CLARUS 400/480 GC

Customer Hardware and Service Guide

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Clarus 400/480 GC Customer Hardware and Service Guide

Release History

Part Number	Release	Publication Date
09936811	C	January 2010

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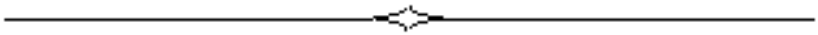

About This Manual

The *Clarus 400/480 GC Hardware and Software Manual* is your complete detailed guide to setting up the Clarus 400/480 GC and integrated autosampler in preparation for running samples.

This manual contains information and procedures for ***all*** of the available injectors and detectors. To benefit the most from this manual, we recommend that you ***read all of the chapters in sequence*** and follow the procedures provided that apply to your specific injectors and detectors as closely as possible. In most cases, reading one chapter is a prerequisite for going on to the next.

For detailed safety information please refer to the *Clarus 400/480 GC Safety and Preparing Your Laboratory Guide (09936813)*.

The manual consists following chapters:

Chapter 1	***Introducing the Clarus 400/480 GC*** provides an introduction to the manual, manual conventions, screen abbreviations, a glossary of Clarus 400/480 GC terms and a glossary of chromatographic terms.
Chapter 2	***System Description*** describes the Clarus 400/480 GC and its major features, including keyboard and display descriptions.
Chapter 3	***Before You Install a Column*** contains general information regarding column installation and the basic procedures you ought to know in order to install a column.
Chapter 4	***Installing a Packed Column*** contains procedures for connecting a packed column to the packed column injector and setting the carrier gas flow using manual pneumatics.
Chapter 5	***Installing a Capillary Column*** contains procedures for connecting a capillary column to a Capillary Injector (CAP). It also describes how to set the gas flows using manual pneumatics.
Chapter 6	***Troubleshooting*** lists the messages that may appear on the screen display, their causes and cures.
Chapter 7	***Maintenance*** includes a variety of routine and preventive maintenance procedures for all injectors and detectors.
Chapter 8	***Practical Hints*** contains useful information on attenuation, filtering detector output, and optimizing FID.
Appendix	A copy of Regulation 10 C.F.R. Section 31.5 of the U.S. Nuclear Regulatory Commission.
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Manual Conventions and Screen Abbreviations

Manual Conventions

Individual keys are displayed in the text by enclosing the name of the key in square brackets. For example, **[Oven Prog]**, **[Enter]**, **[->Set]**, **[Method]**, **[System]**, **[1]**, **[8]**, etc.

All temperatures are in degrees Celsius (□C).

Screen displays are presented throughout the text as a double-lined box:

```
Method 1          READY
                    75°
```

Screen Abbreviations

Autosamp – autosampler

AUX – Auxiliary zone

Cap – capillary split/splitless injector

Cmptr – computer

Ctrl – syringe control parameters

ECD – Electron Capture Detector

Equil – equilibration

Extrn – external

FID – Flame Ionization Detector

Gen – generate

GSV – gas sampling valve

Inj – injector

Inj/Vial – injections per vial

Int – integrator

kPa – kilopascals

NPD – Nitrogen Phosphorus Detector

OnCol – on column

Ovn – oven

Paus – pause

Pkd – packed injector

Pre – # of preinjection syringe washes

Pres – pressure

Prg – autosampler program

Pri – priority sample vial

Psi or psig – pounds per square inch (gauge)

Rec – recorder

Resm – resume

Stpwtch – stopwatch

TCD – Thermal Conductivity Detector

Glossary of Clarus 400/480 GC Terms

The glossary of Clarus 400/480 GC terms are divided into two types:

- Autosampler Terms
- Instrument-Specific Terms

Autosampler Terms

Term	Description
Washes	Washing the syringe.
Pre	The number of prewashes of sample to prime the syringe (no pumping).
Post	The number of post injection syringe washes with a solvent.
Pumps	The number of times the syringe draws up sample and evacuates it before acquiring the volume. This is done to eliminate bubbles.
Mode	The style of injection.
Fast	Fast speed of the syringe during sample injection. This is used to eliminate discrimination in the needle.
Normal	Normal speed of the syringe during sample injection.
Slow	Slow speed of the syringe during sample injection in order to inject directly into a wide-bore capillary column, in hot injection port.
Solv	The number of solvent washes performed before the preinjection sample washes.
Visc	The number of seconds the plunger pauses when drawing up a viscous sample into the syringe.

Instrument-Specific Terms

Term	Description
Background compensation	A routine that automatically subtracts a stored calibrated baseline profile from the signal generated during a GC run.
Ballistically	Changing the oven temperature as quickly as possible to reach a set point.
Detector background	The detector output signal when no components are being eluted.
Equilibration	The delay time after the method set points have been reached before the system becomes READY.
Isothermal method	A method in which the oven temperature remains constant throughout a GC run.
Method	A collection of parameters that control the GC.
Negative-time event	A timed event that you set to occur before the instrument becomes READY.
Parameter	An independent variable used to specify a condition to be met.
Pre-run	The time after equilibration during which negative-time events are executed.
Range	For a Flame Ionization Detector, range means amplification of the detector output signal. For a Thermal Conductivity Detector, range means the bridge current.
Ready	Indicates that all method parameters have reached their set points and that you can start your analysis.
Run	The time from sample injection to the end of the oven temperature program.
Sleep mode	The GC can be set to a predefined method for gas savings.
Timed Events	Events that take place before or during a GC run as specified in a timed events table.
Zone	A heated area in the GC oven, injector, or detector.

Glossary of Chromatographic Terms*

Adsorption – A process that occurs at the surface of a liquid or solid as a result of the attractive forces between the adsorbent and the solute. These forces may be physical or weakly chemical.

Analysis – The complete investigation of a sample by gas chromatographic separation including identification of the sample components and quantitative measurements.

Anode – The negatively charged electrode in any electrical circuit to which charged particles and ions are attracted.

Band Broadening – A process that occurs in the GC whereby the peak width for a component increases the longer the component travels through the column.

Baseline – The detector signal to a recorder or integrator when only the carrier gas is passing through the detector.

Baseline drift – Any regular change occurring in the baseline signal from the detector, usually resulting from column temperature and/or gas flow changes.

Blank run – A run without the sample being injected.

Bleed – The evaporation of the stationary phase from a column.

Capillary column (wall coated open tubular column) – A small-internal-diameter column whose inside wall is coated with a liquid phase.

Carrier gas – The mobile phase of the separation system. An inert gas which transports the sample from the injector through the column to the detector. This gas is usually helium, hydrogen, or nitrogen.

Column conditioning – A process for producing a stable column by heating the column with carrier gas flowing to remove volatile impurities from the stationary phase.

Detectors – Hardware that responds to sample components producing an electrical signal that can be measured to quantitate the amount of each component present.

* Reference: Denney, R.C. *A Dictionary of Chromatography*.

Flow rate – The mass flow of carrier gas or detector gas in milliliters per minute.

Ghost peaks – Peaks that are not due to sample components, for example, peaks produced by carrier gas impurities, septum, or components from previous analyses.

Injection port – The hardware through which the sample is introduced to the column by injection.

Linearity – Quantitatively all detectors will produce a linear response with respect to solute concentration over a defined range, for example, the Linear Range.

Liquid phase – The material in the column that causes the components to separate because of partitioning of the components between the mobile phase (carrier gas) and the stationary phase (liquid phase).

Lowest limit of detection – The smallest amount of sample that can be detected by the detector being used. Usually defined as any signal that is as great as two times the noise level. Also referred to as Minimal Detectable Quantity (MDQ).

Mobile phase – The gas which carries the solute (sample) along and over the column material. This carrier gas is inert and usually helium, nitrogen, or hydrogen.

Noise – Background signal fluctuations arising from a detector response. This response is the result of the column installed, carrier gas purity, electronic components, etc. The response of any detector is defined by the signal-to-noise ratio.

Partition Coefficient – The differential solubility of a substance in two different phases. In the case of gas–liquid chromatography, the sample components reach an equilibrium between the gas phase (mobile) and the liquid phase (stationary). Each component has a different partition coefficient thus causing separation in the column.

Pressure programming – Pressure control through an independent four-step, three-ramp program for each carrier gas channel.

Resolution – The degree of separation between two peaks.

Retention time – The time interval from the point of injection to the appearance of the peak maximum, of a component's signal.

Septum – Silicone rubber material placed in the injection port through which the injection is made. When the needle is withdrawn, the silicone rubber reseals, thus not allowing any sample or carrier gas to escape.

Stationary phase – The liquid or solid adsorbent portion of the column that retains components passing through the GC column.

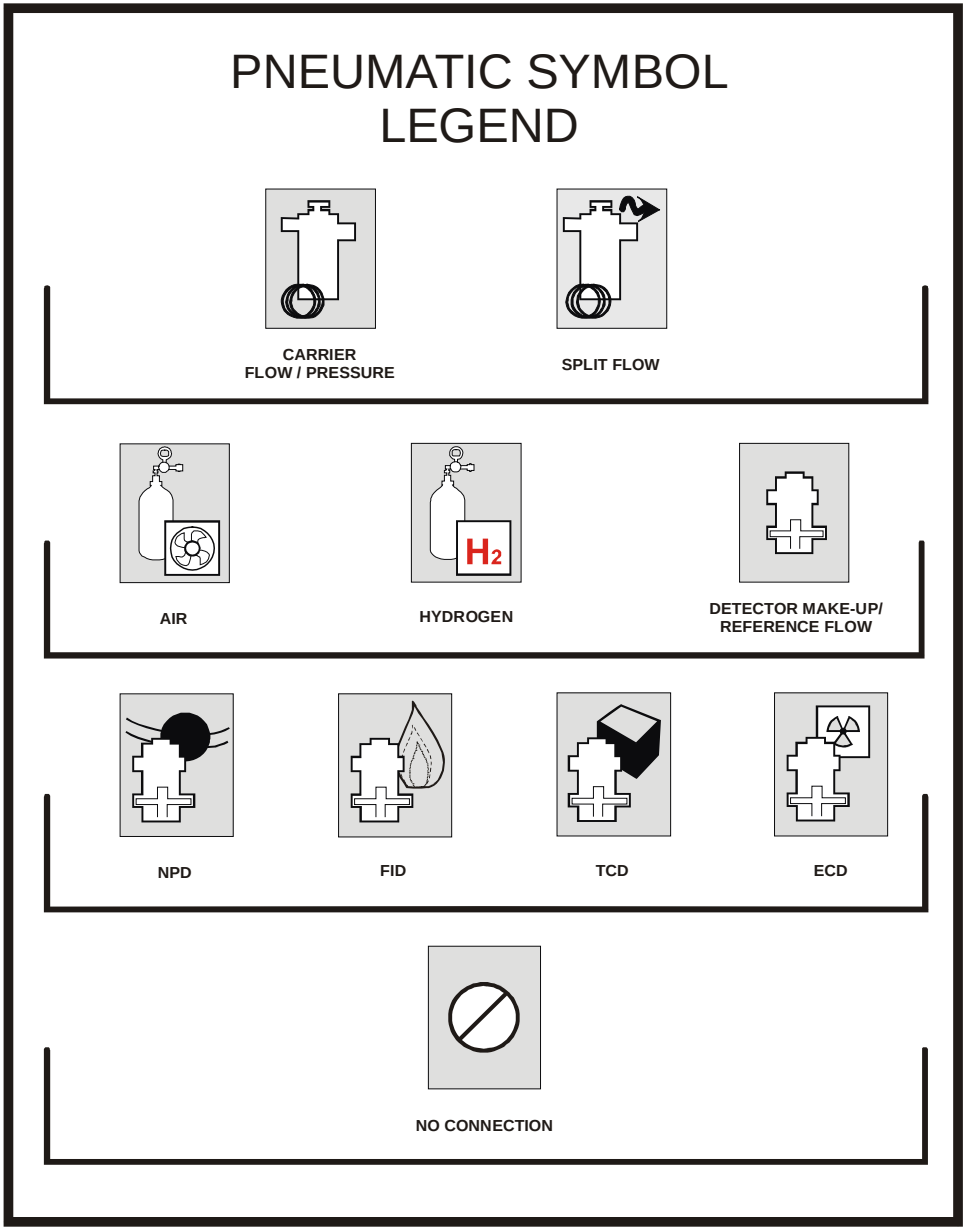
Syringes – Precision dispensing devices used to deliver sample to the GC. Liquid and gas syringes are available.

Tailing – When a peak is not symmetrical or Gaussian shaped but the back end is broadened, it is said to be tailing.

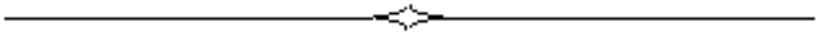
Temperature programming – A technique commonly used to increase the rate of elution of the components. After the sample is injected into the oven at a specific temperature, the temperature program increases the oven temperature to the prescribed temperature at a defined rate (in °C/min).

Unretained peak – A component that is not retained by the column. The time taken for an unretained sample to pass through the column is the same time as the time taken for the carrier gas to pass through.

Symbols Located on the Clarus 400/480 GC



System Description 2



Introduction

The Clarus 400/480 Gas Chromatograph is a 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 400/480 GC is microprocessor controlled, where you enter the operating parameters from the color-coded keyboard and view the prompting text and monitor instrument functions on a large two-line vacuum fluorescence display.

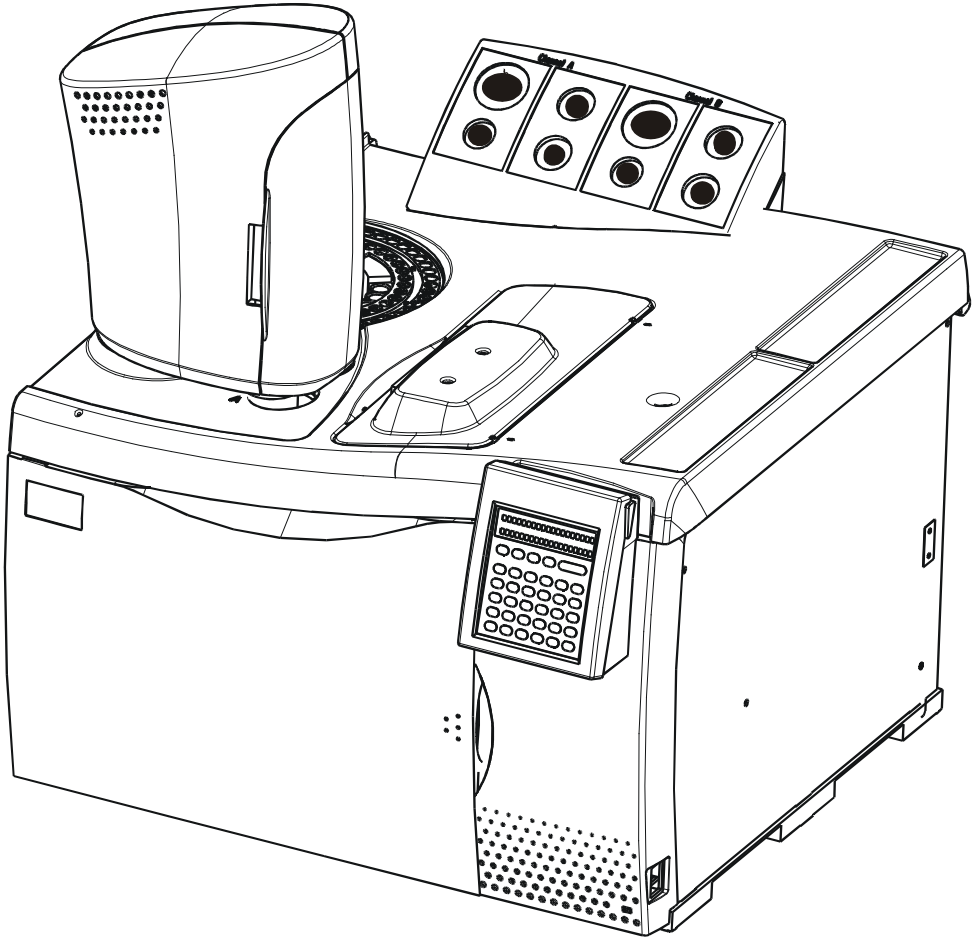


Figure 1. The Clarus 400/480 GC.

Overview of the Clarus 400/480 GC

Your Clarus 400/480 GC may have none, one, or two of the following detectors installed:

- Flame Ionization (FID)
- Electron Capture (ECD)
- Nitrogen Phosphorus (NPD)
- Thermal Conductivity (TCD)

The FID, ECD, TCD, or the NPD, may be installed in either the front or the rear detector position.

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).

Up to two gas sampling valves may be installed.

The Clarus 400/480 is a manual pneumatics instrument.

The carrier gas and detector gas controls are built into the pneumatics control panel on the Clarus 400/480. The carrier gas controls are used to set the flow for packed injectors and the pressure for CAP injectors. The detector gas controls are used to set the hydrogen and air for FID and NPD reference for TCD; and make-up gas for the ECD. Figure 2 is an example of a dual-channel pneumatics control panel with Channel 1 containing a capillary injector and a FID and Channel 2 containing a packed injector and an ECD.

For each channel, the injector-pneumatic controls are on the left and the detector-pneumatic controls are on the right.

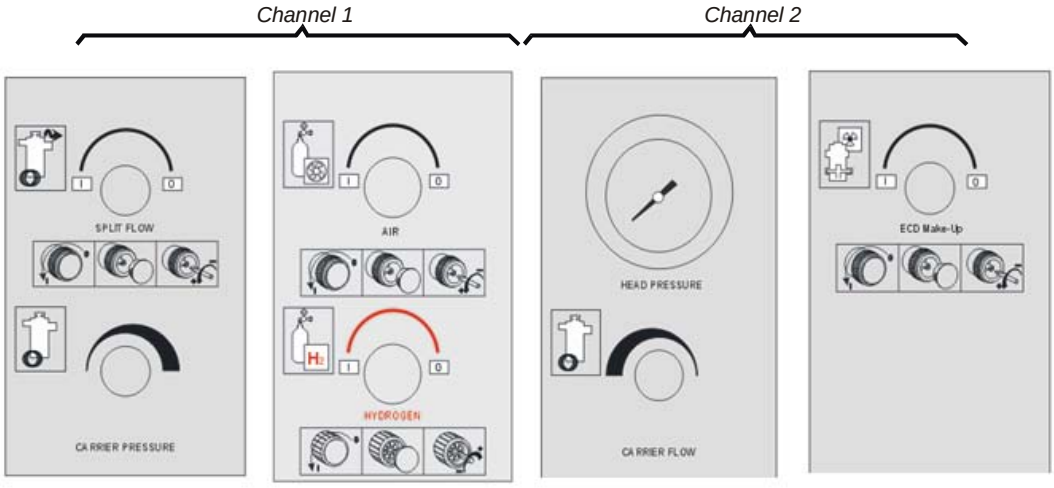


Figure 2. Example of a dual-channel pneumatics control panel in the Clarus 400/480 GC.

Channel 1 designates an injector/detector combination installed in the front position of the instrument, whereas Channel 2 designates a injector/detector combination installed in the rear position.

About the Keyboard

The keyboard is your link to the software. The keyboard has 35 keys divided into the following groups:

- Function keys
- Parameter keys
- Entry keys
- Control keys

NOTE: *As you run this instrument you will see software functions on the display that are **not** supported by the Clarus 400/480 GC. Please ignore these functions and continue with your analysis.*

An audible short beep sounds every time a key is pressed. A long beep sounds when an error has been made. The key groups and their locations are illustrated in Figure 3.

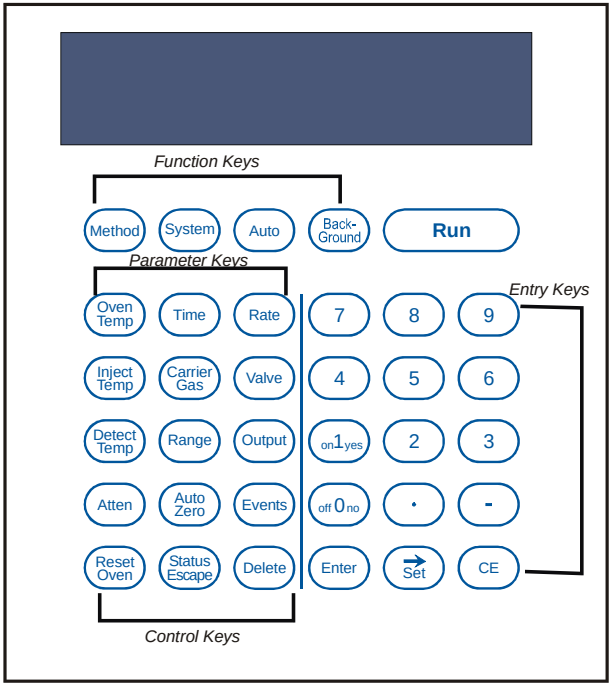


Figure 3. The Clarus 400/480 GC keyboard.

About the Screen

The Clarus 400/480 GC screen is a 2-line by 20-character vacuum fluorescent display.

Figure 4. Vacuum fluorescent screen.

The screen displays status information, error messages, and interactive menus (method, system, configuration, autosampler, and background).

Function Key Descriptions

The four function keys, **[Method]**, **[System]**, **[Auto]**, and **[Background]**, give access to the top-level software menus. The **[System]** menu, in addition to presenting a number of system utility options, provides access to the Configuration Mode.

Pressing a function key displays either a one-page menu or the first page of a two-page menu associated with that key.

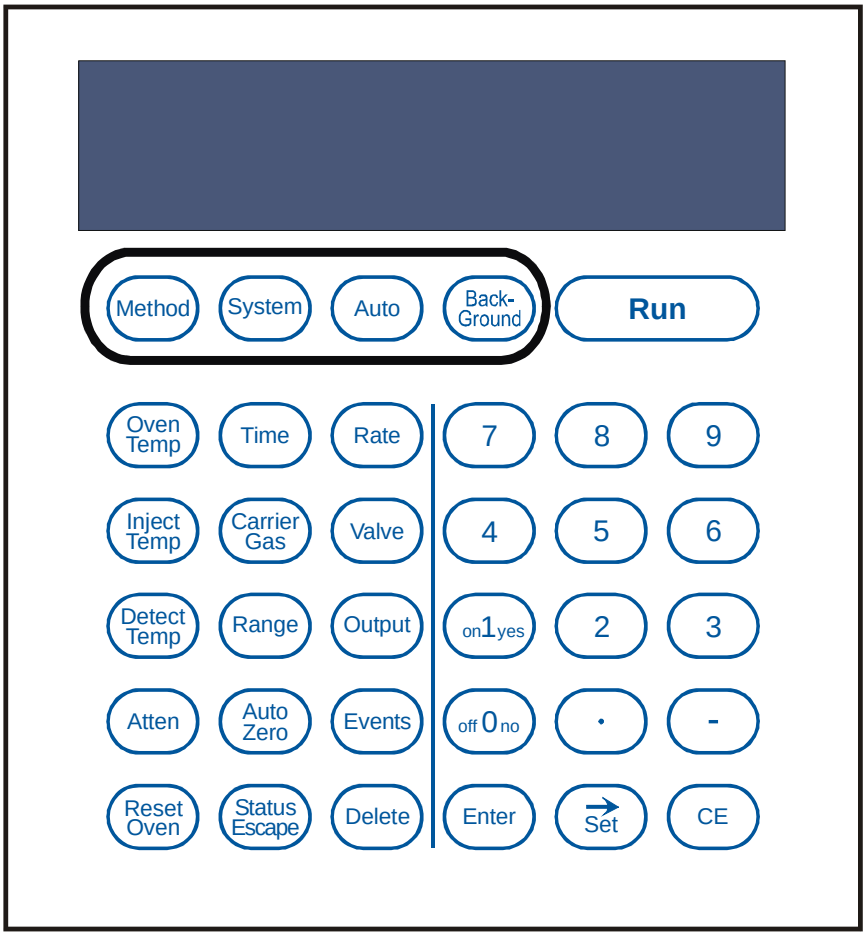
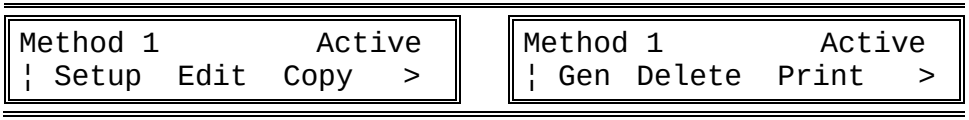


Figure 5. The Function keys.

The Method Function Key

Pressing **[Method]** displays the first page of the two-page Method Menu.



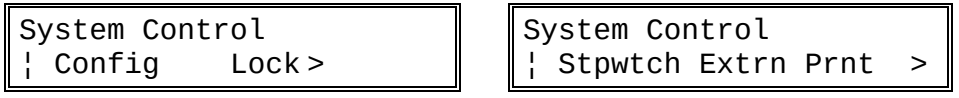
Method Menu, Page 1 **Method Menu, Page 2**

The commands in the Method Menu provide utilities for managing and editing methods. Procedures for using these utilities are given in the Clarus 400/480 Software Guide (0993-6812), *Controlling the Clarus 400/480 GC* chapter. A brief description of these utilities follows.

Command	Description
Setup	Sets up a Stored Method as the Active Method. This option is not available during a GC run or with active automation.
Edit	Allows you to display and edit a Stored Method.
Copy	Copies an existing method to another method number.
Gen	Allows you to generate a new method from the default method.
Delete	Allows you to delete one of the Stored Methods.
Prnt	Prints a method if a printer is attached.

The System Function Key

Pressing **[System]** displays the Page 1 of the two-page **System Control** menu.



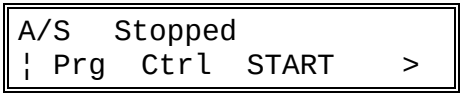
System Control Menu, Page 1 **System Control Menu, Page 2**

The commands in the **System Control** menu provide a number of system utilities. Procedures for using these utilities are given in Clarus 400/480 Software Guide (0993-6812), "*System Utilities.*" A brief description of these utilities follows.

Command	Description
Config	Selecting this option puts the system into the Configuration Mode. The configuration menus allow you to specify configuration details for a variety of hardware options.
Lock	Locks or unlocks the keyboard. For procedural details see Locking and Unlocking the Keyboard in Chapter 13, "System Utilities."
Stpwtch	Accesses the stopwatch function. For procedural details see Chapter 13, "System Utilities."
Extrn	Used to set up an external computer or printer.

The Auto(sampler) Function Key

Pressing **[Auto]** displays the first page of the two-page Autosampler (A/S) menu.



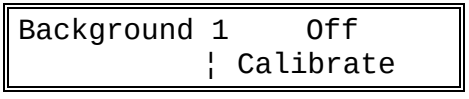
Autosampler Menu, Page 1 **Autosampler Menu, Page 2**

The top line displays the autosampler's status (for example, **Stopped**). The bottom line displays menu options.

Autosampler details are too specialized and extensive to be described in this chapter. All details are provided in Chapter 11, "Controlling the Autosampler."

The Background Function Key

Pressing **[Background]** displays the Background menu on the bottom line and the background status on the top line.



Detailed procedures for using this function are given in Clarus 400/480 Software Guide (0993-6812), "*Background Compensation*."

Control Key Descriptions

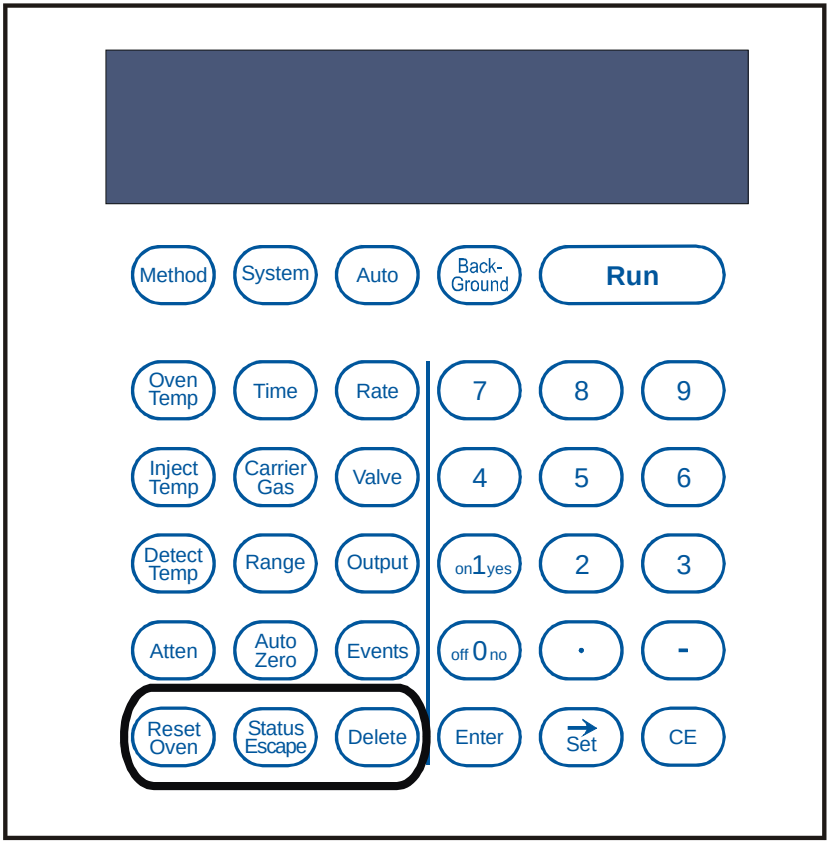


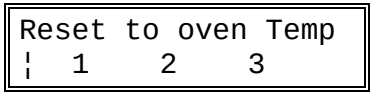
Figure 6. The Control keys.

The Run Key

Press the **[Run]** key to start a GC run after manually injecting a sample, continue to run after an oven hold, or to initiate calibrating a background. Details for using **[RUN]** for the latter purpose are given in Clarus 400/480 Software Guide (09936812), "*Background Compensation.*"

The Reset Oven Key

This key is used to reset the oven temperature during a run.



During the execution of a temperature program, you can elect to heat the oven ballistically to a higher step by selecting the appropriate number from the menu.

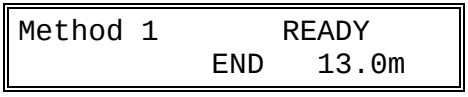
Select **1** to stop a run and reset the instrument to the initial method conditions. Additional details are given in Clarus 400/480 Software Guide (09936812), "*Controlling the Clarus 400/480 GC*."

The Status Escape Key

The **[Status Escape]** key is used to escape from various environments. The top level to which you can escape is the **System Status** screen.

A screen similar to that above appears if you escape from the Method, System, Autosampler, or Background menus.

If you press **[Status Escape]** a second time, the screen displays the Run End Time, as shown below.



In the case of a submenu, escape brings you up to a previous menu level.

The Delete Key

Use this key to delete a timed event or oven temperature program step.

Entry Keys

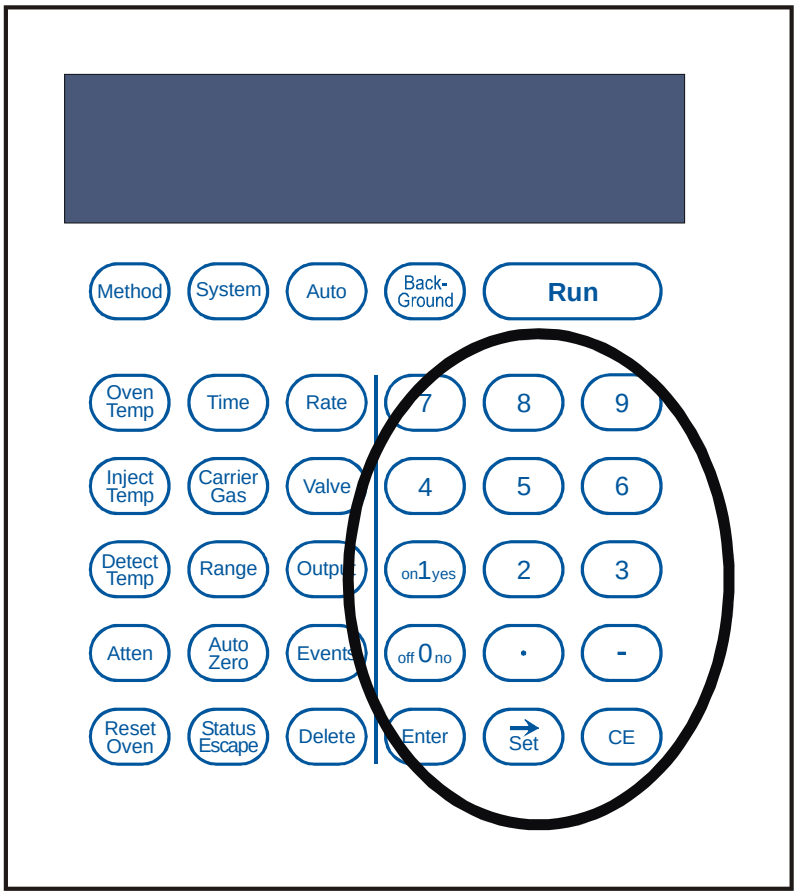


Figure 7. The Entry keys.

All Entry keys, except for the **[->Set]** key, are similar to those on a hand calculator and are used to enter numeric data, clear an entry, etc.

The **[CE]** key (Clear Entry) is used to clear a value before it is entered or to clear certain error messages from the screen.

The **[On/Yes 1]** and **[Off/No 0]** keys are multipurpose keys. In addition to using these keys for entering a numeric 1 or 0, they are used to enter "On" or "Off," "Yes" or "No" in response to questions requiring these answers.

The **[->Set]** key is used to move the screen cursor to a desired screen parameter or menu option for selection. How to use this key for this purpose is described in the next chapter. The**[->Set]** key is also used to activate the Autozero, ignite the FID flame, and actuate connected valves.

Parameter Keys

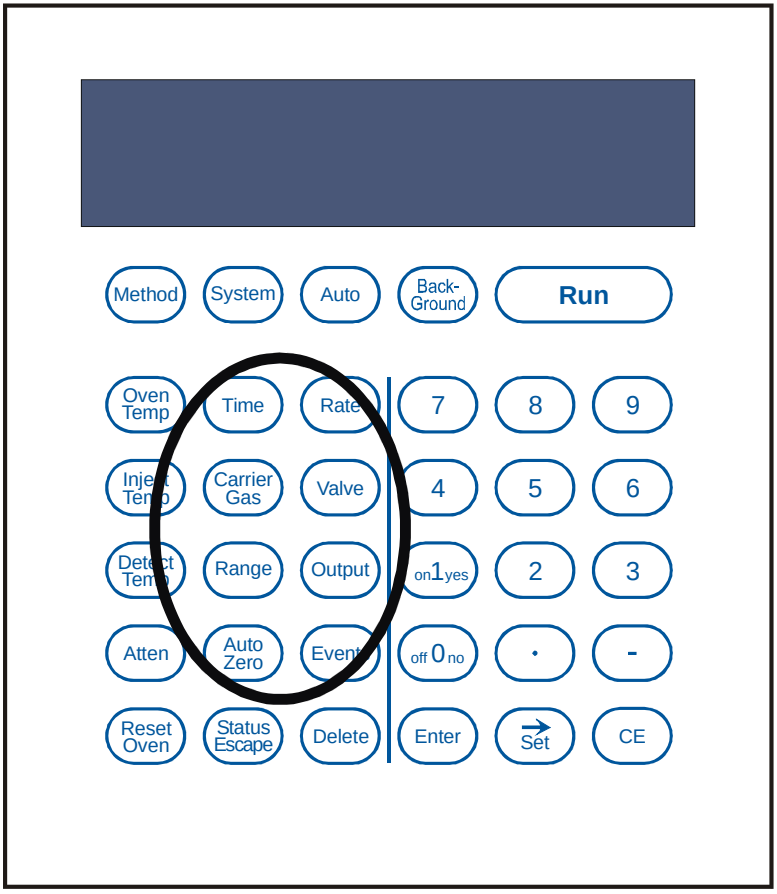
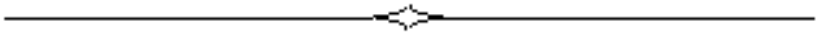


Figure 8. The Parameter keys.

The Parameter keys are used to display operation or configuration parameters. Pressing a Parameter key ***when you not in the Configuration Mode*** displays the operating parameter associated with that key. Operating parameters are described in Clarus 400/480 Software Guide (09936812), "*Controlling the Clarus 400/480 GC* chapter." In the Configuration Mode, pressing a Parameter key displays the configuration of the hardware associated with that key.

Before You Install a Column 3



WARNING

The moment the Clarus 400/480 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.

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

- Protecting your column.
- Turning the oven off and on.
- Turning injector heater(s) off and on.
- Turning detector heater(s) off and on.
- Using the built-in stopwatch.

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 9 shows a capillary injector fitting in the front position and a packed injector fitting in the rear position.

WARNING

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.

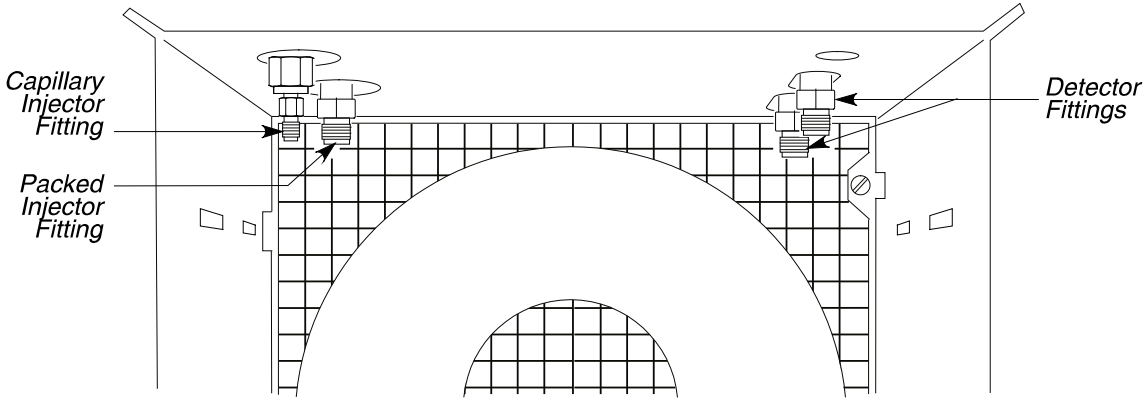


Figure 9. Injector and detector fittings.

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.

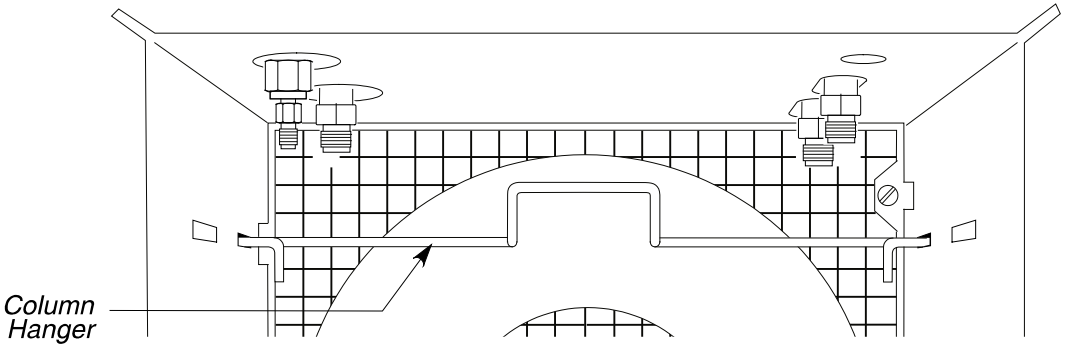


Figure 10. A column hanger installed in the oven rear position.

Protecting Your Column

The Clarus 400/480 GC provides a means for protecting your column(s) from overheating. It does this by not allowing the oven to heat up beyond what we call the Oven Maximum Temperature Limit (OMTL), a value that you set in the Configuration Mode.

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.
The following screen shows an example of an error message:

```
Illegal Oven Temp
Range: xxx ---> yyy
```

Where **xxx** and **yyy** are the permissible minimum and maximum oven temperatures respectively. You enter the OMTL in the Oven Maximum screen which is displayed from the Configuration Mode.

To display the Oven Maximum screen:

1. Press **[System]**.

   The first page of the System Control Menu appears:

```
System Control
¦ Config       Lock  >
```

2. Press **[Enter] [Oven Prog]**.

```
Oven Maximum    Config
Temp Limit       ¦ 450°
```

3. Type in the new OMTL (Oven Maximum Temperature Limit), then press **[Enter]**.

4. Press **[Status Escape]**.

Turning the Oven Off and On

To turn the oven off:

1. Open the oven door.

   The following message appears:

   ```
   OVEN DOOR OPEN
   Press CE to Continue
   ```

   The oven heater turns off.

2. Press **[CE]**.

   A status screen similar to the following appears:

   ```
   Method 1       OVN OFF
   Ovn                 55°
   ```

   Notice that as the oven cools down, the actual temperature is continuously updated on the bottom line. When the oven temperature reaches 40 ºC, the oven fan turns off.

3. Press **[Status Escape]**.

To turn the oven on:

Simply close the oven door.

Turning Injector Heaters Off and On

NOTE: *The examples shown below assume that the Clarus 400/480 GC is in the READY state, position 1 (front) contains a capillary injector, and position 2 (rear) contains a packed injector.*

To turn the injector heaters Off:

1. Press **[Inject Prog]**.

   A screen similar to the following appears:

```
Cap 1              150°
Temperature      ¦ 150°
```

2. Press **[Off/No 0]**.

3. Press **[Enter]**.

   The screen changes to:

```
Cap 1  NOT RDY     149°
Temperature      ¦ Off
```

   Notice that the injector starts to cool.

4. To turn off the second injector heater, display its screen and press **[Inject Prog]** again.

```
Pkd 2              150°
Temperature      ¦ 150°
```

5. Follow steps 2 and 3 above.

6. Press **[Status Escape]**.

To turn the injector heaters on:

1. Display the appropriate Injector Temperature Screen by pressing **[Inject Prog]** once or twice.

2. Enter a temperature set point. For example type: **[1] [5] [0]**, then press **[Enter]**. The screen changes to:

3. Press **[Status Escape]**.

Turning Detector Heaters Off and On

NOTE: *The examples shown below assume that the Clarus 400/480 GC is READY, an FID has been installed in position 1 (front), and an ECD is in position 2 (rear).*

To turn the detector heaters Off:

1. Press **[Detect Control]**.

   A screen similar to the following appears:

```
FID 1                150°
Temperature        ¦ 150°
```

2. Press **[Off/No 0]**.

   The screen changes to:

```
FID 1  NOT RDY       150°
Temperature        ¦   0°
```

Notice that the cursor is blinking, indicating a new value.

3. Press **[Enter]**. The screen changes to:

```
FID 1  NOT RDY      149°
Temperature       ¦ Off
```

   Notice that the detector starts to cool.

4. To turn off the second detector heater, display its screen and press **[Detect Control]** again.

```
ECD 2               150°
Temperature       ¦ 150°
```

5. Follow steps 2 and 3 above.

6. Press **[Status Escape]**.

To turn the detector heaters On:

1. Display the appropriate **Detector Temperature** screen by pressing **[Detect Control]** once or twice.

```
FID 1  NOT RDY       30°
Temperature       ¦ Off
```

2. Enter a temperature set point.

   For example, type: **[1] [5] [0]**, then press **[Enter]**. The screen changes to:

```
FID 1  NOT RDY       40°
Temperature       ¦ 150°
```

3. Press **[Status Escape]**.

Using the Built-in Stopwatch

To measure flows, use the stopwatch function with a soap bubble flowmeter. The following example shows how to measure flows using the built-in stopwatch.

1. Press **[System].**

   The first page of the **System Control** menu appears:

```
System Control
¦ Config       Lock >
```

2. Press **[System]** again.

   The second page of the **System Control** menu appears:

```
System Control
¦ Stpwtch Extrn Print >
```

3. Press **[Enter]**.

   The **Stopwatch** screen appears:

```
Stopwatch          Flow
  0.00 m         Vol ¦ 1
```

4. Enter the volume of the flowmeter you are using.

   For example, change the default to 10 mL by typing **[1] [0]**, then pressing **[Enter]**. The screen changes to:

```
Stopwatch          Flow
  0.00 m        Vol ¦ 10
```

Start the Stopwatch

5. When the bubble reaches the first graduation mark, Press **[Enter]**. This starts the timer.

The elapsed time and calculated flow appear on the screen and are continuously updated.

```
Stopwatch        Flow 20
  0.50 m        Vol ¦ 10
```

Stop the Stopwatch

6. When the bubble reaches the second graduation mark, press **[Enter]**.

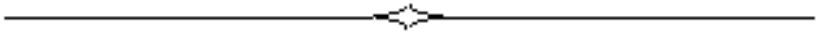
   The system freezes, calculates, and displays the flow in mL/min as of that point in time.

```
Stopwatch       Flow 4.0
  2.50 m        Vol ¦ 10
```

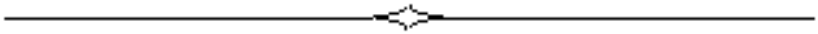
Reset the Stopwatch

7. Press **[CE]**.

8. Press **[Status Escape].**



4 Installing a Packed Column



Packed Column Injector Overview

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 (Part No N6120001) you can convert the injector to accept wide-bore capillary columns.

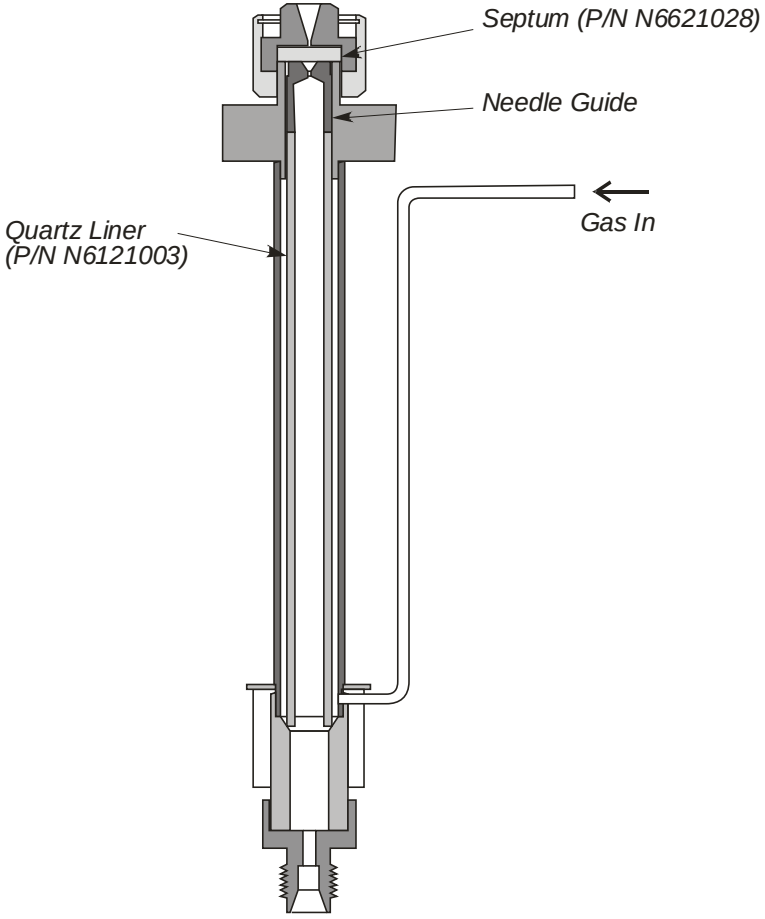


Figure 11. Packed Column Injector.

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 11) and the addition of the adapter fitting (Part No N6100083). 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* (Part No 09938661).

Insert the wide-bore quartz injector liner (Part No N6121003) into the packed column injector with the hourglass portion in the correct position for your desired mode of operation. Figure 11 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 (Part No N610T100), 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.

If you are using the autosampler, install a 0.47-mm O.D. syringe (P/N N6101380) and use the “SLOW" injection mode.

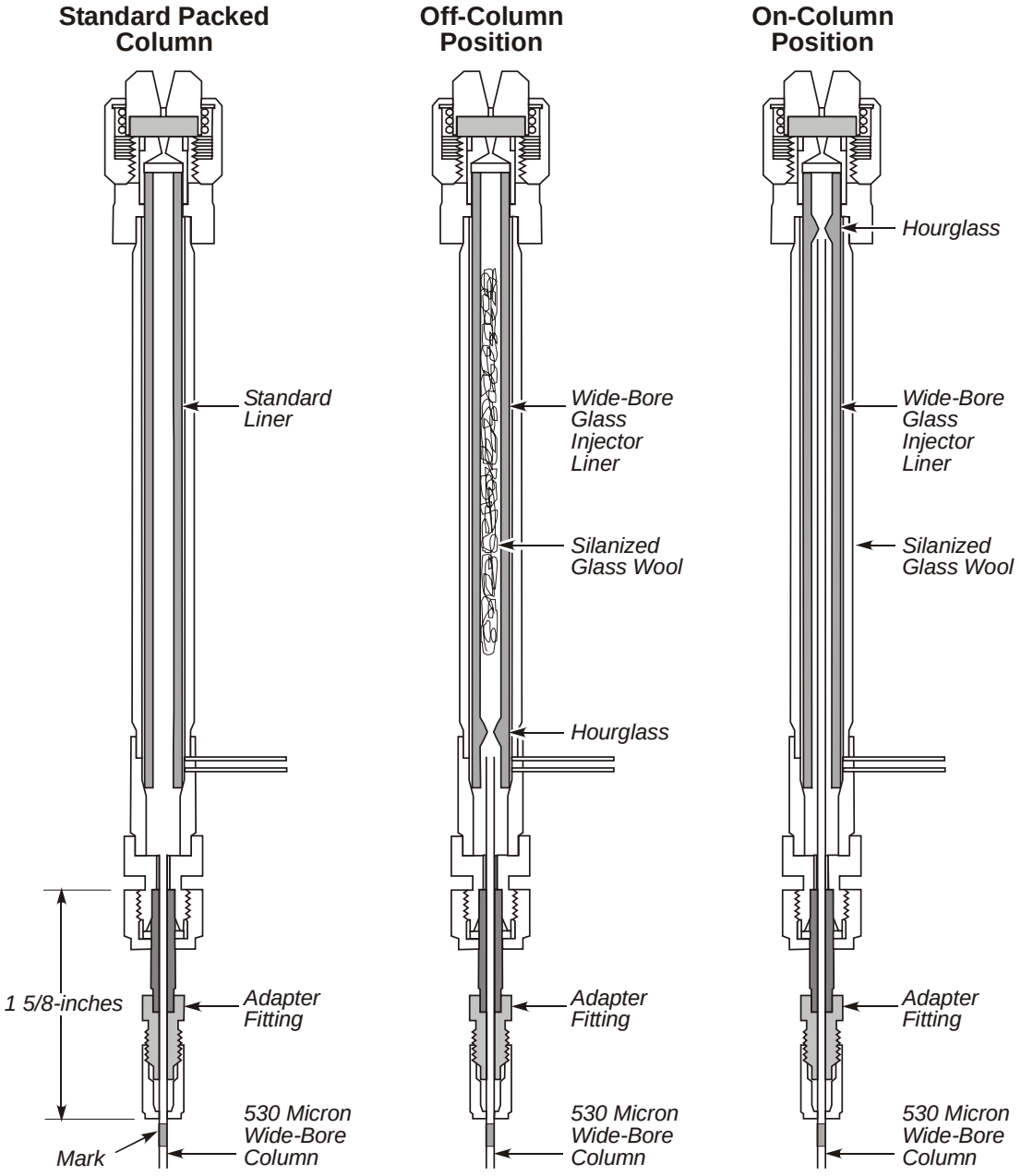


Figure 12. Cross sections of three packed injector configurations with a wide-bore column.

Step 1: Turn off the Heaters

WARNING

The moment the Clarus 400/480 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 400/480 GC Software 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 later in this manual.*

Step 2: Set the Carrier Gas Flow

The following two procedures describe how to set the carrier gas flow for manual pneumatics modules:

- Setting the Carrier Gas Flow Using the Optional Flow Readout.
- Setting the Carrier Gas Flow Using a Soap Bubble or Electronic Flowmeter.

Setting the Carrier Gas Flow Using the Optional Flow Readout

1. Turn on the carrier gas at the tank.
2. Adjust the line pressure to 90 psig (or 620 kPa or 6.2 bar).
3. Press **[Carrier Prog]** until the appropriate screen appears.

```
Flow 1                30
Set          ¦ 30mL/min
```

4. Type the desired flow setpoint value and press **[Enter]**.

5. Adjust the flow by turning the flow control knob (see below) counterclockwise to increase the flow, clockwise to decrease the flow, until the actual flow displayed equals the set point value.

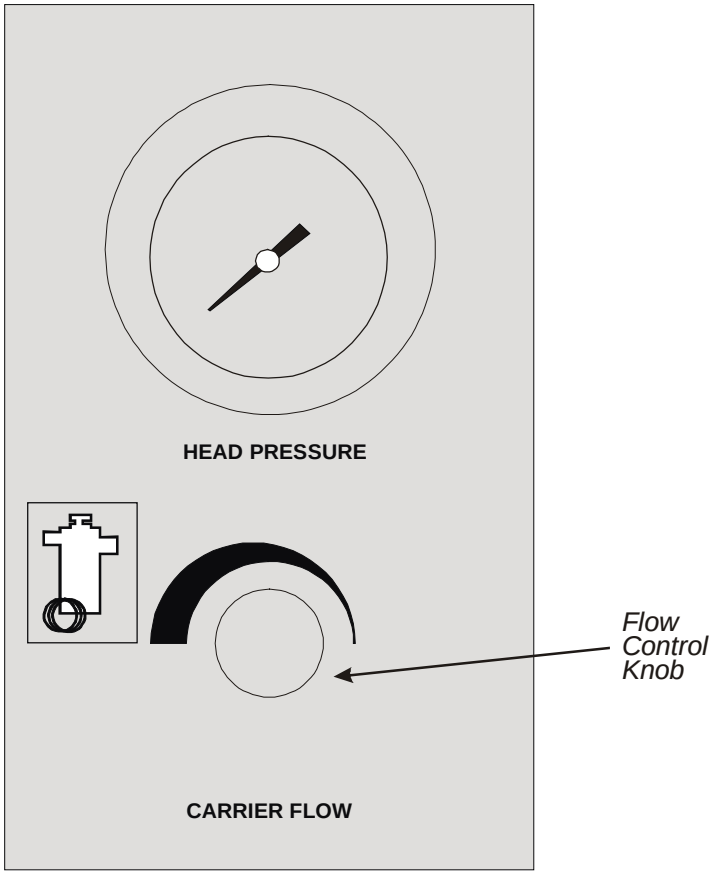


Figure 13. Flow Control Knob.

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 400/480 GC Software Guide (09936812)* before proceeding.

1. Locate the packed injector fitting inside the oven.

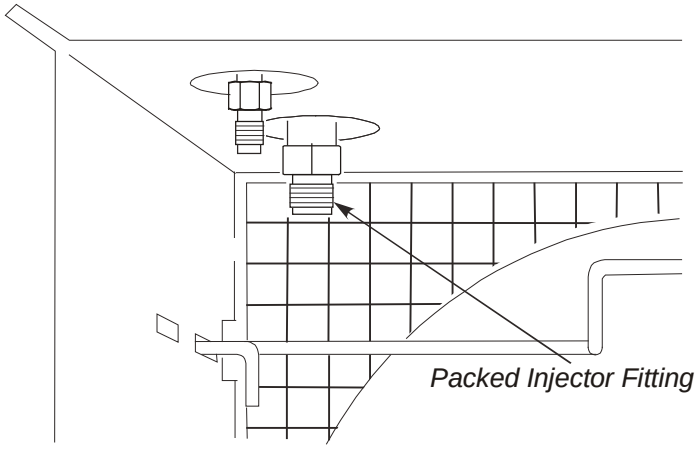


Figure 14. A packed injector fitting.

6. Attach a soap bubble flowmeter to the packed injector fitting.

7. Turn on the carrier gas at the tank and adjust the line pressure to 90 psig.

8. Press **[System] [System] [Enter]** to display the stopwatch screen.

9. Start the carrier gas flowing by turning the flow controller knob counterclockwise.

10. Measure the flow.

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

11. 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.

12. Disconnect the soap bubble flowmeter 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 the following figure).

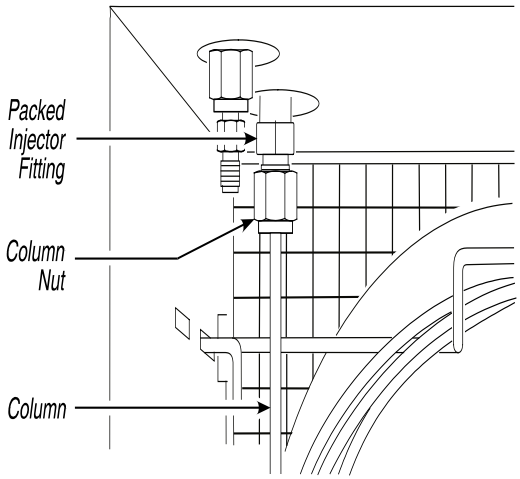


Figure 15. Packed column connected to a packed injector fitting.

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.

CAUTION	*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

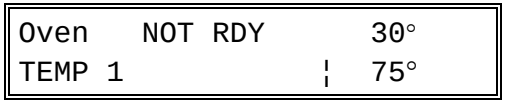
This section contains a suggested temperature program for conditioning a column. The program starts off by holding the oven temperature at a medium value for 10 minutes, gradually increasing the oven temperature at a fixed rate (5 ºC/min) to the column operating temperature, then holding that temperature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

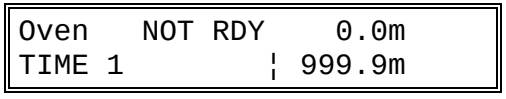
1. Close the oven door, then press **[Oven Prog]**.

   The **Oven Temperature** screen appears.



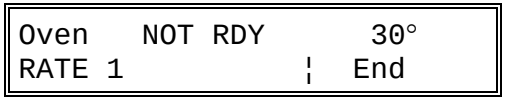
2. Enter an oven temperature set point of 50, then press **[Enter]**.

   The **Oven Time** screen appears:



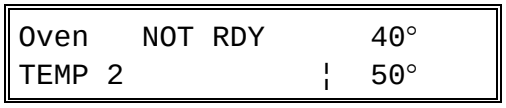
3. Enter a (Hold) TIME of 10, then press **[Enter]**.

   The **Oven Rate** screen appears:



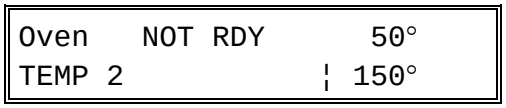
4. To add another program step, enter a RATE of 5 (ºC/min).

A screen similar to the following appears:



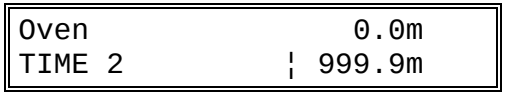
5. For TEMP 2, enter a set point 25 ºC to 50 ºC above your planned analytical operating temperature.

   For example, enter a set point of 150.

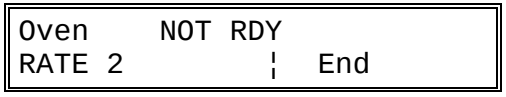


CAUTION	*To avoid damaging the column, do not enter a temperature higher than the maximum operating temperature specified by the column manufacturer.*

6. Press **[Enter]**. The next screen is:



7. Press **[Enter]**. The next screen is:

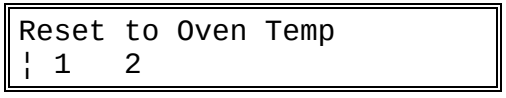


8. Set an Injection Temperature about 50 ºC higher than the TEMP 2 setting.

9. Turn Detector Temperature off. Press **[RUN]** and allow the system to run overnight.

10. The next morning press **[Reset Oven]**.

    A menu similar to the following appears:



11. Press **[Enter]**. This resets the oven temperature set point to that specified for TEMP 1 at the beginning of the temperature program.

12. Open the oven door, then press **[CE]**.

    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.

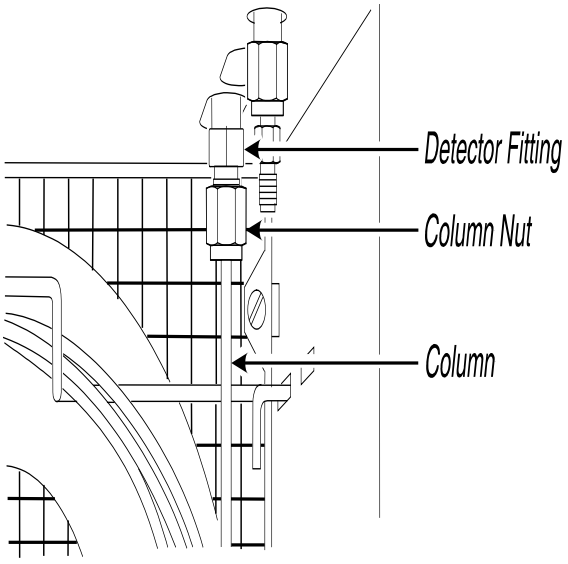


Figure 16. Packed column attached to the rear detector fitting.

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.

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.*

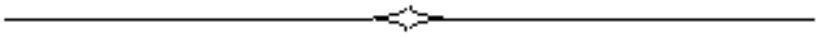
Step 7: Leak Test the Column/Detector Connection

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

With the carrier gas still flowing from the overnight conditioning, test the column/detector connection for leaks using a 50/50 mixture of isopropanol/water or use an electronic leak detector. To prevent contaminating the system, ***DO NOT*** use a soap solution for leak testing. Tighten the connection if a leak is found.

Set up the detector to be used with this column (see *The Clarus 400/480 GC Software Guide*, *Active Method* chapter for information on setting up detectors).

5 Installing A Capillary Column



This chapter describes how to install a capillary column in the Capillary Split/Splitless (CAP)

The information in this chapter is presented as one sequential procedure (Steps **A** through **I**) for all CAP injector with the following procedural steps:

- Setting carrier gas flow using manual pneumatics
- 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	*The CAP injector uses 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 Split/Splitless (CAP) injector.

C. Set the carrier gas to the proper pressure (Set the pressure for the CAP using the optional flow readout or a flowmeter.)

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

- 1/8-inch x 1.0-mm graphite ferrules (P/N 09903394) for 0.53-mm i.d. columns
- 1/16-inch x 0.8-mm graphite ferrules (Part No 09920141)[1] 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 (Part No 09903981)[1] for 0.32/0.25-mm i.d. columns
- One 1/8-inch column nut (Part No 09903453)
- One 1/16-inch graphite ferrule (Part No 09903700)[1] for 0.32/0.25-mm i.d. columns
- One 1/16-inch column nut (Part No 09903392)[1]
- One screwdriver (P/N 0990-7273)[1]
- Deactivated 0.53-mm i.d. fused silica (P/N N6101724)
- Fused-silica universal connector (P/N N9302149)
- Capillary column of your choice
- White-out or felt-tip marker
- Scribe for cutting columns (P/N N9301376)
  (Pointed scribes are ***not*** recommended.)
- Leak-test solution or electronic leak tester

[1] Shipped in the Clarus 400/480 GC Shipping Kit.

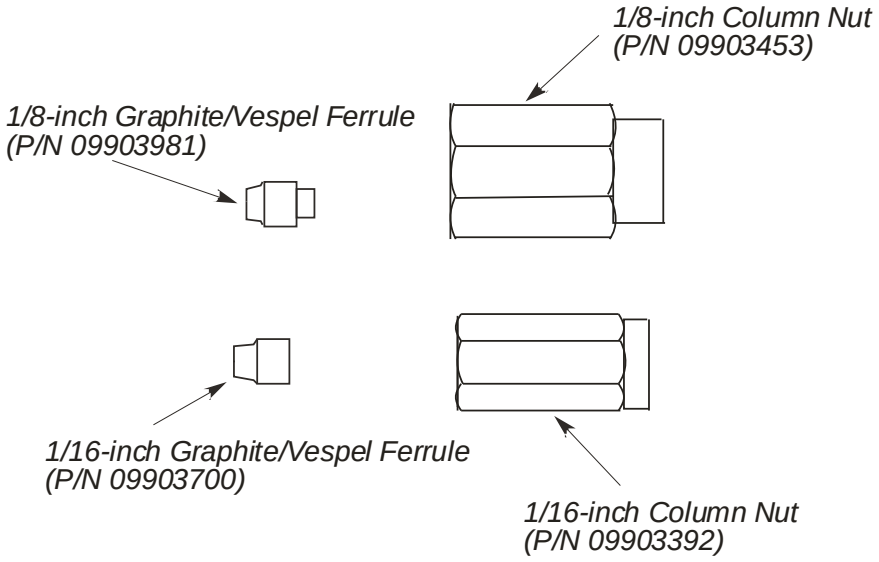


Figure 17. Examples of required fittings.

Step A: Turn the Heaters Off:

CAUTION	*The moment the Clarus 400/480 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 3 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:

This step contains a procedure that describes how to connect a column to Column to the Split/Splitless (CAP) injector:

Step B: 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.

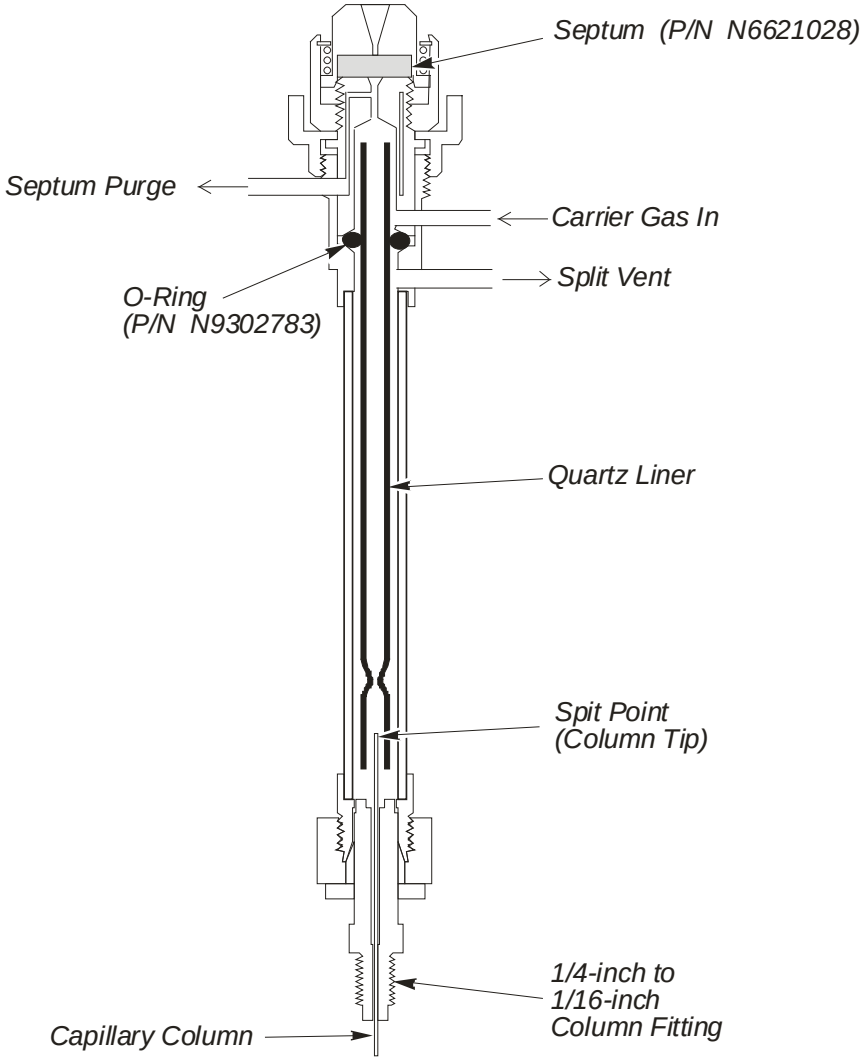


Figure 18. Cutaway view of the Split/Splitless injector (CAP).

About the Injector Liners

The CAP injector uses the following two quartz liners:

- Narrow-bore (2-mm i.d.) liner (P/N N6121002).
- Wide-bore (4-mm i.d.) liner (P/N N6121001).

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 μL. 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 μL 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 μL, 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 μL for hydrocarbon solvents and less than that for high-expansion solvents such as water or CH_2Cl_2. Refer to Table 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 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	333
	2	571
Methanol	1	475
	2	768
Water	1	823
	2	1166

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.

Manual ControlPneumatics

The injector pneumatics consist of a manual pneumatics (flow control valves and pressure regulators) version.

For manual pneumatics, the pneumatics consists of a pressure regulator with an inline pressure transducer for screen readout of the current pressure and a needle valve to control the split vent.

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.

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.

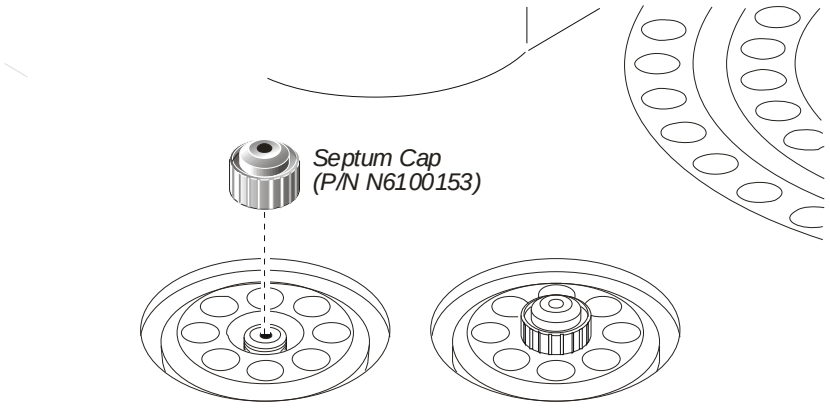


Figure 19. Removing the septum cap.

3. Remove the injector cover.

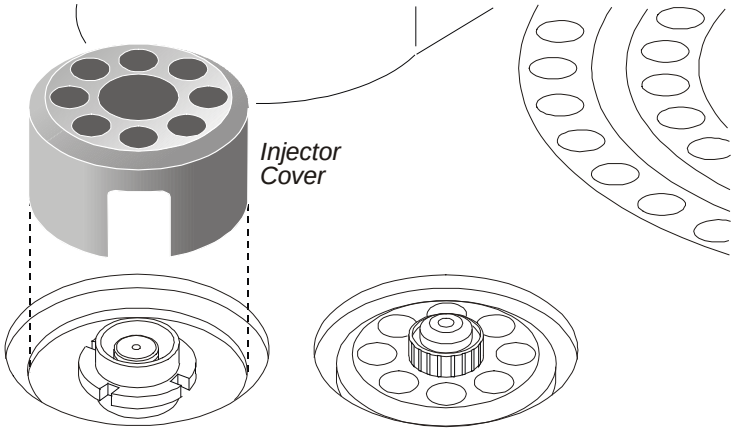


Figure 20. Removing the injector cover.

4. Loosen the threaded collar by using the spanner (P/N N6101359) provided, then remove the threaded collar.

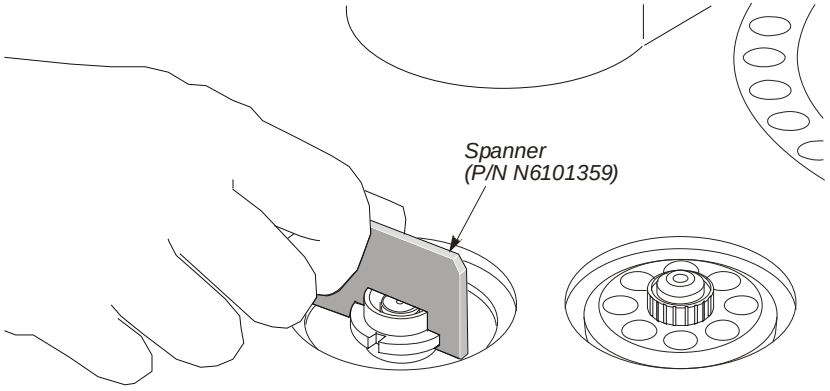


Figure 21. Loosening the threaded collar.

5. Replace the septum cap on the injector.

6. Pull the septum cap upwards to remove the septum purge assembly.

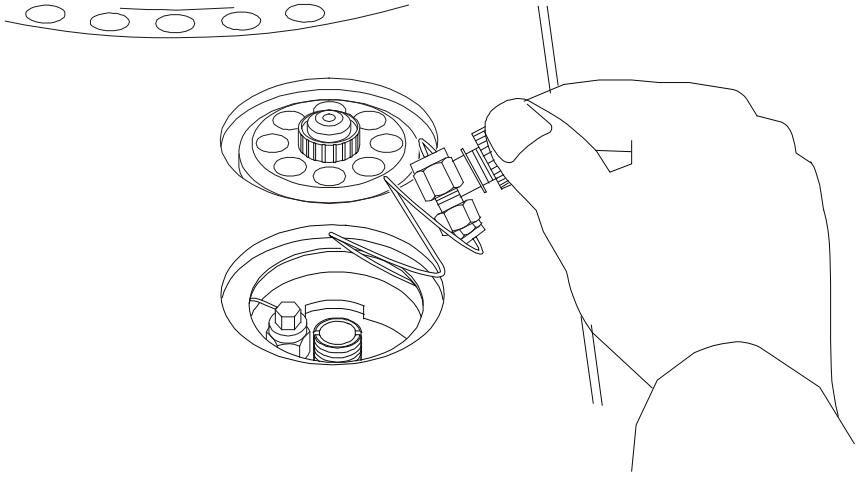


Figure 22. Removing the septum purge assembly.

7. 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.

8. Ensure that the CAP injector liner is cool, then twist the CAP injector liner-removal tool (P/N 02506534, see Figure 24) 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 23. CAP injector liner-removal tool (P/N 02506534).

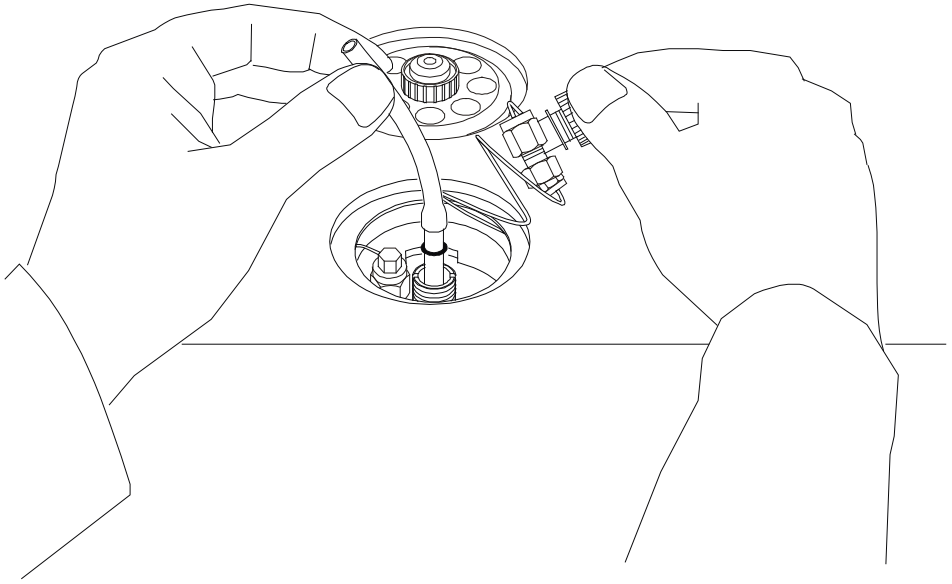


Figure 24. Removing a capillary injector liner.

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 N6121001)
- 2-mm i.d. and 6-mm o.d. CAP injector narrow-bore liner (P/N N6121002)

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, you can 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 limited to a maximum of 2 μL for hydrocarbon solvents and less than that for high-expansion solvents, such as water or CH_2Cl_2. 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 (P/N 6102354) 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 N610T100), push the quartz wool (P/N 6102354) into the liner. Pack the wool tightly[2] 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.

[2] The recovery of high molecular weight components (e.g., C_{40}) may be improved if the liner is loosely packed.

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 N610T100), push the quartz wool (P/N 6102354) 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 27). 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.

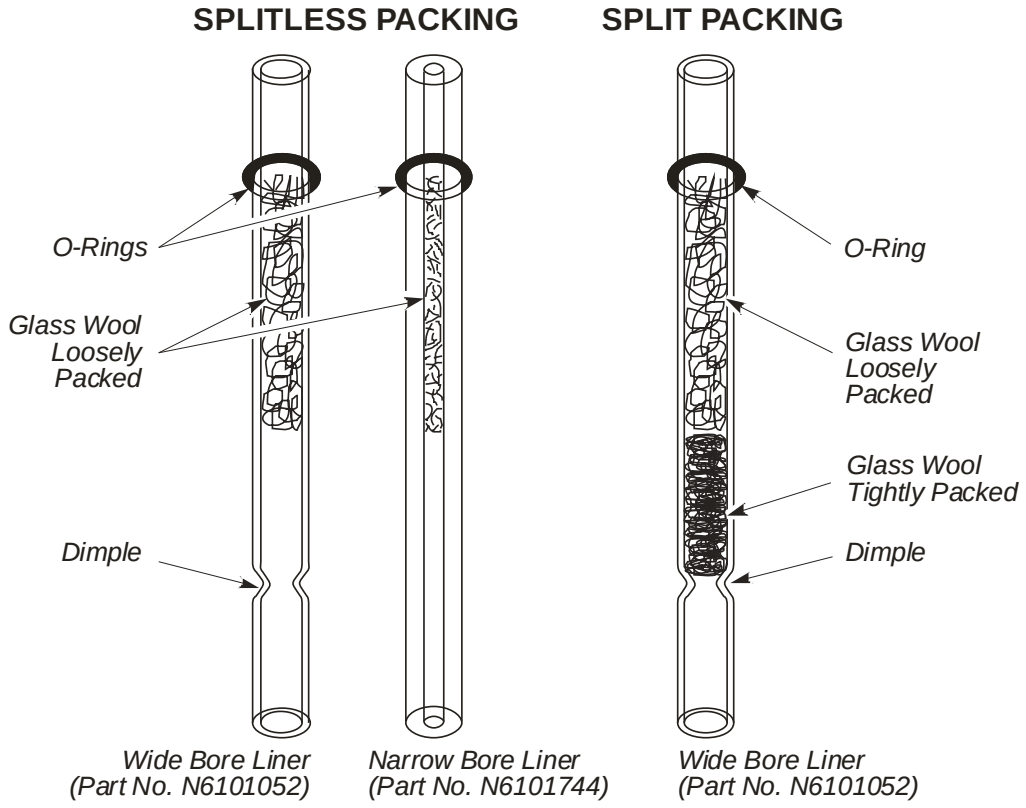


Figure 25. CAP injector liners packed with quartz wool.

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.

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 N6101359).

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 09903392) and 1/16-inch graphite ferrule (0.8 mm i.d., P/N 09920141 or 0.5 mm i.d., P/N 09903700) over one end of the column as shown below:

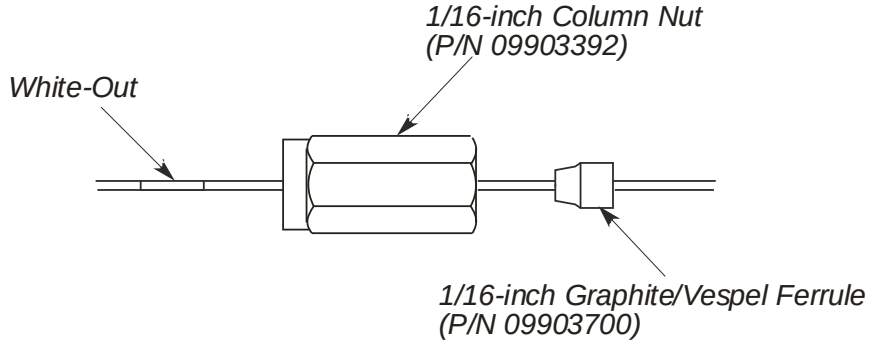


Figure 26. Narrow-bore capillary column, nut, and ferrule on the injector end of a column.

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 N9301376, 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:

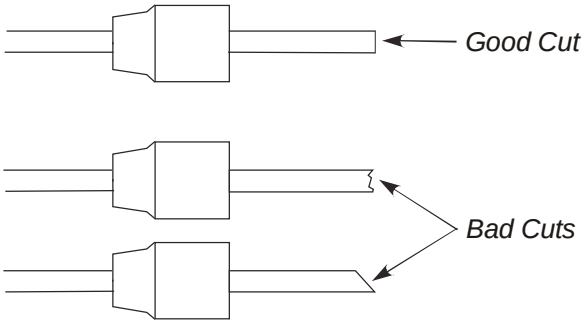


Figure 27. Example of a good cut and bad cuts.

3. Position the column not on the column so that the back of the nut is 4.4 cm to 5.1 (1 ¾ inches to 2 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 26).

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.

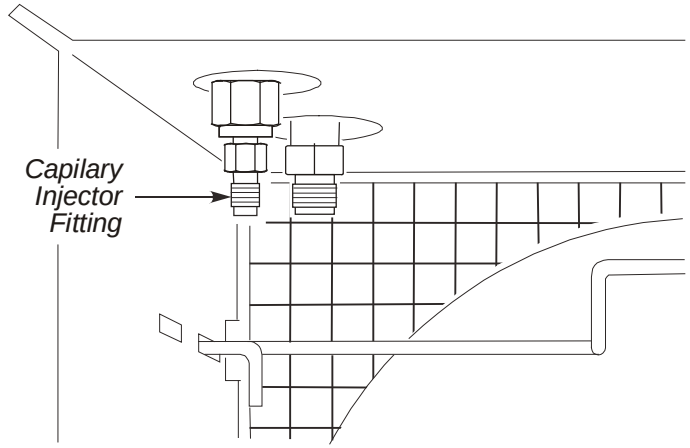


Figure 28. Capillary injector fitting inside the oven.

6. 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.

7. 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.*

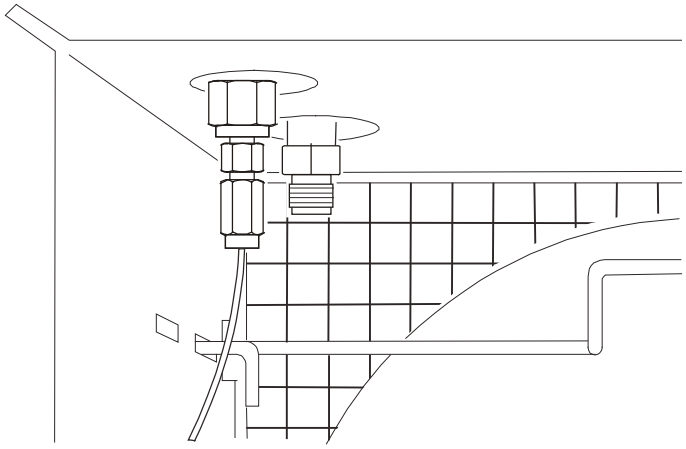


Figure 29. Capillary column attached to capillary injector fitting.

Step C: Set the Carrier Gas Using Manual Pneumatics

This step describes how to set the carrier gas for manual pneumatics modules. Refer to the procedure *Setting the Carrier Gas Using Manual Pneumatics*.

This step includes procedures to set the carrier gas pressure for the split/splitless (CAP).

Setting the Carrier Gas Pressure for the Split/Splitless Injector (CAP)

Carrier gases for the split/splitless injector (CAP) are controlled by adjusting the pressure with the pressure control knob. The location and appearance of the pneumatic controls for a capillary injector are shown in the following figure.

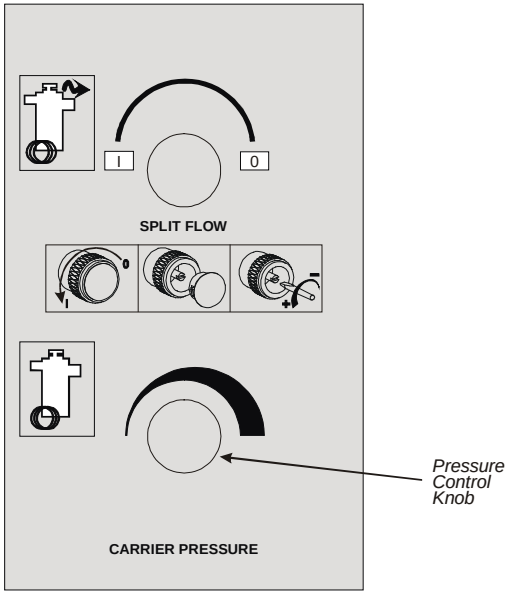


Figure 30. CAP pneumatic controls.

To adjust the carrier gas pressure:

1. Turn on the carrier gas at the tank. Adjust the line pressure to 90 psig (or 620 kPa or 6.2 bar).
2. Press **[Carrier Prog]**.If a capillary injector is in position 1, the Pressure Readout appear.

```
Pres 1          25.0
Set        ¦  25.0 Psi
```

If the capillary injector is in position 2, press **[Carrier Prog]** again.

NOTE: *The Pressure Readout display is factory configured to display the actual pressure as psig. To display the actual pressure as kilopascals (kPa), refer to Clarus 400/480 GC Software Guide (09936812), "Initial Setup Procedures" chapter.*

3. Enter a pressure set point. (See Suggested Capillary Column Pressures below.)

4. Turn the pressure control knob until the readout on the top line equals the set point.

Suggested Capillary Column Pressures:

The following tables are manual pneumatics of the Clarus 400/480 GC.

Table 2. Calculated Pressure Drops (psig) for 10m Column[3]

	Column I.D. (μm)		
$\bar{u}$ [4]	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 3. Calculated Pressure Drops (psig) for 25m Columns[4]

	Column I.D. (μm)		
$\bar{u}$ [5]	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	-

[3] In psig, using helium as a carrier gas at 100 °C.
[4] Average linear velocity (cm/sec).

Table 4. Calculated Pressure Drops (psig) for 50m Columns[4]

	Column I.D. (μm)		
$\bar{u}$ [5]	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	-	-

Step D Leak Test All New Connections:

Manual Pneumatics

Test the connection to the capillary 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 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.

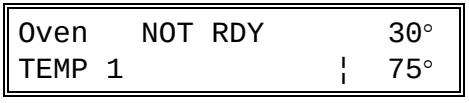
CAUTION	*The temperatures shown in the examples which follow should be used as guidelines. Please refer to the column manufacturer's operating instructions for specific temperature recommendations.*

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

To condition the column:

1. Close the oven door and press **[Oven Prog]**.

   The Oven Temperature screen appears.

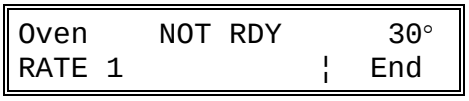


2. Enter an oven temperature set point of 50 ºC and press **[Enter]**.

   The Oven Time screen appears.

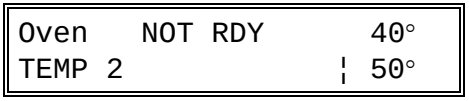
3. Enter a (Hold) TIME of 10 and press **[Enter]**.

The Oven Rate screen appears.



4. To add another program step, enter a RATE of 5(ºC/min).

   A screen similar to the following appears.

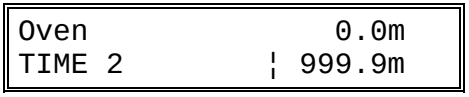


5. For TEMP 2, enter a set point 25 to 50 ºC above your planned analytical operating temperature. For example, enter a set point of 200:

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

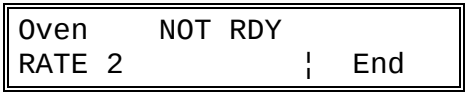
6. Press **[Enter]**.

   The following screen appears:



7. Press **[Enter]**.

   The following screen appears:



8. Configure the injector for the oven mode. Refer to the *Clarus 400/480 GC Software Guide (09936812), Initial Setup Procedures* chapter.

9. Turn the Detector Temperature off.
10. Press **[RUN]**. Allow the system to run overnight.
11. In the morning, press **[Reset Oven]**.
12. A menu similar to the following appears:

```
Reset to Oven Temp
¦  1    2
```

13. Press **[Enter]**.
14. This resets the oven temperature set point to that specified for TEMP 1 at the beginning of the temperature program.
15. Open the oven door, then press **[CE]**.
16. 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:

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:

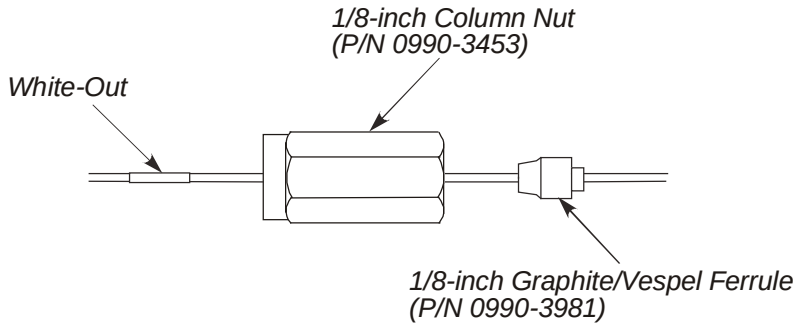


Figure 31. Nut and ferrule on the detector end of a narrow-bore capillary column.

3. Cut about 1 cm (3/8 inch) from the column end using a wafer scribe (P/N N9301376, 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:

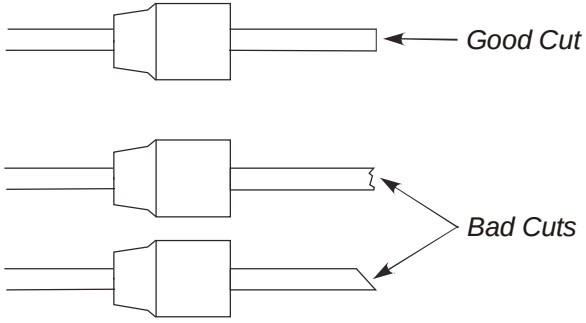


Figure 32. Example of a good cut and bad cuts.

4. Mark the column the following distances from the end using typewriter "white-out" or a felt-tipped pen:

Column Diameter	Detector to which you are attaching the column	Distance from back of nut
≤0.53 mm i.d.	FID	2.75 inches (70 mm)
≤0.53 mm i.d.	ECD[5]	2.5 inches (64 mm)
≤0.53 mm i.d.	TCD	4 inches (103 mm)
≤0.53 mm i.d.	NPD	3 inches (77 mm)

CAUTION

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

5. Locate the detector fitting protruding from the right side of the oven roof.
6. Insert the column into the detector fitting, keeping the mark just behind the column nut.
7. While holding the column in position, hand-tighten the column nut.
8. 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**!

[5] 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.

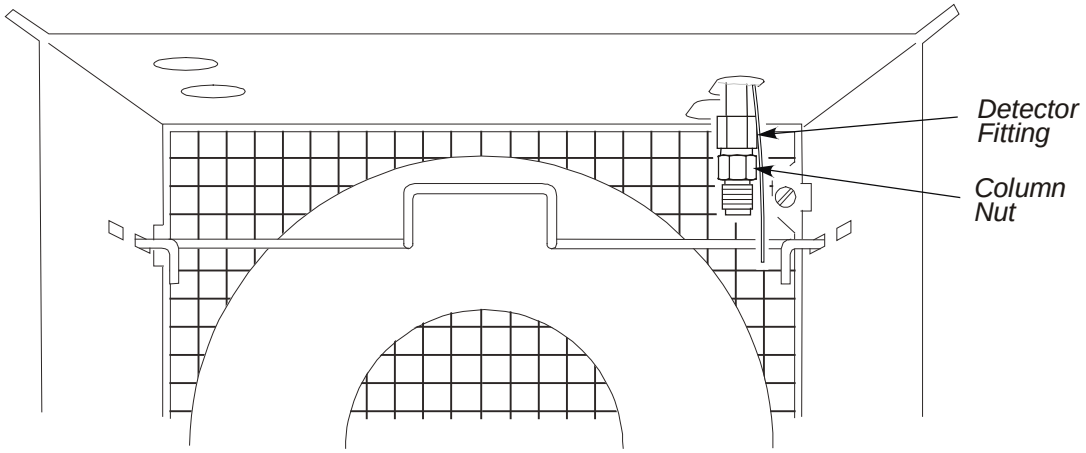


Figure 33. Capillary column connected to the detector fitting.

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 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 manual pneumatics modules. Refer to the procedure that describes your Clarus 400/480 GC manual pneumatics control..

Step H: Setting the Split Mode Using Manual Pneumatics.

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 Manual Pneumatics , Setting up the Splitless Mode for a CAP, Setting the Splitless Mode Using Manual Pneumatics see the *Clarus 400/480 GC Software Guide* (09936625) “Using the Active Method” chapter.

Calculating a Capillary Column Split Ratio

The following procedure and examples show how to calculate the capillary column split ratio in a manual pneumatics version.

Manual Pneumatics Version

1. Calculate the volume of a capillary column:

$$\text{Column Volume} = \frac{(\text{Length of the capillary column in mm})\ (\text{column diameter in mm}/2)^2\ (3.14)}{1000\text{mm}^3/\text{mL}}$$

2. Column flow rate:

$$\text{Column Flow Rate} = \frac{\text{Volume of the column in mL}}{\text{Retention time of methane in min}}$$

3. Calculate the Split Ratio:

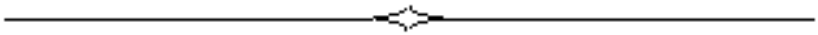
$$\text{Split Rate} = \frac{\text{Flow rate from the split vent in mL/min} + \text{Flow rate of the column}}{\text{Flow rate of the column}}$$

NOTE: *Remember that the split ratio determines how much sample is placed into the capillary column. A larger split ratio means that less sample is placed in the column, therefore less sample is analyzed by the detector.*

In the manual pneumatics version, always note the CAPILLARY HEAD PRESSURE. If the pressure changes, so will the SPLIT RATIO.

In order to reproduce the same chromatographic conditions in the future save the oven temperature program, column head pressure value, and split vent flow rate value.

Troubleshooting 6



This chapter contains the following sections:

- Messages Requiring PerkinElmer Service Assistance
- Background Calibration Error Messages
- Miscellaneous Error Messages
- Illegal Value Error Messages
- GC Troubleshooting

Messages Requiring PerkinElmer Service Assistance

If any of the following messages appear, call your PerkinElmer Service Representative.

A/S control error Elevator not init.	A/S control error Tower not init.
A/S control error Invalid vial	A/S control error Slot not found
A/S control error Invalid syringe size	A/S control error Encoder read error
A/S control error Encoder position err	A/S control error Phase error
A/S control error Bad injection	A/S control error Bad sensor state
A/S control error Bad motor mode	A/S control error Bad motor speed
A/S control error Carousel not init.	A/S control error Bad slot
A/S control error Vial sensor not init.	INSTRUMENT SHUTDOWN xxxxxPRT ERROR
xxxxxERROR PRT NOT FOUND	INSTRUMENT SHUTDOWN xxxxxNO HEAT
INSTRUMENT SHUTDOWN xxxxxTHERM. RUNAWAY	

Background Calibration Error Messages

NOTE: *The following error messages appear before a calibration run starts.*

Message	Cause	Action
B/G CAL ERROR Isothermal Method	You are attempting to perform background calibration using an isothermal method. Background calibration cannot be performed on an isothermal method.	Use a temperature program.
B/G CAL ERROR Negative ramp	Your temperature program has a negative ramp. You cannot calibrate the background when the oven temperature is decreasing.	Check your oven temperature program and remove any negative ramps.
B/G CAL ERROR Run Time < 2.0 m	The run time is less than 2.0 minutes.	Increase the run time to between 2.0 and 999 minutes.
B/G CAL ERROR Run Time > 999 m	The run time is greater than 999 minutes.	Decrease the run time to between 2.0 and 999 minutes.
B/G CAL ERROR HOLD event in Run	Your timed events table contains a HOLD.	Review your timed events table and delete any HOLD events.
B/G CAL ERROR Oven reset	You probably pressed **[Reset]** during a run.	Don't press **[Reset]**.

Miscellaneous Error Messages

Message	Cause	Action
Events table full 32 events maximum	You cannot enter more than 32 events into a timed events table.	Press [CE], then reduce the number of events.
Duplicate event time Enter another time	You entered an event time that already exists. Two events cannot occur simultaneously.	Press [CE], either enter an exclusive new time for this event, or change the time for the duplicate event.
OVEN CONTROL ERROR No Coolant	The oven is trying to cool but cannot. You probably did not turn on the flow of coolant.	Turn on the flow of coolant.
A/S control error Syringe error.	There is no syringe in the autosampler or it is incorrectly installed.	Reinstall syringe.
A/S control error Vial sensor not init	Vial sensor is stuck in the on position when tower is not over a vial.	Check that the vial sensor is moving freely. If the error persists, call your PE rep.
A/S error No vial	In single program mode, no vials in tray. In multiprogram mode vial not found in the range specified in the autosampler program.	Put vials in tray.

A/S error No programs active	In multiprogram mode, all methods specified are Off.	Turn at least one method on.
NO INLET CONFIG Press CE to continue	There are no injectors configured.	Configure at least one injector.
NO DETECTOR CONFIG Press CE to continue	There are no detectors configured.	Configure at least one detector.
Can't select output w/only one detector	You are trying to redirect the detector output with only one detector configured.	Make certain that both detectors are configured.
Filament FIL OVR 100 Temperature 150	TCD filaments exceeded maximum temperature	Make certain that carrier gas is flowing and that the TCD and filament current are within acceptable limits.
A/S Error No w/w vial	A waste or wash vial has been removed.	Replace vial.
Flow too high for 'lo' calibration	When calibrating the flow readout, the measured rate is too high for accurate calibration.	Decrease the flow.
Flow too low for 'hi' calibration	In calibrating the flow readout, the measured rate is too low for accurate calibration.	Increase the flow.

Incorrect keyboard unlock code		Enter the correct code.
Invalid Method # x x x x x	You have entered an invalid Method #.	Enter a valid Method #.
Int. Error XXXXXX Turn Power OFF/ON	Internal Software error.	

Illegal Value Error Messages

The following error messages appear if you enter a value outside of the permissible range. Each screen displays the permissible range for the specific parameter you are entering. To correct the problem, press **[CE]**, then enter a number within the displayed range.

```
Illegal Value
Range 1 - 82
```

```
Illegal Value
Range 1 - 15
```

```
Illegal Value
Range 0.0 - 0.5
```

```
Illegal Value
Range 0.0 - 5.0
```

```
Illegal Value
Range 1 - 3
```

```
Illegal Value
Range x - x
```

GC Troubleshooting

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.

CAUTION	*If the display shows Sleep Mode **do not** activate this mode since this function is only available in the PPC option. The PPC option is not available on the Clarus 400/480 GC. If you inadvertently select this option, reboot the Clarus 400/480 G. Once the instrument is rebooted select the Config screen and switch off the Sleep Mode so the Clarus 400/480 GC will function properly*

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. (See page 116 for part numbers.)
- *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 (P/N N662-1028).
- *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.

1. Note the symptoms - define the problem. Compare your runs with good analyses, that is, with the results normally obtained.

2. 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.

   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

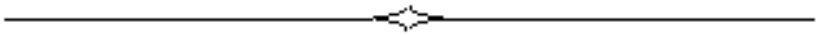
If your GC is a dual-channel system (dual identical detectors and dual identical injectors):

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.

Table 3. READY and START Connections at TB1

Connection	Function
READY OUT TB1-10 (C), 11 (NO), 12 (NC)	*Instrument READY OUT Relay:* These contacts are used to tell an external device that the Clarus 400/480 GC is ready. The normally open contact (NO) is closed in the Ready state.
START OUT TB1-7 (C), 8 (NO), 9 (NC)	*Instrument START OUT Relay:* These contacts are used to start an external device when the Clarus 400/480 GC starts a chromatographic run. The normally open contact is closed for 1 sec when a run is started.
EXT. READY (TB1-5, 6) TB1-6 is signal TB1-5 is ground	*External Ready In:* The Clarus 400/480 GC requires that these contacts be shorted together to become ready and is shipped with a link across them. When using an external device, such as an integrator, remove the link and wire the device to provide a contact closure indicating the Ready state. This will prevent the instrument from becoming ready before the external device is ready.
EXT. START (TB1-3, 4) TB1-4 is signal TB1-3 is ground	*External Start In:* Shorting these contacts will cause the Clarus 400/480 GC to start a chromatographic run. It is equivalent to pressing the RUN key.

Maintenance 7



This chapter contains procedures for:

- Autosampler Maintenance — changing a syringe and replacing a vial-locator mechanism.
- Syringe Maintenance — cleaning the 5-µL and 50-µL syringe plungers and part numbers for syringes.
- Injector Maintenance — changing septa, changing and repacking packed-injector liners, changing and repacking injector liners on the capillary (CAP) 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.
- NPD Maintenance — changing and conditioning the NPD bead and replacing an NPD jet.
- TCD Maintenance

Autosampler Maintenance

Autosampler maintenance consists of changing a syringe and replacing a vial locator mechanism.

Changing a Syringe

1. Press **[Auto] [Auto]**.

```
A/S          STOPPED
¦ Park  Clean  Print >
```

2. Select **Park**, then press **[Enter]**.

   The autosampler tower moves to the park position (facing the front of the Clarus 400/480 GC).

3. Open the tower door on the autosampler tower cover.

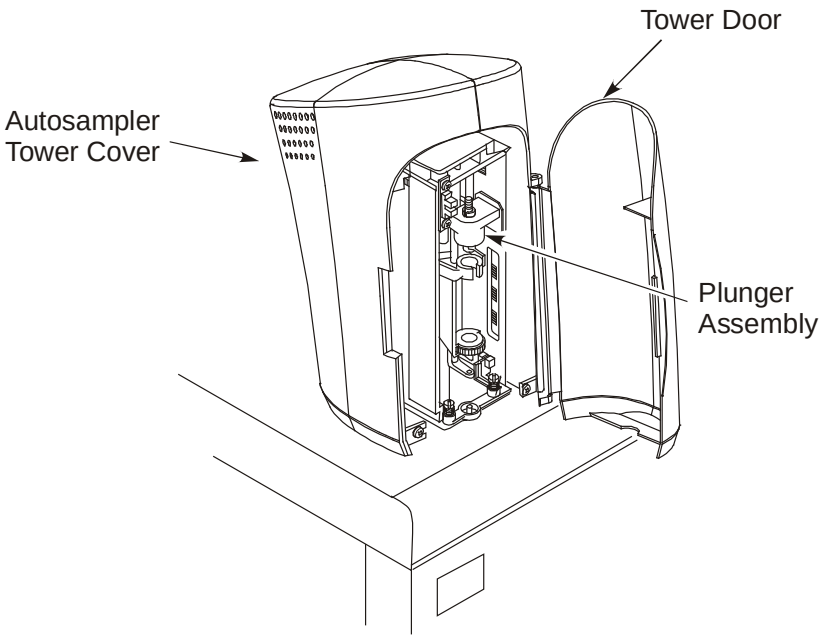


Figure 34. Autosampler tower in the park position.

Removing a Syringe

1. Locate the plunger assembly shown in Figure 34. Then, refer to Figure 35 as you lift up the plunger cap handle and rotate it until it rests on the collar. Then release the plunger cap handle.

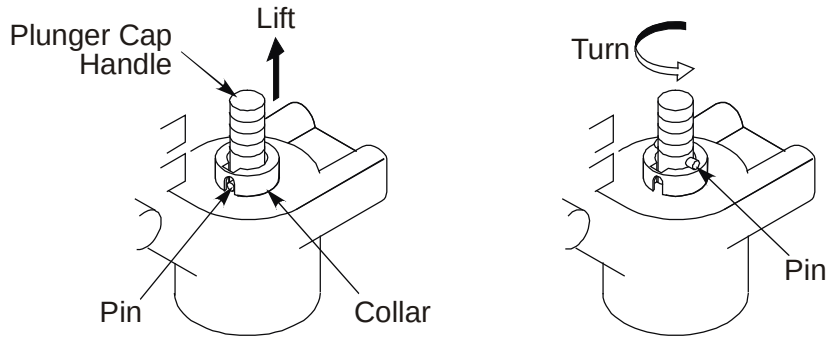


Figure 35. Plunger assembly.

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.

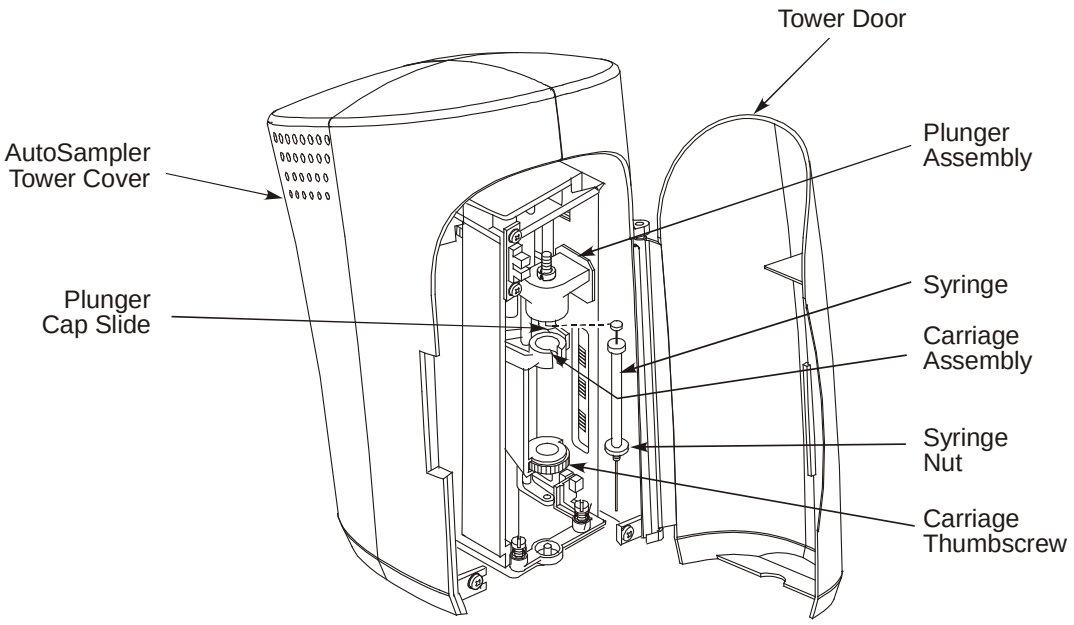


Figure 36. Removing a syringe.

Installing a Syringe

Please refer to Figures 35 through 37 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 needle locator.

5. Tighten the carriage thumbscrew.

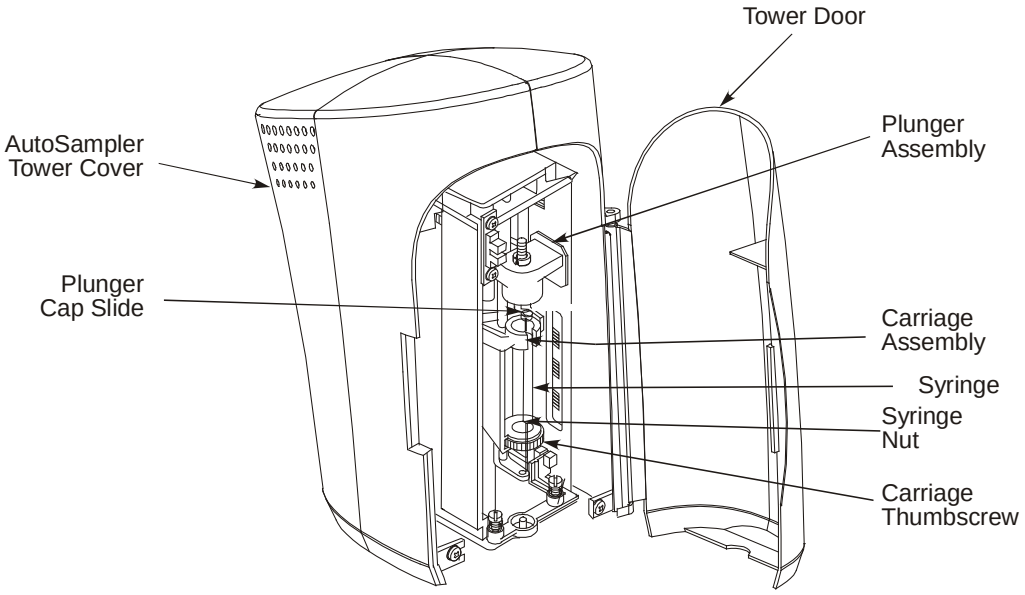


Figure 37. Installing a syringe.

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 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 N6101182) 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.***

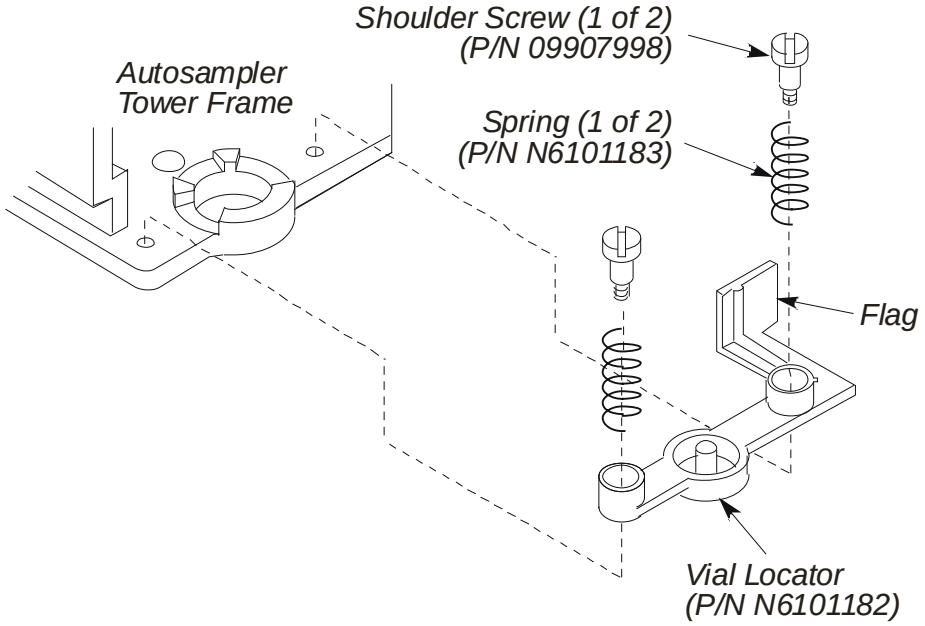


Figure 38. Exploded view of the vial locator.

Cleaning the Autosampler Tray

If you have a spill on the autosampler tray clean with a damp cloth. Do not use any abrasive cleaners on the tray. Remove the tray from the autsampler, clean and return to the autosampler.

Figure 39 Clarus 480 Tray (108 positions) Clarus 400 Tray (82 Positions)

Syringe Maintenance

Syringe maintenance consists of cleaning the 5-µL and 50-µL syringe plungers and servicing idle syringes.

Syringe	Part Number
5-µL (0.63 mm O.D.) Teflon-tipped plunger (Std)	N6101390
0.5- µ L (0.63 mm O.D.)	N6101252
0.5- µ L (0.47 mm O.D.)	N6101253
5- µ L (0.63 mm O.D.)	N6101251
5- µ L (0.47 mm O.D.)	N6101380
50- µ L (0.63 mm O.D.)	N6101760

Cleaning the 5-µL and 50-µL Syringe Plungers

The 5-µL and 50-µL 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 descried in the preceding section.

NOTE: *Only syringes distributed by PerkinElmer should be used with the Clarus 400/480 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 400/480's precision degrading, replace the syringe. The autosampler 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 and repacking CAP liners, changing the charcoal trap or replacing charcoal on the split/splitless CAP 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 N6621028, 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.*

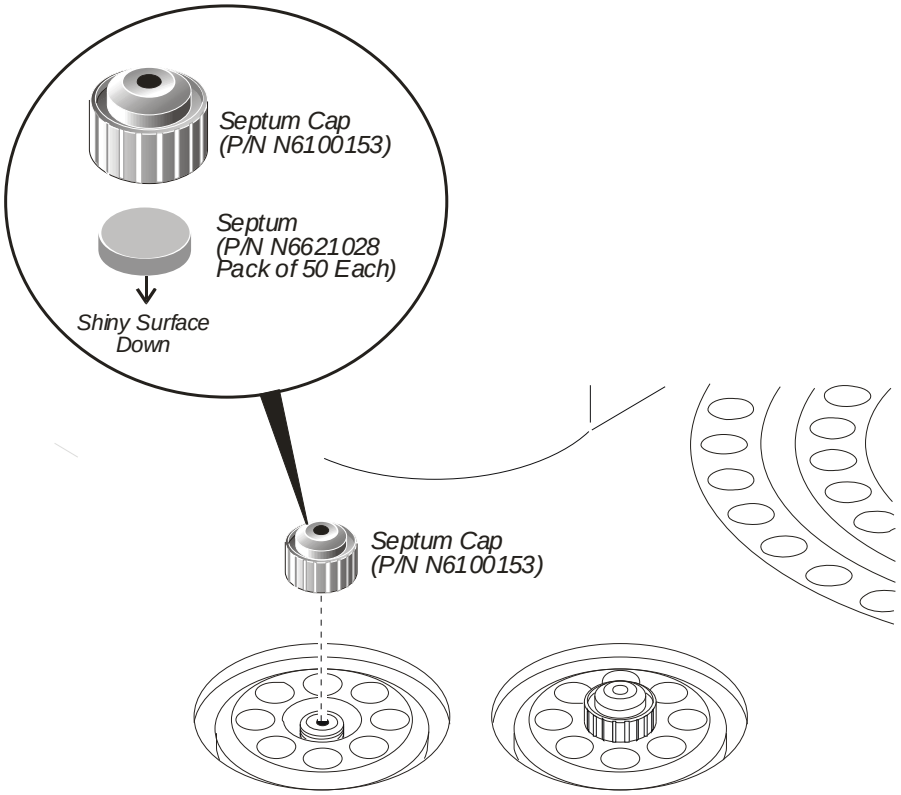


Figure 40. Changing a septum.

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 N6102354) into the top portion of the injector liner (P/N N6101048). 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.

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 39).

3. Remove the septum shield (P/N N6101050) with the large end of the liner-removal tool (P/N N6100102).

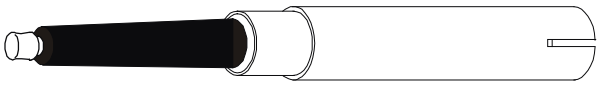


Figure 41. Liner-Removal Tool (P/N N6100102).

4. Press the small end of the liner-removal tool into the injector liner, then pull the injector liner out.

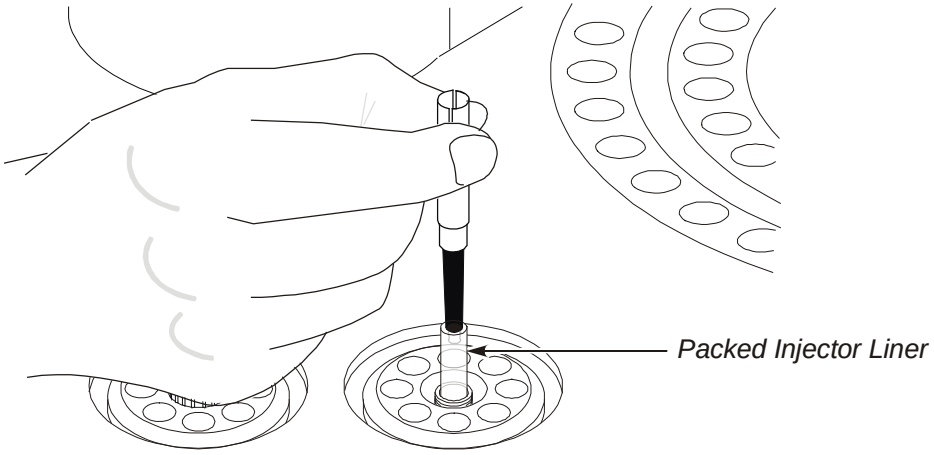


Figure 42. Removing the packed column injector liner.

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 the 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 N610T100), 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 43. Packed column injector liner (P/N N6101048) packed with wool.

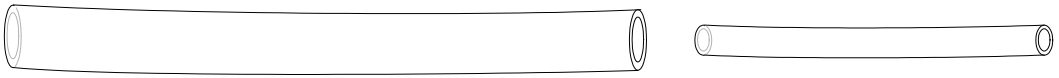
Changing and Repacking Capillary Split/Splitless (CAP) Injector Liners

The procedure below is applicable to the following injector liners:

Injector Liner	Size	Part Number
CAP wide-bore liner	4.0-mm i.d. and a 6.0-mm o.d.	N6121001
CAP narrow-bore liner	2.0-mm i.d. and a 6.0-mm o.d.	N6121002

Removing a CAP 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 02506534) as shown below.



CAP Liner-Removal Tool
P/N 0250-6534

PSS Liner-Removal Tool
P/N 0250-6247

Figure 44. CAP liner-removal tool (P/N 02506534)

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 (<100 °C) will make it difficult to remove the injector liner.**

2. Remove the septum cap.

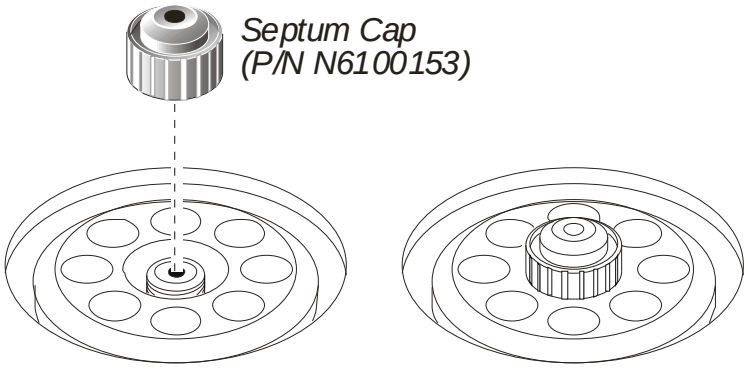


Figure 45. Removing the septum cap.

3. Remove the injector cover.

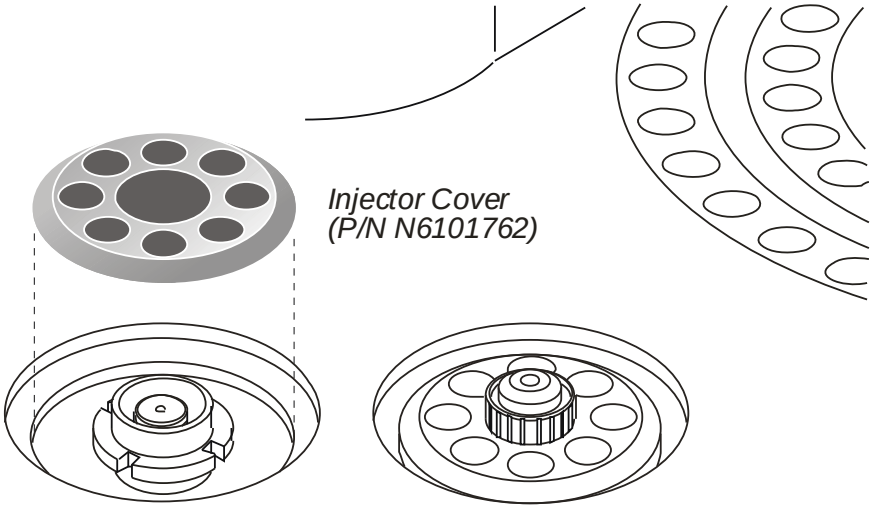


Figure 46. Removing the injector cover.

4. Loosen the threaded collar using the spanner (P/N N6101359) provided, then remove the threaded collar.

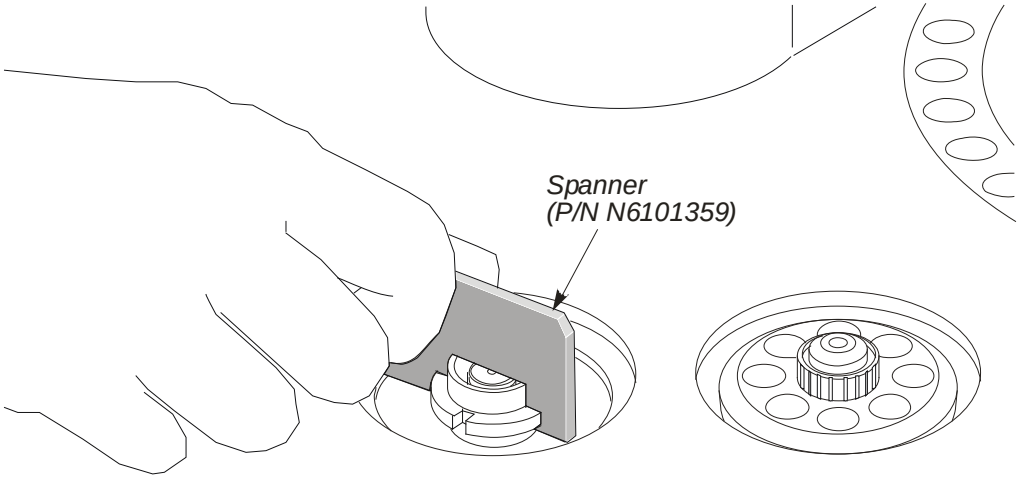


Figure 47. Loosening the threaded collar.

5. Replace the septum cap on the injector.
6. Pull the septum cap upwards to remove the septum purge assembly.
7. 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. Insert the CAP liner-removal tool (P/N 02506534) over the end of the CAP liner and lift the liner out of the injector

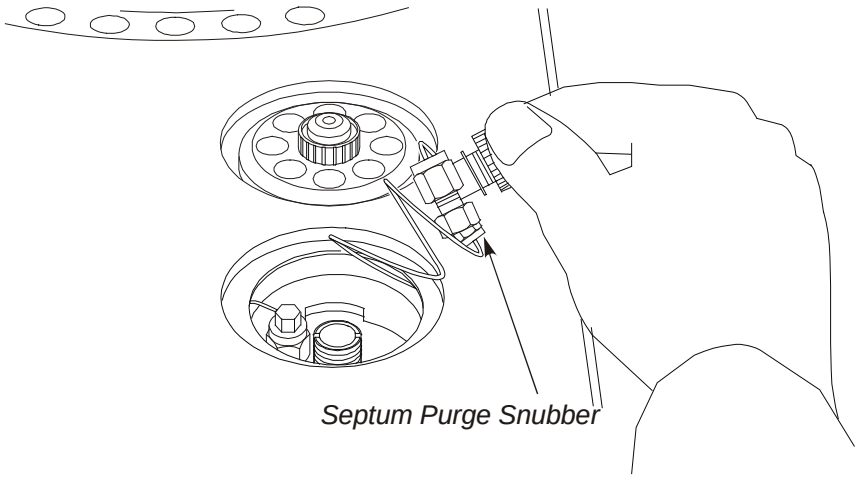


Figure 48. Removing the septum purge assembly.

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, cut off the flared end with a razor blade or scissors.*

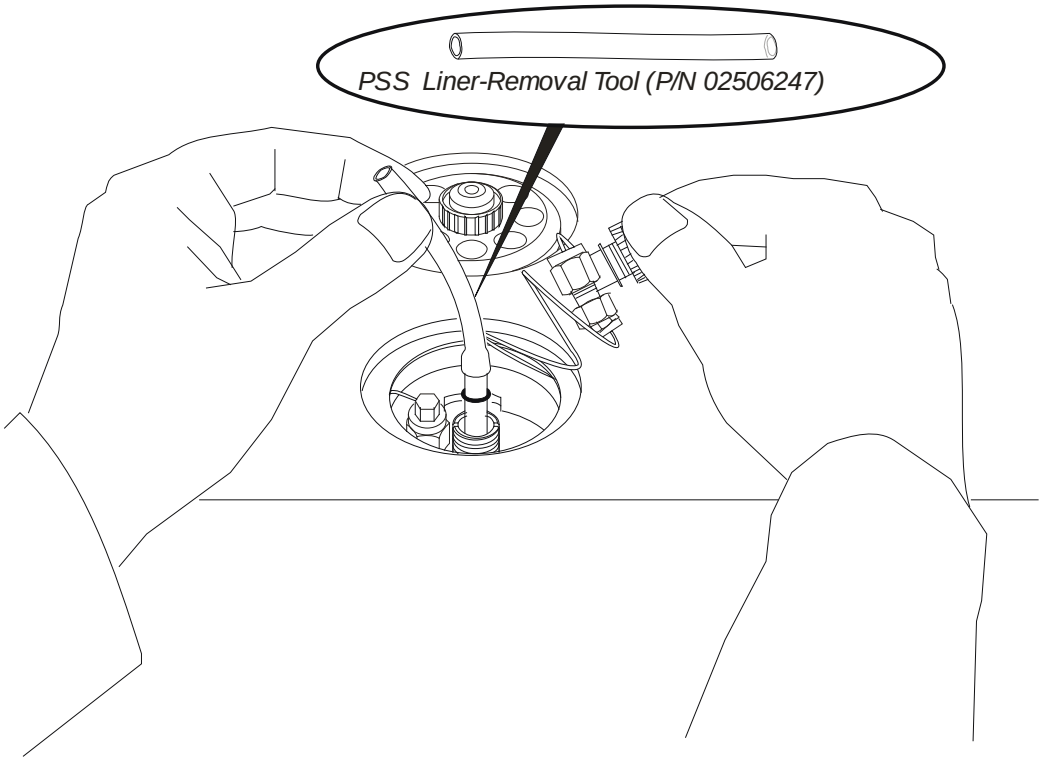


Figure 49. Removing an injector liner.

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.*

NOTE: *Each capillary liner has an O-ring installed on the frosted portion of the CAP liner. 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. See the following page for O-ring part numbers.*

About O-Rings

CAUTION	*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.*

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 in the following table). 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 our web site www.perkinelmer.com or by calling in the U.S. 1-800-762-4000, outside the US contact your local PerkinElmer Sales office. Viton (maximum temperature of 250 ºC) is recommended for the mass spectrometer.*

Injector O-Rings	Recommended Maximum Temperature
CAP Injector	
N610-1374 Silicone (pkg .of 10)	250 ºC
N610-1378 Graphite (pkg. of 5)	450 ºC
N930-2782 Kalrez (pkg. of 1)	450 ºC
N930-2783 Viton (pkg. of 1)	250 ºC (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 N6121001); 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 limited to a maximum of 2 µL for hydrocarbon solvents, and less than that for high-expansion solvents such as water or CH_2Cl_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 (P/N N6102345) 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 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 N610T100), 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.

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 N610T100), 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.

[*] The recovery of high-molecular-weight components (e.g., C_{40} and higher) may be improved if the liner is packed loosely.

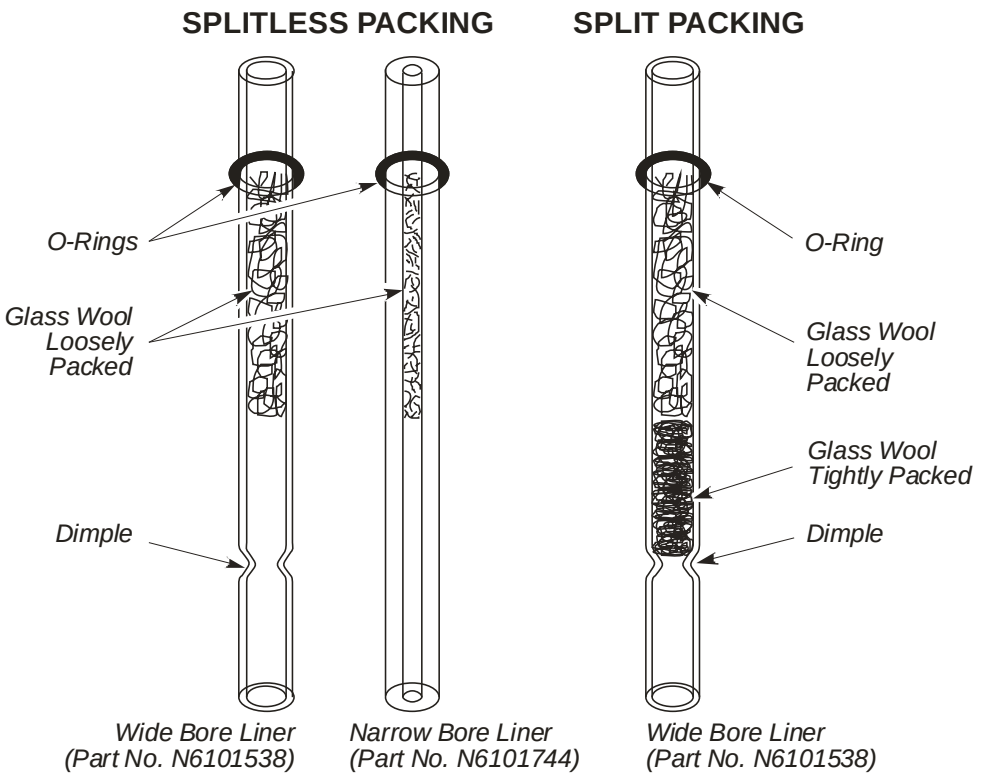


Figure 50. CAP injector liners packed with quartz wool.

NOTE: *As you can see in the previous figure, 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.

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 N610T100), 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.

Packing a CAP Injector Liner for Splitless Operation

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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!*

Changing the Charcoal Trap or Replacing Charcoal on the Split/Splitless CAP Injectors

The charcoal trap will eventually become saturated. When this occurs, ghost peaks and changes in split ratio will be observed.

---

* The recovery of high-molecular-weight components (e.g., C_{40} and higher) may be improved if the liner is packed loosely.

Removing a Charcoal Trap

1. Turn off the Clarus 400/480 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 400/480 GC (see the following figure) and raise the top cover until the cover locks in the raised position.

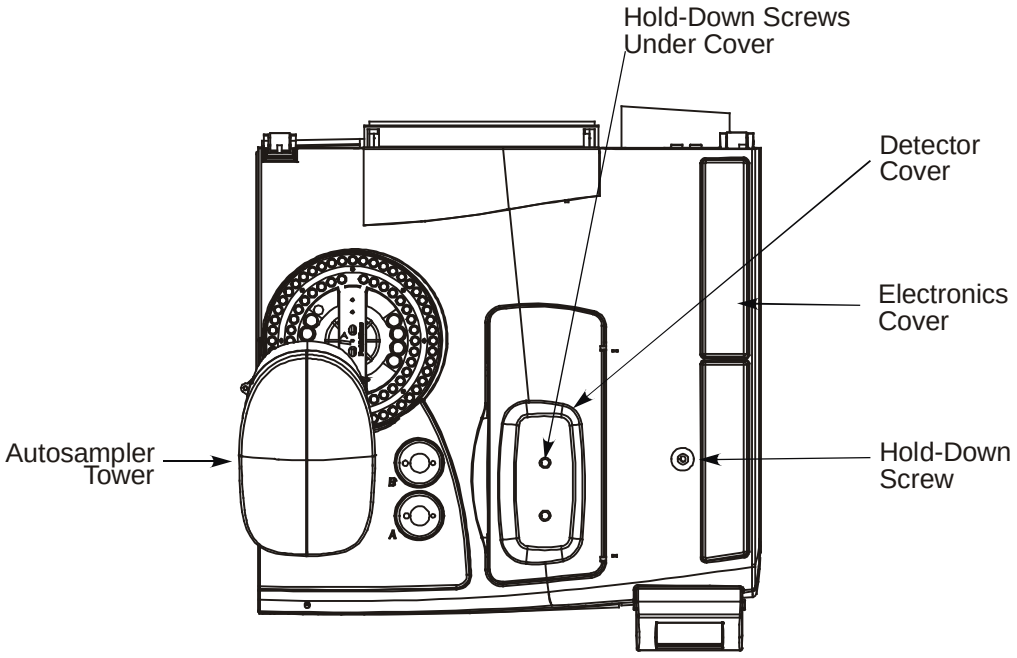


Figure 51. Location of the top cover hold-down screws.

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 N6101359) 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).

Installing a New Charcoal Trap

1. Install the manual pneumatics version charcoal trap (P/N N6100275), or just replace the charcoal in your current trap.

Manual Pneumatics (P/N N6100275)

Replace the charcoal by removing the glass wool plug from the 1/4-inch tubing end of the charcoal filter. Empty the old charcoal from the charcoal filter. Repack the charcoal filter with activated charcoal (30-60 mesh, P/N 03300904). Plug the end of the charcoal filter with a small piece of silanized glass wool. Reinstall the charcoal trap.

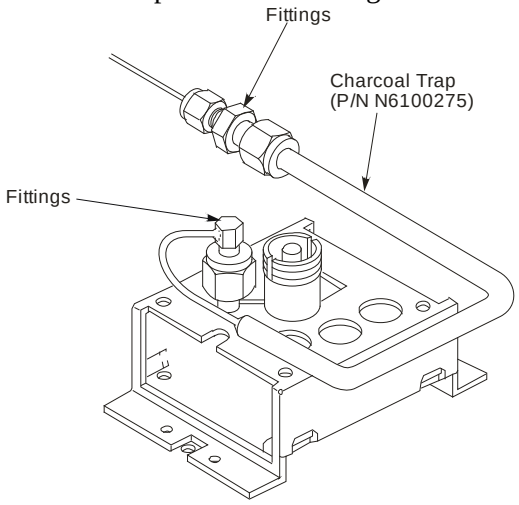


Figure 52. Charcoal Trap on an injector controlled by manual pneumatics (right).

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 400/480 GC top cover and tighten the two hold-down screws.

CAUTION	*To prevent autosampler needle damage after the Clarus 400/480 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. Then move the top cover so that the vial locator is aligned with the center of the septum cap. Secure the Clarus 400/480 GC top cover in this position by tightening the two screws.*

ECD Maintenance

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 400/480 GC display by pressing **[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. Remove the column from the ECD, then cap the ECD with a plug (P/N 09903098).

2. Increase the make-up flow.

   If the background remains the same as the make-up flow increases, the carrier gas could be contaminated. (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 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 09903098).

2. Increase the flow of make-up gas to 60 – 100 mL/min and raise the detector temperature to 450 ^{o}C.

3. Bake the system until the background returns to normal levels. This could take several days.

NOTE: *It may also help to remove the column and increase the oven temperature to 450 ℃ to bake out the lower portion of the ECD body.*

Changing the Charcoal Traps

The ECD is shipped with charcoal traps (P/N N6600037) installed in the make-up and injector pneumatics lines to remove contamination from the needle valve, flow controller, or pressure regulator. The traps should be replaced periodically.

To change charcoal traps:

1. Turn off the Clarus 400/480 GC and allow the injectors/detectors to cool.
2. Loosen the two hold-down screws on the Clarus 400/480 GC top cover (see Figure 50) and raise the top cover until it locks in the raised position.
3. Disconnect the charcoal traps from the make-up gas and injector lines.
4. Install new charcoal traps.

Cleaning the ECD Anode

WARNING

THE FOLLOWING PROCEDURE MUST BE PERFORMED ONLY AT LABS THAT HOLD A SPECIFIC NRC LICENSE, NOT A GENERAL LICENSE. ALL OF THE MATERIALS USED TO CLEAN THE ANODE MUST BE DISPOSED OF IN ACCORDANCE WITH THE NRC REGULATIONS REGARDING RADIOACTIVE MATERIAL.

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 anode in a beaker of hexane 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 to a normal level..

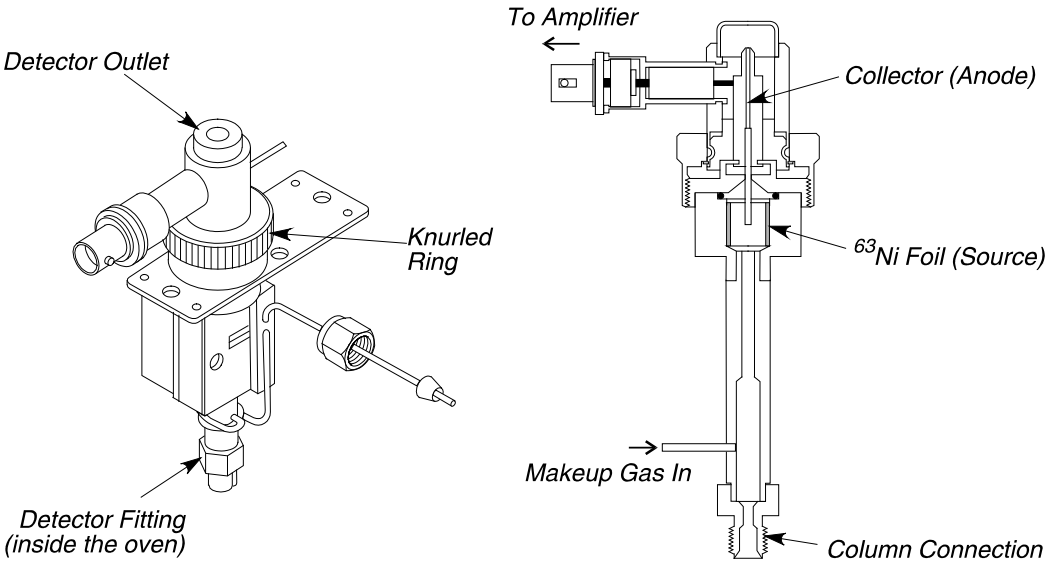


Figure 53. Isometric view and cross section view of the ECD.

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 cool to the touch.
2. Gain access to the detector by lifting the detector cover (see the following figure).

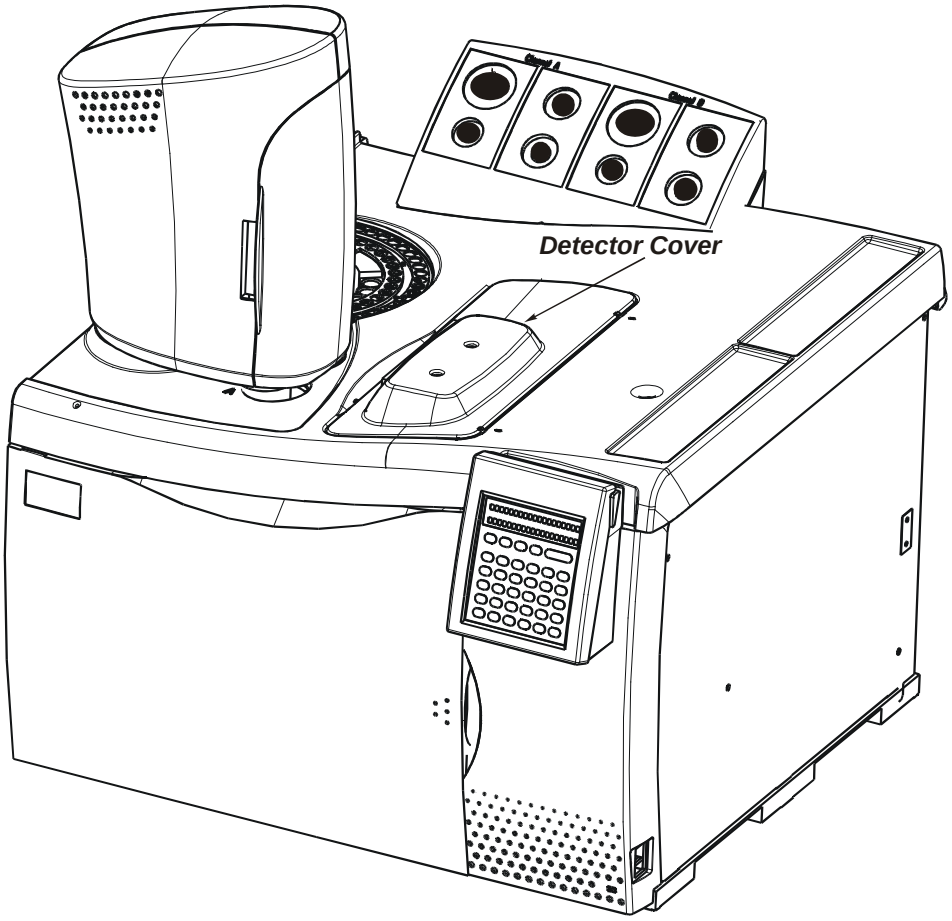


Figure 54. The detector cover.

3. 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!

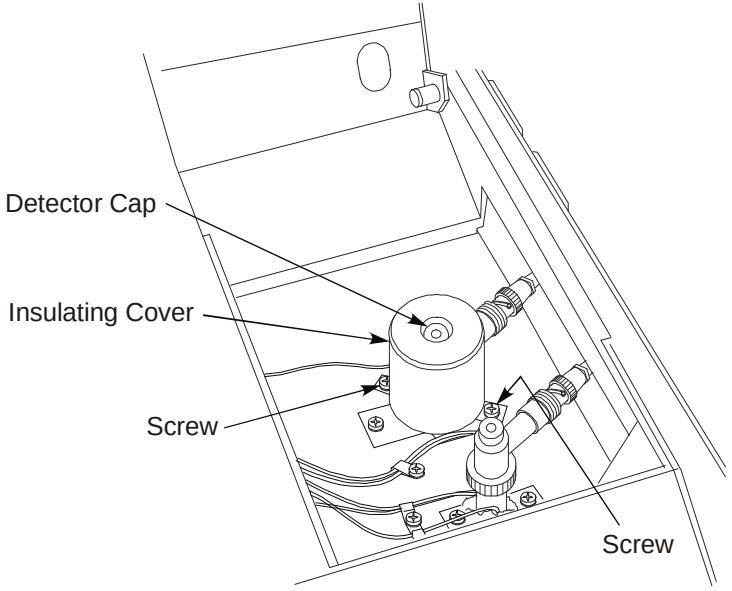


Figure 55. ECD insulating cover.

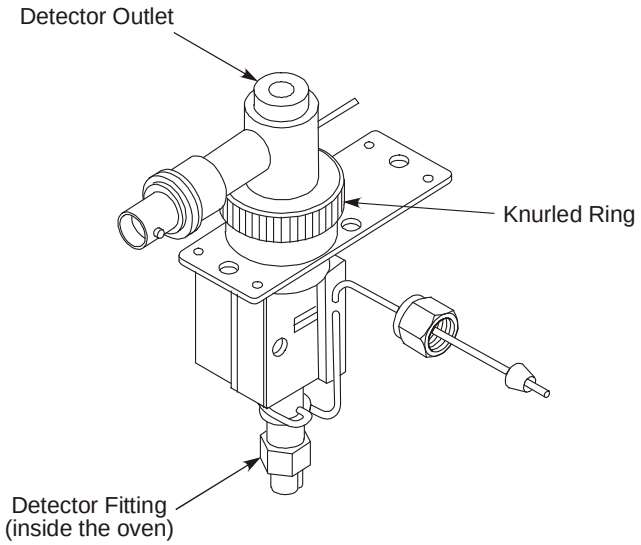


Figure 56. ECD surfaces to wipe.

4. Using the instructions included with the wipe-test kit (P/N 00091667) supplied with the detector, wipe the external surfaces of the items shown in Figure 55 with the "low Activity Source" filter papers:

   - 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 the wipe-test kit. Include a data sheet stating that the wipe test was performed on a PerkinElmer electron capture detector cell (P/N N6100063) 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

NOTE: *The sensitivity of the wipe test is 0.0001 µCi.*

Disposal and Refurbish/Refoil 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 Instruments LLC
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 state and local agency, if applicable.

FID Maintenance

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 by turning the outer knob on the hydrogen needle valve completely clockwise.

1. Turn off the Clarus 400/480 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 53).

3. Remove the polarizing cable from the pin on the polarizing filter assembly.

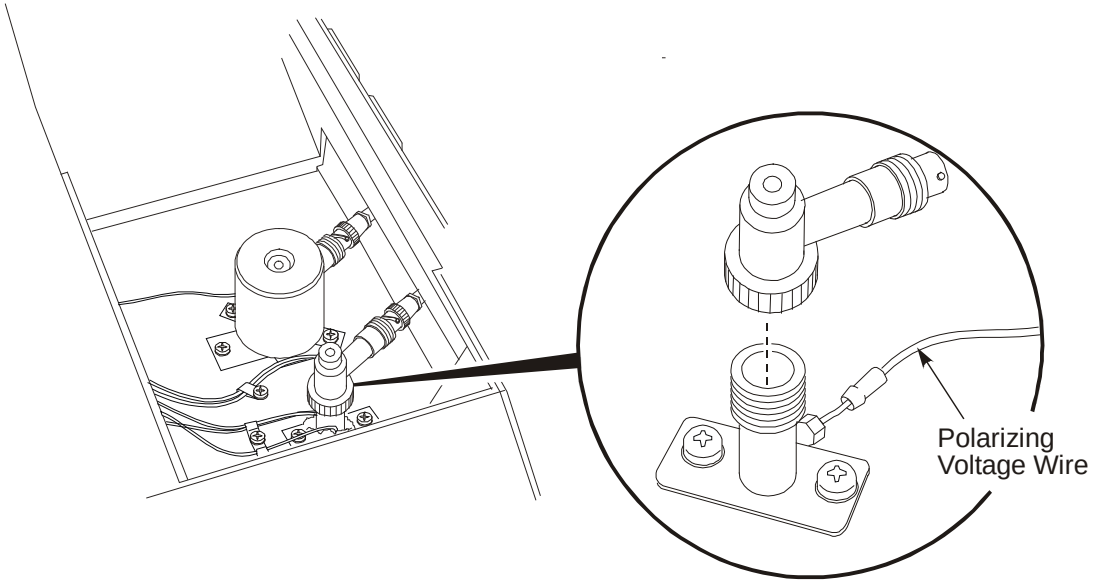


Figure 57. FID polarizing voltage wire.

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 N6103188) into the FID base and lift the nozzle out of the FID base.

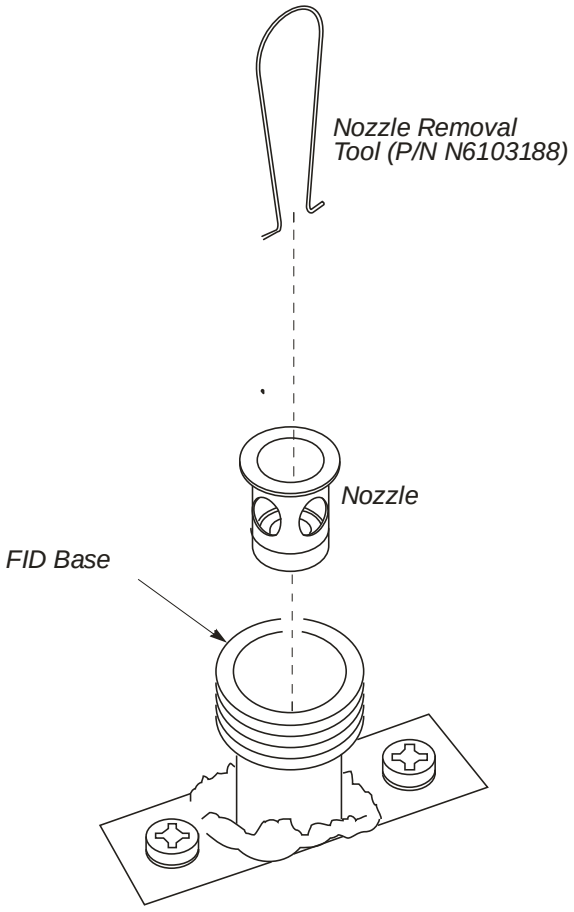


Figure 58. Removing the nozzle assembly from the FID base.

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.

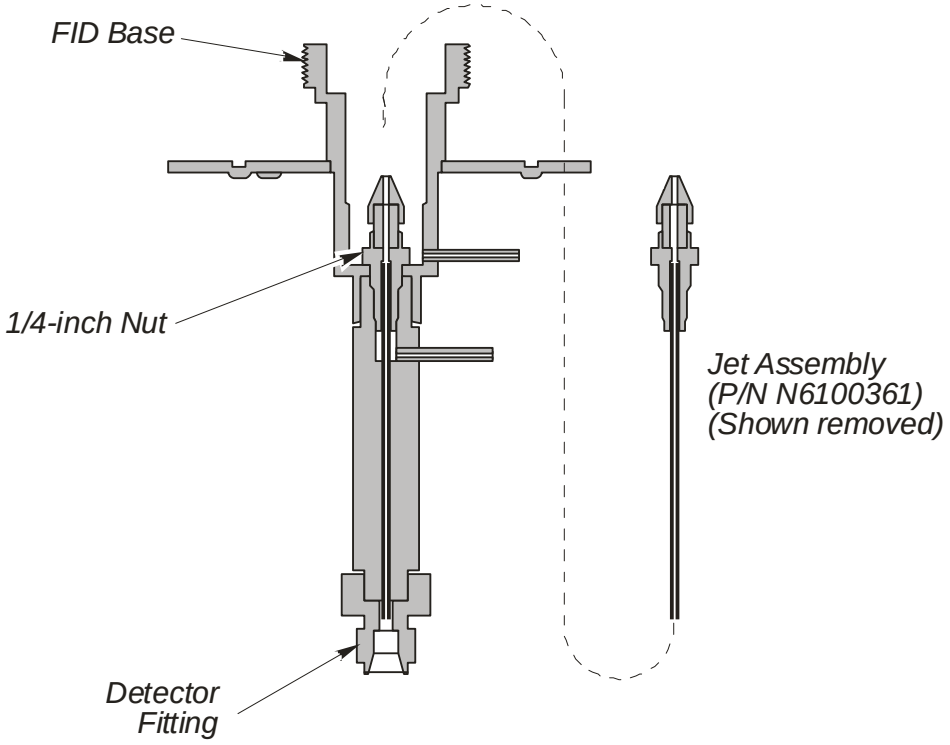


Figure 59. Cross section view of the FID.

8. Insert a new FID jet assembly (P/N N6100361) 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.

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 replace the O-ring in the FID collector:

1. Remove the polarizing voltage wire from the polarizing pin (Figure 56).
2. Loosen the knurled ring, then lift the FID collector off of the FID base.
3. Remove the old O-ring (shown in Figure 59) from the FID collector and insert a new O-ring (P/N 09902143).
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.
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.

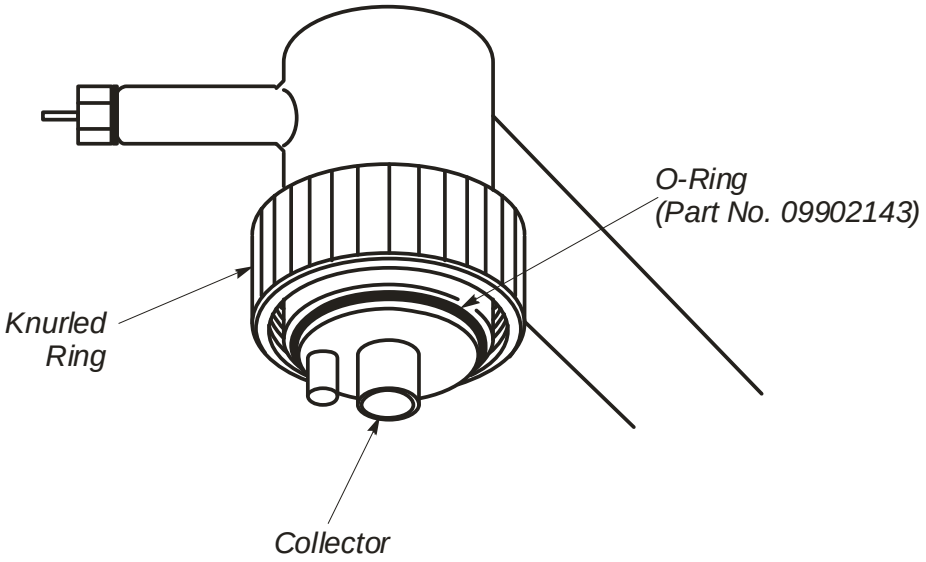


Figure 60. View of the FID collector and O-ring.

NPD Maintenance

NPD maintenance consists of changing and activating the NPD bead and replacing a NPD jet.

Changing the NPD Bead

The Nitrogen Phosphorus Detector utilizes a glass bead that contains alkali metal ions (single bead P/N N6120092 or package of five P/N N6120093) to detect organically bound nitrogen and phosphorus compounds. Examine the NPD and make sure it does not have any broken weld or wires. Operation of the bead in an NPD detector leads to gradual loss of the alkali metal ions, and in time, the bead will not respond and need to be replaced. 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 (in 0.25 mV increments). If you cannot achieve a response at higher settings up to 2mV, then you should recondition the BEAD and be certain that all your gas flows are accurate. Make certain that hydrogen flow is within +/- 10% of 2 ml/minute flow rate and that you precisely followed the conditioning procedure instructions.

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 compartment cover (see Figure 53).

2. Locate the bead potentiometer.

3. 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.

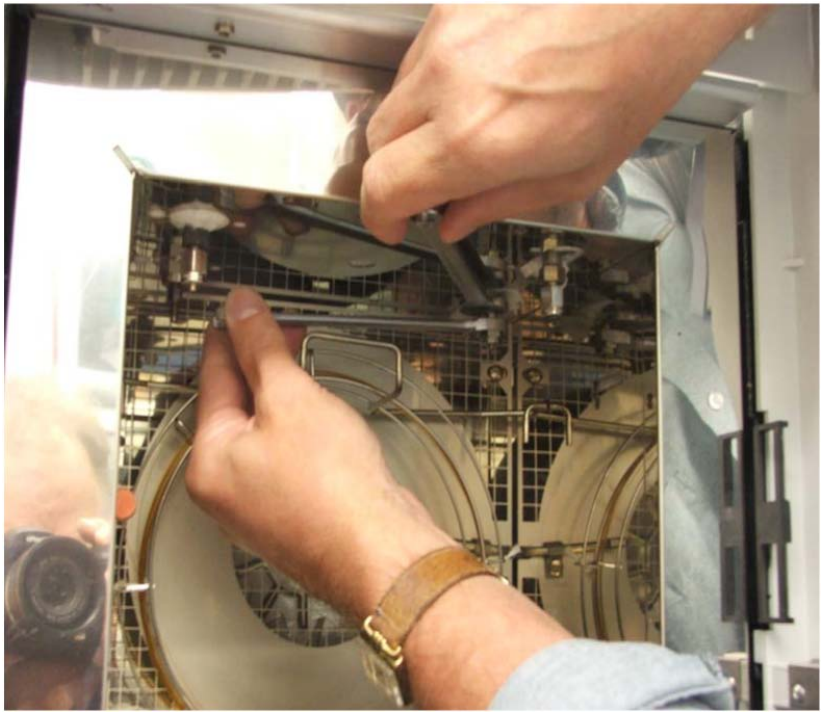
4. Turn the NPD bead off by turning the potentiometer counterclockwise**.**

NOTE: *If two NPDs are installed, turn both beads off, even if you are only replacing one bead.*

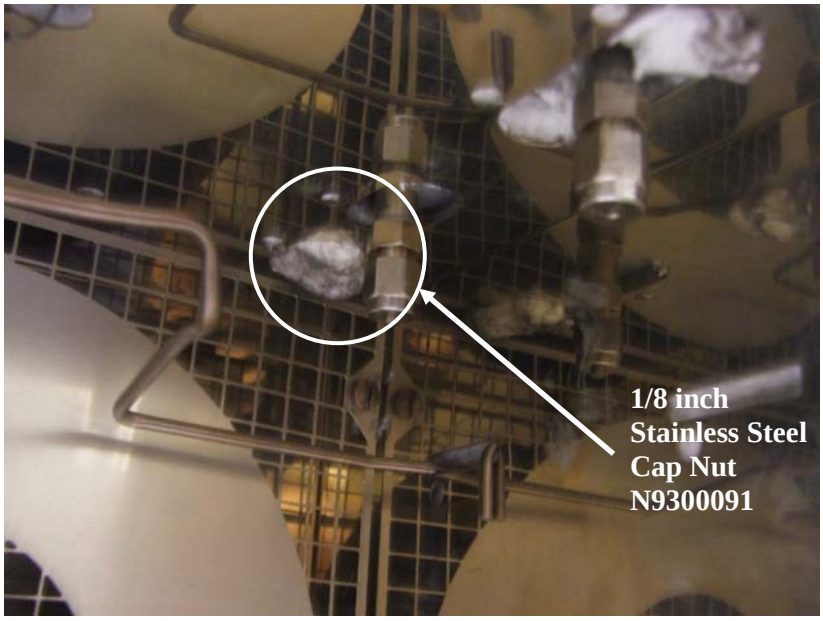
5. Press the **Oven** key and check that the GC oven temperature is set to 50 ºC or below

6. Open the oven door and check to see if a column is attached to the NPD detector on which the bead is to be changed.

7. If a column is attached the NPD detector in question, remove it from the detector with an open-end wrench (7/16 inch wrench for 1/8 inch stainless steel tube nut). Use a second 7/16 inch open-end wrench on the NPD detector base (to avoid applying torque on the detector body brazed fitting). Allow the detector end of the capillary column and column nut to rest on the base of the floor of the oven.



8. Attach a 1/8 inch stainless steel Cap Nut (N9300061) to the NPD detector in place of the 1/8 inch stainless steel tube nut. This step is in preparation for conditioning the new bead, to make sure the specified flows of detector gases pass upwards across a bead to be conditioned.



9. Check the Detector temperatures by pressing the **Detector Temp** Key

10. If the detector temperature of any detector installed is over 100 ºC, set the detector temperature to OFF or the minimum allowed value.

11. When detector temperatures show residual temperature values below 100 ºC, turn off the Clarus 400/480 GC and unplug the line cord from the line voltage for general safety.

WARNING

The gases can remain on during this procedure, but the detector should be cool to the touch to protect you from getting burned.

12. Remove the NPD collector assembly (see Figure 60) 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).*

13. Remove the screw that secures the bead transformer assembly to the top of the Clarus 400/480 GC oven.

14. Carefully remove the bead portion from the detector body by lifting the bead transformer assembly straight up and out of the detector body.

15. Remove the bead assembly from the transformer assembly by unplugging it from the connector (see Figure 60).

16. Plug a new bead assembly (P/N N6120092) into the connector on the bead transformer. The connector is keyed so that the bead assembly can only be inserted one way.

17. Carefully insert the bead portion of the bead assembly in the detector body as shown in Figure 60.

18. Secure the bead transformer to the top of the oven with the screw removed in step 6 of this procedure.

19. 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.

NOTE: *Check that the polarizing wire has not fallen off the detector. If it has, replace it (Figure 62).*

20. Reinsert the line cord in the wall socket and switch on the Clarus 400/480 GC.

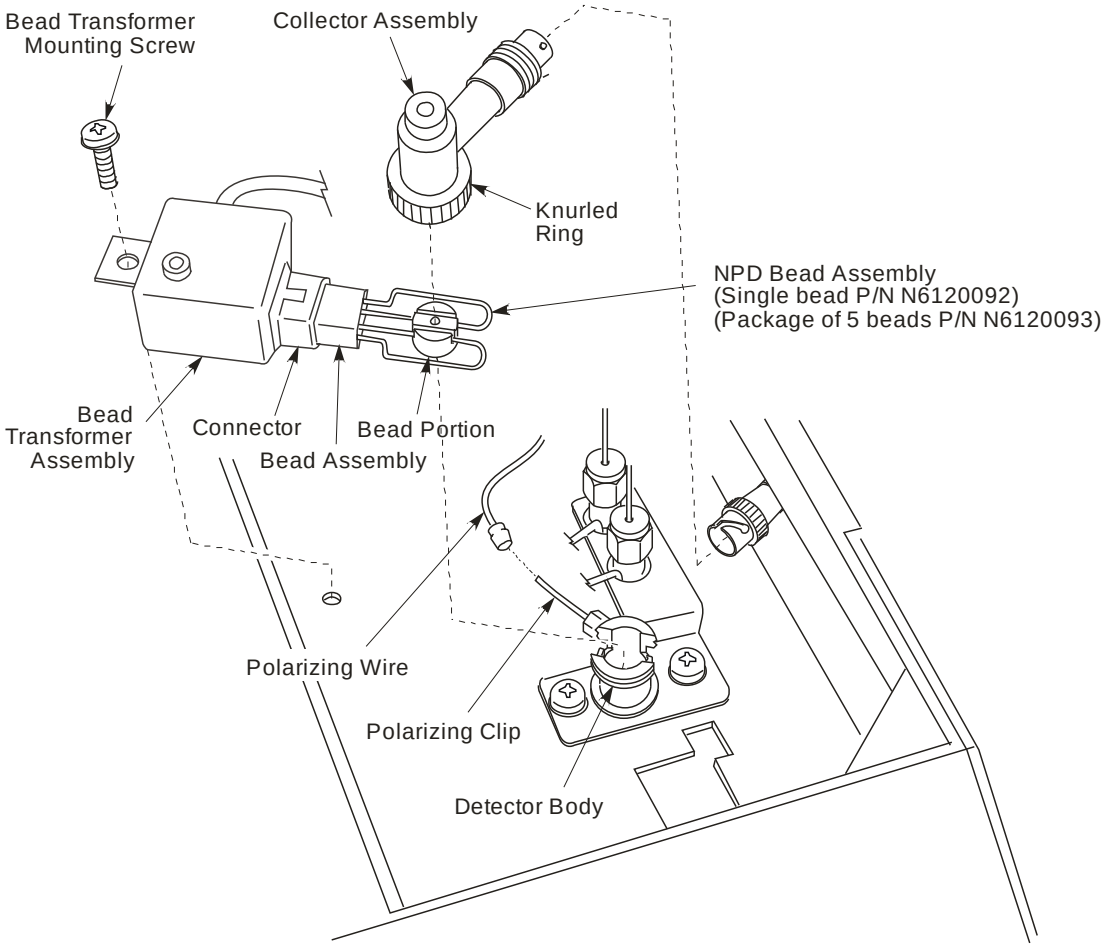


Figure 61. Exploded view of the NPD bead assembly.

NPD Bead Activation Procedure

Select one of the following procedures to activate your NPD bead:

Packed Column Setup:

Disconnect column and cap the oven end of the detector. The recommended Detector Cap Nut is in the 1/8-in. Plug Kit (P/N N9300061).

Capillary Column Setup:

With a capillary column connected, adjust Carrier flow to 0.5 mL/min or less (but not 0), or (preferred) disconnect the column and cap the oven end of the detector.

Reactivating a Used NPD Bead

Preparing to Activate the NPD Bead

To activate a NPD bead:

NOTE: *Never activate a NPD bead with a column connected to the detector fitting. To properly activate the NPD bead, the column must be removed from the detector fitting and the detector fitting must be capped.*

1. Obtain a stopwatch or use the stopwatch function in the GC Utilities screen.
2. Open the oven door and allow the oven to cool.
3. Using a 7/16-inch wrench, loosen and remove the column nut from the detector fitting. Remove the column from the detector fitting.
4. Install a 1/8-inch Swagelok plug (P/N N9300061) on the detector fitting. Provide a leak-free seal by tightening the plug with a 7/16-inch wrench.
5. Close the oven door. Maintain a carrier gas flow through the column.
6. Install the bead in the conventional manner according to the installation procedure in the GC Hardware Guide. Make sure that the bead does not contact any other surfaces.
7. Set the following conditions in the following order:
   - Set the Detector temperature to 150 deg. C. or greater. Wait for the detector temperature to stabilize.

WARNING

If a capillary column is used and is not disconnected from the detector, a high detector temperature can cause bleed at the detector junction with the column. Make certain the detector temperature is lower than the maximum temperature recommended for the capillary column of your application.

- Set the hydrogen and air flow as follows:
  - Air to 100 mL/min
  - Hydrogen to 2.0 mL/min

NOTE: ***Hydrogen flow accuracy is critical to proper NPD bead activation****. Make certain that the H2 flow is within 0.5 mL/min, or closer, to the H2 set point of 2.0 mL/min. Check with a calibrated flow meter at the detector bulkhead that the flow is accurate if activation problems occur.*

NOTE: ***Ensure that the hydrogen line is well purged of entrained air before you make the final setting of hydrogen flow****. Failure to purge the gas line from the hydrogen supply (tank or generator) will result in an incorrect flow setting for operation and can lead to poor operating performance and reduce bead life.*

3. Set the detector range to x1.
4. From the system status screen touch the **signal** icon  to display the detector background.

   When the bead is off, the background value should be 0 ±0.25 mV. Write down the background value that occurs with the bead off.
5. Open the detector cover (see Figure 4-28) and locate the bead potentiometer dial see (the following figure).

   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.

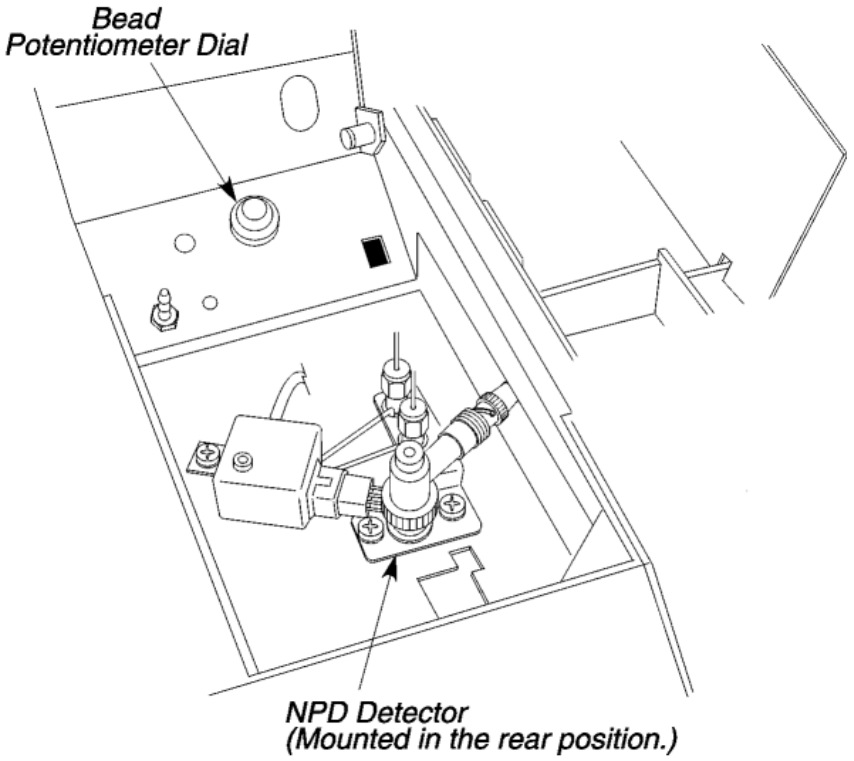


Figure 62. Location of the Potentiometer Dial.

Activating the NPD Bead

The bead requires an activation step prior to use. **Do not inject a sample during Activation.**

To Activate the bead, carefully follow this procedure in sequential steps below:

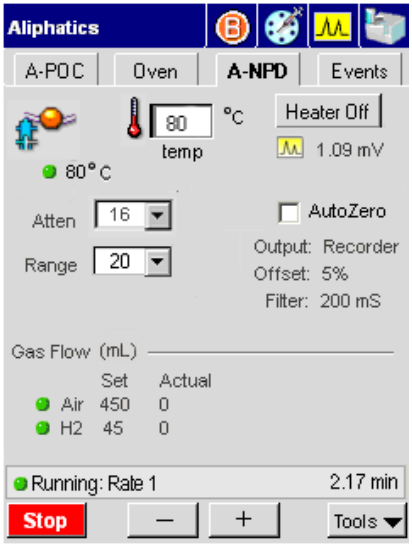
1. Turn the bead potentiometer dial clockwise to apply current to the bead.
2. Slowly increase the NPD control voltage potentiometer to a setting of 700.

NOTE: *The bead will gradually acquire an orange glow at this point.*

3. Wait for 5 minutes then increase the potentiometer to 750.
4. After 1 minute move the potentiometer to 800.
5. After 1 minute move the potentiometer to 850.
6. After 1 minute move the potentiometer to 900.

7. After 1 minute reduce the potentiometer to 850.
8. After 1 minute reduce the potentiometer to 800.
9. After 1 minute reduce the potentiometer to 750.
10. After 1 minute reduce the potentiometer down to 700
11. **For Capillary columns:** Wait 10 minutes, go to the NPD detector screen, then adjust the potentiometer so that the NPD baseline response reads around 0.5 mV on Range 1. Then set the desired column flow for the application to be run.

    For Packed Columns: Wait 10 minutes, go to the NPD detector screen, then adjust the potentiometer down to 500 or below to drop the bead below the active level. Remove the Detector Cap Nut (P/N N9300061) and reconnect the column to the detector. Set the desired column flow for the application to be run, then again increase the bead current potentiometer to give a reading around 0.5 mV on Range 1.
12. Allow the baseline to stabilize for 30 minutes.
13. Using the potentiometer, adjust the baseline between 0.4 and 0.6 mV at Range 1 and the bead ready to use.
14. After conditioning the bead remove the Swagelok plug from the detector fitting and reconnect the column.



15. Make sure the column flow is properly set. Touch the **signal** icon  to display the detector background produced by carrier gas flow. The bead background will be less due to cooling effects.
16. 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.
17. 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.

Adjust the background setting to achieve the sensitivity required for your samples. For example, if you have a concentrated sample, such as drugs, it can be analyzed with a background of 0.25 – 0.50 mV. If you have a low-level sample, such as pesticides, it may require a setting as high as 1 – 2 mV. To get the maximum life from your NPD bead, operate at the lowest setting to achieve your required sensitivity.

NOTE: **(***1) The bead will be stable after operating for several hours, but it 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.

NOTE: *The bead should be turned off by turning the bead potentiometer dial fully counterclockwise before you turn off the Clarus GC. 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).*

Reactivate a Used NPD Bead

To increase the bead life, turn off the bead if the NPD is not continually in use. When you turn off the bead, keep the detector temperature, hydrogen flow, and air flow set to operating conditions. If the column remains connected to the injector and detector in the oven, make sure the carrier gas is flowing through the column. If the column is disconnected from the detector, make sure the Swagelok cap is installed on the NPD fitting.

Prior to using the NPD, adjust the bead background to the same millivolt setting that you were using. Run your standards to determine if the NPD bead is responding at a level of sensitivity equal to the previous run. If not, then reactivate the NPD bead.

To reactivate a NPD bead follow the activation procedure above.

Operating Notes

The bead is operated as a conventional NPD bead; following these instructions will increase the bead lifetime.

Baseline: Set the baseline to a level of between 0.4 – 0.5. A lower baseline will help to extend bead lifetime. Studies have shown that the signal-to-noise will not be adversely affected by the lower baseline.

Column bleed: Material from the column can effect bead lifetime. Keep the maximum oven temperature as low as possible for each application.

Between run operation: If the NPD bead is not in use for a few days it is best to simply lower the potentiometer setting to 600 and maintain the detector temperature at 200 deg. C. or

greater. If the NPD detector will not be in use for an extended period, simply turn the potentiometer to zero. To reuse the bead follow the activation procedure.

Loss in sensitivity – the NPD bead is a consumable and will experience a loss in sensitivity over time. Often, adequate sensitivity can be restored by repeating the activation process.

Maximizing the NPD Bead Life

NPD bead life, optimal performance and sensitivity are greatly influenced by the following external factors: proper conditioning, accurate hydrogen gas flow, purity of gases used, solvents used, and baseline current settings.

In addition exposure to water or the use of methanol, ethanol and/or halogenated compounds can degrade the life and performance of the bead.

The following bullets highlight how you can extend the NPD bead life.

- Use the lowest practical baseline current setting to achieve your desired sensitivity (.5 to .75 is typical).
- Make certain that your H2 flow is accurate with a calibrated H2 meter at the bulkhead H2 line of the NPD detector. The actual flow must not vary more then +/- 0.2 mL/min then the 2.0 mL/min setpoint when measured at the detector bulkhead.
- Make certain the precise conditioning procedure outlined by PerkinElmer is adhered to.
- Turn off the bead when not in use by lowering the potentiometer current.
- Run clean samples and keep the inlet/liner clean to minimize contamination.
- Keep the detector temperature high >150 º C to reduce moisture and other contaminants.
- If the NPD is off for an extended period of time in a high humidity environment, water may accumulate in the detector. To evaporate this water, set the detector temperature to 150°C and maintain it for at least 30 minutes prior to operation.
- NPD beads will need reconditioning depending on the length of time not in use and the ambient environment they are stored in.
- Column bleed from lower grade columns or columns being run near the top of their temperature range can reduce bead life.

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 400/480 GC.
3. Turn off the hydrogen and air flows.
4. Open the detector cover (see Figure 53).

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 (Figure 63).

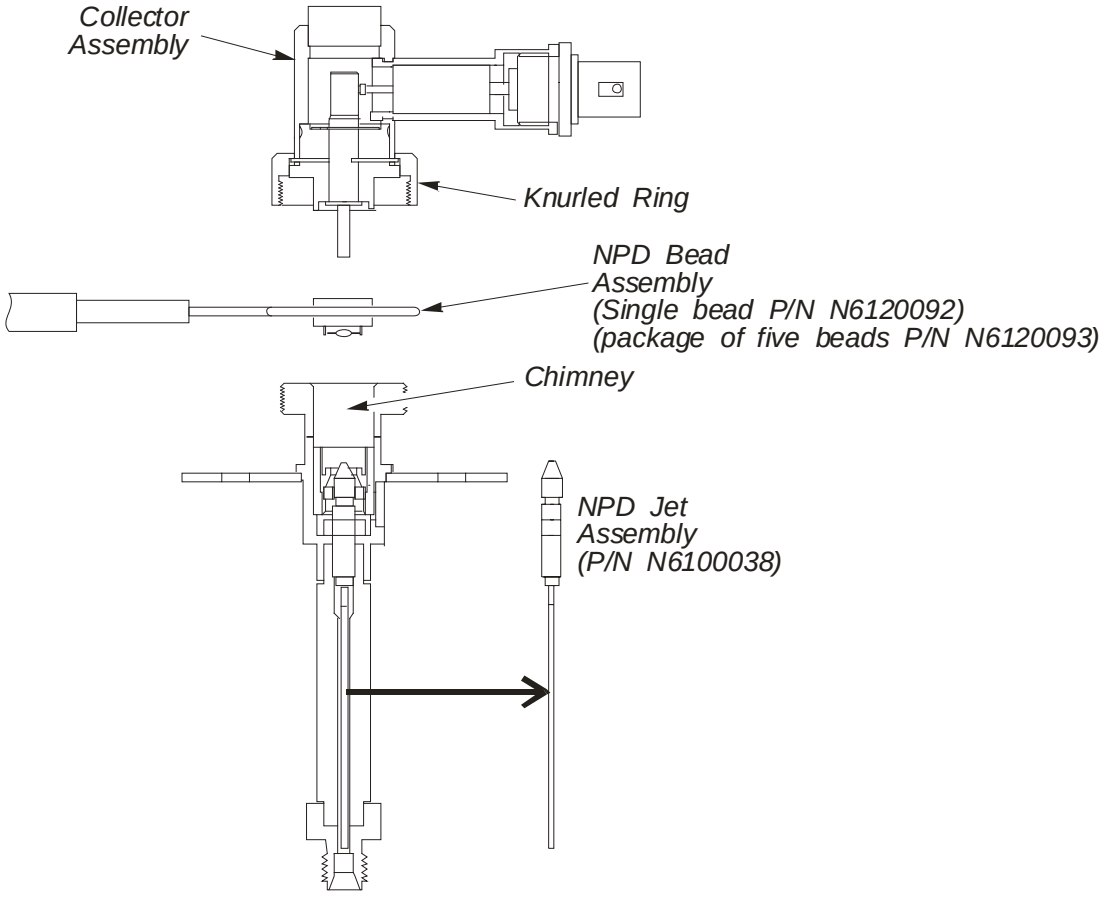


Figure 63. Location of the NPD jet assembly.

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 Figure 60).*

6. Remove the screw that secures the bead transformer assembly to the top of the Clarus 400/480 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 60).

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).

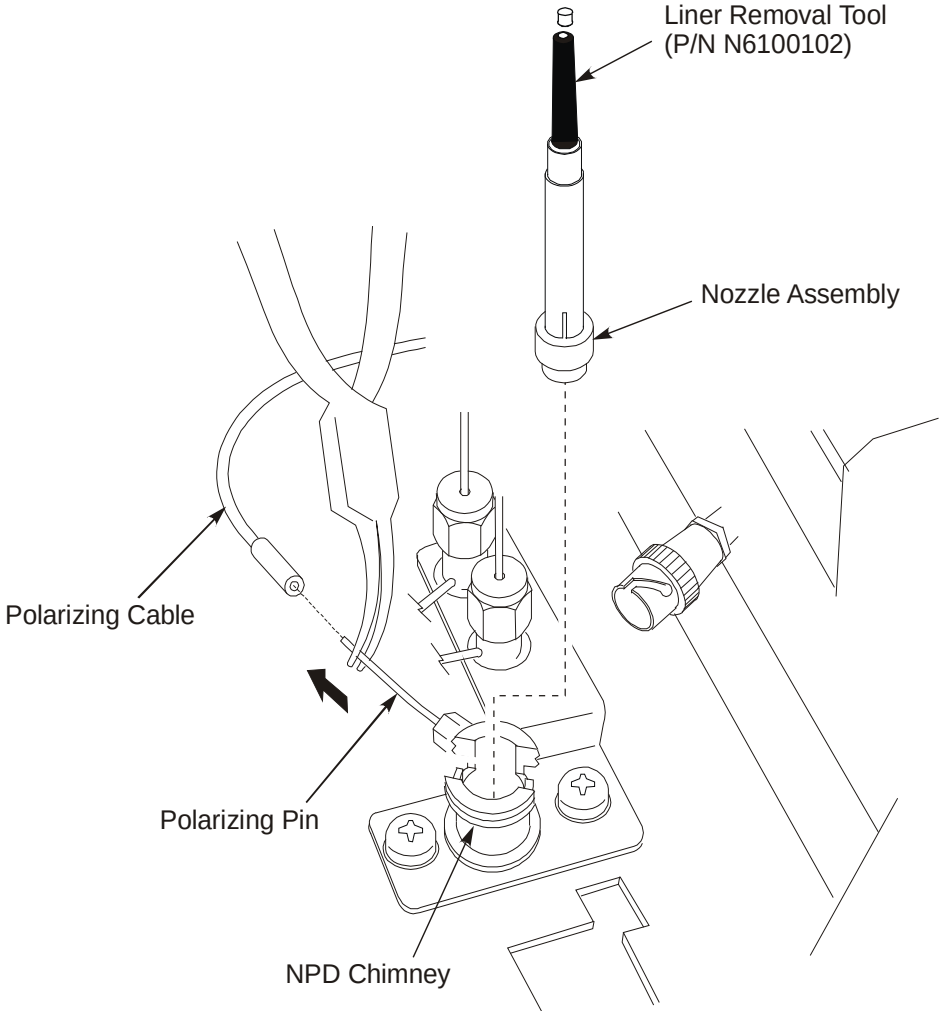


Figure 64. Removing the NPD nozzle assembly and polarizing wire.

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 N6100102) into the NPD chimney so that it engages the nozzle assembly. Then remove the nozzle assembly by lifting it out.

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 N6101297 provided in shipping kit) into the NPD chimney (see Figure 60) 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.

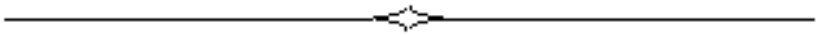
14. Install a new jet assembly (P/N N6100038) by reversing steps 5 through 13.

TCD Maintenance

The TCD requires no specific maintenance.

CAUTION	*The filament of the TCD **will** by damaged if current is applied **without** the gas flow on.*

Practical Hints 8



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. To display a Filter Menu, press **[System] [Enter] [Atten].** A menu similar to the following appears:

```
FID 1 Filter     Config
 50  ¦ 200  800
```

If your column delivers a peak width at half height of ≥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 or NPD (nA, Range 1) – 21.3

FID or NPD (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 and FID (fA) Range 1	NPD and FID (fA) Range 20	TCD (µV)	ECD (Hz)
1	$3.3x10^2$	$6.7x10^3$	8.0	2.3
2	$6.7x10^2$	$1.3x10^4$	1.6x10	4.7
4	$1.3x10^3$	$2.7x10^4$	3.2x10	9.4
8	$2.7x10^3$	$5.3x10^4$	6.4x10	19.0
16	$5.3x10^3$	$1.1x10^5$	$1.3x10^2$	38.0
32	$1.1x10^4$	$2.1x10^5$	$2.6x10^2$	75.0
64	$2.1x10^4$	$4.3x10^5$	$5.1x10^2$	150.0
128	$4.3x10^4$	$8.5x10^5$	$1x10^3$	$3.0x10^2$
256	$8.5x10^4$	$1.7x10^6$	$2x10^3$	$6.0x10^2$
512	$1.7x10^5$	$3.4x10^6$	$4.1x10^3$	$1.2x10^3$
1024	$3.4x10^5$	$6.8x10^6$	$8.2x10^3$	$2.4x10^3$
2048	$6.8x10^5$	$1.4x10^7$	$1.6x10^4$	$4.8x10^3$
4096	$1.4x10^6$	$2.7x10^7$	$3.3x10^4$	$9.6x10^3$
8192	$2.7x10^6$	$5.5x10^7$	$6.6x10^4$	$1.9x10^4$
16384	$5.4x10^6$	$1.1x10^8$	$1.3x10^5$	$3.8x10^4$
32768	$1.1x10^7$	$2.2x10^8$	$2.6x10^5$	$7.7x10^4$
65536	$2.2x10^7$	$4.4x10^8$	$5.2x10^5$	$1.5x10^5$

The following table lists attenuation vs. the maximum detector signal producing a 1-V output to an integrator.

Atten	NPD and FID (pA) Range 1	NPD and FID (pA) Range 20	TCD (mV)	ECD (kHz)
1	$3.3x10^2$	$6.7x10^3$	8.0	2.3
2	$6.7x10^2$	$1.3x10^4$	1.6x10	4.7
4	$1.3x10^3$	$2.7x10^4$	3.2x10	9.4
8	$2.7x10^3$	$5.3x10^4$	6.4x10	19.0
16	$5.3x10^3$	$1.1x10^5$	$1.3x10^2$	38.0
32	$1.1x10^4$	$2.1x10^5$	$2.6x10^2$	75.0
64	$2.1x10^4$	$4.3x10^5$	$5.1x10^2$	150.0

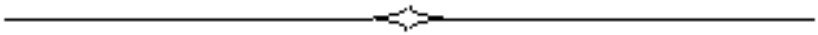
Optimizing ECD Performance

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.*

Appendix U.S. Nuclear Regulations

NOTE: *All USNRC regulations can be obtained through the internet at www.nrc.gov/reading-rm/*



Appendix - SUPPLEMENT 2
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U.S. Nuclear Regulatory Commission Regional Offices

REGION	ADDRESS	TELEPHONE
I **WPI's Region**	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)-816-5100.
(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 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)(vii), 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 20186, Apr. 25, 1995; 66 FR 64738, Dec. 14, 2001; 67 FR 3585, Jan. 25, 2002]

§ 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) A lens 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) A lens dose equivalent exceeding 15 rems (0.15 Sv); or
(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 paragraphs (a) and (b) of this section by telephone to the NRC Operations Center (301) 816-5100.

(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; 63 FR 39483, July 23, 1998]

§ 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
(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[1] 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 and must be clearly labeled "Privacy Act Information: Not for Public Disclosure."
(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 either by mail addressed to

the U.S. Nuclear Regulatory Commission, ATTN: Document Control Desk, Washington, DC 20555-0001; by hand delivery to the NRC's offices at 11555 Rockville Pike, Rockville, Maryland; or, where practicable, by electronic submission, for example, Electronic Information Exchange, or CD-ROM. Electronic submissions must be made in a manner that enables the NRC to receive, read, authenticate, distribute, and archive the submission, and process and retrieve it a single page at a time. Detailed guidance on making electronic submissions can be obtained by visiting the NRC's Web site at *http://www.nrc.gov/site-help/e-submittals.html*, by calling (301) 415-0439, by e-mail to *EIE@nrc.gov*, or by writing the Office of Information Services, U.S. Nuclear Regulatory Commission, Washington, DC 20555-0001. A copy should be sent to the appropriate NRC Regional Office listed in appendix D to this part.
[56 FR 23406, May 21, 1991, as amended at 60 FR 20186, Apr. 25, 1995; 61 FR 65127, Dec. 10, 1996; 68 FR 14309, Mar. 25, 2003; 68 FR 58802, Oct. 10, 2003; 70 FR 69421, Nov. 16, 2005; 72 FR 33386, Jun. 18, 2007]
[1] 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.
[56 FR 23406, May 21, 1991, as amended at 60 FR 20186, 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 or 63 of this chapter; or
(5) Possess spent fuel in an independent spent fuel storage installation (ISFSI) pursuant to part 72 of this chapter; or
(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:

Radionuclide	Quantity of radionuclide[1] in curies
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

[1] 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 by an appropriate method listed in § 20.1007 or via the REIRS Web site at *http://www.reirs.com*.
[56 FR 23406, May 21, 1991, as amended at 56 FR 32072, July 15, 1991; 66 FR 5578, Nov. 2, 2001; 68 FR 58802, Oct. 10, 2003]

§ 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 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 § 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
(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.
(i) Security requirements for portable gauges.
Each portable gauge licensee shall use a minimum of two independent physical controls that form tangible barriers to secure portable gauges from unauthorized removal, whenever portable gauges are not under the control and constant surveillance of the licensee.

[30 FR 8185, June 26, 1965, as amended at 38 FR 33969, Dec. 10, 1973; 43 FR 6922, Feb. 17, 1978; 48 FR 32328, July 15, 1983; 52 FR 1295, Jan. 12, 1987; 52 FR 8241, Mar. 17, 1987; 53 FR 19245, May 27, 1988; 53 FR 23383, June 22, 1988; 54 FR 14061, Apr. 7, 1989; 58 FR 7736, Feb. 9, 1993; 59 FR 61780, Dec. 2, 1994; 65 FR 79187, Dec. 18, 2000; 70 FR 2009, Jan. 12, 2005]

§ 30.35 Financial assurance and record keeping for decommissioning.

(a)(1) 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.

(2) Each holder of, or applicant for, any specific license authorizing the possession and use of sealed sources or plated foils of half-life greater than 120 days and in quantities exceeding 10^{12} times the applicable quantities set forth in appendix B to part 30 (or when a combination of isotopes is involved if R, as defined in § 30.35(a)(1), divided by 10^{12} is greater than 1), shall submit a decommissioning funding plan as described in paragraph (e) of this section. The decommissioning funding plan must be submitted to NRC by December 2, 2005.

(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 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

$1,125,000 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.

(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.

(5) Waste collectors and waste processors, as defined in 10 CFR part 20, Appendix G, must provide financial assurance in an amount based on a decommissioning funding plan as described in paragraph (e) of this section. The decommissioning funding plan must include the cost of disposal of the maximum amount (curies) of radioactive material permitted by license, and the cost of disposal of the maximum quantity, by volume, of radioactive material which could be present at the licensee's facility at any time, in addition to the cost to remediate the licensee's site to meet the license termination criteria of 10 CFR part 20. The decommissioning funding plan must be submitted by December 2, 2005.

(d) Table of required amounts of financial assurance for decommissioning by quantity of material. Licensees required to submit the $1,125,000 amount must do so by December 2, 2004. Licensees required to submit the $113,000 or $225,000 amount must do so by June 2, 2005. Licensees having possession limits exceeding the upper bounds of this table must base financial assurance on a decommissioning funding plan.

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)(1), divided by 10^4 is greater than 1 but R divided by 10^5 is less than or equal to 1.)	$1,125,000
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)(1), divided by 10^3 is greater than 1 but R divided by 10^4 is less than or equal to 1.)	225,000
Greater than 10^{10} but less than or equal to 10^{12} 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)(1), divided by 10^{10} is greater than, 1, but R divided by 10^{12} is less than or equal to 1)	113,000

(e) Each decommissioning funding plan must contain a cost estsimate 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. Cost estimates must be adjusted at intervals not to exceed 3 years. 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 requriements 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 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.

(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; 63 FR 29541, June 1, 1998; 68 FR 57335, Oct. 3, 2003]

§ 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
(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
(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.[1] To the extent that the information is available at the time of notification, the information provided in these reports must include:
(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 NRC using an appropriate method listed in § 30.6(a); and a copy must be sent to the appropriate NRC Regional office listed in appendix D to part 20 of this chapter. 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; 68 FR 58804, Oct. 10, 2003]
[1] The commercial telephone number for the NRC Operations Center is (301) 816-5100.

§

30.51 Records.

(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).

(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]

[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.

§ 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]

§ 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 of the NRC's Office of Nuclear Material Safety and Safeguards, using an appropriate method listed in § 30.6(a). 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; 68 FR 58804, Oct. 10, 2003]

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 institution of proceedings therefor, 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]

§ 30.63 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 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		$3x10^{-4}$
	Sb 124		$2x10^{-4}$
	Sb 125		$1x10^{-3}$
Argon (18)	A 37	$1x10^{-3}$	
	A 41	$4x10^{-7}$	
Arsenic (33)	As 73		5x10_$^{-3}$
	As 74		5x10_$^{-4}$
	As 76		2x10_$^{-4}$
	As 77		8x10_$^{-4}$
Barium (56)	Ba 131		2x10_$^{-3}$
	Ba 140		3x10_$^{-4}$
Beryllium (4)	Be 7		2x10_$^{-2}$
Bismuth (83)	Bi 206		4x10_$^{-4}$
Bromine (35)	Br 82	4x10_$^{-7}$	3x10_$^{-3}$
Cadmium (48)	Cd 109		2x10_$^{-3}$
	Cd 115m		3x10_$^{-4}$
	Cd 115		3x10_-4
Calcium (20)	Ca 45		9x10_-5
	Ca 47		5x10_-4
Carbon (6)	C 14	1x10_-6	8x10_-3
Cerium (58)	Ce 141		9x10_-4
	Ce 143		4x10_-4
	Ce 144		1x10_-4
Cesium (55)	Cs 131		2x10_-2
	Cs 134m		6x10_-2
	Cs 134		9x10_-5
Chlorine (17)	Cl 38	9x10_-7	4x10_-3
Chromium (24)	Cr 51		2x10_-2
Cobalt (27)	Co 57		5x10_-3
	Co 58		1x10_-3
	Co 60		5x10_-4
Copper (29)	Cu 64		3x10_-3
Dysprosium (66)	Dy 165		4x10_-3
	Dy 166		4x10_-4
Erbium (68)	Er 169		9x10_-4
	Er 171		1x10_-3
Europium (63)	Eu 152 (T/2=9.2 Hrs)		6x10_-4
	Eu 155		2x10_-3
Fluorine (9)	F 18	2x10_-6	8x10_-3
Gadolinium (64)	Gd 153		2x10_-3
	Gd 159		8x10_-4
Gallium (31)	Ga 72		4x10_-4
Germanium (32)	Ge 71		2x10_-2
Gold (79)	Au 196		2x10_-3
	Au 198		5x10_-4
	Au 199		2x10_-3
Hafnium (72)	Hf 181		7x10_-4
Hydrogen (1)	H 3.	5x10_-6	3x10_-2

Indium (49)	In 113m		1x10_-2
	In 114m		2x10_-4
Iodine (53)	I 126	3x10_-9	2x10_-5
	I 131	3x10_-9	2x10_-5
	I 132	8x10_-8	6x10_-4
	I 133	1x10_-8	7x10_-5
	I 134	2x10_-7	1x10_-3
Iridium (77)	Ir 190		2x10_-3
	Ir 192		4x10_-4
	Ir 194		3x10_-4
Iron (26)	Fe 55		8x10_-3
	Fe 59		6x10_-4
Krypton (36)	Kr 85m	1x10_-6	
	Kr 85	3x10_-6	
Lanthanum (57)	La 140		2x10_-4
Lead (82)	Pb 203		4x10_-3
Lutetium (71)	Lu 177		1x10_-3
Manganese (25)	Mn 52		3x10_-4
	Mn 54		1x10_-3
	Mn 56		1x10_-3
Mercury (80)	Hg 197m		2x10_-3
	Hg 197		3x10_-3
	Hg 203		2x10_-4
Molybdenum (42)	Mo 99		2x10_-3
Neodymium (60)	Nd 147		6x10_-4
	Nd 149		3x10_-3
Nickel (28)	Ni 65		1x10_-3
Niobium (Columbium) (41).	Nb 95		1x10_-3
	Nb 97		9x10_-3
Osmium (76)	Os 185		7x10_-4
	Os 191m		3x10_-2
	Os 191		2x10_-3
	Os 193		6x10_-4
Palladium (46)	Pd 103		3x10_-3
	Pd 109		9x10_-4
Phosphorus (15)	P 32		2x10_-4
Platinum (78)	Pt 191		1x10_-3
	Pt 193m		1x10_-2
	Pt 197m		1x10_-2
	Pt 197		1x10_-3
Potassium (19)	K 42		3x10_-3
Praseodymium (59)	Pr 142		3x10_-4
	Pr 143		5x10_-4
Promethium (61)	Pm 147		2x10_-3
	Pm 149		4x10_-4
Rhenium (75)	Re 183		6x10_-3
	Re 186		9x10_-4
	Re 188		6x10_-4
Rhodium (45)	Rh 103m		1x10_-1
	Rh 105		1x10_-3
Rubidium (37)	Rb 86		7x10_-4
Ruthenium (44)	Ru 97		4x10_-4
	Ru 103		8x10_-4

	Ru 105		1x10_-3
	Ru 106		1x10_-4
Samarium (62)	Sm 153		8x10_-4
Scandium (21)	Sc 46		4x10_-4
	Sc 47		9x10_-4
	Sc 48		3x10_-4
Selenium (34)	Se 75		3x10_-3
Silicon (14)	Si 31		9x10_-3
Silver (47)	Ag 105		1x10_-3
	Ag 110m		3x10_-4
	Ag 111		4x10_-4
Sodium (11)	Na 24		2x10_-3
Strontium (38)	Sr 85		1x10_-4
	Sr 89		1x10_-4
	Sr 91		7x10_-4
	Sr 92		7x10_-4
Sulfur (16)	S 35	9x10_-8	6x10_-4
Tantalum (73)	Ta 182		4x10_-4
Technetium (43)	Tc 96m		1x10_-1
	Tc 96		1x10_-3
Tellurium (52)	Te 125m		2x10_-3
	Te 127m		6x10_-4
	Te 127		3x10_-3
	Te 129m		3x10_-4
	Te 131m		6x10_-4
	Te 132		3x10_-4
Terbium (65)	Tb 160		4x10_-4
Thallium (81)	Tl 200		4x10_-3
	Tl 201		3x10_-3
	Tl 202		1x10_-3
	Tl 204		1x10_-3
Thulium (69)	Tm 170		5x10_-4
	Tm 171		5x10_-3
Tin (50)	Sn 113		9x10_-4
	Sn 125		2x10_-4
Tungsten (Wolfram) (74)	W 181		4x10_-3
	W 187		7x10_-4
Vanadium (23)	V 48		3x10_-4
Xenon (54)	Xe 131m	4x10_-6	
	Xe 133	3x10_-6	
	Xe 135	1x10_-6	
Ytterbium (70)	Yb 175		1x10_-3
Yttrium (39)	Y 90		2x10_-4
	Y 91m		3x10_-2
	Y 91		3x10_-4
	Y 92		6x10_-4
	Y 93		3x10_-4
Zinc (30)	Zn 65		1x10_-3
	Zn 69m		7x10_-4
	Zn 69		2x10_-2
Zirconium (40)	Zr 95		6x10_-4
	Zr 97		2x10_-4
Beta and/or gamma emitting byproduct material not listed above with half-life less than 3 years.		1x10_-10	1x10_-6

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

[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.[(2)]

(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 § 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 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 bequerel (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, within 30 days after the transfer of a device to a specific licensee or export, furnish a report to the Director of Nuclear Material Safety and Safeguards, ATTN: Document Control Desk/ GLTS, using an appropriate method listed in § 30.6(a) of this chapter. 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 §§ 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: Document Control Desk/GLTS, using an appropriate method listed in § 30.6(a) of this chapter--
(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 providing the Director of the Office of Nuclear Material Safety and Safeguards, by an appropriate method listed in § 30.6(a) of this chapter, a written justification for the request.

(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.
(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 § 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 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.

[39 FR 43532, Dec. 16, 1974, as amended at 40 FR 8785, Mar. 3, 1975; 40 FR 14085, Mar. 28, 1975; 42 FR 25721, May 19, 1977; 42 FR 28896, June 6, 1977; 43 FR 6922, Feb. 17, 1978; 53 FR 19246, May 27, 1988; 56 FR 23471, May 21, 1991; 56 FR 61352, Dec. 3, 1991; 58 FR 67659, Dec. 22, 1993; 64 FR 42275, Aug. 4, 1999; 65 FR 79188, Dec. 18, 2000; 68 FR 58804, Oct. 10, 2003]

[2] 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.

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