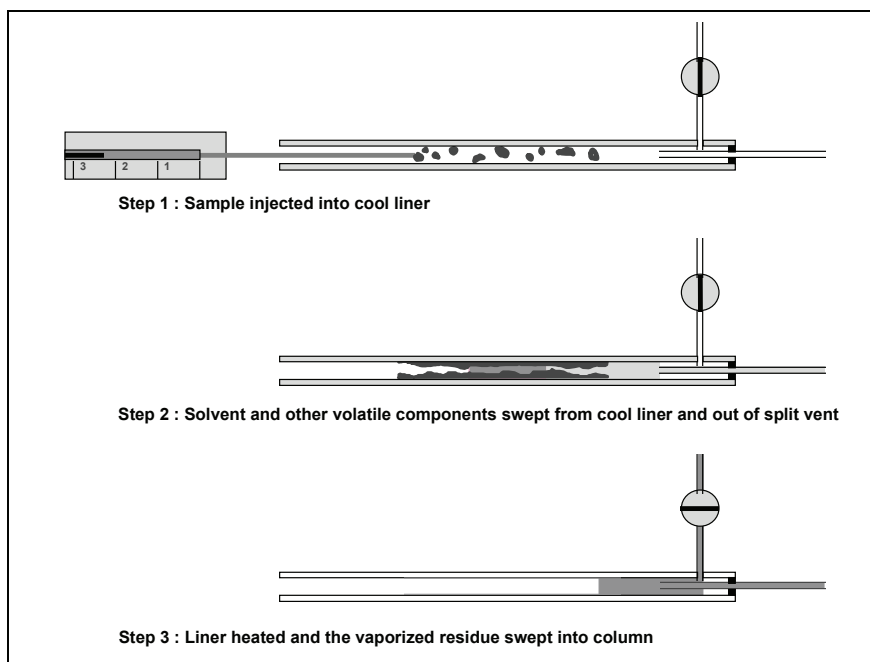


Figure 7-39. Alternative Column Isolation Mode.

## Solvent Purge Technique

This technique uses the ability of the PSS injector to rapidly change its liner temperature according to a user-defined program. Figure 7-40 shows how this ability can be used to remove the solvent from the liner and selectively transfer the less-volatile sample analytes to the column. This technique is best suited to splitless injections of low volatility analytes. While the solvent is being purged from the liner, there is still forward flow into the column and so some of the solvent will still enter the column (and hence the detector). The PreVent system, configured as for the Column Isolation Mode may be used to prevent all traces of the solvent entering the column during the purge process by reversing the flow of carrier gas at the injector column port.



**Figure 7-40. Using the PSS to eliminate solvent from the column.**

Table 7-4 shows typical entries that would be made in a method to enable a 1-minute solvent purge at 100 mL/min and 50 °C. Actual settings will depend upon the type and volume of solvent involved. The initial settings for P1 and P2

## Prevent

are established as described in this chapter. The column oven is programmed as for normal splitless injection except that the temperature is not raised until after the solvent purge step is complete.

**Table 7-4. Typical Method for Solvent Purge Technique**

Time	Event	Comment
INITIAL	SPLIT 1 (F) = 100 mL/min	Purge flow rate.
INITIAL	PSS = 50 °C	Purge temperature.
INITIAL	AUX 1 (P2) = 14 psig	Mid-point pressure at column inlet.
INITIAL	PRESS 1 (P1) = 2 psig	Pressure in injector lower than mid-point to keep solvent out of column.
1.00	SPLIT 1 (F) = OFF	Close split vent to transfer everything to column.
1.01	PRESS 1 (P1) = 20 psig	Raise injector pressure so carrier gas flows from liner to column.
1.02	PSS = 350 °C	Heat liner to vaporize sample residue.
2.00	SPLIT = 50 mL/min	Finally open split vent to clean liner for next run.

The Solvent Purge Technique may be used to support the following actions:

- Elimination of peak distortion caused by solvent flooding effects as a result of excess liquid solvent entering the column and becoming fractionated by the carrier gas flow
- Elimination of hostile solvents from sensitive columns and detectors
- Large volume injections - (See Large Volume Injections below.)

## **Large Volume Injections (LVI)**

The Solvent Purge Technique can also be used to support LVI applications of the PSS injector. The PSS must be used with a 2-mm i.d. liner which is firmly packed with glass wool. A large volume of liquid sample (up to 100  $\mu$ l - a 50- $\mu$ l syringe is available for the autosampler) may be injected into the liner which must be kept at a low temperature to prevent (very) explosive vaporization. The solvent is purged out of the split vent as for the Solvent Purge Technique. The purge time and flow rate need to be sufficient to remove all of the solvent (for example, about 100-200 mL of purge gas is needed to remove 50  $\mu$ l of n-hexane



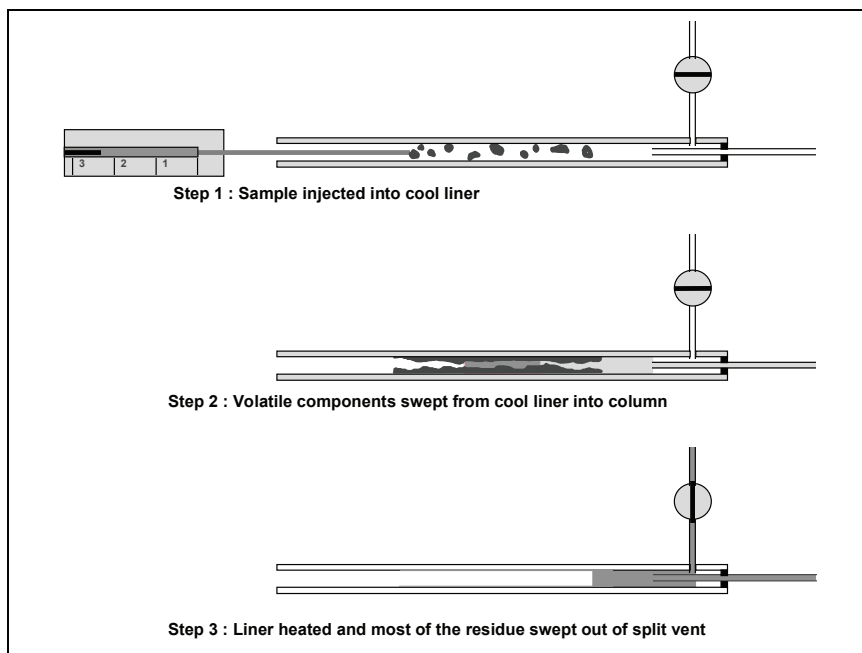
at 50 °C). Extended purge times may result in the loss of some of the analytes and so this technique is only suitable for low volatility compounds (for example, lower than n-decane). Typical method entries are given in Table 7-5.

**Table 7-5. Typical Method for LVI Technique**

Time	Event	Comment
INITIAL	SPLIT 1 (F) = 100 mL/min	Purge flow rate.
INITIAL	PSS = 50 °C	Purge temperature.
INITIAL	AUX 1 (P2) = 14 psig	Mid-point pressure at column inlet.
INITIAL	PRESS 1 (P1) = 2 psig	Pressure in injector lower than mid-point to keep solvent out of column.
2.00	SPLIT 1 (F) = OFF	Close split vent to transfer everything to column.
2.01	PRESS 1 (P1) = 20 psig	Raise injector pressure so carrier gas flows from liner to column.
2.02	PSS = 350 °C	Heat liner to vaporize sample residue.
3.00	SPLIT = 50 mL/min	Finally open split vent to clean liner for next run.

### ***Sample Residue Purge Technique***

The separation described under the Solvent Purge Mode can be reversed so that the volatile sample content enters the column and heavy (unwanted) low volatility sample residue left in the liner can be purged out through the split vent. This technique works best with the detector restrictor PreVent configuration so that the traces of low volatility material that do leave the liner can be backflushed from the column (see next Section). Use of the injector restrictor configuration will not prevent some of the low-volatility material from entering the column during the purge process. The figure below illustrates the separation process inside the PSS liner.



**Figure 7-41. Using the PSS to eliminate low volatility sample material from the column.**

The figures below illustrate how the PreVent system backflushes the heavier sample from the column. For further details on column backflushing refer to the “Time Saver Technique” Table 7-6 lists some typical entries in a method for the Sample Residue Purge technique. The initial settings for P1 and P2 are established as described in this chapter. Split or splitless injection with either a split/splitless or PSS injector is possible and the oven temperature program is set as for normal chromatography.

This technique is useful for the determination of volatile components in heavy oils, natural extracts, etc.

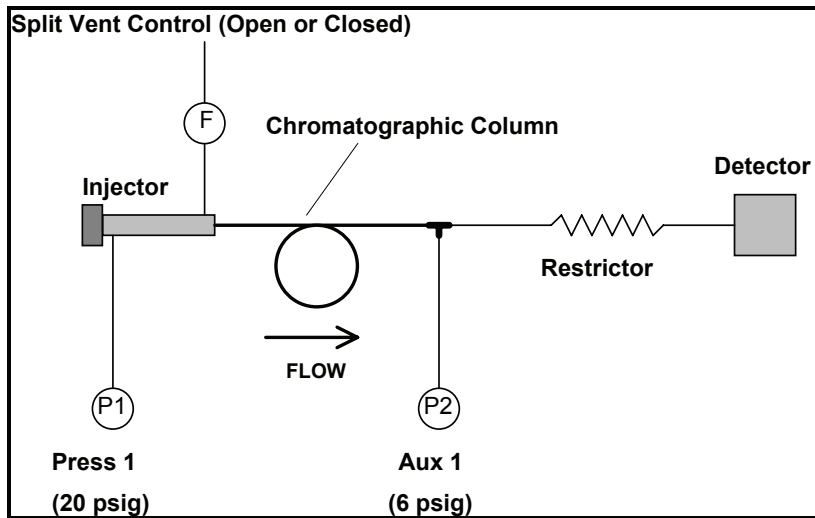


Figure 7-42. Injection and chromatography.

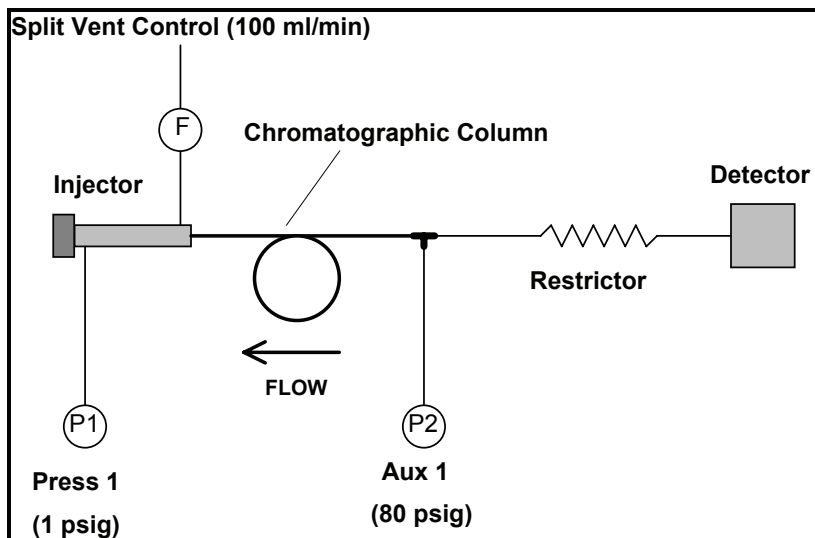


Figure 7-43. Column backflush.

**Table 7-6. Typical Method for Sample Residue Purge Technique**

Time	Event	Comment
INITIAL	SPLIT 1 (F) = 100 mL/min	Injection split flow (can use splitless).
INITIAL	PSS = 200 °C	Injection temperature.
INITIAL	AUX 1 (P2) = 14 psig	Mid-point pressure at column inlet.
INITIAL	PRESS 1 (P1) = 20 psig	Pressure in injector higher than mid-point to allow sample into column.
2.00	SPLIT 1 (F) = 100 mL/min	Open split vent (if not already) to reduce the amount of further sample entering column.
20.00	PRESS 1 (P1) = 5 psig	When last peak of interest has eluted, reduce inlet pressure...
20.01	AUX 1 (P2) = 80 psig	and increase mid-point pressure to backflush any sample material left in column.
20.02	PSS = 450 °C	Heat liner to bake out any sample residue and purge it out of the split vent.

### ***Time Saver Technique***

The PreVent system is able to support the classical, single-column, backflush technique. The use of PPC pneumatics enables the backflush conditions to be optimized for rapid backflush of unwanted sample material after the last peak of interest has eluted. The initial settings for P1 and P2 are established as described in this chapter. Split or splitless injection with either a split/splitless or PSS injector is possible and the oven temperature program is set as for normal chromatography. When the last peak of interest has eluted, the split vent is opened, P1 is reduced and P2 is increased. Any sample material left in the column is rapidly backflushed even at low oven temperatures eliminating the need, in many instances, for temperature programming.

This technique is suitable for the determination of components in any sample where there is unwanted low-volatility material entering the column. By reducing the need for temperature programming to remove such material, it reduces the analysis time and protects the column by not exposing it to high temperatures.

**Table 7-7. Typical Method for Column Backflush Technique**

<b>Time</b>	<b>Event</b>	<b>Comment</b>
INITIAL	SPLIT 1 (F) = 100 mL/min	Injection split flow (can use splitless).
INITIAL	AUX 1 (P2) = 14 psig	Mid-point pressure at column inlet.
INITIAL	PRESS 1 (P1) = 20 psig	Pressure in injector higher than mid-point to allow sample into column.
1.00	SPLIT 1 (F) = 100 mL/min	Open split vent (if not already) to allow backflushed components to escape to vent and help clean liner.
20.00	PRESS 1 (P1) = 5 psig	When last peak of interest has eluted, reduce inlet pressure...
20.01	AUX 1 (P2) = 80 psig	... and increase mid-point pressure to backflush any sample material left in column.

*Prevent*



**7-78**

# PPC Fundamentals 8

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## ***Introduction***

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Programmed Pneumatic Control (PPC) is the electronic control of pressures and flows for inlet, detector, and auxiliary gases. The PPC control modules regulate pressures and flows using electronically driven variable flow restrictors. The control modules also contain pressure and flow transducers to provide feedback for complete monitoring. A PPC controller board drives the variable restrictors on the control modules by comparing actual pressures and flows with setpoints determined from user-entered values.

This chapter presents an overview of the concepts and use of Programmable Pneumatic Control (PPC) of the Clarus 600 GC. In particular, it describes the following microprocessor-controlled pneumatic functions that comprise PPC:

- Carrier-gas flow, pressure, and linear-velocity programming
- Split-flow and split-ratio control
- Detector-gas flow control
- Auxiliary-gas pressure and flow control

The following unique pneumatic devices make these control functions possible:

- Carrier-gas mass-flow controller
- Split-pneumatic controller
- Detector-gas flow controller
- Auxiliary pressure controller

Pressure-readout module

You can combine one or more of these modules to create the control functions required for a specific pneumatic configuration in the GC.

### *PPC Fundamentals*

A dedicated microprocessor control system reads all operating parameters from — and sends control signals to — the pneumatic devices. This PPC controller receives pneumatic setpoints, the ambient temperature, pressure, the oven temperature, and GC status information from the main Clarus 600 GC processor. The PPC controller sends back the pneumatic operating parameters and PPC status information to the main GC processor.

The PPC controller accommodates up to eight flow controllers and/or twelve pressure controllers, for a maximum of twelve installed PPC devices.

The main Clarus 600 GC processor accepts and stores method setpoints and timed events for the configured PPC devices. In addition, the main Clarus 600 GC processor contains PPC functions that include calibration and setup utilities, diagnostics, and fault-condition monitoring.

## **Carrier Gas Control**

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The type of primary PPC carrier gas modules and their operation depends on the inlet (injector) option to which they are connected. For example, the packed-column inlet and the programmed-temperature on-column inlet (POC) use a single carrier gas mass-flow controller plus a pressure-readout module. The split/splitless (capillary) and programmed-temperature split/splitless (PSS) inlets use a combination of a carrier gas mass-flow controller and a split-pneumatic pressure controller. Other carrier-gas controllers may use auxiliary PPC zones (see **Auxiliary Carrier Gas Control**).

### **Packed Column and Programmed-Temperature On-Column Inlets**

The PPC carrier gas mass-flow controller delivers a constant flow rate into a column. The controller operates by increasing or decreasing the column head pressure to maintain a constant mass-flow as the column temperature changes, or if you use different columns. Its internal operation is similar to a conventional mass-flow controller, but instead of utilizing a spring-and-diaphragm mechanism for control, the PPC controller reads and sets the flow rate electronically. Unlike a conventional controller, it constantly monitors the ambient room pressure, the tank pressure, and the temperature of each flow module, and it compensates as required to maintain a constant mass-flow output. The schematic in the following figure illustrates the PPC mass-flow controller configuration for packed column and POC inlets.

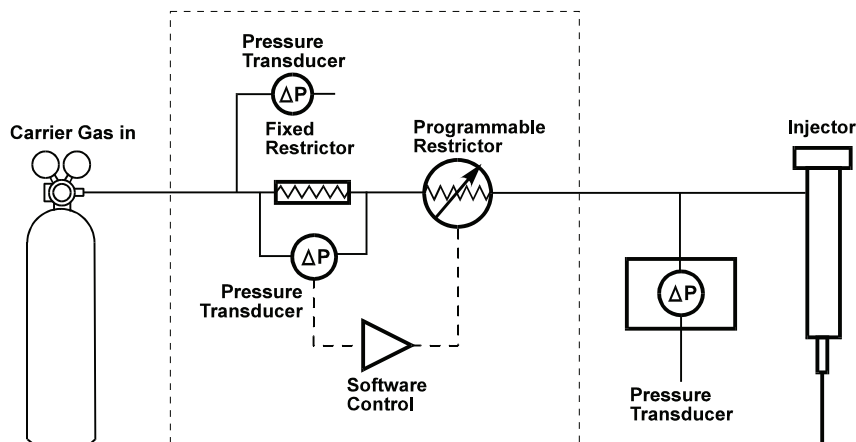


Figure 8-1 Schematic of packed column and POC pneumatics

### **Carrier Gas Mass-Flow Controller Operating Range**

The carrier gas mass-flow controller for packed-column inlets has a nominal flow range of 0 – 30 mL/min for helium carrier gas at 90 psig tank pressure. The controller for the POC inlet has a range of 0 – 10 mL/min helium for capillary column applications. The two controllers differ only in the installed flow-range element (restrictor). By changing the flow-range element, you can use the same controller in different GC configurations. The adapter kit for packed-column installation of 0.53-mm i.d. capillary columns, for example, contains the appropriate 0 – 10 mL/min element. After changing the flow-range element, you must always recalibrate the flow controller.

### **Mass-Flow Controller Setup**

The PPC system stores information about the type, location, and calibration of its pneumatic devices in battery backed-up random access memory (BRAM). This information is written into the BRAM during manufacturing. Normally, there is no need to access or modify the type or location of the PPC controllers. If the PPC hardware configuration is changed — by installing or removing PPC devices — the operator must access the PPC connection menus on the GC touch screen and modify the type and location of the PPC devices.

If the installed hardware does not match the stored configuration, the Clarus 600 GC issues a warning message.

**CAUTION**      *Do not modify the PPC connection information if the PPC hardware configuration has not changed.*

### **Mass-Flow Controller Calibration**

The carrier gas mass-flow controller is factory calibrated to a National Institute of Standards and Technology (NIST) traceable standard with helium carrier gas; however, it requires periodic calibration. Recalibration is required after changing the flow-range element; otherwise the calibration frequency is up to the individual operator. The controller accommodates changing the carrier gas (to other than helium) by approximating a new set of calibration parameters based on the new gas. The approximation is good but controller recalibration is recommended for the best accuracy. It is good laboratory practice to recalibrate all measuring and control devices periodically.

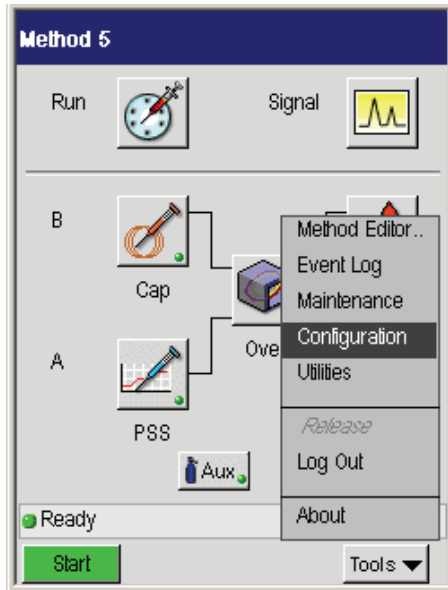
The mass-flow controller calibration procedure consists of specifying the reference temperature and pressure; zeroing the internal sensors in the flow controller with the tank pressure off; specifying the correct carrier gas type and installing the correct flow-range element; measuring the background flow rate at zero-flow with the tank pressure on; and measuring the flow rate at 60% of full-scale flow. This procedure is built into the GC firmware.

Once the PPC is set up and calibrated you must configure it

### **Carrier Gas Mass-Flow Controller Configuration**

Once the carrier gas mass-flow controller has been set up and calibrated for the PPC system, you must configure its operating mode on the GC keypad. For packed-column and POC inlets the selections are **Zero**, **Flow**, or **Pressure**.

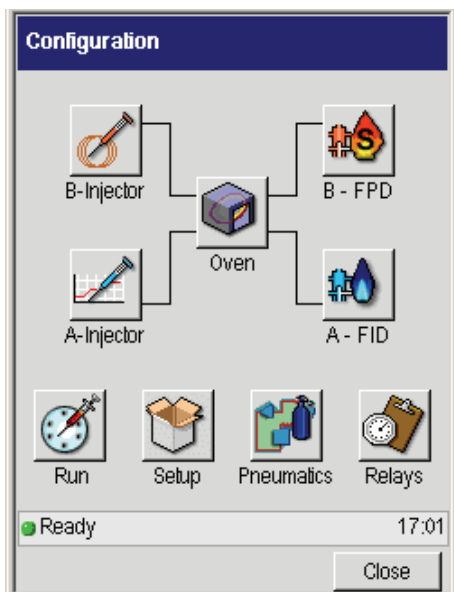
1. From the System Status Screen touch the **Tools** button.



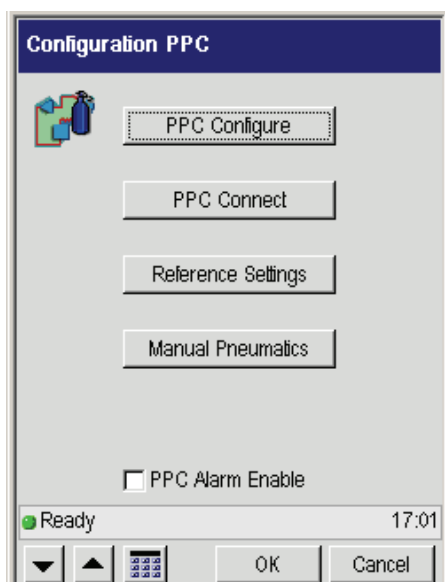
2. From the tools drop down menu select **Configuration**.

3. In the Configuration touch screen touch the Pneumatics button

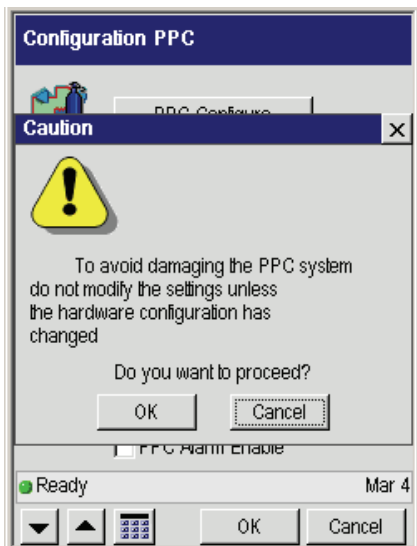




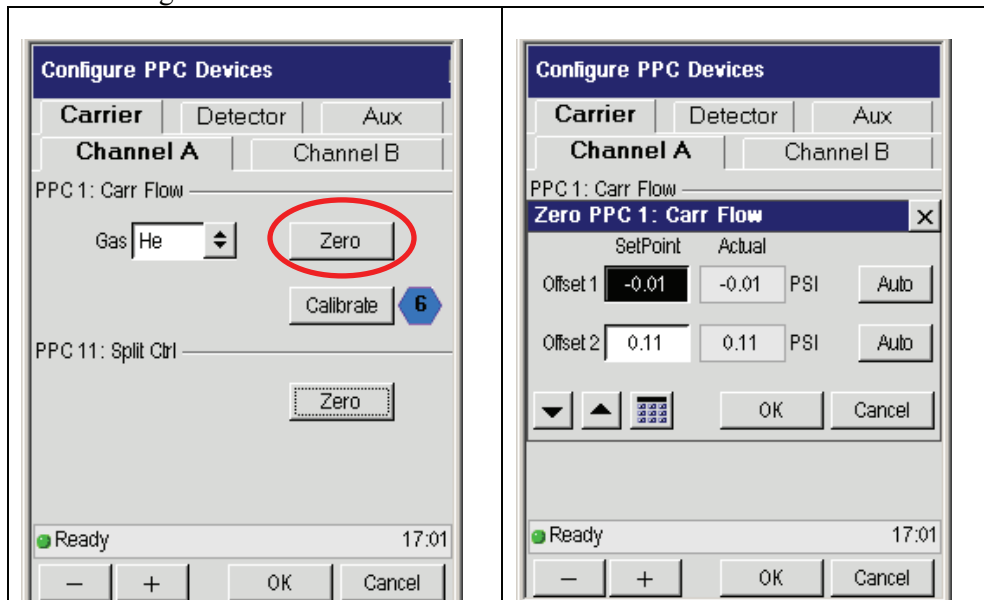
4. In the Configuration PPC touch screen touch **PPC Configure** bar.



5. A popup warning appears, select **OK**.



6. On the **Configure PPC Devices** screen touch the **Zero** button then touch **OK** to set the gas flow to zero.





### **Headspace Analysis**

Headspace analysis is the analysis of the vapor lying in equilibrium over a solid/liquid sample in a sealed vial.

For practical headspace analysis the sample is sealed in a vapor tight vial, placed in an thermostatted oven and heated to a pre-determined temperature.

The sample vial contains the volatile material in equilibrium between the solid/liquid sample and the vapor lying over it.

After equilibrium is reached between solid/liquid phase and the vapor phase, a defined amount of the vapor is taken and carried to the column in the gas chromatograph for analysis. With this technique only highly volatile substances reach the column, the non-volatile substances remain in the sample vial.

Using this technique, samples containing constituents which are unsuitable for injection with a syringe can be analyzed (e.g. polymers, highly viscous liquids).

Suitable fields of application are in the analysis of polymers, certifying of the volatile components in drinks and foodstuffs, blood alcohol levels, water and environmental analysis.

For information on vacuum see the section on **Vacuum Compensation** later in this chapter.

### **Capillary and Programmed Split/Splitless Inlets**

A packed-column or POC inlet requires only a source of flow-controlled carrier gas; however, split-type inlet systems are more complex. The capillary and programmed-temperature split/splitless (PSS) inlets have identical split pneumatics. The split pneumatics occupy two PPC control zones, one for column inlet pressure control and one for split flow rate control.

#### **Split Pneumatic Setup**

The PPC system stores information about the type, location, and calibration of its pneumatic devices in battery-backed-up random access memory (BRAM). This information is written into the BRAM during manufacturing. Normally, there is

## PPC Fundamentals

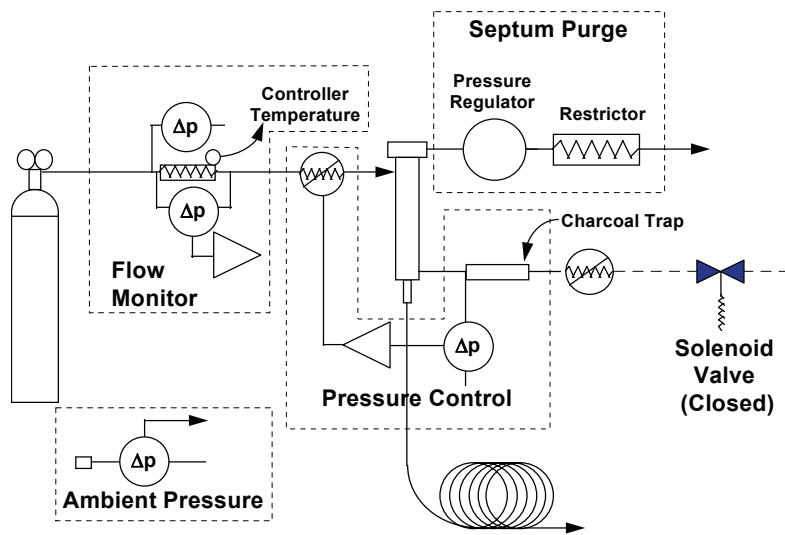
no need to access or modify the type or location of the PPC controllers. If the PPC hardware configuration is changed — by installing or removing PPC devices — you must access the PPC connection menus on the GC keypad and modify the type and location of the PPC devices. If the installed hardware does not match the stored configuration, the GC issues a warning message.

### **CAUTION**

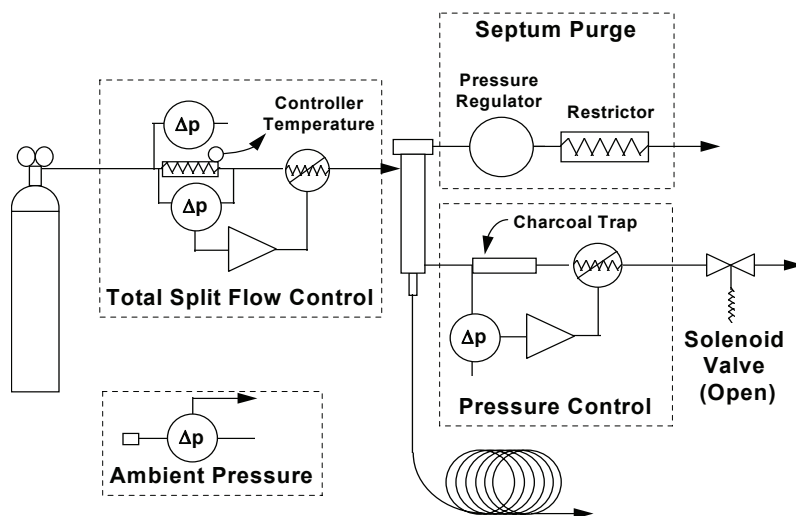
*Do not modify the PPC setup information if the PPC hardware configuration has not changed.*

### **Split Pneumatic Control**

A split system such as the capillary or PSS inlet pneumatics controls three parameters: the split flow, the inlet pressure, and the septum purge flow. See the following figure that shows two diagrams showing the forward pressure mode (splitless control) and the backpressure mode (split control) of the PPC split pneumatics in the Clarus 600 GC.



**Forward Pressure Mode (Splitless Control)**



**Backpressure Mode (Split Control)**

**Figure 8-2 Schematic of the split PPC pneumatics**

**Split Flow** – A mass-flow controller supplies carrier gas to the split or PSS inlet system. This is the same mass-flow controller used for packed or POC inlets, but here it is used with a 0 – 300 mL/min flow-range element (fixed restrictor). Carrier gas passes through the flow controller and into the inlet (injector) at the connection labeled **Carrier In**. Some of the gas flows across the septum and out the *septum purge*, while the rest flows down through the *inlet liner*. At the bottom of the liner, a fraction of the carrier gas enters the column, and the balance flows back up the outside of the liner and out the *split vent*. An electrical solenoid valve provides positive split flow shutoff when required.

**Inlet Pressure** – A pressure transducer connected at the split vent (before the charcoal trap) measures the inlet pressure at the head of the column. The inlet pressure may range from 0 – 100 psig.

**Septum Purge** – A constant-flow device (fixed pressure regulator) regulates the septum purge flow at approximately 3 mL/min, independently of the inlet pressure. The regulator is preset in the factory; it is not adjustable.

### **Split Pneumatic Calibration**

The split flow controller requires periodic calibration. The controller is factory calibrated to a National Institute of Standards and Technology (NIST) traceable standard with helium carrier gas. Recalibration is required upon changing the flow-range element; otherwise, the calibration frequency is up to the individual operator. The PPC controller accommodates changing the carrier gas to other than helium by approximating a new set of calibration parameters based on the new gas. The approximation is good but controller recalibration is recommended for the best accuracy. It is good laboratory practice to recalibrate all measuring and control devices periodically.

The calibration procedure consists of specifying the reference temperature and pressure; zeroing the internal sensors in the flow controller with the tank pressure off; specifying the flow-range element and carrier gas type; measuring the background flow rate at zero-flow with the tank pressure on; and measuring the flow rate at 60% of full-scale flow. This procedure is built into the GC firmware.

### **Split Pneumatic Carrier Gas Configuration**

Once the split pneumatic system has been set up and calibrated on the PPC system you must configure its operating mode by using the GC touch screen. There are configuration choices for column carrier-gas control and split flow or ratio control.

The PPC controller includes a mathematical model of a capillary column. This model is enabled by setting *Capillary Control Mode* ON (see **Capillary Control Mode**, below). The PPC controller uses this model to calculate the column exit flow at ambient pressure and temperature, the column average linear gas velocity, and the inlet split ratio in real time. You may choose to control the column flow or velocity, in which case the PPC controller calculates the pressure required to maintain the flow or velocity setpoint. You may also choose to control the split ratio; in this case the PPC controller calculates the split flow rate required to obtain the setpoint split ratio.

The PPC controller microprocessor carries out the method setpoint program for both carrier-gas channels by taking the setpoint pressure, flow, or velocity value; the column dimensions; the carrier-gas type; the reference temperature and pressure; the oven actual temperature; and the ambient pressure. It calculates the other two parameters in real time. For pressure control, it calculates the flow and velocity; for flow control, it calculates the pressure and velocity; for velocity control, it calculates the pressure and flow. These calculations track ambient pressure changes, changing oven temperatures, and the execution of the multi-ramp carrier-gas program. In each case, the actual pressure in the inlet is controlled by the calculated or method pressure setpoint.

For more information on these internal calculations, see the section on “**Theory of Capillary Column Control**,” in this chapter.

#### **Capillary Control Mode**

The first choice for split carrier-gas control is the **Capillary Control Mode**. This parameter may be set *On* or *Off*. Each carrier gas channel may be setup with or without capillary control.

*Off* – uses direct pressure control of the column through a multi-step pressure program and carrier gas timed events. You can directly control the split flow

## PPC Fundamentals

control through the initial setpoint and timed-event control of the split flow controller. Split ratio control, capillary column flow, and capillary column velocity control and information are not available. This mode of operation does not require knowledge of the capillary column dimensions.

**On** – enables indirect control of capillary column flow or velocity in addition to direct pressure control, each through a multi-step program and timed events. This mode also enables direct split flow or indirect split ratio control of the split flow controller. The display reports actual pressure, flow, velocity, split flow, and split ratio. In this mode you must enter accurate column length and diameter information (except for pressure-controlled operation with oven tracking).

### Carrier Gas Configuration without Capillary Column Control

When capillary control is turned off for a split PPC controller, the choices for carrier-gas control are **None** and **Pressure**.

**None** – removes the split pneumatics from the GC method and turns off the split flow and pressure.

**Pressure** – enables direct pressure control of the split inlet. The PPC controller uses a 5 psig initial setpoint with an infinite hold time when **Pressure** is first selected. The user may add up to three additional setpoints, ramps, and hold times. This mode also enables direct split flow control (see below).

### Carrier Gas Configuration with Capillary Column Control ON

In this mode the choices for carrier-gas control are **None**, **Pressure**, **Flow**, and **Velocity**. With pressure control you may select either **Pressure: Program** or **Pressure: Oventrack**.

**None** – removes the split pneumatics from the GC method and turns off the split flow and pressure.

**Pressure: Program** – enables direct pressure control of the column pressure. The split carrier-gas pressure program mode gives a multi-ramp program with real-time calculation and display of flow and velocity, plus flow control or ratio control of the split flow. The PPC controller uses a 5 psig initial setpoint with an infinite hold time when **Pressure: Program** is first selected. The pressure range

is 0 – 100 psig. You may add up to three additional setpoints, ramps, and hold times.

**Pressure: Oventrack** – also enables direct carrier-gas pressure control. The PPC controller automatically varies the initial inlet pressure so that the capillary column flow rate remains constant as the oven temperature changes away from its initial setpoint value. The PPC controller uses a 5 psig initial setpoint when **Pressure: Oventrack** is first selected. Multi-ramp programming is not available. This mode allows only direct flow control of split flow; however, this mode can be used for constant column flow control when you do not know your column dimensions.

**Flow** – enables indirect capillary column mass-flow control. The PPC controller calculates the inlet pressure required to maintain the method flow program setpoints from the column dimensions, carrier-gas type, oven temperature, and ambient pressure. The display shows the flow, pressure, and calculated average linear gas velocity. The PPC controller uses a 2 mL/min initial setpoint with an infinite hold time when **Flow** is first selected. The column flow has a total flow range of 0 – 500 mL/min (including the split flow). You may add up to three additional setpoints, ramps, and hold times. This mode enables flow control or split ratio control of the split flow.

**Velocity** – enables indirect capillary column average linear gas velocity control. The PPC controller calculates the inlet pressure required to maintain the method velocity program setpoints from the column dimensions, carrier-gas type, oven temperature, and ambient pressure. The display shows the velocity, pressure, and calculated flow rate. The PPC controller uses a 30 cm/s initial setpoint with an infinite hold time when **Velocity** is first selected. You may add up to three additional setpoints, ramps, and hold times. The carrier-gas velocity has a range of 0–200 cm/s. This mode enables flow control or split ratio control of the split flow.

### **Column Dimensions**

After selecting any of the capillary control modes (except *Pressure: Oventrack*) the GC prompts you for the column dimensions. These parameters control the relationships between the pressure, flow, and velocity as calculated by the PPC controller. If the column dimensions are inaccurate, the PPC controller will

produce incorrect readings and will incorrectly control the column flow, velocity, and split ratio.

### **Column Length**

The column length defaults to 25.0 meters. It has a range from 1.0 – 200.0 meters. Small errors in the column length affect the average linear velocity and the flow rate only slightly. See the section “**PPC Tips and Techniques**” at the end of this chapter for procedures to correct errors in column length.

### **Column Inner Diameter**

The column inner diameter defaults to 250  $\mu\text{m}$ . It has a range from 50 – 1000  $\mu\text{m}$ . Small errors in the column inner diameter *strongly* affect the average linear velocity and the flow rate.

### **Vacuum Compensation**

The next Clarus GC split pneumatic configuration parameter in the capillary control mode is vacuum compensation. The PPC controller uses a mathematical model of a capillary column to determine pressure – flow – velocity relationships. The column outlet pressure plays an important role in these calculations. The PPC controller continually monitors the barometric pressure because normal daily atmospheric pressure fluctuations affect the column average linear gas velocity—and thus retention times—if the controlling system does not compensate. Vacuum compensation controls the role of the outlet pressure in the PPC controller. Vacuum compensation may be set **On** or **Off**.

**Off** – causes the PPC controller to assume that the column outlet is at atmospheric pressure.

**On** – causes the PPC controller to assume that the column outlet is at vacuum. This setting is appropriate for GC-MS systems with a direct interface where the column enters the mass-spectrometer source. Vacuum compensation should be turned off when an open-split interface is used since it keeps the GC column outlet close to atmospheric pressure.



### **Split Control Mode Configuration**

The last Clarus GC carrier-gas configuration parameter is the split control mode. The ratio of the total flow through the inlet liner to the column flow rate — the fraction of sample vapor entering the column, or the *split ratio* — controls the quantitative transfer of sample into the column. For split ratio control the PPC system must know both the column flow rate and the total gas flow through the inlet liner.

When capillary control is off, the PPC system does not compute the capillary column flow rate or the split ratio; only split flow control is possible. With capillary control on (except for **Pressure: Oventrack** mode) the PPC controller calculates the column flow rate and the split ratio.

The choices for split-flow control are **Flow** or **Ratio**. There is no choice for **None**, since the split pneumatics will not operate without incoming carrier-gas flow. Setting the carrier-gas configuration to **None** automatically sets the split-flow controller configuration to none.

**Flow** — enables direct split flow control of the carrier gas mass-flow controller. You enter a split vent flow rate in the method. The PPC controller adds an optional offset to obtain the total split flow (see “**Split Flow Offset**” below) and sets that amount at the flow controller. The GC also displays the calculated split ratio if capillary control is on.

**Ratio** — enables indirect split ratio control of the carrier gas mass-flow controller. The user enters a split ratio in the method. The PPC controller calculates the required split vent flow from the column flow rate and adds an optional offset to obtain the total split flow (see “**Split Flow Offset**” below) and sets that amount at the flow controller. The GC also displays the calculated split flow rate defined by the following equation:

$$\text{total split flow} = \text{split flow} + \text{septum purge flow} + \text{column flow}$$

**NOTE:** *Any carrier system leaks will add to the observed total split flow.*

### **Split Flow Offset**

The PPC controller sets and monitors the *total* split flow through the inlet system. The split ratio is based on the flow out the split vent. Since the septum purge flow is taken from the total split flow rate before passing through the inlet liner, across the column entrance, and out the split vent, split ratio calculations must account for the flow reduction. The Clarus 600 GC includes a split flow offset parameter that is accessed from the *CONFIG CARRIER* menus when a split controller is setup. This parameter may be set to **Fixed** or **Auto**.

**Fixed** – prompts the user to enter the septum purge offset value. This offset is the septum purge flow plus the column flow. The initial column flow is displayed on the [Carrier Prog] key when the GC is READY at its initial setpoints with capillary control ON. The PPC controller adds this offset to the method setpoint split flow rate to obtain the actual flow delivered to the split inlet. The septum purge flow is factory set at 3 mL/min. To measure the actual septum purge offset, set the carrier pressure to 5 psig or greater and turn off the split flow (set to 0). The remaining split vent flow displayed is the septum purge flow, plus the column flow. The actual septum purge flow can be measured from the split vent bulkhead fitting.

**Auto** – measures the offset flow rate before each run by momentarily setting the split vent flow to 0, waiting for the remaining flow to stabilize, and then recording its magnitude. During this time the GC status is **PRE-RUN**. This operation occurs after equilibration, and before executing any Pre-Run timed events (events with negative times). The **Auto** measurement includes both column and septum purge flows.

**NOTE:** *The split flow screen (shown below) displays the flow from the injector vent as the setpoint and the total split system flow as the actual values.*

The value in the upper right carrier is the total split flow. The value in the lower right corner is the split vent setpoint.

The flow measured at the split vent fitting on top of the GC is the vent flow plus the septum purge flow.

### **Split Pneumatic Operation**

Once the split system has been configured, you may modify the default setpoints in the active GC method, edit the setpoints in stored methods, or generate a new method and enter split pneumatic setpoints. The following parameters control split pneumatic operation.

#### **Initial Carrier Setpoint**

The PPC controller takes setpoints of flow, velocity or pressure depending on the configured split pneumatic carrier gas mode. It receives its initial setpoint from the active GC method. The initial setpoint is established upon setting up a method, ending a run, or otherwise resetting the instrument. The PPC controller holds the setpoint constant at the initial value if no additional carrier-gas entries are made in the GC method.

#### **Carrier Gas Program**

The GC method accommodates up to three additional carrier-gas program steps consisting of a hold time, ramp rate, and plateau setpoint. Sequential setpoints may be greater than or less than the preceding setpoint; the PPC controller will program the split pneumatics with a positive or negative ramp as required. The carrier gas resets to the initial setpoint value in the method at the end of the GC oven program, even if a carrier-gas program is in progress. The carrier gas also resets if the end of the carrier-gas program occurs before the end of the oven program.

#### **Carrier Gas Timed Events**

You may also program carrier-gas timed events to produce step changes in the setpoint. Carrier-gas timed events can occur before or after a run starts. A carrier-gas timed event supersedes the method setpoint value at the time the event occurs. However, if the carrier-gas program in the method advances to a new step after a carrier-gas event, the new program step or ramp takes control.

#### **Split Flow or Ratio Setpoint**

The GC method includes an initial split flow or ratio setpoint. This setpoint is established upon setting up a method, ending a run, or otherwise resetting the

## *PPC Fundamentals*

instrument. The PPC controller holds the setpoint constant at the initial value if no additional carrier-gas entries are made in the GC method.

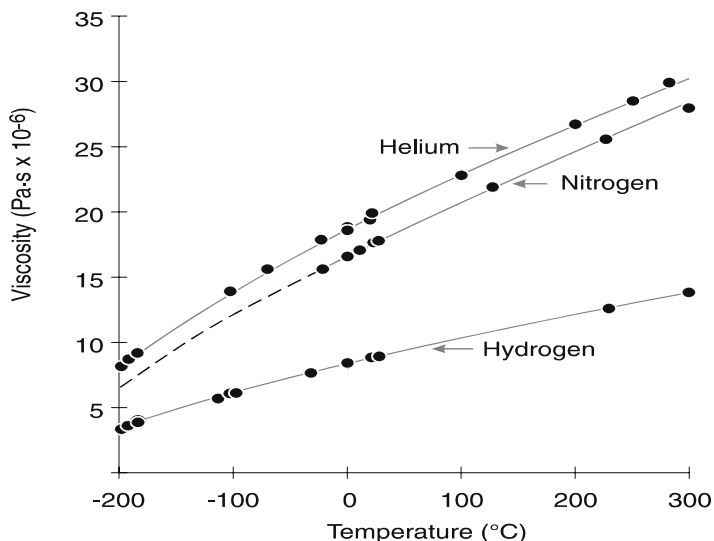
### **Split Flow or Ratio Timed Events**

The split flow or ratio is changed to a new setpoint by a **split** timed event. Split flow or ratio events can occur before or after a run starts. A split flow or ratio event supersedes the method setpoint value at the time the event occurs.

### ***Theory of Capillary Column Control***

This section gives an overview of the theory behind PPC. For more detailed information please refer to Reference [1].

The Clarus 600 GC Programmable Pneumatic Control system can program capillary column pressure, flow, or velocity. It accomplishes this by calculating and maintaining the column pressure drop required by the carrier-gas program and column temperature. The PPC controller then reads the actual pressure and uses that value to calculate the actual column flow rate and average linear gas velocity. The column dimensions, ambient pressure, and carrier-gas type also enter into the equations.

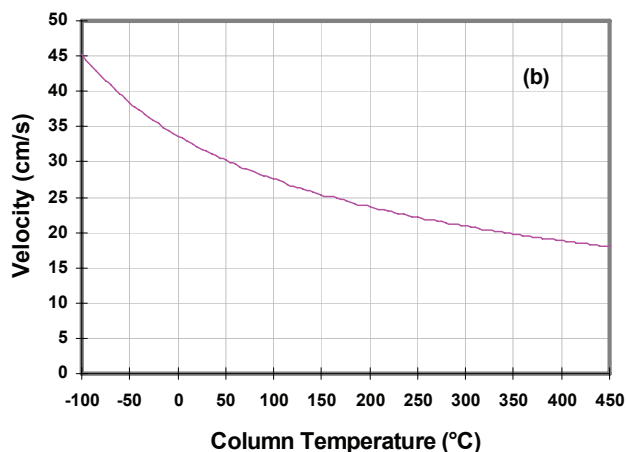
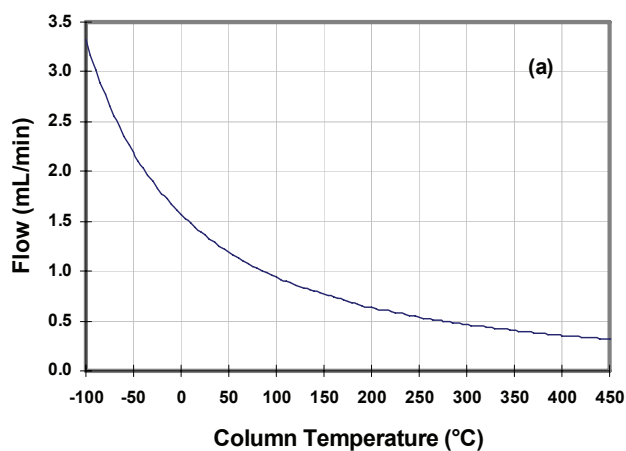
**Column Temperature Effects**

**Figure 8-3 Carrier-gas viscosity-temperature relationships<sup>1</sup>**

The viscosity of the carrier gas changes with the column temperature. As the column temperature increases, so does the carrier-gas viscosity. The increasing viscosity raises the resistance of the carrier gas to being pushed through the column. Thus, higher column pressure drops are required to maintain column flow or average linear velocity at higher temperatures. Conversely, keeping the pressure drop across the column constant while increasing the column temperature causes the flow rate and linear velocity to drop off.

Figure 8-3 illustrates the effect of column temperature on carrier-gas viscosity. Notice that the viscosity is different for each carrier gas, and that the “slopes” of the curves are also different in each case. For each carrier gas, the Clarus 600 GC includes an accurate model of this viscosity-temperature relationship as represented by the lines in Figure 8-3.

<sup>1</sup> J. V. Hinshaw and L.S. Ettre, “Introduction to Open-Tubular Column Gas Chromatography,” (Advanstar Communications, 1994), p. 25. (P/N N930-6007).



**Figure 8-4 Effect of column temperature on (a) flow and (b) velocity**  
Column: 25-m x 250- $\mu$ m i.d. Helium carrier at 12 psig constant pressure drop.

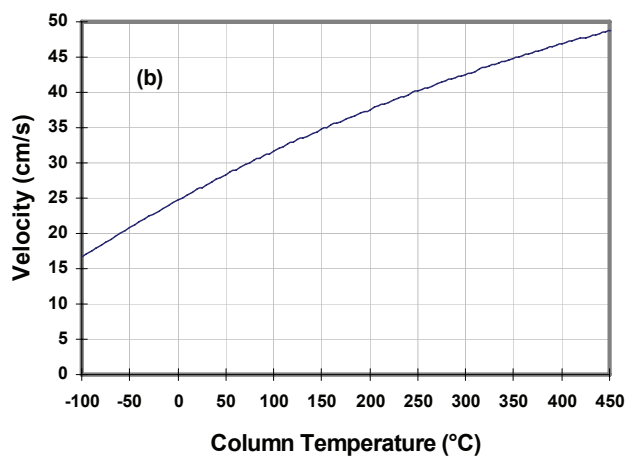
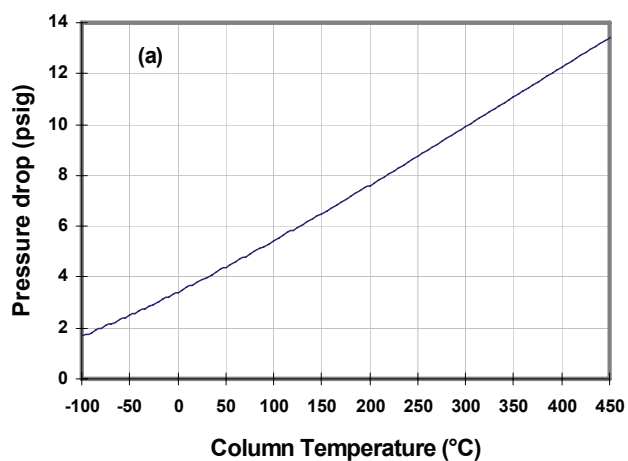
The viscosity changes caused by increasing temperature affect the flow and velocity through a column with a constant pressure drop. As shown in Figure 8-4, both flow and velocity decrease across the column temperature range. This decreasing flow and velocity can cause loss of column efficiency as well as

changes in flow-sensitive detector operation during programmed-temperature analyses. In this case, the column pressure drop was selected so that the average linear velocity was close to 30 cm/s at 50 °C; at 350 °C the velocity drops to around 20 cm/s. The effect on flow is more severe, dropping from 1.19 mL/min at 50 °C to 0.4 mL/min at 350 °C.

Without PPC, compensation for this effect is difficult and limited. Although it is possible to program the PPC controller pressure to crudely compensate, it is much easier and more accurate to use either the flow-programmed or velocity-programmed modes instead.

### ***Flow Programmed Operation***

The PPC controller operates in a pressure-, flow-, or velocity-programmed mode. The flow-programmed mode is useful in applications where the column flow rate must be held constant, or where the operator wishes to express the column carrier program in flow terms. Figure 8-5 shows the pressure program and velocity that result when a 50-m x 530 µm i.d. column operates with nitrogen carrier at a constant 4 mL/min flow across a range of temperatures.



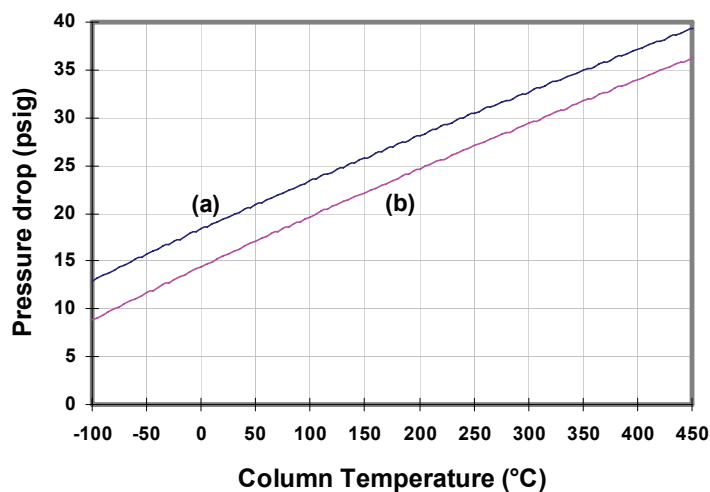
**Figure 8-5 Effect of column temperature on (a) pressure and (b) velocity. Column: 50-m x 530- $\mu$ m i.d. Nitrogen carrier at 4 mL/min constant mass flow**

### ***Velocity Programmed Operation***

Programming the PPC controller in average linear gas velocity mode adjusts the column pressure drop to maintain the setpoint velocity as the oven temperature



and ambient pressure change. This helps ensure better repeatability of chromatograms from one instrument to another — assuming the ovens are calibrated to a common temperature reference standard—by eliminating effects due to differences in altitude or weather. Differences in the columns, however, are not accounted for.

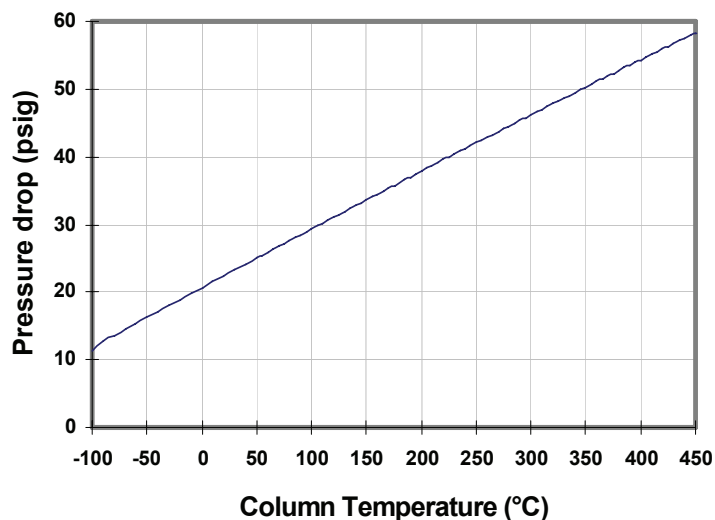


**Figure 8-6 Effect of altitude on pressure programs. Column: 15-m x 100- $\mu$ m i.d. Hydrogen carrier at 30 cm/s constant average linear gas velocity. (a): Sea level; (b): 3000 m altitude**

Figure 8-6 shows two pressure-temperature curves, one for a constant velocity of 30 cm/s on a 15-m x 100  $\mu$ m i.d. column with hydrogen carrier with an atmospheric pressure equivalent to sea level, and the other for the same column at the equivalent of 3000 m altitude. When operating the oven at a constant temperature with identical columns, all solute retention times will be the same in both cases because the velocities are unchanged.

With oven temperature programming, the retention times will be very similar but not quite the same due to slight differences in the velocity profiles across the two columns at different altitudes.

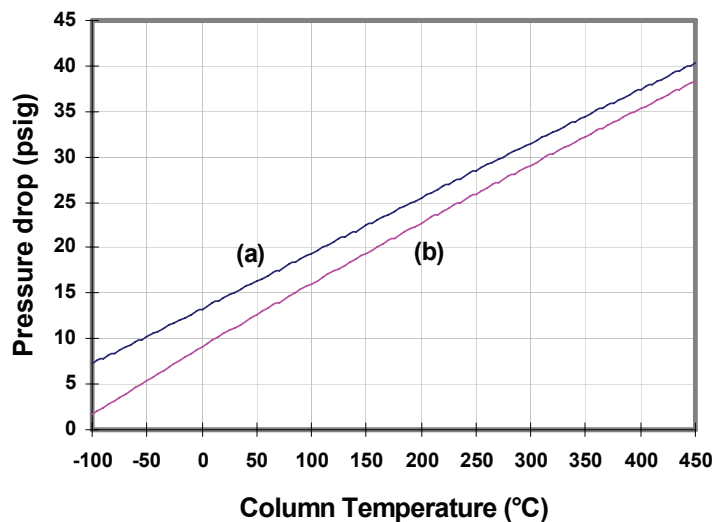
**Pressure / Oventrack Mode**



**Figure 8-7 Pressure program in oventrack mode. Helium carrier at 25 psig, 50 °C initial conditions**

The **Pressure: Oventrack** operating mode maintains a constant column mass flow as the column temperature changes by adjusting the inlet pressure as required. No knowledge of column dimensions is required in this mode: only the initial pressure and temperature are specified. Figure 8-7 shows the pressure-temperature profile for **Pressure: Oventrack** mode with an initial pressure of 25 psig at 50 °C with helium carrier gas. This operating mode is convenient for mass-spectrometric or thermal-conductivity detectors where column dimensions are not important.

**Vacuum Compensation**



**Figure 8-8 Pressure program (a) without and (b) with vacuum compensation. Column: 60-m x 320  $\mu$ m i.d. Helium carrier at 2 mL/min constant flow**

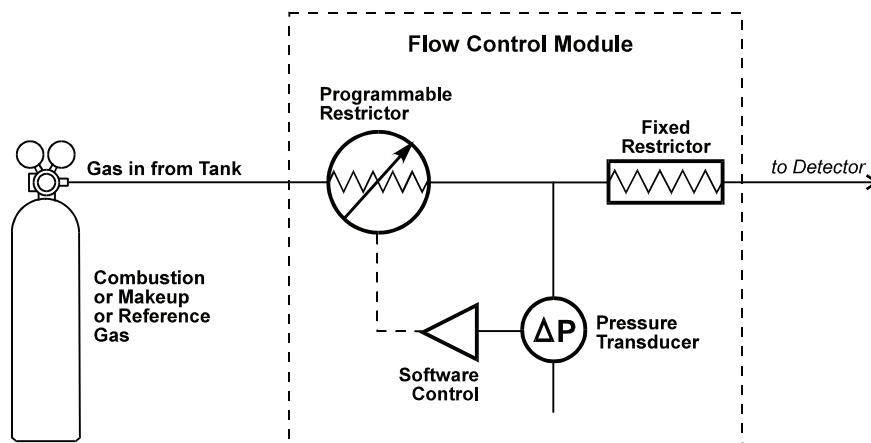
When using a mass-spectrometric detector with the column exit under vacuum, it is necessary to reduce the column pressure drop to account for the reduced exit pressure. The effect of putting the column exit under vacuum is shown in Figure 8-8 for a 60-m x 320  $\mu$ m i.d. column with helium carrier at a 2 mL/min constant flow rate.

## ***Detector Gas Flow Control***

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The Clarus 600 GC can control up to two detectors, each with up to two primary detector gas flow controllers. The GC determines the type of controllers required for each detector by sensing the presence of the detector amplifier board. Certain detectors, such as the EICD/PID combination, use additional auxiliary detector gas control zones (see “**Auxiliary Detector Gas Control**,” below).

The Clarus 600 GC PPC detector gas flow controller delivers a set flow rate of gas into a device (detector) at atmospheric pressure. Unlike the carrier gas controller, the detector gas controller does not compensate for significant changes in its outlet pressure. It is not suitable for providing column flow. The detector gas controller is not affected by normal changes in the incoming gas supply pressure. The PPC controller compensates for changes in each detector gas controller’s internal temperature. Figure 8-9 illustrates the detector flow control module.



**Figure 8-9 Schematic of a detector gas flow control module**

## Detector Gas Flow Controller Operating Range

The detector gas flow controller operating range depends on the detector in use. Each flow controller uses a flow-range element (fixed restrictor) that determines the available flow range for a particular gas. The nominal detector gas flow lies within 20–50% of the full-scale controller range for the factory-installed flow-range elements. The flow-range elements should not be changed unless the controller is to be used with a different detector. Please refer to Chapter 9, “Maintenance,” the Practical Hints section for information on individual flow-range elements for specific detectors.

**NOTE:** *Only the fixed restrictor changes for various requirements.*

## Detector Gas Flow Controller Setup

The PPC system stores information about the type, location, and calibration of its pneumatic devices in battery-backed-up random access memory (BRAM). This information is written into the BRAM during manufacturing; normally there is no need to access or modify the type or location of the PPC controllers. If the PPC hardware configuration is changed — by installing or removing PPC devices — you must access the PPC connection menus on the GC keypad and modify the type and location of the PPC devices. See “Connecting the PPC Modules” in Chapter 13, “**System Utilities**,” for detailed instructions.

**CAUTION**

*Do not modify the PPC connection information if the PPC hardware configuration has not changed.*

## Detector Gas Flow Controller Calibration

The detector gas mass-flow controller requires periodic calibration and the **calibration is gas specific**. The controller is factory calibrated to a National Institutes of Standards Technology (NIST) traceable standard with air, hydrogen, or helium gas, depending on the detector type. Recalibration is required upon

changing the gas or the flow-range element; otherwise, the calibration frequency is up to the individual operator. It is good laboratory practice to recalibrate all measuring and control devices periodically.

The detector gas flow controller calibration procedure consists of specifying the reference temperature and pressure; zeroing the internal sensors in the flow controller with the tank pressure off; specifying the flow-range element; selecting the gas type (for reference or makeup only); then measuring the flow rate at 20% and 40% of full-scale flow. This procedure is built into the Clarus 600 GC firmware. See Chapter 13, “System Utilities,” for detailed instructions.

### **Detector Gas Mass-Flow Controller Operation**

For flame-type detectors, setting up the detector gas flow controllers sets a default flow rate equal to the nominal flow specified for the detectors. For detectors with makeup or reference gases the flow rate defaults to 30 mL/min. Once the detector gas flow controllers have been set up and calibrated on the PPC system, the operator must enter their flow rates on the GC touch screen.

Unlike carrier gas flows, detector gas flow rates do not appear in the GC method. Instead, they are set in the GC configuration. There are no timed events that modify detector flows. Detector gas flow controllers may be set up as Auxiliary gas flow zones if it is necessary to incorporate them into the GC method or to modify them with timed events. See “**Auxiliary Detector Gas Control**” below for further information.

### **Flame Gases**

The Clarus 600 GC automatically determines gas types for flame detectors, including the flame-ionization (FID), flame-photometric (FPD), and nitrogen-phosphorous (NPD).

Once the flame gas controllers are set up, you may modify the default flow rates as required by entering new values under the in the GC configuration.

### **Flame Ignition**

The PPC version of the FID has *flameout* detection and an *auto-ignite* feature. Flameout detection occurs 0.5 minutes into the PRE-RUN time and it measures

the user-entered baseline threshold level in mV. If the threshold level is exceeded, the flame is considered lit; otherwise, the GC detects that the flame is out, and auto-ignite automatically lights the flame before the start of a run. You can also manually light the flame at any time by pressing the ignite button on the touch screen.

## ***Makeup and Reference Gases***

The Clarus 600 GC automatically determines the presence of reference and makeup gases for detectors. The thermal-conductivity detector (TCD) uses reference gas. The electron-capture detector (ECD), photoionization detector (PID), and, optionally, the thermal-conductivity detector (TCD) use makeup gas. Makeup and reference gases operate in a fashion similar to flame gases, except that you may change the gas type in the PPC controller setup.

Once the makeup or reference gas controllers are set up, you may modify the default flow rates as required by entering new values in the active.

Detector makeup gas flow controllers may be set up as Auxiliary gas flow zones if it is necessary to incorporate them into the GC method or to modify them with timed events. See “**Auxiliary Detector Gas Control**” below for further information.

## ***Auxiliary Pressure and Flow Control***

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In addition to carrier and detector gas control, the Clarus 600 GC accommodates up to four auxiliary PPC controllers. The auxiliary zones can control carrier gas mass-flow controllers, pressure controllers, pressure readouts, and detector gas flow controllers. These zones all have an initial setpoint, and incorporate timed-event changes.

### ***Auxiliary Carrier Gas Control***

The Clarus 600 GC controls up to two primary carrier-gas zones and up to four primary detector gas zones. In addition, up to four carrier gas mass flow controllers, pressure controllers, or pressure readouts may be set up as auxiliary PPC carrier zones, subject to a maximum of eight flow controllers (carrier plus detector flow) and twelve zones total in the PPC system. The auxiliary flow control zones do not accommodate split pneumatics, which must be configured as the primary carrier control zones on the GC.

### ***Auxiliary Carrier Gas Setup***

The PPC system stores information about the type, location, and calibration of its pneumatic devices in battery-backed-up random access memory (BRAM). This information is written into the BRAM during manufacturing; normally there is no need to access or modify the type or location of the PPC controllers. Some factory installed options use auxiliary carrier-gas zones. In order to use other auxiliary zones the operator must access the PPC connection menus on the GC touch screen and modify the type and location of the PPC devices. For auxiliary carrier-gas control you may choose either a carrier gas mass-flow controller, a pressure controller, or a pressure readout.

**CAUTION**

*Do not modify the PPC connection information if the PPC hardware configuration has not changed.*



### ***Auxiliary Carrier Gas Mass-Flow Controller Calibration***

The calibration procedure for mass-flow controllers designated as auxiliary carrier gas zones is the same as for controllers designated as primary carrier zones. Please refer to the section “**Mass-Flow Controller Calibration**” in this chapter.

### ***Auxiliary Carrier Gas Zone Operation***

Once the auxiliary carrier gas zone has been set up, you may modify the default setpoints in the active GC method, edit the carrier-gas setpoints in stored methods, or generate a new method and enter carrier-gas setpoints. The following parameters control the auxiliary carrier gas.

#### **Initial Auxiliary Carrier Gas Setpoint**

The auxiliary carrier gas zone takes setpoints of flow or pressure depending on the configured controller. It receives its initial setpoint from the active GC method. The initial setpoint is established upon setting up a method, ending a run, or resetting the instrument. The setpoint is held constant at the initial value if no additional auxiliary carrier entries are made in the GC method. The initial auxiliary carrier gas setpoint appears after touching **Carrier Program** button in the GC method.

#### **Auxiliary Carrier Gas Timed Events**

You may program auxiliary carrier-gas timed events to produce step changes in the setpoint. Auxiliary carrier-gas events can occur before or after a run starts. An auxiliary carrier-gas event supersedes the method setpoint value at the time the event occurs.

### ***Auxiliary Detector Gas Control***

The Clarus 600 GC controls up to four primary detector gas zones and up to two primary carrier-gas zones. In addition, up to four detector gas flow controllers may be set up as auxiliary PPC detector zones, subject to a maximum of eight flow controllers (carrier plus detector flow) and twelve zones total in the PPC system.

### **Auxiliary Detector Gas Setup**

The PPC system stores information about the type, location, and calibration of its pneumatic devices in battery-backed-up random access memory (BRAM). This information is written into the BRAM during manufacturing: normally there is no need to access or modify the type or location of the PPC controllers. Some factory installed detectors use auxiliary detector gas zones. In order to use other auxiliary zones the operator must access the PPC *CONNECTION* menus on the GC touch screen and modify the type and location of the PPC devices.

<p><b>CAUTION</b>      <i>Do not modify the PPC connection information if the PPC hardware configuration has not changed.</i></p>
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### **Auxiliary Detector Gas Flow Controller Calibration**

The calibration procedure for detector gas flow controllers designated as auxiliary detector gas zones is the same as for controllers designated as primary detector zones. Please refer to the section “**Detector Gas Flow Controller Calibration**” in this chapter.

### **Auxiliary Detector Gas Zone Operation**

Once the auxiliary detector gas zone has been set up, you may modify the default setpoints in the active GC method, edit the detector gas setpoints in stored methods, or generate a new method and enter detector gas setpoints. The following parameters control the auxiliary detector gas.

#### **Initial Auxiliary Detector Gas Setpoint**

The auxiliary detector gas zone takes flow setpoints. It receives its initial setpoint from the active GC method. The initial setpoint is established upon setting up a method, ending a run, or resetting the instrument. The setpoint is held constant at the initial value if no additional auxiliary detector gas entries are made in the GC method.

### **Auxiliary Detector Gas Timed Events**

You may program auxiliary detector gas timed events to produce step changes in the setpoint. Auxiliary detector gas events can occur before or after a run starts. An auxiliary detector gas event supersedes the method setpoint value at the time the event occurs.

## **PPC Tips and Techniques**

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This section contains tips and techniques that you will find useful when using PPC.

### **Effect of Flow on Pressurization Rate**

When first turned on, when a higher setpoint pressure is programmed, the PPC carrier-gas zones require some time to come up to pressure. The pressurization rate is a function of the type of inlet, the incoming carrier-gas flow rate and the outgoing column and septum purge flow rate (if applicable).

For typical packed columns the inlet will pressurize within one minute. For a POC inlet where the column flow rate is low it may take longer to reach a steady-state inlet pressure. The PPC gas-leak monitor will not trigger until five minutes have elapsed, which should be enough time in all cases.

For capillary or PSS inlets, setting a high inlet pressure and a low split flow rate (but not a zero flow) can result in the inlet *never* coming to pressure because the incoming split flow is less than the column plus septum purge rates. A zero split-flow rate is treated as a special condition for splitless injection and will not cause this problem. In general, use a minimum split flow rate that is the greater of 20 mL/min or four times the sum of the column and septum-purge flows.

**NOTE:** *If the column pressurization rate (psig/min) is too low, increase the split flow rate during this time.*

*If you want to quickly pressurize the column in the split mode, temporarily increase the split vent flow while pressurizing the column. You can set the split vent flow up to a maximum of 500 mL/min.*

### **Correcting the Column Dimensions**

The PPC controller relies on operator input of column dimensions to calculate pressure – flow and pressure – velocity relationships. If the column dimensions are incorrect, then the calculations will be inaccurate. In general, actual column diameters are within  $\pm 2 \mu\text{m}$  of the nominal specified value. Column lengths may vary for used columns because the ends may have been cut off.

Alternatively, the column dimensions may be unknown. In such cases set the Carrier mode to pressure and oventrack, if you need to maintain a constant column flow rate.

### **Correcting the Column Length**

The average linear gas velocity is the most accurate measure of capillary column conditions, because the low flow rates are difficult to measure. By measuring the average velocity you can compare it to the displayed velocity on the GC, and then make a correction to the column length. This procedure assumes that the column inner diameter entered into the GC is correct.

### **Set Up the Clarus 600 GC**

Install a column and turn on the GC. In **Configuration** set the *CAPILLARY CONTROL MODE ON*, and choose the *VELOCITY* mode. Enter the nominal column length and exact diameter. Select the split *FLOW* mode. In the **Method**, accept the default velocity of 30 cm/s, or choose another value. Set the oven temperature to at least 100 °C, isothermal.

### **Determine the Unretained Peak Time**

Inject 1–5 µL of methane (natural gas) and time how long it takes for the methane peak to elute from the column. This is best done by recording the methane chromatogram with a data handling system and reading the methane peak time off the chromatogram or report. You may use methylene chloride (CH<sub>2</sub>Cl<sub>2</sub>) vapor for an ECD.

### **Calculate the Actual Average Linear Gas Velocity**

Calculate the average linear gas velocity ( $\bar{u}$ ) from this formula:

$$\bar{u} = \frac{L_{PPC}}{t_M} \times 10$$

where  $L_{PPC}$  is the column length (in meters) entered in the Clarus 600 GC and  $t_M$  is the measured methane peak time in seconds.

### **Compute the Correction Factor**

Compare the measured velocity ( $\bar{u}$ ) to the displayed velocity ( $\bar{u}_{PPC}$ ) and compute the column length correction factor ( $f$ ):

$$f = \frac{\bar{u}_{PPC}}{\bar{u}}$$

If the correction factor is less than 0.8 or greater than 1.2, then you should double check the column diameter and nominal length for errors.

### **Enter the Corrected Column Length**

Now, go back to the GC configuration and enter the corrected column length ( $L_{new}$ ):

$$L_{new} = f \cdot L_{PPC}$$

### **Check the New Length**

Upon entering the new length the PPC controller will change the inlet pressure up or down to reflect the new conditions. If the measured velocity was less than the displayed value, then the PPC controller will reduce the pressure so that the measured velocity is now equal to the displayed value. If the measured velocity was higher, then the PPC controller will increase the inlet pressure. Check the new value by measuring the methane peak time again. Your calculated linear velocity should now be very close to the displayed value.

## ***Splitless Injection***

Splitless injection refers to a technique that has a zero split flow rate during injection with an increased split flow rate after injection is complete. There are two ways to accomplish splitless injection on the Clarus 600 GC.

### ***Zero Initial Split Flow***

For this method, you set the initial split flow to zero and enter a timed event after injection (typically at 1–2 minutes) that sets the split vent flow to 50–100

mL/min. The Clarus 600 GC will automatically turn off the split vent flow at the end of the run.

### ***Zero Split Flow by Pre-Run Event***

In some cases — with a PSS inlet in particular — it may not be desirable to have zero split vent flow while the instrument is cooling and recycling for another run. Set the initial split vent flow to 50–100 mL/min. Then enter a timed event at -2 minutes to set the split vent flow to 0, and enter another event after injection (typically at 1–2 minutes) that sets the split vent flow to 50–100 mL/min.

### ***Pressure-Pulse Injection***

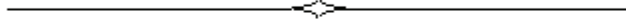
Pressure-pulse injection refers to a technique that facilitates sample transfer from inlet to column by raising the inlet pressure (column flow and velocity) above the normal level for injection, and then lowering it back to a normal setting for peak elution.

To perform pressure-pulse injection, enter a Pre-Run timed event at -1 minute that raises the pressure, flow, or velocity — depending on the operating mode — to a level 2–20 times the method's initial setpoint value. Then enter another timed event 1–2 minutes into the run that reestablishes the initial method setpoint. If you are using a carrier-gas program, then set a 1–2 minute step 1 hold time in the program. The carrier-gas program will “take over” the PPC system at the end of the hold period. To discharge the pressure-pulse, the split flow must have a non-zero volume at the time of discharge. To rapidly change the pressure pulse, the split flow should be zero or as large as possible during pressurization.

### ***Reducing Carrier-Gas Consumption***

You can significantly reduce carrier-gas consumption with a split or PSS inlet by setting the initial split flow to 20 mL/min, using a Pre-Run timed event at -2 minutes to increase the flow for split injection or to set the flow to 0 for splitless injection, and then reducing the split flow back to 20 mL/min after completion of the injection. You should delay the flow reduction until at least 5 minutes or longer after injection, or after a PSS inlet has reached its final temperature.

Don't forget to include a timed event at 1–2 minutes that sets the flow to 50–100 mL/min for splitless injection.





# Maintenance 9

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*Maintenance*

This chapter contains procedures for:


- **Autosampler Maintenance** — changing a syringe and replacing a vial-locator mechanism.
- **Syringe Maintenance** — cleaning the 5- $\mu$ L and 50- $\mu$ L syringe plungers.
- **Injector Maintenance** — changing septa, changing and repacking packed-injector liners, changing and repacking injector liners on the capillary (CAP), programmed split/splitless (PSS), and programmed on-column (POC) injectors, and changing the charcoal trap on the split/splitless injector.
- **ECD Maintenance** — baking out ECD cells, cleaning the ECD anode, and wipe testing an ECD cell.
- **FID Maintenance** — replacing the FID jet, cleaning the FID jet, replacing an O-ring in the FID collector, and cleaning the FID collector and cap.
- **PID Maintenance** — changing PID lamps, cleaning PID lamp windows, changing PID lamp window seals, and positioning disks.
- **NPD Maintenance** — changing and conditioning the NPD bead and replacing an NPD jet.
- **FPD Maintenance** — cleaning or replacing an FPD optical filter assembly, cleaning the detector liner, cleaning/replacing the detector window, replacing the photomultiplier tube, and cleaning and replacing the FPD jet.
- **PPC Maintenance** — replacing PPC module restrictors.

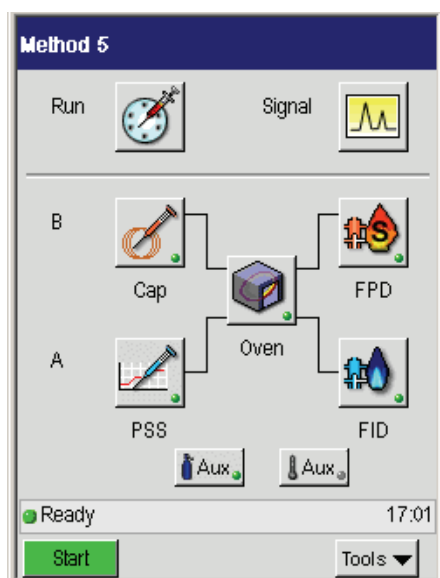
## ***Autosampler Maintenance***

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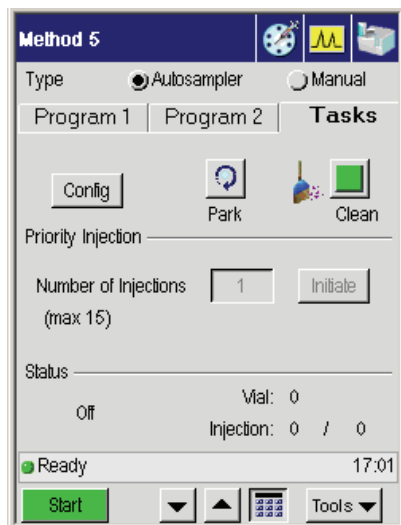
Autosampler maintenance consists of changing a syringe and replacing a vial locator mechanism.

### ***Changing a Syringe***

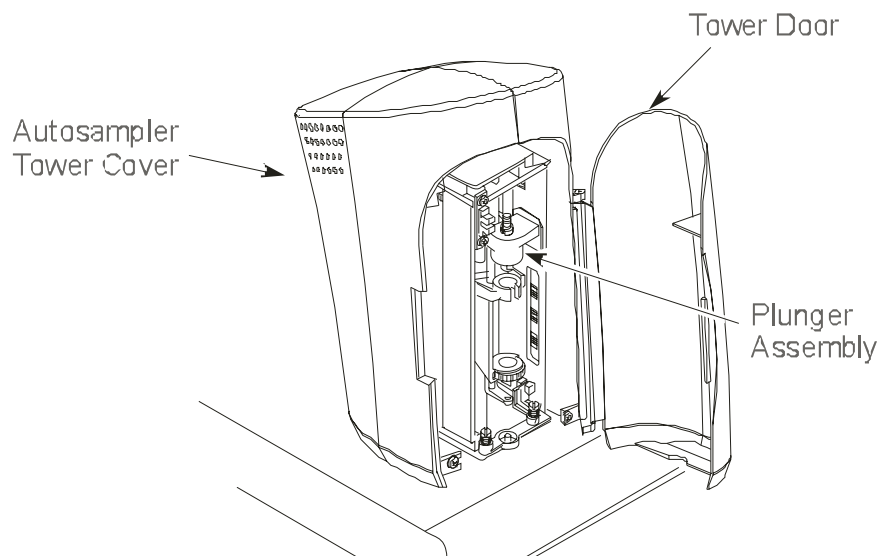
1. From the System Status screen touch the Run button  to display the run screen.



2. On the Run screen touch the **Autosampler** radio button and the **Tasks** tab as shown in the following screen.



3. Touch the **Park** button and the autosampler tower moves to the park position (facing the front of the Clarus 600 GC).
4. Open the tower door on the autosampler tower cover.

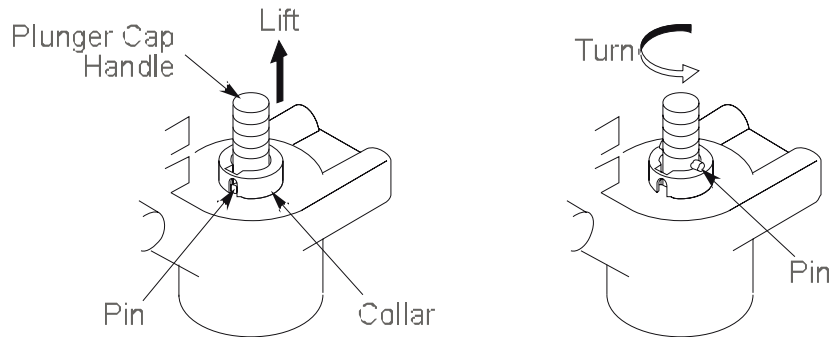


**Figure 9-1 Autosampler tower in the park position**

## Removing a Syringe

1. Locate the plunger assembly shown in the previous illustration.

Then, refer to the following figure, as you lift up the plunger cap handle and rotate it until it rests on the collar. Then release the plunger cap handle.



**Figure 9-2 Plunger assembly**

2. Hold the syringe by the barrel or syringe nut (see the following figure) and turn the carriage thumbscrew clockwise until the syringe is free.
3. Gently pull the top of the syringe forward until it just clears the carriage assembly.
4. Gently lift the syringe out of the carriage assembly.

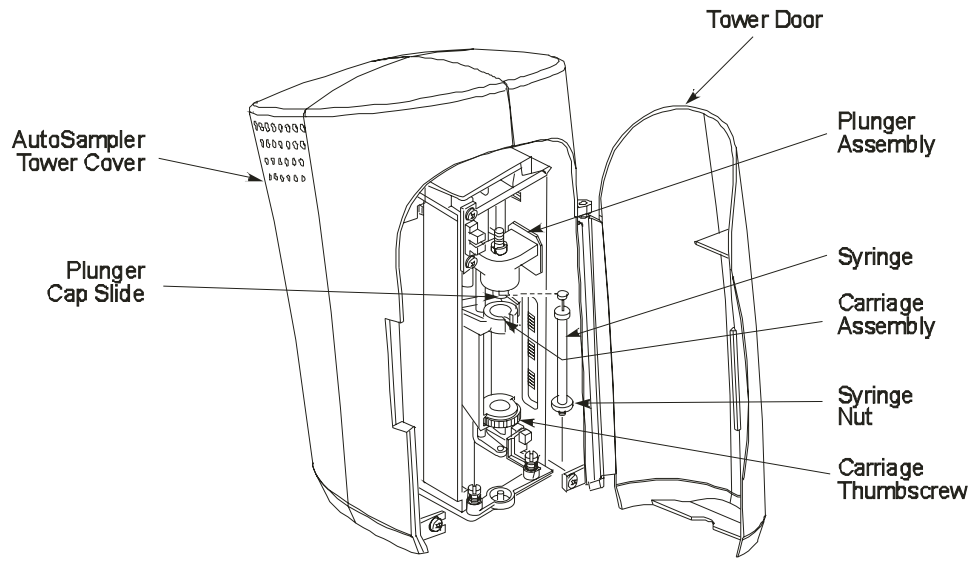


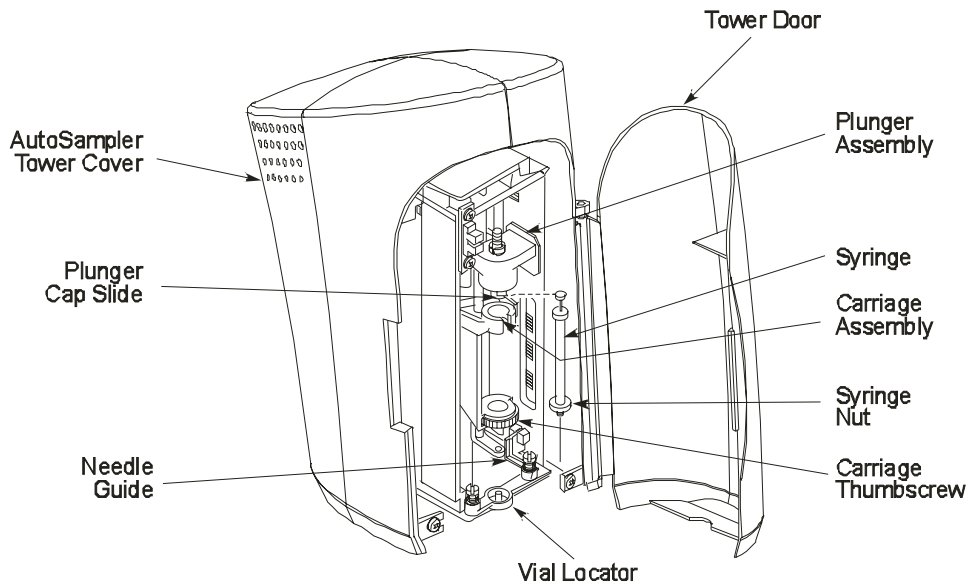
Figure 9-3 Removing a syringe

## ***Installing a Syringe***

---

Please refer to Figures 9-2 through 9-4 as you follow these steps.

1. Guide the needle through the hole in the carriage thumbscrew, then thread the needle through the needle guide. Use your fingers as a guide.
2. Rest the top of the plunger on the plunger cap slide, which is a shelf located on the underside of the plunger assembly.
3. While holding the syringe nut, engage the carriage thumbscrew on the threaded part of the syringe by turning the carriage thumbscrew counterclockwise.
4. Continue turning the thumbscrew counterclockwise. This slowly lowers the needle. Carefully guide the needle through the needle guide into the vial locator.
5. Tighten the carriage thumbscrew.



**Figure 9-4 Installing a syringe**



## ***Replacing the Vial-Locator Mechanism***

---

The vial-locator mechanism will wear out with extended use and require replacement. If the autosampler begins missing vials, or if the hole for the syringe needle begins to plug, it is an indication that you should replace the vial-locator mechanism

To replace a vial-locator mechanism:

1. Remove the two shoulder screws that secure the locator to the autosampler tower frame. Remove the two springs, then remove the vial locator. Discard the vial locator.
2. Mount the new vial locator (P/N N610-1182) on the autosampler tower frame.
3. Install the two shoulder screws through the two springs and into the vial locator. This secures the vial locator to the autosampler tower frame.



**WARNING**

*When securing the vial-locator molding, be sure that the flag is centered (not touching either side) in the sensor. If it touches a side, adjust the flag by loosening and then tightening the screws.*  
***DO NOT ADJUST THE SENSOR.***

Maintenance

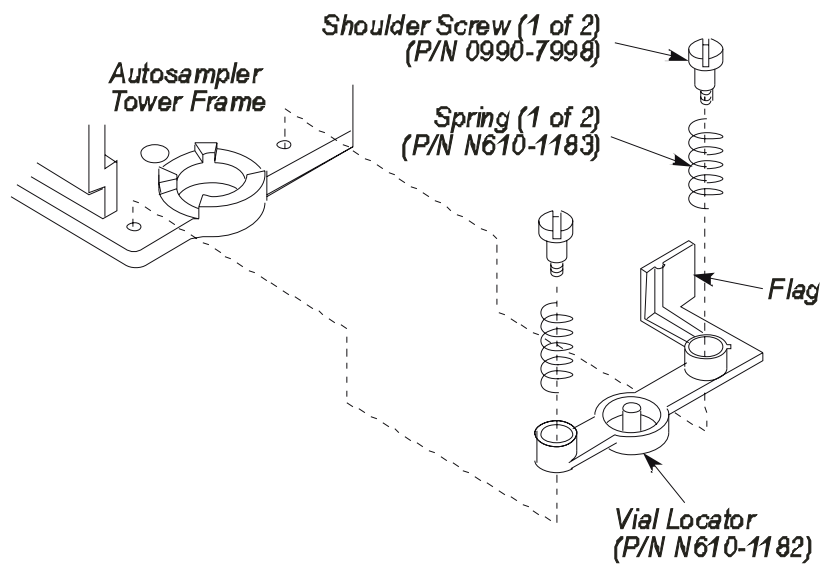


Figure 9-5 Exploded view of the vial locator

## **Syringe Maintenance**

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Syringe maintenance consists of cleaning the 5- $\mu$ L and 50- $\mu$ L syringe plungers and servicing idle syringes.

### **Cleaning the 5- $\mu$ L and 50- $\mu$ L Syringe Plungers**

The 5- $\mu$ L and 50- $\mu$ L syringe plungers should be cleaned regularly, after approximately 500 injections, since insolubles can build up and cause friction.

To clean the syringe plunger:

1. Remove the syringe using the procedure described in the preceding section.
2. Remove the plunger from the syringe barrel.
3. Wipe the plunger with a tissue soaked in an appropriate solvent.
4. Replace the plunger.
5. Pull and expel the same solvent through the barrel several times.
6. Replace the syringe using the procedure described in the preceding section.

**NOTE:** *Only syringes distributed by PerkinElmer should be used with the Clarus GC. Plungers are not interchangeable from syringe to syringe.*

### **Servicing Idle Syringes**

Syringes that are not used for several hours could "freeze," i.e., the syringe plunger will not move. To avoid this condition, **PARK** the tower, then remove and clean the syringe plunger as described above.

**NOTE:** *If you notice the Clarus GC precision degrading, replace the syringe. The Clarus GC syringe is a consumable part. After extended use, you will need to replace it.*

## ***Injector Maintenance***

---

**CAUTION**      *If you are analyzing reactive compounds, you should use deactivated liners and wool which are appropriate for your sample type.*

Injector maintenance consists of changing septa, changing and repacking injector liners, changing the hourglass needle guide on the POC injector, changing and repacking CAP and PSS injector liners, removing a broken liner from the PSS injector body, changing the charcoal trap or replacing charcoal on the split/splitless CAP and PSS injectors.

### ***Changing Septa***

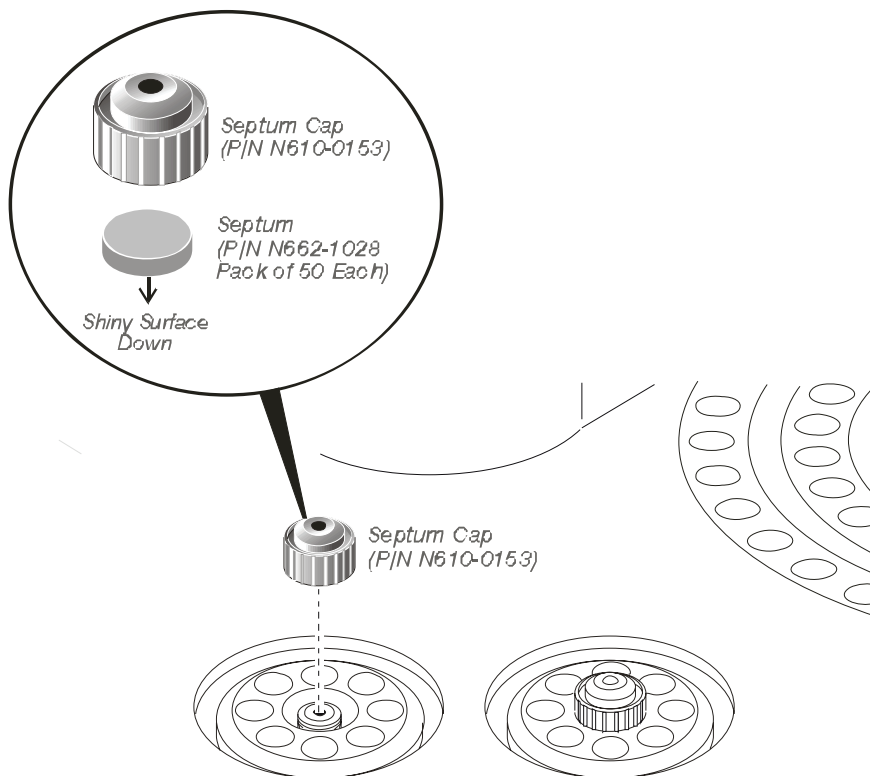
Septa should be replaced on a regular basis. How often depends on the type of septa used, the temperature of the injection port, and the number of injections made.

The septum shipped with your instrument is a Thermogreen LB-2 Septa (P/N N662-1028, package of 50). This septum can handle over 200 injections at moderate temperatures.

To change a septum:

1. Turn off the injector heater and allow the injector to cool.
2. Remove the septum cap.
3. Pry the old septum from the septum cap with a screwdriver.
4. Insert a new septum in the septum cap.
5. Replace the septum cap.

**NOTE:** To minimize the possibility of contamination, avoid unnecessary handling of septa.



**Figure 9-6 Changing a septum**

### ***Changing and Repacking Packed Column Injector Liners***

To improve the performance of the injector used with packed columns, insert a small amount of quartz wool (P/N N610-2354) into the top portion of the injector liner (P/N N610-1048). The quartz wool accomplishes two things: (1) it wipes the end of the syringe needle to insure that reproducible sample volumes are injected, and (2) it retains any nonvolatile components present in your sample, making cleaning the liner easier.

## Maintenance

The injector liner should be removed and the wool packing replaced on a regular basis, particularly if your samples contain nonvolatile components that could build up on the wool. This could cause adsorption of peaks of interest, tailing, and loss of sensitivity.

You can remove the wool with a small hook on the end of a thin wire, or blow it out using compressed air.

To remove a packed injector liner and install new wool:

1. Turn off the injector heater.

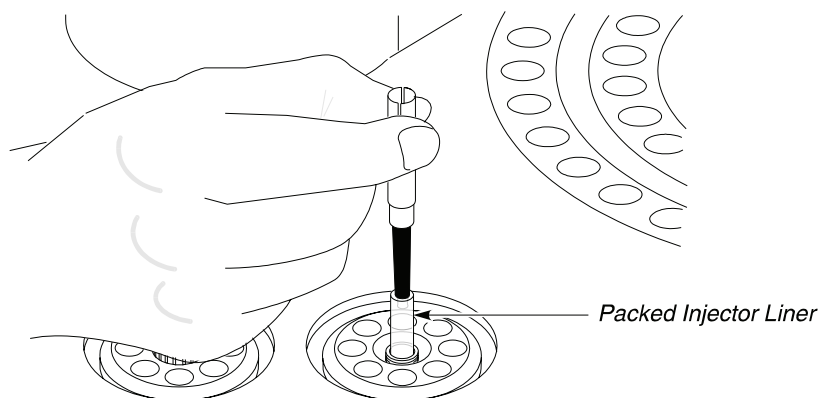
Allow the injector to cool until it is slightly warm to the touch. Cooling the injector to a temperature that is too low (<100 °C) will make it difficult to remove the injector liner.

2. Remove the septum cap (see Figure 9-6).
3. Remove the septum shield (P/N N610-1050) with the large end of the liner-removal tool (P/N N610-0102).



**Figure 9-7 Liner-Removal Tool (P/N N610-0102)**

4. Press the small end of the liner-removal tool into the injector liner, then pull the injector liner out.



**Figure 9-8 Removing the packed column injector liner**

**NOTE:** *To avoid contaminating the quartz wool when packing the injector liner, wear vinyl, powder-free, disposable gloves (for example, the same type of gloves used to perform maintenance on a mass spectrometer).*

5. Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the injector liner. Then, using the supplied 1/16-inch rod (P/N N610-T100), push the quartz wool into the injector liner. *Loosely* pack a 1-inch (2.5 cm) piece of quartz wool into the top portion of the liner (see the following figure).
6. Replace the injector liner, septum shield, and septum cap.



Figure 9-9 Packed column injector liner (P/N N610-1048) packed with wool

### ***Changing the Hourglass Needle Guide on the Programmed On-Column (POC) Injector***

To change the hourglass needle guide:

1. Turn off the injector heater. Allow the injector to cool until it is slightly warm to the touch.
2. Remove the septum cap.

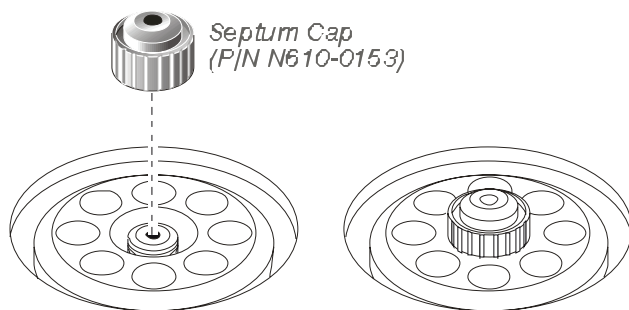
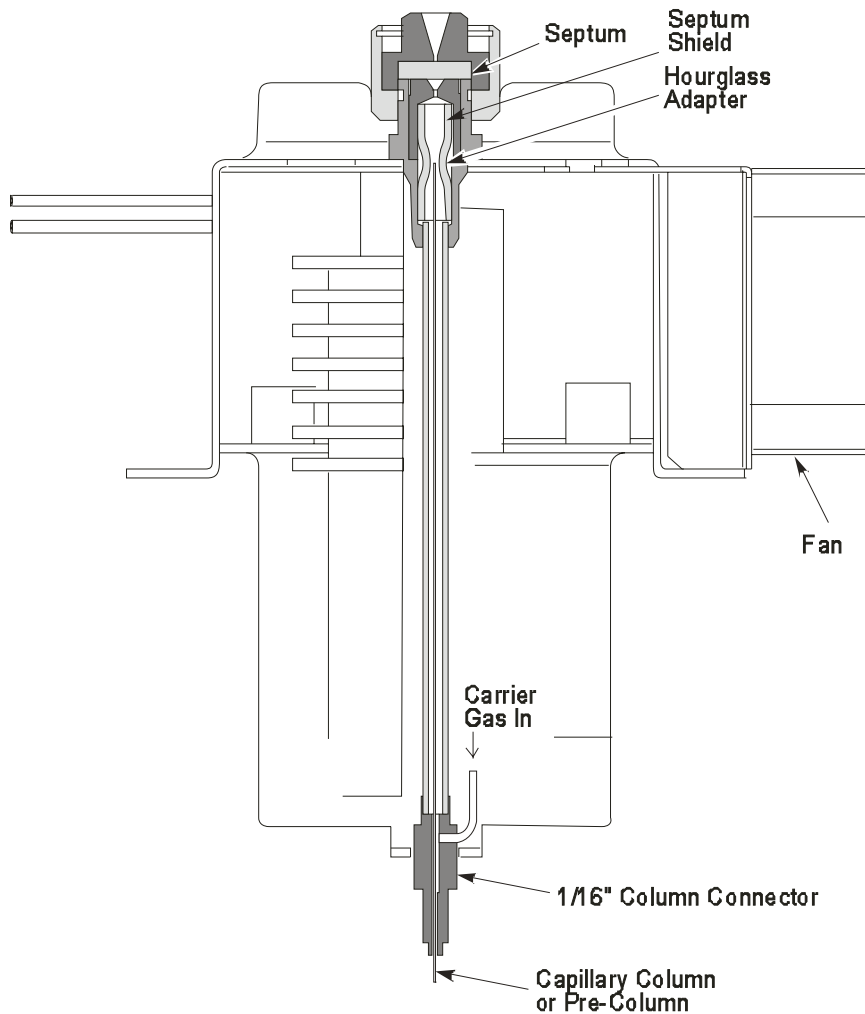


Figure 9-10 Removing a septum cap



3. Remove the septum shield (P/N N610-1702) with the large end of the liner-removal tool (P/N N610-0102).
4. Remove the hourglass needle guide (see the following figure) with a pair of tweezers or small pliers.



**Figure 9-11 Cutaway view of the POC injector showing the location of the hourglass needle guide**

## Maintenance

5. Install the new hourglass needle guide (P/N N610-1703).
6. Replace the septum shield.
7. Replace the septum cap.
8. Reinstall your column.

Refer to Chapter 6, Installing a Capillary Column, for the proper procedures.

## **Changing and Repacking Capillary Split/Splitless (CAP) and Programmed Split/Splitless (PSS) Injector Liners**

The procedure below is applicable to the following injector liners:

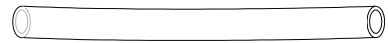
<b>Injector Liner</b>	<b>Size</b>	<b>Part Number</b>
CAP wide-bore liner	4.0-mm i.d. and a 6.0-mm o.d.	N612-1001
CAP narrow-bore liner	2.0-mm i.d. and a 6.0-mm o.d.	N612-1002
PSS wide-bore liner	2.0-mm i.d. and a 4.0-mm o.d.	N612-1004
PSS narrow-bore liner	1.0-mm i.d. and a 4.0-mm o.d.	N612-1006
PSS on-column liner	--	N610-1539

### **Removing a CAP or PSS Injector Liner**

The liner-removal procedure is similar for CAP and PSS wide-bore and narrow-bore liners. To remove the liners, you need a CAP liner-removal tool (P/N 0250-6534) or a PSS liner-removal tool (P/N 0250-6247) as shown below.



**CAP Liner-Removal Tool**  
P/N 0250-6534



**PSS Liner-Removal Tool**  
P/N 0250-6247

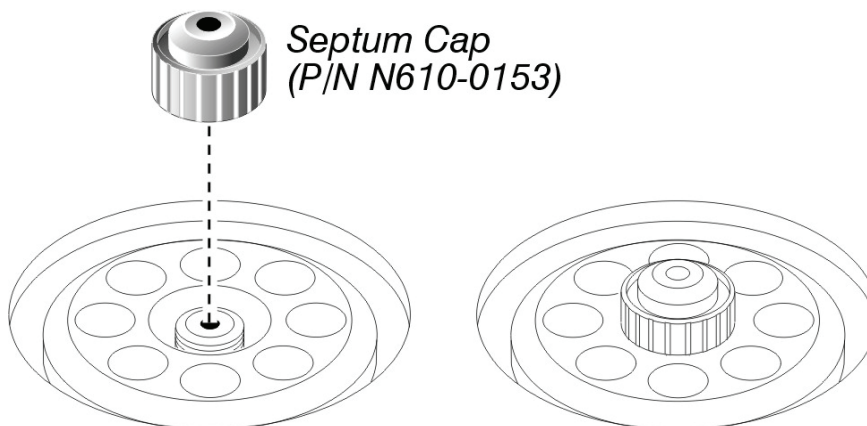
**Figure 9-12 CAP and PSS liner-removal tools**

To remove a capillary injector liner:

1. Turn off the injector heater.

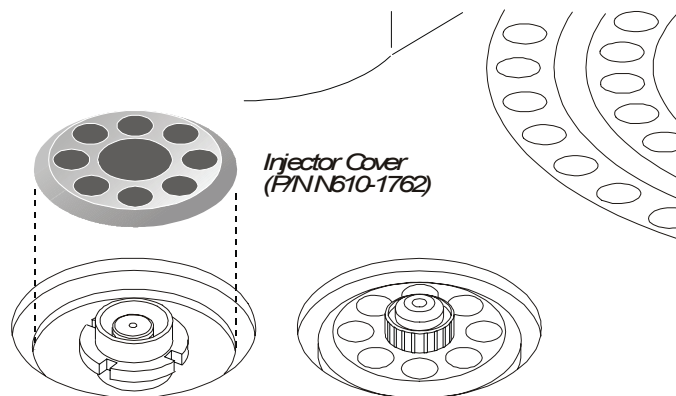
Allow the injector to cool until it is slightly warm to the touch. **Cooling the injector to a temperature that is too low (<80 °C) will make it difficult to remove the injector liner.**

2. Remove the septum cap.



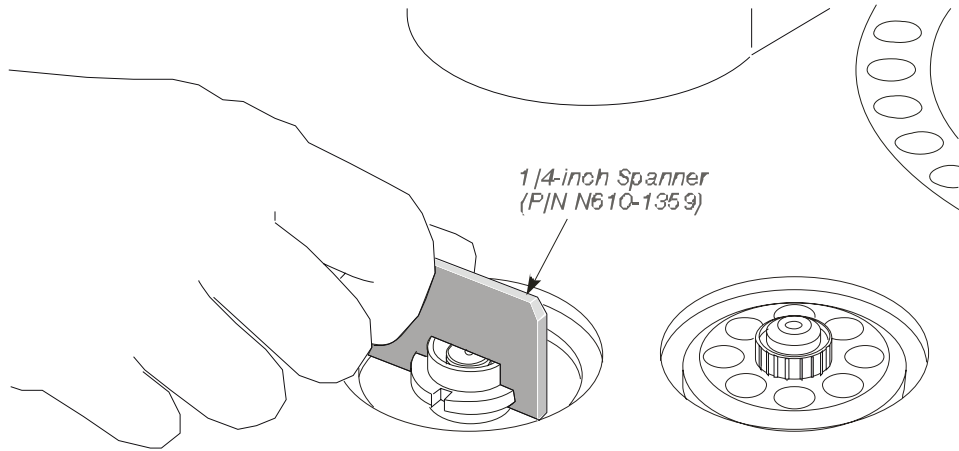
**Figure 9-13 Removing the septum cap**

3. Remove the injector cover.



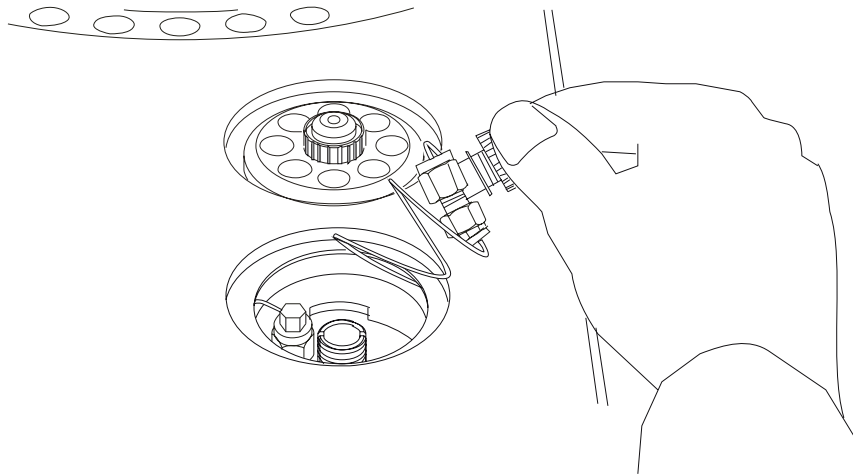
**Figure 9-14 Removing the injector cover**

4. Loosen the threaded collar using the spanner (P/N N610-1359) provided, then remove the threaded collar.



**Figure 9-15 Loosening the threaded collar**

5. Replace the septum cap on the injector.
6. Pull the septum cap upwards to remove the septum purge assembly.



### Figure 9-16 Removing the septum purge assembly

The carrier gas inlet line is coiled. This allows you to pull the septum purge assembly over to the side and gain access to the injector liner.

**NOTE:** *The inlet line used in PPC capillary injectors (CAP and PSS) is coiled but the septum purge assembly does not terminate in a snubber as shown. Instead it is connected to a PPC module. The coil of the inlet line is long enough so that you can pull the septum purge assembly out of the opening in the top cover and gain access to the injector liner.*

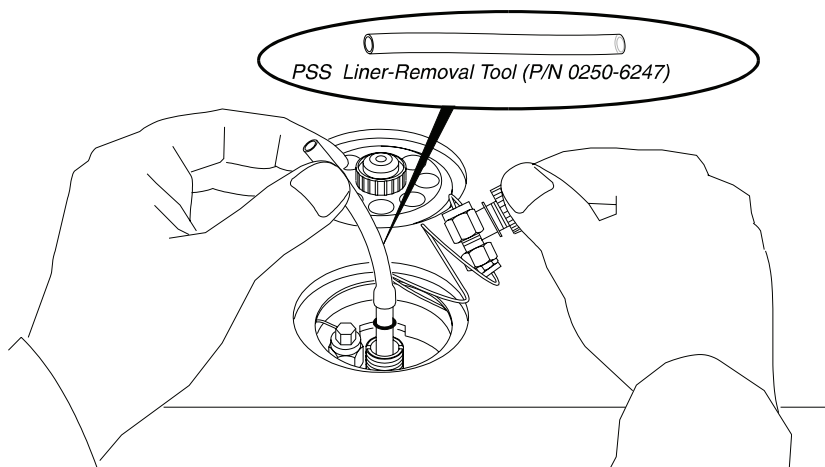
7. Insert the CAP liner-removal tool (P/N 0250-6534) over the end of the CAP liner and lift the liner out of the injector.

**OR**

Insert the PSS liner-removal tool (P/N 0250-6247) over the end of the PSS liner and lift liner out of the injector.

**CAUTION**

*The liner must be cool (no hotter than 100 °C) or the liner-removal tool will melt! The end of the liner-removal tool may flare out with use. If this happens,*



**Figure 9-17 Removing an injector liner**

**NOTE:** *If the quartz liner breaks inside the CAP injector, it can be removed by first removing the column, then removing with a 9/16-inch wrench the 1/4-inch injector fitting that is inside the oven. The liner should fall out of the injector with the fitting. If the liner is stuck, you can push it out from the top or bottom of the injector.*

<b>CAUTION</b>	<i>The PSS injector liner does not have a 1/4-inch fitting like the CAP injector. Be very careful when removing this liner to prevent breaking it. Do not cool the injector below 80 °C. This will make it easier to remove the liner and O-ring. As the injector cools, the O-ring adheres to the metal base.</i>
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**NOTE:** *Each capillary liner has an O-ring installed on the frosted portion of the CAP liner and on the part furthest away from the dimple on the PSS injector. If the O-ring has adhered to the injector, you may not be able to easily remove the liner (step 7 above). If this is the case, use a small screwdriver to dislodge the O-ring before removing the liner and O-ring.*

**About O-Rings**

<b>CAUTION</b>	<i>Each time a capillary injector liner is removed, you should replace the O-ring, especially if the O-ring adheres to the injector body and you had to pry it loose with a screwdriver. This action may damage the O-ring thereby causing a bad seal if the damaged O-ring is reinstalled.</i>
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If your results produce background contamination when a new O-ring is first installed, condition the injector at the maximum temperature of the O-ring (listed below). Depending on the type of column used, you may first want to remove the column before baking it out at a high temperature.

**NOTE:** *High-temperature seals should be used at temperatures of 300 °C or higher. These seals are available in Kalrez or graphite from PerkinElmer's catalog service (in the U.S. dial: 1-800-762-4000) or on-line at [www.perkinelmer.com](http://www.perkinelmer.com). Viton maximum temperature of 250 °C is recommended for the mass spectrometer.*

Injector O-Rings	Recommended Maximum Temperature
<b>CAP Injector</b>	
N610-1374 Silicone (pkg. of 10)	250 °C
N610-1378 Graphite (pkg. of 5)	450 °C
N930-2782 Kalrez (pkg. of 1)	450 °C
N930-2783 Viton (pkg. of 1)	250 °C (not recommended for use with ECD)
<b>PSS Injector</b>	
N610-1751 Graphite (pkg. of 5)	450 °C
0992-1004 Kalrez (pkg. of 1)	450 °C
N610-1747 Viton (pkg. of 10)	250 °C (not recommended for use with ECD)

### ***Selecting an Appropriate CAP Injector Liner***

Select the correct CAP liner for your application and pack it with quartz wool. The CAP injector uses the following two liners:

- CAP wide-bore liner (P/N N612-1001); 4.0-mm i.d. and 6.0-mm o.d.
- CAP narrow-bore liner (P/N N612-1002); 2.0-mm i.d. and 6.0-mm o.d.

The narrow-bore liner is generally used for a splitless injection, and the wide-bore liner is generally used for a split injection. Due to the small internal volume (0.3 mL) of the narrow-bore liner, prevent overfilling the liner with vapor (caused by solvent expansion upon injection) by limiting the amount of sample injected to 0.5 µL. The wide-bore liner is used for splitless injection volumes over 0.5 µL, since its internal volume is 1.25 mL. The sample size should be

## Maintenance

limited to a maximum of 2  $\mu\text{L}$  for hydrocarbon solvents, and less than that for high-expansion solvents such as water or  $\text{CH}_2\text{Cl}_2$ .

If the wide-bore liner is used for splitless injection, the splitless sampling time (the vent-on time) should be more than one minute. Also, lower initial oven temperatures may be required to give good resolution in the first few minutes after the solvent peak elutes. The wide-bore liner should be used with columns having an i.d. of 0.32 mm or greater.

### **Packing the CAP Injector Liner with Quartz Wool**

We recommend packing a small amount of quartz wool in the top portion of the liner to wipe the syringe needle regardless of the liner type or injector mode (for example, split or splitless). This packing assures that reproducible volumes are injected by wiping the syringe needle every time it is inserted.

Remove the liner and replace the quartz wool packing on a regular basis, particularly if your samples contain nonvolatile components that could build up on the wool. This buildup could cause adsorption of peaks of interest, tailing, and loss of sensitivity.

Remove the wool by making a small hook on the end of a thin wire and using that to pull it out, or blow it out using compressed air.

**NOTE:** *To avoid contaminating the quartz wool when packing the injection liner, wear vinyl, powder-free, disposable gloves (for example, the same type of gloves used to perform maintenance on a mass spectrometer).*

### **Packing a CAP Injector Liner for Split Operation**

Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the liner. Then using the supplied 1/16-inch rod (P/N N610-T100), push the quartz wool into the liner. Pack the wool snugly\* from the dimple upwards [about 1 in. (2.5 cm)]. Loosely pack quartz wool in the top portion of the liner to wipe the syringe needle upon injection.

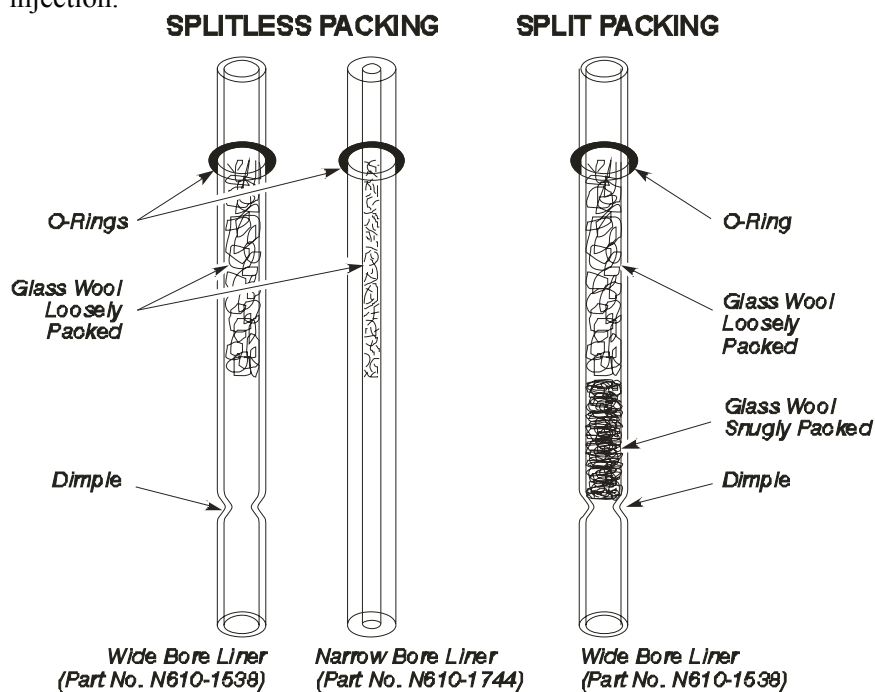
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\* The recovery of high-molecular-weight components (e.g.,  $\text{C}_{40}$  and higher) may be improved if the liner is packed loosely.



**Packing a CAP Injector Liner for Splitless Operation**

Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the liner. Then using the supplied 1/16-inch rod (P/N N610-T100), push the quartz wool into the liner. Pack a 1-inch (2.5 cm) piece of quartz wool *loosely* below the top ground portion of the liner (see the following figure). The sample is then injected into the wool, thereby preventing the delivery of sample beyond the column. The wool also wipes the syringe needle upon injection.



**Figure 9-18 CAP injector liners packed with quartz wool**

## Maintenance

**NOTE:** *As you can see in Figure 9-18, each CAP injector liner has an O-ring installed on the ground portion. If the O-ring has adhered to the liner, it may not be easy to remove the liner. (If this is the case, use a small screwdriver to dislodge the O-ring before removing the liner and O-ring).*

### **Reinstalling the Liner in the CAP Injector**

1. Install a new O-ring near the ground portion of the liner.
2. Insert the liner in the injector body.
3. Place the septum purge assembly over the liner.
4. Press the septum purge assembly down to correctly position the liner in the injector.

### **Selecting an Appropriate PSS Injector Liner**

Select the correct PSS liner for your application and pack it with quartz wool. The PSS injector uses the following three liners:

- PSS injector wide-bore liner (P/N N612-1004); 2.0-mm i.d.
- PSS injector narrow-bore liner (P/N N612-1006); 1.0-mm i.d.
- PSS injector on-column (hourglass) liner (P/N N610-1539)

In general, operate the PSS in the inlet-programmed mode with the 2-mm or 1-mm-i.d. liner for PSS split or splitless injection. For PSS on-column operation, use the hourglass liner and the oven-program mode.

The 2-mm-i.d. PSS liner that is used for either split or splitless operation should be packed with quartz wool as described in this chapter. The 1-mm-i.d. PSS liner may give better early-eluting peak resolution in the split or splitless mode. It should be used for samples with early-eluting peaks for which additional solute trapping/focusing cannot be obtained by lowering the initial oven temperature or by using a column with thicker stationary-phase film.

## **Packing the PSS Injector Liner with Quartz Wool**

**CAUTION**     *Never pack the hourglass liner with quartz wool.*

Pack a small amount of quartz wool in the top portion of the liner to wipe the syringe needle regardless of the liner type or injector mode (for example, split or splitless). Packing assures that reproducible volumes are injected by wiping the syringe needle every time it is inserted.

Remove the liner and replace the quartz wool packing on a regular basis, particularly if your samples contain nonvolatile components that could build up on the wool. This could cause adsorption of peaks of interest, tailing, and loss of sensitivity. You can remove the wool by making a small hook on the end of a thin wire and using that to pull it out, or blow it out using compressed air.

**NOTE:** *To avoid contaminating the quartz wool when packing the injection liner, wear vinyl, powder-free, disposable gloves (for example, the same type of gloves worn when performing maintenance on a mass spectrometer).*

### **Packing a CAP Injector Liner for Split Operation**

Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the liner. Then, using the supplied 1/16-inch rod (P/N N610-T100), push the quartz wool into the liner. Pack the wool ***tightly***\* from the dimple upwards [about 1 in. (2.5 cm)]. ***Loosely*** pack quartz wool in the top portion of the liner to wipe the syringe needle upon injection.

---

\* The recovery of high-molecular-weight components (e.g., C<sub>40</sub> and higher) may be improved if the liner is packed loosely.

### Packing a CAP Injector Liner for Splitless Operation

Take a small piece of quartz wool and twist it into an elongated shape so that you can insert it into the liner. Then, using the supplied 1/16-inch rod (P/N N610-T100), push the quartz wool into the liner. Pack a 1-inch (2.5 cm) piece of quartz wool *loosely* below the top ground portion of the liner (see the following figure). The sample is then injected into the wool, thereby preventing the delivery of sample beyond the column. The wool also wipes the syringe needle upon injection.

**NOTE:** *The narrow-bore liner is more difficult to pack because of its small inner diameter. However, there is a dimple in the middle of the liner to hold the wool in place. Do not pack the wool too tightly!*

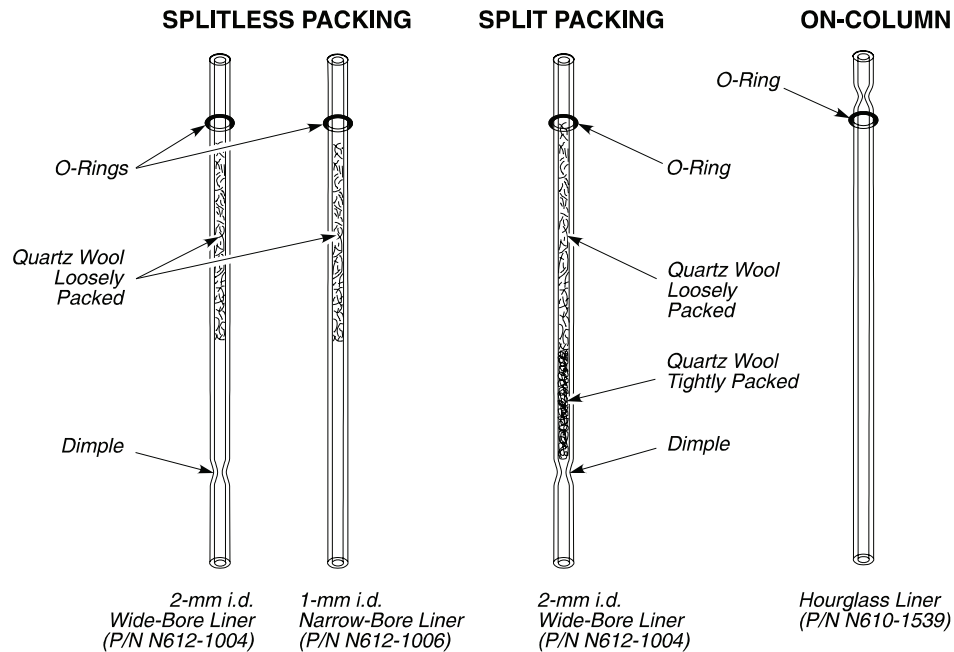


Figure 9-19 PSS injector liners packed with quartz wool

**NOTE:** *As you can see in the preceding figure, each PSS injector liner has an O-ring installed near the end furthest away from the dimple. If the O-ring has adhered to*

*the liner, it may not be easy to remove the liner. If this is the case, use a small screwdriver to dislodge the O-ring before removing the liner and O-ring.*

### **Reinstalling the Liner in the PSS Injector**

1. Install a new O-ring on the top portion of the liner.
2. Insert the liner in the injector body.
3. Place the septum purge assembly over the liner.
4. Press the septum purge assembly down to correctly position the liner in the injector.

Make sure that you **secure** the septum purge assembly **tightly** to the injector base with the 1/4-inch spanner.

### **Removing a Broken Liner from the PSS Injector Body**

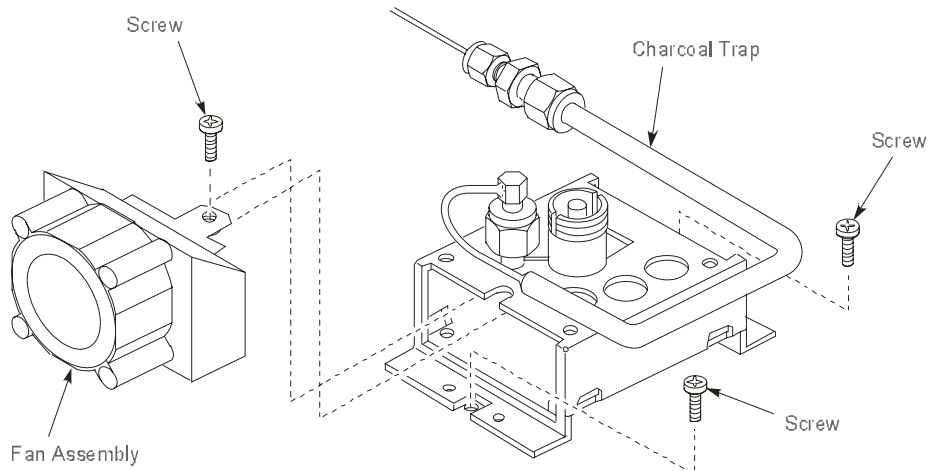
If a liner breaks in the PSS injector body, the best way to remove all pieces of quartz is to remove the injector from the Clarus GC.

To remove a broken PSS liner from the injector:

1. Turn off the Clarus GC. Allow the injector to cool until it is slightly warm to the touch.
2. Disconnect and remove the column from the injector.
3. Loosen the two screws that secure the Clarus GC top cover and raise the top cover until it locks in the raised position.
4. Remove the top cover from the PSS injector. Mark the position of the injector on the metal deck with a pencil.
5. Remove the screw that secures the fan assembly to the PSS injector, then remove the fan assembly (see the following figure).
6. Remove the two screws that secure the PSS injector to the metal deck.

## Maintenance

7. Remove the cable clamps that hold the heater and sensor wires and the gas tubing (see the following figure).
8. Carefully lift the PSS injector out of the Clarus GC. Invert the PSS injector and remove all of the broken quartz liner from it.
9. Reinstall the PSS injector to the Clarus GC and reinstall the fan assembly.
10. Lower the Clarus GC top cover and properly align the PSS injector with the top cover before you completely tighten the PSS injector mounting screws.

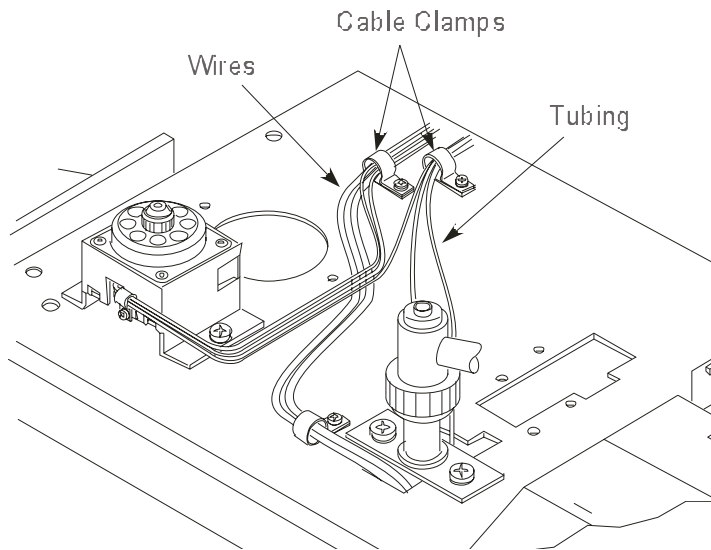


**Figure 9-20 Removing the fan assembly from the PSS injector**

### CAUTION

**To prevent autosampler needle damage after the Clarus GC top cover has been opened and closed, verify that the autosampler tower is aligned with both injectors.**

*Do this by manually rotating the autosampler tower and stopping over injector 1 and injector 2 to check that the vial locator is in the center of the septum cap. If the vial locator does not align with the center of the septum cap, loosen the two hold-down screws that secure the top cover (see Figure 9-22). Then move the top cover so that the vial locator is aligned with the center of the septum cap. Secure the Clarus GC top cover in this position by tightening the two screws.*



**Figure 9-21 Location of the cable clamps that secure the wires and tubing**

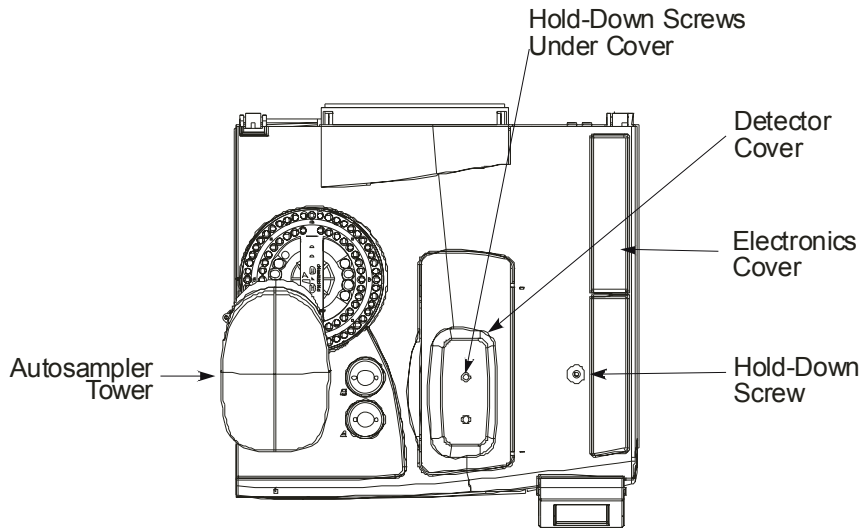
## ***Changing the Charcoal Trap or Replacing Charcoal on the Split/Splitless CAP and PSS Injectors***

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The charcoal trap will eventually become saturated. When this occurs, ghost peaks and changes in split ratio will be observed.

### ***Removing a Charcoal Trap***

1. Turn off the Clarus GC. *Allow the injectors/detectors to become cool to the touch.*
2. Loosen the two hold-down screws on the top cover of the Clarus GC (see following figure) and raise the top cover until the cover locks in the raised position.



**Figure 9-22 Location of the top cover hold-down screws**

3. Remove the septum cap, then remove the top cover from the injector.
4. Loosen the threaded collar using the 1/4-inch spanner (P/N N610-1359) provided, then remove the threaded collar.
5. Replace the septum cap on the injector.

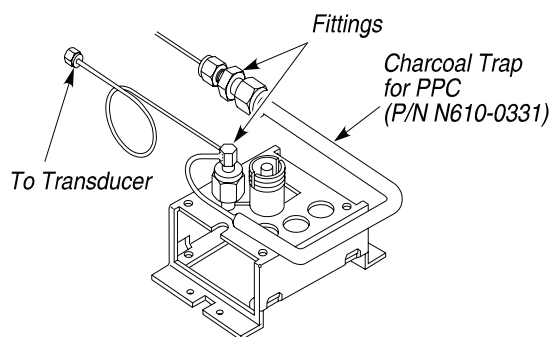


6. Pull the septum cap upwards to remove the septum purge assembly.
7. Using an 1/8-inch wrench, loosen the fittings that are connected to the charcoal trap and remove the charcoal trap (see the following figure). If you have a PPC charcoal trap, use a 1/4-inch wrench to loosen the fitting that connects the trap to the transducer.

### **Installing a New Charcoal Trap**

1. Install a new PPC version charcoal trap (P/N N610-0331) or just replace the charcoal in your current trap.

Replace the charcoal by removing the glass wool plug from the 1/4-inch tubing end. Empty the old charcoal and the glass wool plug at the 1/8-inch end. Push a small glass wool plug from the 1/4-inch end to the end of the 1/4-inch tube (near the 1/8-inch end) then fill with charcoal (P/N 0330-0904) and plug the end with a small piece of silanized glass wool.



**Figure 9-23 Charcoal Trap on an injector controlled by PPC**

2. Replace the septum purge assembly and remove the septum cap.
3. Replace the threaded collar and tighten it using the spanner.
4. Replace the injector cover then replace the septum cap.
5. Lower the Clarus GC top cover and tighten the two hold-down screws.

**CAUTION**

**To prevent autosampler needle damage after the Clarus GC top cover has been opened and closed, verify that the autosampler tower is aligned with both injectors.**

*Do this by manually rotating the autosampler tower and stopping over injector 1 and injector 2 to check that the vial locator is in the center of the septum cap. If the vial locator does not align with the center of the septum cap, loosen the two hold-down screws that secure the top cover (see Figure 9-22). Then move the top cover so that the vial locator is aligned with the center of the septum cap. Secure the Clarus GC top cover in this position by tightening the two screws.*

## ***ECD Maintenance***

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If you observe that the ECD background is higher than normal for your operating conditions, the cell could be contaminated.

You can view the ECD background reading from the Clarus GC touch screen by selecting the signal tab in the ECD tab. Also select the signal tab to display the detector background and the **AutoZero** button. Press the **AutoZero** button to have the system on autozero.

Under normal operating conditions, the ECD background will be up to 7 mV.

If you suspect cell contamination, first eliminate column bleed by lowering the oven temperature to ambient. If bleed is not the problem and the high background coincided with changing the carrier gas tank, the carrier gas may be contaminated. To check for this condition:

1. Cool the ECD to <100 °C.
2. Remove the column from the ECD, then cap the ECD with a 1/8 inch steel plug (P/N 0990-3098).
3. Increase the make-up flow. (The ECD is a concentration-sensitive detector. Increasing the make-up gas flow would normally dilute the contamination and cause a decrease in the background.)

If the background remains the same or increases as the make-up flow increases, the carrier gas could be contaminated. If bleed or carrier gas contamination is not the problem, bake the ECD using the following procedure.

### ***Baking the ECD***

1. Remove the column, then cap the ECD with a plug (P/N 0990-3098).
2. Increase the flow of make-up gas to 60 mL/min and raise the detector temperature to 450 °C.

## Maintenance

3. Bake the system until the background returns to normal levels. This could take from hours to several days.

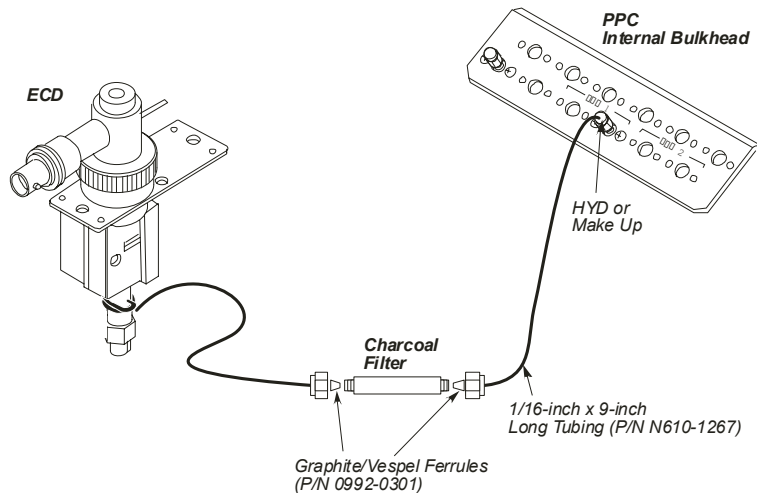
**NOTE:** *It may also help to remove the column and increase the oven temperature to 450 °C to bake out the lower portion of the ECD body.*

## Changing the Charcoal Filters

The ECD is shipped with charcoal filters (P/N N660-0037) installed in the make-up and injector pneumatics lines to remove contamination from the needle valve, flow controller, or pressure regulator. The filters should be replaced periodically.

To change charcoal filters:

1. Turn off the Clarus GC and allow the injectors/detectors to cool.
2. Loosen the two hold-down screws on the GC top cover (see Figure 9-22) and raise the top cover until it locks in the raised position.
3. Disconnect the charcoal filters from the make-up gas and injector lines.
4. Install new charcoal filter filters.



## Cleaning the ECD Anode



### WARNING

*THE FOLLOWING PROCEDURE SHOULD BE PERFORMED ONLY ON ECD'S THAT HAVE A VALID ACCEPTABLE WIPE TEST RESULT WITHIN THE LAST SIX MONTHS.*

*PERFORMING UNAUTHORIZED CLEANING PROCEDURES SUCH AS STEAM CLEANING OR FLUSHING SOLVENTS THROUGH THE ECD ARE STRICTLY FORBIDDEN AND ARE KNOWN TO DISCHARGE RADIOACTIVE CONTAMINATION FROM THE SOURCE. SOURCES WHICH ARE CLEANED IN THIS FASHION SHOULD BE CONSIDERED HOT.*

**NOTE:** *If a dirty or contaminated ECD is suspected, try baking out the detector before using this procedure.*

**NOTE:** *Wear plastic or rubber gloves when cleaning the ECD anode.*

To clean the ECD anode:

1. Turn off the ECD heater and allow the system to cool to room temperature.
2. Unscrew the knurled ring and lift out the anode assembly (see the following figure).
3. Place the collector assembly on top of a beaker of hexane with the anode tube submerged and soak for several minutes. **DO NOT** submerge the side arm in the hexane; submerge only the anode.
4. Remove the anode assembly and wipe it dry with a tissue.
5. Replace the anode assembly, then tighten the knurled ring.
6. Turn on the detector temperature and observe that the background signal has returned down to a normal level.

## Maintenance

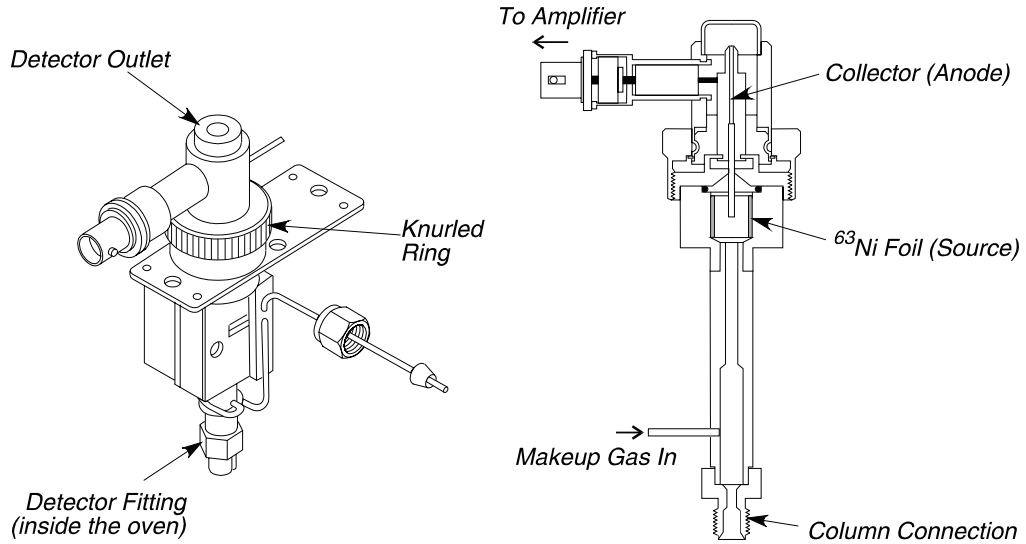


Figure 9-24 Isometric view and cross section view of the ECD

### Wipe Testing an ECD Cell

#### CAUTION

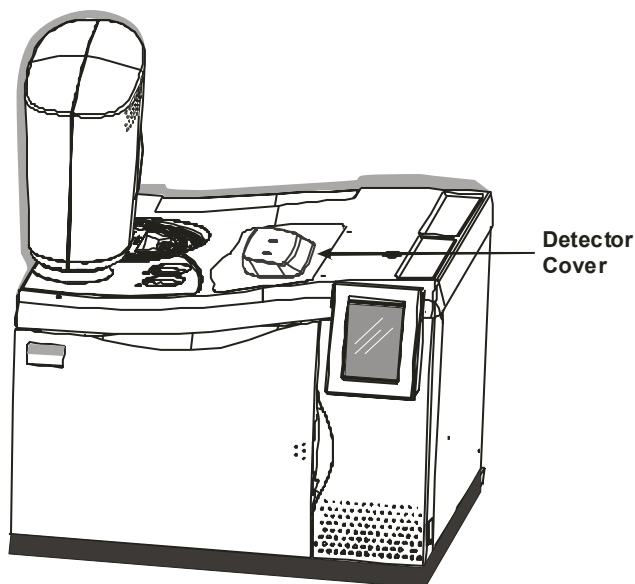
*Until the results of the wipe test are known, use caution and suitable protection when handling the cell and equipment in contact with it. Wear disposable plastic or rubber gloves when performing this test.*

It is strongly recommended that you become familiar with the NRC regulation covering the use of Nickel-63, as well as any other national, state, or local requirements.

To perform the wipe test:

1. Turn the instrument off and allow the detector to become cool to the touch.

- Gain access to the detector by lifting the detector cover (see the following figure).



**Figure 9-25 Detector cover location**

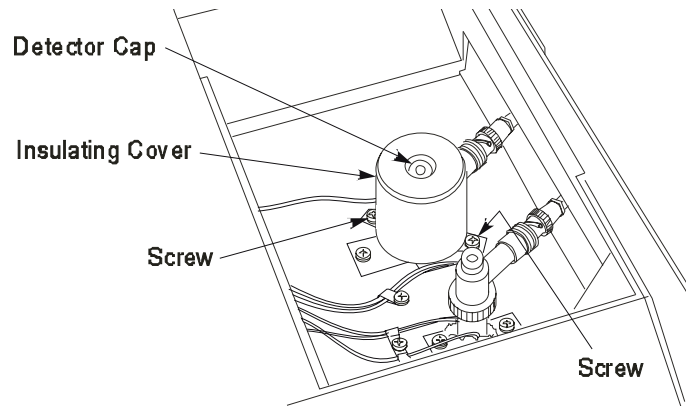
- Remove the two screws holding down the ECD insulating cover, then remove the insulating cover (see the following figure). (Removing the insulating cover exposes the knurled ring and detector outlet.)



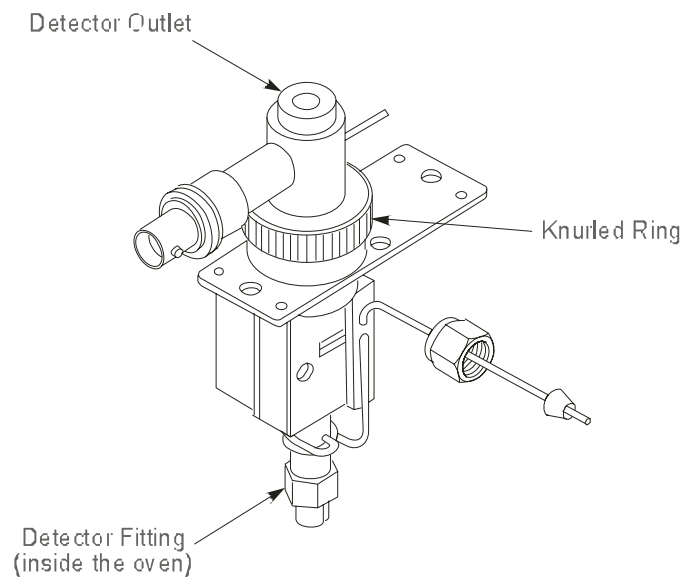
**WARNING**

***DO NOT DISMANTLE THE ECD CELL!***

## Maintenance



**Figure 9-26 ECD insulating cover**



**Figure 9-27 ECD surfaces to wipe**

4. Using the instructions included with the wipe-test kit (P/N 0009-1667) supplied with the detector, wipe the external surfaces of the items shown in the previous figure with the “low Activity Source” filter papers or stick swab:



- Detector outlet
- Knurled ring
- Detector fitting

**CAUTION**

*Do not remoisten the wipe-test paper once it has been moistened or any part of the ECD has been wiped. **Do not allow any of the wipe-test solution to enter the cell.***

5. Place the wipe-test paper in the container provided in your wipe-test kit. Include a data sheet stating that the wipe test was performed on a PerkinElmer electron capture detector cell (P/N N610-0063, 120 volts or P/N N610-0134, 230 volts) and the date of the test.
6. Request a new wipe-test kit to be sent with the test results.
7. Return the envelope to:

**National Leak Test Center  
P.O. Box 486  
North Tonawanda, New York 14120  
Tel: 716-693-0550**

**or**

**NRD  
P.O. Box 310  
Grand Island, New York 14072  
Tel: 800-525-8076  
716-773-7634**

**NOTE:** *The sensitivity of the wipe test is 0.0001  $\mu$ Ci.*

### ***Disposal of an ECD Cell***

If it is necessary to dispose of an ECD cell, contact:

**Nuclear Radiation Development Corp.**  
**2937 Alt. Blvd. North**  
**Grand Island, NY 14072**  
**Tel: (716) 773-7634**  
**Fax: (716) 773-7744**

for disposal instructions and current fees.

In addition, report the ECD cell disposal to:

**PerkinElmer, Inc.**  
**Radiation Safety Officer**  
**710 Bridgeport Ave.**  
**Shelton CT 06484**

and

**Nuclear Material Safety and Safeguard**  
**U.S. Nuclear Regulatory Commission**  
**Washington, DC 20251**

and/or

**your local agency, if applicable.**

### ***Refurbish/Refoil of an ECD Cell***

If it is necessary to refurbish/refoil an ECD cell, contact:

**Nuclear Radiation Development Corp.**  
**2937 Alt. Blvd. North**  
**Grand Island, NY 14072**  
**Tel: (716) 773-7634**  
**Fax: (716) 773-7744**

for instructions and current fees.

## ***FID Maintenance***

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FID maintenance consists of replacing the FID jet, cleaning the FID jet, replacing an o-ring in the collector, and cleaning the FID collector and cap.

### ***Replacing a FID Jet***

**NOTE:** *The FID jet rarely becomes plugged. However, if plugging occurs, it is usually sample dependent. It is recommended that you replace a plugged jet rather than clean it.*

To replace the FID jet:



**WARNING**

*Before you begin, extinguish the flame via the keyboard by setting the hydrogen flow to “0” or if you have manual pneumatics, turn the outer knob on the hydrogen needle valve completely clockwise to off.*

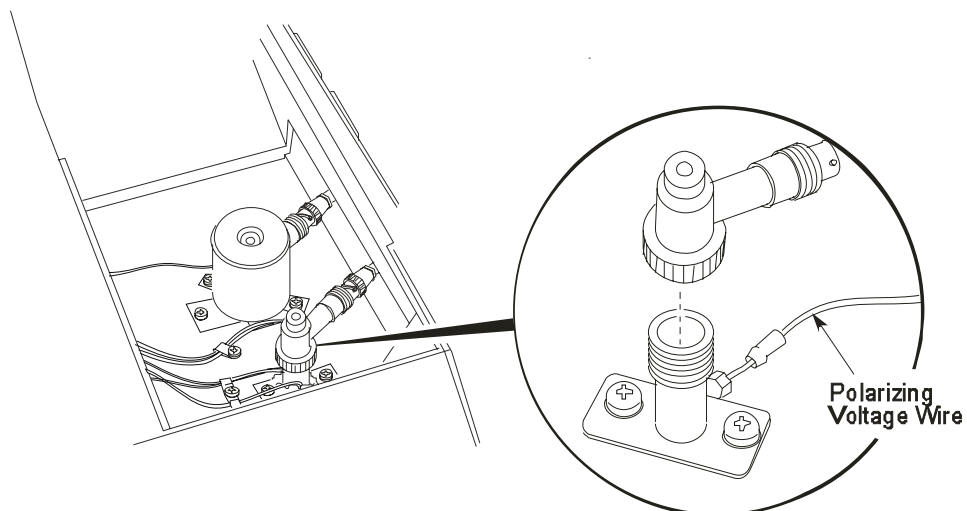
1. Turn off the Clarus GC power.



**WARNING**

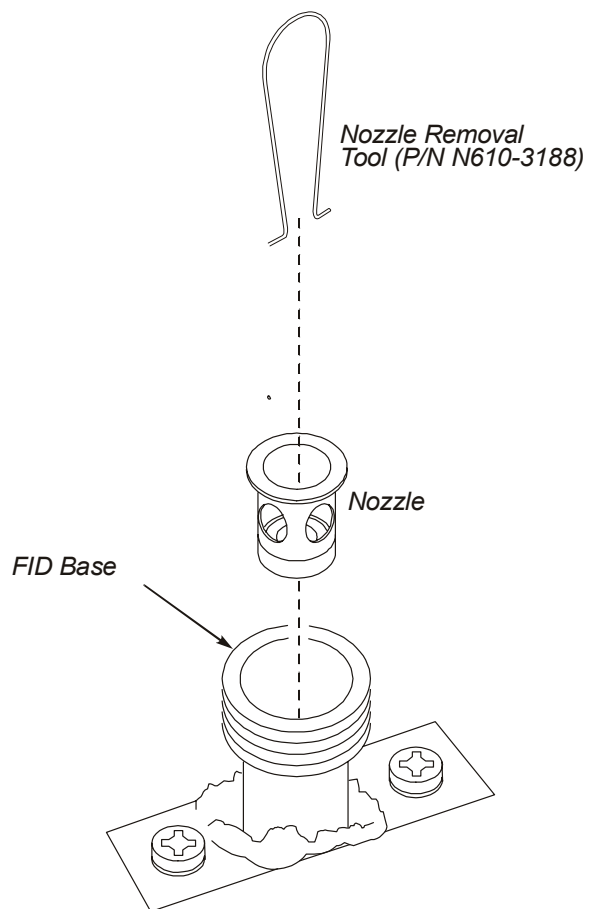
*The FID is hot and can cause serious burns! To prevent injury, allow the detector to become cool to the touch.*

2. Open the detector cover (see Figure 9-25).
3. Remove the polarizing cable from the pin on the polarizing filter assembly.



**Figure 9-28 FID polarizing voltage wire**

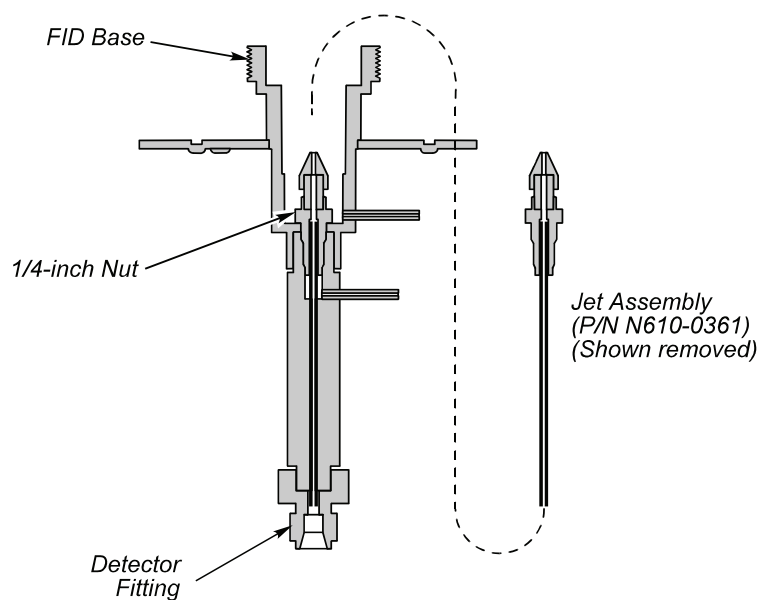
4. Loosen the knurled ring, then lift the FID collector off of the FID base and put it out of the way.
5. Insert the nozzle removal tool (P/N N610-3188) into the FID base and lift the nozzle out of the FID base. Do not unscrew it.



**Figure 9-29 Removing the nozzle assembly from the FID base**

6. Insert a 1/4-inch nutdriver into the FID base to engage the 1/4-inch nut on the FID jet assembly.
7. Loosen the FID jet assembly (turn the 1/4-inch nut counterclockwise) and pull it out of the FID. You should be able to pull out the FID jet assembly with the nutdriver. If not, then pull out the FID jet assembly with a pair of forceps or needle nose pliers.

## Maintenance



**Figure 9-30 Cross section view of the FID**

8. Insert a new FID jet assembly (P/N N610-0361) and secure it in place with the 1/4-inch nut driver.
9. Insert the nozzle assembly into the FID base until you feel it bottom.
10. Insert the FID collector back on the FID base and tighten the knurled ring.
11. Reconnect the polarizing wire to the polarizing pin on the FID collector.
12. Turn on the FID heater and allow it to return to the temperature setting.
13. Re-ignite the flame.

## **Cleaning a FID Jet**

Although it is not recommended, you may try to clean the FID jet as a last resort. Use one or both of the following techniques:

- Based on your analytical application, wash the jet with an appropriate solvent.
- Dislodge the plug with a fine wire such as a syringe needle, then blow out loosened debris using compressed air.

## **Replacing the O-Ring in the FID Collector**

Since the O-ring in the FID collector is in contact with the heated surface of the FID base, you will notice over time that it has become brittle or broken and must be replaced.



***The FID is hot and can cause serious burns! To prevent injury, extinguish the FID flame, turn off the FID heater, and allow the detector to become cool to the touch.***

To replace the O-ring in the FID collector:

1. Remove the polarizing voltage wire from the polarizing pin (Figure 28).
2. Loosen the knurled ring, then lift the FID collector off of the FID base.
3. Remove the old O-ring (see the following figure) from the FID collector and insert a new O-ring (P/N 0990-2143).
4. Insert the FID collector back on the FID base and tighten the knurled ring.
5. Connect the polarizing wire to the polarizing pin on the FID collector.

## Maintenance

6. Turn on the FID heater and allow it to return to the temperature setting.
7. Re-ignite the flame.

### **Cleaning the FID Collector and Cap**

Occasionally clean the collector and cap if you are running samples that may generate soot, for example, carbon disulfide.



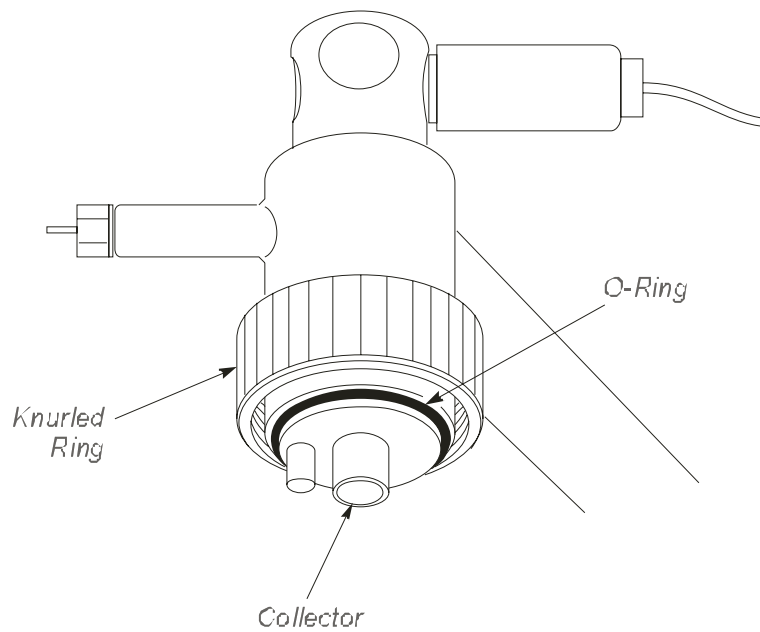
**WARNING**

***The FID is hot and can cause serious burns! To prevent injury, extinguish the FID flame, turn off the FID heater, and allow the detector to become cool to the touch.***

To clean the FID collector:

1. If necessary, disconnect the amplifier coaxial cable, and other wires from the FID collector.
2. Loosen the knurled ring on the collector and remove the collector from the FID base.
3. Using a pipe cleaner, wipe the inside of the collector and then the outside of the collector near the top.
4. Wash the collector with a laboratory soap such as Alconox. Try to keep the side-arm dry.
5. Air dry the collector replace it on the FID base, and tighten the knurled ring to secure the collector in place.
6. If you disconnected the amplifier coaxial cable and any wires from the FID collector, reconnect them to the FID collector.





**Figure 9-31 View of the FID collector and O-ring**

## ***PID Maintenance***

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Routine PID maintenance consists of the following:

- Changing PID lamps
- Cleaning PID lamp windows
- Changing PID lamp window seals and positioning disks

Procedures for performing these tasks follow.



*The PID operates at a high temperature and voltage. To avoid injury, before attempting maintenance procedures, disconnect the PID from line power and allow it to cool to room temperature.*

**CAUTION**

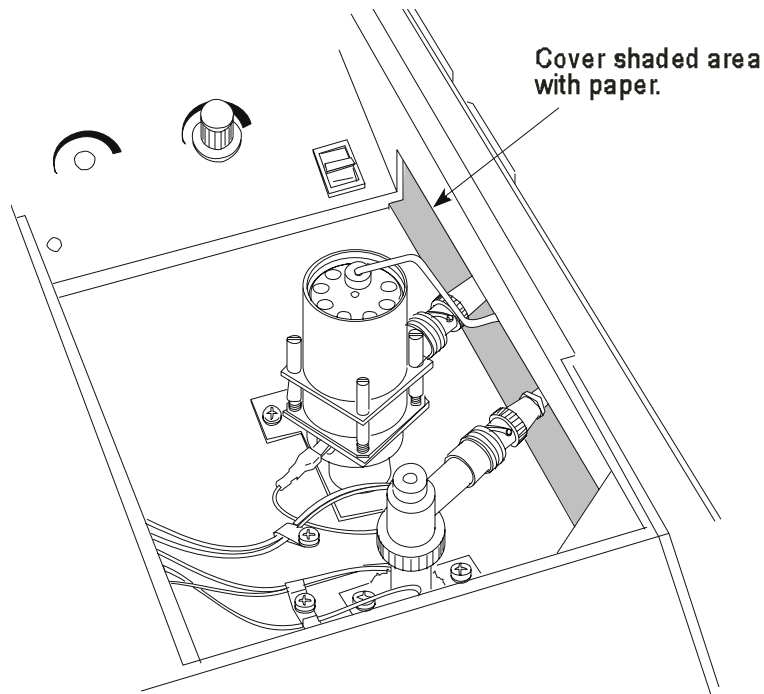
*To avoid contamination, wear rubber gloves or use tweezers when disassembling the PID.*

### ***Changing a PID Lamp***

To change a PID lamp:

1. Disconnect the instrument from line power.
2. Allow the system to cool to room temperature.
3. Place a piece of paper towel to the right of the detector area (see shaded area in the following figure) to cover the space between the oven skin

and the electronics compartment of the Clarus GC. This will prevent the lamp housing fasteners (see next step) from falling into the instrument when they are loosened.



**Figure 9-32 Placing a towel in the space between detector and electronics compartment**

4. Please examine the previous figure. While pressing down on the top of the PID (lamp housing), remove the four fasteners securing the lamp housing to the detector and place them in a safe place.
5. Slowly ease up on the lamp housing. As you do, the spring inside the housing (see the following figure) will push the housing up. Pull off the lamp housing and reveal the spring. The spring is quite powerful and it may shoot off if the lamp housing is removed too quickly.
6. Remove the spring.

## *Maintenance*

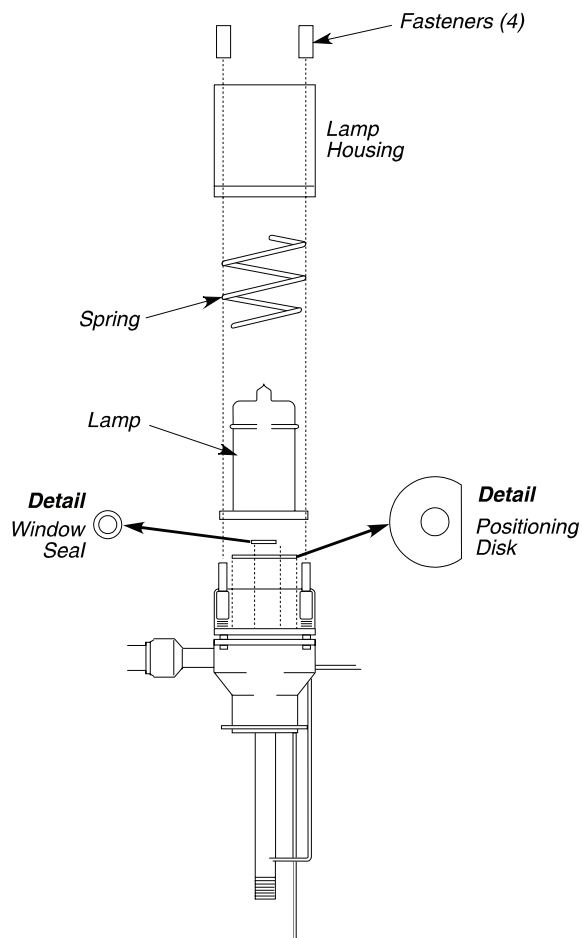
7. Replace the worn out or damaged lamp, window seal, and positioning disk.
8. Replace the spring and housing.
9. While holding the lamp housing down tightly, replace the four fasteners previously removed.
10. Remove the paper inserted in step 3.

## ***Cleaning PID Lamp Windows***

**CAUTION**     *Wear rubber gloves to avoid contaminating the lamp window.*

To clean PID lamp windows:

1. Remove the lamp using steps 1 through 7 in the previous procedure.



**Figure 9-33 Exploded view of the PID**

2. First clean the window with a moist, clean Kimwipe or other lint-free tissue. Use a gentle, circular motion.
3. Complete the cleaning by using a tissue moistened with cleaning compound supplied (P/N 0330-2775).
4. Rinse the window using a warm (30 °C) solution of a mild dishwashing detergent in water.

## Maintenance

5. Rinse with warm (30 °C) distilled water.
6. Dry with air or a lint-free tissue.
7. Ensure that a new lamp window seal and a new positioning disk are in place (see Figure 9-33). Then position the lamp over the window seal, place the spring over the lamp, and place the housing over the lamp and spring.
8. While holding the lamp housing down tightly, replace the four fasteners previously removed.

### **Changing PID Lamp Window Seals and Positioning Disks**

**CAUTION** *New seals and positioning disks must be baked (preferably in a vacuum oven) before use for one to two hours at 240 °C.*

To change PID lamp window seals and positioning disks, refer to the previous figure and follow this procedure:

1. Wearing rubber gloves to avoid contaminating the lamp window, remove the lamp using the appropriate steps in the previous procedure.
2. The lamp window seal will either be in the center of the positioning disk or stuck to the lamp window. Using a pair of tweezers, remove the lamp window seal.
3. Lift out the positioning disk.
4. Replace the positioning disk with a new one, making certain that you do not crease or bend it.
5. Place a new lamp window seal in the center of the positioning disk. Viewing the detector from the end, make certain that the opening in the seal is lined up with the ion chamber opening. The whole window seal must rest on the gold-plated ceramic portion of the ionization chamber.

6. Position the lamp over the seal, place the spring over the lamp, and place the housing over the lamp and spring.
7. Secure the lamp housing with the four fasteners.

**Leak-Test**

You can check for leaks by turning on the make-up gas and checking the flow rates at the detector inlet and outlet to make certain they are equal.

## ***NPD Maintenance***

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NPD maintenance consists of changing and conditioning the NPD bead and replacing a NPD jet.

### ***Changing the NPD Bead***

The Nitrogen Phosphorus Detector utilizes a glass bead containing alkali metal (single bead P/N N612-0092 or package of five P/N N612-0093) to detect organically bound nitrogen and phosphorus compounds. In time, the bead will not respond, or the wire on which the bead is placed may break. The bead is considered a consumable part.

If you cannot achieve a response at your normal operating background (0.25 mV or greater with the detector range set to x1), increase the potentiometer setting (try higher settings). If you cannot achieve a response at higher settings, the bead must be replaced.

**NOTE:** *An indication of a broken bead wire is that the bead does not glow when you increase the bead potentiometer setting (by turning it clockwise).*

To change the NPD bead:

1. Lift open the detector cover.
2. Locate the bead potentiometer.

If the NPD is installed in the front detector position, the bead potentiometer is located on the left side of the detector panel. If the NPD is installed in the rear detector position, the bead potentiometer is located on the right side of the detector panel.

3. Turn the NPD bead off by turning the potentiometer counterclockwise.

**NOTE:** *If two NPDs are installed, turn both beads off, even if you are only replacing one bead.*

4. Turn off the Clarus GC.





**WARNING**

*The gases can remain on during this procedure, but the detector should be cool to the touch to protect you from getting burned.*

5. Remove the NPD collector assembly (see the following figure) by loosening its knurled ring and lifting the collector assembly upward.

**CAUTION**

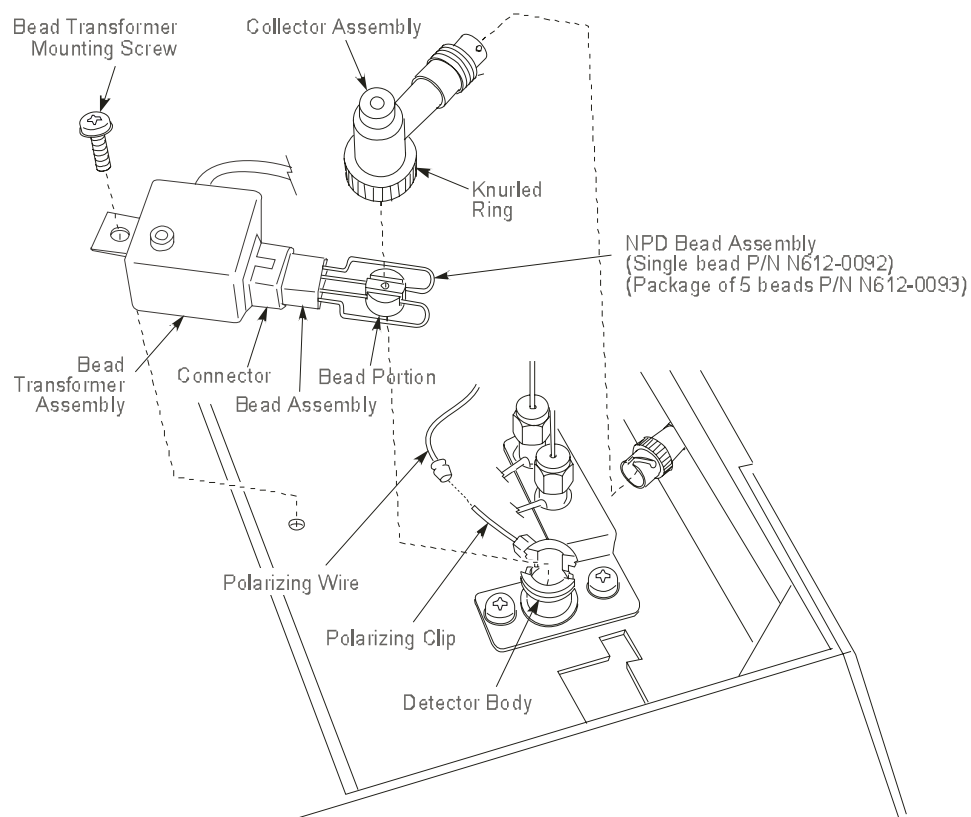
*Lift the collector assembly straight up so that it does not chip the ceramic header of the bead assembly. You may find it easier to remove the coaxial cable from the collector assembly before you remove the collector assembly from the detector body (see the following figure).*

6. Remove the screw that secures the bead transformer assembly to the top of the Clarus GC oven.
7. Carefully remove the bead portion from the detector body by lifting the bead transformer assembly straight up and out of the detector body.
8. Remove the bead assembly from the transformer assembly by unplugging it from the connector (see the following figure).
9. Plug a new bead assembly (P/N N612-0092) into the connector on the bead transformer. The connector is keyed so that the bead assembly can only be inserted one way.
10. Carefully insert the bead portion of the bead assembly in the detector body as shown in (see the following figure).
11. Secure the bead transformer to the top of the oven with the screw removed in step 6 of this procedure.
12. Replace the collector assembly on the detector body, and secure it by tightening the knurled ring. If the coaxial cable was removed, connect it to the collector assembly.

## Maintenance

**NOTE:** Check that the polarizing wire has not fallen off the detector. If it has, replace it (Figure 9-37).

### 13. Turn on the Clarus GC.




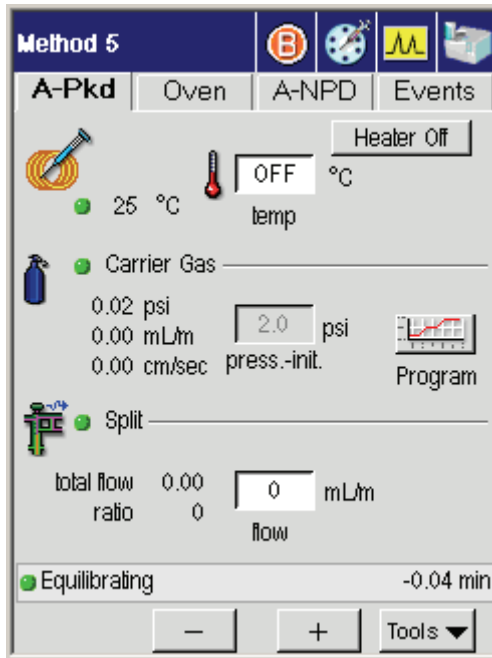
**Figure 9-37 Exploded view of the NPD bead assembly**

## Conditioning a New NPD Bead for Use with a Packed Column

**CAUTION**

*Never condition a NPD bead when a column is connected to the detector fitting. To properly condition the NPD bead, the column must be removed from the detector fitting and the detector fitting must be capped.*

1. Open the oven door and allow the oven to cool.
2. Loosen and remove the column nut from the detector fitting using a 7/16-inch wrench. Remove the column from the detector fitting.
3. Install a 1/8-inch Swagelok plug (P/N N930-0061) on the detector fitting. Provide a leak-free seal by tightening the plug with a 7/16-inch wrench.
4. Close the oven door and continue to flow carrier gas through the column.
5. Set the hydrogen flow, air flow, and all temperatures to operating conditions.
6. Set the detector range to x1.
7. Select the signal tab  to display the detector background and the **AutoZero** button. Press the **AutoZero** button to have the system on autozero.



When the bead is turned off, the background value should be  $0 \pm 0.25$  mV. Write down the background value with the bead off.

8. Open the detector cover and locate the bead potentiometer dial (see Figure 9-33).

If the NPD is detector 1 (front position), the potentiometer is located on the left side of the panel. If the NPD is detector 2 (rear position), the potentiometer is located on the right side of the panel.


9. Turn the bead potentiometer dial clockwise to apply current to the bead. Slowly increase the setting to 700 on the dial.

The millivolt reading on the autozero display will begin to increase. A maximum reading of 997.61 mV at a range setting of x1 is required to condition the bead.

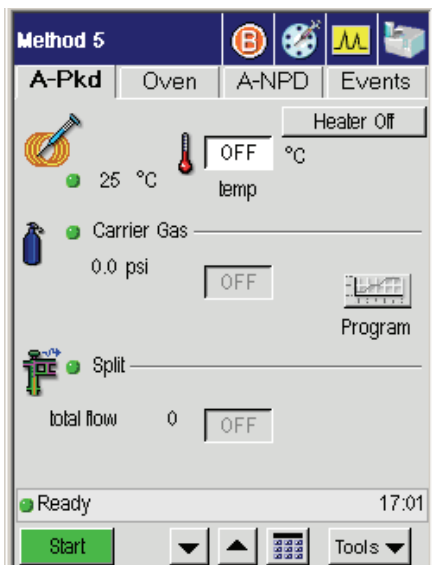
10. Monitor the display for about one minute at this setting. If the millivolt reading is less than 997.61, increase the bead potentiometer setting by an increment of 10 (to 710) and wait about one minute.

Repeat this process until the bead potentiometer setting produces a maximum signal of 997.61 mV.

11. Allow the bead to "condition" at this setting for one hour. The background readout will drift downward during the process.
12. After conditioning the bead, lower the bead potentiometer dial setting to 600. Remove the Swagelok plug from the detector fitting and reinstall the packed column.

13. Make sure the column flow is properly set and select the signal tab  to display the detector background produced by carrier gas flow and the **AutoZero** button. Press the **AutoZero** button to have the system on autozero.

The bead background will be less due to cooling effects.



## Maintenance

14. Turn the bead potentiometer dial clockwise until you achieve a reading of 0.25 mV above the "off" reading, with the detector range set to x1 as noted in step 7 of this procedure.
15. Once the system has stabilized, it is ready for operation.

If you are unable to achieve adequate sensitivity for your standard, increase the background of the NPD. Increasing intervals of 0.25 mV are recommended.

### NOTES:

- *The bead will be stable for several hours of operation but will drift in time. At the start of each day, adjust the background reading to the setting you are using and allow a few minutes for it to stabilize.*
- *Due to the loss of alkali metal with use, the nature of the bead is to drift over time. We strongly recommend using an internal standard for quantitative analysis.*
- *The bead can operate at higher background settings. The higher the setting the greater the signal and noise. Therefore, the signal to noise ratio will not increase dramatically. Operate at the lowest possible setting to achieve the required sensitivity. This will also prolong the life of the bead.*


### CAUTION

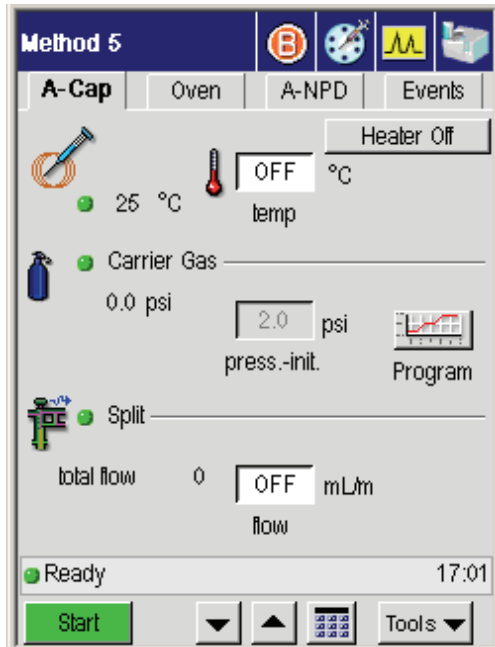
*Before you turn off the Clarus GC, turn off The bead by turning the bead potentiometer dial fully counterclockwise . Remember that the bead has a finite life. You can extend the bead life by turning the bead off when it is not in use for an extended period of time (for example, over the weekend).*

## Conditioning a New NPD Bead for Use with a Capillary Column (0.53 mm i.d. and smaller)

**CAUTION**

*Never condition a NPD bead when a column is connected to the detector fitting. To properly condition the NPD bead, the column must be removed from the detector fitting and the detector fitting must be capped.*

1. Open the oven door and allow the oven to cool.
2. Loosen and remove the column nut from the detector fitting using a 7/16-inch wrench. Remove the column from the detector fitting.
3. Install a 1/16-inch Swagelok plug (P/N N930-0061) on the detector fitting. Provide a leak-free seal by tightening the plug with a 7/16-inch wrench.
4. Close the oven door and continue to flow carrier gas through the column.
5. Set the hydrogen flow, air flow, and all temperatures to operating conditions.
6. Set the hydrogen flow, air flow, and all temperatures to operating conditions.
7. Set the detector range to x1.
8. Select the signal tab  to display the detector background and the **AutoZero** button. Press the **AutoZero** button to have the system on autozero.



When the bead is off, the background value should be  $0 \pm 0.25$  mV. Write down the background value with the bead off.

9. Open the detector cover and locate the bead potentiometer dial.

If the NPD is detector 1 (front position), the potentiometer is located on the left side of the panel. If the NPD is detector 2 (rear position), the potentiometer is located on the right side of the panel.


10. Turn the bead potentiometer dial clockwise to apply current to the bead. Slowly increase the setting to 700 on the dial.

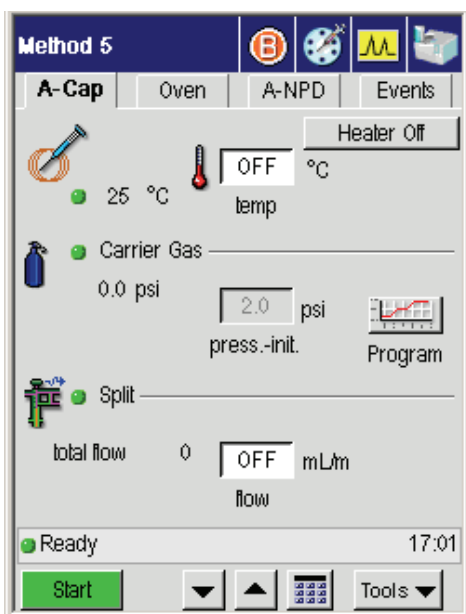
The millivolt reading on the autozero display will begin to increase. A maximum reading of 997.61 mV at a range setting of x1 is required to condition the bead.

11. Monitor the display for about one minute at this setting. If the millivolt reading is less than 997.61, increase the bead potentiometer setting by an increment of 10 (to 710), and wait about one minute.



Repeat this process until the bead potentiometer setting produces a maximum signal of 997.61 mV.

12. Allow the bead to "condition" at this setting for one hour. The background readout will drift downward during this process.
13. After conditioning the bead, readjust the column flow if necessary.
14. Select the signal tab  to display the detector background and the **AutoZero** button. Press the **AutoZero** button to have the system on autozero.



15. Turn the bead potentiometer dial clockwise or counterclockwise until you achieve a reading of 0.25 mV above the "off" reading with the detector range set to x1 (see step 4 of this procedure).
16. Once the system has stabilized, it is ready for operation.

## Maintenance

If you are unable to achieve adequate sensitivity for your standard, increase the background of the NPD. Increasing the background in intervals of 0.25 mV is recommended.

- NOTE:** 1. The bead will be stable for several hours of operation but will drift in time. At the start of each day, adjust the background reading to the setting you are using and allow a few minutes for it to stabilize.
2. Due to the loss of alkali metal with use, the nature of the bead is to drift over time. We strongly recommend using an internal standard for quantitative analysis.
3. The bead can operate at higher background settings. The higher the setting the greater the signal and noise. Therefore, the signal to noise ratio will not increase dramatically. Operate at the lowest possible setting to achieve the required sensitivity. This will also prolong the life of the bead.

### CAUTION

Before you turn off the Clarus GC, turn off the bead by turning the bead potentiometer dial fully counterclockwise. Remember that the bead has a finite life. You can extend the bead life by turning the bead off when it is not in use for long periods of time (for example, over the weekend).

## Replacing an NPD Jet

**NOTE:** The NPD jet rarely becomes plugged. However, if the jet does become plugged, it is usually because of the type of sample used. We recommend replacing a plugged NPD jet.

To replace an NPD jet:

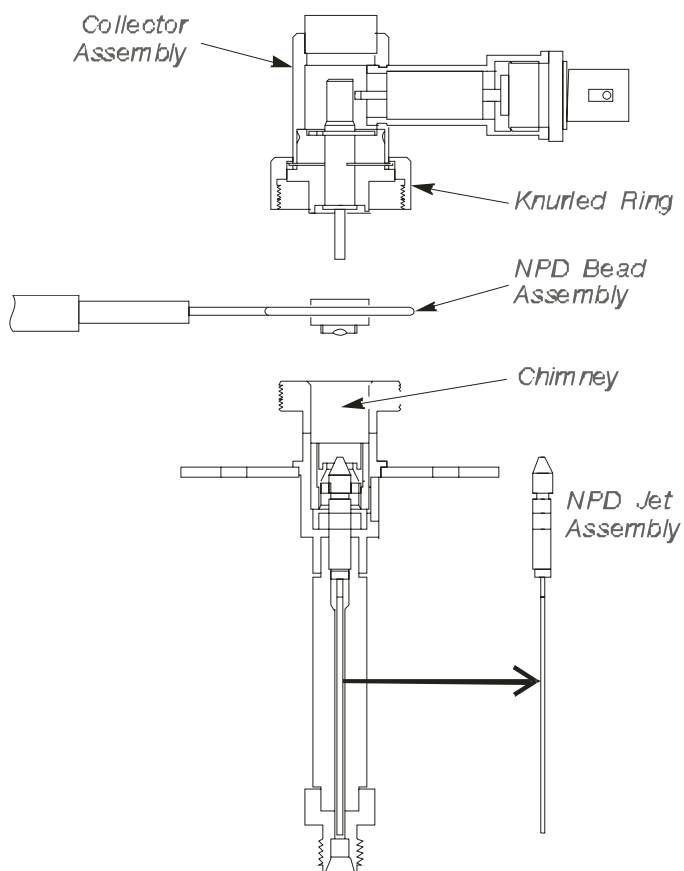
1. Turn off the bead by turning the potentiometer dial fully counterclockwise.
2. Turn off the Clarus GC.
3. Turn off the hydrogen and air flows.
4. Open the detector cover.



**WARNING**

*Wait until the detector is cool to the touch to protect you from getting burned.*

5. Loosen the knurled ring on the collector assembly, then remove the collector assembly (see the following figure).



**Figure 9-38 Location of the NPD jet assembly**

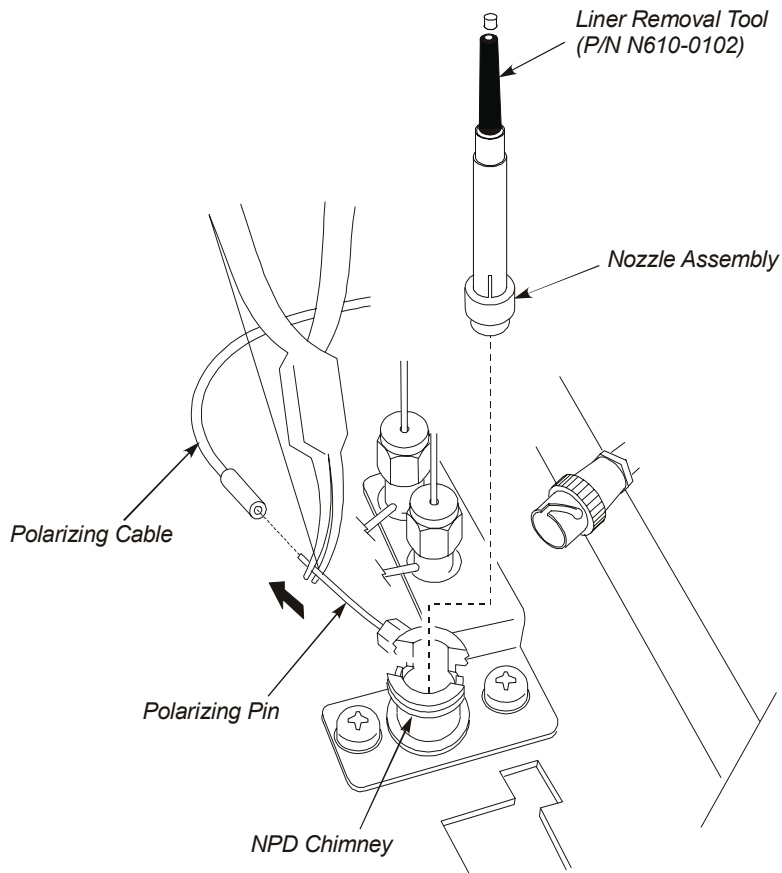
**CAUTION**

*Lift the collector straight up so that it does not chip the ceramic header of the bead assembly. You may find it easier to remove the coaxial cable from the collector before you remove the collector (see the previous figure).*

6. Remove the screw that secures the bead transformer assembly to the top of the Clarus GC oven.
7. Remove the bead assembly from the detector body by lifting the entire bead transformer assembly straight up and out of the detector body (see Figure 9-38).

**NOTE:** *Carefully place the bead transformer assembly out of the way so that the bead is not damaged. You may want to remove (unplug) the bead assembly from the transformer in order to protect the bead.*

8. Remove the polarizing wire. This exposes a spring-loaded polarizing pin, which is a piece of wire about 3/8-inch long (see the following figure).



**Figure 9-39 Removing the NPD nozzle assembly and polarizing wire**

9. Grasp and pull the spring-loaded polarizing pin using a pair of needle nose pliers with your left hand. Maintain a steady pull on the spring-loaded pin.
10. With your right hand, insert the large end of the liner removal tool (P/N N610-0102) into the NPD chimney so that it engages the nozzle assembly. Then remove the nozzle assembly by lifting it out.

## *Maintenance*

11. Still maintaining a steady pull on the spring-loaded polarizing pin, use your right hand to hold a pair of curved pliers or forceps and remove the ceramic insulator (not shown) from the detector body.
12. Still maintaining a steady pull on the spring-loaded polarizing pin, use your right hand to insert a 1/4-inch nutdriver (P/N N610-1297 provided in shipping kit) into the NPD chimney (see Figure 9-39) and engage the nut on top of the NPD jet assembly.
13. Loosen the NPD jet assembly by turning the nut counterclockwise. Then pull the jet assembly out of the NPD with the nutdriver.

If you cannot pull the jet assembly out with the nutdriver, use a pair of forceps or pliers.

14. Install a new jet assembly (P/N N610-0038) by reversing steps 5 through 13.

## ***FPD Maintenance***

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The most common causes of FPD performance deterioration which results in a decrease of the signal-to-noise ratio are as follows:

- Dirty optical filter
- Dirty detector liner
- Dirty window
- Defective photomultiplier tube
- Dirty jet

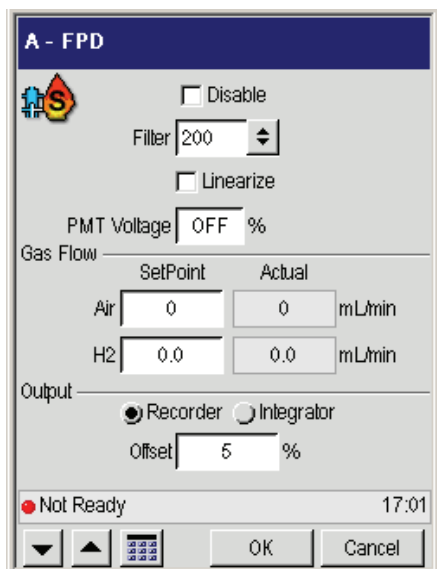
### ***Cleaning/Replacing an Optical Filter Assembly***

Obtain one of the following filters before you begin:

- Sulfur Filter Assembly, P/N N600-0637 (standard shipping filter) (Purple)
- Phosphorus Filter Assembly, P/N N600-0981 (Yellow)
- Tin Filter Assembly, P/N L413-5472 (Orange)

To clean or replace an optical filter assembly:

1. Turn off the photomultiplier voltage by selecting the following screen. Locate **PMT Voltage** on the screen and select **OFF**.



2. Extinguish the flame by turning off the hydrogen.

**CAUTION** *The photomultiplier tube can be damaged if exposed to light while voltage is ON. Never remove the detector cap or Photomultiplier tube WITH the voltage on.*

3. Turn off the Clarus GC and allow the detector to cool.
4. Loosen the knurled nut that secures the photomultiplier tube assembly to the detector body.
5. Slide the photomultiplier tube assembly away from the detector. If the FPD is detector 1 (front position), slide the photomultiplier tube assembly to the left. If the FPD is detector 2 (rear position), slide the photomultiplier tube assembly back.
6. Remove the filter assembly by pulling it out of the photomultiplier tube assembly. Clean the filter assembly with lens paper.

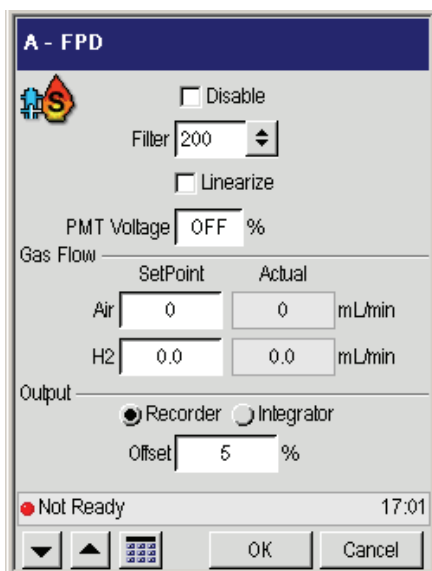


7. Insert the cleaned filter or a replacement filter with the colored side facing the photomultiplier tube assembly. The sulfur filter is purple, the phosphorus filter is yellow, the tin filter is orange.
8. Insert the filter end of the photomultiplier tube assembly into the detector body and secure it with the knurled nut.

### ***Cleaning/Replacing the Detector Liner***

To clean or replace the detector liner:

1. Turn off the photomultiplier voltage by selecting the following screen. Locate **PMT Voltage** on the screen and select **OFF**.

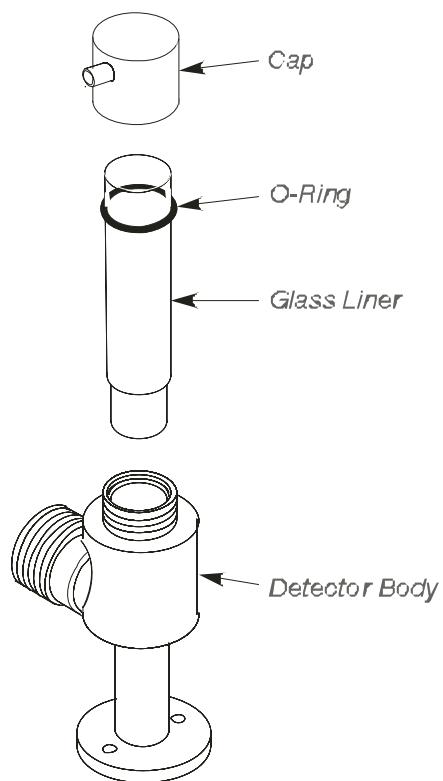


2. Extinguish the flame by turning off the hydrogen needle valve. (Turn the outer knob fully clockwise.)

**CAUTION**

*The photomultiplier tube can be damaged if exposed to light while voltage is ON. Never remove the detector cap or Photomultiplier tube WITH the voltage on.*

3. Turn off the Clarus GC and allow the detector to cool.
4. Remove the cap (see the following figure).
5. Lift out the glass liner and O-ring. Clean the liner by washing it in a soap solution and/or solvent.



**Figure 9-40 Removing a glass liner from the FPD**

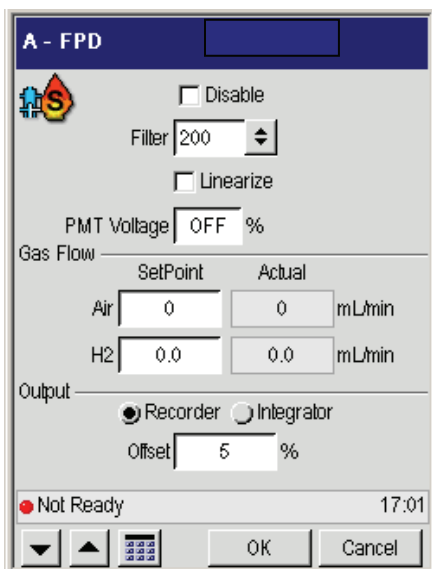
6. Insert the glass liner and O-ring into the detector body, then replace the cap.

**NOTE:** Replace the glass liner (P/N N600-3057) if it is chipped or if it cannot be cleaned. Replace a worn O-ring (P/N 0990-2247).

## **Cleaning/Replacing the Detector Window**

To clean or replace the detector window:

1. Turn off the photomultiplier voltage by selecting the following screen. Locate **PMT Voltage** on the screen and select **OFF**.



2. Extinguish the flame by turning off the hydrogen.

### **CAUTION**

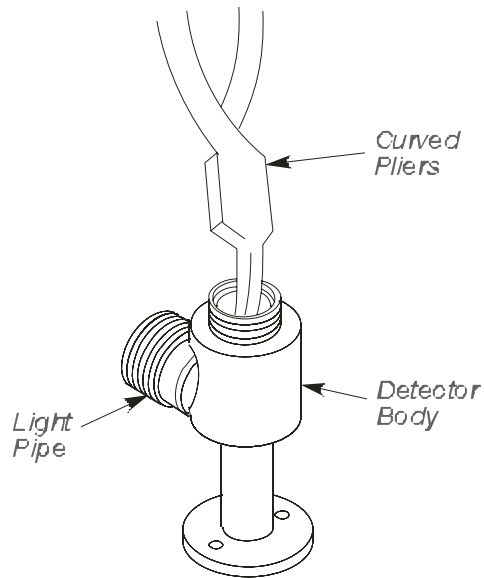
*The photomultiplier tube can be damaged if exposed to light while voltage is ON. Never remove the detector cap or Photomultiplier tube WITH the voltage on.*

## *Maintenance*

3. Turn off the Clarus GC and allow the detector to cool.

### ***Removing the Window***

1. Loosen the knurled nut that secures the photomultiplier tube assembly to the detector body.
2. Slide the photomultiplier tube assembly away from the detector body. If the FPD is detector 1 (front position), slide it to the left. If the FPD is detector 2 (rear position), slide it back.
3. Remove the cap (see the following figure).
4. Lift out the glass liner and O-ring from the detector body.
5. Remove the two hex head screws from the base of the FPD by using a 3/32-inch hex wrench. Then lift the detector body off the base.
6. Remove the spacer from the light pipe on the detector body.
7. Push the detector window out of the light pipe on the detector body (see the following figure). You can do this by inserting the end of a small pair of curved pliers into the top opening on the detector body. Then push the window out of the light pipe.
8. Remove the seal from the window.

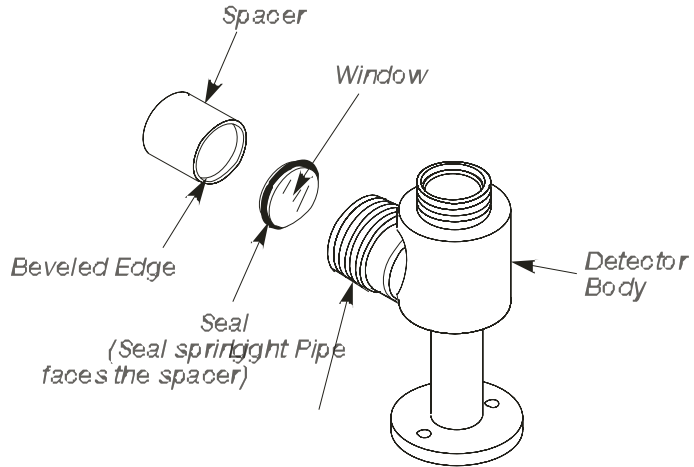


**Figure 9-41 Removing a FPD detector window**

***Cleaning and Replacing the Window***

1. Clean the window with soap and water. If necessary, replace the window and seal (P/N N930-0096).
2. Install the seal on the clean window.
3. Place the detector window on the window holder so that the spring portion of the seal faces the beveled edge in the window holder (see the following figure).
4. Use the spacer to insert the window into the light pipe on the detector body. Push the window and holder completely into the light pipe.

## Maintenance



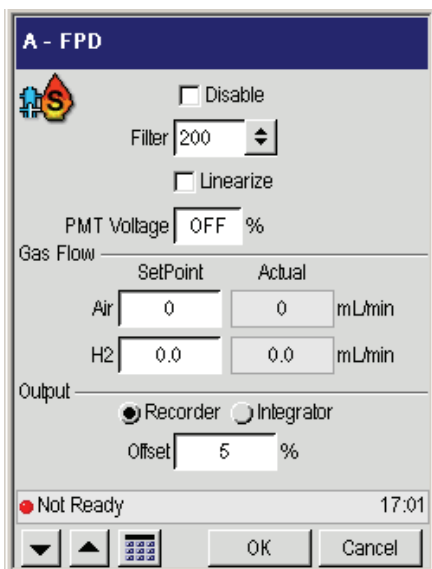
**Figure 9-42. Inserting a seal and window assembly into the FPD body**

5. Insert the detector body on the detector base. Secure the detector body with the hex screws.
6. Replace the detector liner, O-ring, and detector cap on the detector body.
7. Connect the photomultiplier tube assembly to the light pipe on the detector body.
8. Turn on the detector heater. Then turn on the photomultiplier tube and light the flame.

## ***Replacing the Photomultiplier Tube***

To replace the photomultiplier tube:

1. Turn off the photomultiplier voltage by selecting the following screen. Locate **PMT Voltage** on the screen and select **OFF**.

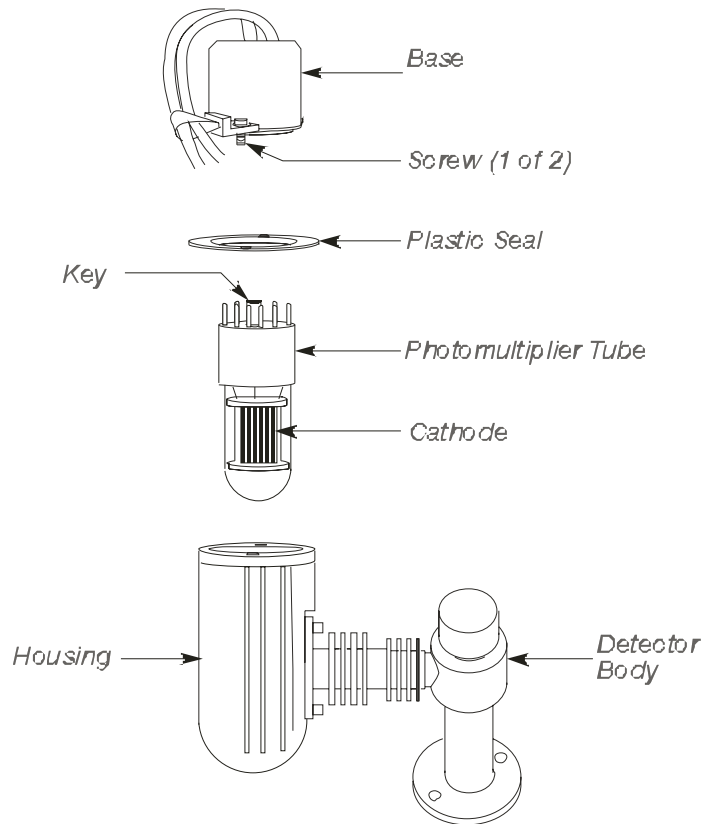


2. Extinguish the flame by turning off the hydrogen.

**CAUTION** *The photomultiplier tube can be damaged if exposed to light while voltage is ON. Never remove the detector cap or Photomultiplier tube WITH the voltage on.*

3. Turn off the Clarus GC and allow the detector to cool.
4. Remove the two screws from the base of the photomultiplier assembly using a 7/64-inch hex wrench (see the following figure).

## Maintenance



**Figure 9-43 Exploded view of a FPD photomultiplier tube**

5. Pull the photomultiplier tube and base out of the housing.
6. Carefully unplug the photomultiplier tube from the base. Be careful not to dislodge the O-ring and plastic seal.
7. Align the key in the new photomultiplier tube with the key hole in the socket.
8. Plug the new photomultiplier tube (P/N 0997-2321) into the socket in the base.



**CAUTION**

*Do not force the key into an incorrect position. Do not touch the glass. Do not expose the photomultiplier tube to any bright, direct light*

9. Position the photomultiplier tube in the housing so that the cathode (the horizontal filament grating; not visible in Figure 9-43) faces the detector body. Align the holes in the plastic seal with the holes in the base.

**CAUTION**

*Make certain that the photomultiplier tube cathode is facing the detector body*

10. Secure the base to the photomultiplier housing with the two screws removed in step 4 of this procedure.
11. Turn on the Clarus GC. Then turn on the hydrogen flow and the photomultiplier tube, and light the flame.

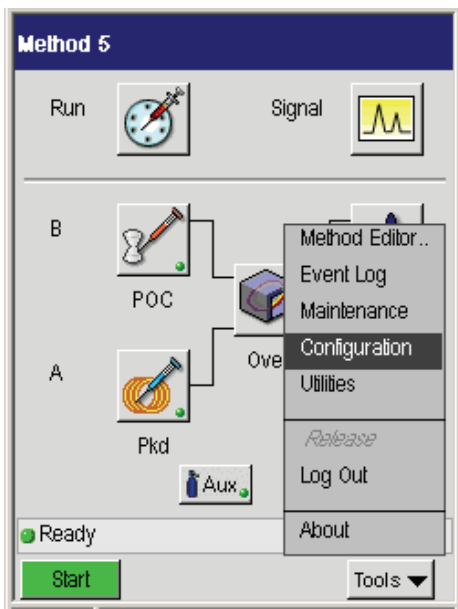
**NOTE:** *A new photomultiplier tube may require several hours to stabilize.*

**CAUTION**

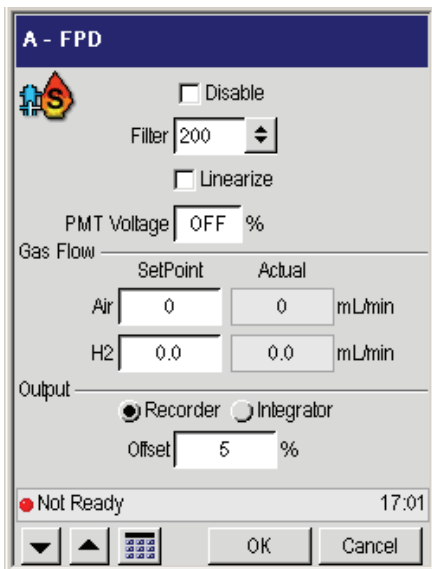
*When a replacement tube is installed, the noise should be about 2% at attenuation 8 with the linearizer off. If it is not, modify the voltage setting until this level of noise is achieved. Adjust the voltage as follows:*

- a. In the System Status screen select Tools for the configuration menu.

## Maintenance



- b. In the Configuration screen select the FPD photomultiplier to view the PMT voltage.



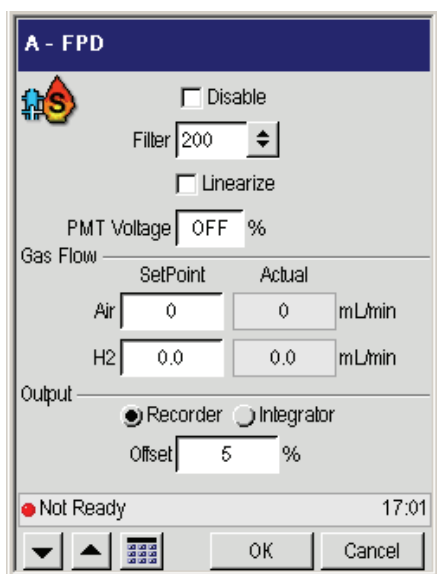
- c. Using the touch pad increase the percent (%) value or to increase the voltage in 0.1% increments or decrease the voltage in 0.1% increments, by using the up or down arrows.

**NOTE:** The millivolt setting is displayed in the upper-right corner. This number will be close to zero and will only change slightly as the photomultiplier tube voltage is modified.

## Cleaning/Replacing the FPD Jet

To clean or replace the FPD jet:

1. Turn off the photomultiplier voltage by selecting the following screen. Locate **PMT Voltage** on the screen and select **OFF**.

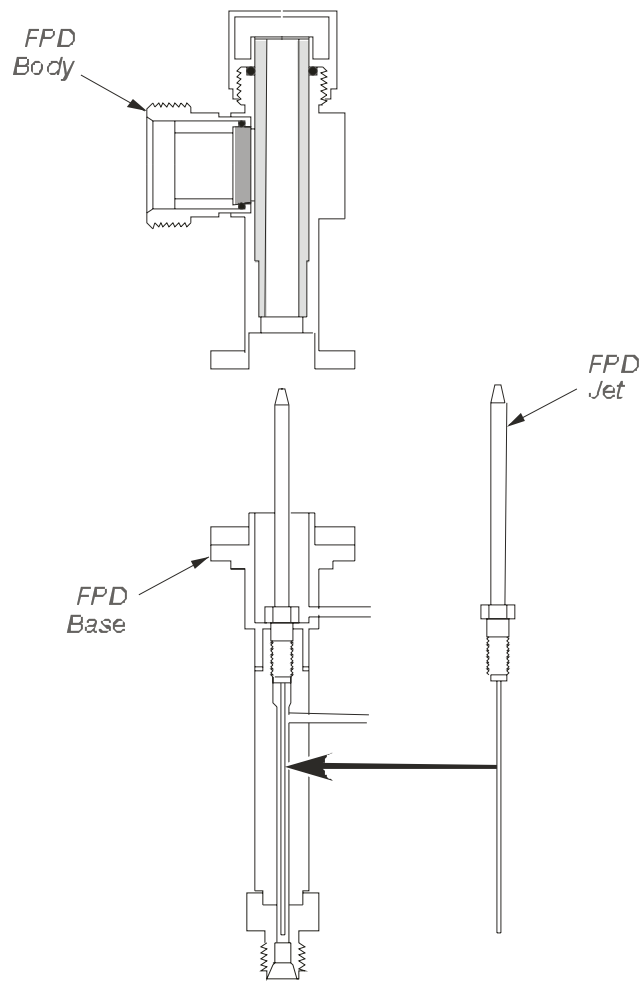


2. Extinguish the flame by turning off the hydrogen.

**CAUTION**

*The photomultiplier tube can be damaged if exposed to light while voltage is ON. Never remove the detector cap or Photomultiplier tube WITH the voltage on.*

3. Turn off the Clarus GC and allow the detector to cool.
4. Remove the two screws from the base of the FPD by using a 3/32-inch hex wrench. Lift the detector body and the attached photomultiplier tube assembly off the detector base. This exposes the FPD jet (see the following figure).
5. Insert a 1/4-inch nutdriver (P/N N610-1297) over the FPD jet.
6. Turn the nutdriver counterclockwise until the jet assembly is completely loosened. Then remove the jet assembly from the detector base.
7. Clean the jet in a suitable solvent (such as methanol or acetone) and scrape off any deposits with a cotton swab on a piece of soft wood. **DO NOT INSERT WIRE OR HARD TOOLS INTO THE JET.** Replacement jets are available from PerkinElmer by ordering P/N N610-0245.
8. Insert the jet in the detector base and secure it with the 1/4-inch nutdriver.
9. Replace the detector body and the attached photomultiplier tube assembly.
10. Turn on the Clarus GC. Then turn on the hydrogen flow and the photomultiplier voltage, and light the flame.



**Figure 9-44** Cutaway view of a FPD showing the jet assembly

## PPC Maintenance

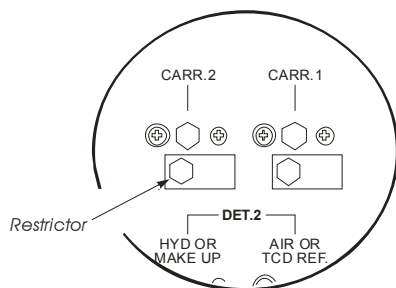
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PPC maintenance consists of replacing a plugged or restricted PPC module restrictor.

### Replacing a Restrictor

To replace a restrictor:

1. Locate the PPC module on the rear of the Clarus GC.



**Figure 9-45 Location of a PPC module restrictor**

2. Turn off all gases to the module on which you are replacing the restrictor.
3. Using a 1/2-inch socket or open-end wrench, loosen and remove the restrictor and install a new restrictor.
4. Turn on the gas and check for leaks.
5. Calibrate the PPC module.

### Zeroing the Pressure

The Clarus GC PPC modules are zeroed at the factory, prior to shipping, but it should be periodically checked to identify any change. This is particularly important if there have been large changes in the ambient temperature from

22 °C. For information about zeroing the pressure, refer to the “Clarus Users Guide.”

## ***PPC Restrictor Information***

### ***Available Restrictors***

<b>Restrictor Number</b>	<b>Color</b>	<b>Helium Flow at 30 PSIG, vent to atmosphere</b>
1	Silver	1 mL/min
2	Red	3 mL/min
3	Yellow	10 mL/min
4	Black	30 mL/min
5	Green	100 mL/min
6	Blue	300 mL/min

**Restrictors for Carrier Gas Control**

<b>Injector</b>	<b>Restrictor Number</b>	<b>Helium Flow (mL/min) (with a 90 psig inlet pressure and a 10 psig drop across the restrictor)</b>
Packed	4 (std)	30
Split/Splitless or PSS	6 (std)	300
Split/Splitless or PSS	4 (acc)	30
POC	3 (std)	10
POC	2 (acc)	3



**Restrictors for Detector Gas Control****NOTE:** *Maintain an inlet pressure between 60 and 90 psig.*

<b>Detector</b>	<b>Gas</b>	<b>Restrictor Number</b>	<b>Nominal Flow Required (mL/min)</b>
FID	Air	6	450
FID	Hydrogen	4	45
NPD	Air	5	100
NPD	Hydrogen	1	2
FPD	Air	5	90
FPD	Hydrogen	4	75
ECD	Argon/Methane	4	30
ECD	Nitrogen	4	30
TCD	Helium	4	30
TCD	Hydrogen	3	30

## **Practical Hints**

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### **Reversing TCD Polarity**

The following examples indicate when you may want to change the TCD polarity.

- When one of the components being analyzed has a higher thermal conductivity than the carrier gas.
- For example, if hydrogen is a sample component and helium the carrier gas, set up a timed event to reverse the polarity of the TCD prior to elution of the hydrogen (to generate a positive peak for the hydrogen). Then change the polarity back for the remaining components.
- If negative peaks are produced when two packed injectors are installed, each has a different column attached, and you are running two different analyses. Change the polarity to produce positive peaks.
- When two packed injectors are installed with two identical columns and the TCD is being operated at maximum sensitivity. In this case, alternate the column into which the sample is being injected in order to expose both sets of filaments (reference and sample) to sample, thus keeping the filaments more electrically balanced.
- To change the TCD polarity, enter a negative detector range. For example, TCD ranges of +2 and -2 have opposite polarities.

### **Optimizing FID Performance**

FID sensitivity is affected primarily by the hydrogen flow. The optimum hydrogen flow varies slightly if the column flow changes dramatically. For example, if you go from a packed column with a flow rate of 30 mL/min or higher to a capillary column with flows of 2 mL/min or less, the optimum hydrogen flow will be a different value.

The hydrogen flows recommended in this manual assume packed column flow rates. If you switch from a packed to a capillary column, re-optimizing the hydrogen flow will help to improve the FID sensitivity.

The following is the suggested FID optimization procedure after you have switched from a packed to a capillary column.

1. Prepare a one component standard.
2. Set up the carrier gas flow.
3. Set up the hydrogen and air flows, then ignite the flame.
4. Make 2 to 3 injections at varying hydrogen flows.

The optimum hydrogen flow is that which produces the maximum area counts.

### ***Filtering Detector Output***

You can select one of three software filters from the Filter Menu.

If your column delivers a peak width at half height of  $\geq 1$  s, select 200. If your column delivers a peak width at half height of  $< 1$  s, select 50.

A value of 800 should be used only with caution to minimize the signal-to-noise ratio. Try 800 with your application. If the peak height and area are not affected but the noise is decreased, then 800 will improve the signal-to-noise ratio.

**NOTE:** *The ECD has values of 200 and 800 only.*

### ***Autozero Display Sensitivity***

The maximum detector signals for various detectors that produce a 1-V reading on the Autozero Display are as follows:

FID, NPD, or PID (nA, Range 1) – 21.3

FID, NPD, or PID (nA, Range 20) – 426

TCD (mV) – 510

ECD (KHz) – 150

## Attenuation vs. Detector Output

The following table lists attenuation vs. the maximum detector signal producing 100% deflection on a 1-mV recorder.

Atten	NPD, PID and FID (fA) Range 1	NPD, PID and FID (fA) Range 20	TCD ( $\mu$ V)	ECD (Hz)
1	$3.3 \times 10^2$	$6.7 \times 10^3$	8.0	2.3
2	$6.7 \times 10^2$	$1.3 \times 10^4$	$1.6 \times 10$	4.7
4	$1.3 \times 10^3$	$2.7 \times 10^4$	$3.2 \times 10$	9.4
8	$2.7 \times 10^3$	$5.3 \times 10^4$	$6.4 \times 10$	19.0
16	$5.3 \times 10^3$	$1.1 \times 10^5$	$1.3 \times 10^2$	38.0
32	$1.1 \times 10^4$	$2.1 \times 10^5$	$2.6 \times 10^2$	75.0
64	$2.1 \times 10^4$	$4.3 \times 10^5$	$5.1 \times 10^2$	150.0
128	$4.3 \times 10^4$	$8.5 \times 10^5$	$1 \times 10^3$	$3.0 \times 10^2$
256	$8.5 \times 10^4$	$1.7 \times 10^6$	$2 \times 10^3$	$6.0 \times 10^2$
512	$1.7 \times 10^5$	$3.4 \times 10^6$	$4.1 \times 10^3$	$1.2 \times 10^3$
1024	$3.4 \times 10^5$	$6.8 \times 10^6$	$8.2 \times 10^3$	$2.4 \times 10^3$
2048	$6.8 \times 10^5$	$1.4 \times 10^7$	$1.6 \times 10^4$	$4.8 \times 10^3$
4096	$1.4 \times 10^6$	$2.7 \times 10^7$	$3.3 \times 10^4$	$9.6 \times 10^3$
8192	$2.7 \times 10^6$	$5.5 \times 10^7$	$6.6 \times 10^4$	$1.9 \times 10^4$
16384	$5.4 \times 10^6$	$1.1 \times 10^8$	$1.3 \times 10^5$	$3.8 \times 10^4$
32768	$1.1 \times 10^7$	$2.2 \times 10^8$	$2.6 \times 10^5$	$7.7 \times 10^4$
65536	$2.2 \times 10^7$	$4.4 \times 10^8$	$5.2 \times 10^5$	$1.5 \times 10^5$

## Maintenance

The following table lists attenuation vs. the maximum detector signal producing a 1-V output to an integrator.

Atten	NPD, PID and FID (pA) Range 1	NPD, PID and FID (pA) Range 20	TCD (mV)	ECD (kHz)
1	$3.3 \times 10^2$	$6.7 \times 10^3$	8.0	2.3
2	$6.7 \times 10^2$	$1.3 \times 10^4$	1.6x10	4.7
4	$1.3 \times 10^3$	$2.7 \times 10^4$	3.2x10	9.4
8	$2.7 \times 10^3$	$5.3 \times 10^4$	6.4x10	19.0
16	$5.3 \times 10^3$	$1.1 \times 10^5$	$1.3 \times 10^2$	38.0
32	$1.1 \times 10^4$	$2.1 \times 10^5$	$2.6 \times 10^2$	75.0
64	$2.1 \times 10^4$	$4.3 \times 10^5$	$5.1 \times 10^2$	150.0

### ***Prolonging the Life of a PID UV Lamp***

Lamp life decreases at elevated temperatures. (For example, when conditioning the detector above 250 °C.) To increase lamp life, substitute a disc (P/N N0330-2989) for the UV lamp during such procedures.

### ***Optimizing ECD Performance***

ECDs are extremely sensitive. Therefore, care should be taken to avoid contamination from any part of the system (for example, pneumatics, injector, column, gases, etc.). To help assure a clean system: condition the column, bake out the injector and detector, use clean tubing, and use pure filtered gases.

**CAUTION**

*To minimize detector contamination, run the ECD hot, at a temperature of at least 375 °C.*

## **Optimizing FPD Performance**

### **About Ionization Gas Flows**

FPD sensitivity is affected by both the hydrogen flow and the air flow. Hydrogen has a true optimum flow; that is, above or below a certain flow there is less response. Air, on the other hand, is more critical. That is, below a certain flow there is no response at all. The flows provided in the *Clarus Users Guide* are in the optimum range, but there may be slight variation from detector to detector.

### **Optimizing FPD Gas Flows**

To optimize gas flows:

1. Set the hydrogen flow to 65 mL/min.
2. Set the air flow to 90 mL/min.
3. Set the detector temperature to 250 °C, and wait for the detector to reach that temperature.
4. Light the flame.
5. Prepare a one-component standard (sulfur, phosphorus, or tin, depending on the filter installed).
6. Make two or three injections.
7. Increase the hydrogen flow by 5 mL/min and repeat the injections.
8. Continue increasing the hydrogen, and running the sample, until a maximum signal is obtained. Set the hydrogen at that flow.
9. Repeat the above procedure, varying the air flow by 5 mL/min (both below and above 90 mL/min).
10. Set the air flow to at least 10 mL/min above the flow that previously produced no response.

### **Quenching**

When hydrocarbons co-elute with the sulfur compound (or phosphorus or tin compound depending on which filter is being used), they will “quench” the flame, thereby causing a diminished response, or no response at all, to the peak of interest. To avoid this problem, complete separation and elution of all peaks is necessary. Because the FPD is a selective detector, hydrocarbons may not show as significant peaks. We recommend that you first profile your sample by using the same column with either an FID or TCD. All peaks will then be seen and you can determine the necessary conditions to completely separate and elute all compounds.

### **PSS and POC Operating Hints**

Both of the Programmed Split/Splitless (PSS) and Programmed On-Column (POC) injectors can be operated in either the oven programming mode or the inlet programming mode. For specific operation, refer to the *Clarus 600 Users Guide*.

#### **Oven Programming Mode**

This is the default mode for both the POC and the PSS injectors. This mode is the easiest to use, since only the oven temperature program needs to be entered into the method. In this mode, the injector will follow the oven temperature profile plus 5 °C. In this mode, the sample should be introduced into the injector when the temperature of the inlet is at the boiling point of the solvent. Then temperature program the injector and oven.

If the initial temperature of the oven is above the boiling point of the solvent you are using, then it would be better to modify the oven program to start at a lower temperature or to configure the injector for the Inlet mode and set a temperature for the injector separate from the oven temperature.

#### **Inlet Programming Mode**

This mode permits the use of independent injector temperatures and rates that you define in the method. The injector will be programmed for injector temperature 1, injector time 1, injector rate 1, injector temperature 2, injector



time 2, etc. You can program up to three temperatures and two ramps for each PSS or POC configured in the inlet mode.

It is important to set the initial injector temperature to about the boiling point of the solvent you are using.

**CAUTION**

*The PSS injector can be used in the "hot" split or splitless mode. However, this is not recommended due to liner size and could cause solvent flashback in the injector. This mode should be used with caution, depending upon the solvent and temperatures you choose.*

**CAUTION**

*When using the PSS injector in the on-column mode or the POC injector with the autosampler, you must use a special syringe that has a needle o.d. of 0.47 mm (P/N N610-1253 or N610-1380). Refer to the Clarus 600 User's Guide for more detail. You must use only the "Norm" injection speed with this syringe in the on-column mode. The "Fast" injection speed will bend this thin needle and the "Slow" injection speed may produce peak break-up or distorted peaks. You can achieve better precision in the on-column mode when sample volumes of 1.0  $\mu$ L or greater are injected.*

**CAUTION**

*If you have the subambient option, the POC and PSS injectors are linked to the oven subambient option; therefore, you cannot operate the injectors below the oven subambient temperature.*

If a column is used extensively at high temperatures (350 °C or greater), the polyimide may become very brittle. This brittleness will cause the column to fracture when you try to seal it in the universal adapter. If you wish to continue to use the brittle column, a low dead volume union may be a helpful alternative.

**PPC Restrictor Information****Available Restrictors**

<b>Restrictor Number</b>	<b>Color</b>	<b>Helium Flow at 30 PSIG, vent to atmosphere</b>
1	Silver	1 mL/min
2	Red	3 mL/min
3	Yellow	10 mL/min
4	Black	30 mL/min
5	Green	100 mL/min
6	Blue	300 mL/min

**Restrictors for Carrier Gas Control**

<b>Injector</b>	<b>Restrictor Number/Color</b>	<b>Helium Flow (mL/min) (with a 90 psig inlet pressure and a 10 psig drop across the restrictor)</b>
Packed	4 (std)/Black	30
Split/Splitless or PSS	6 (std)/Blue	300
Split/Splitless or PSS	4 (acc)/Black	30
POC	3 (std)/Yellow	10
POC	2 (acc)/Red	3

**Restrictors for Detector Gas Control**

**NOTE:** *Maintain an inlet pressure between 60 and 90 psig.*

<b>Detector</b>	<b>Gas</b>	<b>Restrictor Number/Color</b>	<b>Nominal Flow Required (mL/min)</b>
FID	Air	6/Blue	450
FID	Hydrogen	4/Black	45
NPD	Air	5/Green	100
NPD	Hydrogen	1/Silver	2
FPD	Air	5/Green	90
FPD	Hydrogen	4/Black	75
ECD	Argon/Methane	4/Black	30
ECD	Nitrogen	4/Black	30
TCD	Helium	4/Black	30
TCD	Hydrogen	3/Yellow	30

# Troubleshooting **10**

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*Troubleshooting*

This chapter contains the following sections:

- Instrument Error Messages
- Autosampler Error Messages
- Data Manager (DM) Error Messages
- GC Troubleshooting

## **Error Messages**

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The error messages are shown on the touch screen display. On a Warning message the user can touch OK and continue. On a Fatal Error there is no OK and the user must turn off the instrument and resolve the issue.

**Table 10-1 Instrument Error Messages**

### **Error Messages**

#### **Timed Events Error**

Events table is full.

32 events maximum.

#### **Timed Events Error**

Duplicate event time.

Enter a value at least 0.1 minute different.

#### **RS 232 Timeout Error.**

**Device failed to respond within specified time frame.**

#### **Oven Control Error**

No Coolant available. Check coolant supply.

#### **PPC Hardware Fault**

Power instrument OFF, wait 10 seconds and then power ON. If problem persists Call your PerkinElmer Service representative.

#### **Autosampler Error**

No vial found at location xx.

#### **Autosampler Error**

No autosampler programs active.

#### **Background Calibration Error**

Isothermal Method selected. Background calibration required temperature program method.

#### **Background Calibration Error**

Negative temperature ramp specified. Background calibration requires positive temperature ramps.

#### **Background Calibration Error**

Run Time is less than 1.0 minute.



**Background Calibration Error**

Run Time is greater than 999 minutes.

**Background Calibration Error**

HOLD Event is present in temperature program ramp. This is not permitted.

**No Inlet Configured**

Select Configuration on the Tools menu to access configuration settings.

**No Detector Configured**

Select Configuration on the Tools menu to access configuration settings.

**Flow Calibration Error**

Flow too high for 'low' calibration.

Check flow and repeat calibration.

**Flow Calibration Error**

Flow too low for 'hi' calibration.

Please check flow and repeat calibration.

**Autosampler Error**

Access door open.

Close door.

**Autosampler Error**

No post wash specified. Set post-inject field in configuration to a minimum of 1.

**Relay Fault**

Turn power OFF for 10 seconds and then ON. If problem persists Call your PerkinElmer Service representative.

**Temperature Error**

Unable to reach specified temperature.

Check coolant.

**PPC Error**

PPC Carrier xx not connected.

Check PPC Carrier module connections.

## *Troubleshooting*

### **PPC Error**

PPC Detector xx not connected.

Check PPC Detector module connections.

### **PPC Error**

The AUX flow PPC module being calibrated is not connected.

Connect the AUX PPC flow module and calibrate.

### **PPC Error**

Selected connection is currently assigned.

Select another connection.

### **PPC Error**

Warning: the PPC Alarm is OFF! If this is correct, press OK.

To enable the PPC Alarm, select PPC Alarm ON in configuration setup.

### **Ignite Error**

The detector xx failed to ignite.

Check the hydrogen and air flows.

### **Ignite Error**

The detector xx failed to ignite.

The hydrogen and air supplies are OFF. Turn the gases ON.

**Table 10-2 Oven Error Messages.**

<b>Oven Error Messages</b>
<p><b>INSTRUMENT SHUTDOWN! "CHECK OVEN FAN MOTOR"</b>                      Actual oven cooling fan speed is out of tolerance (+/- 20% of set speed) for 20 seconds. Fatal error, call your PerkinElmer Service Center.</p>
<p><b>INSTRUMENT SHUTDOWN! "CHECK STEPPER MOTOR"</b>                      One or both of the 2 stepper motors for the oven doors (inlet/exhaust) stalled. This is determined by the firmware monitoring a PRT. Fatal error, call your PerkinElmer Service Center.</p>
<p><b>INSTRUMENT SHUTDOWN! "CHECK OVEN FAN DRIVE"</b>                      Oven cooling fan motor reports a generic fault. Fatal error, call your PerkinElmer Service Center.</p>
<p>Temperature sensor fault OPEN. Cycle the power OFF for 10 seconds then ON. If the error persists call the PerkinElmer Service Center.</p>
<p>Temperature sensor fault SHORT. Cycle the power OFF for 10 seconds then ON. If the error persists call the PerkinElmer Service Center.</p>
<p>No Heat. Cycle the power OFF for 10 seconds then ON. If the error persists call the PerkinElmer Service Center.</p>
<p>Temperature exceeds setpoint! Instrument has shut down. Cycle the power OFF for 10 seconds then ON. If the error persists call your PerkinElmer Service representative.</p>
<p><b>OVEN NOT CONFIGURED "Press OK to continue"</b>                      Oven is not configured (std/high power heater and line voltage). All instruments should have the oven configured before being shipped. Customer should not see this. Resolution must be by service.</p>
<p><b>OVEN CAL FAILURE "Press OK to continue"</b>                      Startup oven calibration procedure of inlet/exhaust doors failed. Warning</p>
<p><b>A/S control error "Pre-wash Timeout"</b>                      A sample pre-wash has taken place (reduced injection wait time) and system has not come ready within 30 minutes to inject. Warning</p>

*Troubleshooting*

**Table 10-3 Autosampler error messages.**

<b>Autosampler Error Messages</b>
Bad Sensor state
Bad motor mode
Bad motor speed
Elevator not initializing
Syringe error. Input value exceeds maximum volume of installed syringe.
Carousel not initializing
Tower not initializing
No W/W vial present
Invalid vial number
Invalid injection port
Slot not found firmware to clarify Tower location sensor not finding slot.
No vial at position xx.
Invalid syringe size
Encoder read error (reserved)
Encoder position err
Bar Code error (reserved)
Encoder position err
Bar Code error (reserved)
Vial Sensor not initializing
Phase error
Reserved for barcode reader
Bad slot firmware to clarify Tower slot is wrong width.
Bad volume firmware to clarify Incorrect volume for syringe.
Bad injection firmware to clarify Injection failed.

Bad motor id firmware to clarify Tried to move invalid motor.
Illegal command firmware to clarify Illegal command to Autosampler

**Table 10-4 –Formatted error messages.**

<b>Error Messages</b>
<b>Cannot delete oven</b> program step <step number>
<b>Cannot delete inlet</b> program step <step number>
<b>Invalid Method</b> <method number> in sequence
<b>Carrier</b> <carrier number> <b>unable to</b> <b>maintain pressure</b>
<b>Split leaking</b> <b>flow above max</b>
<b>FID flame out</b> Touch <b>OK</b> to continue
<b>Warning: Car</b> <inlet number> <b>Inlet</b> <b>&lt; 50 Psi</b> , Touch <b>OK</b> to continue

## Troubleshooting

**Table 4 Data Manager (DM) error messages.**

The DM Errors consist of a DM number and an associated index. The following table lists the errors, indices and gives a brief explanation of the error.

<b>Number</b>	<b>Index</b>	<b>Explanation</b>
1	1	Autosampler Resume Validation error
2	1	Autosampler Program Validation error
3	1	Background Compensation Validation error
4	PPC Channel 0 - 11	PPC Calibration Mode Validation error
5	PPC Channel 0 - 11	PPC Calibration Maximum Validation error
6	PPC Channel 0 - 11	PPC Calibration Minimum Validation error
7	11	Configuration Validation error
8	1	Cryo Configuration Validation error
9	1	Extended Configuration Validation error Valves 5 and 6, FPD PMT % voltages
10	1	PPC Configuration Validation error
11	1	Time Configuration Validation error
12	1	Autosampler Control Validation error
13	1	Flame Off Validation error
14	1	Flow Calibration Validation error
15	1	GC Serial Number Validation error
16	1	GC Tune Validation error
17	1	GC Tune Flag Validation error
18	1	GC Tune Mode Validation error
19	1	GC Tune Valid Flag Validation error
20	1	Heat Delay Validation error

21	1	HS 40 Mode Validation error
22	1	PPC Hardware Configuration Validation error
23	1	PPC Map Validation error
24	1	PPC Limits Validation error
25	1	Maxget Validation error
26	Method index 0 - 4	Method Extension Validation error Valves 5 and 6, PSS and POC Inlet 1 and 2 programs
27	Method index 0 - 4	Method Validation error
28	Method index 0 - 4	PPC Method Validation error
29	1	Oven Calibration Validation error
30	1	Priority Vial Cycles Validation error
31	1	Data Collection Rate Validation error
32	1	Sleep Validation error
33	1	Split Autozero Validation error
34	1	Start Structure Validation error
35	1	PPC References Validation error
36	1	TCD Protection Validation error
37	1	Valve Validation error
38	1	PPC Zero Offset Validation error

## **GC Troubleshooting**

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The cardinal rule in troubleshooting your gas chromatograph is "***If it ain't broke, don't fix it.***" When things are working fine, leave well enough alone, but when problems occur, this section will help you identify what could be wrong and how to solve your problem.

There are several sources of problems in gas chromatography:

***The operator:*** When the operator is new to chromatography and/or a new instrument, problems can be introduced during the learning curve. Once the operator becomes familiar with both the technique and the instrument, this problem source diminishes greatly.

***The sample:*** Unlike clean standards, real world samples such as environmental samples, can introduce problems because they are difficult to handle, have complicated matrices, contain unknown constituents, etc..

***The column:*** The column is most often the major factor contributing to poor analyses. The more a column is used, the greater the possibility of contamination, loss of substrate, etc. Columns do not last forever and should be changed when results become suspect.

***The gas flow system:*** Leaks are a major concern in gas chromatography and can lead to many problems.

***The electronics:*** The problem must be identified as either chromatographic or hardware. Electronics used in the system can malfunction.

***Data handling:*** Today, most chromatographers rely on sophisticated data handling systems to integrate their results. Some problems can be related to the incorrect setting of data handling parameters.



## **Spare Components.**

Following is a list of items you should have on hand to help solve problems.

*New syringes* – a syringe can break, become plugged or begin leaking. Always have spares available.

*Duplicate columns* – a column does not last forever; therefore, a duplicate column should be on hand in the event that your separation begins to degrade. Also, capillary columns can be damaged if oxygen is introduced at high temperatures. A duplicate column will allow you to identify if the column is the cause of the problem.

*Septa* – this is the one area of the gas chromatograph which requires routine maintenance. Always have spare septa available.

*Leak detector* – the gas flow system can be a problem as fittings wear with age and can begin to leak. A leak detector should be available to help find and fix leaks.

*Injector liners* – are made of glass and can be easily broken when removed. A supply of spare liners should therefore be kept on hand. Please remember that you cannot run satisfactory analyses without an injector liner.

## **Logical Troubleshooting Steps.**

There are some simple steps that should be taken when trying to locate the problem. Use the following guide to troubleshoot your GC.

Note the symptoms - define the problem. Compare your runs with good analysis, that is, with the results normally obtained.

Systematically eliminate possible causes.

The first rule here is, "What did you change last?" Many times a problem arises when a change is made to the system, such as changing a gas tank, septum, or glass liner. If the problem occurred after such a change, then the change is the most likely cause of the problem.

### *Troubleshooting*

Change the simplest thing first. For example, if you suspect a gas leak, the easiest change to make is the GC septum instead of replumbing the internal pneumatics.

Change only one GC parameter at a time and check for its effect. If you change three items at once and your problem goes away, you may not know which of the three moves or combination of moves corrected the problem. This way, if the problem happens again, you will know exactly what corrective action to take.

### ***Dual Identical Channels Only***

If your GC is a dual-channel system (dual identical detectors and dual identical injectors):

1. Try switching the column to the second channel. If the problem is corrected, then the problem was caused by the detector, injector, amplifier, or the pneumatics.
2. Replace each of the above components one at a time to identify which one is defective. If the problem is the same as before you switched the column, you should suspect the column, syringe, standard or sample, electronics, or data handling device.

# Appendix I U.S. Nuclear Regulations



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**NOTE:** *All USNRC regulations can be obtained through the internet at  
[www.nrc.gov/reading-rm/](http://www.nrc.gov/reading-rm/)*

*Appendix I*

*Appendix 1-2*

Appendix I - SUPPLEMENT 2  
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U.S. Nuclear Regulatory Commission Regional Offices

<b>REGION</b>	<b>ADDRESS</b>	<b>TELEPHONE</b>
I <b>WPI's Region</b>	U.S. Nuclear Regulatory Commission, Region I 475 Allendale Road King of Prussia, PA 19406-1415	(800) 432-1156
II	U.S. Nuclear Regulatory Commission, Region II 101 Marietta St., N.W., Suite 2900 Atlanta, GA 30323-0199	(800) 577-8510
III	U.S. Nuclear Regulatory Commission, Region III 801 Warrenville Road Lisle, IL 60137-5927	(800) 522-3025
IV	U.S. Nuclear Regulatory Commission, Region IV 611 Ryan Plaza Drive, Suite 400 Arlington, TX 76011-8064	(800) 952-9677
Walnut Creek Field Office	U.S. Nuclear Regulatory Commission 1450 Maria Lane Walnut Creek, CA 94596-5368	(800) 882-4672

## **Nuclear Regulatory Commission Regulations**

The following NRC regulations are from Title 10 Energy in the Code of Federal Regulations revised as of June 30, 1996.

### **Subpart M-Reports**

Source: 56 FR 23406, May 21, 1991, unless otherwise noted.

#### **§ 20.2201 Reports of theft or loss of licensed material.**

- (a) Telephone reports. (1) Each licensee shall report by telephone as follows:
  - (i) Immediately after its occurrence becomes known to the licensee, any lost, stolen, or missing licensed material in an aggregate quantity equal to or greater than 1,000 times the quantity specified in Appendix C to Part 20 under such circumstances that it appears to the licensee that an exposure could result to persons in unrestricted areas; or
  - (ii) Within 30 days after the occurrence of any lost, stolen, or missing licensed material becomes known to the licensee, all licensed material in a quantity greater than 10 times the quantity specified in Appendix C to Part 20 that is still missing at this time.
- (2) Reports must be made as follows:
  - (i) Licensees having an installed Emergency Notification System shall make the reports to the NRC Operations Center in accordance with § 50.72 of this chapter, and
  - (ii) All other licensees shall make reports by telephone to the NRC Operations Center (301-951-0550).
- (b) Written reports. (1) Each licensee required to make a report under paragraph (a) of this section shall, within 30 days after making the telephone report, make a written report setting forth the following information:
  - (i) A description of the licensed material involved, including kind, quantity, and chemical and physical form; and
  - (ii) A description of the circumstances under which the loss or theft occurred; and
  - (iii) A statement of disposition, or probable disposition, of the licensed material involved; and
  - (iv) Exposures of individuals to radiation, circumstances under which the exposures occurred, and the possible total effective dose equivalent to persons in unrestricted areas; and
  - (v) Actions that have been taken, or will be taken, to recover the material; and
  - (vi) Procedures or measures that have been, or will be, adopted to ensure against a recurrence of the loss or theft of licensed material.
- (2) Reports must be made as follows:
  - (i) For holders of an operating license for a nuclear power plant, the events included in paragraph (b) of this section must be reported in accordance with the procedures

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described in § 50.73(b), (c), (d), (e), and (g) of this chapter and must include the information required in paragraph (b)(1) of this section, and

(ii) All other licensees shall make reports to the Administrator of the appropriate NRC Regional Office listed in appendix D to Part 20.

(c) A duplicate report is not required under paragraph (b) of this section if the licensee is also required to submit a report pursuant to §§ 30.55(c), 40.64(c), 50.72, 50.73, 70.52, 73.27(b), 73.67(e)(3)(vi), 73.67(g)(3)(iii), 73.71, or 150.19(c) of this chapter.

(d) Subsequent to filing the written report, the licensee shall also report any additional substantive information on the loss or theft within 30 days after the licensee learns of such information.

(e) The licensee shall prepare any report filed with the Commission pursuant to this section so that names of individuals who may have received exposure to radiation are stated in a separate and detachable part of the report.

[56 FR 23406, May 21, 1991, as amended at 58 FR 69220, Dec. 30, 1993; 60 FR 20183, Apr. 25, 1995]

### **§ 20.2202 Notification of incidents.**

(a) Immediate notification. Notwithstanding any other requirements for notification, each licensee shall immediately report any event involving byproduct, source, or special nuclear material possessed by the licensee that may have caused or threatens to cause any of the following conditions-

(1) An individual to receive-

(i) A total effective dose equivalent of 25 rems (0.25 Sv) or more; or

(ii) An eye dose equivalent of 75 rems (0.75 Sv) or more; or

(iii) A shallow-dose equivalent to the skin or extremities of 250 rads (2.5 Gy) or more; or

(2) The release of radioactive material, inside or outside of a restricted area, so that, had an individual been present for 24 hours, the individual could have received an intake five times the annual limit on intake (the provisions of this paragraph do not apply to locations where personnel are not normally stationed during routine operations, such as hot-cells or process enclosures).

(b) Twenty-four hour notification. Each licensee shall, within 24 hours of discovery of the event, report any event involving loss of control of licensed material possessed by the licensee that may have caused, or threatens to cause, any of the following conditions:

(1) An individual to receive, in a period of 24 hours-

(i) A total effective dose equivalent exceeding 5 rems (0.05 Sv); or

(ii) An eye dose equivalent exceeding 15 rems (0.15 Sv); or

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(iii) A shallow-dose equivalent to the skin or extremities exceeding 50 rems (0.5 Sv); or  
(2) The release of radioactive material, inside or outside of a restricted area, so that, had an individual been present for 24 hours, the individual could have received an intake in excess of one occupational annual limit on intake (the provisions of this paragraph do not apply to locations where personnel

are not normally stationed during routine operations, such as hot-cells or process enclosures).

(c) The licensee shall prepare any report filed with the Commission pursuant to this section so that names of individuals who have received exposure to radiation or radioactive material are stated in a separate and detachable part of the report.

(d) Reports made by licensees in response to the requirements of this section must be made as follows:

(1) Licensees having an installed Emergency Notification System shall make the reports required by paragraphs (a) and (b) of this section to the NRC Operations Center in accordance with 10 CFR 50.72; and

(2) All other licensees shall make the reports required by paragraph (a) and (b) of this section by telephone to the NRC Operations Center (301) 816-5100 and by telegram, mailgram, or facsimile to the Administrator of the appropriate NRC Regional Office listed in appendix D to 10 CFR part 20.

(e) The provisions of this section do not include doses that result from planned special exposures, that are within the limits for planned special exposures, and that are reported under § 20.2204.

[56 FR 23406, May 21, 1991, as amended at 56 FR 40766, Aug. 16, 1991; 57 FR 57879, Dec. 8, 1992; 59 FR 14086, Mar. 25, 1994]

**§ 20.2203 Reports of exposures, radiation levels, and concentrations of radioactive material exceeding the limits.**

a) *Reportable events.* In addition to the notification required by §20.2202, each licensee shall submit a written report within 30 days after learning of any of the following occurrences:

(1) Any incident for which notification is required by §20.2202; or

(2) Doses in excess of any of the following:

(i) The occupational dose limits for adults in §20.1201; or

(ii) The occupational dose limits for a minor in §20.1207; or

(iii) The limits for an embryo/fetus of a declared pregnant woman in §20.1208; or

(iv) The limits for an individual member of the public in §20.1301; or

(v) Any applicable limit in the license; or

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- (vi) The ALARA constraints for air emissions established under §20.1101(d); or
  - (3) Levels of radiation or concentrations of radioactive material in --
    - (i) A restricted area in excess of any applicable limit in the license; or
    - (ii) An unrestricted area in excess of 10 times any applicable limit set forth in this part or in the license (whether or not involving exposure of any individual in excess of the limits in §20.1301); or
  - (4) For licensees subject to the provisions of EPA's generally applicable environmental radiation standards in 40 CFR part 190, levels of radiation or releases of radioactive material in excess of those standards, or of license conditions related to those standards.
- (b) *Contents of reports.* (1) Each report required by paragraph (a) of this section must describe the extent of exposure of individuals to radiation and radioactive material, including, as appropriate:
- (i) Estimates of each individual's dose; and
  - (ii) The levels of radiation and concentrations of radioactive material involved; and
  - (iii) The cause of the elevated exposures, dose rates, or concentrations; and
  - (iv) Corrective steps taken or planned to ensure against a recurrence, including the schedule for achieving conformance with applicable limits, ALARA constraints, generally applicable environmental standards, and associated license conditions.
- (2) Each report filed pursuant to paragraph (a) of this section must include for each occupationally overexposed<sup>(2)</sup> individual: the name, Social Security account number, and date of birth. The report must be prepared so that this information is stated in a separate and detachable part of the report.
- (c) For holders of an operating license for a nuclear power plant, the occurrences included in paragraph (a) of this section must be reported in accordance with the procedures described in §50.73(b), (c), (d), (e), and (g) of this chapter and must also include the information required by paragraph (b) of this section. Occurrences reported in accordance with §50.73 of this chapter need not be reported by a duplicate report under paragraph (a) of this section.
- (d) All licensees, other than those holding an operating license for a nuclear power plant, who make reports under paragraph (a) of this section shall submit the report in writing to the U.S. Nuclear Regulatory Commission, Document Control Desk, Washington, DC 20555, with a copy to the appropriate NRC Regional Office listed in appendix D to part 20.

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[56 FR 23406, May 21, 1991, as amended at 60 FR 20186, Apr. 25, 1995; 61 FR 65127, Dec. 10, 1996]

<sup>7</sup> With respect to the limit for the embryo-fetus (§20.1208), the identifiers should be those of the declared pregnant woman.

**§20.2204 Reports of planned special exposures.**

The licensee shall submit a written report to the Administrator of the appropriate NRC Regional Office listed in appendix D to Part 20 within 30 days following any planned special exposure conducted in accordance with § 20.1206, informing the Commission that a planned special exposure was conducted and indicating the date the planned special exposure occurred and the information required by § 20.2105.

[Amended 60 FR 20183, Apr. 25, 1995]

**§ 20.2205 Reports to individuals of exceeding dose limits.**

When a licensee is required, pursuant to the provisions of §§20.2203, 20.2204, or 20.2206, to report to the Commission any exposure of an identified occupationally exposed individual, or an identified member of the public, to radiation or radioactive material, the licensee shall also provide a copy of the report submitted to the Commission to the individual. This report must be transmitted at a time no later than the transmittal to the Commission.

[60 FR 36043, July 13, 1995]

**§20.2206 Reports of individual monitoring.**

(a) This section applies to each person licensed by the Commission to-

(1) Operate a nuclear reactor designed to produce electrical or heat energy pursuant to §50.21(b) or §50.22 of this chapter or a testing facility as defined in §50.2 of this chapter; or

(2) Possess or use byproduct material for purposes of radiography pursuant to Parts 30 and 34 of this chapter; or

(3) Possess or use at any one time, for purposes of fuel processing, fabricating, or reprocessing, special nuclear material in a quantity exceeding 5,000 grams of contained uranium-235, uranium-233, or plutonium, or any combination thereof pursuant to part 70 of this chapter; or

(4) Possess high-level radioactive waste at a geologic repository operations area pursuant to part 60 of this chapter; or

(5) Possess spent fuel in an independent spent fuel storage installation (ISFSI) pursuant to part 72 of this chapter; or

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(6) Receive radioactive waste from other persons for disposal under part 61 of this chapter; or

(7) Possess or use at any time, for processing or manufacturing for distribution pursuant to parts 30, 32, 33 or 35 of this chapter, byproduct material in quantities exceeding any one of the following quantities:

<b>Radionuclide</b>	<b>Quantity of radionuclide<sup>1</sup> in curies</b>
Cesium-137	1
Cobalt-60	1
Gold-198	100
Iodine-131	1
Iridium-192	10
Krypton-85	1,000
Promethium-147	10
Techetium-99m	1,000

<sup>1</sup> The Commission may require as a license condition, or by rule, regulation, or order pursuant to § 20.2302, reports from licensees who are licensed to use radionuclides not on this list, in quantities sufficient to cause comparable radiation levels.

(b) Each licensee in a category listed in paragraph (a) of this section shall submit an annual report of the results of individual monitoring carried out by the licensee for each individual for whom monitoring was required by § 20.1502 during that year. The licensee may include additional data for individuals for whom monitoring was provided but not required. The licensee shall use Form NRC 5 or electronic media containing all the information required by Form NRC 5.

(c) The licensee shall file the report required by § 20.2206(b), covering the preceding year, on or before April 30 of each year. The licensee shall submit the report to the REIRS Project Manager, Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission, Washington, DC 20555.

[56 FR 23406, May 21, 1991, as amended at 56 FR 32072, July 15, 1991]

**§ 30.34 Terms and conditions of licenses.**

(a) Each license issued pursuant to the regulations in this part and the regulations in parts 31 through 36 and 39 of this chapter shall be subject to all the provisions of the Act, now or hereafter in effect, and to all valid rules, regulations and orders of the Commission.

(b) No license issued or granted pursuant to the regulations in this part and parts 31 through 36, and 39 nor any right under a license shall be transferred, assigned or in any manner disposed of, either voluntarily or involuntarily, directly or indirectly, through transfer of control of any license to any person, unless the Commission shall, after

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securing full information, find that the transfer is in accordance with the provisions of the Act and shall give its consent in writing.

(c) Each person licensed by the Commission pursuant to the regulations in this part and parts 31 through 36 and 39 shall confine his possession and use of the byproduct material to the locations and purposes authorized in the license. Except as otherwise provided in the license, a license issued pursuant to the regulations in this part and parts 31 through 36 and 39 of this chapter shall carry with it the right to receive, acquire, own, and possess byproduct material. Preparation for shipment and transport of byproduct material shall be in accordance with the provisions of part 71 of this chapter.

(d) Each license issued pursuant to the regulations in this part and parts 31 through 36 and 39 shall be deemed to contain the provisions set forth in section 183b. - d., inclusive, of the Act, whether or not these provisions are expressly set forth in the license.

(e) The Commission may incorporate, in any license issued pursuant to the regulations in this part and parts 31 through 36 and 39, at the time of issuance, or thereafter by appropriate rule, regulation or order, such additional requirements and conditions with respect to the licensee's receipt, possession, use and transfer of byproduct material as it deems appropriate or necessary in order to:

(1) Promote the common defense and security;

(2) Protect health or to minimize danger to life or property;

(3) Protect restricted data;

(4) Require such reports and the keeping of such records, and to provide for such inspections of activities under the license as may be necessary or appropriate to effectuate the purposes of the Act and regulations thereunder.

(f) Licensees required to submit emergency plans by §30.32(i) shall follow the emergency plan approved by the Commission. The licensee may change the approved without Commission approval only if the changes do not decrease the effectiveness of the plan. The licensee shall furnish the change to the appropriate NRC Regional Office specified in §30.6 and to affected offsite response organizations within six months after the change is made. Proposed changes that decrease, or potentially decrease, the effectiveness of the approved emergency plan may not be implemented without prior application to and prior approval by the Commission.

(g) Each licensee preparing technetium-99m radiopharmaceuticals from molybdenum-99/technetium-99m generators shall test the generator eluates for molybdenum-99 breakthrough in accordance with §35.204 of this chapter. The licensee shall record the results of each test and retain each record for three years after the record is made.

(h)(1) Each general licensee that is required to register by Sec. 31.5(c)(13) of this chapter and each specific licensee shall notify the appropriate NRC Regional Administrator, in writing, immediately following the filing of a voluntary or involuntary petition for bankruptcy under any chapter of title 11 (Bankruptcy) of the United States Code by or against:

(i) The licensee;

(ii) An entity (as that term is defined in 11 U.S.C. 101(14)) controlling the licensee or listing the license or licensee as property of the estate; or

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- (iii) An affiliate (as that term is defined in 11 U.S.C. 101(2)) of the licensee.
- (2) This notification must indicate:
  - (i) The bankruptcy court in which the petition for bankruptcy was filed; and
  - (ii) The date of the filing of the petition.

**§ 30.35 Financial assurance and record keeping for decommissioning.**

a) Each applicant for a specific license authorizing the possession and use of unsealed byproduct material of half-life greater than 120 days and in quantities exceeding  $10^5$  times the applicable quantities set forth in appendix B to part 30 shall submit a decommissioning funding plan as described in paragraph (e) of this section. The decommissioning funding plan must also be submitted when a combination of isotopes is involved if  $R$  divided by  $10^5$  is greater than 1 (unity rule), where  $R$  is defined here as the sum of the ratios of the quantity of each isotope to the applicable value in appendix B to part 30.

(b) Each applicant for a specific license authorizing possession and use of byproduct material of half-life greater than 120 days and in quantities specified in paragraph (d) of this section shall either --

(1) Submit a decommissioning funding plan as described in paragraph (e) of this section; or

(2) Submit a certification that financial assurance for decommissioning has been provided in the amount prescribed by paragraph (d) of this section using one of the methods described in paragraph (f) of this section. For an applicant, this certification may state that the appropriate assurance will be obtained after the application has been approved and the license issued but before the receipt of licensed material. If the applicant defers execution of the financial instrument until after the license has been issued, a signed original of the financial instrument obtained to satisfy the requirements of paragraph (f) of this section must be submitted to NRC before receipt of licensed material. If the applicant does not defer execution of the financial instrument, the applicant shall submit to NRC, as part of the certification, a signed original of the financial instrument obtained to satisfy the requirements of paragraph (f) of this section.

(c)(1) Each holder of a specific license issued on or after July 27, 1990, which is of a type described in paragraph (a) or (b) of this section, shall provide financial assurance for decommissioning in accordance with the criteria set forth in this section.

(2) Each holder of a specific license issued before July 27, 1990, and of a type described in paragraph (a) of this section shall submit, on or before July 27, 1990, a decommissioning funding plan as described in paragraph (e) of this section or a certification of financial assurance for decommissioning in an amount at least equal to \$750,000 in accordance with the criteria set forth in this section. If the licensee submits the certification of financial assurance rather than a decommissioning funding plan, the licensee shall include a decommissioning funding plan in any application for license renewal.

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(3) Each holder of a specific license issued before July 27, 1990, and of a type described in paragraph (b) of this section shall submit, on or before July 27, 1990, a decommissioning funding plan as described, in paragraph (e) of this section, or a certification of financial assurance for decommissioning in accordance with the criteria set forth in this section.

(4) Any licensee who has submitted an application before July 27, 1990, for renewal of license in accordance with §30.37 shall provide financial assurance for decommissioning in accordance with paragraphs (a) and (b) of this section. This assurance must be submitted when this rule becomes effective November 24, 1995.

(d) Table of required amounts of financial assurance for decommissioning by quantity of material.

greater than  $10^4$  but less than or equal to  $10^5$  times the applicable quantities of appendix B to part 30 in unsealed form. (For a combination of isotopes, if R, as defined in §30.35(a), divided by  $10^4$  is greater than 1 but R divided by  $10^5$  is less than or equal to 1.) ....

\$750,000

greater than  $10^3$  but less than or equal to  $10^4$  times the applicable quantities of appendix B to part 30 in unsealed form. (For a combination of isotopes, if R, as defined in §30.35(a), divided by  $10^3$  is greater than 1 but R divided by  $10^4$  is less than or equal to 1.) ....

\$150,000

greater than  $10^{10}$  times the applicable quantities of appendix B to part 30 in sealed sources or plated foils. (For a combination of isotopes, if R, as defined in §30.35(a), divided by  $10^{10}$  is greater than 1). ....

\$75,000  
(e) Each decommissioning funding plan must contain a cost estimate for decommissioning and a description of the method of assuring funds for decommissioning from paragraph (f) of this section, including means for adjusting cost estimates and associated funding levels periodically over the life of the facility. The decommissioning funding plan must also contain a certification by the licensee that financial assurance for decommissioning has been provided in the amount of the cost estimate for decommissioning and a signed original of the financial instrument obtained to satisfy the requirements of paragraph (f) of this section.

(f) Financial assurance for decommissioning must be provided by one or more of the following methods:

(1) Prepayment. Prepayment is the deposit prior to the start of operation into an account segregated from licensee assets and outside the licensee's administrative control of cash or liquid assets such that the amount of funds would be sufficient to pay decommissioning costs. Prepayment may be in the form of a trust, escrow account, government fund, certificate of deposit, or deposit of government securities.

(2) A surety method, insurance, or other guarantee method. These methods guarantee that decommissioning costs will be paid. A surety method may be in the form of a surety bond, letter of credit, or line of credit. A parent company guarantee of funds for decommissioning costs based on a financial test may be used if the guarantee and test are as contained in appendix A to this part. A parent company guarantee may not be used in combination with other financial methods to satisfy the requirements of this section. For

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commercial corporations that issue bonds, a guarantee of funds by the applicant or licensee for decommissioning costs based on a financial test may be used if the guarantee and test are as contained in appendix C to this part. For commercial companies that do not issue bonds, a guarantee of funds by the applicant or licensee for decommissioning costs may be used if the guarantee and test are as contained in appendix D to this part. For nonprofit entities, such as colleges, universities, and nonprofit hospitals, a guarantee of funds by the applicant or licensee may be used if the guarantee and test are as contained in appendix E to this part. A guarantee by the applicant or licensee may not be used in combination with any other financial methods used to satisfy the requirements of this section or in any situation where the applicant or licensee has a parent company holding majority control of the voting stock of the company. Any surety method or insurance used to provide financial assurance for decommissioning must contain the following conditions:

(i) The surety method or insurance must be open-ended or, if written for a specified term, such as five years, must be renewed automatically unless 90 days or more prior to the renewal date, the issuer notifies the Commission, the beneficiary, and the licensee of its intention not to renew. The surety method or insurance must also provide that the full face amount be paid to the beneficiary automatically prior to the expiration without proof of forfeiture if the licensee fails to provide a replacement acceptable to the Commission within 30 days after receipt of notification of cancellation.

(ii) The surety method or insurance must be payable to a trust established for decommissioning costs. The trustee and trust must be acceptable to the Commission. An acceptable trustee includes an appropriate State or Federal government agency or an entity which has the authority to act as a trustee and whose trust operations are regulated and examined by a Federal or State agency.

(iii) The surety method or insurance must remain in effect until the Commission has terminated the license.

(3) An external sinking fund in which deposits are made at least annually, coupled with a surety method or insurance, the value of which may decrease by the amount being accumulated in the sinking fund. An external sinking fund is a fund established and maintained by setting aside funds periodically in an account segregated from licensee assets and outside the licensee's administrative control in which the total amount of funds would be sufficient to pay decommissioning costs at the time termination of operation is expected. An external sinking fund may be in the form of a trust, escrow account, government fund, certificate of deposit, or deposit of government securities. The surety or insurance provisions must be as stated in paragraph (f)(2) of this section.

(4) In the case of Federal, State, or local government licensees, a statement of intent containing a cost estimate for decommissioning or an amount based on the Table in paragraph (d) of this section, and indicating that funds for decommissioning will be obtained when necessary.

(5) When a governmental entity is assuming custody and ownership of a site, an arrangement that is deemed acceptable by such governmental entity.

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(g) Each person licensed under this part or parts 32 through 36 and 39 of this chapter shall keep records of information important to the decommissioning of a facility in an identified location until the site is released for unrestricted use. Before licensed activities are transferred or assigned in accordance with §30.34(b), licensees shall transfer all records described in this paragraph to the new licensee. In this case, the new licensee will be responsible for maintaining these records until the license is terminated. If records important to the decommissioning of a facility are kept for other purposes, reference to these records and their locations may be used. Information the Commission considers important to decommissioning consists of --

(1) Records of spills or other unusual occurrences involving the spread of contamination in and around the facility, equipment, or site. These records may be limited to instances when contamination remains after any cleanup procedures or when there is reasonable likelihood that contaminants may have spread to inaccessible areas as in the case of possible seepage into porous materials such as concrete. These records must include any known information on identification of involved nuclides, quantities, forms, and concentrations.

(2) As-built drawings and modifications of structures and equipment in restricted areas where radioactive materials are used and/or stored, and of locations of possible inaccessible contamination such as buried pipes which may be subject to contamination. If required drawings are referenced, each relevant document need not be indexed individually. If drawings are not available, the licensee shall substitute appropriate records of available information concerning these areas and locations.

(3) Except for areas containing only sealed sources (provided the sources have not leaked or no contamination remains after any leak) or byproduct materials having only half-lives of less than 65 days, a list contained in a single document and updated every 2 years, of the following:

(i) All areas designated and formerly designated restricted areas as defined in 10 CFR 20.1003 (For requirements prior to January 1, 1994, see 10 CFR 20.3 as contained in the CFR edition revised as of January 1, 1993.);

(ii) All areas outside of restricted areas that require documentation under §30.35(g)(1).

(iii) All areas outside of restricted areas where current and previous wastes have been buried as documented under 10 CFR 20.2108; and

(iv) All areas outside of restricted areas that contain material such that, if the license expired, the licensee would be required to either decontaminate the area to meet the criteria for decommissioning in 10 CFR part 20, subpart E, or apply for approval for disposal under 10 CFR 20.2002.

(4) Records of the cost estimate performed for the decommissioning funding plan or of the amount certified for decommissioning, and records of the funding method used for assuring funds if either a funding plan or certification is used.

[53 FR 24044, June 27, 1988, as amended at 56 FR 23471, May 21, 1991; 58 FR 39633, July 26, 1993; 58 FR 67659, Dec. 22, 1993; 58 FR 68730, Dec. 29, 1993; 59 FR 1618, Jan. 12, 1994; 60 FR 38238, July 26, 1995; 61 FR 24673, May 16, 1996; 62 FR 39090, July 21, 1997]

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**§ 30.41 Transfer of byproduct material.**

(a) No licensee shall transfer byproduct material except as authorized pursuant to this section.

(b) Except as otherwise provided in his license and subject to the provisions of paragraphs (c) and (d) of this section, any licensee may transfer byproduct material:

- (1) To the Department;
- (2) To the agency in any Agreement State which regulates radioactive material pursuant to an agreement under section 274 of the Act;
- (3) To any person exempt from the licensing requirements of the Act and regulations in this part, to the extent permitted under such exemption;
- (4) To any person in an Agreement State, subject to the jurisdiction of that State, who has been exempted from the licensing requirements and regulations of that State, to the extent permitted under such exemption;
- (5) To any person authorized to receive such byproduct material under terms of a specific license or a general license or their equivalents issued by the Atomic Energy Commission, the Commission, or an Agreement State;
- (6) To a person abroad pursuant to an export license issued under part 110 of this chapter; or

(7) As otherwise authorized by the Commission in writing. (c) Before transferring byproduct material to a specific licensee of the Commission or an Agreement State or to a general licensee who is required to register with the Commission or with an Agreement State prior to receipt of the byproduct material, the licensee transferring the material shall verify that the transferee's license authorizes the receipt of the type, form, and quantity of byproduct material to be transferred.

(d) The following methods for the verification required by paragraph (c) of this section are acceptable:

- (1) The transferor may have in his possession, and read, a current copy of the transferee's specific license or registration certificate;
- (2) The transferor may have in his possession a written certification by the transferee that he is authorized by license or registration certificate to receive the type, form, and quantity of byproduct material to be transferred, specifying the license or registration certificate number, issuing agency and expiration date;
- (3) For emergency shipments the transferor may accept oral certification by the transferee that he is authorized by license or registration certificate to receive the type, form, and quantity of byproduct material to be transferred, specifying the license or registration certificate number, issuing agency and expiration date: Provided, That the oral certification is confirmed in writing within 10 days;
- (4) The transferor may obtain other sources of information compiled by a reporting service from official records of the Commission or the licensing agency of an Agreement State as to the identity of licensees and the scope and expiration dates of licenses and registration; or

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(5) When none of the methods of verification described in paragraphs (d)(1) to (4) of this section are readily available or when a transferor desires to verify that information received by one of such methods is correct or up-to-date, the transferor may obtain and record confirmation from the Commission or the licensing agency of an Agreement State that the transferee is licensed to receive the byproduct material.

[38 FR 33969, Dec. 10, 1973, as amended at 40 FR 8785, Mar. 3, 1975; 43 FR 6922, Feb. 17, 1978]

## **RECORDS, INSPECTIONS, TESTS, AND REPORTS**

### **§ 30.50 Reporting requirements.**

(a) Immediate report. Each licensee shall notify the NRC as soon as possible but not later than 4 hours after the discovery of an event that prevents immediate protective actions necessary to avoid exposures to radiation or radioactive materials that could exceed regulatory limits or releases of licensed material that could exceed regulatory limits (events may include fires, explosions, toxic gas releases, etc.).

(b) Twenty-four hour report. Each licensee shall notify the NRC within 24 hours after the discovery of any of the following events involving licensed material:

(1) An unplanned contamination event that:

(i) Requires access to the contaminated area, by workers or the public, to be restricted for more than 24 hours by imposing additional radiological controls or by prohibiting entry into the area;

(ii) Involves a quantity of material greater than five times the lowest annual limit on intake specified in appendix B of §§ 20.1001-20.2401 of 10 CFR part 20 for the material; and

(iii) Has access to the area restricted for a reason other than to allow isotopes with a half-life of less than 24 hours to decay prior to decontamination.

(2) An event in which equipment is disabled or fails to function as designed when:

(i) The equipment is required by regulation or license condition to prevent releases exceeding regulatory limits, to prevent exposures to radiation and radioactive materials exceeding regulatory limits, or to mitigate the consequences of an accident;

(ii) The equipment is required to be available and operable when it is disabled or fails to function; and

(iii) No redundant equipment is available and operable to perform the required safety function.

(3) An event that requires unplanned medical treatment at a medical facility of an individual with spreadable radioactive contamination on the individual's clothing or body.

(4) An unplanned fire or explosion damaging any licensed material or any device, container, or equipment containing licensed material when:

(i) The quantity of material involved is greater than five times the lowest annual limit on intake specified in appendix B of §§ 20.1001-20.2401 of 10 CFR part 20 for the material; and

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(ii) The damage affects the integrity of the licensed material or its container.

(c) Preparation and submission of reports. Reports made by licensees in response to the requirements of this section must be made as follows:

(1) Licensees shall make reports required by paragraphs (a) and (b) of this section by telephone to the NRC Operations Center. To the extent that the information is available at the time of notification, the information provided in these reports must include:

The commercial telephone number for the NRC Operations Center is (301) 816-5100.

(i) The caller's name and call back telephone number;

(ii) A description of the event, including date and time;

(iii) The exact location of the event;

(iv) The isotopes, quantities, and chemical and physical form of the licensed material involved; and

(v) Any personnel radiation exposure data available.

(2) Written report. Each licensee who makes a report required by paragraph (a) or (b) of this section shall submit a written follow-up report within 30 days of the initial report. Written reports prepared pursuant to other regulations may be submitted to fulfill this requirement if the reports contain all of the necessary information and the appropriate distribution is made. These written reports must be sent to the U.S. Nuclear Regulatory Commission, Document Control Desk, Washington, DC 20555, with a copy to the appropriate NRC Regional office listed in appendix D of 10 CFR part 20. The reports must include the following:

(i) A description of the event, including the probable cause and the manufacturer and model number (if applicable) of any equipment that failed or malfunctioned;

(ii) The exact location of the event;

(iii) The isotopes, quantities, and chemical and physical form of the licensed material involved;

(iv) Date and time of the event;

(v) Corrective actions taken or planned and the results of any evaluations or assessments; and

(vi) The extent of exposure of individuals to radiation or to radioactive materials without identification of individuals by name.

(3) The provisions of § 30.50 do not apply to licensees subject to the notification requirements in

§ 50.72. They do apply to those part 50 licensees possessing material licensed under part 30, who are not subject to the notification requirements in § 50.72.

[56 FR 40767, Aug. 16, 1991, as amended at 59 FR 14086, Mar. 25, 1994]

### **§ 30.51 Records.**

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(a) Each person who receives byproduct material pursuant to a license issued pursuant to the regulations in this part and parts 31 through 36 of this chapter shall keep records showing the receipt, transfer, and disposal of the byproduct material as follows:

(1) The licensee shall retain each record of receipt of byproduct material as long as the material is possessed and for three years following transfer or disposal of the material.

(2) The licensee who transferred the material shall retain each record of transfer for three years after each transfer unless a specific requirement in another part of the regulations in this chapter dictates otherwise.

(3) The licensee who disposed of the material shall retain each record of disposal of byproduct material until the Commission terminates each license that authorizes disposal of the material.

(b) The licensee shall retain each record that is required by the regulations in this part and parts 31 through 36 of this chapter or by license condition for the period specified by the appropriate regulation or license condition. If a retention period is not otherwise specified by regulation or license condition, the record must be retained until the Commission terminates each license that authorizes the activity that is subject to the recordkeeping requirement.

(c)(1) Records which must be maintained pursuant to this part and parts 31 through 36 of this chapter may be the original or a reproduced copy or microform if such reproduced copy or microform is duly authenticated by authorized personnel and the microform is capable of producing a clear and legible copy after storage for the period specified by Commission regulations. The record may also be stored in electronic media with the capability for producing legible, accurate, and complete records during the required retention period. Records such as letters, drawings, specifications, must include all pertinent information such as stamps, initials, and signatures. The licensee shall maintain adequate safeguards against tampering with and loss of records.

(2) If there is a conflict between the Commission's regulations in this part and parts 31 through 36 and 39 of this chapter, license condition, or other written Commission approval or authorization pertaining to the retention period for the same type of record, the retention period specified in the regulations in this part and parts 31 through 36 and 39 of this chapter for such records shall apply unless the Commission, pursuant to § 30.11, has granted a specific exemption from the record retention requirements specified in the regulations in this part or parts 31 through 36 and 39 of this chapter.

(d) Prior to license termination, each licensee authorized to possess radioactive material with a half-life greater than 120 days, in an unsealed form, shall forward the following records to the appropriate NRC Regional Office:

(1) Records of disposal of licensed material made under §§ 20.2002 (including burials authorized before January 28, 1981 {1}), 20.2003, 20.2004, 20.2005; and | {1} A previous § 20.304 permitted burial of small |quantities of licensed materials in soil before January |28, 1981, without specific Commission authorization. |See § 20.304 contained in the 10 CFR, parts 0 to 199, |edition revised as of January 1, 1981. (2) Records required by § 20.2103(b)(4).

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(e) If licensed activities are transferred or assigned in accordance with § 30.34(b), each licensee authorized to possess radioactive material, with a half-life greater than 120 days, in an unsealed form, shall transfer the following records to the new licensee and the new licensee will be responsible for maintaining these records until the license is terminated:

(1) Records of disposal of licensed material made under §§ 20.2002 (including burials authorized before January 28, 1981 {1}), 20.2003, 20.2004, 20.2005; and | {1} A previous § 20.304 permitted burial of small |quantities of licensed materials in soil before January |28, 1981, without specific Commission authorization. |See § 20.304 contained in the 10 CFR, parts 0 to 199, |edition revised as of January 1, 1981.

(2) Records required by § 20.2103(b)(4).

(f) Prior to license termination, each licensee shall forward the records required by § 30.35(g) to the appropriate NRC Regional Office.

[41 FR 18301, May 5, 1976, as amended at 43 FR 6922, Feb. 17, 1978; 52 FR 8241, Mar. 17, 1987; 53 FR 19245, May 27, 1988; 58 FR 7736, Feb. 9, 1993; 61 FR 24669, May 16, 1996]

### **§ 30.52 Inspections.**

(a) Each licensee shall afford to the Commission at all reasonable times opportunity to inspect byproduct material and the premises and facilities wherein byproduct material is used or stored.

(b) Each licensee shall make available to the Commission for inspection, upon reasonable notice, records kept by him pursuant to the regulations in this chapter.

[30 FR 8185, June 26, 1965]

### **§ 30.53 Tests.**

Each licensee shall perform, or permit the Commission to perform, such tests as the Commission deems appropriate or necessary for the administration of the regulations in this part and parts 31 through 36 and 39 of this chapter, including tests of:

- (a) Byproduct material;
- (b) Facilities wherein byproduct material is utilized or stored;
- (c) Radiation detection and monitoring instruments; and
- (d) Other equipment and devices used in connection with the utilization or storage of byproduct material.

[30 FR 8185, June 26, 1965, as amended by 43 FR 6922, Feb. 17, 1978; 52 FR 8241, Mar. 17, 1987; 58 FR 7736, Feb. 9, 1993]

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**§ 30.55 Tritium reports.**

(a)-(b) [Reserved]

(c) Except as specified in paragraph (d) of this section, each licensee who is authorized to possess tritium shall report promptly to the appropriate NRC Regional Office listed in appendix D of part 20 of this chapter by telephone and telegraph, mailgram, or facsimile any incident in which an attempt has been made or is believed to have been made to commit a theft or unlawful diversion of more than 10 curies of such material at any one time or more than 100 curies of such material in any one calendar year. The initial report shall be followed within a period of fifteen (15) days by a written report submitted to the appropriate NRC Regional Office which sets forth the details of the incident and its consequences. Copies of such written report shall be sent to the Director, Office of Nuclear Material Safety and Safeguards, U.S. Nuclear Regulatory Commission, Washington, DC 20555. Subsequent to the submission of the written report required by this paragraph, the licensee shall promptly inform the Office of Nuclear Material Safety and Safeguards by means of a written report of any substantive additional information, which becomes available to the licensee, concerning an attempted or apparent theft or unlawful diversion of tritium.

(d) The reports described in this section are not required for tritium possessed pursuant to a general license provided in part 31 of this chapter or for tritium contained in spent fuel.

[37 FR 9208, May 6, 1972, as amended at 38 FR 1271, Jan. 11, 1973; 38 FR 2330, Jan. 24, 1973; 41 FR 16446, Apr. 19, 1976; 43 FR 6922, Feb. 17, 1978; 46 FR 55085, Nov. 6, 1981; 49 FR 24707, June 15, 1984; 52 FR 31611, Aug. 21, 1987]

**ENFORCEMENT**

**§ 30.61 Modification and revocation of licenses.**

(a) The terms and conditions of each license issued pursuant to the regulations in this part and parts 31 through 35 of this chapter shall be subject to amendment, revision or modification by reason of amendments to the Act, or by reason of rules, regulations and orders issued in accordance with the terms of the Act.

(b) Any license may be revoked, suspended or modified, in whole or in part, for any material false statement in the application or any statement of fact required under section 182 of the Act, or because of conditions revealed by such application or statement of fact or any report, record or inspection or other means which would warrant the Commission to refuse to grant a license on an original application, or for violation of, or failure to observe any of the terms and provisions of the Act or of any rule, regulation or order of the Commission.

(c) Except in cases of willfulness or those in which the public health, interest or safety requires otherwise, no license shall be modified, suspended or revoked unless, prior to the

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institution of proceedings therefore, facts or conduct which may warrant such action shall have been called to the attention of the licensee in writing and the licensee shall have been accorded an opportunity to demonstrate or achieve compliance with all lawful requirements.

[30 FR 8185, June 26, 1965, as amended at 35 FR 11460, July 17, 1970; 43 FR 6922, Feb. 17, 1978]

§ 30.62 Right to cause the withholding or recall of byproduct material. The Commission may cause the withholding or recall of byproduct material from any licensee who is not equipped to observe or fails to observe such safety standards to protect health as may be established by the Commission, or who uses such materials in violation of law or regulation of the Commission, or in a manner other than as disclosed in the application therefore or approved by the Commission.

[30 FR 8185, June 26, 1965, as amended at 40 FR 8785, Mar. 3, 1975]

### **§ 30.63 Violations.**

(a) The Commission may obtain an injunction or other court order to prevent a violation of the provisions of-

(1) The Atomic Energy Act of 1954, as amended; (2) Title II of the Energy Reorganization Act of 1974, as amended; or  
(3) A regulation or order issued pursuant to those Acts.

(b) The Commission may obtain a court order for the payment of a civil penalty imposed under section 234 of the Atomic Energy Act:

(1) For violations of-

(i) Sections 53, 57, 62, 63, 81, 82, 101, 103, 104, 107, or 109 of the Atomic Energy Act of 1954, as amended;

(ii) Section 206 of the Energy Reorganization Act;

(iii) Any rule, regulation, or order issued pursuant to the sections specified in paragraph

(b)(1)(i) of this section;

(iv) Any term, condition, or limitation of any license issued under the sections specified in paragraph (b)(1)(i) of this section.

(2) For any violation for which a license may be revoked under section 186 of the Atomic Energy Act of 1954, as amended.

[57 FR 55072, Nov. 24, 1992]

### **§ 30.64 Criminal penalties.**

(a) Section 223 of the Atomic Energy Act of 1954, as amended, provides for criminal sanctions for willful violation of, attempted violation of, or conspiracy to violate, any regulation issued under sections 161b, 161i, or 161o of the Act. For purposes of section

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223, all the regulations in part 30 are issued under one or more of sections 161b, 161i, or 161o, except for the sections listed in paragraph (b) of this section.

(b) The regulations in part 30 that are not issued under sections 161b, 161i, or 161o for the purposes of section 223 are as follows:

§§ 30.1, 30.2, 30.4, 30.5, 30.6, 30.8, 30.11, 30.12, 30.13, 30.15, 30.16, 30.31, 30.32, 30.33, 30.37, 30.38, 30.39, 30.61, 30.62, 30.63, 30.64, 30.70, 30.71, and 30.72.

[57 FR 55072, Nov. 24, 1992]

### Schedules

#### § 30.70 Schedule A-exempt concentrations.

[See footnotes at end of this table]

Element (atomic number)	Isotope	Col. I Gas concentration μCi/ml {1}	Col. II Liquid and solid concentration μCi/ml {2}
Antimony (51)	Sb 122		3x10 <sup>-4</sup>
	Sb 124		2x10 <sup>-4</sup>
	Sb 125		1x10 <sup>-3</sup>
Argon (18)	A 37	1x10 <sup>-3</sup>	
	A 41	4x10 <sup>-7</sup>	
Arsenic (33)	As 73		5x10 <sup>-3</sup>
	As 74		5x10 <sup>-4</sup>
	As 76		2x10 <sup>-4</sup>
	As 77		8x10 <sup>-4</sup>
Barium (56)	Ba 131		2x10 <sup>-3</sup>
	Ba 140		3x10 <sup>-4</sup>
Beryllium (4)	Be 7		2x10 <sup>-2</sup>
Bismuth (83)	Bi 206		4x10 <sup>-4</sup>
Bromine (35)	Br 82	4x10 <sup>-7</sup>	3x10 <sup>-3</sup>
Cadmium (48)	Cd 109		2x10 <sup>-3</sup>
	Cd 115m		3x10 <sup>-4</sup>
	Cd 115		3x10 <sup>-4</sup>
Calcium (20)	Ca 45		9x10 <sup>-5</sup>
	Ca 47		5x10 <sup>-4</sup>
Carbon (6)	C 14	1x10 <sup>-6</sup>	8x10 <sup>-3</sup>
Cerium (58)	Ce 141		9x10 <sup>-4</sup>
	Ce 143		4x10 <sup>-4</sup>
	Ce 144		1x10 <sup>-4</sup>
Cesium (55)	Cs 131		2x10 <sup>-2</sup>
	Cs 134m		6x10 <sup>-2</sup>
	Cs 134		9x10 <sup>-5</sup>
Chlorine (17)	Cl 38	9x10 <sup>-7</sup>	4x10 <sup>-3</sup>
Chromium (24)	Cr 51		2x10 <sup>-2</sup>
Cobalt (27)	Co 57		5x10 <sup>-3</sup>
	Co 58		1x10 <sup>-3</sup>
	Co 60		5x10 <sup>-4</sup>
	Co 64		3x10 <sup>-3</sup>
Copper (29)	Cu 64		3x10 <sup>-3</sup>
Dysprosium (66)	Dy 165		4x10 <sup>-3</sup>
	Dy 166		4x10 <sup>-4</sup>

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Erbium (68)	Er 169		9x10 <sup>-4</sup>
	Er 171		1x10 <sup>-3</sup>
Europium (63)	Eu 152		6x10 <sup>-4</sup>
	(T/2=9.2 Hrs)		
	Eu 155		2x10 <sup>-3</sup>
Fluorine (9)	F 18	2x10 <sup>-6</sup>	8x10 <sup>-3</sup>
Gadolinium (64)	Gd 153		2x10 <sup>-3</sup>
	Gd 159		8x10 <sup>-4</sup>
<b>Element (atomic number)</b>	<b>Isotope</b>	<b>Col. I</b>	<b>Col. II</b>
Gallium (31)	Ga 72		4x10 <sup>-4</sup>
Germanium (32)	Ge 71		2x10 <sup>-2</sup>
Gold (79)	Au 196		2x10 <sup>-3</sup>
	Au 198		5x10 <sup>-4</sup>
	Au 199		2x10 <sup>-3</sup>
Hafnium (72)	Hf 181		7x10 <sup>-4</sup>
Hydrogen (1)	H 3.	5x10 <sup>-6</sup>	3x10 <sup>-2</sup>
Indium (49)	In 113m		1x10 <sup>-2</sup>
	In 114m		2x10 <sup>-4</sup>
Iodine (53)	I 126	3x10 <sup>-9</sup>	2x10 <sup>-5</sup>
	I 131	3x10 <sup>-9</sup>	2x10 <sup>-5</sup>
	I 132	8x10 <sup>-8</sup>	6x10 <sup>-4</sup>
	I 133	1x10 <sup>-8</sup>	7x10 <sup>-5</sup>
	I 134	2x10 <sup>-7</sup>	1x10 <sup>-3</sup>
Iridium (77)	Ir 190		2x10 <sup>-3</sup>
	Ir 192		4x10 <sup>-4</sup>
	Ir 194		3x10 <sup>-4</sup>
Iron (26)	Fe 55		8x10 <sup>-3</sup>
	Fe 59		6x10 <sup>-4</sup>
Krypton (36)	Kr 85m	1x10 <sup>-6</sup>	
	Kr 85	3x10 <sup>-6</sup>	
Lanthanum (57)	La 140		2x10 <sup>-4</sup>
Lead (82)	Pb 203		4x10 <sup>-3</sup>
Lutetium (71)	Lu 177		1x10 <sup>-3</sup>
Manganese (25)	Mn 52		3x10 <sup>-4</sup>
	Mn 54		1x10 <sup>-3</sup>
	Mn 56		1x10 <sup>-3</sup>
Mercury (80)	Hg 197m		2x10 <sup>-3</sup>
	Hg 197		3x10 <sup>-3</sup>
	Hg 203		2x10 <sup>-4</sup>
Molybdenum (42)	Mo 99		2x10 <sup>-3</sup>
Neodymium (60)	Nd 147		6x10 <sup>-4</sup>
	Nd 149		3x10 <sup>-3</sup>
Nickel (28)	Ni 65		1x10 <sup>-3</sup>
Niobium (Columbium) (41).	Nb 95		1x10 <sup>-3</sup>
	Nb 97		9x10 <sup>-3</sup>
Osmium (76)	Os 185		7x10 <sup>-4</sup>
	Os 191m		3x10 <sup>-2</sup>
	Os 191		2x10 <sup>-3</sup>
	Os 193		6x10 <sup>-4</sup>
Palladium (46)	Pd 103		3x10 <sup>-3</sup>
	Pd 109		9x10 <sup>-4</sup>
Phosphorus (15)	P 32		2x10 <sup>-4</sup>
Platinum (78)	Pt 191		1x10 <sup>-3</sup>

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<b>Element (atomic number)</b>	<b>Isotope</b>	<b>Col. I</b>	<b>Col. II</b>
	Pt 193m		1x10 <sup>-2</sup>
	Pt 197m		1x10 <sup>-2</sup>
	Pt 197		1x10 <sup>-3</sup>
Potassium (19)	K 42		3x10 <sup>-3</sup>
Praseodymium (59)	Pr 142		3x10 <sup>-4</sup>
	Pr 143		5x10 <sup>-4</sup>
	Pm 147		2x10 <sup>-3</sup>
Promethium (61)	Pm 149		4x10 <sup>-4</sup>
Rhenium (75)	Re 183		6x10 <sup>-3</sup>
	Re 186		9x10 <sup>-4</sup>
	Re 188		6x10 <sup>-4</sup>
Rhodium (45)	Rh 103m		1x10 <sup>-1</sup>
	Rh 105		1x10 <sup>-3</sup>
Rubidium (37)	Rb 86		7x10 <sup>-4</sup>
Ruthenium (44)	Ru 97		4x10 <sup>-4</sup>
	Ru 103		8x10 <sup>-4</sup>
	Ru 105		1x10 <sup>-3</sup>
	Ru 106		1x10 <sup>-4</sup>
Samarium (62)	Sm 153		8x10 <sup>-4</sup>
Scandium (21)	Sc 46		4x10 <sup>-4</sup>
	Sc 47		9x10 <sup>-4</sup>
	Sc 48		3x10 <sup>-4</sup>
Selenium (34)	Se 75		3x10 <sup>-3</sup>
Silicon (14)	Si 31		9x10 <sup>-3</sup>
Silver (47)	Ag 105		1x10 <sup>-3</sup>
	Ag 110m		3x10 <sup>-4</sup>
	Ag 111		4x10 <sup>-4</sup>
Sodium (11)	Na 24		2x10 <sup>-3</sup>
Strontium (38)	Sr 85		1x10 <sup>-4</sup>
	Sr 89		1x10 <sup>-4</sup>
	Sr 91		7x10 <sup>-4</sup>
	Sr 92		7x10 <sup>-4</sup>
Sulfur (16)	S 35	9x10 <sup>-8</sup>	6x10 <sup>-4</sup>
Tantalum (73)	Ta 182		4x10 <sup>-4</sup>
Technetium (43)	Tc 96m		1x10 <sup>-1</sup>
	Tc 96		1x10 <sup>-3</sup>
Tellurium (52)	Te 125m		2x10 <sup>-3</sup>
	Te 127m		6x10 <sup>-4</sup>
	Te 127		3x10 <sup>-3</sup>
	Te 129m		3x10 <sup>-4</sup>
	Te 131m		6x10 <sup>-4</sup>
	Te 132		3x10 <sup>-4</sup>
Terbium (65)	Tb 160		4x10 <sup>-4</sup>
Thallium (81)	Tl 200		4x10 <sup>-3</sup>
	Tl 201		3x10 <sup>-3</sup>
	Tl 202		1x10 <sup>-3</sup>
	Tl 204		1x10 <sup>-3</sup>
Thulium (69)	Tm 170		5x10 <sup>-4</sup>
	Tm 171		5x10 <sup>-3</sup>
Tin (50)	Sn 113		9x10 <sup>-4</sup>
	Sn 125		2x10 <sup>-4</sup>
Tungsten (Wolfram) (74)	W 181		4x10 <sup>-3</sup>
	W 187		7x10 <sup>-4</sup>

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Vanadium (23)	V 48		3x10 <sup>-4</sup>
Xenon (54)	Xe 131m	4x10 <sup>-6</sup>	
	Xe 133	3x10 <sup>-6</sup>	
	Xe 135	1x10 <sup>-6</sup>	
<b>Element (atomic number)</b>	<b>Isotope</b>	<b>Col. I</b>	<b>Col. II</b>
Ytterbium (70)	Yb 175		1x10 <sup>-3</sup>
Yttrium (39)	Y 90		2x10 <sup>-4</sup>
	Y 91m		3x10 <sup>-2</sup>
	Y 91		3x10 <sup>-4</sup>
	Y 92		6x10 <sup>-4</sup>
	Y 93		3x10 <sup>-4</sup>
Zinc (30)	Zn 65		1x10 <sup>-3</sup>
	Zn 69m		7x10 <sup>-4</sup>
	Zn 69		2x10 <sup>-2</sup>
Zirconium (40)	Zr 95		6x10 <sup>-4</sup>
	Zr 97		2x10 <sup>-4</sup>
Beta and/or gamma emitting byproduct material not listed above with half-life less than 3 years.		1x10 <sup>-10</sup>	1x10 <sup>-6</sup>

-----  
Footnotes to Schedule A:

{1} Values are given only for those materials normally used as gases.  
{2} æCi/gm for solids.

**Note 1:** Many radioisotopes disintegrate into isotopes which are also radioactive. In expressing the concentrations in Schedule A, the activity stated is that of the parent isotope and takes into account the daughters.

**Note 2:** For purposes of § 30.14 where there is involved a combination of isotopes, the limit for the combination should be derived as follows:

Determine for each isotope in the product the ratio between the concentration present in the product and the exempt concentration established in Schedule A for the specific isotope when not in combination. The sum of such ratios may not exceed "1" (i.e., unity).

Example:

Concentration of Isotope A in Product

-----  
Exempt concentration of Isotope A

Concentration of Isotope B in Product

-----<= 1

Exempt concentration of Isotope B

**Appendix 1-30**



[30 FR 8185, June 26, 1965, as amended at 35 FR 3982, Mar. 3, 1970; 38 FR 29314, Oct. 24, 1973; 59 FR 5520, Feb. 7, 1994]

**§ 31.5 Certain detecting, measuring, gauging, or controlling devices and certain devices for producing light or an ionized atmosphere.<sup>(2)</sup>**

(a) A general license is hereby issued to commercial and industrial firms and research, educational and medical institutions, individuals in the conduct of their business, and Federal, State or local government agencies to acquire, receive, possess, use or transfer, in accordance with the provisions of paragraphs (b), (c) and (d) of this section, byproduct material contained in devices designed and manufactured for the purpose of detecting, measuring, gauging or controlling thickness, density, level, interface location, radiation, leakage, or qualitative or quantitative chemical composition, or for producing light or an ionized atmosphere.

(b)(1) The general license in paragraph (a) of this section applies only to byproduct material contained in devices which have been manufactured or initially transferred and labeled in accordance with the specifications contained in--

(i) A specific license issued under Sec. 32.51 of this chapter; or

(ii) An equivalent specific license issued by an Agreement State.

(2) The devices must have been received from one of the specific licensees described in paragraph (b)(1) of this section or through a transfer made under paragraph (c)(9) of this section.

(c) Any person who acquires, receives, possesses, uses or transfers byproduct material in a device pursuant to the general license in paragraph (a) of this section:

(1) Shall assure that all labels affixed to the device at the time of receipt and bearing a statement that removal of the label is prohibited are maintained thereon and shall comply with all instructions and precautions provided by such labels;

(2) Shall assure that the device is tested for leakage of radioactive material and proper operation of the on-off mechanism and indicator, if any, at no longer than six-month intervals or at such other intervals as are specified in the label; however:

(i) Devices containing only krypton need not be tested for leakage of radioactive material, and

(ii) Devices containing only tritium or not more than 100 microcuries of other beta and/or gamma emitting material or 10 microcuries of alpha emitting material and devices held in storage in the original shipping container prior to initial installation need not be tested for any purpose;

(3) Shall assure that the tests required by paragraph (c)(2) of this section and other testing, installation, servicing, and removal from installation involving the radioactive materials, its shielding or containment, are performed:

(i) In accordance with the instructions provided by the labels; or

(ii) By a person holding a specific license pursuant to parts 30 and 32 of this chapter or from an Agreement State to perform such activities;

(4) Shall maintain records showing compliance with the requirements of paragraphs (c)(2) and (c)(3) of this section. The records must show the results of tests. The records

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also must show the dates of performance of, and the names of persons performing, testing, installing, servicing, and removing from the installation radioactive material and its shielding or containment. The licensee shall retain these records as follows:

- (i) Each record of a test for leakage or radioactive material required by paragraph (c)(2) of this section must be retained for three years after the next required leak test is performed or until the sealed source is transferred or disposed of.
- (ii) Each record of a test of the on-off mechanism and indicator required by paragraph (c)(2) of this section must be retained for three years after the next required test of the on-off mechanism and indicator is performed or until the sealed source is transferred or disposed of.
- (iii) Each record that is required by paragraph (c)(3) of this section must be retained for three years from the date of the recorded event or until the device is transferred or disposed of.

(5) Shall immediately suspend operation of the device if there is a failure of, or damage to, or any indication of a possible failure of or damage to, the shielding of the radioactive material or the on-off mechanism or indicator, or upon the detection of 185 becquerel (0.005 microcurie) or more removable radioactive material. The device may not be operated until it has been repaired by the manufacturer or other person holding a specific license to repair such devices that was issued under parts 30 and 32 of this chapter or by an Agreement State. The device and any radioactive material from the device may only be disposed of by transfer to a person authorized by a specific license to receive the byproduct material in the device or as otherwise approved by the Commission. A report containing a brief description of the event and the remedial action taken; and, in the case of detection of 0.005 microcurie or more removable radioactive material or failure of or damage to a source likely to result in contamination of the premises or the environs, a plan for ensuring that the premises and environs are acceptable for unrestricted use, must be furnished to the Director of Nuclear Material Safety and Safeguards, ATTN: GLTS, U.S. Nuclear Regulatory Commission, Washington, DC 20555-0001 within 30 days. Under these circumstances, the criteria set out in Sec. 20.1402, "Radiological criteria for unrestricted use," may be applicable, as determined by the Commission on a case-by-case basis;

(6) Shall not abandon the device containing byproduct material;

(7) Shall not export the device containing byproduct material except in accordance with part 110 of this chapter;

(8)(i) Shall transfer or dispose of the device containing byproduct material only by export as provided by paragraph (c)(7) of this section, by transfer to another general licensee as authorized in paragraph (c)(9) of this section, or to a person authorized to receive the device by a specific license issued under parts 30 and 32 of this chapter, or part 30 of this chapter that authorizes waste collection, or equivalent regulations of an Agreement State, or as otherwise approved under paragraph (c)(8)(iii) of this section.

(ii) Shall furnish a report to the Director of Nuclear Material Safety and Safeguards, ATTN: GLTS, U.S. Nuclear Regulatory Commission, Washington, DC 20555-0001

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within 30 days after the transfer of a device to a specific licensee or export. The report must contain--

(A) The identification of the device by manufacturer's (or initial transferor's) name, model number, and serial number;

(B) The name, address, and license number of the person receiving the device (license number not applicable if exported); and

(C) The date of the transfer.

(iii) Shall obtain written NRC approval before transferring the device to any other specific licensee not specifically identified in paragraph (c)(8)(i) of this section.

(9) Shall transfer the device to another general licensee only if--

(i) The device remains in use at a particular location. In this case, the transferor shall give the transferee a copy of this section, a copy of Secs. 31.2, 30.51, 20.2201, and 20.2202 of this chapter, and any safety documents identified in the label of the device. Within 30 days of the transfer, the transferor shall report to the Director of Nuclear Material Safety and Safeguards, ATTN: GLTS, U.S. Nuclear Regulatory Commission, Washington, DC 20555-0001--

(A) The manufacturer's (or initial transferor's) name;

(B) The model number and the serial number of the device transferred;

(C) The transferee's name and mailing address for the location of use; and

(D) The name, title, and phone number of the responsible individual identified by the transferee in accordance with paragraph (c)(12) of this section to have knowledge of and authority to take actions to ensure compliance with the appropriate regulations and requirements; or

(ii) The device is held in storage by an intermediate person in the original shipping container at its intended location of use prior to initial use by a general licensee.

(10) Shall comply with the provisions of §§20.2201, and 20.2202 of this chapter for reporting radiation incidents, theft or loss of licensed material, but shall be exempt from the other requirements of parts 19, 20, and 21, of this chapter.

(11) Shall respond to written requests from the Nuclear Regulatory Commission to provide information relating to the general license within 30 calendar days of the date of the request, or other time specified in the request. If the general licensee cannot provide the requested information within the allotted time, it shall, within that same time period, request a longer period to supply the information by submitting a letter to the Director, Office of Nuclear Material Safety and Safeguards, U.S. Nuclear Regulatory Commission, Washington, DC 20555-0001 and provide written justification as to why it cannot comply.

(12) Shall appoint an individual responsible for having knowledge of the appropriate regulations and requirements and the authority for taking required actions to comply with appropriate regulations and requirements. The general licensee, through this individual, shall ensure the day-to-day compliance with appropriate regulations and requirements.

This appointment does not relieve the general licensee of any of its responsibility in this regard.

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(13)(i) Shall register, in accordance with paragraphs (c)(13)(ii) and (iii) of this section, devices containing at least 370 MBq (10 mCi) of cesium-137, 3.7 MBq (0.1 mCi) of strontium-90, 37 MBq (1 mCi) of cobalt-60, or 37 MBq (1 mCi) of americium-241 or any other transuranic (i.e., element with atomic number greater than uranium (92)), based on the activity indicated on the label. Each address for a location of use, as described under paragraph (c)(13)(iii)(D) of this section, represents a separate general licensee and requires a separate registration and fee.

(ii) If in possession of a device meeting the criteria of paragraph (c)(13)(i) of this section, shall register these devices annually with the Commission and shall pay the fee required by Sec. 170.31 of this chapter. Registration must be done by verifying, correcting, and/or adding to the information provided in a request for registration received from the Commission. The registration information must be submitted to the NRC within 30 days of the date of the request for registration or as otherwise indicated in the request. In addition, a general licensee holding devices meeting the criteria of paragraph (c)(13)(i) of this section is subject to the bankruptcy notification requirement in Sec. 30.34(h) of this chapter.

(iii) In registering devices, the general licensee shall furnish the following information and any other information specifically requested by the Commission--

(A) Name and mailing address of the general licensee.

(B) Information about each device: the manufacturer (or initial transferor), model number, serial number, the radioisotope and activity (as indicated on the label).

(C) Name, title, and telephone number of the responsible person designated as a representative of the general licensee under paragraph (c)(12) of this section.

(D) Address or location at which the device(s) are used and/or stored. For portable devices, the address of the primary place of storage.

(E) Certification by the responsible representative of the general licensee that the information concerning the device(s) has been verified through a physical inventory and checking of label information.

(F) Certification by the responsible representative of the general licensee that they are aware of the requirements of the general license.

(iv) Persons generally licensed by an Agreement State with respect to devices meeting the criteria in paragraph (c)(13)(i) of this section are not subject to registration requirements if the devices are used in areas subject to NRC jurisdiction for a period less than 180 days in any calendar year. The Commission will not request registration information from such licensees.

(14) Shall report changes to the mailing address for the location of use (including change in name of general licensee) to the Director of Nuclear Material Safety and Safeguards, ATTN: GLTS, U.S. Nuclear Regulatory Commission, Washington, DC 20555-0001 within 30 days of the effective date of the change. For a portable device, a report of address change is only required for a change in the device's primary place of storage.

(15) May not hold devices that are not in use for longer than 2 years. If devices with shutters are not being used, the shutter must be locked in the closed position. The testing required by paragraph (c)(2) of this section need not be performed during the period of

## *Appendix I-34*

storage only. However, when devices are put back into service or transferred to another person, and have not been tested within the required test interval, they must be tested for leakage before use or transfer and the shutter tested before use. Devices kept in standby for future use are excluded from the two-year time limit if the general licensee performs quarterly physical inventories of these devices while they are in standby.

(d) The general license in paragraph (a) of this section does not authorize the manufacture or import of devices containing byproduct material.

<sup>2</sup> Persons possessing byproduct material in devices under a general license in Sec. 31.5 before January 15, 1975, may continue to possess, use, or transfer that material in accordance with the labeling requirements of Sec. 31.5 in effect on January 14, 1975.

*Appendix I*



# Appendix II Ionization Potential



***Appendix II-2***



For your convenience, we have reproduced ionization potentials for representative compounds in the following categories:

- Some atoms and simple molecules
- Paraffins and cycloparaffins
- Alkyl halides
- Aliphatic alcohols, ethers, thiols, and sulfides

**Some Atoms and Simple Molecules**

	IP(eV)
H	13.595
C	11.264
N	14.54
O	13.614
Si	8.149
S	10.357
F	17.42
Cl	13.01
Br	11.84
I	10.48
H <sub>2</sub>	15.426
N <sub>2</sub>	15.580
O <sub>2</sub>	12.075
CO	14.01
CN	15.13
NO	9.25
CH	11.1
OH	13.18
F <sub>2</sub>	15.7
Cl <sub>2</sub>	11.48
Br <sub>2</sub>	10.55
I <sub>2</sub>	9.28
HF	15.77
HCl	12.74
HBr	11.62
HI	10.38
SO <sub>2</sub>	12.34
CO <sub>2</sub>	13.79
COS	11.18
CS <sub>2</sub>	10.08
N <sub>2</sub> O	12.90
NO <sub>2</sub>	9.78
O <sub>3</sub>	12.80
H <sub>2</sub> O	12.59
H <sub>2</sub> S	10.46
H <sub>2</sub> Se	9.88
H <sub>2</sub> Te	9.14
HCN	13.91
C <sub>2</sub> N <sub>2</sub>	13.8
NH <sub>3</sub>	10.15
CH <sub>3</sub>	9.84
CH <sub>4</sub>	12.98
NH <sub>3</sub>	10.15
CH <sub>3</sub>	9.84
CH <sub>4</sub>	12.98

**Paraffins and Cycloparaffins**

	IP(eV)
methane	12.58
ethane	11.65
propane	11.07
n-butane	10.63
i-butane	10.57
n-pentane	10.35
i-pentane	10.32
2,2-dimethylpropane	10.35
n-hexane	10.18
2-methylpentane	10.12
3-methylpentane	10.08
2,2-dimethylbutane	10.06
2,3-dimethylbutane	10.02
n-heptane	10.08
2,2,4-trimethylpentane	9.86
cyclopropane	10.06
cyclopentane	10.53
cyclohexane	9.88
methylcyclohexane	9.85

**Alkyl Halides**

	IP(eV)
HCl	12.74
Cl <sub>2</sub>	11.48
CH <sub>4</sub>	12.98
methyl chloride	11.28
dichloromethane	11.35
trichloromethane	11.42
ethyl chloride	10.98
1,2-dichloroethane	11.12
1-chloropropane	10.82
2-chloropropane	10.78
1,2-dichloropropane	10.87
1,3-dichloropropane	10.85
1-chlorobutane	10.67
2-chlorobutane	10.65
1-chloro-2-methylpropane	10.66
2-chloro-2-methylpropane	10.61
HBr	11.62
Br <sub>2</sub>	10.55
methyl bromide	10.53
dibromomethane	10.49
tribromomethane	10.51
CH <sub>2</sub> BrCl	10.77
CHBr <sub>2</sub> Cl	10.59

**Alkyl Halides (cont)**

	IP(eV)
ethyl bromide	10.29
1,1-dibromoethane	10.19
1-bromo-2-chloroethane	10.63
1-bromopropane	10.18
2-bromopropane	10.075
1,3-dibromopropane	10.07
1-bromobutane	10.13
2-bromobutane	9.98
1-bromo-2-methylpropane	10.09
2-bromo-2-methylpropane	9.89
1-bromopentane	10.10
HI	10.38
I <sub>2</sub>	9.54
methyl iodide	9.54
diiodomethane	9.34
ethyl iodide	9.33
1-iodopropane	9.26
2-iodopropane	9.17
1-iodobutane	9.21
2-iodobutane	9.09
1-iodo-2-methylpropane	9.18
2-iodo-2-methylpropane	9.02
1-iodopentane	9.19
F <sub>2</sub>	15.7
HF	15.77
CFCl <sub>3</sub> (Freon 11)	11.77
CF <sub>2</sub> Cl <sub>2</sub> (Freon 12)	12.31
CF <sub>3</sub> Cl	12.91
CHClF <sub>2</sub>	12.45
CFBr <sub>3</sub>	10.67
CF <sub>2</sub> Br <sub>2</sub>	11.07
CH <sub>3</sub> CF <sub>2</sub> Cl (Genetron 101)	11.98
CFCl <sub>2</sub> CF <sub>2</sub> Cl	11.99
CF <sub>3</sub> CCl <sub>3</sub> (Freon 113)	11.78
CFHBrCH <sub>2</sub> Br	10.75
CF <sub>2</sub> BrCH <sub>2</sub> Br	10.83
CF <sub>3</sub> CH <sub>2</sub> I	10.00
n-C <sub>3</sub> F <sub>7</sub> I	10.36
n-C <sub>3</sub> F <sub>7</sub> CH <sub>2</sub> Cl	11.84
n-C <sub>3</sub> F <sub>7</sub> CH <sub>2</sub> I	9.96

**Aliphatic Alcohols, and Ethers**

	IP(eV)
H <sub>2</sub> O	12.59
methyl alcohol	10.85
ethyl alcohol	10.48
n-propyl alcohol	10.2
i-propyl alcohol	10.16
n-butyl alcohol	10.04
dimethyl ether	10.00
diethyl ether	9.53
n-propyl ether	9.27
i-propyl ether	9.20

**Aliphatic Thiols and Sulfides**

	IP(eV)
H <sub>2</sub> S	10.46
methanethiol	9.44
ethanethiol	9.285
1-propanethiol	9.195
1-butanethiol	9.14
dimethyl sulfide	8.685
ethyl methyl sulfide	8.55
diethyl sulfide	8.430
di-n-propyl sulfide	8.30

**Aliphatic Aldehydes**

	IP(eV)
CO <sub>2</sub>	13.79
formaldehyde	10.87
acetaldehyde	10.21
propionaldehyde	9.98
n-butyraldehyde	9.86
isobutyraldehyde	9.74
n-valeraldehyde	9.82
isovaleraldehyde	9.71
acrolein	10.10
crotonaldehyde	9.73
benzaldehyde	9.53

**Aliphatic Ketones**

	IP(eV)
acetone	9.69
methyl ethyl ketone	9.53
methyl n-propyl ketone	9.39
methyl i-propyl ketone	9.32
diethyl ketone	9.32
methyl n-butyl ketone	9.34
methyl i-butyl ketone	9.30
3,3-dimethyl butanone	9.17

2-heptanone	9.33
cyclopentanone	9.26
cyclohexanone	9.14
2,3-butanedione	9.23
2,4-pentanedione	8.87

**Aliphatic Acids**

	IP(eV)
CO <sub>2</sub>	13.79
formic acid	11.05
acetic acid	10.37
propionic acid	10.24
n-butyric acid	10.16
isobutyric acid	10.02
n-valeric acid	10.12

**Aliphatic Esters**

	IP(eV)
methyl formate	10.185
ethyl formate	10.61
n-propyl formate	10.54
n-butyl formate	10.50
isobutyl formate	10.46
methyl acetate	10.27
ethyl acetate	10.11
n-propyl acetate	10.04
isopropyl acetate	9.99
n-butyl acetate	10.01
isobutyl acetate	9.97
sec-butyl acetate	9.91
methyl propionate	10.15
ethyl propionate	10.00
methyl n-butyrate	10.07
methyl isobutyrate	9.98

**Aliphatic Amines and Amides**

	IP(eV)
NH <sub>3</sub>	10.15
methyl amine	8.97
ethyl amine	8.86
n-propyl amine	8.78
i-propyl amine	8.72
n-butyl amine	8.71
i-butyl amine	8.70
s-butyl amine	8.70
t-butyl amine	8.64
dimethyl amine	8.24
diethyl amine	8.01
di-n-propyl amine	7.84

di-i-propyl amine	7.73
di-n-utyl amine	7.69
trimethyl amine	7.82
triethyl amine	7.50
tri-n-propyl amine	7.23
formamide	10.25
acetamide	9.77
N-methyl-acetamide	8.90
N,N-dimethyl formamide	9.12
N,N-dimethyl acetamide	8.81
N,N-diethyl formamide	8.89
N,N-diethyl acetamide	8.60

**Other Aliphatic Molecules containing Nitrogen**

	IP(eV)
nitromethane	11.08
nitroethane	10.88
1-nitropropane	10.81
2-nitropropane	10.71
HCN	13.91
acetonitrile	12.22
propionitrile	11.84
n-butyronitrile	11.67
acrylonitrile	10.91
3-butene-nitrile	10.39
ethyl nitrate	11.22
n-propyl nitrate	
methyl thiocyanate	10.065
ethyl thiocyanate	9.89
methyl isothiocyanate	9.25
ethyl isothiocyanate	9.14

**Olefins, Polenes and Acetylenes**

	IP(eV)
ethylene	10.515
propylene	9.73
1-butene	9.58
2-methylpropene	9.23
trans-2-butene	9.13
cis-2-butene	9.13
1-pentene	9.50
2-methyl-1-butene	9.12
3-methyl-1-butene	9.51
3-methyl-2-butene	8.67
1-hexene	9.46
1,1-butadiene	9.07
isoprene	8.845
cyclopentene	9.01
cyclohexene	8.945
4-methylcyclohexene	8.91
4-vinylcyclohexene	8.93
cyclo-octatetraene	7.99
acetylene	11.41
propyne	10.36
1-butyne	10.18

**Some Olefin Derivatives**

	IP(eV)
vinyl chloride	9.995
cis-dichloroethylene	9.65
trans-dichloroethylene	9.66
trichloroethylene	9.45
tetrachloroethylene	9.32
vinyl bromide	9.80
1,2-dibromoethylene	9.45
tribromoethylene	9.27
3-chloropropene	10.04
2,3-dichloropropene	9.82
1-bromopropene	9.30
3-bromopropene	9.70
CF <sub>3</sub> CCl=CCLCF <sub>3</sub>	10.36
n C <sub>5</sub> F <sub>11</sub> CF=CF <sub>2</sub>	10.48
acrolein	10.10
crotonaldehyde	9.73
mesityl oxide	9.08
vinyl methyl ether	8.93
allyl alcohol	9.67
vinyl acetate	9.19

**Heterocyclic Molecules**

	IP(eV)
furan	8.89
2-methyl furan	8.39
2-furaldehyde	9.21
tetrahydrofuran	9.54
dihydropyran	8.34
tetrahydropyran	9.26
thiophene	8.86
2-chlorothiophene	8.68
2-bromothiophene	8.63
pyrrole	8.20
pyridine	9.32
2-picoline	9.02
3-picoline	9.04
4-picoline	9.04
2,3-lutidine	8.85
2,4-lutidine	8.85
2,6-lutidine	8.85

**Aromatic Compounds**

	IP(eV)
benzene	9.245
toluene	8.82
ethyl benzene	8.76
n-propyl benzene	8.72
i-propyl benzene	8.69
n-butyl benzene	8.69
s-butyl benzene	8.68
t-butyl benzene	8.68
o-xylene	8.56
m-xylene	8.56
p-xylene	8.445
mesitylene	8.40
durene	8.025
styrene	8.47
α-methyl styrene	8.35
ethynylbenzene	8.815
naphthalene	8.12
1-methylnaphthalene	7.96
2-methylnaphthalene	7.955
biphenyl	8.27
phenol	8.50
anisole	8.22
phenetole	8.13
benzaldehyde	9.53
acetophenone	9.27
benzenethiol	8.33
phenyl isocyanate	8.77
phenyl isocyanate	8.52
benzonitrile	9.705
nitrobenzene	9.92

aniline	7.70
fluorobenzene	9.195
chlorobenzene	9.07
bromobenzene	8.98
iodobenzene	8.73
o-dichlorobenzene	9.07
m-dichlorobenzene	9.12
p-dichlorobenzene	8.94
1-chloro-2-fluorobenzene	9.155
1-chloro-3-fluorobenzene	9.21
1-chloro-4-fluorobenzene	8.99
o-fluorotoluene	8.915
m-fluorotoluene	8.915
p-fluorotoluene	8.785
o-chlorotoluene	8.83
m-chlorotoluene	8.83
p-chlorotoluene	8.70
o-bromotoluene	8.81
m-bromotoluene	8.81
p-bromotoluene	8.67
o-iodotoluene	8.62
m-iodotoluene	8.61
p-iodotoluene	8.50
benzotrifluoride	9.68
o-fluorophenol	8.66

**Miscellaneous Comps**

	IP(eV)
ethylene oxide	10.565
propylene oxide	10.22
p-dioxane	9.13
dimethoxymethane	10.00
diethoxymethane	9.70
1,1-dimethoxyethane	9.65
propiolactone	9.70
methyl disulfide	8.46
ethyl disulfide	8.27
diethyl sulfite	9.68
thiolacetic acid	10.00
acetyl chloride	11.02
acetyl bromide	10.55
cyclo C <sub>6</sub> H <sub>11</sub> CF <sub>3</sub>	10.46
(n-C <sub>3</sub> F <sub>7</sub> )(CH <sub>3</sub> )C=O	10.58
trichlorovinylsilane	10.79
(C <sub>2</sub> F <sub>5</sub> ) <sub>3</sub> N	11.7
isoprene	9.08
phosgene	11.77

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## WEEE Instructions for PerkinElmer Products



or



A label with a crossed-out wheeled bin symbol and a rectangular bar indicates that the product is covered by the Waste Electrical and Electronic Equipment (WEEE) Directive and is not to be disposed of as unsorted municipal waste. Any products marked with this symbol must be collected separately, according to the regulatory guidelines in your area.

The objectives of this program are to preserve, protect and improve the quality of the environment, protect human health, and utilize natural resources prudently and rationally. Specific treatment of WEEE is indispensable in order to avoid the dispersion of pollutants into the recycled material or waste stream. Such treatment is the most effective means of protecting the customer's environment.

Requirements for waste collection, reuse, recycling, and recovery programs vary by regulatory authority at your location. Contact your local responsible body (e.g., your laboratory manager) or authorized representative for information regarding applicable disposal regulations. Contact PerkinElmer at the web site listed below for information specific to PerkinElmer products.

Web address:

<http://las.perkinelmer.com/OneSource/Environmental-directives.htm>

For Customer Care telephone numbers select "Contact us" on the web page.

Products from other manufacturers may also form a part of your PerkinElmer system. These other producers are directly responsible for the collection and processing of their own waste products under the terms of the WEEE Directive. Please contact these producers directly before discarding any of their products.

Consult the PerkinElmer web site (above) for producer names and web addresses.