



VARIAN

CP-3800 GC

Operator's Manual

*3800 Keyboard and Display ♦ Sample Introduction
Detectors ♦ Communications ♦ Local Automation*



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3800 Keyboard and Display

The 3800 keyboard and display is an integrated user interface designed for maximum ease of learning and ease of use. This section describes the keyboard and display layout and functions, and gives step-by-step instructions for configuring and building methods on the 3800 Gas Chromatograph.

The material is presented in the same order that you should familiarize yourself with the system, e.g., first learn the 3800 display, then the basic keyboard functions such as the ENTRY and INSTRUMENT keys, and finally, the method specific functions.

The 3800 Display

The 3800 display allows easy access to all GC functions. Screens are presented in pages that contain up to 11 lines of text. Generally, all information pertaining to a specific component or function is presented on a single page. However, two or more pages are used for some functions, such as building the detector section of a method. In these cases the page numbers are clearly shown at the top of the screen, e.g., page 1 of 2, etc.

Some screen pages contain tables for time programmed parameters such as injector or column temperature programs, external event controls, or column pressure. An example of a 3800 screen page with a timed events table to program the split state of the 1079 injector is shown below.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME												
Method 1	Method 1		0.00	20.00												
Set (°C): 250	Actual (°C): 250		Split Ratio: Off													
FRONT 1079, Page 3 of 3																
<table border="1"><thead><tr><th>Time</th><th>Split State</th><th>Split Ratio</th></tr></thead><tbody><tr><td>Initial</td><td>OFF</td><td>OFF</td></tr><tr><td>0.75</td><td>ON</td><td>100</td></tr><tr><td>1.50</td><td>ON</td><td>5</td></tr></tbody></table>					Time	Split State	Split Ratio	Initial	OFF	OFF	0.75	ON	100	1.50	ON	5
Time	Split State	Split Ratio														
Initial	OFF	OFF														
0.75	ON	100														
1.50	ON	5														
																

The top line of each page identifies the method that is currently active, the method that is currently available to edit, whether the GC is under local or remote control (if the 3800 is under remote control then a computer icon appears in the middle of the top line of the display), and finally, the current run time and the end time of the active method.

The ACTIVE method represents the method parameters that are currently active and are reflected on the second line of the display in the status section. The 1079 Injector screen above shows the set and actual temperature and the current split ratio.

The EDIT method is the method that was last chosen as the method to edit, and its parameters are shown in the middle section of the display. Page 1 of the 3800 Column Oven method parameters is shown below. Any changes made to the method will be automatically updated. However, for these changes to affect the next GC analysis, the edited method must be activated. After editing a method, you must activate it to ensure its parameter settings are used during the next analysis.

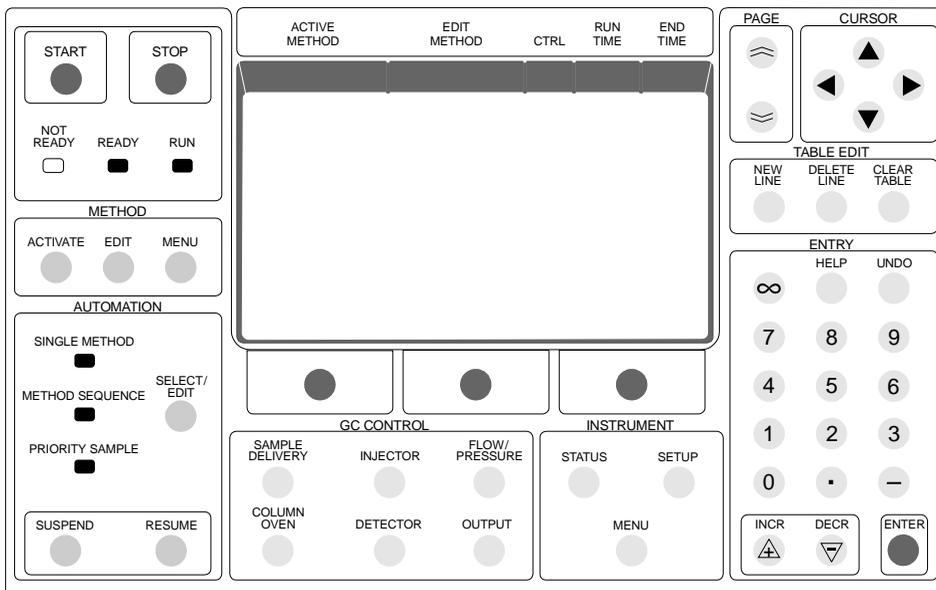
Each 3800 screen has a prompt line directly beneath the method parameters section. Based on the field the display cursor is currently on, the prompt line indicates the available range of values for this field.

The bottom line of the 3800 display is reserved for soft keys, i.e., specific functions that are active in certain 3800 screens. For example, the Column Oven method screen, below, contains Turn Oven On and Turn Oven Off softkeys to allow programming the column oven power on and off.

ACTIVE METHOD		EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1		Method 1		0.00	20.00
Set (°C): 50				Actual (°C): 50	
Stabilization Time (mi n): 2.00				Column Oven: 0n	
Column Oven, Page 1 of 2					
Step	Temp (°C)	Rate (°C/mi n)	Hold (mi n)	Total (mi n)	
Initial	50	-	2.00	2.00	
1	150	10.0	1.00	13.00	
2	250	20.0	5.00	23.00	
Turn Oven On		End Stabilization		Turn Oven Off	
○		○		○	

Note that the Status section of the display and the Method Parameters section are separated by a bold line.

The 3800 Keyboard

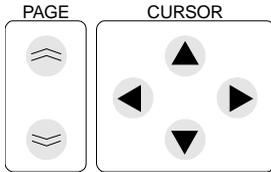


The 3800 keyboard is laid out around a multi-line display with the following sections:

<i>Section</i>	<i>Description</i>
<i>Page and Cursor</i>	Used to navigate around the display and move between screens.
<i>Table Edit</i>	Used to edit table entries, e.g., temperature programs or timed events.
<i>Entry</i>	Contains the numeric keys (0-9, decimal point, minus sign, and infinity), increment (INCR) and decrement (DECR) keys, and the ENTER key. The HELP key gives a context-sensitive explanation for the parameter that the cursor is on.
<i>Instrument</i>	Contains the Setup and Status keys that allow you to modify the GC configuration and view the status of the installed components.
<i>GC Control</i>	Allows immediate access to the injector, column, detector, etc. sections when either building a method or viewing the status.
<i>Automation</i>	Pressing Select/Edit will display a menu to set Automation Mode and modify automation parameters. Local Automation will preempt remote control by a Star Workstation.
<i>Method</i>	Allows you to edit or activate any of the eight available methods.
<i>Start/Stop</i>	Contains the START and STOP keys to manually start or stop a run. Not Ready, Ready, and Run lights indicate the GC status.

These sections are described in detail in the following paragraphs.

PAGE and CURSOR Keys



The PAGE and CURSOR keys allow you to move around the display and between screens.

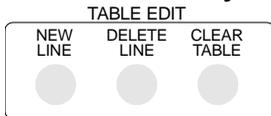
Pressing the UP arrow key backs up to the previous page; likewise, the DOWN arrow key advances to the next page. If no next or previous page exists, then pressing these keys has no effect.

If a screen contains parameters that can be modified, then a cursor will be present. The Cursor Arrow Keys are used to move the cursor from one parameter to the next. These four Cursor Arrow keys (up, down, left, and right) are oriented for the direction to move the cursor on the screen. The left and right keys are used primarily to move across a table of entries. If the cursor is not in a table, then pressing the right or left key has the same effect as moving the down or up key, e.g. the cursor moves down to the next or back to the previous parameter on the screen.

Use the cursor keys to navigate through a 3800 method. When a parameter is entered in a field and one of the cursor keys is pressed, the field is updated to the new parameter setting and the cursor moves to the next available field.

The up and down cursor keys can also be used to move through a list of numbered menu items. Selecting the menu item requires first placing the cursor on the appropriate item, then pressing ENTER to select. The same action can be accomplished by pressing the number of the menu item on the ENTRY keypad followed by pressing the ENTER key.

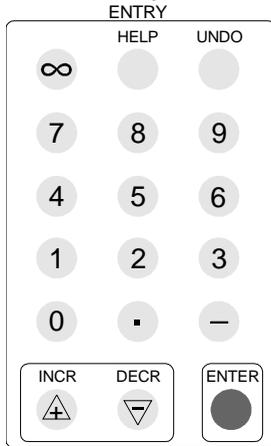
TABLE EDIT Keys



The TABLE EDIT section contains keys to edit method tables, and they are used only with pages that contain tables.

Key	Description
NEW LINE	Adds a new line after the selected line in the table. All tables have an initial series of default entries. A table is built by adding new lines to the initial line.
DELETE LINE	Deletes the selected line in the table. When a line is deleted, then the table reformats to move the succeeding rows up to fill the void if line 1 is already on the display, and move the preceding rows down if line 1 is not on the display.
CLEAR TABLE	Clears all entries in the table except for the initial line.

ENTRY Keys



The ENTRY section of the keypad is used to navigate around the 3800 keyboard and make parameter entries into the display fields.

Key	Description
Numeric	Allows entry of numeric values (0-9), including decimal points, negative numbers, and infinity. These keys also allow you to select a specific menu item by number.
HELP	Pressing this key will display a description of the parameter that the cursor is currently on.
UNDO	Pressing this key will reverse the last edited action and replace it with the previous parameter as long as the ENTER key has not been pressed.
INCR/DECR	Used to select a specific value when the choices are a defined set of possible values, e.g., detector attenuation. The INCR/DECR keys can also be used to increase or decrease a numeric parameter.
ENTER	Verifies the parameter that you entered on the keyboard or advances the cursor to the next parameter. If you press ENTER without changing the parameter, then its value remains unchanged, and the cursor advances to the next parameter. If the parameter is changed, pressing ENTER will implement the new value but not advance the cursor. ENTER can then be pressed a second time to advance the cursor.

After selecting the Edit Setup menu choice, you are presented with the following SETUP menu screen. Note that once you choose to Edit Setup, the 3800 will always reinitialize when you exit Setup. This is to check whether the 3800 configuration has changed. Note that the “Save and Exit” softkey is the only way to exit Edit Setup. Use the Page Up and Page Down keys to move between individual screens in Setup.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
EDIT INSTRUMENT SETUP MENU [1] Edit Time and Date [2] Edit Heated Zones [3] Edit EFC [4] Edit Column Parameters [5] Edit Sample Delivery [6] Edit Ethernet Setup [7] Edit Miscellaneous Setup Parameters <div style="border: 1px solid black; display: inline-block; padding: 2px 10px;">Save and Exit</div>				
○	○			○

Setup Parameters

TIME AND DATE

This is used to set the local time and date. Selection of the date format and the language for display is also performed here.

HEATED ZONES

The 3800 has six available heated zones, excluding the column oven (see figure below). These are numbered 1 - 6, with all zones having temperature programming capability and three (1 - 3) having cryogenic cooling capability. Typically, zones 1 - 3 are assigned to injectors and zones 4 - 6 to detectors. However, occasionally this guideline may change, particularly with valved systems.

Each installed 3800 option is identified by its location designated as “front”, “middle” and “rear” corresponding to the location of its controlling electronics. For example, if the 3800 is equipped with three detectors - an FID, TSD and ECD located from front to back on the top of the instrument - these would be identified as “front FID”, “middle TSD” and “rear ECD.” The SETUP screen below shows a typical 3800 configuration where four of the available heated zones are configured and two are free.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
EDIT HEATED ZONES				
Zone	Type	Temp Limit	Location	Coolant
Col Oven	Col Oven	450 (°C)	Col Oven	None
1	1079	450 (°C)	Front	LCO ₂
2	1079	450 (°C)	Middle	LCO ₂
3	None	450 (°C)	Rear	None
4	FID	450 (°C)	Front	None
5	FID	450 (°C)	Middle	None
6	None	450 (°C)	Rear	None
Save and Exit				
				

The following are the available choices of heated zone type, temperature limit, location, and coolant.

	Default	Range
Heat Zone		Col oven 1,2,3,4,5,6
Type	None	1177,1079,1041,1061,1093 TCD, FID, TSD, ECD, PPF Large Valve Oven, Small Valve Oven, Methanizer, SPT
Temp Limit (°C)	450 °C	50 - 450 °C ¹
Location	Front	Front, Middle, Rear
Coolant	None	None, LN ₂ , LCO ₂

EFC

The 3800 GC can be configured with either manual pneumatics or electronic flow control (EFC). Nine types of EFC modules exist, and each type is associated with a specific GC inlet system or detector. The Setup menu identifies each EFC type and its associated inlet system or detector by location (front, middle, rear). Below is a description of each EFC type, display, and associated parameters. When using AutoCalibrate for injector EFC, verify that the inlet pressure to the EFC module is at least 60 psi, a column and septa must be installed for this test.

¹ Large and small valve ovens are limited to 350°C. Micro-TCD is limited to 110°C.

Type 1 EFC This is the electronic flow control used for the CP-1177 and 1079 injectors, a back-pressure regulated system with a split flow controller.

Type 1 EFC has the following parameters:

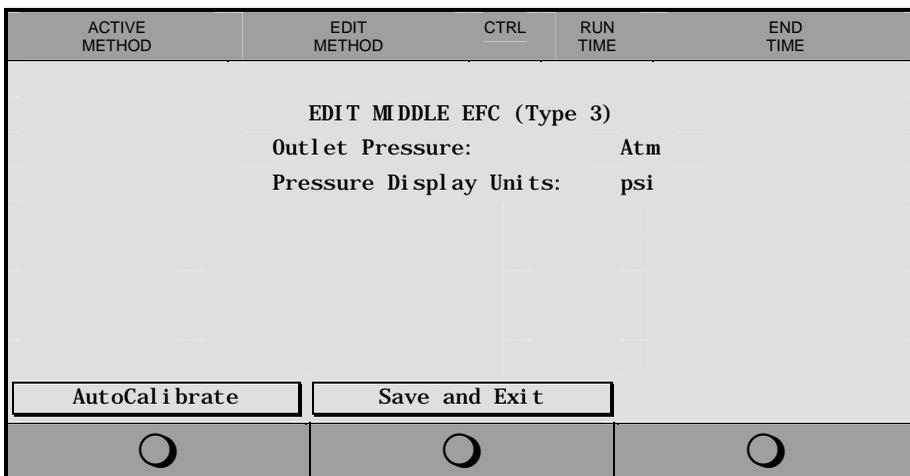
<i>For this Parameter ...</i>	<i>Enter...</i>
Outlet Pressure	Atm if the column vents to atmospheric pressure Vacuum if the column is connected to a vacuum source, e.g., a mass spectrometer
Pressure Display Units	psi, bar, kPa, depending on which pressure units that you want to display
Splitless Vent Flow	The flow the system will vent when the split valve is left closed for the duration of the run such as during an on-column injection. This flow is diverted before the injector and thus does not influence the splitless nature of the run. This entry is ignored during split and splitless runs in which the split vent is opened. This parameter should not be set to less than 20 mL/min.
Gas Saver Flow	The lowest system flow rate that will be maintained during the gas saver period. This has a range of 1-100 mL/min and defaults to 20 mL/min.
Gas Saver Timeout	A non-zero value entered for this parameter will cause the system to initiate a timer once a method is activated. If no system actions such as a run start or another method activation occurs before the Gas Saver Timeout is reached; then the system will reduce the flow through the system to the Gas Saver Flow. The GC will advance to a faulted state (all three LEDs will be lit). A method activation action will clear the Gas Saver Flow and bring the system to Ready for subsequent injections. A zero value entered for the Gas Saver Timeout will cause the Gas Saver Flow to be ignored and no Gas Saver actions will be performed

Type 1 EFC has the following softkeys:

<i>Press this softkey</i>	<i>If you want to...</i>
AutoCalibrate	Automatically calibrate the EFC system. When it is finished, a screen will display either a successful completion or detected problem message. No other GC functions can be performed during AutoCalibration. <i>Note that AutoCalibration should be done on a semiannual basis, or whenever an EFC module is changed.</i>
Septum Purge Calibrate	Calibrate the septum purge. After installing a new column, the septum purge should be calibrated. Set the desired column head pressure and press the "Enter" key. Then measure the actual septum purge flow rate and enter this value in the septum purge field. The septum purge flow rate is adjustable but is typically set at 3 - 5 mL/min.
Save and Exit	Save the changed parameters and reinitialize the 3800.

Type 3 and 4 EFC

Type 3 EFC is a flow controlled system that is used with the 1041 On-Column and 1061 Flash-Vaporization injectors. Type 4, used for valves, is a pressure controlled system with a flow controller in parallel with a pressure regulator. It can supply a rapid increase in inlet flow rate if the pressure drops suddenly, such as after a valve switch.



Type 3 and 4 EFC have the same SETUP screens.

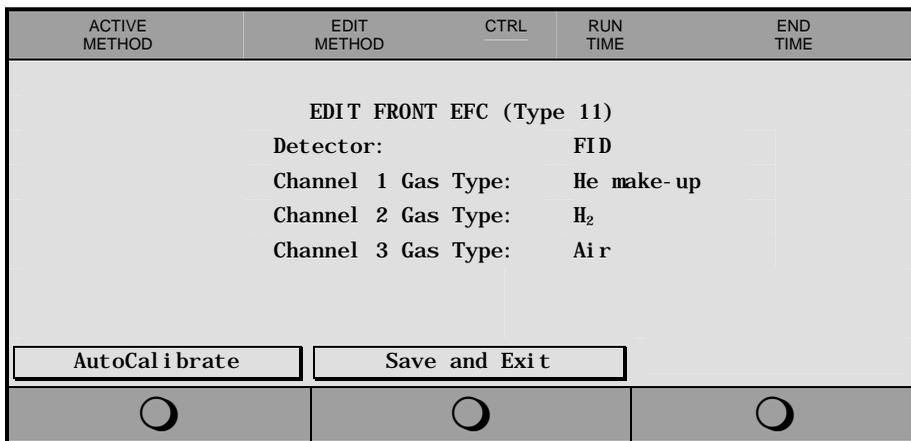
<i>For this Parameter ...</i>	<i>Enter...</i>
Outlet Pressure	Atm if the column exit is to atmospheric pressure Vacuum if the column is connected to a vacuum source, e.g., Varian Mass Spectrometer.
Pressure Display Units	psi, bar, kPa, depending on which pressure units that you want to display.

Type 3 and 4 EFC have the following softkeys:

<i>Press this softkey</i>	<i>If you want to...</i>
AutoCalibrate	Automatically calibrate the EFC system. When it is finished, a screen will display either a successful completion or detected problem message. No other GC functions can be performed during AutoCalibration. <i>Note that AutoCalibration should be done on a semiannual basis, or whenever an EFC module is changed.</i>
Save and Exit	Save the changed parameters and reinitialize the 3800.

Type 11 EFC

Type 11 EFC is a detector EFC module specifically designed for the Flame Ionization Detector (FID). It is a flow controlled system with three independent channels, channel 1 for make-up flow, channel 2 for hydrogen flow and channel 3 for air flow. Note that the only parameter than can be changed in the type 11 Setup screen is the choice of make-up gas. This can be set to Helium (He), Nitrogen (N₂) or Argon (Ar).



<i>For this Parameter ...</i>	<i>Enter...</i>
Channel 1 Gas Type	He, N ₂ or Ar.

Type 11 EFC has the following softkeys:

<i>Press this softkey</i>	<i>If you want to...</i>
AutoCalibrate	Automatically calibrate the EFC system. When it is finished, a screen will display either a successful completion or detected problem message. No other GC functions can be performed during AutoCalibration. <i>Note that AutoCalibration should be done on a semiannual basis, or whenever an EFC module is changed.</i>
Save and Exit	Save the changed parameters and reinitialize the 3800.

Type 12 EFC

Type 12 EFC is a detector EFC module specifically designed for the Thermionic Specific Detector (TSD). It is a flow controlled system with three independent channels, channel 1 for make-up flow, channel 2 for hydrogen flow and channel 3 for air flow. Note that the only parameter that can be changed in the type 12 Setup screen is the choice of make-up gas. This can be set to Helium (He), Nitrogen (N₂) or Argon (Ar).

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
EDIT FRONT EFC (Type 12) Detector: TSD Channel 1 Gas Type: He make-up Channel 2 Gas Type: H ₂ Channel 3 Gas Type: Air				
AutoCalibrate		Save and Exit		
○		○		○

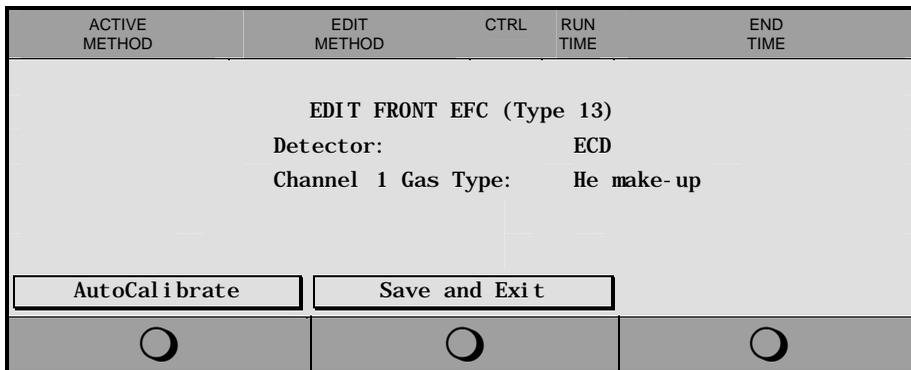
<i>For this Parameter ...</i>	<i>Enter...</i>
Channel 1 Gas Type	He, N ₂ or Ar.

Type 12 EFC has the following softkeys:

<i>Press this softkey</i>	<i>If you want to...</i>
AutoCalibrate	Automatically calibrate the EFC system. When it is finished, a screen will display either a successful completion or detected problem message. No other GC functions can be performed during AutoCalibration. <i>Note that AutoCalibration should be done on a semiannual basis, or whenever an EFC module is changed.</i>
Save and Exit	Save the changed parameters and reinitialize the 3800.

Type 13 EFC

Type 13 EFC is a detector EFC module designed for the Electron Capture Detector (ECD). It is a flow controlled system with one channel which supplies make-up gas. The gas type can be set to Nitrogen (N₂) or Argon (Ar).



<i>For this Parameter ...</i>	<i>Enter...</i>
Channel 1 Gas Type	He, N ₂ or Ar.

Type 13 EFC has the following softkeys:

<i>Press this softkey</i>	<i>If you want to...</i>
AutoCalibrate	Automatically calibrate the EFC system. When it is finished, a screen will display either a successful completion or detected problem message. No other GC functions can be performed during AutoCalibration. <i>Note that AutoCalibration should be done on a semiannual basis, or whenever an EFC module is changed.</i>
Save and Exit	Save the changed parameters and reinitialize the 3800.

Type 14 EFC

Type 14 EFC is a detector EFC module specifically designed for the Thermal Conductivity Detector (TCD). It is a flow controlled system with two independent channels, channel 1 for make-up flow and channel 2 for reference flow (or make-up flow if two capillary columns are used with the TCD). The gas type for both channels can be set to Helium (He), Nitrogen (N₂) or Argon (Ar).

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
EDIT FRONT EFC (Type 14) Detector: TCD Channel 1 Gas Type: He make-up Channel 2 Gas Type: He reference				
AutoCalibrate		Save and Exit		
○		○		○

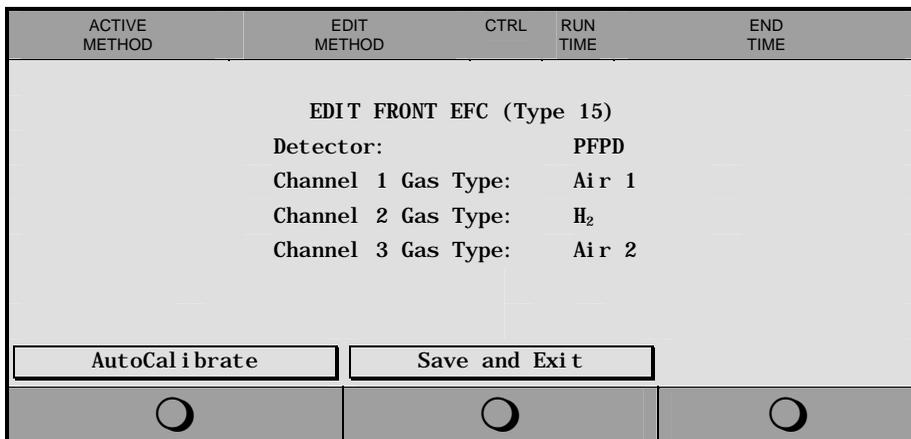
<i>For this Parameter ...</i>	<i>Enter...</i>
Channel 1 Gas Type	He, N ₂ or Ar. Make-up.
Channel 2 Gas Type	He, N ₂ or Ar. Make-up or reference.

Type 14 EFC has the following softkeys:

<i>Press this softkey</i>	<i>If you want to...</i>
AutoCalibrate	Automatically calibrate the EFC system. When it is finished, a screen will display either a successful completion or detected problem message. No other GC functions can be performed during AutoCalibration. <i>Note that AutoCalibration should be done on a semiannual basis, or whenever an EFC module is changed.</i>
Save and Exit	Save the changed parameters and reinitialize the 3800.

Type 15 EFC

Type 15 EFC is a detector EFC module specifically designed for the Pulsed Flame Photometric Detector (PFPD). It is a flow controlled system with three independent channels; channel 1 for air 1 flow, channel 2 for hydrogen flow and channel 3 for air 2 flow. There are no user selectable parameters with EFC type 15.

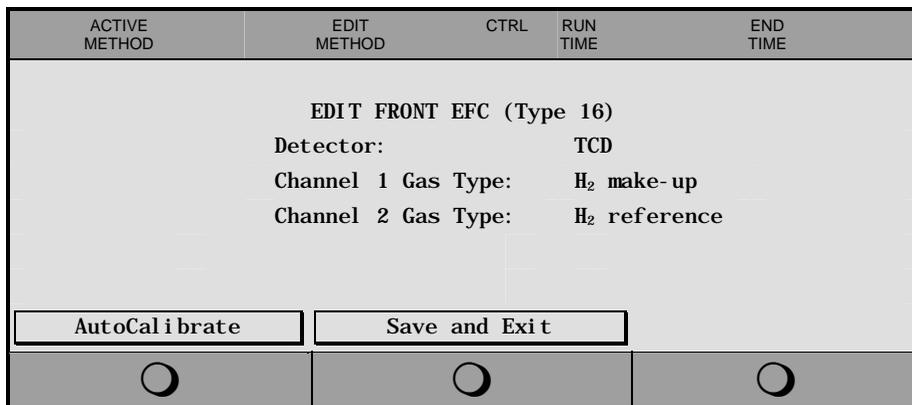


Type 15 EFC has the following softkeys:

<i>Press this softkey</i>	<i>If you want to...</i>
AutoCalibrate	Automatically calibrate the EFC system. When it is finished, a screen will display either a successful completion or detected problem message. No other GC functions can be performed during AutoCalibration. <i>Note that AutoCalibration should be done on a semiannual basis, or whenever an EFC module is changed.</i>
Save and Exit	Save the changed parameters and reinitialize the 3800.

Type 16 EFC

Type 16 EFC is a detector EFC module specifically designed for the Thermal Conductivity Detector (TCD) when used with Hydrogen gas. It is a flow controlled system with two independent channels, channel 1 for make-up flow and channel 2 for reference flow (or make-up flow if two capillary columns are used with the TCD). The gas type for either channel can be set to make-up or reference.



<i>For this Parameter ...</i>	<i>Enter...</i>
Channel 1 Gas Type	Make-up or reference
Channel 2 Gas Type	Make-up or reference

Type 16 EFC has the following softkeys:

<i>Press this softkey</i>	<i>If you want to...</i>
AutoCalibrate	Automatically calibrate the EFC system. When it is finished, a screen will display either a successful completion or detected problem message. No other GC functions can be performed during AutoCalibration. <i>Note that AutoCalibration should be done on a semiannual basis, or whenever an EFC module is changed.</i>
Save and Exit	Save the changed parameters and reinitialize the 3800.

Column Parameters

Column Parameters defines the carrier gas and the capillary column dimensions used with the specific sample introduction device (injector or valve). The location (front, middle, or rear) refers to the Sample Introduction device to which the column is connected. The carrier gas and column dimension information is used for EFC calculations. If a packed column is used, then "0" is set for length and ID. The parameters and their ranges are shown below:

	Default	Range
Location	Front	[Front, Middle, Rear]
Carrier Gas	He	[He, H ₂ , N ₂]
Length (meters)	30.00	[0 – 250.00]
ID (μm)	250	[0 - 999]

The following example is shown for a 3800 with two capillary columns installed.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
EDIT COLUMN PARAMETERS				
Location	Carrier	Length	ID	
Front	He	30.00	250	
Middle	He	30.00	320	
Rear	He	0.00	0	
<input type="button" value="Save and Exit"/>				

**Sample
Delivery
Setup**

This section has parameters for an autosampler, if installed, and valving.

Autosampler

In this section you configure which injectors you want the 8400 to injector into. You also select the syringe capacity.

Valves

This section configures the seven external events available on the 3800 that are typically used to control the CP-1177 and 1079 split valve with manual pneumatics (non-EFC) or actuators for sampling or switching valves. Each valve is identified in SETUP by a name that indicates its application. Names are selected using the INCR and DECR keys from a table of 22 names. When a name is chosen this also defines the default states of the valve. These states will be visible when the valve is being programmed in the valve table section of the 3800 method.

Below is an example of a 3800 equipped with a manual pneumatics 1079 injector, a CP-1177 injector and two automated gas sampling valves (GSV). Valve numbers 1 and 2 control the GSVs, and 3 and 4 control the CP-1177 and 1079 split valves. Each split valve type is identified based on the location of its associated injector.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
---------------	-------------	------	----------	----------

EDIT VALVES, Page 1 of 2

Number	Valve Type
1	Gas Sampling Valve
2	Gas Sampling Valve
3	Front Split Valve
4	Rear Split Valve

Save and Exit



The following table summarizes the various valve types, abbreviations, and associated default (de-energized) and energized states.

Valve Type	Abbreviation	Default State (-)	Energized State (+)
Front Split Valve	FSV	ON (split)	OFF (s/less)
Middle Split Valve	MSV	ON (split)	OFF (s/less)
Rear Split Valve	RSV	ON (split)	OFF (s/less)
Gas Sampling Valve	GSV	Fill	Inject
Liquid Sampling Valve	LSV	Fill	Inject
Sample	S	OFF	ON
Internal Standard	IS	OFF	ON
Surrogate Standard	SS	OFF	ON
Series Bypass	SB	Series	Bypass
Backflush to Detector	BD	Forward	Backflush
Backflush to Vent	BV	Forward	Backflush
Column Selection	CS	Column 1	Column 2
Injection + Backflush to Detector	IBD	Backflush	Inject
Injection + Backflush to Vent	IBV	Backflush	Inject
Alternate Injection	AI	Column 1	Column 2
Simultaneous Injection	SI	Fill	Inject
Methanizer Bypass	MB	Series	Bypass
Sample Preconcentration Trap	SPT	SPT Desorb	SPT Trap
Event A	EA	OFF	ON
Event B	EB	OFF	ON
Event C	EC	OFF	ON
Event D	ED	OFF	ON
None			

Ethernet Setup

If you are connecting your 3800 to a company network, you can enter an IP Address, Netmask, and Default Gateway provided by your company network administrator. If you are using Star WorkStation on an isolated network (3800 connected to PC only) Do Not enter values here, instead use Star WorkStation BOOTP to assign an IP Address to the 3800.

Instrument STATUS Key

The Instrument STATUS key allows you to view the current status of the GC components used in the active method. Several status screens are provided based on component type or GC channel (front, middle or rear). In addition, you can view the individual components' status in more detail by pressing the appropriate GC CONTROL keys (INJECTOR, DETECTOR, etc.).

Instrument component status is presented on these pages in the order: front, middle, and rear channels with each screen showing a set of injector, column, and detector status. The column oven temperature appears on all pages. The PAGE UP/DOWN keys can be used to move between the pages

or the CURSOR UP/DOWN keys can be used to scroll one line at a time. Status parameters appear in the same order as the sample flows through the system, i.e., Injector, Flow / Pressure, Column Oven, Detector, and Output. Local Automation status indicates the current automation status if a series of runs has been started from the local keyboard of the 3800.

The status page also indicates whether a component has failed to reach its setpoint or has a fault. If a setpoint is not reached, the message NOT READY is displayed next to the component, while if a fault is present the message FAULTED appears. The Column Oven field also indicates when it is stabilizing.

**Miscellaneous
Setup
Parameters**

- Is Ready-In a closed contact (Yes/No). Allows you to specify what switch state corresponds to External Device Ready.
- Enable check for (Front/Middle/Rear) FID flameout (Yes/No).

If you have an FID electrometer installed, this field allows the GC to ignore flameout test results. This could be used if you have a very low background system, or when the FID electrometer is used to collect signal from another low-background detector, such as a PID.

Ethernet Setup Ethernet address - can enter IP address, Netmask, and Default Gateway when provided by your network administrator. If using Star WorkStation allow the WorkStation BootP server to assign IP addresses.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
INSTRUMENT STATUS (FRONT), Page 1 of 3				
Component		Setpoint	Actual	
1079 Oven (°C)		50	50	
Column Flow (mL/min)			1.0	
Column Pressure (psi)		15.0	15.0	
Column Oven Temp (°C)		100	100	
FID Oven (°C)		300	300	
FID Output (mV)			8.25	
Software Ver.			View Faults	
○	○	○		

The Software Ver. Softkey displays the current version of installed software. The View Faults softkey displays the most recently detected fault conditions.

Instrument MENU Key

The Instrument MENU has four sub categories: Remote / Local, Standby Mode, 8400 Miscellaneous, and Column Mode. Each of these four items are discussed below.

The Remote / Local screen allows you to select between remote (Star Workstation) or local control. Disabling remote control prevents a remote user from gaining control of the instrument, which may be an important safeguard when conducting instrument maintenance. This screen also displays the Ethernet Address, IP address, and Alias assigned by the remote user. This information cannot be configured locally on the GC but has to be assigned from the Star Workstation. Refer to the Communications section of this manual for more detailed information on configuring the 3800 with a Star Workstation.

The screen below shows a typical configuration where the 3800 is under remote control. To disable remote control, move the cursor to the Enable field and toggle to "Prevent" using the INCR or DECR key.

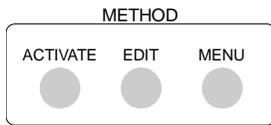
ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
REMOTE / LOCAL CONTROL				
<input type="button" value="Enable"/>				
Ethernet Address: XX: XX: XX: XX: XX: XX IP Address: XXX. XXX. XX. XXX Alias: 3800A				

The Standby Mode allows you to set a standby temperature for the column oven. This standby temperature will be invoked after the timer counts to zero, provided another run is not started before the timer has timed out. Once the timer had timed out a method activation will restore the column temperature to the method temperature. A timer value of zero disables the standby temperature function.

8400 Miscellaneous allows you to: turn the autosampler buzzer On/Off, change the plunger stroke warning limit, turn the vial detector On/Off, and Find home at Start. On page two you can change the needle depth into the injector and change the injection speed. These parameters are also discussed in more detail in the 8400/8410 Operator Manual.

Column mode defaults to Standard. If this 3800 is part of a GC/MS system Rapid MS mode can be selected from MS Workstation. Once selected, the linear velocity will not be displayed and the column dimensions will be dashed out in View Setup Column parameters.

METHOD



The method keys allow you to activate, edit, and copy methods. Because you can edit one method while another is active, the status bar on top of the screen always identifies both the active method and the method available to edit.

ACTIVATE Method Key

The Activate Method key allows you to activate one of eight methods. Select the method by using the INCR and DECR keys to toggle between method numbers then press the “Activate Now” softkey to activate the method. The Activate Now softkey is disabled while local automation is running. Below is an example of the Activate Method screen.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
ACTIVATE METHOD				
	Last Modified		Last Modified	
Method 1	XX/XX/XX XX.XX	Method 5	XX/XX/XX XX.XX	
Method 2	XX/XX/XX XX.XX	Method 6	XX/XX/XX XX.XX	
Method 3	XX/XX/XX XX.XX	Method 7	XX/XX/XX XX.XX	
Method 4	XX/XX/XX XX.XX	Method 8	XX/XX/XX XX.XX	
Activate Method:		Method 1		
<div style="border: 1px solid black; padding: 2px; display: inline-block;">Activate Now</div>				

EDIT Method Key

The EDIT key allows you to select one of the eight methods to edit. The active method is the default selection to edit, which allows quick access to modifying the active method. The components available to edit are those that have been configured earlier in the Instrument SETUP section. Below is an example of the edit screen showing Method 1 as the active method. If you want to edit another method, use the INCR/DECR keys to toggle to another method, then press ENTER. The Injector Method screen will appear first. If there is more than one Injector installed, a menu page will appear instead, so that the desired injector may be selected for editing. Method components appear in the following order: Injector, Flow/Pressure, Column Oven, Detector, and Output. If you want to go directly to one of the method sections, press the appropriate GC Control key. To access the Valve Table or local 8200 AutoSampler method, press the Sample Delivery key.

Note: The method parameters that appear on the display represent the current EDIT method and not necessarily the ACTIVE method. The edited method parameters will only be implemented when activated. Always remember to reactivate the active method if you have made changes to it.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
EDIT METHOD				
Edit Method: Method 1				
				

**Method
MENU Key**

The method MENU key allows you to select less frequently accessed method operations: method copy, preset to defaults, and lock/unlock methods. A menu screen is presented when this key is pressed to allow selection of one of these options.

**Method
COPY**

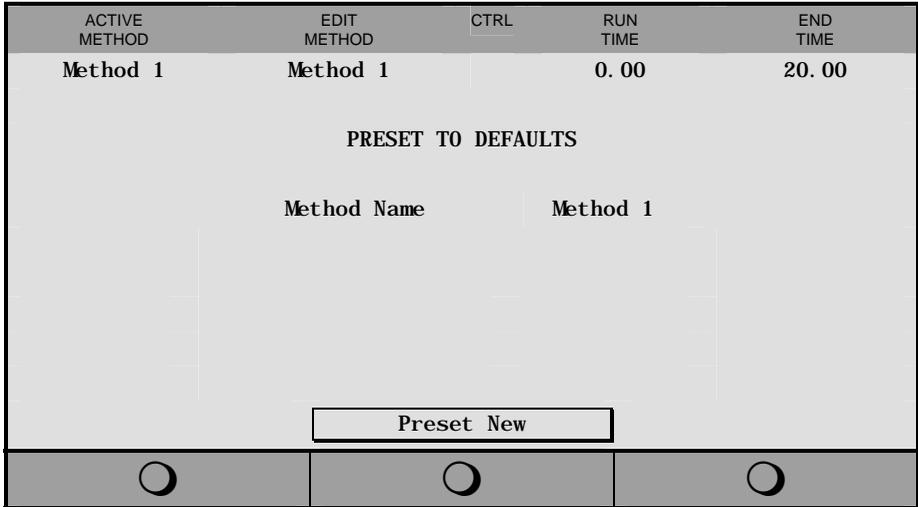
This allows you to copy the contents of one method to another. However, a method that is currently running cannot be overwritten, i.e., another method cannot be copied to it. Follow the instructions shown in the screen below, then press the COPY NOW softkey to implement the copy method action.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
COPY METHOD				
Copy From	Method 1	Copy To	Method 2	
COPY NOW				



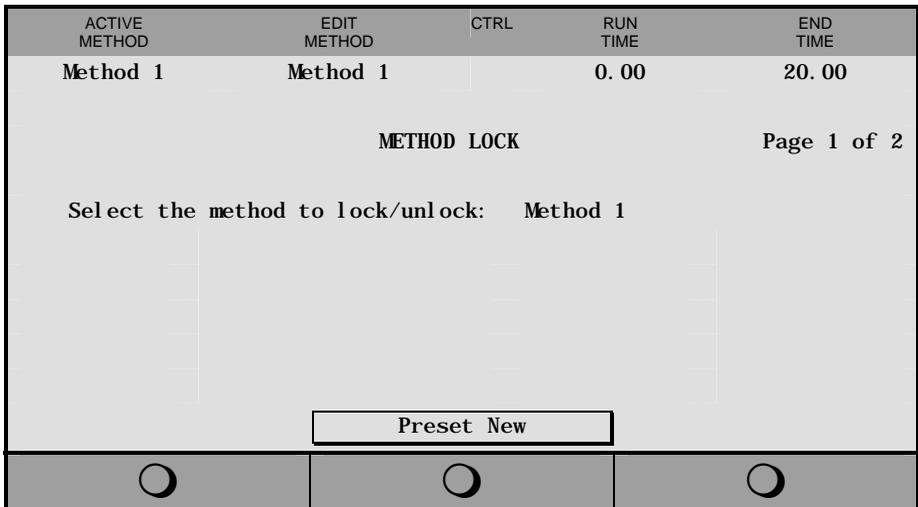
Preset to Defaults

This allows you to set all of the parameters in the selected method back to default values.



Lock/Unlock Methods

This allows you to prevent unauthorized changes to a method.

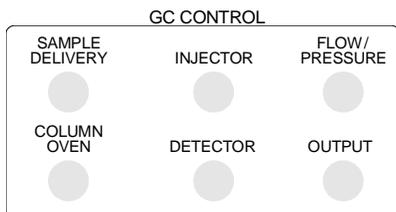


ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
METHOD UNLOCK				Page 2 of 2
Method:	Method 1		Status:	Unlocked
Password: XXXXXX				
Note: Enter password and press "Toggle Lock" to lock/unlock the selected method.				
<div style="border: 1px solid black; display: inline-block; padding: 2px 10px;">Preset New</div>				

A method may be locked by pressing the Menu Key, selecting Lock/Unlock Method, and entering the desired method number. Note that Method 8 cannot be locked, as it is needed for Workstation control. When the Password field appears, type a number between 0 and +999.999, then press the Toggle Lock softkey. The method status will change from Unlocked to Locked.

To unlock a locked method, follow the same procedure, typing in the password used when the method was locked, then press Toggle Lock.

GC CONTROL



The GC Control keys are used to access each method section directly when either checking the GC status or editing a method.

GC Control screens for each component have the following format:

- Status
- Name and Location
- Method Section
- Softkey Commands

Pressing a GC Control key will cause the appropriate screen to be displayed. If more than one component of that type is installed on the GC, e.g., two injectors, you will be asked to choose which one you wish to program or check the status of.

SAMPLE DELIVERY Key

The Sample Delivery section contains the status and method instructions for programming the valve options configured in Instrument SETUP, including manual control of the 1177 or 1079 split valve. In addition, this section allows programming the 8200 or 8400 AutoSampler, if it is being controlled by the local 3800 keyboard.

If more than one SAMPLE DELIVERY device is installed, then you are presented with a selection menu.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
SAMPLE DELIVERY				
[1.] 8200				
[2.] Valve Table:				
				

Valve Table

The following method screens show an example of a 3800 configuration with two automated gas sampling valves installed in a valve oven and two manual pneumatics 1177 or 1079 injector split valves. Note that if an 1177 or 1079 is equipped with EFC, the split state is controlled in the injector section of the method and is not programmed in the Sample Delivery Section of the method.

The position of the valve is indicated by its energized (+) or de-energized (-) state. When the cursor is moved to a specific table field, the prompt line below the table indicates the full name of the valve and a description of the state of the valve, e.g., if the cursor were moved to the first table entry (initial time) of the GSV in position 1, the prompt line indicates that the valve is in the FILL position, which is equivalent to the de-energized state (-). The

status line above the table indicates the current state of all of the valves in the valve table.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME			
Method 1	Method 1		0.00	20.00			
Valve: State 4 RSV ON	1 GSV Fill 5 None	2 GSV Fill 6 None	3 FSV ON 7 None				
VALVE TABLE							
Time	1 GSV	2 GSV	3 FSV	4 RSV	5 None	6 None	7 None
Initial	-	-	-	-	-	-	-
0.01	+	+	+	+	-	-	-
0.75	-	-	-	-	-	-	-
○		○			○		

Below is the complete set of valve names and associated states that are available for programming.

Valve Type	Abbreviation	Default State (-)	Energized State (+)
Front Split Valve	FSV	ON (split)	OFF (s/less)
Middle Split Valve	MSV	ON (split)	OFF (s/less)
Rear Split Valve	RSV	ON (split)	OFF (s/less)
Gas Sampling Valve	GSV	Fill	Inject
Liquid Sampling Valve	LSV	Fill	Inject
Sample	S	OFF	ON
Internal Standard	IS	OFF	ON
Surrogate Standard	SS	OFF	ON
Series Bypass	SB	Series	Bypass
Backflush to Detector	BD	Forward	Backflush
Backflush to Vent	BV	Forward	Backflush
Column Selection	CS	Column 1	Column 2
Injection + Backflush to Detector	IBD	Fill + Backflush	Inject
Injection + Backflush to Vent	IBV	Fill + Backflush	Inject
Alternate Injection	AI	Column 1	Column 2
Simultaneous Injection	SI	Fill	Inject
Methanizer Bypass	MB	Series	Bypass
Sample Preconcentration Trap	SPT	SPT Desorb	SPT Trap
External Event A	EA	OFF	ON
External Event B	EB	OFF	ON
External Event C	EC	OFF	ON
External Event D	ED	OFF	ON
None			

INJECTOR Key

The Injector section of the method can contain liquid injectors, valve ovens or an SPT (Sample Preconcentration Trap). The Sample Preconcentration Trap has a separate user's manual.

Liquid Injectors

Injector	Description
CP-1177 Split/Splitless Capillary	Dedicated Split/Splitless isothermal injector. Capillary columns only.
1079 Universal Capillary	Split, splitless, and on-column injector. Temperature programmable with cryogenic capability Capillary columns only
1041 On-column	Isothermal, largebore and packed columns
1061 Flash Vaporization	Isothermal, largebore and packed columns

The 1041 and 1061 injectors can accommodate either packed columns, using optional packed column adapter kits or large bore capillary (0.53 mm ID) columns. However, no differentiation is made in the INJECTOR section. The 3800 method regards both 1041 and 1061 as isothermal heated zones.

You will be presented with a menu when the INJECTOR key is pressed if more than one injector is installed and configured in Instrument SETUP. For example, a 3800 with a CP-1177 in the front, 1079 in the middle and a 1041 in the rear will display the following.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
INJECTOR MENU				
[1.] Front 1177:				
[2.] Middle 1079				
[3.] Rear 1041				



The Injector Component Screen (below) is displayed once you select from the above Injector menu or if only one injector installed. This screen contains the Injector Status information [Setpoint and Actual Temperature, Split State (if EFC equipped)], Injector Component Method instructions, a prompt line, and softkeys.

CP-1177 Injector

The CP-1177 method has two pages:

Page	Description
1	Temperature control
2	Split state programming (if type 1 EFC is installed) If the CP-1177 has manual pneumatics, then the split valve is programmed in the valve table located in SAMPLE DELIVERY.

The following are typical method parameters for a CP-1177.

Temperature is entered in page 1.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 250		Actual (°C): 250		
FRONT CP- 1177, Page 1 of 2				
1177 Oven Power:			ON	
1177 Temperature (°C)			250	
<input type="radio"/> <input type="radio"/> <input type="radio"/>				

	Default	Range
Temperature	<u>50</u>	[50 - 450]
Injector On/Off	<u>Off</u>	[On/Off]

A CP-1177 equipped with Electronic Flow Control includes a second page for programming the split state and split ratio. If the split state is ON, then the sample is split according to the split ratio specified. If the split state is OFF, then the entire sample enters the column.

The example below is for a splitless injection. At the initial time (prior to run start), the Split State is OFF indicating that when the sample is injected the entire sample vapor should enter the column. After 0.75 minutes, the split state is switched to ON with a 100:1 split ratio.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME												
Method 1	Method 1		0.00	20.00												
Set (°C): 50		Actual (°C): 50	Split Ratio: OFF													
FRONT 1177, Page 2 of 2.																
<table border="1" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th>Time</th> <th>Split State</th> <th>Split Ratio</th> </tr> </thead> <tbody> <tr> <td>Initial</td> <td>OFF</td> <td>OFF</td> </tr> <tr> <td>0.75</td> <td>ON</td> <td>100</td> </tr> <tr> <td></td> <td></td> <td></td> </tr> </tbody> </table>					Time	Split State	Split Ratio	Initial	OFF	OFF	0.75	ON	100			
Time	Split State	Split Ratio														
Initial	OFF	OFF														
0.75	ON	100														
○		○		○												

	<i>Default</i>	<i>Range</i>
Time (min)	Initial	[0.00 - 999.99]
Split State	Off	[On/Off]
Split Ratio	100	[Off, 1 to 10,000]

If the injector is equipped with manual pneumatics, then the injector split state is programmed in the VALVE TABLE of the SAMPLE DELIVERY section.

1079 Injector

The 1079 has three pages:

Page	Description
1	Temperature control and programming
2	Oven power and cryogenic parameters (if configured in Setup)
3	Split state programming (if type 1 EFC is installed) If the 1079 has manual pneumatics, then the split valve is programmed in the valve table located in SAMPLE DELIVERY.

The following are typical method parameters for a 1079 with cryogenics and temperature programming.

Temperature programs are entered in page 1.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 50	Actual (°C): 50		Split Ratio: OFF	
FRONT 1079, Page 1 of 3				
Step	Temp (°C)	Rate (°C/min)	Hold (min)	Total (min)
Initial	50	-	0.00	0.00
1	200	150	15.00	16.00

	Default	Range
Step	Initial	[1 - 4]
Initial temperature (°C):	<u>50</u>	[-99 - 450°C]
Rate (°C/min):	<u>X</u>	[1 - 200]
Hold Time	<u>X</u>	[0.01 - 999.99]
Total	<u>X</u>	[0.01 - 999.99]

Oven power and cryogenics are entered in page 2.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 50		Actual (°C): 50	Split Ratio: OFF	
FRONT 1079, Page 2 of 3				
1079 Oven Power			On	
Coolant ON/OFF			On	
Enable Coolant at (°C)			250	
Coolant Time-out (min)			5	

	<i>Default</i>	<i>Range</i>
1079 Oven Power	Off	[On/Off]
Coolant ON/OFF	On	[On/Off]
Enable Coolant at (°C)	<u>250</u>	[30 - 450]
Coolant Time-out (min):	<u>20</u>	[0.00 - 999]

The coolant enable temperature is the temperature the coolant valve turns on at when the zone is cooling down.

Coolant time-out is a safety device to save coolant after a specified time if either (1) a zone fails to reach its set temperature or (2) it reaches the set temperature and the GC does not go into run. The valve will be turned off if the coolant time-out is reached, and no further coolant will be applied. A value of zero disables the coolant time-out function.

A 1079 equipped with Electronic Flow Control includes a third page for programming the split state and split ratio. If the split state is ON, then the sample is split according to the split ratio specified. If the split state is OFF, then the entire sample enters the column.

The example below is for a splitless injection. At the initial time (prior to run start), the Split State is OFF indicating that when the sample is injected all the sample vapor should enter the column. After 0.75 minutes, the split state is switched to ON with a 100:1 split ratio.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME												
Method 1	Method 1		0.00	20.00												
Set (°C): 50		Actual (°C): 50		Split Ratio: OFF												
FRONT 1079, Page 3 of 3.																
<table border="1"> <thead> <tr> <th>Time</th> <th>Split State</th> <th>Split Ratio</th> </tr> </thead> <tbody> <tr> <td>Initial</td> <td>OFF</td> <td>OFF</td> </tr> <tr> <td>0.75</td> <td>ON</td> <td>100</td> </tr> <tr> <td></td> <td></td> <td></td> </tr> </tbody> </table>					Time	Split State	Split Ratio	Initial	OFF	OFF	0.75	ON	100			
Time	Split State	Split Ratio														
Initial	OFF	OFF														
0.75	ON	100														
																

	<i>Default</i>	<i>Range</i>
Time (min)	Initial	[0.00 - 999.99]
Split State	Off	[On/Off]
Split Ratio	100	[Off, 1 to 10,000]

If the injector is equipped with manual pneumatics, then the injector split state is programmed in the VALVE TABLE of the SAMPLE DELIVERY section.

***Isothermal
(1041 / 1061)
Injector***

The 1041 and 1061 isothermal injectors display one screen to set the temperatures or view the status.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 250		Actual (°C): 250		
MIDDLE 1041				
1041 Oven Power:			ON	
1041 Temperature (°C):			250	
				

	<i>Default</i>	<i>Range</i>
1041 Oven Power:	Off	[On/Off]
1041 Temperature (°C):	50	[50 - 450 °C]

Valve Oven

The Valve Oven is an optional, isothermal heated zone that is specified in Instrument Setup as “Front, Middle, or Rear” and either Large Valve Oven or Small Valve Oven. Separate method screens are available for each configured valve oven.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 200		Actual (°C): 200		
FRONT SMALL VALVE OVEN				
Valve Oven:			ON	
Valve Oven Temp (°C):			200	

FLOW / PRESSURE Key

The FLOW / PRESSURE section displays the Electronic Flow Control status or method parameters for injectors and valves controlled by EFC modules. Three types of EFC modules exist; all are described in Instrument SETUP of this section and in more detail in the Sample Introduction Section of the manual.

If more than one EFC module is installed, then a menu screen is displayed showing the module types and injector or valve location associated with the device each controls. Select the module that you want to view the status of/or change method parameters.

Type 1 EFC

Type 1 EFC, used with either the CP-1177 or 1079 injector, allows you to maintain a constant pressure or to build a pressure program to maintain constant column flow while temperature programming the column oven.

An example of a Type 1 EFC method is shown below. The status field displays the column head pressure (in the units chosen in Setup), calculated column flow rate, calculated column linear velocity, total flow and split ratio status. Total flow represents the column flow septum purge flow and the split vent flow. The split ratio status is either off if the split state is set to OFF, or a whole number. Additionally, the table below is a typical pressure program used in conjunction with a column oven temperature program to maintain constant flow.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Col Press (psi): 10.0		Col Flow (mL/min): 2.0		
Linear Vel. (cm/sec): 3.5		Total Flow (mL/min): 50		
Split Ratio: Off				
FRONT EFC Type 1 Page 1 of 2				
Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	10.0	-	0.75	0.75
1	20.0	2.00	2.00	5.75
2	35.0	5.00	5.25	14.00
				

Constant Flow Programming

The second page of the TYPE 1 EFC allows constant flow mode to be selected. Selecting Constant Flow will allow you to set a Pressure Pulse program accessed via the Pressure Pulse softkey that will be displayed once you have selected Constant Flow. When Constant Flow Programming is enabled, a pressure program based on the column parameters and the column oven temperature program will be built whenever the method is activated. If the desired column flow, or the column oven temperature program is changed, the method must be reactivated to derive a new pressure program for these new conditions.

After activating your method, you may disable Constant Flow Programming. The calculated pressure program will be retained and may be edited. If you edit the calculated pressure program, you should make sure that Constant Flow Programming is disabled on Page 2. Failure to do so will result in your edits to the pressure program being overwritten when the method is reactivated.

Gas Saver

For type 1 EFC a specific Gas Saver Flow and a timeout period may be set. **A time entry of 0 turns the Gas Saver feature off.** If a non-zero time is entered, the Gas Saver timer will start incrementing upon a method activation. If a run is not started or another method not activated before the timer expires, the set Gas Saver flow rate will be started. A message will be logged in the instrument log. **The fault is cleared by activating a method.** EFC setup for type 1 modules (CP-1177 and 1079 injectors) also contains a septum purge calibration and S/L Vent Flow. Septum purge calibration involves measuring a flow rate at a set pressure and entering those values into the system to correctly calculate the settings for split and column flow. The S/L Vent Flow provides a vent purge for runs in which the split valve remains closed for the duration of the run.

All EFC modules can be calibrated, if necessary, using the Autocalibrate softkey. This procedure should be carried out every six months, or when a new EFC module is installed. Failure to AutoCalibrate may cause a mismatch between set and actual flowrates resulting in a Not Ready condition in the CP-3800.

Type 3 EFC

Type 3 EFC, used with the 1041 and 1061 injectors, allows you to set a constant column flow or to program the column flow. An example of a Type 3 EFC method is shown below. The status field displays the column pressure (in the units chosen in Setup), actual flow rate, calculated linear velocity. The table below is the method parameters to establish the carrier flow into the injector/column system.

ACTIVE METHOD		EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1		Method 1		0.00	20.00
Col Press (psi): 5.0		Act Flow (mL/min): 10.0			
MIDDLE EFC, Type 3					
Step	Fl (mL/min)	Rate (mL/min/min)	Hold (min)	Total (min)	
Initial	10.0	-	20.00	20.00	
○		○		○	

Type 4 EFC

Type 4 EFC is used with valved systems where the inlet head pressure will typically be kept constant, but the total flow can be changed to maintain this head pressure in the event of a sudden drop in system pressure (as is usual when a sample at atmospheric pressure is loaded into the system). Pressure programming the inlet head pressure, similar to type 1 EFC, is also possible to maintain a constant column flow rate during a column oven temperature program. The programming of type 4 EFC is done on 2 pages (see below). Page 1 includes a ramp table for programming the inlet pressure. Page 2 is a timed events table for setting the total flow rate. In this example the column head pressure is held constant during the analytical run, but the total flow into the system is increased significantly at the beginning of the run to allow the system to respond more rapidly to pressure transients by sampling valve actuation.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Col Press (psi): 10.0		Col Flow (mL/min): 15		
Linear Vel. (cm/sec): 125		Total Flow (mL/min): 30		
REAR EFC TYPE 4, Page 1 of 2				
Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	10.0	-	20.00	20.00
				

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Col Press (psi): 10.0		Column Flow (mL/min): 15		
Linear Vel. (cm/sec): 125 cm/sec		Total Flow (mL/min): 30		
REAR EFC Type 4, Page 2 of 2				
Time	Total Flow (mL/min)			
Initial	30			
0.00	100			
0.50	30			
				

COLUMN OVEN Key

The COLUMN OVEN section allows you to establish the column oven temperature, build a temperature program, or view the column oven status. The status field for the COLUMN OVEN indicates the programmed set temperature, the actual measured temperature, the set or remaining stabilization time and whether the column oven is turned on or off. Stabilization time is the period the column oven will equilibrate for before allowing the system to go READY. It will count down from the set value then display 0.00 min when the stabilization period is over. The TURN OVEN ON and the TURN OVEN OFF softkeys are used to turn the column oven heater and fan OFF and ON. The END STABILIZATION softkey is used to bypass the remainder of the stabilization time.

Similar to other programming tables, the initial or isothermal line will always appear on the first line of the table. You can then build a temperature program by adding new lines. Below is a column oven program with an initial temperature, initial hold time, programmed ramp, and final hold. Note that the temperature range of the column oven is -99 to 450 °C and that the allowable ramp rates are from 0.1 to 100°C/min.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 50		Actual (°C): 50		
Stabilization Time (mi n): 2.00		Column Oven: 0n		
COLUMN OVEN, Page 1 of 2				
Step	Temp(°C)	Rate(°C/mi n)	Hold (mi n)	Total (mi n)
Initial	50	-	2.00	2.00
1	150	10.0	1.00	13.00
2	250	20.0	5.00	23.00
<div style="display: flex; justify-content: space-around; margin-top: 10px;"> <div style="border: 1px solid black; padding: 5px; width: 30%;">Turn Oven ON</div> <div style="border: 1px solid black; padding: 5px; width: 30%;">End Stabilization</div> <div style="border: 1px solid black; padding: 5px; width: 30%;">Turn Oven Off</div> </div>				
<div style="display: flex; justify-content: space-around; margin-top: 10px;"> <div style="width: 30%; text-align: center;">○</div> <div style="width: 30%; text-align: center;">○</div> <div style="width: 30%; text-align: center;">○</div> </div>				

	<i>Default</i>	<i>Range</i>
Step	Initial	[1 - 8]
Initial temperature (°C):	50	[-99 - 450°C]
Rate (°C/min):	X	[0.1 - 100.0]
Hold Time	20.00	[0.01 - 999.99]

A second page allows establishing optional column coolant parameters and the stabilization time.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 50			Actual (°C): 50	
Stabilization Time (min): 2.00			Column Oven: On	
COLUMN OVEN, Page 2 of 2				
Stabilization Time (min)			2.00	
Coolant On/Off			On	
Enable Coolant at (°C)			50	
Coolant Time-out (min)			20.00	
○	○		○	

	<i>Default</i>	<i>Range</i>
Coolant ON/OFF	Off	[On/Off]
Activate Coolant at (°C)	50	[30 - 450]
Coolant Time-out (min):	20.00	[0.00 - 999]
Stabilization Time (min)	0.50	[0.00 - 10.00 min]

DETECTOR Key

The DETECTOR section allows you to modify the detector method parameters and view the status of individual detectors. If more than one detector is installed, then pressing DETECTOR will prompt you to select from a menu of the installed detectors. The menu screen indicates the name of the detector and its location.

Below is an example for a Flame Ionization Detector. Further details for programming other detectors can be found in the detector section of this manual. Each detector method consists of three or four pages. Page one includes initial parameters for the detector oven temperature, turning on the detector oven power and electronics, and selecting the range. Page two establishes time programs to either change the range or Autozero the detector at specific times. The adjustments page or pages are accessed by pressing the adjustments softkey. For the FID, the first page of adjustments allows you to select between a fast or slow detector time constant. The normal setting for this parameter is fast.

If detector EFC is installed, then page 2 of adjustments allows setting the appropriate flow rates for an FID.

FID (Flame Ionization Detector)

The screen below indicates the set and actual detector oven temperature and the FID analog signal level.

The method parameters, default settings, and range are:

	Default	Range
Oven Power	Off	[On/Off]
Temperature (°C)	50	[50 - 450 °C]
Electronics	Off	[On/Off]
Range	12	[9, 10, 11, 12]

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 300	Actual (°C): 300		Signal (mV): 5.15	
FRONT FID, Page 1 of 2				
	Oven Power:		0N	
	Temperature (°C):		300	
	Electronics:		0N	
	Range:		12	
IGNITE		AUTOZERO		ADJUSTMENTS
○		○		○

The IGNITE softkey applies power to the FID ignitor filament for 5 seconds. Note that there is a flame re-ignition diagnostic which re-lights the FID if a flame-out is detected. The AUTOZERO softkey applies an immediate zeroing of the detector signal. Note that this is a single Autozero event and not a continuous Autozero.

Press the Page Down key to access the time programming events.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME												
Method 1	Method 1		0.00	20.00												
Set (°C): 300 Actual (°C): 300 Signal (mV): 5.15																
FRONT FID Page 2 of 2.																
<table border="1" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th>Time</th> <th>Range</th> <th>Autozero</th> </tr> </thead> <tbody> <tr> <td>Initial</td> <td>12</td> <td>NO</td> </tr> <tr> <td>5.00</td> <td>12</td> <td>YES</td> </tr> <tr> <td>6.50</td> <td>11</td> <td>NO</td> </tr> </tbody> </table>					Time	Range	Autozero	Initial	12	NO	5.00	12	YES	6.50	11	NO
Time	Range	Autozero														
Initial	12	NO														
5.00	12	YES														
6.50	11	NO														
IGNITE		AUTOZERO		ADJUSTMENTS												
○		○		○												

The parameters, default settings and range are:

	Default	Range
Time	Initial	[0.00 - 999.99]
Range	12	[9, 10, 11, 12]
A/Z	No	[yes/no] - set to yes at initial time means that FID A/Z is on continuously before the run starts.

Press the “Adjustments” softkey to access FID time constant setting.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 300 Actual (°C): 300 Signal (mV): 5.15				
FRONT FID ADJUSTMENTS, Page 1 of 2				
Time Constant: Fast				
<div style="border: 1px solid black; padding: 2px; display: inline-block;">Clear AUTOZERO</div>				
○		○		○

Page two, on GCs purchased with detector EFC parameters, is accessed by pressing the PAGE DOWN key.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Make-up Flow (ml/min): 25 H ₂ Flow (ml/min): 30				
Air Flow (ml/min): 300				
FRONT FID ADJUSTMENTS, Page 2 of 2				
Make-up Flow (ml/min)			25	
H ₂ Flow (ml/min)			30	
Air Flow (ml/min)			300	

The upper portion of the screen indicates the actual flow rates for the various detector gases. The method parameters, default settings and range are:

	<i>Default</i>	<i>Range</i>
Make-up Flow	25 ml/min	0 - 50 ml/min
H ₂ Flow	30 ml/min	0 - 50 ml/min
Air Flow	300 ml/min	0 - 500 ml/min

METHANIZER

A methanizer is an option that converts carbon monoxide and carbon dioxide to methane. It is associated with a specific FID detector; consequently, it is accessed in the DETECTOR section. A methanizer is presented as one of the detector options in the detector menu screen and is identified by location as “front”, “middle, and “rear”. Below is a sample screen for a Methanizer.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 380		Actual (°C): 380		
REAR METHANIZER				
Methanizer Oven Temperature			ON	
			380	

OUTPUT Key

The OUTPUT section configures the analog output ports to send an analog signal to external integrators or data systems that have internal analog to digital conversion. The 3800 three analog output ports which can be configured to any of the installed detectors.

The front, middle, and rear detectors are by default associated with analog signals A, B, and C, respectively (see below).

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
OUTPUT MENU [1] Program Analog Output Port A [2] Program Analog Output Port B [3] Program Analog Output Port C				

By selecting Analog Output A (above), the following screen appears. You can time program the port to switch the detector signal source or attenuation during a run. The attenuation defaults to 1 and should not be changed if the signal is going to an integrator or data system.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Detector: Front: FID Middle: TSD Rear: FID				
ANALOG OUTPUT PORT A				
Time		Signal Source	Attenuation	
Initial		Front Detector	1	
10.00		Middle Detector	1	

Sample Introduction

Sample introduction devices refer to the hardware used to introduce the sample into the Gas Chromatograph. These include liquid injectors, gas sampling valves and liquid sampling valves. This section also covers the pneumatic systems associated with the various sample introduction systems. The various pneumatic systems are described with the individual sample introduction systems.

Liquid Injectors

There are four liquid injectors available on the 3800 Gas Chromatograph:

1. The **CP-1177 Split/Splitless Capillary Injector** is optimized especially for split and splitless injections. The injector is designed to accept a variety of inserts allowing tailored configurations for either the Split or Splitless injection method.
2. The **1079 Universal Capillary Injector** can be used in a variety of sampling modes including isothermal split and splitless, temperature ramped splitless, on-column and large volume injection. The 1079 can be temperature programmed and cooled by cryogenics.
3. The **1041 On-Column Injector** is designed for use with largebore capillary columns or packed columns. The standard injector is supplied with an adapter for largebore capillary columns. Optional adapter kits are available for 1/8" and 1/4" packed columns.
4. The **1061 Flash Vaporization Injector** is also designed for use with largebore capillary or packed columns. This injector uses a glass insert inside the injector body. The standard injector is supplied with an insert for use with largebore capillary columns. Optional adapters are available for 1/8" and 1/4" packed columns.

Sampling Valves

The 3800 can be configured with a wide variety of gas and liquid sampling valves. These sampling valves can be unheated or installed in a valve oven. Typically a sampling valve is installed in series with a liquid injector and thus shares the pneumatic system of the injector, but valves can also be installed with their own pneumatic systems. Optional actuators are available to automate the function on the sampling valves. In addition to sampling valves, Varian also supplies a large number of switching valve configurations.

External Sample Introduction Systems

In addition to liquid injectors and sampling valves, several external sampling systems can be used with the 3800. The most common are the 8400/8410, 8200 AutoSamplers, Purge, and Trap System, and the Heated Headspace System. These sampling systems are supplied with individual operator's manuals.

Pneumatic Systems

Most sample introduction systems may be equipped with manual pneumatics or Electronic Flow Control (EFC). The 1041 and 1061 injectors are only available with Electronic Flow Control. The following table summarizes the available pneumatics systems for liquid injectors, sampling valves, purge and trap and heated headspace systems.

Table 1 Pneumatic Systems for Liquid Injectors and Sampling Valves

<i>Sample Introduction System</i>	<i>Standard Pneumatics System</i>	<i>Optional Pneumatics System</i>
CP-1177 Liquid Injector	Type 1 EFC	Manual pneumatics - inlet flow controller/back pressure regulator with pressure gauge.
1079 Liquid Injector	Type 1 EFC	Manual pneumatics - inlet flow controller/back pressure regulator with pressure gauge.
1041 Liquid Injector	Type 3 EFC	Type 4 EFC
1061 Liquid Injector	Type 3 EFC	Type 4 EFC
Gas or Liquid Sampling Valves	Type 4 EFC	<ol style="list-style-type: none"> 1. Manual digital flow controller with pressure gauge 2. Manual digital flow controller and pressure regulator in parallel with gauge.
Purge and Trap with either CP-1177 or 1079	Type 5 EFC	Manual pneumatics - inlet flow controller/backpressure regulator
Genesis Headspace with either CP-1177 or 1079	Type 5 EFC	Manual pneumatics - inlet flow controller/back pressure regulator

Note that when a valve is plumbed in series with a 1041 or 1061 injector it is recommended to upgrade from type 3 EFC to type 4 EFC.

EFC types are described fully in the individual sample introduction device sections.

The CP-1177 Split/Splitless Capillary Injector

The CP-1177 is a dedicated Split/Splitless Isothermal Capillary Injector which can be operated in two modes:

- Split (Isothermal)
- Splitless (Isothermal)

The CP-1177 Injector can be used with a wide range of narrow bore to large bore (100 to 530 μm ID) capillary columns. The basic pneumatics design is a wide range inlet flow controller and back pressure regulated column head pressure. This is the case whether manual pneumatics or Electronic Flow Control is used.

Features of the CP-1177 injector

- The CP-1177 can be operated isothermally from 50 to 450 °C.
- Changing from one injection mode to another typically involves a change of injector insert and a modification to the injector and EFC program. Glass inserts can be easily changed from the top of the injector.
- The internal injector temperature profile keeps the septum cool while maintaining the point of injection at the setpoint temperature.
- The CP-1177 injector design facilitates large volume injection (5 - 100 μL).
- The CP-1177 uses positive septum purge to minimize the adsorption of sample onto the injector septum and to prevent contaminants from the septum entering the column.
- The CP-1177 uses a unique dual split vent that allows effective sweeping of the entire injector body.

Automatic Start Switch

The automatic start switch is a spring loaded actuator that fits over, and is aligned with, the injection port of the CP-1177 injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually started by pressing START on the keyboard.



WARNING:
BURN HAZARD

The injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands.

CP-1177 Injector Assembly

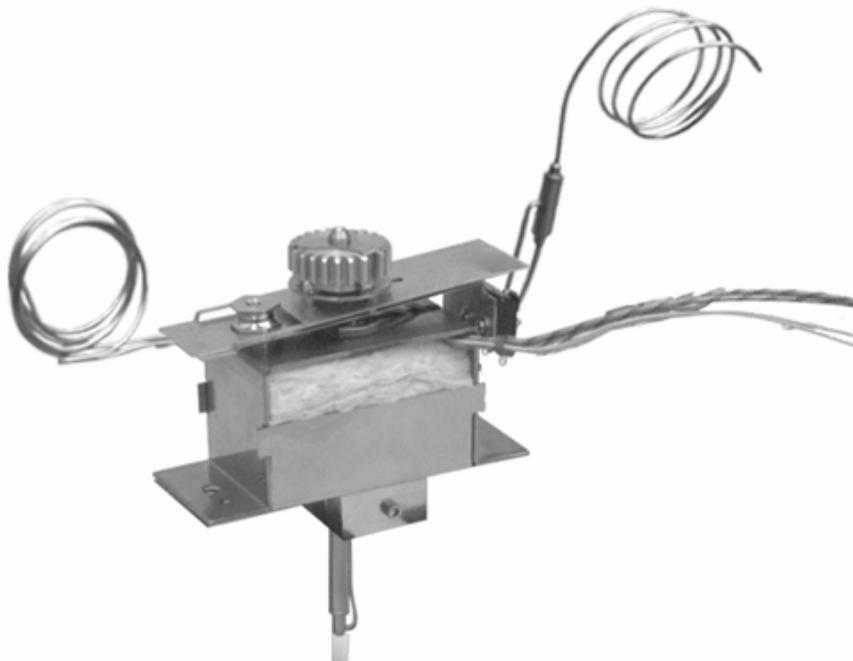
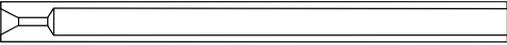
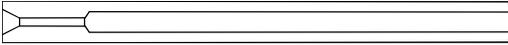
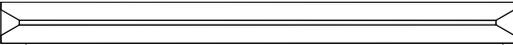
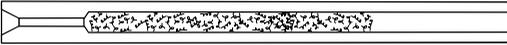
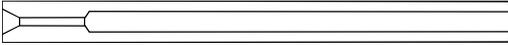


Figure 1 CP-1177 Injector Assembly

CP-1177 Injector Inserts

Note that all CP-1177 injector inserts are deactivated for maximum inertness.

Mode	Insert	Description	Part Number
Split Mode	Packed 4.0 mm ID	Deactivated glass wool packing . Enhances sample mixing to ensure linear split.	03-926119-36
			
Split Mode	Unpacked 4.0 mm ID	The insert be packed with quartz wool, glass beads, etc. Can be used with the full range of capillary columns.	03-926119-27
			
Splitless Mode (isothermal)	Open Single Goose Neck 2 mm ID	The narrow bore minimizes dead volume, ensuring efficient transfer of sample to the column. Loosely pack with quartz wool for hot splitless injections	03-926119-26
			
Splitless	Open 2.0 mm ID	Trace analysis. The low surface area makes it ideal for thermolabile and polar compounds. Used with narrow to wide bore columns (50 - 320 μm).	03-926119-24
			
Split/Splitless	Packed 2 mm ID	Deactivated glass wool packing. Can be used for split, splitless.	03-925999-03
			
SPME	Open .75mm ID		03-926119-98
			

CP-1177 Modes of Injection

The CP-1177 injection mode is defined by the Split Vent program and by the choice of insert. The Split Vent is controller differently based on the pneumatics installed, if EFC the split valve is controlled from the Injector section, or if manual pneumatics the external events control the split valve. The following are brief descriptions of the various modes of injection. More detailed information on each mode is given later in this section.

Split Mode

The split injection mode is preferred for the analysis of relatively concentrated samples. The sample is split in the injector with a representative portion entering the column. The split injection mode provides the shortest sampling time which leads to sharp chromatographic peaks. Use the 4.0 mm open insert, or the packed 4.0 mm ID insert when operating in the split mode.

In the split injection mode, the sample volume is typically 2 μL or less. Early eluting compounds usually appear as very sharp peaks. In some cases, the peak width is less than one second. Thus, it is important that you inject the sample as quickly as possible. If you are using the CP-8400 AutoSampler use the Standard Split/Splitless mode of injection. If you are using a non-Varian AutoSampler or you are using the User Defined Mode of operation on either the CP-8400 or the 8200 AutoSamplers, if the sample injection time (the time between the insertion and removal of the syringe needle from the injector) exceeds the peak width, peaks can broaden, tail or chromatographic performance will be degraded. With broader, later eluting peaks, it is less important that you inject the sample quickly. The split ratio (fraction of sample that enters the column) is the ratio of the flow of carrier gas out the split vent to the flow through the column.

Splitless Mode

The 4 mm insert packed with glass wool is typically used for isothermal splitless injection. The small capillary section of the glass insert fits snugly around the syringe needle and restricts backflash of the sample vapor during injection. In the splitless injection mode, the sample enters the column during a variable sampling time at the beginning of the analysis. This period is typically 30-90 seconds during which there is no flow from the injector to the split vent. After the sampling time, the injector is vented to remove any residual solvent and sample out of the injector.

CP-1177 Flow Diagram

Refer to Figure 6 for the flow path in the CP-1177 Injector.

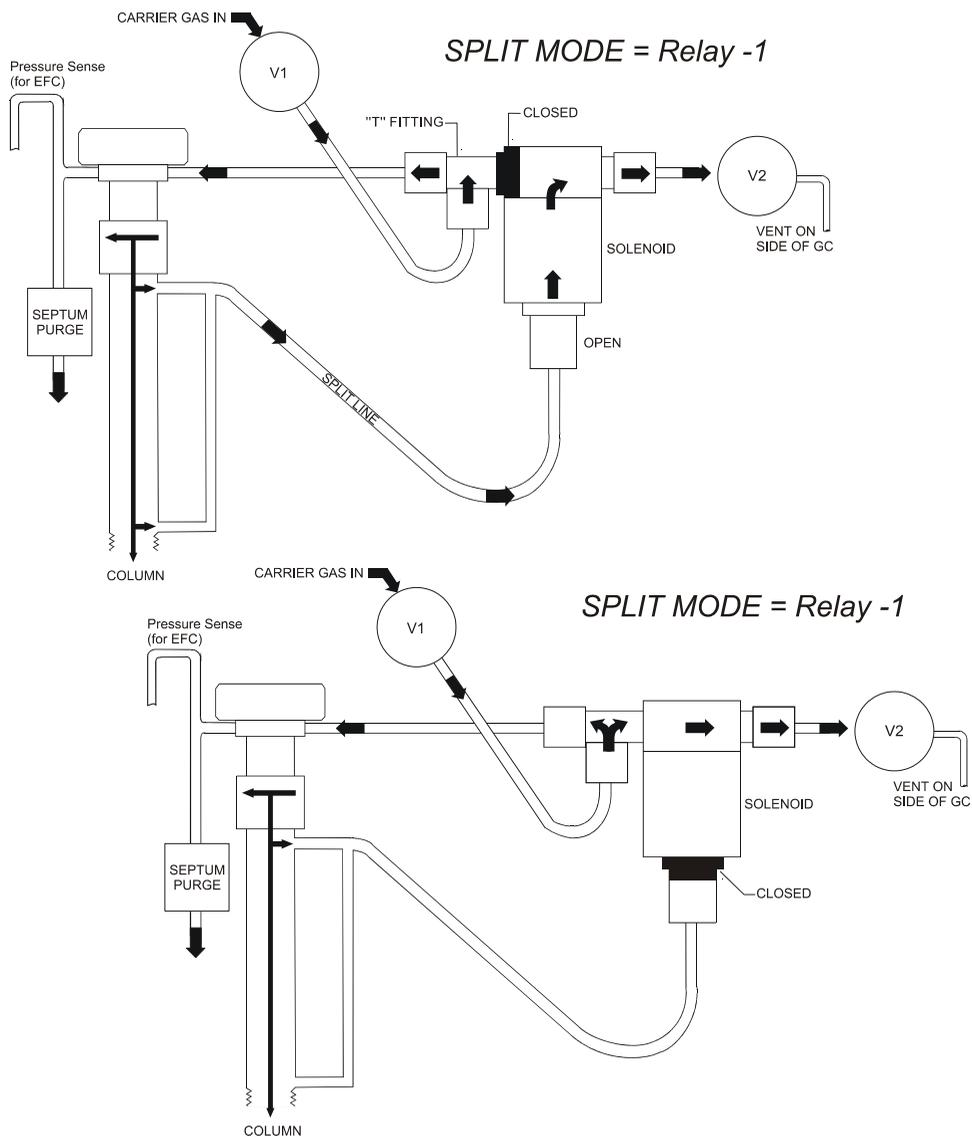


Figure 2 CP-1177 Injector Flow Path

CP-1177 Pneumatics

CP-1177 Electronic Flow Control

The Electronic Flow Control module used on a CP-1177 injector is identified as a type 1 EFC. This EFC type is designed specifically for the CP-1177 and 1079 injectors to support their various modes of operation. In simple terms it duplicates the behavior of the Split/Splitless manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the injector and a pressure control valve downstream from the injector which sets the injector pressure. As injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection. The type 1 EFC flow diagram shown below is an indication of the control mechanism of this type of EFC module.

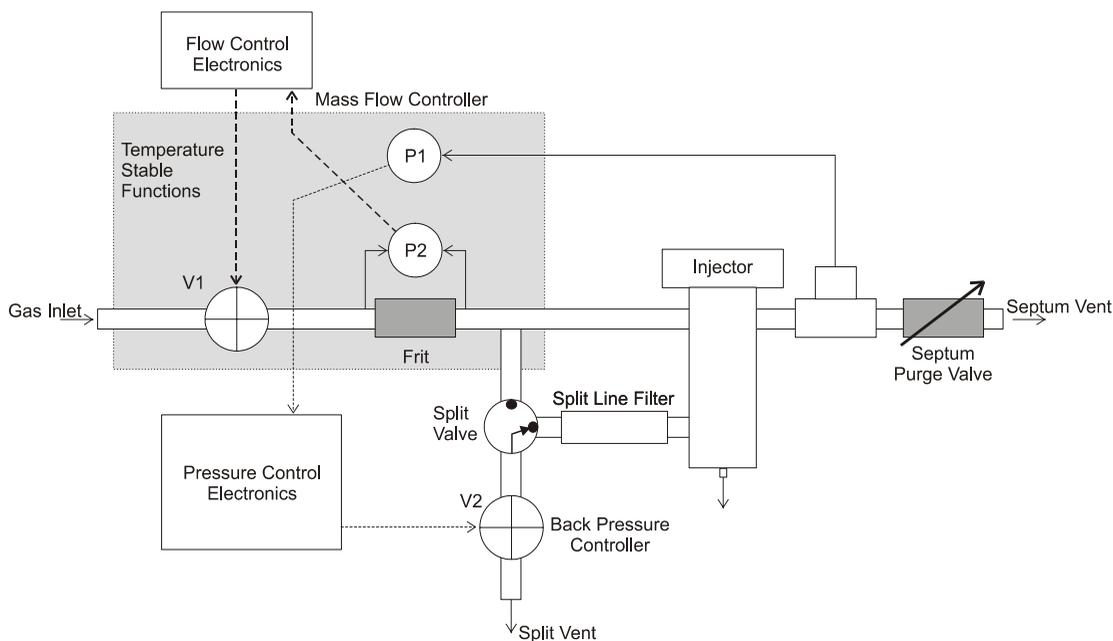


Figure 3 Type 1 EFC Flow Diagram

Type 1 EFC allows the user set constant injector pressure or constant flow. In addition the split ratio can be programmed.

**CP-1177
Manual
Pneumatics**

The total flow into the system is adjusted using a 0–800 mL/min manual flow controller. The CP-1177 Injector uses a flow controller to supply the total flow into the system with a back pressure regulator to control the column head pressure. When the injector is operated in the split mode the flow out the split vent relative to the flow through the column is defined as the split ratio.

In the splitless mode, gas does not flow through the split line from the injector to the 3-way solenoid valve (see diagram below). Rather, gas from the flow controller by-passes the injector to pass through the 3-way solenoid valve. The back pressure regulator uses this flow to control column head pressure, which in turn controls carrier gas flow through the column.

Operation of the CP-1177 Injector

The following section describes how to operate the CP-1177 Split/Splitless Capillary Injector with the Varian 3800 Gas Chromatograph. It is separated into a series of procedures, starting with installation of a column and basic programming of the injector from the keyboard, followed by detailed information on the various modes of injection.

**Column
Installation**

The following procedure describes the installation (or reinstallation) of a capillary column in a Varian 3800 GC equipped with a CP-1177 injector. Each step of the procedure is described in detail:

**Prepare the
Injector end
of the
Column**

Refer to Figure 8 when preparing the end of a capillary column for installation into the CP-1177 injector.

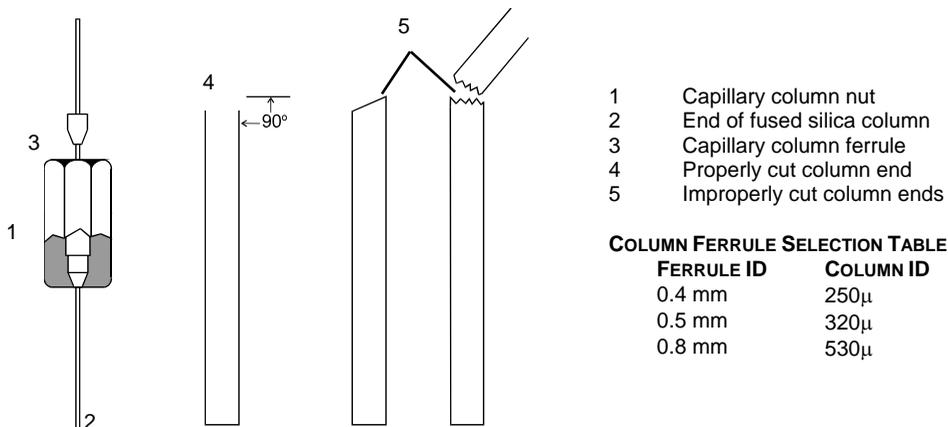


Figure 4 1079 Injector Capillary Installation

Tools and equipment needed

- Ceramic scoring wafer (01-900158-00) or a scoring tool
- Magnifying lens (00-997369-00)

Follow these steps to prepare the end of the column for insertion into the 1177 Injector.

1	Slide the capillary column nut over the fused silica column end.
2	Install the appropriate ferrule over the column end. See above table. Make sure that the tapered end of the ferrule is toward the inside of the column nut.
3	Use the scoring tool to score the column once lightly about 1 to 2 cm from the end of the column.
4	With your thumbs and forefingers, grasp the column on each side of the score and bend the column carefully away from the score mark until it breaks at the score mark.
5	Examine the column with a magnifying lens to determine whether it was properly cut.

Follow these steps to install the chromatographic column into the 1177 Injector.

Note: This procedure is for installation in an injector that is equipped with an open style insert, e.g., the split, splitless or temperature ramped splitless modes.

1	<p>Set the column oven temperature to 50°C and wait for it to cool to this temperature. Turn the column oven power off by pressing the “Turn Oven Off” softkey. Cool the injector to 70°C or lower.</p> <div style="text-align: center;">  <p>WARNING: BURN HAZARD</p> </div>
2	<p>The column should extend 3.7 cm from bottom of the column nut. In the following procedure, you will mark the 3.7 cm insertion depth on the column and then adjust the nut and ferrule so that 3.7 cm extends beyond the column nut. Move the nut and ferrule to within 3 cm of the column end. Measure 3.7 cm from the end of the column and mark this distance with a felt pen or typewriter correction fluid or a small piece of tape.</p> <p>Note: <i>DO NOT allow the column nut and ferrule to fall past the mark because the ink or correction fluid can contaminate the ferrule.</i></p>

3	Partially insert the column into the lower end of the injector. Thread up the capillary column nut and ferrule finger-tight.
4	Gently push the column into the injector until the mark on the column is aligned with the bottom edge of the column nut.
5	Tighten the column nut by hand until it is finger-tight. Hold the column and use a 5/16" wrench to tighten the column nut in place. Tighten the column nut only enough to seal the column and hold it firmly in place.

Note: An optional Capillary Column Quick Connect Kit is available that simplifies the installation of a capillary column into injectors and detectors. It contains split capillary column nuts, re-usable graphite ferrules (for 250 μm , 320 μm , 530 μm ID columns), and a column depth scale. The ferrule is set on the column using this depth scale. No felt pen, typewriter correction fluid, or tape is needed. (The part number of the Quick Connect Kit is 03-925751-90.)

Condition the Column

Analytical columns are conditioned according to the recommendations of the manufacturer. In general, condition the analytical column at 20 °C below the maximum isothermal operating temperature recommended by the column manufacturer.

As an example, a typical conditioning procedure for a non polar column such as a CP-Sil 5 CB would be to set the injector temperature to 300 °C and ramp the column slowly (10 °C/min) to 300 °C. The system is then maintained at these temperatures for a minimum of 2 - 4 hours.



CAUTION

Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged by exposure to oxygen at elevated temperatures. Allow 10 – 15 minutes to purge the column before heating.

Column Installation in Detector

To insert the column into the GC detector, follow the same procedure as installing into the injector. A summary of detector installation depths is given below. These depths refer to the distance from the tip of the column to the bottom edge of the column nut. Note that the TCD refers to the installation distance with a make-up adapter installed.

Table 2 Detector Installation Depths

<u>Detector</u>	<u>Depth (cm)</u>
TCD	3.9
FID/TSD	9.5
PFPD	9.7
ECD	10.5

**Tips for
Tightening
Column
Ferrules**

Refer to the table below for instructions to tighten the capillary column nut with a variety of ferrule types. Do not over tighten capillary column nuts - use a new ferrule if the column does not seal. Over tightening capillary nuts can destroy some ferrules, particularly graphite.

Table 3 Column Ferrule Selection

Type of ferrule...	Recommendation	Comments
New graphite ferrule	1/4-turn past finger-tight	1/4-to 1/2-turn past finger-tight may be required to achieve a seal.
Used graphite ferrule	1/4-turn past finger-tight	If a different size of column is used, more tightening may be required.
New graphite-Vespel® ferrule	1/4- to 1/2-turn past finger-tight	May require retightening after first or second programmed run.
Used graphite-Vespel® ferrule	1/4-turn past finger-tight	Reuse only on same size column.
New Vespel® ferrule	1/4- to 1/2-turn past finger-tight	May require retightening after first or second programmed run.
Used Vespel® ferrule	1/4-turn past finger-tight	Reuse only on same size column.

Setting CP-1177 Gas Flow Rates

The gas flow rates for the CP-1177 Split/Splitless Capillary Injector can be set using manual pneumatics or Electronic Flow Control. Note that a positive flow through the column must be set before heating the column.



CAUTION

Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged.

EFC Pneumatics

Type 1 EFC used with the CP-1177 injector allows the user to set a constant column head pressure, build a pressure program, or set a constant column flow. In addition, a split ratio can be set or time programmed. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is activated.

The CP-1177 is a pressure-controlled injector; thus the column flow decreases with increasing column temperature if the pressure remains

constant. EFC method parameters and status are accessed via the FLOW/PRESSURE key on the 3800 keyboard.

The Type 1 EFC status field displays the actual column head pressure (in the units chosen in Setup), calculated column flow rate, calculated column linear velocity, and the split ratio. The split ratio status is either off if the split state is set to OFF, or a whole number. Page two allows you to view/edit constant flow mode. The following screen is an example of a type 1 EFC method.

The example shown below is a constant pressure method.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Col Press (psi): 10.0		Col Flow (mL/min): 2.0		
Linear Vel (cm/sec): 35		Total Flow (mL/min): 25		
Split Ratio: OFF				
FRONT EFC, TYPE 1		Page 1 of 2		
Step	Pressure (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	10.0	-	2.00	2.00
○		○		○

Constant Column Flow

Type 1 EFC with the CP-1177 will automatically building a pressure program to keep the column flow rate constant during temperature programming of the column oven when Constant Flow is selected. Constant Flow is enabled on page two of the EFC method section. The same general guidelines should be followed for all injection modes. If the column is operated isothermally then the pressure should be kept constant. If the column is temperature programmed then the pressure can either be held constant or programmed. Programming the injector pressure generally has no significant impact on chromatography, other than a slight reduction in analysis time. In some instances resolution may either improve or degrade depending on the settings. For larger volume hot splitless injections, a technique called Pressure Pulse is also available for use with Constant Flow programs with Type 1 EFC. Use the following guide for setting injector pressure and/or building a pressure program.

Temperature programming the column oven results in an increase in carrier gas viscosity which results in a decrease in column flow rate. This effect can be offset by applying the appropriate column pressure program. The second page of the TYPE 1 EFC allows constant flow mode to be selected. If Constant Flow Programming is enabled, a pressure program based on the column parameters and the column oven temperature program will be automatically built whenever the method is activated. If the desired column

flow or the column oven temperature program is changed, the method must be reactivated to derive a new pressure program for these new conditions.

When entering a pressure program to maintain constant column flow rate, the program is based on the column temperature program. If the column oven is operated isothermally, then constant pressure is maintained to achieve a desired flow rate.

Pressure Pulse

Once you have selected Constant Flow programming, a softkey will be displayed on the second page of the Type 1 EFC section. This softkey is labeled Pressure Pulse. Selecting Pressure Pulse will give you the option of enabling a pressure pulse, setting the desired pressure, and the duration for which the pressure should last. Typically, the pressure pulse pressure will be held for between 30 and 90 seconds. The purpose of the pressure pulse is two fold. The higher pressure will prevent the solvent vapor cloud from becoming excessively large allowing larger injection volumes and providing more efficient passage of the sample into the column. The higher pressure also causes higher flow rates into the column making the transfer quicker and thus preventing excessive residence times in the injector with consequent decomposition of labile compounds. **If you elect to use Pressure Pulse technique you will need to perform septa purge calibration using the higher pressure pulse pressure.**

- First the GC will go Ready based on the calculated constant flow conditions.
- Once the initial Ready conditions are achieved, the pressure pulse pressure will be applied to the system.
- When the Pressure Pulse pressure has been achieved, the GC will go to Ready and, if an autosampler is connected, the autosampler will begin sampling in preparation for injections; if you are performing manual injections, you may inject your sample as soon as the Ready LED turns green.

Gas Saver

For type 1 EFC a specific Gas Saver Flow and a timeout period may be set. **A time entry of 0 turns the Gas Saver feature off.** If a non-zero time is entered, the Gas Saver timer will start incrementing upon a method activation. If a run is not started or another method not activated before the timer expires, the set Gas Saver flow rate will be started. A message will be logged in the instrument log. **The fault is cleared by activating a method.**

Septum Purge Calibration

With type 1 EFC, the septum purge calibration routine should be carried out when the instrument is first set up or a new column is installed. Press the SETUP key and choose the Edit Setup Option. Select the Setup EFC menu option for the specific type 1 EFC and press the “Septum Purge Calibrate” softkey. Enter the desired pressure by typing in the number and pressing the ENTER key. After a few seconds the following messages will be displayed: “Enter Septum Purge Flow Rate”. Measure the septum purge flow rate and adjust the septum purge valve (located behind the column oven door) to the desired flow, enter this value in the appropriate field. Press “Save and Edit” softkey to exit setup.

Manual Pneumatics

Tools and equipment needed

- Bubble or electronic flowmeter, e.g., Varian Intelligent Digital Flowmeter, P/N 01-900115-00

1	Turn the CP-1177 Split Flow Controller (on the GC pneumatics panel) counterclockwise to open the split flow controller.
2	Adjust the Back Pressure Regulator (on the GC pneumatics panel) to establish a positive column head pressure (monitored on the pressure gauge). Note: Set the column head pressure based on the column installed in the GC. For example, for a 30M x 250 µm ID column, set the column head pressure to 12-15 psig to achieve ~1 mL/min column flow rate at 50 °C oven temperature (helium).
3	Connect the flowmeter to the split vent on the left side of the GC and measure the split vent flow rate. Turn the Split Flow Controller valve to adjust the split vent flow rate to 50 mL/min.
4	Adjust the Septum Purge Needle valve to adjust the septum purge flow rate to 3 - 5 mL/min. Readjust the split vent flow rate to 50 mL/min.
5	Before heating the column, purge the system with carrier gas for 10 - 15 minutes.

1177 Modes of Operation

The 1177 can be operated in several modes, depending on the nature of the sample and requirements of the analysis. When designing an injection method the most important parameters are:

- the injector insert used;
- the injector and column temperature when the injection is made;
- the carrier gas flow profile through the injector.

The following is a brief description of the method parameters used for each injection mode. In all cases Electronic Flow Control is used for carrier gas control.

Note: In many cases switching from one 1177 mode to another involves changing the glass insert. A detailed stepwise procedure to carry out this task is given in the Maintenance section of the 3800 Getting Started manual. In most injection modes the insert is installed with an o-ring.

Split Injection

The split mode is used when samples are relatively concentrated and for neat samples. This mode of injection involves rapid vaporization of the sample followed by sample splitting. Splitting involves directing a portion of the sample into the column while the remainder is vented. The split ratio is defined as the proportion of sample vented to the sample entering the column. With EFC carrier gas control this parameter can be set automatically in the 1177 method.

Table 4 Split Injection Typical Conditions

Injector Insert	4 mm ID open insert packed with glass wool
Column Installation	3.7 cm from the bottom of the column nut at the base of the injector
Injector Temperature	Isothermal 250 °C.
Column Temperature	50 °C initial for 0.1 min, ramp to 250 °C at 20 °C/min, hold 5 min.
Carrier Gas Control	Set the split mode to ON for the duration of the run in the 1177 method section, and set the split ratio to 100. The sample will be split upon injection and a representative portion representing 1/100 th of the amount injected will enter the column. Note that the split ratio is a method specific parameter and should be set appropriately for individual analyses. In addition the injector pressure or pressure ramp should be set to achieve the desired column flow rate.

Splitless Injection

The classical splitless injection technique involves vaporizing the sample in a hot injector and slow transfer to the column. The split state is OFF during the sampling period; therefore, all of the injected sample should enter the column. The initial column temperature is maintained for at least the sampling time to trap all sample components at the head of the column. At the end of the sampling period (typically 0.5 to 1.5 minutes) the split state is turned ON to vent any residual sample or solvent from the injector.

The following table describes typical method parameters for an isothermal splitless injection.

Table 5 Splitless Injection Method Parameters

Injector Insert	4 mm ID open insert packed with glass wool
Column Position	3.7 cm from the bottom of the column nut at the base of the injector.
Injector Temperature	Initial temperature isothermal 250 °C.
Column Temperature	50 °C initial for 1 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Set the initial split mode to OFF and time program it to ON after 0.75 minutes. In this case the splitless sampling time is 0.75 minutes. The split ratio during the split ON period should be set to 50. Set the appropriate pressure or pressure ramp to achieve the desired column flow rate.

Note that in all the above cases the parameters given are generic and will have to be optimized (including column position) for specific applications. Particular care should be taken with the large volume mode of injection where the initial injector temperature and timing of the split states have to be carefully selected.

Testing the 1177 Injector Performance

The following procedure describes how to test the performance of the 1177 Capillary Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The following table lists the series of test samples available for Varian GC.

Table 6 1177 Test Samples

Test Sample	Part Number	Concentrations of Test Compounds
TCD	82-005048-01	3.00 µg/µL of C ₁₄ , C ₁₅ , and C ₁₆ in iso-octane.
ECD	82-005048-02	33.0 pg/µL of lindane and aldrin in iso-octane.
PFPD	82-005048-03	20.0 ng/µL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/µL of n-pentadecane in iso-octane.
TSD	82-005048-04	2.00 ng/µL of azobenzene, methyl parathion; 4.00 ng/µL malathion and 4.00 µg/µL C ₁₇ in iso-octane.
FID	82-005048-07	30 ng/µL of C ₁₄ , C ₁₅ , and C ₁₆ in iso-octane.

Note: If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.

To run one of these test samples, use the chromatographic conditions listed previously for the injection technique you are currently using. The detector should be operated at the most sensitive range, e.g., 12 for FID and TSD, 0.05 for TCD, 10 for PFPD, and 1 for ECD. The resultant chromatogram should approximate that shown in the detector section of this manual. Some chromatographic interpretation information is given in the troubleshooting section of this manual.

The 1079 Universal Capillary Injector

The 1079 is a Universal Capillary Injector which can be operated in five modes:

- Split (Isothermal)
- Splitless (Isothermal)
- Temperature Ramped Splitless (Temperature Programmed)
- On-Column (Temperature Programmed)
- Large Volume (Temperature Programmed)

The 1079 Injector can be used with a wide range of narrow bore to large bore (100 to 530 µm ID) capillary columns. The basic pneumatics design is a wide range inlet flow controller and back pressure regulated column head pressure. This is the case whether manual pneumatics or Electronic Flow Control is used.

Features of the 1079 Injector

- The 1079 can be operated isothermally or temperature programmed. The temperature range of the injector is from -99 to 450 °C. Sub-ambient temperatures are achieved using cryogenic cooling.
- Changing from one injection mode to another typically involves a change of injector insert and a modification to the injector program. Glass inserts can be easily changed from the top of the injector.
- Temperature programming in the splitless mode gives better recovery of labile analytes and is useful for wide boiling point mixtures.
- The injector temperature profile keeps the septum cool while maintaining the point of injection closer to the setpoint temperature.
- The 1079 injector design facilitates large volume injection (5 - 100 µL).
- The 1079 uses positive septum purge to minimize the adsorption of sample onto the injector septum and to prevent contaminants from the septum entering the column.

Automatic Start Switch

The automatic start switch is a spring loaded actuator that fits over, and is aligned with, the injection port of the 1079 universal injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually started by pressing START on the keyboard.



WARNING:
BURN HAZARD

The injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands.

1079 Injector Assembly

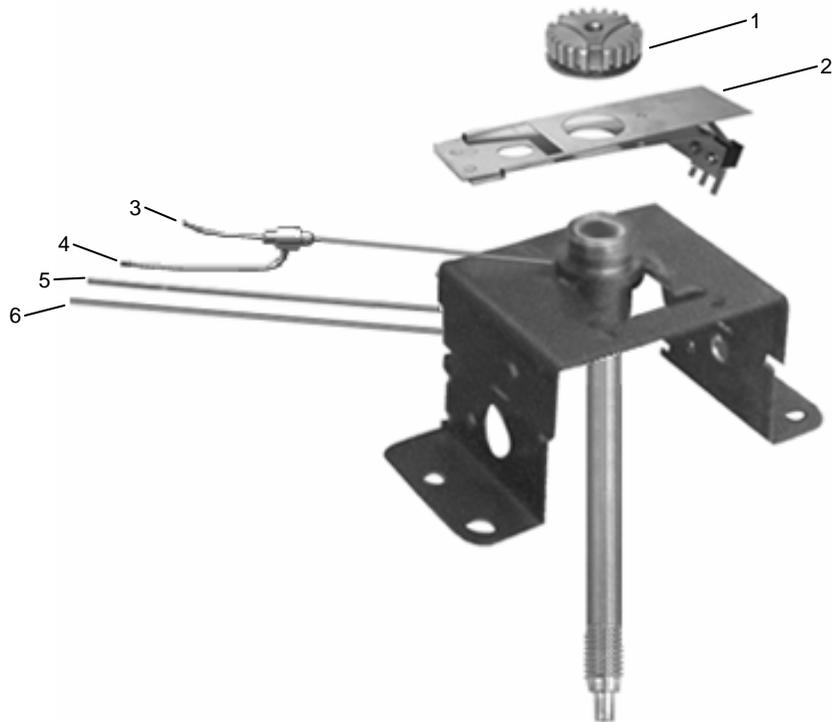
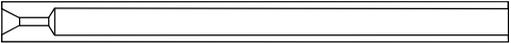
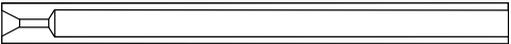
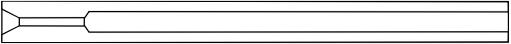
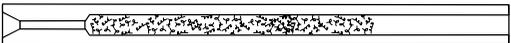
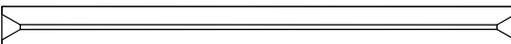
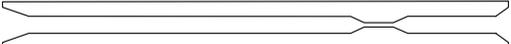


Figure 5 1079 Injector Assembly

- 1 Injector Nut
- 2 Automatic Start Switch
- 3 Septum purge outlet
- 4 Pressure sense line
- 5 Carrier gas inlet
- 6 Split vent outlet

1079 Injector Inserts

Note that all 1079 injector inserts are deactivated for maximum inertness.

Mode	Insert	Description	Part Number
Split Mode	Fritted 3.4 mm ID	Enhances sample mixing to ensure linear split, instantaneous sample vaporization.	03-926119-46
			
	Open 3.4 mm ID	Can be packed with quartz wool, glass beads, etc. Can be used with the full range of capillary columns.	03-926119-45
			
	Packed 3.4 mm ID	Packed with 10% OV-101 on Chromosorb W-HP. Can be used with the full range of capillary columns.	03-926119-54
			
Splitless Mode (isothermal)	Open 3.4 mm ID	Can be packed with quartz wool, glass beads, etc. Can be used with the full range of capillary columns.	03-926119-45
			
Splitless Mode (isothermal)	Open 2 mm ID	The narrow bore minimizes dead volume, ensuring efficient transfer of sample to the column	03-926119-47
			
Splitless (Temperature Ramp Mode)	Open 0.5 mm ID	Trace analysis. The low surface area makes it ideal for thermolabile and polar compounds. Used with narrow to wide bore columns (50 - 320 μm).	03-926119-49
			
Split/Splitless (Temperature Ramp Mode)	Packed 2 mm ID	Deactivated glass wool packing. Can be used for split, splitless and temperature ramp modes.	03-926119-53
			
SPME Mode	Open 0.8 mm ID	Mainly used with SPME, this insert is designed for maximum analyte transfer from the SPME fiber.	03-926119-48
			
High Performance Mode	Tapered insert for column sealing	The tapered internal design allows sealing 0.18 - 0.32 mm ID capillary columns inside the insert. The low surface area makes it ideal for thermolabile and polar compounds.	01-900109-06
			
On-Column Mode	Tapered insert for column sealing	On-column insert for largebore (0.53 mm) capillary columns. Like the high performance insert the column is sealed within the insert.	01-900109-07
			

1079 Modes of Injection

The 1079 injection mode is defined by the Split Vent program and by choice of insert. The Split Vent is controller differently based on the pneumatics installed, if EFC the split valve is controlled from the Injector section, or if manual pneumatics the external events control the split valve. The following are brief descriptions of the various modes of injection; more detailed information on each mode is given later in this section.

Split Mode

The split injection mode is preferred for the analysis of relatively concentrated samples. The sample is split in the injector with a representative portion entering the column. The split injection mode provides the shortest sampling time which leads to sharp chromatographic peaks. Use the 3.4 mm open insert, the 3.4 mm ID fritted insert, or the packed 3.4 mm ID insert when operating in the split mode.

In the split injection mode, the sample volume is typically 1 μL or less. Early eluting compounds usually appear as very sharp peaks. In some cases, the peak width is less than one second. Thus, it is important that you inject the sample as quickly as possible. If the sample injection time, (time between the insertion and removal of the syringe needle from the injector) exceeds the peak width, peaks can broaden, tail or chromatographic performance will be degraded. With broader, later eluting peaks, it is less important that you inject the sample quickly. The split ratio (fraction of sample that enters the column) is the ratio of the flow of carrier gas out the split vent to the flow through the column.

Isothermal Splitless Mode

The 3.4 mm insert packed with glass wool is typically used for isothermal splitless injection. The small capillary section of the glass insert fits snugly around the syringe needle and restricts backflash of the sample vapor during injection. In the splitless injection mode, the sample enters the column during a variable sampling time at the beginning of the analysis. This period is typically 30 - 90 seconds during which there is no flow from the injector to the split vent. After the sampling time, the injector is vented to clean any residual sample out of the injector.

Temperature Ramp Splitless Mode The splitless temperature ramp mode is preferred for compounds that are altered by higher temperatures (thermolabile). Also, the splitless temperature ramp mode is used with wide boiling range mixtures (e.g., hydrocarbon mixtures). Two glass inserts are recommended when operating in the splitless temperature ramp mode: 1) The 2 mm ID glass wool packed insert is used for non-polar compounds at levels >1 ng; and 2) the 0.5 mm ID open insert is used for thermolabile and/or polar compounds at trace levels (pg level).

In the splitless temperature ramp mode, the injector is held at a temperature that is equal to or slightly below the boiling point of the injection solvent. The sample is deposited on the surface of the insert. After injection, the temperature of the injector is increased rapidly (ramped). As the temperature of the injector increases, the sample then vaporizes and is swept onto the column.

On-Column Modes In the on-column modes the column is sealed to the glass insert (0.18 - 0.53 mm columns). This ensures that there is maximum transfer of sample to the column. In the on-column modes the sample is normally injected at or below the solvent boiling point and then the injector is ramped to transfer the sample onto the column.

Large Volume In the large volume mode of injection typically > 5 μL of sample is deposited into the injector slowly, the solvent is vented and then the components of interest are transferred to the column. This is done using a special split vent program and an injector temperature ramp. The 8200 AutoSampler allows automated injection of up to 100 μL of sample.

1079 Flow Diagram

Refer to Figure 6 for the flow path in the 1079 Injector.

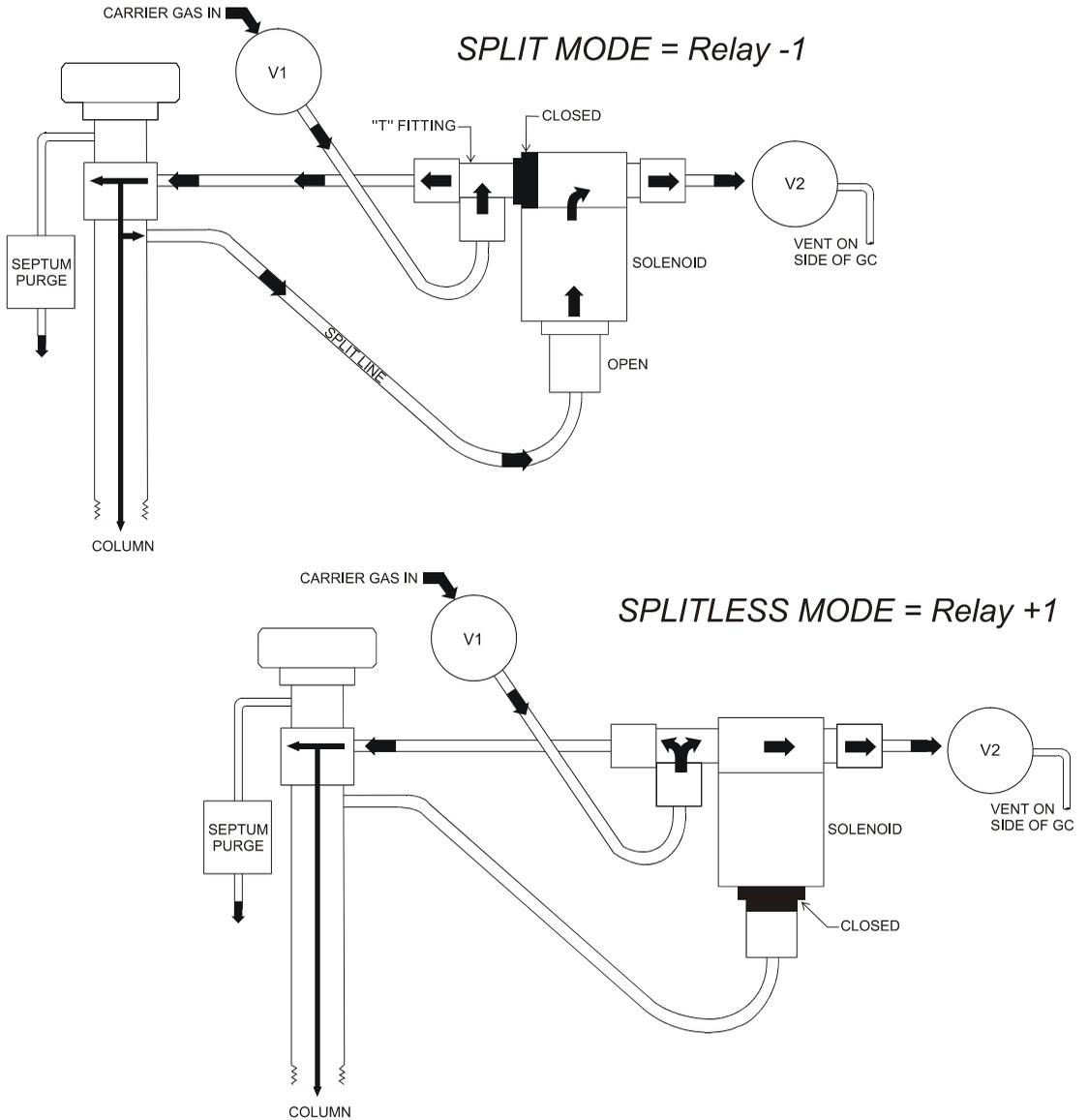


Figure 6 1079 Injector Flow Path

1079 Pneumatics

1079 Electronic Flow Control

The Electronic Flow Control module used on a 1079 injector is identified as a type 1 EFC. This EFC type is designed specifically for the 1079 injector to support its various modes of operation. In simple terms it duplicates the behavior of the 1079 manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the injector and a pressure control valve downstream from the injector which sets the injector pressure. As injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection. The type 1 EFC flow diagram shown below is an indication of the control mechanism of this type of EFC module.

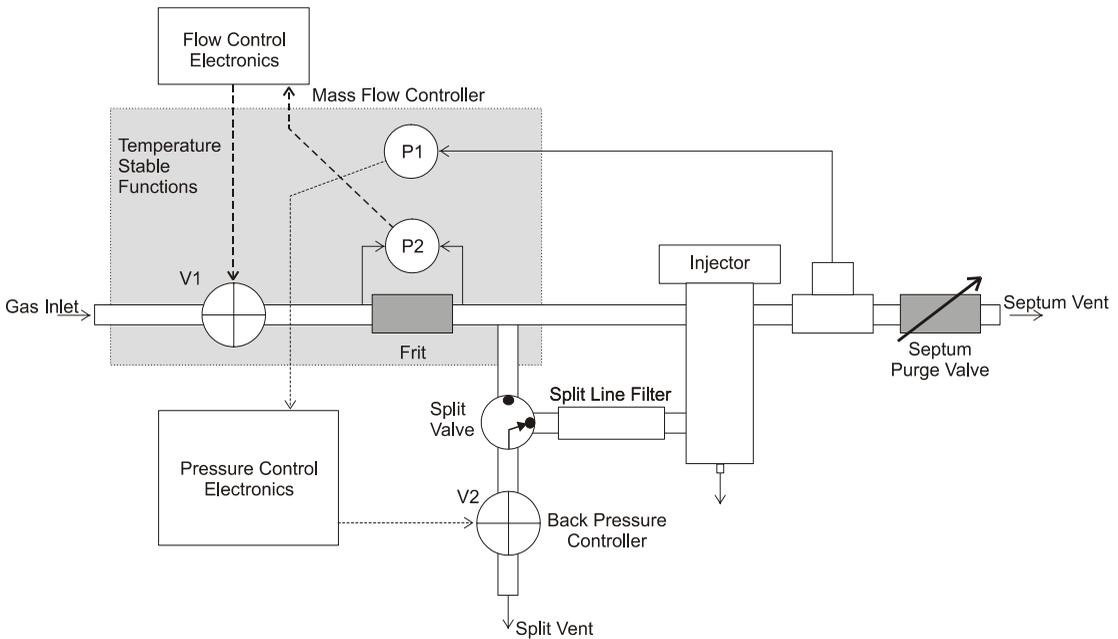


Figure 7 EFC Flow Diagram

1079 Manual Pneumatics

The total flow into the system is adjusted using a 0–800 mL/min manual flow controller. The 1079 Injector uses a flow controller to supply the total flow into the system with a back pressure regulator to control the column head pressure. When the injector is operated in the split mode the flow out the spilt vent relative to the flow through the column is defined as the split ratio.

In the splitless mode, gas does not flow through the split line from the injector to the 3-way solenoid valve (see diagram below). Rather, gas from the flow controller by-passes the injector to pass through the 3-way solenoid valve. The back pressure regulator uses this flow to control column head pressure, which in turn controls carrier gas flow through the column.

Operation of the 1079 Injector

The following section describes how to operate the 1079 Universal Capillary Injector with the Varian 3800 Gas Chromatograph. It is separated into a series of procedures, starting with installation of a column and basic programming of the injector from the keyboard, followed by detailed information on the various modes of injection.

Column Installation

The following procedure describes the installation (or reinstallation) of a capillary column in a Varian 3800 GC equipped with a 1079 injector. Each step of the procedure is described in detail:

Prepare the Injector end of the Column

Refer to Figure 8 when preparing the end of a capillary column for installation into the 1079 injector.

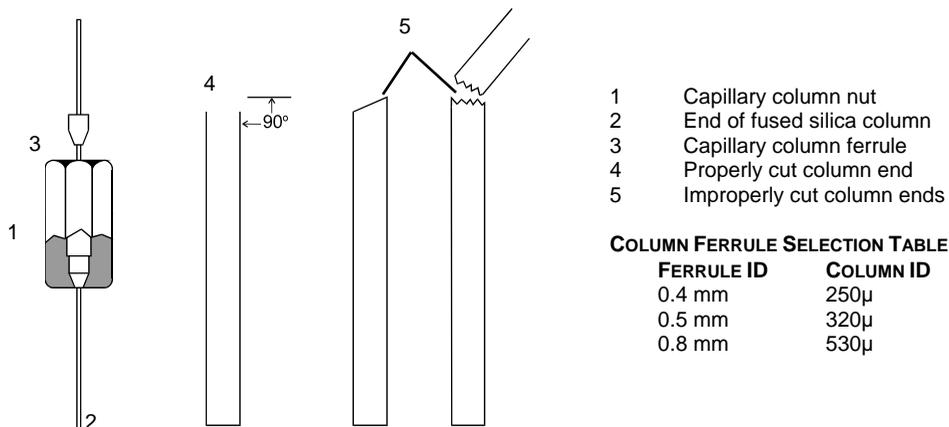


Figure 8 1079 Injector Capillary Installation

Tools and equipment needed

- Ceramic scoring wafer (01-900158-00) or a scoring tool
- Magnifying lens (00-997369-00)

Follow these steps to prepare the end of the column for insertion into the 1079 Injector.

1	Slide the capillary column nut over the fused silica column end.
2	Install the appropriate ferrule over the column end. See previous page. Make sure that the tapered end of the ferrule is toward the inside of the column nut.
3	Use the scoring tool to score the column once lightly about 1 to 2 cm from the end of the column.
4	With your thumbs and forefingers, grasp the column on each side of the score and bend the column carefully away from the score mark until it breaks at the score mark.
5	Examine the column with a magnifying lens to determine whether it was properly cut.

Follow these steps to install the chromatographic column into the 1079 Injector.

Note: This procedure is for installation in an injector that is equipped with an open style insert, e.g., the split, splitless or temperature ramped splitless modes. Instructions are provided later in this section for the on column modes.

1	<p>Set the column oven temperature to 50 °C and wait for it to cool to this temperature. Turn the column oven power off by pressing the “Turn Oven Off” softkey. Cool the injector to 70 °C or lower.</p> <div style="text-align: center;">  <p>WARNING: BURN HAZARD</p> </div>
2	<p>Move the nut and ferrule to within 5 cm of the column end. Measure 7.5 cm from the end of the column and mark this distance with a felt pen or typewriter correction fluid or a small piece of tape.</p> <p>Note: DO NOT allow the column nut and ferrule to fall past the mark because the ink or correction fluid can contaminate the ferrule.</p>
3	Partially insert the column into the lower end of the injector. Thread up the capillary column nut and ferrule finger-tight.
4	Gently push the column into the injector until the mark on the column is aligned with the bottom edge of the column nut.

5	Tighten the column nut by hand until it is finger-tight. Hold the column and use a 5/16" wrench to tighten the column nut in place. Tighten the column nut only enough to seal the column and hold it firmly in place.
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Note: An optional Capillary Column Quick Connect Kit is available that simplifies the installation of a capillary column into injectors and detectors. It contains split capillary column nuts, re-usable graphite ferrules (for 250 μm , 320 μm , 530 μm ID columns), and a column depth scale. The ferrule is set on the column using this depth scale. No felt pen, typewriter correction fluid, or tape is needed. (The part number of the Quick Connect Kit is 03-925751-90.)

**Condition
the Column**

Analytical columns are conditioned according to the recommendations of the manufacturer. In general, condition the analytical column at 20°C below the maximum isothermal operating temperature recommended by the column manufacturer.

As an example, a typical conditioning procedure for a non polar column such as a CP-Sil 5 CB would be to set the injector temperature to 300 °C and ramp the column slowly (10 °C/min) to 300 °C. The system is then maintained at these temperatures for a minimum of 2 - 4 hours.



CAUTION

Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged by exposure to oxygen at elevated temperatures. Allow 10 – 15 minutes to purge the column before heating.

**Column
Installation
in Detector**

To insert the column into the GC detector, follow the same procedure as installing into the injector. A summary of detector installation depths is given below. These depths refer to the distance from the tip of the column to the bottom edge of the column nut. Note that the TCD refers to the installation distance with a make-up adapter installed.

Table 7 Detector Installation Depths

<i>Detector</i>	<i>Depth (cm)</i>
TCD	3.9
FID/TSD	9.5
PFPD	9.7
ECD	10.5

**Tips for
Tightening
Column
Ferrules**

Refer to the table below for instructions to tighten the capillary column nut with a variety of ferrule types. Do not over tighten capillary column nuts - use a new ferrule if the column does not seal. Over tightening capillary nuts can destroy some ferrules, particularly graphite.

Table 8 Column Ferrule Selection

Type of ferrule...	Recommendation	Comments
New graphite ferrule	1/4-turn past finger-tight	1/4-to 1/2-turn past finger-tight may be required to achieve a seal.
Used graphite ferrule	1/4-turn past finger-tight	If a different size of column is used, more tightening may be required.
New graphite-Vespel® ferrule	1/4- to 1/2-turn past finger-tight	May require retightening after first or second programmed run.
Used graphite-Vespel® ferrule	1/4-turn past finger-tight	Reuse only on same size column.
New Vespel® ferrule	1/4- to 1/2-turn past finger-tight	May require retightening after first or second programmed run.
Used Vespel® ferrule	1/4-turn past finger-tight	Reuse only on same size column.

Setting 1079 Gas Flow Rates

The gas flow rates for the 1079 Universal Capillary Injector can be set using manual pneumatics or Electronic Flow Control. Note that a positive flow through the column must be set before heating the column.



CAUTION

Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged. Allow 10 – 15 minutes to purge the column before heating.

EFC Pneumatics

Type 1 EFC used with the 1079 injector allows the user to set a constant column head pressure, build a pressure program, or set a constant column flow. In addition, a split ratio can be set or time programmed. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is activated.

The 1079 is a pressure-controlled injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessed via the FLOW/PRESSURE key on the 3800 keyboard.

The Type 1 EFC status field displays the actual column head pressure (in the units chosen in Setup), calculated column flow rate, calculated column linear velocity, and the split ratio. The following screen is an example of a type 1 EFC method.

The example shown below is a constant pressure method.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Col Press (psi): 10.0		Col Flow (mL/min): 2.0		
Linear Vel (cm/sec): 35		Total Flow (mL/min): 25		
Split Ratio: OFF				
FRONT EFC Type 1		Page 1 of 2		
Step	Pressure (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	10.0	-	2.00	2.00
<div style="display: flex; justify-content: space-around; align-items: center;"> ○ ○ ○ </div>				

**Constant
Column
Flow**

Type 1 EFC with the CP-1079 will automatically building a pressure program to keep the column flow rate constant during temperature programming of the column oven when Constant Flow is selected. Constant Flow is enabled on page 2 of the EFC method section. The same general guidelines should be followed for all injection modes. If the column is operated isothermally then the pressure should be kept constant. If the column is temperature programmed then the pressure can either be held constant or programmed. Programming the injector pressure generally has no significant impact on chromatography, other than a slight reduction in analysis time. In some instances resolution may either improve or degrade depending on the settings. For larger volume hot splitless injections, a technique called Pressure Pulse is also available for use with Constant Flow programs with Type 1 EFC. Use the following guide for setting injector pressure and/or building a pressure program.

Temperature programming the column oven results in an increase in carrier gas viscosity which results in a decrease in column flow rate. This effect can be offset by applying the appropriate column pressure program. The second page of the TYPE 1 EFC allows constant flow mode to be selected. If Constant Flow Programming is enabled, a pressure program based on the column parameters and the column oven temperature program will be automatically built whenever the method is activated. If the desired column flow or the column oven temperature program is changed, the method must be reactivated to derive a new pressure program for these new conditions.

When entering a pressure program to maintain constant column flow rate, the program is based on the column temperature program. If the column oven is operated isothermally, then constant pressure is maintained to achieve a desired flow rate.

Pressure Pulse

Once you have selected Constant Flow programming, a softkey will be displayed on the second page of the Type 1 EFC section. This softkey is labeled Pressure Pulse. Selecting Pressure Pulse will give you the option of enabling a pressure pulse, setting the desired pressure, and the duration for which the pressure should last. Typically, the pressure pulse pressure will be held for between 30 and 90 seconds. The purpose of the pressure pulse is two fold. The higher pressure will prevent the solvent vapor cloud from becoming excessively large allowing larger injection volumes and providing more efficient passage of the sample into the column. The higher pressure also causes higher flow rates into the column making the transfer quicker and thus preventing excessive residence times in the injector with consequent decomposition of labile compounds. **If you elect to use Pressure Pulse technique you will need to perform septa purge calibration using the higher pressure pulse pressure.**

- First the GC will go Ready based on the calculated constant flow conditions.
- Once the initial Ready conditions are achieved, the pressure pulse pressure will be applied to the system.
- When the Pressure Pulse pressure has been achieved, the GC will go to Ready and, if an autosampler is connected, the autosampler will begin sampling in preparation for injections; if you are performing manual injections, you may inject your sample as soon as the Ready LED turns green.

Gas Saver

For type 1 EFC a specific Gas Saver Flow and a timeout period may be set. A time entry of 0 turns the Gas Saver feature off. If a non-zero time is entered, the Gas Saver timer will start incrementing upon a method activation. If a run is not started or another method not activated before the timer expires, the set Gas Saver flow rate will be started. A message will be logged in the instrument log. The fault is cleared by activating a method.

Manual Pneumatics

Tools and equipment needed

- Bubble or electronic flowmeter, e.g., Varian Intelligent Digital Flowmeter, 01-900115-00

1	Turn the 1079 Split Flow Controller (on the GC pneumatics panel) counterclockwise to open the split flow controller.
2	Adjust the Back Pressure Regulator (on the GC pneumatics panel) to establish a positive column head pressure (monitored on the pressure gauge). Note: Set the column head pressure based on the column installed in the GC. For example, for a 30M x 250 μ m ID column, set the column head pressure to 12-15 psig to achieve ~1 mL/min column flow rate at 50 °C oven temperature (helium).
3	Connect the flowmeter to the split vent on the left side of the GC and measure the split vent flow rate. Turn the Split Flow Controller valve to adjust the split vent flow rate to 50 mL/min.
4	Adjust the Septum Purge Needle valve to adjust the septum purge flow rate to 3-5 mL/min. Readjust the split vent flow rate to 50 mL/min.
5	Before heating the column, purge the system with carrier gas for 10 - 15 minutes.

1079 Modes of Operation

The 1079 can be operated in several modes, depending on the nature of the sample and requirements of the analysis. When designing an injection method the most important parameters are:

- the injector insert used and the position of the column within the insert;
- the injector and column temperature when the injection is made;
- the carrier gas flow profile through the injector.

The following is a brief description of the method parameters used for each injection mode. In all cases Electronic Flow Control is used for carrier gas control.

Note: In many cases switching from one 1079 mode to another involves changing the glass insert. A detailed stepwise procedure to carry out this task is given in the Maintenance section of the 3800 Getting Started manual. In most injection modes the insert is installed with a graphite ferrule. Use the special fixture supplied with the 1079 accessory kit to install the ferrule correctly on the insert.

**Isothermal
Split
Injection**

The split mode is used when samples are relatively concentrated and for neat samples. This mode of injection involves rapid vaporization of the sample followed by sample splitting. Splitting involves directing a portion of the sample into the column while the remainder is vented. The split ratio is defined as the proportion of sample vented to the sample entering the column. With EFC carrier gas control this parameter can be set automatically in the 1079 method.

Table 9 Split Injection Typical Conditions

Injector Insert	Install the fritted split insert (03-926119-46) or unpacked split insert (03-926119-45). See instructions in the Maintenance section of the Getting Started manual (03-914647-00) for changing the 1079 insert.
Column Installation	7.5 cm from the bottom of the column nut at the base of the injector
Injector Temperature	Isothermal 250 °C.
Column Temperature	50 °C initial for 0.1 min, ramp to 250 °C at 20 °C/min, hold 5 min.
Carrier Gas Control	Set the split mode to ON for the duration of the run in the 1079 method section, and set the split ratio to 100. The sample will be split upon injection and a representative portion representing 1/100 th of the amount injected will enter the column. Note that the split ratio is a method specific parameter and should be set appropriately for individual analyses. In addition the injector pressure or pressure ramp should be set to achieve the desired column flow rate.

**Isothermal
Splitless
Injection**

The classical splitless injection technique involves vaporizing the sample in a hot injector and slow transfer to the column. The split state is OFF during the sampling period; therefore, all of the injected sample should enter the column. The initial column temperature is maintained for at least the sampling time to trap all sample components at the head of the column. At the end of the sampling period (typically 0.5 to 1.5 minutes) the split state is turned ON to vent any residual sample or solvent from the injector.

The following table describes typical method parameters for an isothermal splitless injection.

Table 10 Isothermal Splitless Injection Method Parameters

Injector Insert	Install the standard 3.4 mm ID splitless insert (P/N 03-926119-45).
Column Position	7.5 cm from the bottom of the column nut at the base of the injector.
Injector Temperature	Initial temperature isothermal 250 °C.
Column Temperature	50 °C initial for 1 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Set the initial split mode to OFF and time program it to ON after 0.75 minutes. In this case the splitless sampling time is 0.75 minutes. The split ratio during the split ON period should be set to 50. Set the appropriate pressure or pressure ramp to achieve the desired column flow rate.

Temperature Ramped Splitless Injection This is a similar technique to isothermal splitless except the sample is vaporized slowly, utilizing temperature programming. This controlled mode of vaporization reduces the risk of mass discrimination and thermal breakdown of sample components during the sampling period. The sample is deposited in the injector as a liquid, therefore the initial injector temperature must be close to the solvent boiling point and there must be a nearby surface for the sample to be retained on. For this mode of injection either a narrow bore (0.5 mm) insert or a glass wool packed insert is used. This facilitates efficient transfer of the sample to a surface from which it is then vaporized.

Note that the glass wool packet insert is not recommended for low levels of polar analytes.

The following are typical method parameters for a temperature ramped splitless injection. In this example hexane is used as the solvent which has a boiling point of 68 °C. Note that the sampling time and initial column hold time are 2 minutes to allow the injector get to maximum temperature.

Table 11 Temperature Ramped Splitless Injection

Injector Insert	Install either the narrow bore 0.5 mm ID insert (03-926119-49) or the glass wool packed 2 mm ID insert (03-926119-53) or the open 3.4 mm ID splitless insert (03-926119-45).
Column Position	7.5 cm from the bottom of the column nut at the base of the injector.
Injector Temperature	Initial temperature 65 °C, hold for 0.1 minute, ramp to 250 °C at 150 °C/minute, hold 10 minutes.
Column Temperature	50 °C initial for 2 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Set the initial split mode to OFF and time program it to ON after 2.00 minutes. In this case the splitless sampling time is 2.00 minutes. The split ratio during the ON period should be set to 50. Set the appropriate pressure or pressure ramp to achieve the desired column flow.

On Column Injection

There are two techniques used for on column injection with the 1079 injector. The high performance mode used a narrow bore insert with a taper for sealing a narrow to wide bore capillary column (0.18 - 0.32 mm ID) within the insert. With this technique the sample is deposited on the insert at the head of the column. The large-bore on-column mode uses a wider bore insert with a taper to allow insertion of a 0.53 mm ID capillary column inside the insert. A standard gauge GC syringe (26s) can then be used to deposit sample inside the column. Note that if this true on-column technique is required for narrow to wide bore columns a short length of uncoated 0.53 mm ID tubing can be used with an appropriate press-fit seal to connect it to the analytical column. As the column is sealed to the insert the entire sample enters the column. The injector is temperature ramped to minimize sample decomposition due to thermal effects or active sites.

The on column techniques involve some special setup of the injector. The following instructions refer to setting up the 1079 in the on-column (0.53 mm ID columns) and high performance (0.18 - 0.32 mm ID columns) modes.

*In the on-column modes the column has to be sealed within the insert. The injector nut, septum, septum support and insert are removed from the injector. The column is pushed up through the injector until it protrudes past the top of the injector. The tapered on-column insert is then pushed onto the column to make a seal between the polyimide coating on the column and the glass surface. The insert is then lowered into the injector and the septum support installed **without** a graphite ferrule. The septum support must not be screwed all the way down or the septum will not seal.*

The following are typical conditions for carrying out an on-column injection. The injector should be maintained at 10 - 20 °C below the solvent boiling point at injection. In this example the solvent is hexane which has a boiling point of 68 °C.

Note: With the high performance insert the taper is **towards the bottom** of the injector. With the on-column insert (530 µm) the taper is **towards the top** of the injector.

Table 12 On-Column Injection

Injector Insert	Install either the high performance tapered insert (P/N 01-900109-06) for narrow to wide bore columns or the on column insert (P/N 01-900109-07) for large bore (0.53 mm ID) columns.
Seal/Position Column	Seal the column within the tapered insert.
Injector Temperature	Initial temperature 50°C, hold for 0.1 minute, ramp to 250°C at 150°C/minute, hold 10 minutes.
Column Temperature	50°C initial for 2 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Split does not apply in on-column mode. The Splitless vent flow must be set to 20 ml/min or greater in Setup. Delete any splitter program and set initial state to off in the Injector section.

**Large
Volume
Injection**

The large volume injection technique is used where the absolute lowest level of detection is required. Up to 100 µL of sample may be introduced into the 1079 injector using a 100 µL syringe (03-925414-01). Note that if this technique needs to be automated using the 8200 AutoSampler a Large Volume Injection kit should be ordered (03-925413-91). The sample is injected at a very slow rate while the injector temperature is set a few degrees below the solvent boiling point. In the example below hexane is used as the solvent which has a boiling point of 68 °C.

Using the large volume injection technique the injector is maintained in the split ON state at the beginning of the run to vent most of the solvent. The sample components are trapped in the injector insert so the same type of insert is used as for temperature ramped splitless injection. The split state is then programmed to OFF and the injector temperature ramped to transfer the sample components to the head of the column. The following are typical conditions for a large volume injection.

Table 13 Large Volume Injection

Injector Insert	Install either the narrow bore 0.5 mm ID insert (03-926119-49) or the glass wool packed 2 mm ID insert (03-926119-53).
Column Position	7.5 cm from the bottom of the column nut at the base of the injector.
Injector Temperature	Initial temperature 66°C, hold for 1 minute, ramp to 250 °C at 150 °C/minute, hold 10 minutes.
Column Temperature	50 °C initial for 3.00 min, ramp to 250 °C at 20 °C/min, hold 5 min.
Carrier gas control	Set the initial split mode to ON, time program it to OFF after 1.00 minutes and then back to ON after 3.00 minutes. The split ratio should be set to 50 during the split ON periods.

Note that in all the above cases the parameters given are generic and will have to be optimized for specific applications. Particular care should be taken with the large volume mode of injection where the initial injector temperature and timing of the split states have to be carefully selected.

Testing the 1079 Injector Performance

The following procedure describes how to test the performance of the 1079 Universal Capillary Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The following table lists the series of test samples available for Varian GC.

Table 14 1079 Test Samples

Test Sample	Part Number	Concentrations of Test Compounds
TCD	82-005048-01	3.00 µg/µL of C ₁₄ , C ₁₅ , and C ₁₆ in iso-octane.
ECD	82-005048-02	33.0 pg/µL of lindane and aldrin in iso-octane.
PFPD	82-005048-03	20.0 ng/µL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/µL of n-pentadecane in iso-octane.
TSD	82-005048-04	2.00 ng/µL of azobenzene, methyl parathion; 4.00 ng/µL malathion and 4.00 µg/µL C ₁₇ in iso-octane.
FID	82-005048-07	30 ng/µL of C ₁₄ , C ₁₅ , and C ₁₆ in iso-octane.

Note: If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.

To run one of these test samples, use the chromatographic conditions listed previously for the injection technique you are currently using. The detector should be operated at the most sensitive range, e.g., 12 for FID and TSD, 0.05 for TCD, 10 for PFPD, and 1 for ECD. The resultant chromatogram should approximate that shown in the detector section of this manual. Some chromatographic interpretation information is given in the troubleshooting section of this manual.

The 1041 On-Column Injector

The 1041 on-column injector is designed for use with 0.53 mm ID capillary columns or 1/8 - 1/4" packed columns. Switching from one mode to another involves changing the injector and detector column mounting hardware. The 1041 is supplied with capillary mounting hardware as standard. If you wish to operate the injector in the packed column mode then a packed column adapter kit must be ordered (03-925588-91). This kit contains injector and detector hardware to facilitate use of the injector with 1/8" metal packed columns. In addition, adapter kits are available for 1/4" columns. Full instructions on converting the 1041 to packed column use are included with the packed column kit.

Automatic Start Switch

The automatic start switch is a spring loaded actuator that fits over, and is aligned with the injection port of the 1041 universal injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually started by pressing START on the keyboard.



WARNING:
BURN HAZARD

The injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands.

Injector Assembly and Insert

A cross-sectional view of the 1041 Universal Injector with insert and 530 μ ID fused silica capillary column is shown in Figure 9.

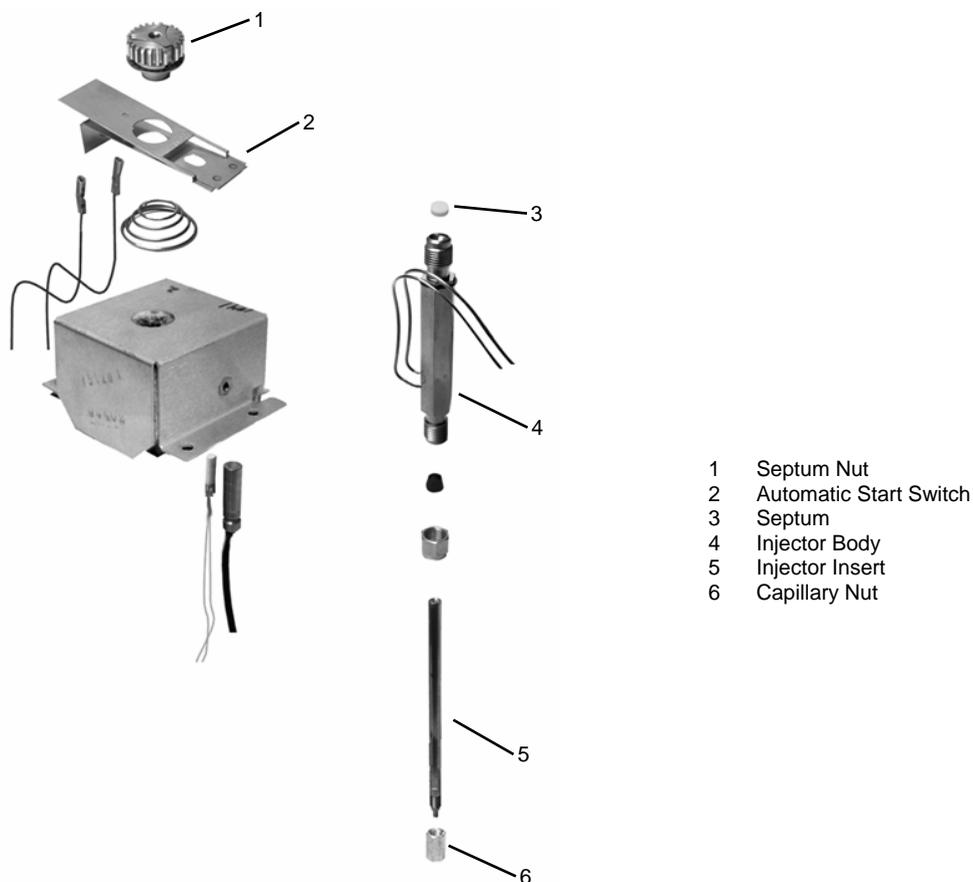


Figure 9 1041 Universal Injector with Insert

Column Installation

The following instructions apply to installing a 0.53 mm ID capillary column in the 1041 injector. Note that the thin polymeric coating on fused silica columns will give some protection against breakage; however, fused silica columns are somewhat fragile and must be handled with care.

Mounting the Column

During operation, the 0.53 mm ID capillary column hangs on the capillary column holder in the column oven.

Note: To prevent the column from unwinding, weave both of the column ends through and around the column loop several times. This is particularly useful if your column is not mounted in a cage.

Hang the coiled column on the capillary column holder then prepare column end and complete column connections to both the injector and detector fittings, as detailed in the following paragraphs.

Preparing Column Ends

To ensure a leak-free connection of capillary columns, the ends of the column must be cut squarely and as smoothly as possible. To achieve this, a proper scoring tool is required. A ceramic scoring wafer (01-900158-00) is recommended. Before cutting the sealed ends of the column, slide the capillary column nut over the column end. Install the appropriate Vespel, graphite/Vespel, or graphite ferrule onto the column with the tapered end toward the capillary column nut. This procedure prevents contaminating the column with ferrule material.

1	Grasp the column securely between the thumb and forefinger of one hand. Score the column once lightly with the cutting tool.
2	Per Figure 10, bend column slightly to break it at the score mark. A magnifying lens (recommended 20X magnifier, 00-997369-00) is necessary to determine the quality of the cut. (If small splinters of silica or some of the outer coating remains on the column end, it is advisable to make a fresh cut.)

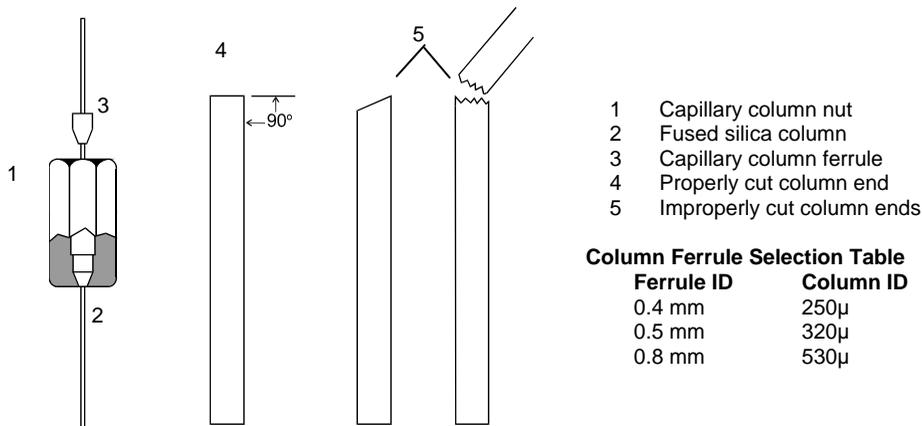


Figure 10 Column Ends

Table 15 General Tightening and Retightening for Common Ferrules

Ferrule Type	Recommendation	Comments
New graphite ferrule	¼ - turn past finger-tight	¼ - to ½ - turn past finger-tight may be required to achieve a seal.
Used graphite ferrule	¼ - turn past finger-tight	If a different size of column is used, more tightening may be required.
New polyimide/graphite ferrule	¼ - to ½ -turn past finger-tight	May require retightening after first or second programmed run. Over tightening will destroy ferrule and seal.
Used polyimide/graphite ferrule	¼ - turn past finger-tight	Re-use only on same size column.
New polyimide ferrule	Not recommended with 1041	
Used polyimide ferrule	Not recommended with 1041	



Column Connection to Injector

IMPORTANT ALIGNMENT OF THE CAPILLARY COLUMN: When a 0.53 mm ID capillary column is used in combination with the Varian 1041 injector, alignment of the column is critical. Use the following steps to precisely align the capillary column. Unless this procedure is followed, the injection syringe may damage the top of the column or peak tailing may result.

The pure graphite ferrule must not be used on the detector (effluent) end of the column when connected to an Electron Capture Detector (ECD). Graphite tends to absorb, then desorb compounds that may contaminate the ECD. Graphite/Polyimide ferrules are recommended for ECD use.

1	Install the capillary injector insert nut and the reversed ferrule on the column.
2	Uncoil about 20 cm of the injector end of the column.
3	Gently push the column fully up into the injector (approximately 3.5 inches), until the column comes to a firm stop. This correctly positions the column against the upper end of the injector insert. The column must go in the full distance. If it does not, the injector will not function properly. Tighten the column nut just enough to prevent column movement.

4	Manually insert the syringe that will be used with the column into the 1041 injector, checking that the syringe goes into the injector completely with no binding or interference. If interference occurs, repeat the above column insertion procedure until the syringe moves smoothly into the injector.
5	Continue to hold the column firmly in place as you tighten the column nut carefully and only enough to hold the column firmly in place and obtain a good seal. Remove the syringe from the injector.
6	After the column is installed at the injector end, make several dry injections to ensure that the needle enters the column easily.
7	Refer to the Detector section of this manual for column connection to the installed detector.

Setting the Carrier Gas Flow

The 1041 injector is supplied with Electronic Flow Control as standard. Type 3 EFC is used with the 1041 and 1061 injectors.

With type 3 Electronic Flow Control equipped injectors a flow range of 0 – 100 mL/min may be set from the 3800 keyboard. Press the Flow/Pressure key and select from the menu of available injectors. The only user settable parameter with the 1041 type of EFC is the desired flow rate.

Flow Rates for Operation

Recommended flow rates differ, depending on the operating mode used. Refer to the appropriate paragraph for your operational mode.

Capillary Mode

When operating the injector in the capillary mode, the following carrier gas flow rates are recommended:

Table 16 Carrier Gas Flow Rates for 1041

Carrier Gas	Column Flow Rate (Optimum)	Column Flow Rate (Typical)
Nitrogen	1.5 mL/min	3 - 15 mL/min
Helium	2.4-4.0 mL/min	4 - 15 mL/min
Hydrogen	4.0-8.0 mL/min	8 - 20 mL/min

Adjust the make-up gas flow to give a total of 30 mL/min.

Packed Mode

When operating in the packed column mode, adjust the carrier gas flow to applicable packed column flow rates (20 to 50 mL/min).

1041 Operation

This section contains the specific operating information and procedures required for optimum performance of the 1041 on column injector. All installations for both the instrument and the injector must be completed before continuing further. Note that the 1041 injector is equipped with Electronic Flow Control as standard.

1041 Injector Method

The 1041 injector method component on the 3800 keyboard contains one screen for setting the required injector temperature and viewing the status information. The 1041 is an isothermal injector and cannot be temperature programmed.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 250, Actual (°C): 250				
MIDDLE 1041				
1041 Oven Power		ON		
1041 Temperature (°C):		250		
				

**Electronic
Flow Control
Operation**

With EFC installed there is one additional screen to program accessed by pressing the FLOW / PRESSURE key. Type 3 EFC allows the user to set a constant flow or to program the column flow. The status field displays the column pressure, actual flow and column linear velocity. Note that type 3 actually sets a required carrier flow into the injector/column system. A leak in the system would be indicated to the user by a drop in inlet pressure.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Col Press: 5.0 psi, Act. Flow: 10.0 mL/min Linear Vel.: 125 cm/sec				
MIDDLE EFC Type 3				
Step	Flow (mL/min)	Rate (mL/min/min)	Hold (min)	Total (min)
Initial	10.0	-	20.00	20.00
<div style="display: flex; justify-content: space-around;"> ○ ○ ○ </div>				

The screen above represents a typical application of type 3 EFC with an isothermal injector. Normally this type of injection system is maintained at constant flow during the analytical run. However, if required the flow rate could be programmed during the run.

Testing the 1041 Injector

The most effective method of testing injector performance is by running a test sample. The following procedure describes how to test the performance of the 1041 on column injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The following table lists the series of test samples available for Varian GC detectors.

Note: Due to airline regulations, test samples are not shipped outside the USA. Refer to the compound concentrations in the table below to make your own test samples.

Table 17 1041 Test Compounds

Test Sample	Part Number	Concentrations of Test Compounds
TCD	82-005048-01	3.00 µg/µL of C ₁₄ , C ₁₅ , and C ₁₆ in iso-octane.
ECD	82-005048-02	33.0 pg/µL of lindane and aldrin in iso-octane.
PFPD	82-005048-03	20.0 ng/µL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/µL of n-pentadecane in iso-octane.
TSD	82-005048-04	2.00 ng/µL of azobenzene, methyl parathion; 4.00 ng/µL malathion and 4.00 µg/µL C ₁₇ in iso-octane.
FID	82-005048-07	30 ng/µL of C ₁₄ , C ₁₅ , and C ₁₆ in iso-octane.
Note: <i>If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.</i>		

To run one of these test samples, use the chromatographic conditions listed below. The detector should be operated at its most sensitive range, e.g., 12 for FID and TSD, 0.05 for TCD, 10 for PFPD, and 1 for ECD. The resultant chromatogram should approximate that shown in the detector section of this manual.

Table 18 Chromatographic conditions for testing the 1041 injector

Injector temperature	250 °C
Column temperature	50 °C initial, ramp to 250 °C at 20 °C / minute and hold for 5 minutes
Injection Volume	1 µL

Observe the eluting peaks for symmetry, separation, and elution time. Abnormally wide or skewed peaks, excessive elution times, abnormally small peaks, and noisy or drifting baselines indicate faulty performance.

The 1061 Flash Vaporization Injector

The 1061 flash vaporization injector is designed for use with large-bore fused silica columns of 0.53 mm ID but can be operated in two modes: the capillary column mode or the packed column mode. Switching from one mode to another involves changing the injector and detector column mounting hardware. The 1061 is supplied with capillary mounting hardware as standard. If you wish to operate the injector in the packed column mode then a packed column adapter kit must be ordered (03-925588-92). This kit contains injector and detector hardware to facilitate use of the injector with 1/8" metal packed columns. Complete instructions are provided in this kit for installing and operating the injector in the packed column mode.

Automatic Start Switch

The automatic start switch is a spring loaded actuator that fits over, and is aligned with the injection port of the 1061 universal injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually started by pressing START on the keyboard.

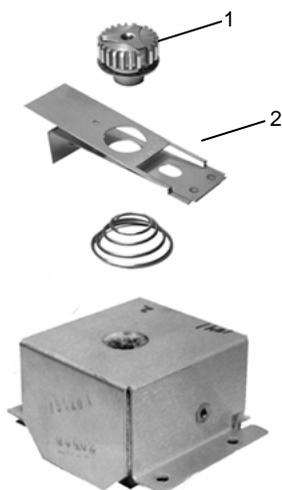


WARNING:
BURN HAZARD

The injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands.

Injector Assembly and Insert

The 1061 Universal Injector with insert and 530 μ ID fused silica capillary column is shown in Figure 11.



These tools are needed to replace the glass insert:

- Tweezers or septum pick (72-000084-00)
- Injector nut wrench (03-908423-00)
- Clean laboratory tissue



- 1 Septum Nut
- 2 Automatic Start Switch
- 3 Septum
- 4 Injector Insert
- 5 Injector Body

Figure 11 1061 Flash Vaporization Injector with Insert

Note that the 1061 injector contains a glass insert with a tapered section on the bottom. This allows a large-bore capillary column seal with the insert.

The sample is vaporized in the insert and then swept onto the column. The use of glass wool in the insert allows the analysis of samples containing non-volatile components. The non-volatile material is trapped on the glass wool while the volatile components vaporize onto the column. The packed column kit for the 1061 also contains a glass insert, in this case the packed column butts up against the bottom of the insert.

Column Installation

The following instructions apply to installing a 0.53 mm ID capillary column in the 1061 injector. Note that the thin polymeric coating on fused silica columns will give some protection against breakage; however, fused silica columns are somewhat fragile and must be handled with care.

Mounting the Column

During operation, the fused silica capillary column hangs on the capillary column holder in the column oven.

Note: To prevent the fused silica column from unwinding, weave both of the column ends through and around the column loop several times. This is particularly useful if your column is not mounted in a cage.

Hang the coiled column on the capillary column holder then prepare column end and complete column connections to both the injector and detector fittings, as detailed in the following paragraphs.

Preparing Column Ends

To ensure a leak-free connection of capillary columns, the ends of the column must be cut squarely and as smoothly as possible. To achieve this, a proper scoring tool is required. A ceramic scoring wafer (01-900158-00) is recommended. Before cutting the sealed ends of the column, slide the capillary column nut over the column end. Install the appropriate Vespel, graphite/ Vespel, or graphite ferrule onto the column with the tapered end toward the capillary column nut. This procedure prevents contaminating the column with ferrule material.

1	Grasp the column securely between the thumb and forefinger of one hand. Score the column once lightly with the cutting tool.
2	Per Figure 12, bend column slightly to break it at the score mark. A magnifying lens (recommended 20X magnifier, 00-997369-00) is necessary to determine the quality of the cut. (If small splinters of silica or some of the outer coating remains on the column end, it is advisable to make a fresh cut.)
3	Install a capillary injector nut and reversed ferrule on the column.
4	Push the column into the injector until it stops.

5	Continue to hold the column as you tighten the capillary column nut carefully and only enough to obtain a good seal and hold the column firmly in place. Refer to Table 20 for tightening recommendations for common ferrules.
----------	--

Table 19 Detector Installation Depths

<i>Detector</i>	<i>Depth (cm)</i>
TCD	3.9
FID/TSD	9.5
PFPD	9.7
ECD	10.5

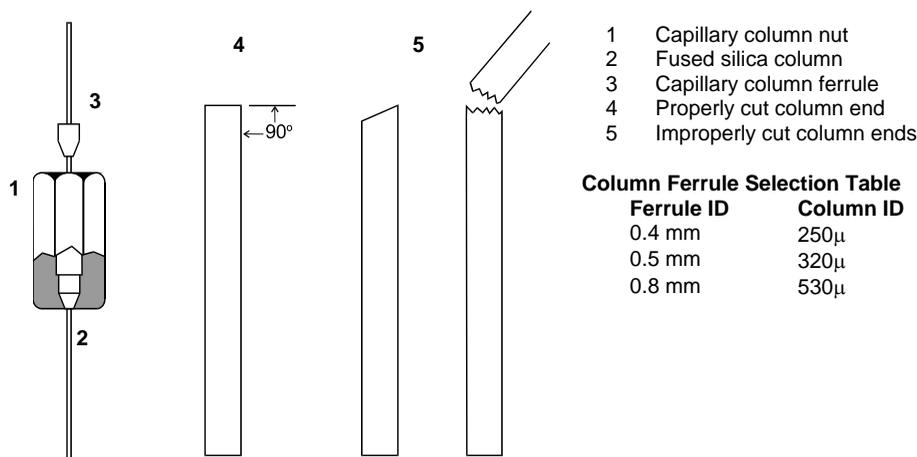


Figure 12 Column Ends

Table 20 General Tightening and Re-tightening for Common Ferrules

<i>Ferrule Type</i>	<i>Recommendation</i>	<i>Comments</i>
New graphite ferrule	¼ - turn past finger-tight	¼ - to ½ -turn past finger-tight may be required to achieve a seal.
Used graphite ferrule	¼ - urn past finger-tight	If a different size of column is used, more tightening may be required.
New polyimide/graphite ferrule	¼ - to ½ -turn past finger-tight	May require retightening after first or second programmed run. Over tightening will destroy ferrule and seal.
Used polyimide/graphite ferrule	¼ - turn past finger-tight	Re-use only on same size column.
New polyimide ferrule	Not recommended with 1061	
Used polyimide ferrule	Not recommended with 1061	

Setting the Carrier Gas Flow

The 1061 injector is equipped with type 3 Electronic Flow Control. With this type of EFC a column flow of 0 - 100 mL/min may be set from the 3800 keyboard.

Flow Rates for Operation

Recommended flow rates differ, depending on the operating mode used. Refer to the appropriate paragraph for your operational mode.

Capillary Mode

When operating the injector in the capillary mode, the following carrier gas flow rates are recommended:

Table 21 Carrier Gas Flow Rates for 1061

Carrier Gas	Column Flow Rate (Optimum)	Column Flow Rate (Typical)
Nitrogen	1.5 mL/min	3 - 15 mL/min
Helium	2.4-4.0 mL/min	4 - 15 mL/min
Hydrogen	4.0-8.0 mL/min	8 - 20 mL/min

Adjust the make-up gas flow to give a total of 30 mL/min.

Packed Mode

When operating in the packed column mode, adjust the carrier gas flow to applicable packed column flow rates (20 to 50 mL/min).

1061 Operation

This section contains the specific operating information and procedures required for optimum performance of the 1061 flash vaporization injector. All installations for both the instrument and the injector must be completed before continuing further. Note that the 1061 is equipped with Electronic Flow Control as standard.

All EFC adjustments are made from the GC keyboard. The 1061 uses a type 3 EFC module which is a flow controller. The status feedback on the 3800 display is the column head pressure, actual flow rate, and the calculated linear velocity.

1061 Injector Method

The 1061 injector method component on the 3800 keyboard contains one screen for setting the required temperature for the injector and viewing the status information. The 1061 is an isothermal injector and cannot be temperature programmed.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 250, Actual (°C): 250				
MIDDLE 1061				
1061 Oven Power		ON		
1061 Temperature		250		

Electronic Flow Control Operation

With EFC installed there is one additional screen to configure accessed by pressing the FLOW/PRESSURE key. Type 3 EFC allows the user to set a constant flow or to program the flow. The status field displays the column pressure, actual flow and column linear velocity. Note that type 3 actually sets a required carrier flow into the injector/column system. A leak in the system would be indicated to the user by a drop in inlet pressure.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Col Press: 5.0 psi, Col Flow: 10.0 mL/min, Linear Vel: 125 cm/sec				
MIDDLE EFC Type 3				
Step	Flow	Rate	Hold	Total
Initial	10.0	-	20.00	20.00

The screen above represents a typical application of type 3 EFC with an isothermal injector. Normally this type of injection system is maintained at constant flow during the analytical run. However, if required the column flow rate could be programmed during the run.

Testing the 1061 Injector

The most effective method of testing injector performance is by running a test sample. The following procedure describes how to test the performance of the 1061 flash vaporization injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The following table lists the series of test samples available for Varian G.C. detectors.

Note: Due to airline regulations, test samples are not shipped outside the USA. Refer to the compound concentrations in the table below to make your own test samples.

Table 22 1061 Test Compounds

Test Sample	Part Number	Concentrations of Test Compounds
TCD	82-005048-01	3.00 µg/µL of C ₁₄ , C ₁₅ , and C ₁₆ in iso-octane.
ECD	82-005048-02	33.0 pg/µL of lindane and aldrin in iso-octane.
PFPD	82-005048-03	20.0 ng/µL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/µL of n-pentadecane in iso-octane.
TSD	82-005048-04	2.00 ng/µL of azobenzene, methyl parathion; 4.00 ng/µL malathion and 4.00 µg/µL C ₁₇ in iso-octane.
FID	82-005048-07	30 ng/µL of C ₁₄ , C ₁₅ , and C ₁₆ in iso-octane.

Note: If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.

To run one of these test samples, use the chromatographic conditions listed below. The detector should be operated at its most sensitive range, e.g., 12 for FID and TSD, 0.05 for TCD, 10 for PFPD, and 1 for ECD. The resultant chromatogram should approximate that shown in the detector section of this manual.

Table 23 Chromatographic Conditions for Testing the 1061 Injector

Injector temperature	250 °C
Column temperature	50 °C initial, ramp to 250 °C at 20 °C/minute and hold for 5 minutes.
Injection Volume	1 µL

Observe the eluting peaks for symmetry, separation, and elution time. Abnormally wide or skewed peaks, excessive elution times, abnormally small peaks, and noisy or drifting baselines indicate faulty performance.

Valved Systems

The 3800 can be equipped with a variety of valving options, including gas sampling valves, liquid sampling valves and switching valves. There are numerous locations for valves on the 3800, some of which are unheated and others that are heated. All valves can be automated regardless of location. The following are the typical locations for valves on the 3800:

- Unheated Valves located on the top panel of the instrument, beneath the injector cover. The area behind the injectors can accommodate up to two unheated valves.
- Single or dual valve ovens which replace one or two injector positions on the 3800. The single valve oven can accommodate one valve and an optional 1041 or 1061 injector. The dual valve oven accommodates one or two valves.
- A multi-position valve oven which can accommodate up to six standard valves with 4 - 10 ports, or one 12 port or larger valve (including a Stream Selector Valve) and four standard valves. This valve oven is mounted over the rear injector position and utilizes one heated zone.
- Up to three valves can be installed on the right side of the column oven. This is the least preferred location due to temperature cycling of the column oven.

Heated Zones for Valves

If valves are located in a valve oven then they are configured in 3800 Setup as front, middle or rear valve ovens. The valve oven temperature is programmed in the Sample Delivery section of the method. Note that when multiple valves are installed in one valve oven the temperature of the oven should always be set lower than the lowest temperature rated valve. In general valves have upper temperature ratings of 220 °C or 350 °C.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Col Press (psi): 10		Column Flow (mL/min): 15		
Linear Velocity (cm/sec): 10		Total Flow (mL/min): 30		
FRONT EFC TYPE 4, Page 2 of 2				
	Time (min)	Total Flow (mL/min)		
	Initial	30		
	0.01	500		
	1.50	30		

**Flow Rates
for
Operation**

Recommended total flow rates differ, depending on the operating mode used. Normally the total flow rate is set about twice the required column flow rate. For example if the desired column flow rate is 15 mL/min, set the total flow rate to 30 mL/min. When a valve switch is programmed, the total flow should be increased to offset any effects of the pressure disturbance. In this case program the total flow to 500 mL/min for one minute and then revert back to the original total flow. The total flow program can be further optimized by adjusting the duration of the higher flow rate or reducing the total flow.

Detectors

Introduction

The 3800 can accommodate up to three installed detectors and have all three running simultaneously. The standard detectors available on the 3800 are the Flame Ionization Detector (FID), Thermal Conductivity Detector (TCD), Electron Capture Detector (ECD), Thermionic Specific Detector (TSD), and Pulsed Flame Photometric Detector (PFPD). Except for the TCD and PFPD any combination of three detectors can be installed. In the case of the PFPD, only two may be installed but any one additional detector may also be installed including a TCD. In the case of the standard TCD, only two can be installed and then no additional detectors can be installed. However, a dual TCD option is available as a custom solution which allows installation of an additional ionization detector. The dual TCD consists of two detector cells in one housing.

Detectors are mounted on the top of the 3800, above the left side of the column oven. The position of the detector, however, is determined by the location of the detector's electrometer in the electronics cabinet. Detector electrometers are installed on the left side of the GC accessed by removing the left side panel. All cables connecting the electrometers to the detectors are accessible by removing the two covers on the top left of the 3800, i.e., the detector top cover and the cover over the keyboard/display.

Flame Ionization Detector

The following instructions refer to the installation and operation of a 3800 Flame Ionization Detector (FID). The FID is installed on a detector base, directly above the column oven. The FID electrometer is installed in the electronics cabinet on the left of the instrument. The electronics of the FID are controlled from the 3800 keyboard, the gas flows are set and controlled either from the 3800 keyboard (if detector EFC is installed) or at the pneumatics panel.

Initial Set-Up

To set up the FID, proceed as follows. Note that when the 3800 is first powered up all heated zones with the exception of the column oven are powered off, and all detector electronics are turned off.

1	Connect the air, hydrogen, and carrier gas lines to the appropriate bulkhead fittings on the GC.
2	Check the gas supply pressures. The 3800 flow rates have previously been factory set at the following pressures: air = 60 psig; carrier + make-up = 80 psig, and H ₂ = 40 psig. Reset the gas flows if you will be using different supply pressures. If you are operating with capillary columns, a make-up gas (at 80 psig) is also required. (Refer to the Pre-Installation Instructions for additional information on gas supply requirements.)
3	Press the SETUP key on the 3800 front panel to display the Instrument Setup Menu. Select "View Instrument Setup" and then the Heated Zones menu item (item 2) and press Enter to display the location of all installed heated zones. The FID should be listed as Zone 4, 5, or 6, and its location (Front, Middle or Rear) should appear in the Location column. Check that the location corresponds with the physical location of the FID electrometer board in the electronics cabinet on the left side of the 3800, and note the location of the FID detector tower to which this electrometer is connected. If your GC is equipped with detector EFC, page down to the detector EFC section of Setup and verify the makeup gas you will be using with your FID.
4	Install a chromatographic column in the GC (refer to the 3800 Getting Started manual). If the analytical column is not pre-conditioned, use a no-hole ferrule in the detector column nut while conditioning the column and leave the detector end of the column loose in the oven. If the analytical column is pre-conditioned, follow the usual procedure for column installation.
5	<p>If detector EFC is installed, set the following flow rates in the adjustments section of the FID method. Press the DETECTOR key in the GC CONTROL section of the 3800 keyboard and select the FID being used. Press the ADJUSTMENTS softkey and then select page 2. Set the make-up flow to 25 mL/min, the hydrogen flow to 30 ml/min and the air flow to 300 mL/min. Note that the combined column + make-up flow should be 30 mL/min, so some adjustment of the make-up flow may be necessary.</p> <p>If manual pneumatics are installed, attach a bubble or electronic flowmeter to the top of the FID tower using the adapters supplied in the FID accessory kit (Note that the most reliable measure of flow is directly from the flame tip. This prevents misadjustment due to leaks in the overall tower assembly). Check the following flow rates and set them if necessary: combined carrier gas + make-up flow to 30 mL/min, the hydrogen flow to 30 mL/min and the air flow to 300 mL/min.</p>
6	Press the detector key on the 3800 keyboard, select the FID by its location and turn on the FID oven power. Set the oven temperature to 300 °C. Verify that the FID electronics is turned OFF.

Operation

Check the detector temperature status on the FID screen. Check the FID range setting and set to range 12, if necessary.

Note: Generally, to prevent water condensation in the detector assembly, the detector should be operated at a temperature above the column temperature and not below 150 °C. If the detector is operated at a lower temperature, condensation can lead to excessive noise.

After the FID oven has reached its setpoint temperature, turn on the FID electronics. Note that the FID will light automatically when the electronics are turned ON. The 3800 GC monitors the FID background current continuously and will attempt to light the flame if the current drops below a specified threshold. Up to three attempts to light the flame will be made before the 3800 reports a flame-out fault. If a flame-out fault is reported, correct the cause of the flame-out and then clear the fault by pressing the FID ignite softkey, or by turning the FID electronics OFF and ON.



**WARNING:
EYE HAZARD**

DO NOT look directly into the detector tower when attempting to ignite the flame.

You can verify that the flame is lit by monitoring the FID signal in the status region of the FID display. Initially the signal will read a very high value but should then stabilize. Typically the signal will be > 50 mV at range 12 when the flame is first lit but should drop to < 10 mV within 30 minutes.



**WARNING:
EXPLOSION HAZARD**

To avoid a possible fire or explosion, always turn off the flow of hydrogen when the column is removed or when the detector is not being used. This prevents the accumulation of hydrogen.

Detector Program

The DETECTOR section allows you to modify the detector method parameters and view the status of individual detectors. If more than one detector is installed, then pressing DETECTOR will prompt you to select from a menu of the installed detectors. The menu screen indicates the name of the detector and its location.

Below is an example for a Flame Ionization Detector. Each detector method includes three or four pages (four if detector EFC is installed). Page one includes initial parameters for the detector oven temperature, turning on the detector oven power and electronics, and selecting the range. Page two establishes time programs to either change the range, or Autozero the detector at specific times.

The third and fourth pages, located by pressing the adjustments softkey, include detector-specific parameters not routinely adjusted. For the FID, these pages allow you to select between a fast or slow detector time constant (page 1) and to set detector gas flows if EFC is installed (page 2). The normal setting for the time constant is fast.

Page 1 of the FID method:

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 300	Actual (°C): 300		Signal (mV): 5.15	
FRONT FID Page 1 of 2				
	Oven Power:		ON	
	Temperature (°C):		300	
	Electronics:		ON	
	Range:		12	
IGNITE		AUTOZERO		ADJUSTMENTS
○		○		○

The IGNITE softkey applies power to the FID ignitor filament for 5 seconds. The AUTOZERO softkey applies an immediate zeroing of the detector signal.

Page 2 allows time programming events such as range and Autozero.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME												
Method 1	Method 1		0.00	20.00												
Set (°C): 300	Actual (°C): 300		Signal (mV): 5.15													
FRONT FID Page 2 of 2.																
<table border="1"> <thead> <tr> <th>Time</th> <th>Range</th> <th>Autozero</th> </tr> </thead> <tbody> <tr> <td>Initial</td> <td>12</td> <td>NO</td> </tr> <tr> <td>5.00</td> <td>12</td> <td>YES</td> </tr> <tr> <td>6.50</td> <td>11</td> <td>NO</td> </tr> </tbody> </table>					Time	Range	Autozero	Initial	12	NO	5.00	12	YES	6.50	11	NO
Time	Range	Autozero														
Initial	12	NO														
5.00	12	YES														
6.50	11	NO														
IGNITE		AUTOZERO		ADJUSTMENTS												
○		○		○												

The following page is accessed by pressing the Adjustments softkey.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 300	Actual (°C): 300		Signal (mV): 5.15	
FRONT FID ADJUSTMENTS, Page 1 of 2				
Time Constant: Fast				
<div style="border: 1px solid black; display: inline-block; padding: 2px 10px;">Clear AUTOZERO</div>				

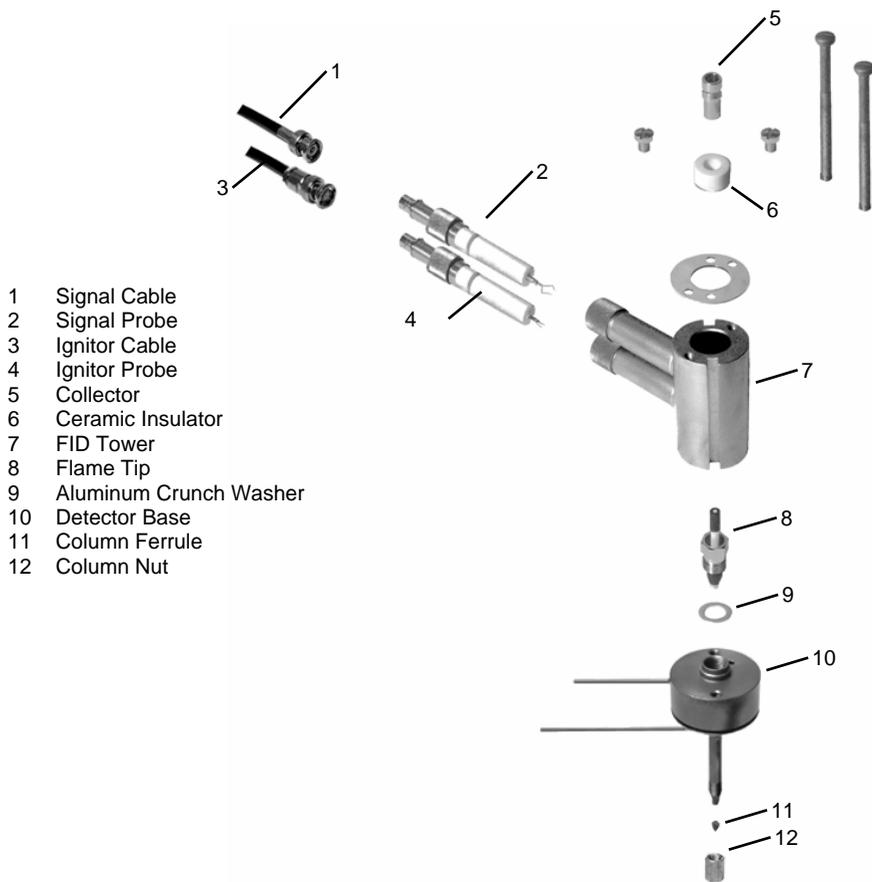
The second adjustments page is only accessible if detector EFC is installed.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Make-up Flow (ml/min): 25	H ₂ Flow (ml/min): 30			
Air Flow (ml/min): 300				
FRONT FID ADJUSTMENTS, Page 2 of 2				
Make-up Flow (ml/min)	25			
H ₂ Flow (ml/min)	30			
Air Flow (ml/min)	300			

Installation/Disassembly

At times, it is necessary to remove the detector components to inspect, to clean, to replace parts, or to install another detector. Follow the disassemble/reassemble instructions below for both the FID and the detector oven.

Figure 13 is an exploded view of the FID. Refer to these figures as you disassemble the detector.



- 1 Signal Cable
- 2 Signal Probe
- 3 Ignitor Cable
- 4 Ignitor Probe
- 5 Collector
- 6 Ceramic Insulator
- 7 FID Tower
- 8 Flame Tip
- 9 Aluminum Crunch Washer
- 10 Detector Base
- 11 Column Ferrule
- 12 Column Nut

Figure 13 FID Exploded View

Disassemble the FID To disassemble the FID, proceed as follows:

1	Set the FID to 50 °C and wait for it to cool before disassembly. Turn the detector electronics and detector oven OFF in the active method. Remove the GC top covers.
2	Turn off the main GC gas supplies to the detector at the pneumatics panel. These are the make-up, hydrogen and air supplies.
3	Disconnect the signal and ignitor cables from their probes on the detector tower.

4	Remove the signal and ignitor probes from the detector tower. <i>DO NOT</i> rotate the probes as you disconnect them from the electrical contacts in the tower. Place the probes on a clean surface such as a lint-free laboratory tissue.
5	Remove the two 8-32 x 2-3/4-inch tower mounting screws from the top of detector tower.
6	Remove the tower assembly from the detector oven, lifting straight up until clear of the flame tip. Remove the collector tube and insulator from the detector tower. Avoid contamination of the ceramic insulator and probes. If the detector is not completely cool, use a metal tool (such as tweezers or a hooked wire) to remove parts from the tower assembly. Place parts on a clean Kimwipe®. Never place them on a counter or painted surface.
7	Remove the flame-tip assembly from the detector base. Take care not to break the ceramic flame tip tube or the Vespel®/graphite or graphite ferrule.
8	Remove and discard the aluminum seal washer from the detector base. Always use a new aluminum seal washer each time you reassemble the detector. Note that the aluminum seal washer may remain in the detector tower when the tower is removed from the base.

**Reassemble
the FID**

Refer to Figure 13 to reassemble the FID detector. To reassemble the FID, proceed as follows:

1	If you removed the detector oven from the plenum, restore the oven to its former position and reinstall the four screws holding the oven to the plenum.
2	Install the flame tip in the detector base. The FID and TSD flame tips are identical. <ul style="list-style-type: none"> • If you are installing the flame tip with a new Vespel/graphite ferrule, tighten the assembly finger-tight plus an extra 1/6-turn. • If you are installing the flame tip assembly with a used graphite ferrule, tighten about 1/3-turn past finger tight.
3	The Vespel/graphite ferrule supplied with the GC has a maximum temperature limit of 350 °C. If it is necessary to operate the detector above 350 °C, you may need to replace the Vespel/graphite ferrule with a graphite ferrule.
4	Install a new aluminum seal washer onto the shoulder of the detector base. To ensure a reliable tower seal, use a NEW aluminum washer each time you install the detector.
5	Place the detector tower on the detector base and secure it with the two 8-32 x 2-3/4-inch tower mounting screws. Alternately tighten these screws a ½-turn as the tower tightens into place.



CAUTION

Handle the ceramic insulator and probes with tweezers to avoid contamination.

6	Carefully insert the ignitor probe into the lower arm of the detector tower. Align the probe key with the tower arm slot. Check the orientation of the ignitor elements by looking down through the top of the detector tower. The spring clip should slip around the flame tip and make good contact (see Figure 14). The ignitor coil must not touch the flame tip assembly nor be positioned directly above it. Tighten the knurled nut to secure the probe.
7	Make sure the notch in the detector tower arm does not cut the O-ring seal.
8	Insert the insulator into the detector tower, then insert the collector tube into the tower. The collector tube must not touch the ignitor coil.
9	Insert the signal probe into the upper arm of the detector tower. The probe clip should fit around the tapered section on the collector tube tightly enough to exert a downward force (see Figure 15). Secure the signal probe by tightening the knurled nut. Make sure the notch in the detector tower arm does not cut the o-ring seal.
10	Connect the ignitor cable to the ignitor probe and the signal cable to the signal probe.

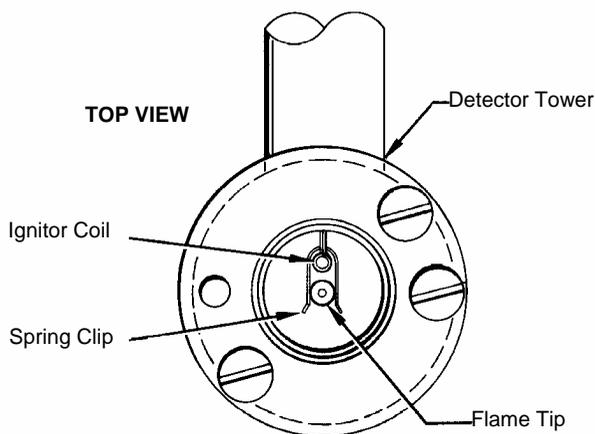


Figure 14 Orientation of Ignitor Probe Elements in FID Tower

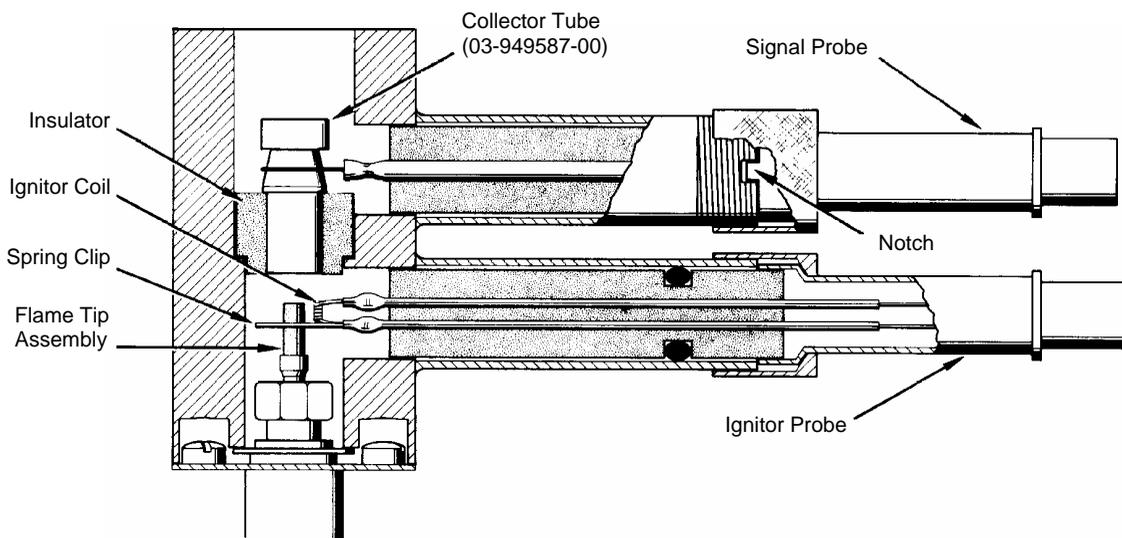


Figure 15 FID Cross-sectional View

**Remove /
Install the FID
PC Board**

To remove the FID PC Board, proceed as follows:



**WARNING:
SHOCK HAZARD**

Dangerous voltages exposed. Turn the GC power OFF when you remove or install PC boards. Failure to do so may result in accidental contact with dangerous voltages, or in damage to the PC board or GC.

1	Turn the GC power OFF.
2	Disconnect the signal and ignitor cables from the electrometer.
3	Remove the left side panel from the GC by removing the 8- 32 screws.
4	Remove the 8-32 screw securing the FID electrometer board in the detector bay. Slide the PC board straight out of the guide slots in the cabinet. If possible, do not touch the edge connectors. Place the PC board in a clean anti-static bag.
5	To install the PC board, align the groove in the electrometer cover with the guide rail at the top of the detector bay and slide the electrometer into the opening until the edge connector seats in the socket. Never force the board into the cabinet. Make sure that all cables are out of the way before you insert the board. Reconnect the cables to the detector. Check that the connectors mate.

Thermionic Specific Detector

These instructions refer to the installation and operation of the Thermionic Specific Detector (TSD) on the 3800 Gas Chromatograph. The TSD detector tower is installed on a 3800 detector oven. The TSD Electrometer Board is installed in the GC electronics cabinet on the left side of the instrument.

The TSD is controlled through the keyboard on the front panel of the GC. Gas flows are controlled at the pneumatics panel of the GC, or in the Adjustments section of the method if Detector EFC is installed.

Initial Set-Up

To set up the TSD for operation, proceed as follows:

1	Connect the air, hydrogen, and carrier gas lines to the appropriate bulkhead fittings on the GC.
2	Check the gas supply pressures. The GC is factory set for the following inlet pressures: air = 60 psig; H ₂ = 40 psig; carrier = 80 psig. Reset the gas flow rates if you use different supply pressures. If you are operating with capillary columns, a make-up gas (at 80 psig) is also required. (Refer to the 3800 Pre-Installation Instructions for additional information on gas supply requirements.)
3	Press the SETUP key on the 3800 front panel to display the Instrument Setup Menu. Select View Instrument Setup, then Heated Zones (2) and press Enter to display the location of all installed heated zones. The TSD will be listed as Zone 4, 5, or 6, and its location (Front, Middle or Rear) should appear in the Location column. Check that the location corresponds with the physical location of the TSD electrometer board in the electronics cabinet on the left side of the 3800, and note the location of the TSD detector tower to which this electrometer is connected. If your 3800 GC is equipped with detector EFC, page down to the detector EFC section of Setup and verify the makeup gas you will be using with your TSD.
4	Install a chromatographic column in the GC (refer to the Column installation information in the 3800 Getting Started manual). If the analytical column is not well conditioned, use a no-hole ferrule in the detector column nut while conditioning the column and leave the column end loose in the oven. If the analytical column is well conditioned, follow the usual procedure for column installation.
5	Condition the TSD bead. The following bead conditioning and optimization procedures assume use of a short, non-polar column such as the Varian test column, 03-912300-30 (packed) or 03-912805-99 (capillary). If you use a different type of column, you should adjust conditions accordingly.

Operation and Operating Recommendations

TSD operation requires hydrogen, air, and either nitrogen or helium carrier gas. The amount of hydrogen relative to the other gases will affect nitrogen-

6	Page forward to page 2 and turn the initial Autozero OFF. Set the bead current to 2.400 A. Turn on the power to the bead by setting Electronics to ON. Allow the bead to condition for at least 15 minutes.
7	Increase the bead current in steps of 0.100 Ampere, waiting at least 15 minutes for the bead to equilibrate between increases. When increasing the bead current causes the TSD baseline value to go above 64 mV, as shown at the top of the display, the bead has reached its ignition point. Allow the bead current to remain at this setting for a minimum of 12 hours (preferably overnight). This completes the initial conditioning of the TSD bead probe. Now optimization of the bead probe for N/C selectivity is required.

Page 1 of the TSD method:

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 300	Actual (°C): 300		Signal (mV): 15.35	
MIDDLE TSD Page 1 of 2				
	Oven Power:		ON	
	Temperature (°C):		300	
	Electronics		ON	
	Range:		12	
	Bead Current:		2.4	
	AUTOZERO			ADJUSTMENTS
○	○			○

The AUTOZERO softkey applies an immediate zeroing of the detector signal.

Page 2 allows time programming events such as range changes.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME																
Method 1	Method 1		0.00	20.00																
Set (°C): 300 Actual (°C): 300 Signal (mV): 15.35																				
MIDDLE TSD Page 2 of 2																				
<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th>Time</th> <th>Range</th> <th>Autozero</th> <th>Bead Power</th> </tr> </thead> <tbody> <tr> <td>Initial</td> <td>12</td> <td>NO</td> <td>ON</td> </tr> <tr> <td>5.00</td> <td>12</td> <td>YES</td> <td>ON</td> </tr> <tr> <td>6.50</td> <td>11</td> <td>NO</td> <td>ON</td> </tr> </tbody> </table>					Time	Range	Autozero	Bead Power	Initial	12	NO	ON	5.00	12	YES	ON	6.50	11	NO	ON
Time	Range	Autozero	Bead Power																	
Initial	12	NO	ON																	
5.00	12	YES	ON																	
6.50	11	NO	ON																	
		AUTOZERO		ADJUSTMENTS																

The following pages are accessed by pressing the Adjustments softkey.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 300 Actual (°C): 300 Signal (mV): 15.35				
MIDDLE TSD ADJUSTMENTS, Page 1 of 2				
Time Constant: Fast				
<div style="border: 1px solid black; padding: 5px; display: inline-block;">Clear AUTOZERO</div>				

The Clear Autozero softkey removes the Autozero offset so that you can observe the detector background signal.

The second Adjustments page is only accessible if detector EFC is installed.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Make-up Flow (ml/min): 25 H ₂ Flow (ml/min): 2.5				
Air Flow (ml/min): 175				
MIDDLE TSD ADJUSTMENTS, Page 2 of 2				
	Make-up Flow (ml/min)		25	
	H ₂ Flow (ml/min)		4.5	
	Air Flow (ml/min)		175	

Optimization of the TSD Using the Varian Test Sample

The sensitivity of the TSD is highly dependent on bead temperature and the selectivity is dependent on hydrogen flow. In addition, there are slight variations from bead to bead and a bead changes slowly as it ages. Therefore, optimum operating conditions for a given TSD cannot be specified exactly; the user should determine optimum operating conditions for the application and TSD bead.

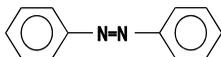
The detector responds to both nitrogen and phosphorus. It is not possible to significantly change the relative response of nitrogen to phosphorus.

Determining Optimum Hydrogen Flow

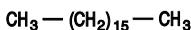
Although a nominal hydrogen flow of 4.0 mL/min is suggested, the nitrogen-to-carbon selectivity versus hydrogen flow often differs slightly from bead to bead. Consequently, for each new bead it is useful to chromatograph the detector test sample at several values of hydrogen flow (from 4 to 5.5 mL/min) to determine the best hydrogen flow for that bead.

1	Zero the TSD signal. Inject 1 µL of TSD test sample (82-005048-04). A normal response will result in the bead quenching during the solvent peak (i.e., a negative deflection of the pen will occur), and a positive response for the azobenzene, heptadecane, methyl parathion, and malathion peaks. Adjust the detector range to keep the peaks on scale.
2	If the azobenzene peak (see the appropriate chromatogram below) is less than 3 times the height of the heptadecane peak, decrease the hydrogen flow rate and re-inject the sample. If injection of the test sample results in quenching by the solvent peak and the bead fails to re-ignite, simply increase the bead current slowly, with intervening pauses of ~15 minutes, until the bead re-ignites. Then repeat step 1.
3	Over the next few days, the background current of the bead will decrease, and the sensitivity will also decrease to a steady level. At this point the sensitivity of the bead will remain relatively constant, with low baseline drift, and low noise.

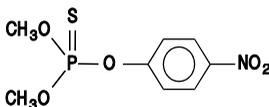
1. Azobenzene:
2 ng Azobenzene contains 310 pg nitrogen



2. Heptadecane:
4 µg heptadecane contains 3.4 µg carbon



3. Methylparathion:



4. Malathion:
4 ng Malathion contains 380 pg phosphorus

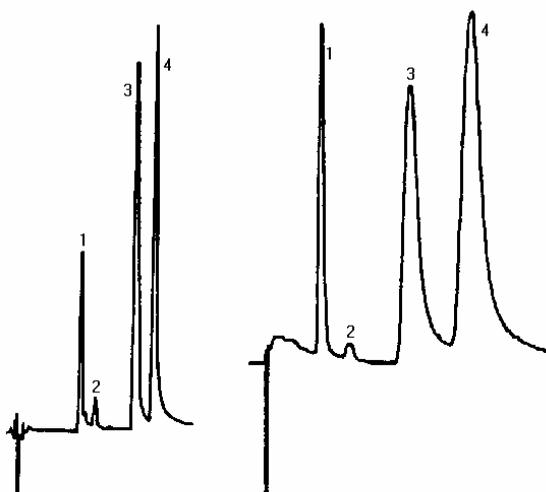
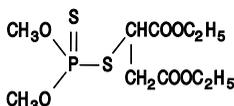


Figure 16 Structure of Components in the TSD Test Sam*ple

Maintaining Optimum Conditions

If the detector is used daily, the power to the bead may be left on when not in use at 2.4 to 2.6 Amps. As the bead ages, it will require a higher current to maintain a given bead sensitivity. Therefore, detector background signal should be checked every few days. Eventually sensitivity will be lost and it will be necessary to replace the bead (03-906074-00). The hydrogen flow does not need to be changed during the lifetime of a bead.

Column Bleed

To minimize contamination of the detector by column bleed, all new columns should be conditioned before they are connected to the detector. Condition new columns for a minimum of 24 hours at 40 °C below the maximum temperature of the column packing liquid phase.

Since the TSD detects compounds containing nitrogen or phosphorus, column packing materials containing these elements should be avoided. Common liquid phases containing nitrogen are OV-225, OV-275, FFAP, XE-60, and TCEP. If you must use such columns, it is important that they be well conditioned and operated at as low a temperature as possible in order to minimize column bleed. High column bleed produces high background signals, making it difficult or impossible to set the bead current, and can result in the occurrence of negative peaks in the chromatogram.

Solvent Considerations When the TSD is used for trace level analysis, only solvents free of nitrogen, phosphorus, sulfur, or halogenated compounds should be used. Depending on the volatility of the impurity in a solvent, it can produce excessive solvent tailing or extraneous chromatographic peaks.

Chlorinated Solvents Chlorinated solvents can be used with the TSD. However, these solvents generally cause an abrupt increase in both detector background signal and sample response. These effects appear to be associated with a temporary adsorption of chlorinated species onto the surface of the hot alkali-ceramic bead. Repetitive injections of samples in chlorinated solvents yield constant responses. However, once the injection of chlorinated solvents is stopped, the background and sample response will decay back to the response levels that existed before the initial injection of the chlorinated solvent. Therefore, when chlorinated solvents are used, calibration standards should be run frequently.

Bead Power Interruption You can minimize the effect of chlorinated solvents on the bead by turning off bead current during the time that the solvent elutes, using the time programming table of the detector method. The total time that the bead is turned off should be less than 2 minutes in order to minimize thermal re-equilibration times of the detector. (Any peaks eluting during this off time will not be measured.)

Effect of Silylation Reagents Silylation reagents affect the TSD in two ways. First, such reagents sometimes contain nitrogen which will cause tailing of the solvent peaks. Second, decomposition of the reagent on the hot bead produces SiO₂ which deposits on the bead surface causing loss of response. Generally, SiO₂ deposits can be removed by cleaning the bead and usually produce no permanent damage to the bead.

Table 24 TSD Gases, Pressures, Purity, and Flow Rates

Gas	Inlet Pressure at Gas Cylinders	Recommended Purity	Flow Rate (mL/min)
<i>Carrier (packed columns):</i>			
He or N ₂	80 psig	99.999%	30 mL/min
<i>Detector Make-Up (capillary columns):</i>			
He or N ₂	80 psig	99.999%	25 ±1 mL/min
<i>Detector Gases:</i>			
Air	60 psig	CGA Grade E	175 ±5 mL/min
H ₂	40 psig	99.999%	4.0 ±0.2 mL/min

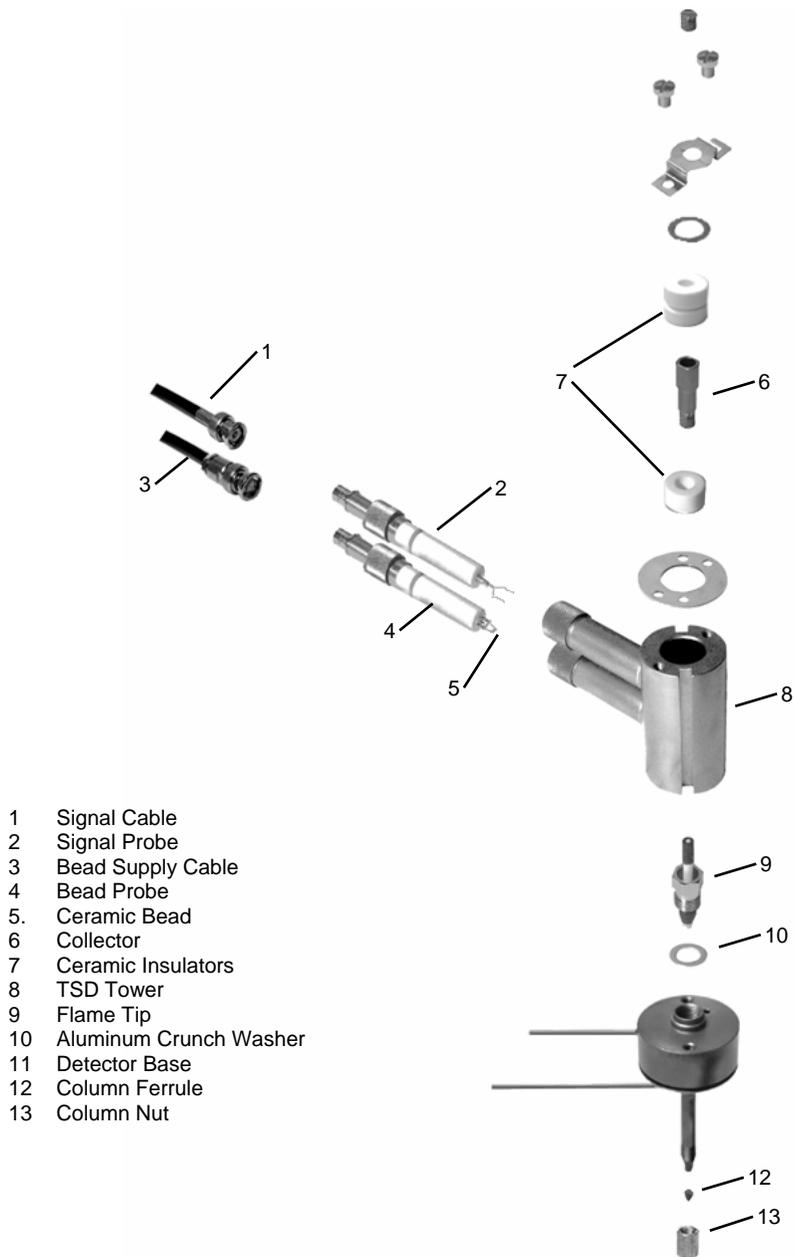
TSD Assembly

It may be necessary to remove the TSD detector components for inspection, cleaning, parts replacement, or installation of another detector. Disassembly/re-assembly instructions are detailed below for both the TSD and the detector oven.

TSD Disassembly

The TSD is mounted on the ion detector base in the detector oven.

Figure 17 is an exploded view of the TSD. GC covers should be removed.



- 1 Signal Cable
- 2 Signal Probe
- 3 Bead Supply Cable
- 4 Bead Probe
- 5. Ceramic Bead
- 6 Collector
- 7 Ceramic Insulators
- 8 TSD Tower
- 9 Flame Tip
- 10 Aluminum Crunch Washer
- 11 Detector Base
- 12 Column Ferrule
- 13 Column Nut

Figure 17 TSD Exploded View

1	Turn the TSD Electronics and TSD Oven Power OFF. Allow the detector to cool.
2	Turn off the main GC gas supplies to the detector at the pneumatics panel.
3	Disconnect the signal and the ignitor cables from the TSD tower.
4	Remove the two 8-32 x 3-inch tower mounting screws and lift the detector tower vertically from the detector base until clear of the flame tip. Remove the two 8-32 x ¼ - inch screws from the top clamp. Note: Use clean cotton gloves or tweezers when handling internal parts of the TSD.



CAUTION

Avoid contamination of ceramic insulators and probes. If the detector is not completely cool, use a metal tool (such as tweezers or a hooked wire) to remove parts from the tower assembly.

5	Remove the upper insulator.
6	Remove the signal probe. There may be some resistance to removal, due to the o-ring seals. Do not rotate probe as it is withdrawn from electrical contacts in tower.
7	Remove the collector and the lower insulator with tweezers.
8	Remove the bead probe. There may be some resistance to removal, due to the o-ring seals. Do not rotate probe as it is withdrawn from electrical contacts in tower.
9	Carefully remove the flame tip assembly from the detector base.
10	Remove and discard the aluminum seal washer from detector base. In some cases this washer may remain in the detector tower. If so, be sure to remove and discard this washer so that the detector is not accidentally reassembled with both the old washer and a new washer.

TSD Re-assembly

Refer to Figure 18 for re-assembly orientation.

1	If you removed the detector oven from the plenum, restore the oven to its former position and reinstall the four screws holding the oven to the plenum.
2	Install the flame tip in the detector base. This is a 0.020-inch ID ceramic flame tip. The TSD and FID flame tips are identical. DO NOT use an 0.010-inch flame tip for the TSD. <ul style="list-style-type: none"> If you are installing the flame tip assembly with a used Vespel® ferrule, tighten the assembly finger-tight plus an extra 1/6-turn. If you are installing the flame tip assembly with a new Vespel ferrule, tighten about 1/3-turn past finger-tight.
3	The Vespel ferrule supplied with the GC has a maximum temperature limit of 350°C. If it is ever necessary to operate the detector above 350 °C, replace the Vespel ferrule with a graphite ferrule.

4	Install a new aluminum seal washer onto the shoulder of the detector base. Note: For a reliable tower seal, use a new aluminum washer each time the detector is installed.
5	Measuring the nominal 4.0 mL/min H ₂ flow for the TSD may be simplified at this time by attaching a tube to the flame tip, and then connecting the tube to a flowmeter. Make sure the makeup gas is turned off and adjust the hydrogen flow. This method eliminates flow measurement errors which may be caused by leaks due to poor seals.
6	Replace the detector tower on the detector base and secure with the two 8-32 screws. Tighten screws alternately as tower is tightened into place.



CAUTION

Handle the ceramic insulators and probes with tweezers to avoid contamination. Use care when handling to make sure bead probe is not bent or dropped, as the ceramic bead on the probe is fragile.

7	Carefully insert bead probe into lower arm of detector tower. Align probe key with tower arm slot.
8	Secure bead probe by tightening knurled nut.
9	Insert lower insulator with shoulder down into detector tower, then insert collector tube with grid down. Make sure the punch mark on the top edge of the collector is toward the probe arm.
10	Insert signal probe into upper arm of detector tower. Carefully align the spring clip with the collector. Be sure the key on the probe and the tower arm are aligned. Secure signal probe by tightening knurled nut.
11	Assemble the upper insulator and top clamp over the collector. The O-ring will provide slight resistance to complete insertion. Take care not to tear the O-ring or the tower will not seal properly.
12	Look down the tower assembly to verify that the bead does not touch the collector. Carefully adjust the collector as necessary.
13	Assemble the top clamp to the tower and secure with the two 8-32 screws.
14	Connect the signal cable to the signal probe (top) and the ignitor cable to the bead probe (bottom). If cables were totally disconnected from the GC, reconnect signal cable and the bead current supply cable at the top of the TSD Electrometer Board.

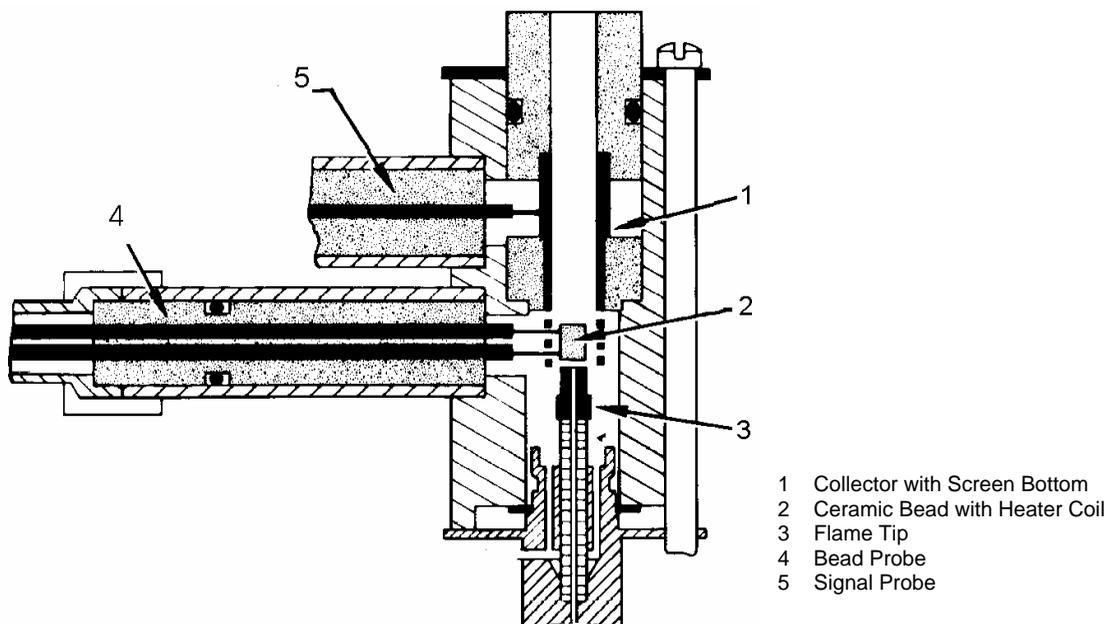


Figure 18 TSD Cross-Sectional View

***PC Board
Removal/
Installation***

To remove the TSD PC Board, proceed as follows:



*Turn the GC power OFF when
removing or installing PC Boards*

1	Turn the GC power OFF.
2	Disconnect the signal and ignitor cables from detector.
3	Remove the left side panel from the GC by removing the 8 -32 screws.
4	Remove the 8 -32 screw securing the TSD electrometer board in the detector bay. Slide the PC board straight out of the guide slots in the cabinet. If possible, do not touch the edge connectors. Place the PC board in a clean anti-static bag.
5	To install the PC board, align the groove in the electrometer cover with the guide rail at the top of the detector bay and slide the electrometer into the opening until the edge connector seats in the socket. Never force the board into the cabinet. Make sure that all cables are out of the way before you insert the board. Reconnect the cables to the detector. Check that the connectors mate.

Thermal Conductivity Detector

The Thermal Conductivity Detector (TCD) is mounted directly above the column oven on the 3800 GC. The TCD PC Board is installed in the electronics cabinet on the left side of the instrument.

TCD operating parameters are set at the 3800 keyboard: These include carrier gas type, detector temperature, range, temperature limit, and filament temperature. Carrier gas flow is set and controlled at the pneumatics panel, and in the Adjustments section of the method if Detector EFC is installed.

Initial Set-Up

To set up the TCD, proceed as follows. Note that the TCD is a two channel detector and requires both a sample flow of carrier gas and a reference flow of the same gas. Either channel can be used as the sample stream but there must always be a reference stream.

1	Connect the gas lines to the appropriate bulkhead fittings on the GC.
2	Check the gas supply pressures. Flow controllers with turn dials are calibrated for Helium at 80 psig. Recalibrate the flow controllers if you use a different supply pressure or carrier gas.

3	Establish carrier gas flow through the Sample (right) side of the TCD and an equal Reference gas flow through the Reference (left) side of the detector. If you are using a capillary column, the total of column flow and makeup flow should equal the Reference gas flow. Perform a Leak Check on both the Sample and Reference pneumatics before proceeding to apply power to the TCD filaments. Even a small leak can supply enough oxygen to cause filament oxidation and consequent baseline drift. If your TCD is equipped with detector EFC, enter Setup. Select the EFC section for your TCD and select the gas type you will use for makeup and reference. When you have completed changes to Setup, press the Save and Exit softkey to store your changes and restart the GC.
----------	--

TCD Adjustments

There are two TCD parameters which need to be set before using the TCD for analysis. These are the choice of carrier gas and the filament temperature limit.

1	Press the Detector key and select the TCD by location. Press the Adjustments softkey and select the carrier gas setting. If you are using helium or hydrogen as carrier gas, select the He setting. For all other carrier gases, select the N ₂ /Ar setting.
2	Set the filament temperature limit. To protect the filament, always operate the detector with the 390 °C limit selected unless you require the maximum dynamic range available to the TCD.

The following page is accessed by pressing the Adjustments softkey.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 120		Actual (°C): 120		Signal (mV): 10.835
Filament Current (mA): 220		Balance (%): -42		
FRONT TCD ADJUSTMENTS, Page 1 of 2				
Time Constant::		Fast		
Carrier Gas:		He		
Filament Temp Limit:		390		
<div style="border: 1px solid black; display: inline-block; padding: 2px 10px;">CLEAR AUTOZERO</div>				
				

With prolonged use, the filaments slowly oxidize and their resistances increase. At some point this will cause the largest peaks in your chromatogram to become flat topped, indicating that the filament protection software has been activated. (Note that if the TCD signal is sufficiently large

the output may go to zero and become unresponsive. If this occurs, adjust your injection site and conditions to avoid over range conditions.) To adjust for this increase in resistance, set the Filament Temperature Limit to 490 °C, even for filament temperature setpoints below 390 °C.

The TCD filament is automatically protected in the GC. The GC turns off the filament current after four minutes if it detects that the carrier gas flow has stopped or that the filament current is low. Also, the GC turns off filament current when it detects that carrier gas is flowing through one side of the detector cell but not through the other (e.g., a septum or column is not installed, or a leak exists in one cell and not the other).

If you select Helium or H₂ as the carrier gas, the GC operates in the standard way to protect the TCD filament. However, because the filament protection feature in the GC operates through the detection of air (or N₂) in the detector cells, the filament protection feature is disabled when you use nitrogen or argon as the carrier gas.

Set the Filament Temperature Limit to 390 °C or 490 °C, according to the highest filament temperature setpoint required. With the Limit set to 390 °C, the peak sample filament temperature is limited to approximately 450 °C. The temperature limit is approximately 550 °C at the 490 °C setting. The 450°C limit protects filaments from oxidation when exposed to air over an extended period of time. However, at the 550 °C temperature limit the filament can degrade after only a few minutes.



CAUTION

If you select N₂/Ar as the carrier gas, the filament protection feature is disabled. Because the GC does not turn off the filament current, the TCD filaments can rapidly oxidize if you operate the TCD filament temperature above 390 °C without carrier gas flowing through the TCD cells. If you plan to operate the TCD after the carrier gas has been turned off, purge the air from the TCD cells with carrier gas for five minutes before operating the TCD.

The second adjustments page is only accessible if detector EFC is installed. Note that a TCD can be used with two different types of EFC. The following example is for type 14, which provides make-up flow to one channel of the TCD and reference flow to the other channel.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Make-up Flow (ml/min): 25		Ref. Flow (ml/min): 30		
FRONT TCD ADJUSTMENTS, Page 2 of 2				
Make-up Flow (ml/min):		25		
Reference Flow (ml/min):		30		

The other EFC choice for TCD is 16. Type 16 provides two channels of Hydrogen, one for makeup and one for Reference gas.

Operation

To operate, the TCD requires only carrier gas. Helium or hydrogen are recommended as carrier gases. You can use nitrogen as a carrier gas, but you may lose some sensitivity and see an increase in the detector noise.



WARNING:
FIRE HAZARD

If hydrogen is used as a carrier gas, vent the hydrogen from the TCD to a safe place. Hydrogen is very flammable.

Before Operating the TCD

Do not operate the TCD until you have connected carrier gas to both inlets of the TCD. The TCD accommodates two columns (a column for the reference cell and a column for the detector cell) or a column and a reference line. The columns can be packed or capillary columns and you can combine a capillary column with a packed column. To avoid contaminating the detector, condition all packed columns before you connect them to the TCD.

For the following discussion, assume that the analytical column is attached to the right side of the TCD and the reference column or reference gas supply to the left side of the TCD. However, you can configure the GC with the analytical column connected to the left side of the TCD, simply reverse the TCD polarity.

Note: *If you plan to operate the TCD after the carrier gas has been turned off for an extended period of time, turn on the carrier gas for five minutes before turning on the filament current to purge air from the system. This prevents oxidation of the TCD filaments.*

At the factory, the carrier gas flow rate for TCD is set at 30 mL/min. (Carrier gas: He) through both sides of the detector. The carrier gas flow rate is adjusted at the pneumatics cabinet and measured at the two exit ports on the TCD. Once the carrier gas is flowing through the TCD, set up the TCD for operation as follows:

1	Set the TCD oven to the desired temperature and allow it to come to the setpoint. Usually, set the TCD temperature at least 20 °C higher than the maximum temperature the column oven reaches in your analysis.
2	Set TCD filament temperature in the detector section. A filament temperature 50 °C above the detector temperature should provide a good starting point for most applications. After the TCD oven has reached its set temperature, turn the TCD Electronics ON to begin heating the filaments.
3	Allow system to equilibrate 10 to 15 minutes.
4	Monitor the filament current through the detector status display. Initially, the current is greater than the final operating current. Gradually, however, the current decreases as the detector thermally equilibrates. For best performance of the detector, do not permit the filament temperature to exceed the detector temperature by more than 200 °C. Generally, use the lowest detector current possible for the sensitivity your application requires.
5	Page forward to page 2 of the TCD section and set the initial Autozero OFF. Press the Autozero soft key to set the initial signal to 0. Monitor the signal on the detector status display. A continuous drift, either positive or negative, may indicate an air leak in either the carrier or reference gas stream.
6	Note that it is normal for the TCD baseline to drift until the detector thermally equilibrates. The time it takes to reach equilibrium always exceeds the time it takes for the detector oven to reach its temperature. When the baseline is stable, you can begin your analysis. Turn initial Autozero back ON. Autozero is automatically disabled when the GC method is in RUN.

Page 1 of the TCD method:

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 120		Actual (°C): 120		Signal (mV): 10.835
Filament Current (mA): 220		Balance (%): -42		
FRONT TCD, Page 1 of 2				
TCD Oven Power:			ON	
Temperature (°C):			120	
Electronics			ON	
Range:			0.5	
Filament Temperature:			170	
		AUTOZERO		ADJUSTMENTS

The AUTOZERO softkey applies an immediate zeroing of the detector signal.

Page 2 allows time programming events such as range changes.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME																
Method 1	Method 1		0.00	20.00																
Set (°C): 120		Actual (°C): 120		Signal (mV): 10.835																
Filament Current (mA): 220		Balance (%): -42																		
MIDDLE TCD, Page 2 of 2.																				
<table border="1" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th>Time</th> <th>Range</th> <th>Autozero</th> <th>Polarity</th> </tr> </thead> <tbody> <tr> <td>Initial</td> <td>0.5</td> <td>NO</td> <td>Positive</td> </tr> <tr> <td>5.00</td> <td>0.5</td> <td>YES</td> <td>Positive</td> </tr> <tr> <td>6.50</td> <td>0.5</td> <td>NO</td> <td>Positive</td> </tr> </tbody> </table>					Time	Range	Autozero	Polarity	Initial	0.5	NO	Positive	5.00	0.5	YES	Positive	6.50	0.5	NO	Positive
Time	Range	Autozero	Polarity																	
Initial	0.5	NO	Positive																	
5.00	0.5	YES	Positive																	
6.50	0.5	NO	Positive																	
		AUTOZERO		ADJUSTMENTS																

Initial Autozero = YES allows the system to balance the bridge automatically before each run for maximum dynamic range. The current status of the balance control, from -99% to +99%, is shown on the display.

A continuous drift in the balance value over time may indicate you have an air leak which is causing one set of filaments to oxidize, changing their resistance and unbalancing the detector bridge.

Range Setting

Use the 0.05 mV range in the following cases:

- Low currents (100-150 mA)
- High Currents (300 mA)
- Output Signal Fed to Computer or Integrator

Low Currents (100-150 mA)

Analyses that had required a high filament current and a less sensitive range can often be performed at lower currents and a more sensitive range. For example, a filament current of 120 mA and a range of 0.05 mV yields about the same output signal as a filament current of 300 mA and a range of 0.5 mV. At the lower current, the detector noise is lower, the stability is higher, and the filament lifetime is extended. Thus, Varian recommends that, whenever possible, you operate the TCD at the lowest practical filament current.

The chromatograms in Figure 19 illustrate the high sensitivity and detectivity (18 ppm of Neon in air) that is possible with the TC Detector. Also, a comparison of chromatogram A with chromatogram B shows that with the use of the bridge output signal amplifier, you can obtain the same analytical results at half the filament current, thereby extending the lifetime of the TCD filament. The Neon peak in chromatogram C illustrates how you can use a high filament current (300 mA) with a high amplification setting to increase the effective sensitivity of the TCD.

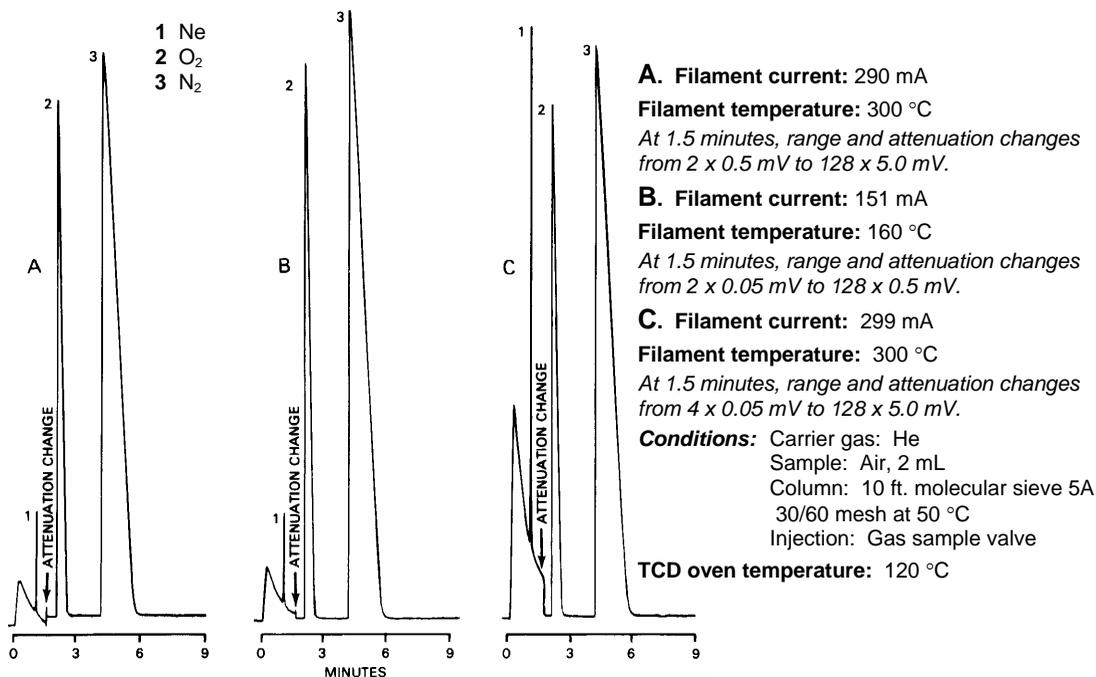


Figure 19 TCD Sensitivity and Amplification

High Currents (300 mA)

To obtain the highest possible sensitivity, operate the TCD at the 0.05 mV range with a high filament current. In this configuration, TCD noise is primarily noise derived from filament vibration and fluctuations in carrier gas flow through the detector. However, at high filament currents, the detector is very susceptible to air leaks into the gas lines, improperly conditioned columns, imbalanced reference and analytical flow rates, or impure carrier gas supply, which cause the baseline to drift. Baseline drift from any of these sources generally increases as the absolute filament temperature increases. Therefore, operate the TCD with as low a filament current as practical for your application.

Also, operate the TCD with the lowest filament and detector temperatures appropriate. If possible, avoid operating the TCD with a filament temperature greater than 390 °C. To operate the TCD with filament temperatures between 390 °C and 490 °C, set the Filament Temperature Limit to 490 °C. See Figure 19 for an example of the performance of the TCD at a high current.

**Calculate
Detector
Sensitivity**

To calculate the sensitivity of the TCD, proceed as follows:

EXAMPLE 1: Known Sample Weight

$$S = PF_C/W$$

Where:

S = Sensitivity in mV x mL/mg

P = Integrated peak area in mV x min

W = Weight of sample in carrier gas in mg

F_C = Carrier gas flow in mL/min corrected to detector temp

$$F_C = F_O (T_D/T_A)(1-P_W/P_A)$$

Where:

F_O = Flow rate measured at detector outlet (at ambient temperature) in mL/min.

T_D = Detector temperature in °K

T_A = Ambient temperature in °K

P_W = Partial pressure of H₂O at ambient temperature in torr

P_A = Ambient pressure in torr

Note: The $(1-P_W/P_A)$ expression applies only if using a bubble flowmeter.

EXAMPLE 2: Known Concentration of Sample in Detector

$$S = E/C_D$$

Where:

S = Sensitivity in mV x mL/mg

E = Detector signal (peak height) in mV

C_D = Concentration of test substance in the measured peak volume in mg/mL

Peak volume =

Peak width at half height (in min.) x gas flow rate at detector
(in mL/min) = $W \cdot h/2 \times FC$

**Calculate
Detector
Detectivity**

To calculate the detectivity of the TCD, proceed as follows:

$$\text{Detectivity} = \frac{2 \times \text{Noise}}{\text{Sensitivity}}$$

**Reverse
Polarity**

If you inject a standard or sample into the reference side of the TCD, peaks appear in the negative direction. If you want to integrate or process these peaks with a data system, you must convert the polarity of the peaks from negative to positive. Reference the TCD section to set initial detector polarity either Positive or Negative.

You can also time program when you would like the polarity to be automatically reversed during a chromatographic run. Set a time-programmed polarity reversal in the detector method section.

Adjust TCD Carrier Gas Flow Rates

All 3800 GCs equipped with TCDs are factory tested at the flow rate listed below. Adjustments to the carrier gas flow rate are made at the pneumatics panel of the GC.

Carrier Gas:	Helium
Inlet Pressure:	80 psig
Purity:	99.999%
Column Flow Rate:	30 mL/min
Reference Column Flow Rate:	30 mL/min

Note: *Tightly cap gas filters when not used. Prolonged exposure of the contents of the filter to room air degrades the performance of the filters.*

**Remove/
Install the
TCD PC
Board**

To remove the TCD PC Board from the GC, proceed as follows:



CAUTION

Turn OFF the power to the GC when removing or installing PC Boards.

1	Turn the GC power OFF.
2	Disconnect the TCD power/signal cable from the TCD PC Board.
3	Remove the left side panel from the GC by removing the 8 -32 screws.
4	Remove the 8 -32 screw securing the TCD controller board in the detector bay. Slide the PC board straight out of the guide slots in the cabinet. If possible, do not touch the edge connectors. Place the PC board in a clean anti-static bag.
5	Place the PC board in an anti-static bag and set aside.

To install the TCD PC Board, proceed as follows:

1	To install the PC board, align the groove in the controller cover with the guide rail at the top of the detector bay and slide the controller into the opening until the edge connector seats in the socket. Never force the board into the cabinet. Make sure that all cables are out of the way before you insert the board.
2	Reconnect the TCD signal cable. Make sure that the connectors mate.

Electron Capture Detector



WARNING: RADIATION SOURCE

The Electron Capture Detector contains a beta-emitting radioactive isotope, ^{63}Ni . Users of this detector are required by regulations of the Nuclear Regulatory Commission (NRC) to read the radiation safety procedures described in the Radiation Safety Manual (03-913999-00).

The ECD is installed on the detector oven, directly above the column oven. The ECD PC Board is installed in the electronics cabinet. The electronics of the ECD are controlled through the 3800 keyboard, whereas gas flows are set and controlled at the pneumatics panel and in the Adjustments section of the Method if Detector EFC is installed.

An ECD equipped with a captive detector cell is included in the ECD Kit. Varian is authorized to distribute this kit to General Licensees, as defined in the Radiation Safety Manual.

Due to the NRC regulations on the testing, packaging, and labeling of radioactive materials, additional or replacement ECD detector cells are available only as kits.

Initial Set-Up

1	Connect gas lines to the appropriate bulkhead fittings on the GC.
2	Check the gas supply pressures. The GC is set for: N_2 , He, or Ar/CH_4 at 80 psig. Reset the gas flows if you intend to use different supply pressures. (Refer to the 3800 Pre-Installation Instructions for additional information on gas supply requirements.) If your ECD is equipped with detector EFC, enter Setup, select the EFC section for your ECD, and select the gas type you will be using as makeup. When you have completed all changes to Setup, press the Save and Exit softkey to store your changes and restart the GC.

Page one includes initial parameters for the detector oven temperature, turning on the detector oven power and electronics, and selecting the range. Page two establishes time programs to either change the range or Autozero the detector at specific times.

The third and fourth pages (located by pressing the adjustments softkey) include detector-specific parameters not routinely adjusted. For the ECD, these pages allow you to select between a fast or slow detector time constant, Cell Current and Contact Potential which are described later in this section. Page 2 of adjustments allows setting detector gas flows if detector EFC is installed. The normal setting for the time constant is fast.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 300	Actual (°C): 300		Signal (mV): 2.15	
FRONT ECD, Page 1 of 2				
	ECD Oven Power:		ON	
	Temperature (°C):		300	
	Electronics:		ON	
	Range:		10	
		AUTOZERO		ADJUSTMENTS

The AUTOZERO softkey applies an immediate zeroing of the detector signal.

Page 2 allows time programming events such as range and Autozero.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME												
Method 1	Method 1		0.00	20.00												
Set (°C): 300	Actual (°C): 300		Signal (mV): 2.15													
FRONT ECD, Page 2 of 2.																
<table border="1"> <thead> <tr> <th>Time</th> <th>Range</th> <th>Autozero</th> </tr> </thead> <tbody> <tr> <td>Initial</td> <td>10</td> <td>YES</td> </tr> <tr> <td>5.00</td> <td>10</td> <td>YES</td> </tr> <tr> <td>6.50</td> <td>1</td> <td>NO</td> </tr> </tbody> </table>					Time	Range	Autozero	Initial	10	YES	5.00	10	YES	6.50	1	NO
Time	Range	Autozero														
Initial	10	YES														
5.00	10	YES														
6.50	1	NO														
		AUTOZERO		ADJUSTMENTS												

ECD Adjustments

There are ECD adjustments which should be set before operating the detector. These are located by pressing the Adjustments softkey in the ECD method.

Cell-Current In order to obtain the optimum linear and dynamic ranges of response, you must select the proper ECD cell current. Your choice of cell current depends on the specific carrier gas that is used and the baseline signal (frequency, f_0) with only carrier gas present. One of four values may be selected for the cell current: Ar/CH₄ (1710 pA), CAP (480 pA), N₂STD (290 pA), and N₂HIGH (146 pA). In addition, a Zero cell current setting is provided for convenience in setting the cell contact potential (see below).

For normal operation with nitrogen carrier gas, use the N₂STD setting. When a greater linear range is desired, select the Ar/CH₄ setting and use 90% Argon/10% Methane as the carrier gas.

Set the switch to CAP when using capillary columns. For capillary columns, the column bleed is low and the baseline is usually below 1 mV when current is set to N₂STD on Range 1.

You may encounter applications in which high column bleed is unavoidable when using nitrogen as the carrier gas. In such instances, the baseline signal may be too high. Select the N₂HIGH setting in these situations.

In general, the baseline signal should never exceed 25 mV when measured on Range 1. A value above 25 mV indicates that you have selected the wrong reference current for the chromatographic conditions (column, phase, sample matrix). A baseline signal above 25 mV decreases the useful linear range of the ECD detector.

Time Constant Switch The time constant has two settings: SLOW (200 msec) and FAST (50 msec). For most applications, select the SLOW setting. For capillary applications with peak widths at half height of less than 2 seconds, select the FAST setting.

Setting the Contact Potential An extremely clean ECD system can yield a contact potential that must be nulled for optimum performance. The contact potential may vary with time and detector temperature for several days after the cell is first put into operation, therefore you may need to repeat this adjustment procedure daily until the contact potential stabilizes.

Install a conditioned column and establish carrier gas and makeup flow. If the column is not well conditioned, remove the column from the detector inlet and cap off the inlet to the ECD. Condition the column as recommended by the supplier, then reconnect the column to the detector. **NEVER** operate the ECD cell at high temperature without a source of inert gas, either carrier gas from the column or makeup gas. Conditioning a column connected to the ECD detector can severely contaminate the detector, requiring a costly cleaning to restore proper function!

Set the ECD detector to the desired operating temperature for your application, typically 300 °C, by pressing the Detector key on the GC front panel, selecting the ECD, entering the desired temperature, and setting the oven power to ON. Turn the Electronics ON and set the Range to 1. Page forward to page two and set the initial Autozero to NO. Allow the detector to come to temperature and equilibrate for several hours, or preferably overnight.

From the ECD screen, press the soft key labeled Adjustments to display the Cell Current control. Press the “Clear Autozero” softkey. Select a cell current of Zero. Set the contact potential to -760 mV. The ECD signal on the 3800 display should read between -12.7 and -13.0 mV. Increase the contact potential to +760 mV. The ECD signal should increase by several mV, but may still be negative. Now adjust the contact potential in a negative direction until the ECD signal is within 0.5 mV of its original value, e.g., if the signal was -12.7 mV, adjust the contact potential until the ECD signal reads -12.2 mV. Now set the cell current to the desired value.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Set (°C): 300	Actual (°C): 300		Signal (mV): 2.15	
FRONT ECD Adjustments, Page 1 of 2				
	Time Constant:		Fast	
	Cell Current:		CAP	
	Contact Potential (mV):		230	
<div style="border: 1px solid black; padding: 2px; display: inline-block;">CLEAR AUTOZERO</div>				
○	○		○	

The Clear Autozero softkey clears the previously stored detector background signal level.

The second adjustments page is only accessible if detector EFC is installed.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Make-up Flow (ml/min): 25				
FRONT ECD ADJUSTMENTS, Page 2 of 2				
Make-up Flow (ml/min)		25		

**ECD
Operation**

Observe the following procedures when operating with an ECD.

**Column and
Septum
Bleed**

The ECD can become contaminated by chemicals that bleed from columns and septa. Such column and/or septum bleed can seriously decrease the ECD cell current which may result in an unacceptable increase in the frequency/output signal. To prevent such contamination, condition all columns and septa before you use them with the ECD.

**Column
Conditioning**

NEVER condition a column while it is connected to the ECD detector. Instead, remove the column from the detector, cap the ECD column fitting with a nut, and a no-hole ferrule. Maintain makeup gas flow to the detector to keep the ECD cell clean. Condition new columns for a minimum of 10 hours at 40 °C below the maximum temperature of the column stationary phase, or as directed by the column supplier. Complete the column conditioning phase before you connect the column to the ECD.

**Septa
Conditioning**

Place septa in a clean glass beaker or dish and bake the septa in the GC oven at 150 °C for 30 minutes. Complete the septa conditioning phase before you place the septa in the injector.

**Column Oven
and Detector
Oven
Temperature
Selection**

In selecting ECD oven temperatures, consider the following:

- The maximum permissible operating temperature of the radioactive foil is 400 °C. This foil temperature cannot be reached, even when the detector oven temperature is set to 450 °C. Do not add additional insulation material around the detector tower. Added insulation can raise the foil temperature and reduce its operating lifetime.
- A high detector oven temperature reduces the likelihood of contaminating the foil with material bleeding from the column.
- The higher the temperature of the foil, the greater the rate of migration of the ⁶³Ni into the backing material. Rapid migration of the ⁶³Ni reduces the electron flux and the lifetime of the foil.
- Background noise increases with an increase in the detector and column oven temperatures.

Use these guidelines to select the detector oven temperature:

- Detector temperature should be at least 30° higher than the maximum column temperature, but not less than 150 °C.
- To prevent column bleed from fouling the detector, wait for the detector to reach its operating temperature before you increase the column temperature.

ECD Pneumatics

The 3800 GC is tested at the factory at the flow rates listed in the *Getting Started* manual, Table 2. Use these flow rates as a starting point. Adjust the flow rate at the pneumatics panel before you use the detector. Measure the flow rate at the exit tube of the ECD cell.

You may need periodically to clean the detector cell with carrier gas. Flow rates for the thermal cleaning procedure are also listed in the *Getting Started* manual, Table 2.

Refer to the installation section for a list of gas filters recommended for packed column or capillary applications.

Note: Each ECD kit is equipped with a Teflon® flow tube assembly. When setting/checking the flow rate, attach the end of the flow tube with the ferrule to the 1/16-inch OD exit tube of the ECD cell. Attach the other end of the flow tube assembly to a flowmeter. To prevent cross-contamination between detectors, use the flow tube only with the ECD and store the assembly with the ECD kit.



WARNING: RADIATION SOURCE

After prolonged use of the ECD detector, radioactive contaminants may build-up on the detector cap and exit tube. In such cases, it is possible that you could come in contact with radioactive material when checking or adjusting the carrier gas flow rate. Refer to Radiation Safety Manual. Always wash your hands after handling the top of the ECD.

Table 25 ECD Gases, Pressures, Purity, and Flow Rates

Gas	Inlet Pressure at Gas Cylinder	Minimum Purity	Recommended Flow Rate (mL/min)
Carrier: N ₂ , He, or Ar/CH ₄ (90:10)	80 psig	99.999%	30 (packed column) ≤5 (capillary columns)
Detector Make-Up (capillary): N ₂ or Ar/CH ₄	80 psig	99.999%	20 to 30

Installation/Disassembly

If you need to remove the detector components to inspect, clean, or replace parts, or to install another detector, refer to the following disassembly/re-assembly instructions for the ECD and the ionization oven.



WARNING: RADIATION SOURCE

In the ECD Kit (P/N 02-001972-00) the detector cell, which contains the radioactive ionization source, is fixed within the detector tower and cannot be removed. Do not attempt to remove the cell from the tower. Regulations for the handling of radioactive materials strictly prohibit unlicensed disassembly. Evidence of removal voids the warranty. Violations of these regulations are reported to NRC.

1	Cool the ECD to 50 °C.
2	Remove the detector tower cap and insulation.
3	Remove the signal probe and pulser probe.
4	Insert the installation tool into the detector tower assembly (located over the detector cell). Loosen the hex nut until the cell turns freely
5	Remove the two 8-32 x 3-inch tower mounting screws.
6	Remove the detector from the detector oven.

After removing the ECD, place the cap on the tower, and place the cooled detector and installation tool in the ECD case. Store the detector in an area that is clean and of a low humidity. Refer to the Radiation Safety Manual for instructions on the storage and shipping of the ECD detector.

If the ECD is to be transferred to another GC, transfer the "CAUTION RADIOACTIVE MATERIAL" label to the front of the other GC as well. However, if you removed the detector only to gain access to the detector oven, and you plan to reinstall the detector, place the ECD in a clean, uncontaminated area and proceed to the next paragraph.

ECD Installation

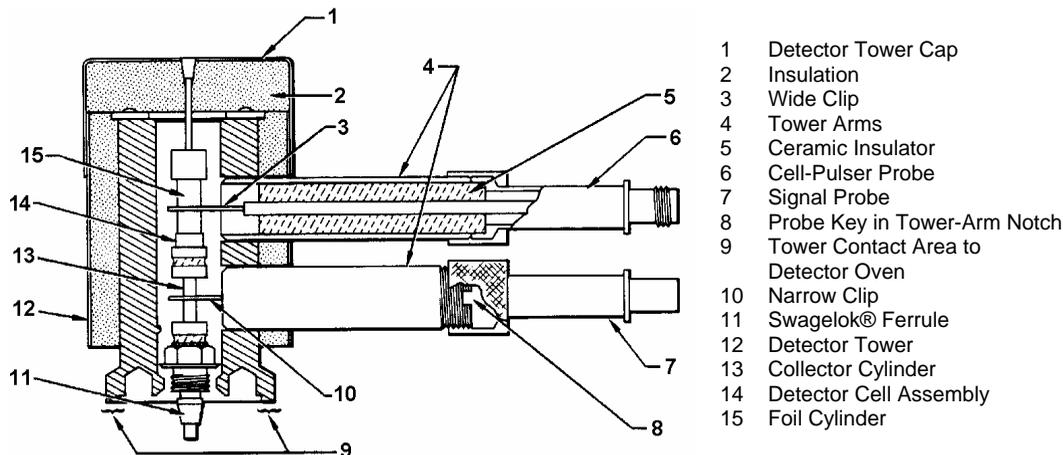


Figure 20 Cross-Sectional View of the ECD

Note: Because of the sensitivity of the ECD to oxygen, check the pneumatics for leaks prior to its installation.

1	Turn the detector oven power OFF.
2	Remove the detector cover and the GC top cover.
3	Remove both probes (pulsar and signal) from the tower arms (see Figure 20) of the detector assembly. Note: Be sure both probes have been removed from the detector tower arms before attempting to install the detector tower. If you insert the installation tool before you have removed the probes you will damage the probe electrode contacts.



WARNING

NON-SERVICEABLE ASSEMBLY. Do not attempt to remove the cell assembly which is fixed inside the tower.

4	Check to see whether the detector tower, base, and detector cell fittings are clean. Install the ECD shim on the detector base. Note: Poor metal-to-metal contact between the tower contact area and the detector oven can produce a temperature gradient.
5	Remove the insulated detector cap.

6	<p>Position the tower assembly on the detector base and install the two 8-32 x 3-inch tower mounting screws loosely in the tower assembly.</p> <p>Note: <i>Do not cross-thread the tower mounting screws.</i></p>
7	<p>Insert the installation tool into the detector tower assembly (located over the detector cell). Engage the hex nut at the lower end of the cell assembly. Tighten the cell assembly into the base to provide a leak-free seal. <i>Do not over-tighten.</i></p>
8	<p>Alternating between the two 8-32 x 3-inch tower mounting screws, evenly tighten the tower into place.</p>
9	<p>Carefully install the signal and pulser probes in the tower arms. The signal probe (bottom) has a twist-lock cable connector and a narrow clip to engage the collector cylinder. The pulser probe (top) has a threaded cable connector and a wide clip to engage the foil cylinder. Insert the probes into the tower arms with the keys on the probes lined up with the notches in the tower arms. If you encounter any resistance during the insertion of the probes, check that the probes are being installed in the correct positions.</p>
10	<p>Install the insulated detector tower cap. The label shown in Figure 21 is factory installed on a detector tower registered as a General License Device.</p>
11	<p>Connect the pulser and signal cables to the appropriate probes of the ECD. Refer to the designation on each cable connector.</p>
12	<p>Cable connections for the operation of the ECD require installation of the ECD Electrometer Board.</p>
13	<p>Connect the gas supplies at the rear of the GC.</p>
14	<p>Replace the GC top cover and the detector cover.</p>
15	<p>Install the "CAUTION RADIOACTIVE MATERIAL" label (P/N 31-000347-00) in a location on the front of the GC that is clearly visible. This label describes the ECD radioactive isotope and the amount of radioactivity (Figure 22).</p>
16	<p>Return the installation tool to the ECD case. You will use this tool each time you remove the ECD. Retain the ECD case should you need to store the ECD or to return the detector to Varian. See the Radiation Safety Manual.</p>

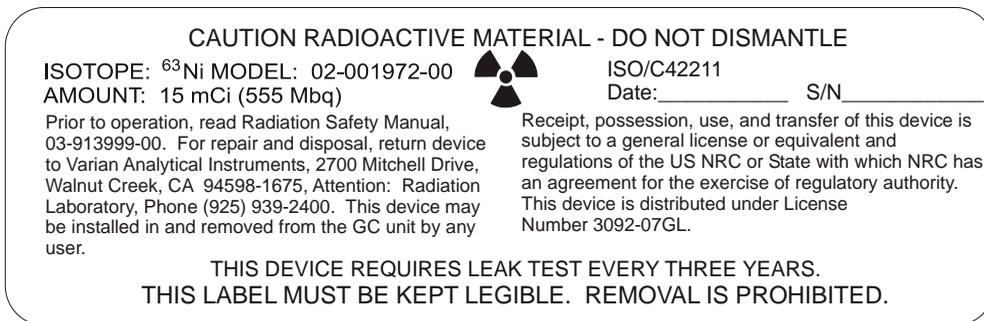


Figure 21 Radioactive Caution Label - Factory Installed

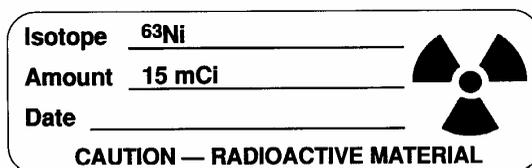


Figure 22 Radioactive Caution Label - User Installed

**PC Board
 Removal/
 Installation**

The ECD PC Board is a slide-in module. To remove the ECD PC Board, proceed as follows:



**WARNING:
 SHOCK HAZARD**

Dangerous voltages exposed. Turn the GC power OFF when you remove or install PC boards. Failure to do so may result in accidental contact with dangerous voltages, or damage to the PC board or GC.

1	Turn the GC power OFF.
2	Disconnect the signal and pulser cables from the electrometer.
3	Remove the left side panel from the GC by removing the 8-32 screws.
4	Remove the 8-32 screw securing the ECD electrometer board in the detector bay. Slide the PC board straight out of the guide slots in the cabinet. If possible, do not touch the edge connectors. Place the PC board in a clean anti-static bag.
5	To reinstall the electrometer board, insert the PC board into the guide slot on the top of the electrometer bay, then slide the board into the connector on the mother board. Never force a board into the cabinet. Make sure that all cables are out of the way before you insert the board. Reconnect the cables to the detector. Check that the connectors mate.

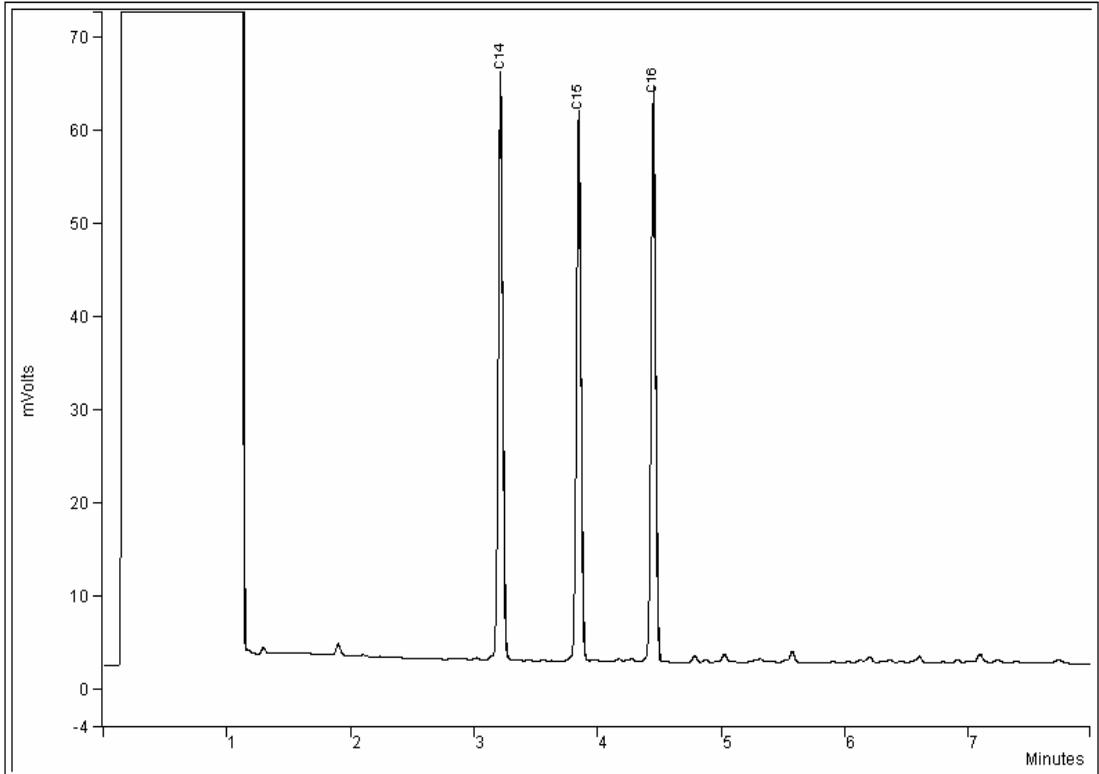
Pulsed Flame Photometric Detector

The PFPD is supplied with a separate manual, P/N 03-914649-00. All 3800 GCs equipped with a PFPD will be supplied with this manual which contains complete setup and operating information for the PFPD.

Test Chromatograms

The following figures show typical chromatograms of the Varian test mixes for the CP-3800 detectors.

File Edit View Results Edit Method Help



FID Test Mix

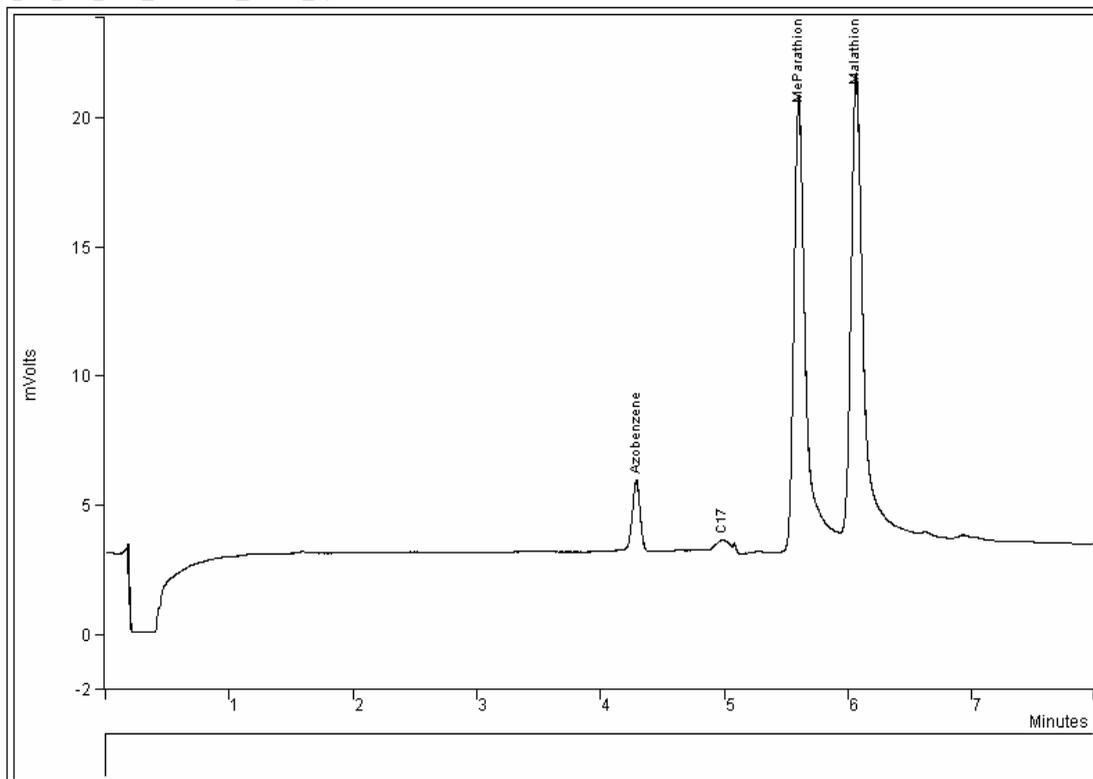
Column: Non-polar, e.g., 1 μ film thickness, 15m x 0.25 mm, CP-Sil 5 CB

Carrier: Nitrogen, 5 mL/minute

Injection: 1079, 200 °C, 1 μ L splitless

Temperature Program: 80 °, hold 1 minute, 20°/minute to 200°, hold 1 minute

Detector: 300 °C, Range 12



TSD Test Mix

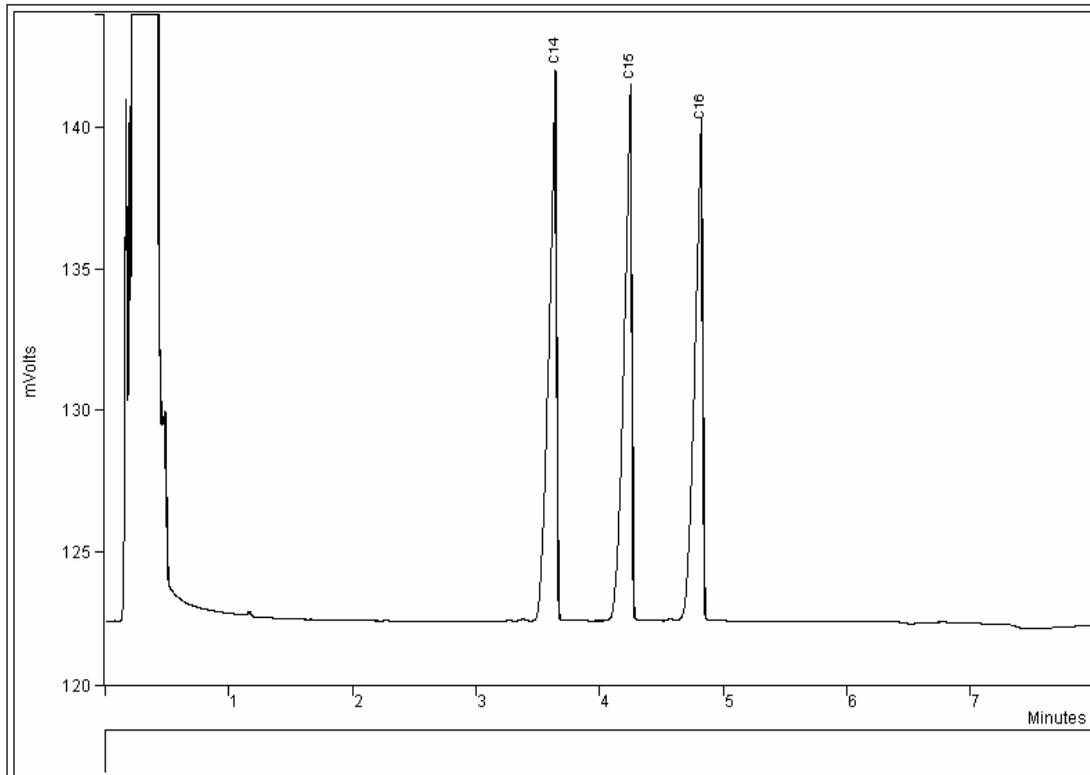
Column: Non-polar, e.g., 1 μ film thickness, 15m x 0.25 mm, CP-Sil 5 CB

Carrier: Nitrogen, 5 mL/minute

Injection: 1079, 200 °C, 1 μ L splitless

Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute

Detector: 300 °C, Range 12



TCD Test Mix

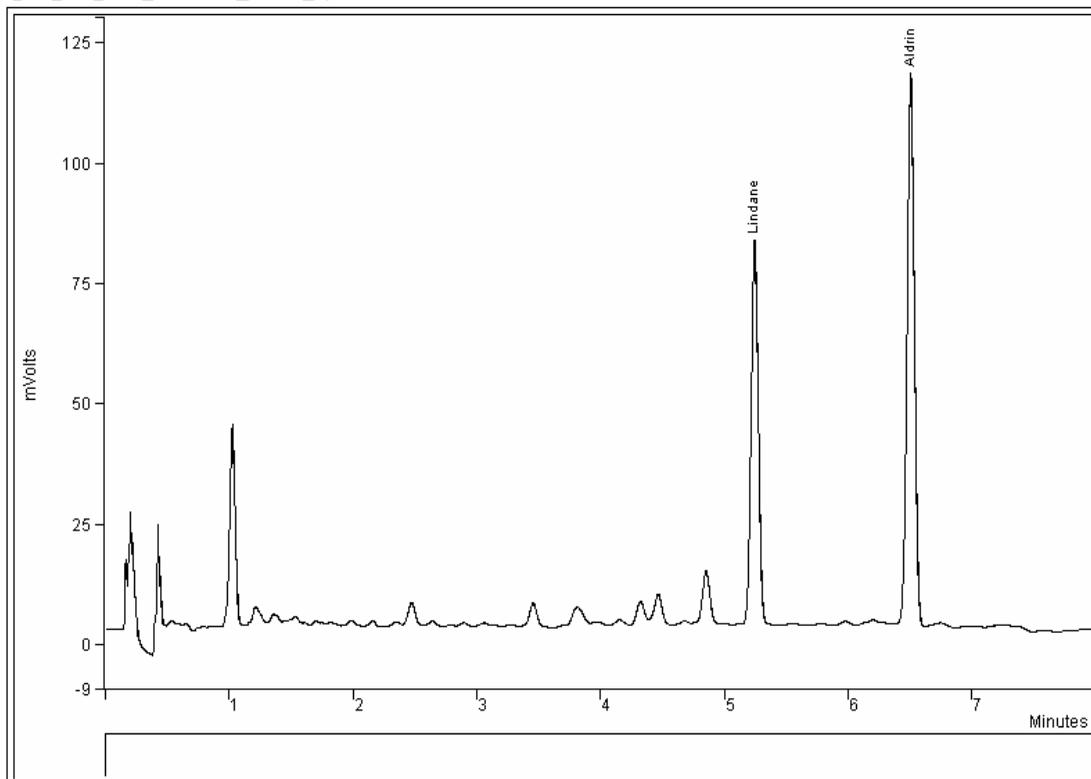
Column: Non-polar, e.g., 1 μ film thickness, 15m x 0.25 mm, CP-Sil 5 CB

Carrier: Helium, 5 mL/minute

Injection: 1079, 200 °C, 1 μ L splitless

Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute

Detector: 220 °C, Range 0.05, Filament Temperature 270° \pm 20°, Filament current 200 \pm 10 mA



ECD Test Mix

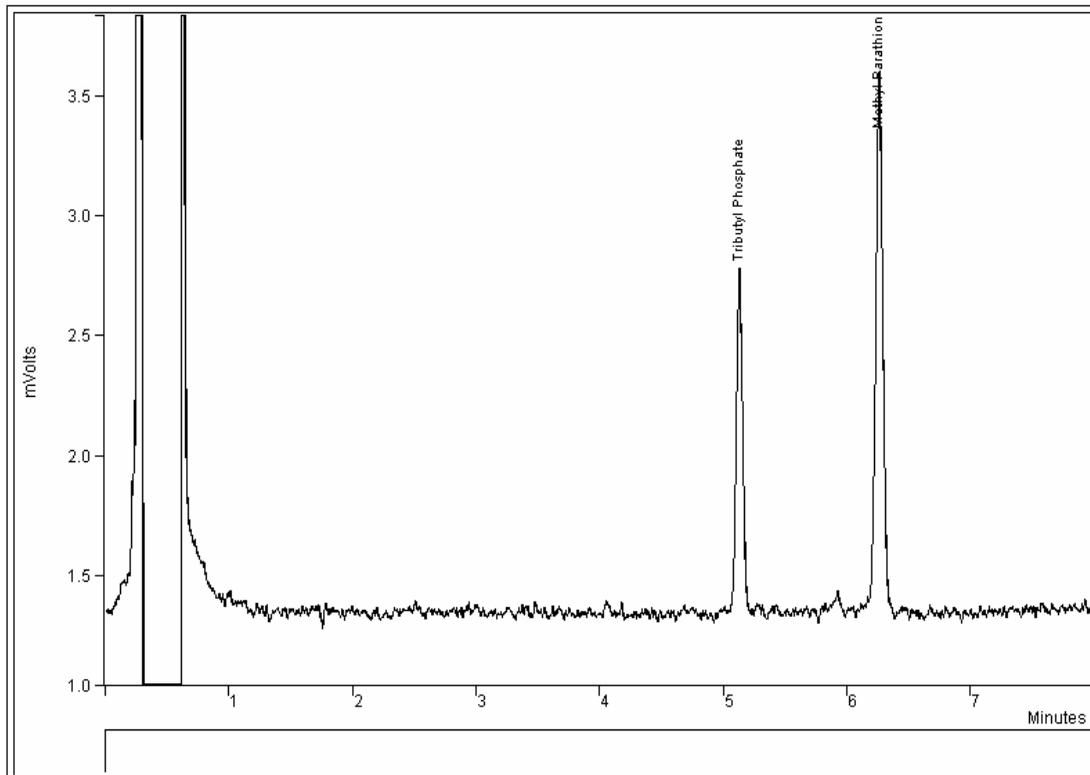
Column: Non-polar, e.g., 1 μ film thickness, 15m x 0.25 mm, CP-Sil 5 CB

Carrier: Nitrogen, 5 mL/minute

Injection: 1079, 200 °C, 1 μ L splitless

Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute

Detector: 300 °C, Range 1, Capillary current setting



PFPD P Mode

Column: Non-polar, e.g., 1 μ film thickness, 15m x 0.25 mm, CP-Sil 5 CB

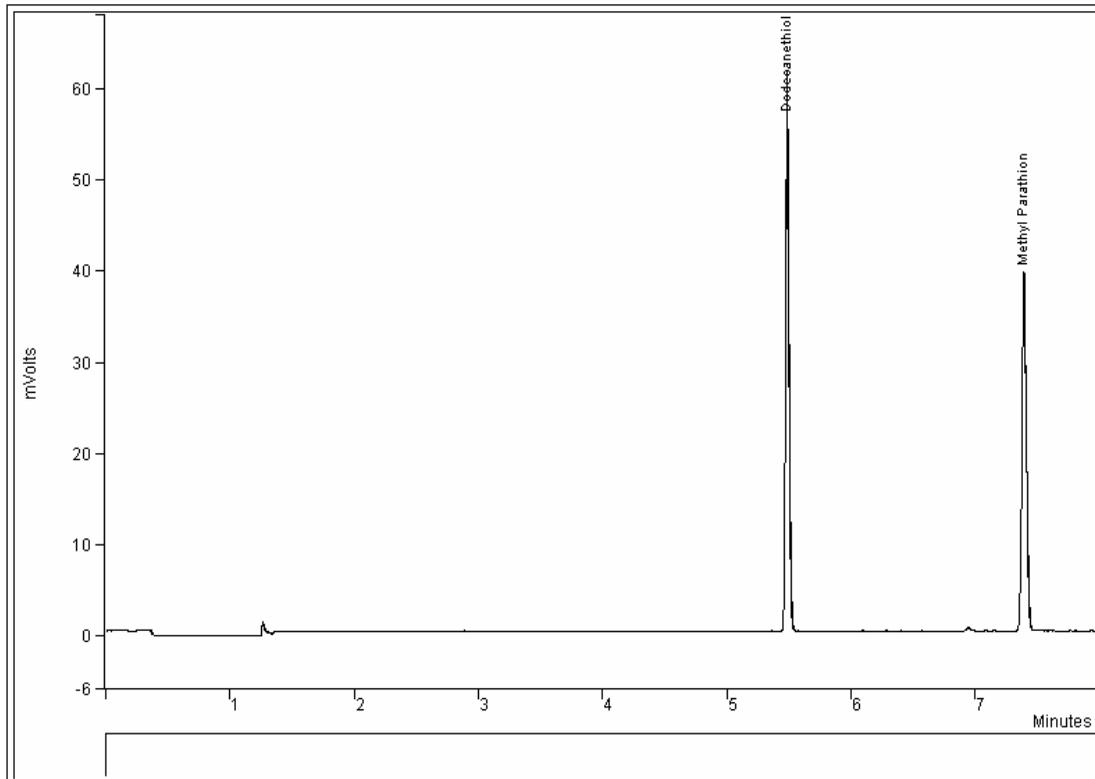
Carrier: Nitrogen, 2 mL/minute

Injection: 1079, 220 °C, 1 μ L splitless of a 1:30 dilution of FPD test mix in iso-octane

Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute

Detector: 300 °C, Range 10, Air1 17 mL/min., Hydrogen 14 mL/min., Air2 10 mL/min.

Gate Delay 4 msec., Gate Width 10 msec, Trigger level 200 mV



PFPD S Mode

Column: Non-polar, e.g., 1 μ film thickness, 15m x 0.25 mm, CP-Sil 5 CB

Carrier: Nitrogen, 2 mL/minute

Injection: 1079, 220 °C, 1 μ L splitless of a 1:30 dilution of FPD test mix in iso-octane

Temperature Program: 80°, hold 1 minute, 20°/minute to 200°, hold 1 minute

Detector: 300 °C, Range 10, Air1 17 mL/min., Hydrogen 14 mL/min., Air2 10 mL/min.

Gate Delay 6 msec., Gate Width 20 msec, Trigger level 200 mV

Communications

Introduction

The 3800 Gas Chromatograph uses Ethernet® as the method of communications for instrument control and for sending digitized data to an external data system. In addition, the 3800 is equipped with analog output ports which allow analog data to be sent to external integrators or other analog data systems.

The 3800 is equipped to handle digital and analog data simultaneously. The Ethernet communications option allows Star Workstation control of the 3800 and transmits up to three channels of detector data to the Workstation. In addition, the standard 3800 is equipped with an analog connection which can be configured with up to three analog outputs and status signals. Various cable options are available for sending analog data to integrators and data systems.

Ethernet Communications

Ethernet refers to the type of hardware connection used to transfer digital data between the 3800 GC and the data system to which it is attached. The optional 3800 GC Communications Board has a 10Base2 Ethernet connector located on the rear of the GC. This BNC style connector accepts RG58C/U cable connections. The standard 3800 Communications kit contains a six foot cable and a BNC Tee. The BNC Tee is connected to the GC BNC connector. The Ethernet cable connects the BNC Tee on the 3800 GC to the Star Chromatography Workstation Ethernet board or to a cable or hub on a company network. If only one GC is connected to the Star Workstation or is the last in a series of GCs connected, then a terminator is used on the open end of the Tee. Otherwise, another cable can be connected to the BNC Tee to attach to another GC.

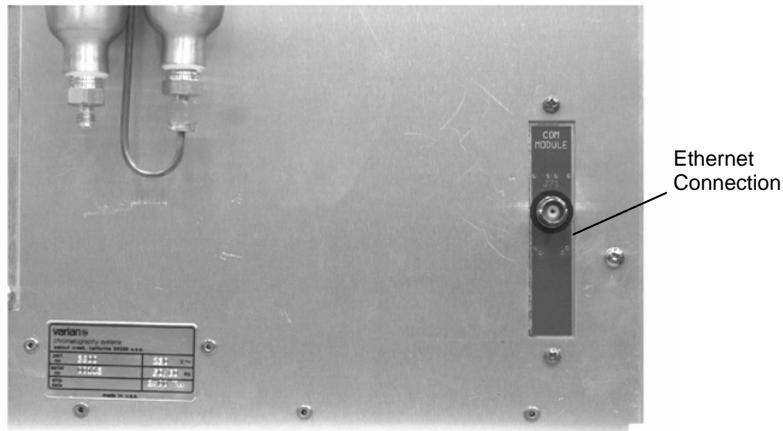


Figure 23 3800 GC Ethernet Connection

The following is the start-up sequence for a 3800 that has the Communications board installed:

1. If there is no Communications Board installed, the GC will boot up normally into local control mode.
2. If a Communications Board is installed, the GC will send out its Ethernet address and wait for an IP address and host name to be delivered by the Star Workstation or a central BOOTP server. The user can bypass this process and boot up in local mode by pressing any key on the 3800 keyboard. However, if this is done, the 3800 will have to be powered off and on again to re-establish communications. Connection with the Star Workstation may take as long as one minute. If after one minute the 3800 has not connected to the workstation, power the GC off and on to repeat the sequence.

10Base2 Ethernet Cable Components

In addition to the 10Base2 Ethernet cable, the components shown below are used to build a 10Base2 Ethernet segment. For a 10Base2 Ethernet segment, two BNC (Bayonet Nut Connector) terminators are used – one at each end of the cable segment. The side branch of the BNC Tee connects directly to the Communications Board in the 3800 GC or the Star Workstation Ethernet Board in the computer.

Note: Do not try to connect an Ethernet cable between the BNC Tee and the 3800 GC Communications Board or the Star Workstation Ethernet Board.

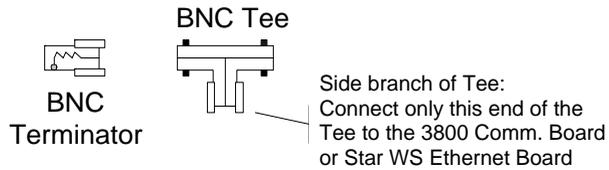


Figure 24 10Base2 Ethernet Components

Connecting a 3800 GC to a Star WS Computer

In the simplest case, one 3800 GC will be connected to one Star Workstation computer. Figure 25 shows schematically how this cabling would look when viewed from above.

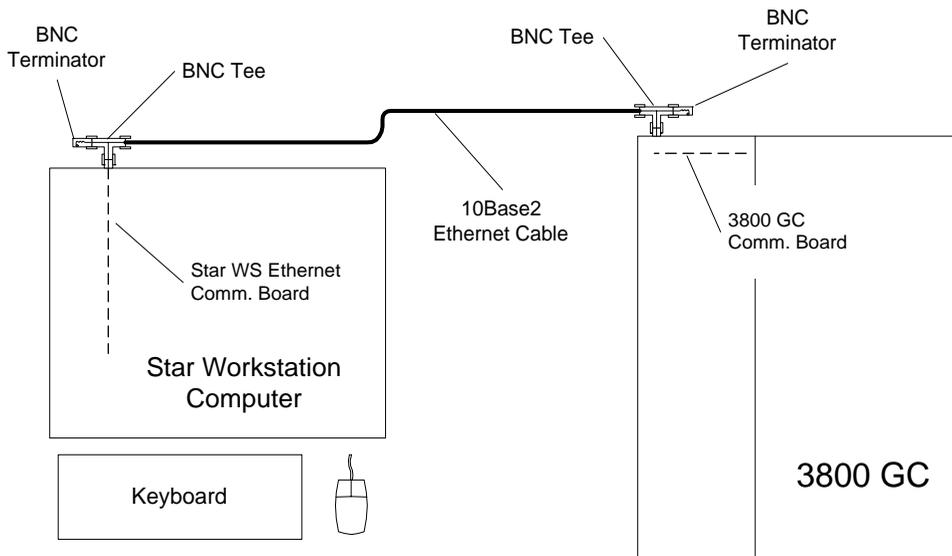


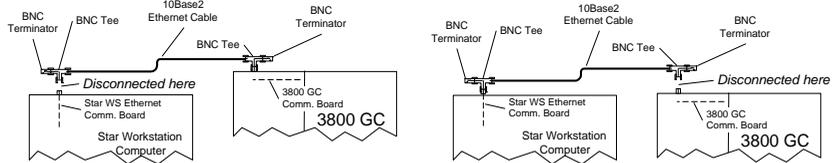
Figure 25 3800 GC to Star Workstation Connection

Connecting Additional 3800 GCs and Star WS Computers

Additional 3800 GCs or Star Chromatography Workstation Computers can easily be added to a 10Base2 Ethernet segment. The rules to follow are:

1. Whenever any portion of the cable that is connected between the two BNC terminators is to be disconnected, all devices on that Ethernet segment must be turned off or disconnected from the Ethernet segment. You may disconnect and reconnect devices from the BNC Tees without disrupting the communication between other devices on the segment, but you **CANNOT** disconnect an Ethernet cable or BNC terminator from a BNC Tee without disrupting the communication among all devices on the segment.

Acceptable Disconnections



Unacceptable Disconnections

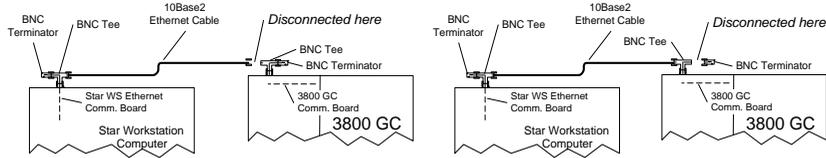


Figure 26 Acceptable and Unacceptable Disconnection Examples

2. The side branch of the BNC Tee connector must either connect directly to the Ethernet Communications Board in the device or be left disconnected. Leaving disconnected BNC Tees in the 10Base2 Ethernet segment allows additional devices to be added to the segment at a later date.
3. The Ethernet cables and BNC Tees must form an unbroken chain linking the two BNC terminators together. There cannot be more than two BNC terminators in a 10Base2 Ethernet segment.
4. The total cable length of a 10Base2 Ethernet segment should not exceed 200 meters.

Notice how the Ethernet cables (shown by the heavy black line) along with the BNC Tees provide a single unbroken path from one BNC terminator to the other.

More detailed information on setting up Star Workstation communications is provided in the Star 3800 GC Control Software Installation and Operation manual. Ethernet communication troubleshooting information is provided in the troubleshooting section of the same manual.

Analog Communications

The 3800 is equipped with three analog output ports. These are located on connector J6 on the top of the GC, accessed by removing the top left cover. This connector contains all three analog channels plus status signals for “Ready In” to the GC and “Start Out” from the GC. The individual analog ports can be assigned to any installed detector on the 3800. The ports are identified as analog port A, analog port B and analog port C with the default assignment being front detector, middle detector and rear detector respectively.

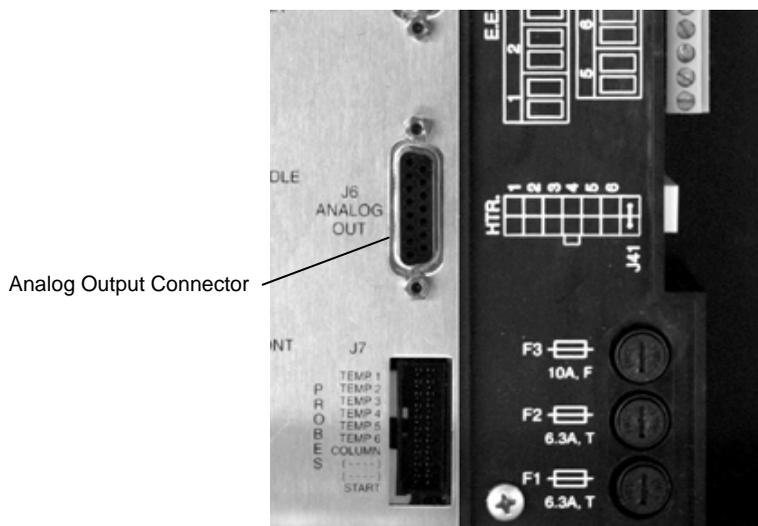


Figure 28 Analog Output Connector

The analog output ports are programmed in the 3800 method by pressing the OUTPUT key on the keyboard. This brings up a menu screen from which you choose one of the three ports to program; A, B or C. The following screen appears when Analog Port A is chosen.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Detectors: Front: TCD Middle: FID Rear: None				
ANALOG OUTPUT A				
Time	Signal Source	Attenuation		
Initial	Front Detector	1		
10.00	Middle Detector	1		
				

The screen shown is used to time program the signal source of the analog output. By default analog output A is assigned to the front detector. In the example above, the 3800 is configured with a TCD in the front detector position and an FID in the middle detector position. The analog port is programmed to transmit the TCD signal for the first 10 minutes of the run and then switch to the FID for the remainder of the run. This mode of operation is known as detector switching.

Note that the attenuation should normally be set to 1. Most of the devices to which the analog output will be connected, such as integrators and analog data systems, are equipped with attenuation control. The signal sent from the GC in this case should not be attenuated.

The 3800 has a number of analog cable options, depending on the device to which the cable is connected. All cables have a 15 pin D-shell connector on one end to attach to J6 on the 3800 and have the appropriate connectors on the other end of the cable for the devices to which they are being attached. The following cables are available:

- Two channel cable for Varian ADC Board: 03-925672-01. This cable is equipped with two 9 pin D-shell connectors for the ADC Board. The Varian ADC Board can collect a maximum of two channels of analog data. Analog Output Port A is connected to the Varian ADC Board Channel A. Similarly, Analog Output Port B is connected to the Varian ADC Board Channel B. Analog Output Port C is not connected.

- Two channel cable for 4400 Integrator: 03-925303-01. This cable is equipped with a status connector (ready in/start out) and spade lug terminations for two analog channels. The 4400 Integrator can collect a maximum of two channels of analog data. Analog Output Port A is connected to the 4400 Integrator Channel A. Similarly, Analog Output Port B is connected to the 4400 Integrator Channel B. Analog Output Port C is not connected.
- Three channel analog cable with status connector: 03-925675-01. This cable is equipped with three sets of shielded analog signal pairs, terminated with spade lugs and two sets of sync signals, a "ready in" to the 3800 GC and a "start out" from the 3800 GC.

Local Automation

Introduction

This section of the manual deals with local automation from the 3800 keyboard / display. For information regarding automation control from the Star Workstation please refer to the Star Workstation documentation. Local automation control from the 3800 keyboard is completely independent from Workstation automation control and was designed for those users who do not use the Star Workstation.

Note: If Local 3800 Automation is selected on a 3800 that has a Star Workstation connected, the Star Workstation will be disconnected from the 3800. In this mode the 3800 and autosampler, if installed, cannot be controlled from the Star Workstation and data cannot be transmitted over the Ethernet. To restore Star Workstation communication the Manual Injection mode must be selected in Automation Select / Edit and then Remote Control must be enabled in the Instrument Menu section of the 3800 keyboard.

3800 local automation consists of two distinct types of automation control. One type refers to automation using a device that can be controlled from the 3800 through a communications interface called a Sample Introduction Device. This type of automation is known as *addressable automation*. The most common example of addressable automation is control of the Varian 8400 GC AutoSampler.

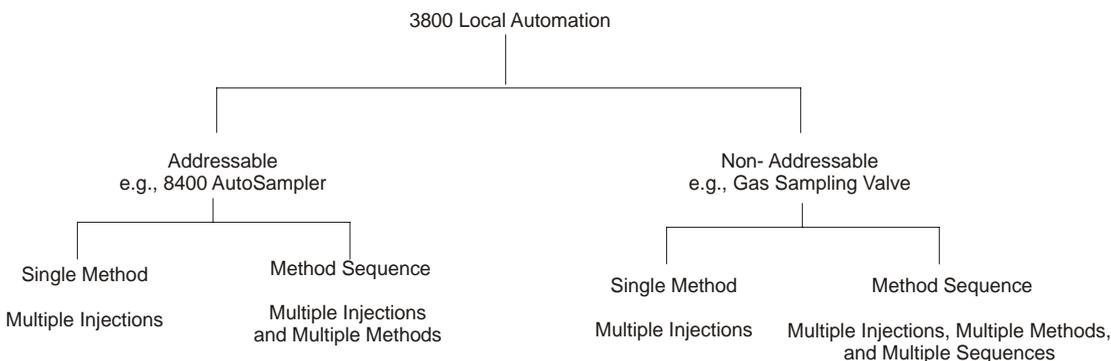


Figure 29 3800 Local Automation

The second type of automation control simply involves running a series or sequence of methods without control of an addressable device such as an AutoSampler. This is referred to as non-addressable automation. An example of *non-addressable automation* would be to run a series of analyses where sampling is accomplished using a gas sampling valve controlled from one of the 3800 valve drivers. Non-addressable automation will be covered first as this is the simplest type of automation.

Non-Addressable Automation

Non-addressable automation refers to running more than one unattended analysis on the 3800. The simplest example of non-addressable automation is where the same 3800 method is run a number of times. This is called Single Method Automation. A typical application of Single Method Non-Addressable Automation is a series of analyses using a gas sampling valve. In this instance a sample stream is purged through a gas sampling valve installed on the 3800. The gas sampling valve is controlled using one of the seven available valve drivers on the 3800. The two gas sampling valve states are identified as *Fill* and *Inject*.

Fill refers to the state when the sample is purging through the sample loop of the gas sampling valve. *Inject* refers to the valve switch which loads the sample into the analytical system and starts the run.

By building a Single Method automation method the user can program the 3800 to carry out up to 999 analyses. In addition, the user can select Method Sequence automation where methods can be switched during automation. For example, the user could run an analysis using method 1 followed by an analysis using method 3.

Addressable Automation

Addressable automation refers to a series of analyses where an ancillary device, such as an AutoSampler, is controlled from the 3800. The 8400 GC AutoSampler is the most common device used with addressable automation. In this instance the Single Method or Method Sequence automation is run in conjunction with control of an 8400 AutoSampler. Each analysis consists of a sample injection using the specific conditions programmed in the 8400 section of the 3800 method.

Automation Control

Local 3800 automation is accessed by pressing the Select / Edit key in the Automation section of the 3800 keyboard. This presents the following menu screen where an automation method can be selected or edited.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
AUTOMATION SELECT				
[1] Select Automation Mode				
[2] Edit Single Method Automation				
[3] Edit Method Sequence Automation				
[4] Select Priority Sample				
				

Select Automation Mode allows the user to choose from Manual Injection, Single Method Automation, or Method Sequence Automation. Manual Injection is the default when the 3800 is first powered on.

In the Manual Injection mode local automation is inactive. The user can program the 3800 from the keyboard or download methods, including automation, from the Star Workstation. When a local automation method is selected, either Single Method or Method Sequence, the 3800 is disconnected from the Star Workstation. Do not select one of the local automation modes unless you actually want to run automation from the 3800. If you are running local automation and later want to add Star Workstation communication you must select the Manual Injection mode and enabling remote control found by pressing the Instrument Menu key and selecting enable.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
<p>AUTOMATION MODE SELECT</p> <p>Automation Mode: <u>Manual Injection</u></p> <p>Note: Selecting Single Method or Method Sequence Automation will disable remote control.</p>				
○	○	○		

The automation mode is selected using the increment or decrement key on the 3800 keyboard. Press the Enter key to activate the required automation mode. If Single Method or Method Sequence automation is chosen, the corresponding LED will light in the automation section of the 3800 keyboard.

An automation method may be suspended by pressing the *Suspend* key in the automation section of the 3800 keyboard. To resume the automation method press the *Resume* key.

If the 3800 Stop key is pressed while automation is in progress the automation method will be terminated.

Automation Methods

Local 3800 automation methods differ depending on whether or not an addressable device is detected. Addressable devices are connected to one of the serial interface ports on the top of the 3800. These ports are labeled as SID1 and SID2 (Sample Introduction Device 1 and 2). When the 3800 is powered up it monitors these ports to determine if an addressable device is present. If a device such as an 8400 AutoSampler is detected, the 3800 defaults to the addressable automation mode. Note that the default port for an 8400 AutoSampler is SID1. If there are no devices connected to SID1 or SID2, the 3800 defaults to the non-addressable automation mode. The primary difference between addressable and non-addressable automation is that the addressable methods include a sample range. For example, the 8400 AutoSampler contains a sample range of 0 to 99 sample vials.

Single Method, Non-Addressable Automation

If an addressable device such as an 8400 AutoSampler is not present, the 3800 defaults to the non-addressable mode on power up. In Single Method Automation the same 3800 method can be run up to 999 times.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
SINGLE METHOD AUTOMATION				
Method: <u>Method 1</u> Injections: <u>1</u>				
○	○	○		

Select from one of the available eight 3800 methods using the increment or decrement keys. *Injections* refers to how many times this method should be run. The allowable entries are 1 to 999.

To start a Single Method Automation, press the Select / Edit key in the Automation section of the 3800 keyboard, then select Single Method as the automation choice. Press the 3800 Start key to start automation. Note that if the method specified in Single Method Automation is different from the currently active 3800 method, this may result in a delay of several minutes before the first analytical run starts.

Method Sequence, Non-Addressable Automation

Method Sequence Automation allows running an automation sequence where the method can be changed from run to run. The sequence of methods is built by adding lines to the automation table. In addition, a method sequence automation table may be run multiple times. The method sequence automation table can consist of up to 25 lines. Lines are added by pressing the New Line key on the 3800 keyboard. Page 2 of the Method Sequence allows repetition of the sequence up to 999 times.

Method Sequence automation, page 1 of 2

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
METHOD SEQUENCE AUTOMATION, Page 1 of 2				
Method		Injections		
Method 1		5		
Method 2		5		
<input type="radio"/> <input type="radio"/> <input type="radio"/>				

Injections refers to the number of times the method specified in that line of the table should be run. The allowable entries are 1 - 999.

Method Sequence automation, page 2 of 2.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
METHOD SEQUENCE AUTOMATION, Page 2 of 2				
Number of Sequences: <u> 1 </u>				
<input type="radio"/> <input type="radio"/> <input type="radio"/>				

Number of Sequences refers to the number of times the sequence list is to run.

Single Method, Addressable Automation

If an addressable device such as an 8400 AutoSampler is present, the 3800 defaults to the addressable mode on power up. If an 8400 is detected, the following automation screens will appear. Note that the 8400 must be connected to port SID1 on the 3800. In addressable automation methods with the 8400 AutoSampler, the range of sample vials must be specified in addition to the number of injections / sample. The sample range for an 8400 AutoSampler is 0 to 99, and the range of injections per sample is 1 to 99.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Page 1 of 2				
SINGLE METHOD AUTOMATION				
Method:		Method 1		
Initial Sample:		0		
Final Sample:		99		
Injections / Sample:		1		
Injection Volume (ul):		1.0		
				

Select from one of the available eight 3800 methods using the increment or decrement keys. Initial sample is the first 8400 vial to be sampled and final sample is the final 8400 vial to be sampled. The automation method will sample all vials that are present in the 8400 carousel in this sample range. As an example, if the initial sample was specified as 2 and the final sample specified as 10, but sample vials were only present in positions 2, 5 and 10, three runs would be made assuming the injections / sample parameter was set to 1. The 8400 would sample from vial 2, then from vial 5, and finally from vial 10. Note that the final sample must be greater than the initial sample. Injections / sample refers to how many times the 8400 should sample from each vial. The allowable entries are 1 to 99. Page two allows using Prep-Ahead. Prep-Ahead allows the autosampler to begin preparing the next sample for injection while the current run is in progress. You will have to experiment with the Prep-Ahead count down timer if you wish to have the next sample ready to inject exactly when the GC becomes ready after the current run ends.

To start a Single Method Automation, press the Select / Edit key in the Automation section of the 3800 keyboard, then select Single Method as the automation choice. Press the 3800 Start key to start automation. Note that if the method specified in Single Method Automation is different from the currently active 3800 method, the method specified in automation will be activated upon pressing the Start key. Depending on the GC parameters of the respective methods, this may result in a delay of several minutes before the first analytical run starts.

Method Sequence, Addressable Automation

Method Sequence Automation allows running an automation sequence where the method can be changed from run to run. The sequence of methods is built by adding lines to the automation table. Unlike non-addressable method sequences, the automation table may not be run multiple times. The method sequence automation table can consist of up to 25 lines. Lines are added by pressing the New Line key on the 3800 keyboard. Note that for any given line, the final sample must be greater than the initial sample. Prep-Ahead allows the autosampler to begin preparing the next sample for injection while the current run is in progress. You will have to experiment with the Prep-Ahead count down timer if you wish to have the next sample ready to inject exactly when the GC becomes ready after the current run ends.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Page 1 of 2				
METHOD SEQUENCE AUTOMATION				
Method	Initial	Final	Inj / Vial	Inj Vol
Method 1	0	12	1	1.0
Method 2	13	24	1	1.0
<input type="radio"/> <input type="radio"/> <input type="radio"/>				

Priority Sample

Priority Sample is only available while local Automation is running. You may designate which vial location contains the priority and which Method (1-8) you wish to use with it. Pressing the Run Now soft key will cause the selected vial position to be sampled using the specified method **after** the

Automation Status

The 3800 has a status screen which is dedicated to automation status. This screen is accessed by pressing the status key on the 3800 keyboard and using the Page Down key to access Local Automation Status. The information displayed on the screen varies depending on whether addressable or non-addressable automation is active.

Addressable Automation status screen (8400 Automation)

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Automation Status				
Automation State:		Ready		
Injection Number:		1		
Table Line Number:		1		
Sample Vial:		1		
Plunger Stroke Count		126		
Prep-Ahead Delay (min)		0.00		
○	○	○		

The Automation Status field indicates whether the automation status is ready, running or suspended. Injection number refers to the specific 8400 vial that is being sampled. Table line number indicates the line number if a method sequence is being run and sample vial refers to the specific 8400 vial being sampled (0 - 99). Note that table line number is blank if Single Method automation is active.

Running Priority Sample

The priority sample is a powerful tool for temporarily interrupting the execution of automation in order to run an urgent sample. While Local Automation is running, you may select the Priority Sample Option. You will be presented with options for which AutoSampler Vial Position and Method you wish to use. After entering the pertinent information, pressing the Run Now softkey will cause the priority sample to be run after the current injection is complete. Regular automation will continue from where it was interrupted after the Priority Sample is complete. If you select the Priority Sample on the last run of either Single Method or Method Sequence automation, the system will return to the beginning of Automation and continue. Press the Stop key to abort running the same Automation a second time.

Non-addressable Automation status screen

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1	Method 1		0.00	20.00
Automation Status				
Automation State: Ready				
Injection Number: 1				
Table Line Number: 1				
Sequence Number: 1				
○	○		○	

The Automation Status field indicates whether the automation status is ready, running or suspended. Injection number refers to the number of injections of the specific method. Table line number indicates the line number if a method sequence is being run and sequence number refers to number of the sequence (page 2 of the Method Sequence method). Note that table line number and sequence number are blank if Single Method automation is active.