

# CP-3800 GC Getting Started Manual



Varian Analytical Instruments 2700 Mitchell Drive Walnut Creek, CA 94598-1675/usa

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# Read Before Operating Important Safeguards

The following items are frequently not recognized or are overlooked by new users while learning to operate a Gas Chromatograph. They are brought to your attention to safeguard against damage to your equipment.

- Carrier gas must be flowing through the column before heating the column oven. Carefully evaluate columns that have been heated with no carrier gas flowing through and discard, if damaged. Ensure the injector contains a septum and there is a positive flow of carrier gas before heating the column.
- Become familiar with the location and identity of injector and detector options prior to making settings at the GC keyboard and pneumatics panel. For example, determine the physical location of the injector you want to use (front, middle, or rear) and identify the specific pneumatic controls associated with it (manual pneumatics or Electronic Flow Control). Similarly, determine the physical location of the detector you want to use and identify the specific pneumatic controls for this detector.
- Heating an ECD to elevated temperatures without carrier or make-up gas flowing through it can damage the ECD cell. This can happen inadvertently especially when the ECD is not in use. For this reason, it is recommended that a low flow of carrier or make-up gas be maintained through an ECD at all times.
- If your system has a TCD, gas must be flowing through both channels to protect the filaments. Turn power to filaments off when not in use.

# **Important Tips Regarding 3800 Operation**

- After editing the active method, it must be re-activated before running the next analysis. Always remember to activate a method after you have made edits, if you intend to run that method next.
- Note that the GC control keys allow access to both the active method status (top of the display) and the current method available to edit (middle of the display). If you want to edit the active method then you must make it the current edit method. Both the current active method and edit method are listed on the top line of the 3800 display.
- If a potentially hazardous fault is detected, such as a thermal runaway, the 3800 shuts down the affected component and reports the fault. After correcting the fault, normal operation is restored by powering the 3800 OFF then ON.

# **Table of Contents**

INTRODUCTION	5
Guide to the 3800 User Manuals	5
Finding Information in these Manuals	5
Abbreviations	5
Brief Description of the 3800 Gas Chromatograph	6
Configuration and Options	7
	•
Site Deguirements for CC Installation	9
Site Requirements for GC installation	9
Power Requirements	9
Space Requirements	10
Environmental Requirements	10
Peripheral Connectors	11
GC Installation	12
Inspection	12
Unpacking	12
Remove Protective Devices	12
Connect Data Handling Peripherals	12
Select Gases	13
Install Gas Regulators and Set Pressures	13
Connect Gases	13
Connect GC to Power	14
Turn Power On	14
Factory Default States and Settings	14
Quick Guide to the 3800 Keyboard and Display	15
Getting Around the Keyboard	15
Column Installation and Conditioning	29
Connect Column to Injector	29
Nuts and Ferrules for Capillary Columns	29
Connect Capillary Column to Injector	31
Nuts and Ferrules for Packed Columns	32
Connect a Packed Column to the Injector	32
Varian Test Column	33
Identify Carrier Gas Controls and Turn on Flow	33
1079 Split Vent Flow and Septum Purge Flow	34
Set Carrier Gas Flow Rate to Condition Column	35
Note for Capillary Column Users	35
Conditioning the Injector and Column	35
Connect Capillary Column to Detector	36
Column Insertion Depths for Varian Detectors	37
Connect Metal Packed Column to Detector	38
Check for Leaks	38

BASIC OPERATION	39
Setting Carrier and Detector Gas Flow Rates	39
Condition the GC System	41
Build a Method to Analyze a Test Sample	41
Method to Analyze a Varian Test Sample on a Varian Test Column	42
Prepare the Test Sample	43
Solvents for GC	43
Sample Volume and Injection Rate	44
Inject the Test Sample	45
MAINTENANCE	47
Overview	47
General GC Maintenance	48
Check and Renew Gas Supplies	
Leak Check	
Gas Purifier Replacement	50
Injector Maintenance	52
1079 Injector	53
Remove the Glass Insert	55
Replace the Glass Insert	56
Clean the Glass Insert	57
Deactivate the Glass Insert	
1041 Injector	60
Hardware Replacement and Cleaning Procedures	60
Septum Replacement	
Needle/Syringe Cleaning	
Leak Checking	
1061 Injector	
Replacing the 1061 Glass Insert	
Remove the Glass Insert	
Clean the Clean Insert	
Clean the Glass Insen	00
Clean the Flame Tin and Internal Parts	00 89
Replace the Ferrule	00 03
Thermionic Specific Detector	03 70
Cleaning/Maintenance	70
Bead Cleaning	70
Flame Tip and Internal Parts Cleaning	70
Ferrule Replacement	
Electron Capture Detector	
Thermal Cleaning With Carrier Gas	
Thermal Conductivity Detector	
Pulsed Flame Photometric Detector	73
Micro Thermal Conductivity Detector	73

PARTS AND SUPPLIES	75
To Order Parts and Supplies	75
Injectors	75
1079 Injector	75
1041/1061 Injector	76
Detectors	76
Flame Ionization Detector	76
Thermionic Specific Detector	76
Electron Capture Detector	76
Thermal Conductivity Detector	77
Pulsed Flame Photometric Detector	77
Micro-TCD	77

Figure 1 Connectors for Peripheral Equipment	11
Figure 2 3800 GC Keyboard	15
Figure 3 1079 Injector - Cross-Sectional View	54
Figure 4 1041 Injector Assembly	61
Figure 5 1061 Injector Assembly	64
Figure 6 Flame Ionization Detector Assembly	69
Figure 7 Thermionic Specific Detector Assembly	71
5 i j	

Table 1 Physical Dimensions of the 3800 GC System Components10Table 2 Flow Rates for Carrier and Detector Gases for the 3800 Gas Chromatograph39

# Introduction

# Guide to the 3800 User Manuals

The purpose of these User Manuals is to provide instructions for the safe installation and operation of the Varian 3800 Gas Chromatograph.

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**Abbreviations** The following abbreviations appear in this Manual:

ac	alternating current
dc	direct current
ECD	Electron Capture Detector
EFC	Electronic Flow Control
FID	Flame Ionization Detector
GC	Gas Chromatograph
GC/MS	Gas Chromatograph/Mass Spectrometer
ID	Inner Diameter
OD	Outer Diameter
P/N	Part Number
PFPD	Pulsed Flame Photometric Detector
TCD	Thermal Conductivity Detector
TSD	Thermionic Specific Detector
Micro-TCD	Micro Thermal Conductivity Detector

## Brief Description of the 3800 Gas Chromatograph

The following are some of the key features of the 3800:

- Three injectors, columns and detectors can be installed and operated simultaneously.
- The 1079 Universal Capillary Injector provides five modes of injection isothermal split and splitless, temperature ramped splitless, on-column and large volume.
- Electronic or manual control of carrier gas.
- Ethernet® communications with full networking capability.
- A full suite of universal and selective detectors FID, TCD, ECD, TSD (N, P), PFPD (S, P, N, C, Metals), Micro-TCD and Ion Trap MS.
- Large, swing-out pneumatics for easy access.
- Seven heated zones and seven external events for maximum flexibility and custom configurations.
- Up to eight separately programmed and stored analytical methods.
- Intuitive, function-based keyboard with large, dot matrix display for ease of use in method building and viewing instrument status.
- Built-in Custom Solutions capability valved systems, custom plumbing, six-position valve oven, methanizer.
- Large column oven for easy access and installation of up to three analytical columns.
- A complete range of GC automation and sample preparation techniques the 8200 AutoSampler with AutoDrive accessory, dual 8200 AutoSamplers, Solid Phase MicroExtraction, Purge and Trap, Heated Headspace, and the Archon Purge and Trap AutoSampler.

# **Configuration and Options**

The 3800 has seven heated zones, one of which is always configured as the column oven. The following options may be configured in the other six available heated zones:

- 1079 Universal Capillary Injector
- 1041 On-Column Injector for large bore (0.53 mm ID) or packed columns
- 1061 Flash Vaporization Injector for large bore or packed columns
- Single, dual, or multi-position Valve Oven
- Flame Ionization Detector
- Thermal Conductivity Detector
- Electron Capture Detector
- Thermionic Specific Detector
- Pulsed Flame Photometric Detector
- Methanizer
- Micro Thermal Conductivity Detector

Any combination of three injectors may be installed on the 3800. If a valve oven is installed, it normally replaces two injector positions.

Any combination of three detectors can be installed with the following exceptions: Only two TCD or PFPD detectors can be installed. If two standard TCDs are installed, then no other detectors can be accommodated. However, a dual TCD (two cells in one housing) is available as a Custom Solution which allows installation of another detector, such as an FID, behind it.

The methanizer typically occupies the same location as one of the detectors. Dual 8200 AutoSamplers restrict the 3800 configuration to two injectors and two detectors.

Installed options on the 3800 are generally identified by their location. The options mounted on the top of the instrument, such as those listed above, are designated as Front, Middle and Rear to coincide with their relative location or the location of their respective electronic control modules.

# Installation

## Site Requirements for GC Installation

PowerPower SourceRequirementsEach 3800 GC requires a separate circuit with the following<br/>characteristics:

101 Vac  $\pm$  10%, 50 or 60 Hz  $\pm$  2%, 25 Amps, 2.5 Kilowatts

120 Vac  $\pm$  10%, 60 Hz  $\pm$  2%, 20 Amps, 2.4 Kilowatts

230 Vac  $\pm$  15%, 50 Hz  $\pm$  2%, 10 Amps, 2.3 Kilowatts

Installation Category: II

#### Ground

It is essential to have a safety ground connection to this instrument.

#### Power Cord

In Europe the instrument will be supplied with an IEC 320 style socket. The power cord assembly can be changed to suit your local power and socket requirements. In the United States and Japan the instrument is equipped with a fixed power cord and plug.

#### Heat Dissipation

Calculate heat dissipation using the following formula:

Watts x 3.4129 = BTU/hr.

Actual dissipation will depend on method parameters and instrument configuration.



Heat from the column oven is vented through the rear of the GC. Make certain that the hot column oven exhaust is not directed onto other electronic instruments, cables or gas lines.

#### Fuses

There are four fuses accessible on the top of the 3800:

F1: 6.3A T, F2: 6.3A T, F3: 10.0A F, F4: 6.3A T

**Space** Allow sufficient bench space to permit installation of workstations, integrators, autosamplers and other peripheral GC equipment. The table below lists the physical dimensions and weight of the GC and the peripheral instruments which may be installed near it. Allow six inches of space at the sides and rear of the GC to permit free air circulation.

> The power cord, gas inlets and the optional Ethernet® communications port are located on the rear panel of the instrument. The power switch is to the rear of the top cover. Signal output connectors for peripheral instrumentation such as autosamplers and integrators are located under the left top cover of the instrument.

	Hei	ght	Wi	dth	De	pth	We	ight
Instrument	in.	cm	in.	cm	in.	cm	lb.	kg
3800 GC	20	51	26	66	22	56	95	43
8200 AutoSampler	20	51	6	16	16	41	24	11
Tekmar 3000 Purge and Trap	19	48	9	23	18	46	37	16.8
Archon Purge and Trap AutoSampler	17	43	21.5	55	21	53	80	36
Tekmar ALS-2016	27	69	15	38	15	38	35	13.1
Genesis Headspace AutoSampler	22	56	28	72	18	46	110	50
Star Chromatography Workstation (computer with monitor, approximate values)	17	43	17	43	21	53	35	16

#### Table 1 Physical Dimensions of the 3800 GC System Components

#### Environmental Requirements

Pollution degree: 2

*Humidity:* 5% to 95% RH

*Temperature:* 10° to 40°C operating, -20° to 65°C non-operating

The 3800 Gas Chromatograph is intended for indoor use and certified for operation up to an altitude of 2000m, however, the 3800 is designed for use at higher altitudes. Varian GCs should be protected from corrosive chemicals or gases, dust/ particulate accumulation, and direct venting of air conditioners, heaters, furnaces or fans.

Peripheral	The 3800 Gas Chromatograph is equipped with various connectors on the
Connectors	rear panel and top of the instrument intended for use with peripheral
	equipment such as autosamplers and data handling devices. The following are the standard connectors on the 3800 and their intended uses:

*Rear Panel* Ethernet® Connector: BNC connector for 10 base 2 cable. *Printer Connector:* 25 pin "D" connector, intended for future use *AC Power Input:* 230V units only.

#### Top of Instrument (under detector cover)



J45 and J46

Aux Output, intended for future use.

#### Figure 1 Connectors for Peripheral Equipment

## **GC** Installation

- Inspection The 3800 Gas Chromatograph will arrive packed in one large palletized box and one or more smaller cartons. Inspect the cartons carefully for damage or signs of rough handling. Report damage to the carrier and to your local Varian office.
- **Unpacking** Unpack the GC and accessories carefully and transfer to the work area, using proper handling techniques. The GC should be lifted from the bottom. Inspect the GC and accessories and immediately report any damage to the carrier and your local Varian office.



Avoid back strain or injury by following all safety precautions when lifting heavy objects.

Remove Protective	The instrument has been protected during shipment by various caps, plugs and restraints. Prior to operating, remove:			
Devices	<ul> <li>Shipping dowel for oven fan motor from hole in GC rear panel</li> </ul>			
	Detector tower plugs under detector cover			
	Gas bulkhead fitting plugs on rear panel			
	Any plastic plugs inside the column oven			
Connect Data Handling Peripherals	Workstations, integrators and other data handling devices are connected to the cable connectors on the back panel or the top of the instrument.			
	• If the Varian Star Chromatography Workstation with 3800 control is being installed follow the installation instructions in the communications section of the 3800 Operator's manual. This installation simply involves making a connection to the Ethernet® connector on the rear panel of the GC. Additional information on 3800 Workstation control is provided in the Star Workstation documentation.			
	<ul> <li>If an analog output signal is being used, follow the specific installation instructions provided with the analog interface kit.</li> </ul>			

**Select Gases** The following gases are commonly used in Gas Chromatography for the following functions. Note that the recommended purity for carrier make-up and detector fuel gases is 99.999%. In addition, the use of gas filters to remove hydrocarbons, oxygen, and water from carrier gases is recommended.

Gas	Function
He, N <sub>2</sub> , H <sub>2</sub> , Ar, Ar/CH <sub>4</sub>	Carrier gas
H <sub>2</sub> , Air	Detector fuel gases
He, N <sub>2</sub> , Ar/CH <sub>4</sub>	Make-up gas for capillary column use
Air	AutoSampler operation
LN <sub>2</sub> , LCO <sub>2</sub>	Cryogenics

Install Gas	Carrier gas, air and H <sub>2</sub> supplied from cylinders or manifolds should have a
Regulators	two-stage regulator having a zero to 100 psi low-pressure stage. Set
and Set	cylinder regulator pressure to match the GC gas inlet pressures, listed
Pressures	below:

Gas Inlet	Pressure		
Carrier gas	80 psi		
Make-up	80 psi		
Air	60 psi		
H <sub>2</sub>	40 psi		

Connect

Follow the steps below to connect gas supplies to your 3800 GC:

#### Gases

-	
1	Cut required lengths of heat-cleaned 1/8" copper tubing for the carrier and fuel gas plumbing. Clean the ends of the tubing with a metal file.
2	Slide a 1/8" Swagelok® nut, back ferrule, and front ferrule over one end of the copper tubing and attach to the outlet fitting on the cylinder regulator.
3	Push the copper tubing into the outlet fitting as far as it will go, then pull back very slightly and tighten 3/4-turn past finger-tight with a 7/16" wrench.
4	Slide a 1/8" Swagelok nut, back ferrule, and front ferrule over the other end of the copper tubing and connect it to the corresponding gas inlet on the GC rear panel.



5	Push the copper tubing into the bulkhead fitting as far as it will go, then pull back very slightly and tighten 3/4-turn past finger-tight with a 7/16" wrench.
6	Make sure the gas supply control valve, which controls gas pressure to the GC, is completely closed. Open this valve slowly and monitor the pressure gauges on the dual stage regulator. The first pressure gauge should now read the pressure of the cylinder.
7	Cautiously turn the regulator valve to supply gas to the GC. Watch the pressure gauge closely and adjust the pressure according to the table above.
8	Leak check all fittings.

Connect GC to Power	Plug the GC power cord into an appropriate source of power. For site power requirements, refer to page 9.			
Turn Power	Turn on the GC power switch located at the top left of the GC.			
On	<ul> <li>The GC display should illuminate after a few moments and go through an initialization sequence.</li> </ul>			
	<ul> <li>The 3800 automatically enters the INSTRUMENT STATUS screen on power up. Press the SETUP key and select view SETUP to verify the 3800 configuration.</li> </ul>			
Factory Default States	Your GC is shipped from the factory with default settings. The following is relevant information on the factory default states and settings:			
and Settings	<ul> <li>When the GC is turned on, all installed heated zones except for the column oven default to power OFF.</li> </ul>			
	<ul> <li>The default active method is method 1. The column oven defaults to power ON and a set temperature of 50°C.</li> </ul>			
	• When installed components that use a heated zone are turned ON in the			

 When installed components that use a heated zone are turned ON in the active method, they default to a set temperature of 50°C.

## Quick Guide to the 3800 Keyboard and Display

Getting Around the Keyboard The 3800 user interface is designed for maximum ease of use. The keyboard is laid out in functional sections allowing quick access to the necessary information. This subsection of the manual gives a brief overview of the functionality associated with each section of the keyboard. For more detailed information refer to the Operator's Manual: *Section 1, The 3800 Keyboard and Display*.



### Figure 2 3800 GC Keyboard



The PAGE and CURSOR keys are used to navigate around the 3800 display and move between screens. Pressing the PAGE UP arrow key backs up to the previous page; pressing the PAGE DOWN arrow key advances to the next page. The cursor arrow keys move the screen cursor from one field to another.

To select a menu item, the cursor can be placed on the item and the ENTER key pressed or if the menu item has a number, the same menu item can be selected by pressing the appropriate number followed by the ENTER key.

Because the pertinent information cannot always fit on one screen, multiple pages are sometimes used. Where multiple pages are used this is always indicated on the title line of the display, e.g., Column Oven, Page 1 of 2.





If you are modifying a table such as a temperature ramp table or timed events table, the TABLE EDIT keys can be used to add new lines to the table, delete the highlighted line from the table, or clear the table of all entries except for initial default conditions.

The ENTRY section of the keyboard contains the numeric keys (0 -9, decimal point, minus sign, and infinity), the increment (INCR) and decrement (DECR) keys, the ENTER key, the HELP key, and the UNDO key. The INCR and DECR keys can be used to select from a range of values for a discrete parameter or used to increment or decrement a numeric parameter. Pressing the UNDO key will undo the last editing action. The ENTER key either advances the cursor to the next parameter if the value in the current field is not updated or updates the parameter value if a new value is entered. In the latter case the ENTER key must be pressed again to advance to the next parameter. A more convenient way to enter parameters is to use the cursor keys to move from one field to another after a parameter value has been changed.

Pressing the HELP key gives you a context sensitive explanation for the parameter at the cursor location.



The INSTRUMENT and GC CONTROL sections are most commonly accessed. The INSTRUMENT section is where the 3800 is configured in terms of installed options and allowable operating parameters, and where an overview of instrument status can be viewed. The configuration information is accessed using the SETUP key and the instrument status information is accessed using the STATUS key.



Methods are built in the GC CONTROL section. The user can build and store up to 8 methods on the 3800. Methods are built based on the components installed on the 3800 and identified in SETUP.

The following is more detailed information on the 3800 SETUP, STATUS and GC CONTROL functions.

SETUP The Instrument SETUP key allows access to the instrument configuration screens for the purpose of viewing or modifying the 3800 configuration. Note that the 3800 has already been configured at the factory and requires only minor input from the user. At the time of installation, the SETUP screens are normally accessed by the user to update the local time and date information, update the EFC display parameters and column parameters, or verify the instrument configuration. If the instrument configuration is changed, such as when adding a field upgrade kit, the instrument SETUP screens must be updated.

The same screens are accessed whether you choose to VIEW or EDIT Instrument SETUP. However, the contents of the screens can only be changed if you choose the EDIT option. Do not choose the Edit Option unless you want to change the 3800 configuration.

As shown in the screen below, there are five menu choices in Instrument SETUP.

ACTIVE METHOD	E ME	EDIT CT ETHOD	rrl ri	JN ME	END TIME
Method l	Met	hod 1	0.1	י סכ	20.00
	ED:	IT INSTRUMENT	SETUP MEN	U	
	LLJ Edit	Time and Date			
	TST FOIL	Heated Zones			
	E3] Edit	EFC			
	E4] Edit	Column Parame	ters		
	E5] Edit	Valves			
	E6] Edit	Miscellaneous	Setup Par	ameters	
		Save and	Exit		
Ο		Ο		C	

**Time and Date** is used to update to the current local time and date. Selection of the desire date format and language for display is also performed here.

**Heated Zones** identify the type of device installed in each of the heated zones of the 3800, the upper temperature limit of these devices, their location and what type of coolant, if any, the device uses. Column oven always occupies one heated zone. The other available heated zones are numbered 1 through 6 and can be occupied by one of the following devices: 1079 injector, 1041 injector, 1061 injector, SPT, FID, TCD, ECD, TSD, PFPD, Micro-TCD, large valve oven, small valve oven, or methanizer. SPT is allowed only in zone 3.

The location typically refers to whether the device is installed in the front, middle, or rear position on the top of the 3800. In the case of detectors the location also refers to the relative positions of the detector electrometers installed on the main electronics board. By default, zones 1, 2, and 3 are identified as front, middle, and rear and zones 4, 5, and 6 are identified as front, middle, and rear. Zones 1, 2 and 3 are normally used for injectors and zones 4, 5, and 6 are normally used for detectors.

The temperature limit for all zones defaults to  $450^{\circ}$ C but can be set to a lower value. The coolant choices are None, LN<sub>2</sub>, or LCO<sub>2</sub>. Note that zones 4 - 6 cannot be used with coolant.

The following screen shows an example of a configuration screen for the 3800 heated zones. This instrument is configured with two 1079 injectors, both of which have an  $LCO_2$  coolant option, and two FIDs.

ACTIVE METHO	E D	EDIT METHOD	CTRL	RUN TIME	END TIME
Ndelt ho		Ndeltho		<b>0.0</b> 0	<b>200.0</b> 0
		E HERONE S			
<b>žo</b> ne	<b>ëyp</b> e	"pe≝nimit		Location	Coolant
(lon)ven	(Loniven	()° (2 ()		(Loniven	Neone
l	3079	99°C)		Firont	NDC 0 2
2	3079	99°C)		Neiddle	NDC 0 2
З	Neone	99°C)		Rear	Neone
4	ÐID	99°C)		Firont	Neone
5	ÐID	199°C)		Neiddle	Neone
ь	Neone		89°C) Rear		Neone
Raddetxit					
0		(	C		Ο

**EFC** (Electronic Flow Control) identifies any installed EFC modules and their type. Types 1, 3, and 4 are used with inlet systems and Types 11 through 16 with detectors. Detector EFC Setup involves selecting the relevant gas type and, in some cases, the detector type. Inlet types allow you to set the pressure display units (psi, bar or Kpa), outlet pressure and minimum flow (type 1 EFC only). Outlet pressure is used to distinguish between a column venting to atmosphere or a column venting to a vacuum source such as a mass spectrometer. The Atm setting is used for the standard 3800 detectors such as FID, TCD, ECD, TSD, and PFPD. Minimum flow is the split vent flow rate that type 1 EFC maintains during the gas saver period of operation. EFC setup for type 1 modules (1079) also contains a septum purge calibration.

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 mis-match between set and actual flowrates resulting in a Not Ready condition in the 3800.

**Column Parameters** define values used for EFC calculations such as column length, internal diameter, and choice of carrier gas. Note that if Argon or Argon/Methane carrier gas is used, the Nitrogen option should be selected. The EFC calculations for Nitrogen, Argon, and Argon/Methane are essentially the same.

**Valves** defines the assignment of the seven available external events on the 3800. These are typically used with either manually controlled 1079 split valves (non-EFC) or actuators for sampling or switching valves. The setup screen identifies the valve type and default state, i.e., the state it is in when the valve is not energized. The following table summarizes the available valve types and their states. Note that the valves are normally identified on the 3800 display by their abbreviation, but when the display cursor is placed on a specific valve its full name is given on the prompt line of the display.

Abbr.	Default State (-)	Activated State (+)
FSV	ON (split)	OFF (splitless)
MSV	ON (split)	OFF (splitless)
RSV	ON (split)	OFF (splitless)
GSV	Fill	Inject
LSV	Fill	Inject
S	OFF	ON
IS	OFF	ON
SS	OFF	ON
SB	Series	Bypass
BD	Forward	Backflush
BV	Forward	Backflush
CS	Column 1	Column 2
IBD	Backflush	Inject
IBV	Backflush	Inject
AI	Column 1	Column 2
SI	Fill	Inject
MB	Series	Bypass
EA	OFF	ON
EB	OFF	ON
EC	OFF	ON
ED	OFF	ON
	Abbr. FSV MSV RSV GSV LSV S IS SS BD BV CS IBD IBV AI SI MB EA EB EC ED	Abbr.Default State (-)FSVON (split)MSVON (split)RSVON (split)GSVFillLSVFillSOFFISOFFSBSeriesBDForwardBVForwardCSColumn 1IBDBackflushAlColumn 1SIFillMBSeriesEAOFFEBOFFEDOFF

The miscellaneous setup parameters page allows changing the polarity of the "Ready-In" sync signal. Also, if an FID electrometer is installed it allows enabling/disabling of the flameout diagnostics.

STATUS The Instrument STATUS key allows the user to view the current status of the various components of the 3800 instrument. Several status screens are available based on component type or GC channel (front, middle, or rear). Note that the current status of individual components can also be viewed by pressing the relevant key in the GC CONTROL keyboard section.

The primary status information provided for each component using the STATUS key is the actual component temperature, carrier gas flow and pressure (if EFC is installed), and detector analog output signal. The following is an example of a status screen for a 3800 equipped with a 1079 injector and FID. Note that the status screen reflects the current state of the instrument. If the instrument is running a method, the status fields will update at a rate of once per second as the values change during the run.

ACTIVE METHOD		EDIT METHOD	CTRL	RUN TIME	END TIME
Method 1		Method 1		0.00	20.00
INSTRUMENT STATUS (FRONT)				L of E	
	Compor	ient	Status	5	
	1079 0	ven (°C)	250		
	Columr	n Flow (ml∕min)	1.0		
	Columr	• Pressure (psi)	15.0		
	Columr	ı Oven Temp (°C)	50		
	FID Ov	ven (°C)	300		
	FID Ou	itput (mV)	8.25		
0		0		Ο	

Note that status information may extend beyond one screen depending on the number of installed options on the GC. If this is the case, the PAGE UP/ DOWN keys can be used to move between pages. Status information is presented in the order: front channel, middle channel, and rear channel.

In addition to the above status information, specific status information on individual components can be accessed by pressing the relevant key on the 3800 keyboard. For example to view the current status of the column oven, the COLUMN OVEN key is pressed. The status information is shown at the top of the display above the heavy line separating the status information from the edit method information.

The following is an example of the screen shown when the COLUMN OVEN key is depressed. The column oven status field is shown at the top of the screen. Note that the stabilization time is also shown. This represents the period of time for which the instrument will stabilize after reaching its set temperatures and before going to the ready state.

ACTIV METHO	/E OD	EDIT CTRI METHOD	- RUN TIME	END TIME			
Metho	d l	Method 1	0.00	2 <b>0</b> · 00			
Set (°C):	50		Act	tual (°C): 50			
Stabiliza	tion Time (m	in): 2.00	Co	lumn Oven: On			
_	C.	DLUMN OVEN <sub>7</sub> Pag	je 1 of 2	_			
Step	Temp (°C)	Rate (°C/min)	Hold (mi	n) Total (min)			
Initial	50	-	5.00	2.00			
L	150	10.0	1.00	13.00			
2	250	20.0	5.00	23.00			
Turn Oven ON Turn Oven Off							
0		Ο		Ο			



The 3800 contains eight separately programmed analytical methods. On power up the system defaults to method 1. To activate another method the user can press the ACTIVATE key in the METHOD section and select from Methods 1 - 8. To edit a method the user can press the EDIT key in the METHOD section and select the method they wish to edit. The currently active method is by default the method that will be edited unless the user chooses a different method to edit. Both the currently active method and the method being edited are identified on the top line of the 3800 display.

A method is edited by entering method parameters into the various components of the GC CONTROL keyboard section. Components are presented in the order that the sample goes through the GC system i.e. Sample Delivery > Injector > Flow / Pressure > Column Oven > Detector > Output. The method can be edited by selecting any particular component and pressing that GC Control key or by stepping through the entire analytical method in the order shown above, i.e., if you enter values for the injector component, you are automatically taken to the Flow / Pressure component.

The following is an example of a simple analytical method consisting of a 1079 isothermal splitless injection using EFC pneumatics, basic column temperature ramp and FID detection. Only four method components are built: the injector, flow/pressure, column oven and detector.

# Note that all heated zones, with the exception of the column oven, are OFF when the 3800 is first powered up.

INJECTOR

The 1079 method uses three pages of screen displays, a page for setting the injector temperature (isothermal or temperature programmed), a page for fixed parameters such as coolant ON / OFF, and a page for programming the split state of the injector. Note that the injector is identified by its model number and location.

	ACTIVE METHOD		EDIT METHOD	CTRL	RUN TIME		END TIME
	Method	1	Method :	1	0.00		20.00
Set	(°C): 50	Actu	al (°C):	50	Split	Ratio: 0	FF
		F	RONT 107	79 Page l	of 3		
		Step	Temp	Rate	Hold	Total	
		Initial	250	-	20.00	20.00	
	Ο			Ο		(	0

The above screen represents the temperature settings for the 1079 injector, operated in typical isothermal mode. If the injector were temperature programmed, a new line would be added to add a ramp rate and additional hold time.

The other important 1079 parameter to program is the split state, which is programmed differently depending on whether the 1079 is equipped with manual pneumatics or EFC. The split state controls the flow of carrier gas through the injector during the analytical run. When the split state is OFF, most of the sample injected is directed onto the column. When the split state is ON, the sample is split in the injector with typically the smaller fraction entering the column and the larger fraction being vented.

The example shown below is a splitless injection with EFC pneumatics. In this case the injector is held in the split OFF state for a period of time at the beginning of the run (the sampling time). The injector is then switched to the split ON state, typically after 0.5 - 1.5 minutes, to vent the injector of any residual solvent. After the injector has been vented, the split ratio can be reduced to conserve carrier gas. In this example the split ratio is reduced to 5:1 after 1.5 minutes. The 1079 SPLIT STATE screen is accessed by pressing the PAGE DOWN key on the 3800 keyboard until page 3 of 3 is presented.

ACTIVE METHOD	EDI METH	T OD	CTRL	RUN TIME		END TIME
Method 1	Metho	d L		0.00		20.00
Set (°C): 25	Actual	(°C):	250	Split	Ratio:	OFF
	FR	NT 1079	Page 3	of 3		
	Time	Split S	tate	Split Ratio		
	Initial	0FF		0FF		
	0.75	ON		100		
	1.50	ON		5		
		-				
0		(	C			Ο

If the 1079 is equipped with manual pneumatics, the split state is programmed in the VALVE TABLE section of SAMPLE DELIVERY. For further information on this topic refer to the Sample Introduction section of the 3800 Operator's Manual.

Page two of the 1079 method contains fixed parameters which are rarely changed. These include turning on and off power to the injector heater and the coolant supply to the injector.

ACTIVE METHOD	EDIT METHOD	CTRL	RUN TIME	END TIME
Method l	Method 1		0.00	20.00
Set (°C): 50	Actual (°C):	50 Sp	lit Ratio: O	)FF
	FRONT 1079	Page 2 of 3		
	1079 Oven Power	0 N		
	Coolant ON/OFF	ON		
	Enable Coolant a	t (°C) 250	1	
	Coolant Timeout	(min) 5		
0	(	C	(	C

The 1079 oven must be turned off if the injector is being disassembled for maintenance. Coolant ON / OFF turns on or off the cryogenic supply to the injector, if installed. Enable coolant refers to the temperature at which the coolant is turned on when the injector is set to a specific temperature. For example if the injector is cooling from 300°C to 50°C, and the coolant enable is set to 250°C, the coolant will be turned on when the injector reaches 250°C. Coolant time-out refers to the period of time for which the coolant will remain on after it is turned on. For example, if the set temperature has not been reached or the run has not started within the specified time, the coolant supply will be turned off.

FLOW/ PRESSURE If the 3800 is equipped with Electronic Flow Control, then pressing the FLOW/PRESSURE key will bring up the screens associated with programming EFC. EFC modules are identified by type and location to coincide with the sample delivery or injection device with which they are associated. Type 1 EFC is used with the 1079 Universal Capillary Injector. The following screen is a typical EFC program for type 1 EFC. In this case, a pressure program is applied to keep the column flow rate constant while the column temperature is ramped. For further information, refer to the Sample Introduction section of the 3800 Operator's Manual.

ACTIVE METHOD		EDIT METHOD	CTRL	RU TIM	N E	END TIME	
Method	l M	ethod 1		0.0	10	20.00	
Column Pressure (psi): 10.0 Column Flow (ml/min): 2							
Linear Vei	locity (cm/	sec):35 T	otal Flo	w (m1/	min): 10		
Split Rat:	io: Off						
	FR0	NT EFC, Type	Ъ	Page	lof 2		
Step	Pressure (psi)	Rate (psi/	min)	Hold	(min)	Total (min)	
Initial	10.0	-		2.00		2.00	
Г	1.5.0	0.50		5.00		17.00	
Ο		0				0	

COLUMN OVEN

The column oven method section has two screens, one with the column oven temperature conditions and the second with fixed parameters. The temperature control screen could have one line of initial conditions in the case of isothermal operation, or a table in the case of temperature programmed operation.

The total time represents the cumulative time up to that point in the run. The Turn Oven Off softkey turns off the column oven heater and fan to allow access to the oven. Similarly, the Turn Oven On softkey is used to turn the column oven heater and fan back on.

ACTIVE METHO	) D	EDIT METHOD	CTRL	RUN TIME		EN TIM	D 1E
Method	1 P	lethod l		0.00	1	20.	00
Set (°C):	50				Actual	(°C):	50
Stabilizat	ion Time (mi	n): 2.00			Column	0ven:	0 n
	C <b>0</b> I	UMN OVEN	Page 1	of 2			
Step	Temp (°C)	Rate (°C	/min)	Hold	(min)	Total	(min)
Initial	50	-		2.00		2.00	
l	250	20.0		5.00		17.00	
Turn Oven On Turn Oven Off							
0		(	)			Ο	

Page 2 of the Column Oven parameters consists of fixed parameters which are rarely changed. These consist of coolant on/off, coolant activation temperature, coolant time-out and stabilization. The first three operate the same as described above for the 1079. Stabilization time is the time taken for the column oven to stabilize at its set temperature before allowing the GC to go to the ready state.

DETECTOR The 3800 detector methods generally have two method parameter pages and an additional adjustments page. The adjustments page is generally used for advanced features of the specific detectors and also for setting detector gas flows, if EFC is installed. The following is an example of an FID method. For the FID the only adjustments item is the detector time constant (Fast or Slow).

ACTIVE METHOD	EDIT CTRL METHOD	RUN TIME	1 E	END TIME
Method 1	Method l	0.0	0	20.00
Set (°C): 300	Actual (°C): 300	Si	gnal (mV)	: 5.15
	FRONT FID Page 1	of 2		
	≬ven Power∶	ON		
	Temperature (°C):	300		
	Electronics:	ON		
	Range:	75		
		-		
IGNITE	AUTOZERO		A⊅JU	ISTMENTS
Ο	Ο			0

The second page of method parameters for the FID allows the user to time program certain events such as range and autozero during the analytical run.

ACTIVE METHOD	EDIT METHO	CTRL D	RUN TIME	END TIME
Method 1	Method	<u>г</u>	0.00	20.00
Set (°C): 30	O Actua	1 (°C): 300	Signal	(mV): 5.15
	FROM	NT FID Page	2 of 2	_
	Time	Range	Autozero	
	Initial	75	NO	
	5.00	75	YEZ	
	6.50	77	NO	
IGNITE		AUTOZERO		ZTNAMTZULGA
Ο		0		Ο

## **Column Installation and Conditioning**

Connect Column to Injector Your GC arrives ready for installation of an analytical column. On first installation, a new column needs to be conditioned. **Conditioning is performed with the detector end of the column unattached.** 



You may need to install and condition a new column after your GC has been in use for some time. Reset all injector, detector and column ovens to  $\leq 50^{\circ}$ C to avoid burn hazards when connecting column to injector and detector parts inside the oven.

Prior to installing your column, check that the factory-installed injector is equipped with the proper insert for your application. Factory-installed injectors, other than the 1041, have a glass insert suitable for a variety of applications. If your application calls for a different insert, replace the factory-installed insert prior to installing your column. Refer to the Injector section of the Operator's Manual for instructions to replace the insert. The factory-installed inserts for the injectors are shown below.

Injector	Insert Information	Part Number
1061	4 mm ID Flash (530μ)	03-918339-00
1079	Open 2 mm ID Splitless	03-918466-00

Nuts and Ferrules for Capillary Columns Capillary columns are generally installed with a capillary column nut (P/N 03-949551-00) and a single reversed ferrule, as shown in the diagram. For connection to the TCD, a special capillary adapter (P/N 03-925605-01) is used.

 A Quick Connect Kit (P/N 03-925355-90) facilitates easy connection of capillary column to injector and detector. The kit comes with instructions and contains: column depth scale, knurled split nuts (2), and graphite jacketed ferrules (2 each of 0.4, 0.5 and 0.8 mm ID, for 250 µm, 320 µm, and 530 µm columns respectively).



Three types of ferrules are common: graphite, Vespel® or polyimide, and graphite/Vespel.

- Do not use graphite with ECD or GC/MS systems.
- Ferrule ID is based on the diameter of the capillary column. See the following table.

			Part Number		
Ferrule ID (mm)	Fitting Size (inches)	Column ID (microns)	Polyimide (10/pk)	Graphite (10/pk)	Polyimide/ Graphite (10/pk)
No hole	1/16" fitting	—	28-694503-01	—	28-694590-01
0.4 mm	1/16" fitting	50 - 250	28-694586-01	28-694583-01	28-694580-01
0.5 mm	1/16" fitting	320	03-908361-01	28-694561-01	28-694581-01
0.8 mm	1/16" fitting	530	28-694552-01	28-694042-01	28-694582-01

Capillary column connection to the Micro-TCD requires a special series of graphite/Vespel ferrules (see table below).



Column ID	Ferrule ID	Ferrule Part Number
0.1 mm	0.4 mm	CP85889
0.25 mm	0.4 mm	CP85889
0.32 mm	0.5 mm	CP470100
0.53 mm	0.8 mm	CP470101

Follow steps 1-7 to install a capillary column into your injector.

#### Connect Capillary Column to Injector

		1041 (530μ)	1061 (530µ)	1079 (50-530µ)
1	Cut off sealed section at each end of new column.	~	1	✓
2	Uncoil 10 cm at injector end of column.	✓	✓	~
3	Slide appropriate column nut and ferrule on each end of column.	~	~	✓
4	Make a clean cut with the cutting tool 1-2" (2-4 cm) from end of the column. Inspect the cut to verify it is square and smooth.	~	~	~
5	Move nut and ferrule to within 5 cm of column end.	✓	✓	
	<i>For 1079 injector (except "on-column" modes):</i> Measure and mark column at 7.5 cm from end.			$\checkmark$
6	Holding column in one hand, gently slide column into injector until it stops OR	~	✓	
	For 1079 injector (except on column modes): Engage the column nut on the injector and then slide the column into the injector until the mark on the column aligns with the bottom of the nut.			✓ 
7	Finger-tighten the column nut while holding the column in place, then tighten a further 1/4 to 1/2-turn with a 5/16" wrench.	~	~	$\checkmark$

Nuts and Ferrules for Packed Columns

- Metal packed columns are usually installed with a metal 2-piece ferrule. (A commonly used metal packed column is 1/8" OD stainless steel. It uses a 1/8" ID stainless steel ferrule.)
- **Glass** packed columns are 1/4" OD and are installed with a graphite front ferrule and a metal back ferrule installed backwards. See diagram below.



- A Two Piece Ferrule
- B Column Nut
- C Back Ferrule
- D Front Ferrule
- E Column Nut
- F Metal Back Ferrule Installed Backwards
- G Graphite Ferrule

### Connect a Packed Column to the Injector

Follow the steps below to install a new packed column into your 1041 or 1061 injector. Note that the injector must first be converted for packed column use by installing the appropriate adapters.

1	Place nuts and ferrules on both ends of column as shown in the previous diagram.
2	If your column is metal, insert it into the injector fitting and gently push until it stops. Manipulate the column so the detector end points into the oven OR If your column is glass, insert it into the injector, rotate it slightly so
	the detector end is out of alignment with the detector and push gently until the end is near but not touching the oven ceiling.
3	Hold the column in place and thread the nuts by hand.
4	Tighten the nut finger-tight, then give it a further 3/4-turn with a small wrench.

Varian TestAt points throughout this manual, specific values and conditions are given<br/>for the analysis of Varian Test Samples on Varian Test Columns. The<br/>following table provides information on Varian Test Columns.

Column Description	For use with Injector Models	Part Number
Packed 50 cm x 1/8" OD stainless steel OV-101.	1041/1061 Packed Adapters	03-912300-30
Capillary 4m x 320µ ID DB-1 fused silica.	1079	03-912805-99
Capillary 4m x 320µ ID DB-1 fused silica (a) connected to a short pre-column of 0.53 mm fused silica (b) using a Press-Fit <sup>™</sup> connector (c). The above parts are also available as a kit (d).	1041/1061	03-912805-99 (a) 00-997564-03 (b) 00-997668-03 (c) 03-912809-90 (d)

#### Varian Test Columns

IdentifyGases are plumbed at the factory through the rear bulkhead fittings into the<br/>inlet manifold, then to the pneumatic controls in the pneumaticsControls and<br/>Turn on FlowCompartment. The pneumatic controls can be either electronically controlled<br/>and are different for each injector system.

lf your injector model is a	Carrier gas flow is regulated by a
1041/1061	Type 3 EFC or upgrade to type 4 with valving.
1079	Type 1 EFC <b>or</b> Flow controller (controls flow into injector) <i>and a</i> Back pressure regulator (controls flow through column)

- Carrier gas flow is adjusted at the user interface for EFC or at the pneumatics panel for manual pneumatics. For manual pneumatics carrier gas systems, identify the controls for carrier gas flow at the pneumatics panel for your injector system. These pneumatic controls are identified by their location, i.e., front, middle, rear.
- Turn on carrier gas flow by adjusting the manual flow controller or setting a positive pressure or flow on the EFC system.
- Verify that there is a positive pressure on the pneumatics panel pressure gauge or EFC status screen.

1079 Split Vent
Flow and
Septum Purge
Flow
Whether you are making an injection in the split or splitless mode, a split vent flow rate needs to be set. In this example, the split vent flow is set to 100 mL/min. The split state is set in the Injector section of the 3800 method if EFC is installed and in the VALVE TABLE section of SAMPLE DELIVERY if manual pneumatics are installed. In addition to a split state a septum purge flow rate should also be set. The following information is for setting a split vent flow rate with EFC installed. For a 1079 with manual pneumatics see the Injector section of the Operator's Manual for further information.

- 1. Press the FLOW PRESSURE key and set the appropriate head pressure for your analytical column.
- 2. Press the INJECTOR key in the GC CONTROL section of the 3800 keyboard and select the 1079 injector by location.
- 3. Press the PAGE down key to access the SPLIT STATE screen (page 3 of 3). When Electronic Flow Control is installed, in addition to programming the split state, the split ratio can also be programmed.
- 4. Set the initial split state to ON and set the split ratio to 100.
- 5. Open the column oven door and adjust the septum purge needle valve located above the column oven until the flow rate is in the range 3 5 mL/min. For EFC equipped systems this value has to be entered in the septum purge calibration routine. Use the following procedure to carry out septum purge calibration:
  - Press the STATUS key and note the current column pressure for the channel you are using (front, middle, or rear).
  - Select the edit SETUP option and select EFC. Select the injector you are setting up by specifying the location (front, middle or rear).
  - Press the SEPTUM PURGE CALIBRATION softkey. You will be prompted to enter the injector pressure and septum purge flow rate. Enter the pressure value noted above and the septum purge flow rate measured earlier. The septum purge calibration is completed automatically when you press the "Save and Exit" softkey.

Set Carrier Gas Flow Rate to Condition Column	To condition the column, flow rates need only be approximate. For packed columns and large bore (0.53 mm ID) capillary columns set the flow rate to 15 - 30 mL/min. Press the FLOW / PRESSURE key, select the appropriate injector by location, and set the initial flow to 15 - 30 mL/min (15 mL/min for a large bore column and 30 mL/min for a packed column). Use the following guide to set an appropriate column flow for the 1079 injector with narrow bore capillary columns. With manual pneumatics the pressure is set using the 1079 back pressure regulator on the pneumatics panel. If the 1079 has EFC press the FLOW / PRESSURE key, select the appropriate injector by location and set the pressure as follows:			
		Column ID F	lead Pres	sure
		250 µm	25 psi	
		320 µm	15 psi	
		530 μm	5 psi	
Note for Capillary Column Users	Verify gas is flow column into a be carrier gas flow	wing through the c eaker containing a should be indicate	olumn by c solvent, s ed by a stre	lipping the detector end of uch as isopropanol. Positive am of bubbles.
Conditioning the Injector and Column	It is important to temperature for column or can b allowable tempe conditioning pur above the maxin below the maxin lower.	establish the max the installed colur be obtained from the erature for the colu poses. In general mum temperature num operating ten	kimum reco nn. This ir ne column imn does r the colum used in the nperature o	ommended conditioning formation is provided with the manufacturer. The maximum ot have to be used for n should be conditioned at 20°C e analytical method, or 20°C of the column, whichever is



A GC column may be irreversibly damaged if it is heated with air or moisture present in the pneumatic system. It is important to purge the column with inert carrier gas before heating the column. After the column flow is established, allow the carrier gas to purge through the column for 15 - 20 minutes before heating it. In this example the injector and column are conditioned at 250°C for two hours. The column is programmed to 250°C at 5°C/min as a gentle temperature program is generally recommended for column conditioning. After this conditioning is complete, allow the column oven to cool and retighten the column nut attaching the column to the injector. This is especially important when graphite ferrules are used.

To condition the injector press the INJECTOR key in the GC CONTROL section of the 3800 keyboard. Set the injector temperature to 250 (initial temperature for a 1079). If a 1079 injector is being used, ensure that the initial split state is ON and that no program is present to time program the split state (the split state table should only consist of the initial line). Verify that the split ratio is set to 100.

To condition the column press the COLUMN key in the GC CONTROL section of the 3800 keyboard and build the following column oven method.

Initial Column Temperature: 50°C Hold Time: 5 min Step 1 temperature: 250°C Step 1 ramp rate: 5°C/min Step 1 Hold Time: 120 min

When the 3800 reaches the READY state indicated by the READY LED being lit, press the START key to begin the conditioning process.

Connect Capillary Column to Detector After the column has been conditioned, follow the steps below to connect the capillary column to the detector.



Hot surfaces are exposed.

Check that the capillary column nut and ferrule are installed on the column. Note that if the TCD is used with capillary columns, a special capillary adapter (PN 03-925605-01) is used on the TCD inlet. DO NOT use graphite ferrules with the ECD.
Uncoil about 20 cm from end of column.
3 Move the column nut and ferrule to within 5 cm of the column end. Find the column insertion depth for your detector in the diagram below. Measure the correct distance from the end of the column and mark this distance. Refer to diagram below for correct distances. DO NOT let the column nut and ferrule fall past this mark. The marking medium can contaminate the ferrule.



For the Micro-TCD, the end of the column should be flush with the end of the ferrule when installed on the detector fitting.



4	Place the column into the base of the detector.
5	Thread the capillary column nut and ferrule finger-tight.
6	Gently slide the column into the detector until the mark on the column aligns with the bottom edge of the column nut.
7	Continue to hold the column as you tighten the nut just enough to hold the column firmly in place and to create a good seal.

Connect Metal<br/>PackedAfter conditioning your column and cooling the GC, follow the steps below<br/>to connect your metal packed column to the detector. Note that the<br/>appropriate packed column adapter must be installed.Detector



#### Hot Surfaces are Exposed.

1	If using a detector other than TCD, locate the packed column detector adapter in the Packed Column Kit and install on the inlet to the detector. The TCD inlet is designed for packed column installation.
2	Insert the open end of the packed column into the detector inlet or adapter, and push until it reaches a stop.
3	Hold the column in place and finger-tighten the column nut. Then, with a 7/16" wrench, tighten a further 3/4-turn past finger-tight. On subsequent installations, tighten the nut 1/4 to 1/2-turn past finger-tight.

Check for Leaks Inspect your GC system for leaks before heating. An electronic leak detector is the most reliable method to check the injector and detector fittings for carrier gas leaks.



Never use soaps, such as Snoop®. These may contaminate the system and cause permanent damage to capillary columns. If a liquid leak detector is desired, try a 50/50 mixture of isopropanol/water.

## **Basic Operation**

# **IMPORTANT Note:** At points throughout this section, specific values and conditions are given for analysis of the Varian test samples on Varian test columns. Your analytical column and application may require different values and conditions. Please ascertain the range of operating temperatures for your analytical column and make sure your settings are within this range.

#### **Setting Carrier and Detector Gas Flow Rates**

The following table indicates the typical values for carrier and detector gas flow rates. For practical purposes a generalized carrier gas flow rate of 2 mL/min is used for capillary columns. Note that the carrier gas and make-up combined flow is typically 30 mL/min.

Detector	Carrier Gas	Carrier Ga (mL Packed	is Flow Rate /min) Capillary	Make-Up Gas	Make-Up + Carrier Gas Flow Rate (mL/min)	Detector Gas Air 1 (mL/min)	Detector Gas Air 2 (mL/min)	Detector Gas H <sub>2</sub> (mL/min)
ECD	N <sub>2</sub> or Ar/CH <sub>4</sub>	30 ±1	1 - 15	N <sub>2</sub> or Ar/CH <sub>4</sub>	30 ±1	—	_	_
FID	N <sub>2</sub> , H <sub>2</sub> or He	30 ±1	1 - 15	N <sub>2</sub> or He	30 ±1	300 ±10	—	30 ±1
TCD*	N <sub>2</sub> , He or H <sub>2</sub>	30 ±1	1 - 15	Same as Carrier Gas	30 ±1	—	—	—
PFPD	N <sub>2</sub> , He or H <sub>2</sub>	30 ±1**	1 - 10	None		17 ±1	10 ± 1	13 ±1
TSD	N <sub>2</sub> or He	30 ± 1	1 - 15	Same as Carrier Gas	30 ±1	175 ±10	_	4 ±0.2
Micro- TCD***	H <sub>2</sub> , or He		1 – 5	None	—			
*TCD Reference Flow – Rate for carrier gas flow through the reference cell is 30 ±1 mL/min.								
** <b>PFPD with packed columns</b> – an effluent splitter is used to reduce the carrier flow to the detector to < 5 mL/min.								
***Micro-TCD reference flow is provided by a reference column of the same length and ID as the analytical column.								

 Table 2 Flow Rates for Carrier and Detector Gases for the 3800 Gas Chromatograph

The following instructions are for instruments configured with manual pneumatics. Gas flows for detectors with EFC are set in the Adjustments Section of the detector method.

Follow the steps below to set all gas flow rates on the 3800 Gas Chromatograph. Flows are typically set with carrier gas first followed by make-up and detector fuel gases. The most convenient way to measure or verify gas flows is at the detector exit. Adapters are provided in the individual detector accessory kits for this purpose.

1	Attach a bubble or digital flowmeter to the detector outlet using the appropriate adapter.
2	Turn off make-up and detector gases at the pneumatics panel. For a needle valve this is accomplished by turning the needle valve cartridge knob fully clockwise. For a flow controller this is accomplished by turning the flow controller knob fully counter- clockwise or until the turns counting dial reads zero.
3	Observe reading on flowmeter. Ensure it is within the range described in the table for carrier gas flow rates.
4	Adjust the carrier gas flow using the manual pneumatics controls on the pneumatics panel or by adjusting the Electronic Flow Control to achieve the desired flow rate.
5	For a capillary system, turn on the make-up needle valve and adjust to achieve the desired flow rate. The combined carrier and make-up flow should be 30 mL/min. Adjustment is done with a small flat screwdriver inside the needle valve cartridge knob.
6	Turn on the relevant detector fuel gases in the sequence hydrogen followed by air. Use the table below to calculate the combined flows for the specific detector. For the TSD adjust the hydrogen flow controller until the turns dial reads 400.

Detector	Carrier + make-up	Carrier + make- up + hydrogen	Carrier + make-up + hydrogen + air1	Carrier + make-up + hydrogen +air1 + air2
FID	30	60	360	
TSD	30	34	209	
PFPD	2	15	32	42

#### **Condition the GC System**

As the injector and column were conditioned earlier, this procedure represents a quick conditioning of the entire GC system including the detector. Follow the steps below to build an analytical method to condition the GC system.

1	Press the INJECTOR key in the GC CONTROL section of the 3800 keyboard and verify that the injector temperature is 250°C. If the injector is a 1079 verify that the initial split state is ON and that there is no split time program. Set the split ratio to 100.
2	Press the COLUMN key and set the initial column oven temperature to 250°C.
3	Press the DETECTOR key and set the detector oven temperature to 300°C. If the detector is an FID, wait for the detector zone to get above 100°C and then press the ignite softkey to ignite the flame.
4	Allow the system to condition for one hour.

#### Build a Method to Analyze a Test Sample

The conditions for running a test sample depend on the sample chosen. The test sample is typically one of the following:

- the column test mixture supplied by the column manufacturer,
- a Varian Test Sample, or
- a test mixture you select as an indicator of system performance for a particular analysis.

#### Method to Analyze a Varian Test Sample on a Varian Test Column

For use with	Sample Composition	Part Number
ECD	33 ppb Lindane and Aldrin in iso-octane	82-005048-02
FID (Packed Column)	0.03% C14, C15, C16 in iso-octane	82-005048-00
FID (Capillary Column)	0.003% C14, C15, C16 in iso-octane	82-005048-07
TCD	0.3% C14, C15, C16 in iso-octane	82-005048-01
PFPD	0.002% n-dodecanethiool, tributyl phosphate and methyl parathion, and 0.4% n-pentadecane in iso-octane	82-005048-03
TSD	2 ppm azobenzene and methyl parathion, 4 ppm malathion, and 4000 ppm heptadecane in iso-octane	82-005048-04
Micro-TCD	68 mg/mL n-hexane in iso-octane	CP84886

#### Varian Test Samples

**Note:** These conditions may not conform to the requirements of your analytical column. Do not exceed your column's T<sub>max</sub>. See Column Manufacturer's specifications for your column.

#### For Systems with 1041/1061 Injectors: Method Settings to Analyze Varian Test Samples on Varian GCs Installed with Varian Test Columns

Note: Capillary columns use a temperature program while packed columns use isothermal conditions.

	ECD	FID	TCD <sup>1</sup>	TSD	PFPD	Micro-TCD <sup>2</sup>
Initial column temp (°C)	80	80	80	80	80	40
Initial column hold time (min)	1	1	1	1	1	1
Final column temp (°C)	200	200	200	200	200	200
Final colum hold time (min)	5	5	5	5	5	5
Initial column temp (°C) (Isothermal operation)	190	140	140	175	175	_
Initial column hold time (min) (Isothermal operation)	15	15	15	15	15	—
Injector temp (°C)	220	220	220	220	220	150
Detector temp (°C)	300	300	220	300	300	110
Range	10	10	0.05	12	10	0.5
Initial Autozero	On	On	On	On	On	On
TSD bead current (amps)				$3.0\pm0.3$		

<sup>1</sup> TCD Filament temp: 270 ±20°C; filament current: 200 ±10 mA, polarity: positive

<sup>2</sup> **Micro-TCD** Test sample must be diluted 1:20 before injection on a 1041/1061 injector.

	ECD	FID	TCD <sup>1</sup>	TSD	PFPD	Micro-TCD
Initial column temp (°C)	80	80	80	80	80	40
Initial column hold time (min)	1	1	1	1	1	1
Program 1 final temp (°C)	200	200	200	200	200	200
Program 1 ramp rate (°C/min)	20	20	20	20	20	20
Program 1 hold time (min)	5	5	5	5	5	1
Injector temp (°C)	220	220	220	220	220	150
Detector temp (°C)	300	300	220	300	300	110
Range	10	11	0.05	12	10	0.5
Initial Autozero	On	On	On	On	On	On
TSD bead current (amps)	-	-	-	3.0 ±0.3	-	-
Injection mode	Splitless					Split
Initial split state	OFF					On
Step 1 time (min)	0.75					-
Step 1 split state	ON					-
Step 1 split ratio	100					25

For Systems with 1079 Injectors Method Settings to Analyze the Varian Test Sample on Varian GCs Installed with the Varian Test Column

<sup>1</sup> **TCD** Filament temp: 270 ±20°C; filament current: 200 ±10 mA, polarity: positive

#### **Prepare the Test Sample**

You are now ready to prepare your sample. For preparation of samples for automated injection, please refer to the *8200/SPME AutoSampler Operator's Manual, P/N 03-914692-00.* 

You may inject your sample neat or diluted in a solvent. The Varian PFPD test sample should be diluted 1:20 in iso-octane if you intend to use it for a splitless injection. The choice of solvents depends on a number of factors, including component solubility, type of detector, and polarity of the analytical column. Select solvents to match the polarity of your column.

#### Solvents for GC

Following are some recommended solvents for non-polar, intermediate, and polar phase columns.

Column Phase	Recommended Selvent	Dailing Daint
Column Phase	Recommended Solvent	Bolling Point
		(°C)
Non-Polar	Pentane	36.1
<ul> <li>100% Methyl</li> </ul>	n-Hexane	69.0
<ul> <li>5% Phenyl, 95% Methyl</li> </ul>	Cyclohexane	80.7
	<ul> <li>Iso-octane</li> </ul>	99.3
	Benzene	80.1
	Toluene	110.6
	Ethyl Ether	34.6
	<ul> <li>Methyl tert-butyl ether</li> </ul>	55.2
	Methylene Chloride	39.8
	Chloroform	61.2
	Carbon Disulfide	46.5
Intermediate	Ethyl Acetate	77.0
<ul> <li>50% Phenyl, 50% Methyl</li> </ul>	Acetone	56.5
	<ul> <li>Methyl iso-butyl ketone</li> </ul>	127.0
	Acetonitrile	81.6
Polar	Methanol	64.7
<ul> <li>Polyethyene Glycol</li> </ul>	Ethanol	78.5
	2-Propanol	82.5
	n-Butanol	117.7

#### **Recommended Solvents for Gas Chromatography**

#### **Sample Volume and Injection Rate**

The quality of your chromatogram can be greatly affected by the sample volume and injection rate. These vary according to the type of injector and column you are using. Shown below are some recommended sample volumes and injection rates. Draw the appropriate volume of sample into your syringe and inject at specified rate.

Injector	On-Column, Flash Vaporizing (1041/1061)	Split (1079)	Splitless (1079)
Column	<ul> <li>For</li> <li>Large-bore Capillary Columns (0.53 mm ID)</li> <li>Packed Columns</li> </ul>	<i>For</i> All Capillary Columns	<i>For</i> All Capillary Columns
Sample Volume in μL	<ul><li>0.1-1</li><li>1 - 5</li></ul>	0.2-1	1-5
Injection Rate, in μL/sec	<ul><li>0.5-5</li><li>1 - 10</li></ul>	5	1

#### Inject the Test Sample

Use 1µL of the Varian test sample for all detectors. In addition, if you are using a PFPD, make a 1:20 dilution of the test sample in iso-octane. With the READY light on at the GC and the appropriate volume of test sample in your injection syringe, you are ready to make your injection. For automated injection of samples, please refer to the 8200/SPME AutoSampler Operator's Manual, P/N 03-914692-00.

- To inject your sample, insert the syringe needle into the injector inlet, press the syringe barrel against the injector switch and depress it completely, simultaneously pressing down the syringe plunger. Remove the syringe from the injector immediately.
- The RUN light comes on and stays on until the analysis ends. The run may be stopped by pressing the STOP key.
- Compare the resulting chromatogram with your known test chromatogram.
- If you are analyzing the Varian Test Sample on a Varian Test Column, you may compare your resulting chromatogram with the appropriate test chromatogram in the Detector section of the Operator's Manual. The retention times will be close to those shown in the manual. The peak heights may be considerably different.
- Failure to duplicate the results you expected may indicate an installation, operation or instrument problem. This problem must be corrected before proceeding.

### Maintenance

#### Overview

It is important for the GC user to learn general maintenance techniques and carry these procedures out on a regular basis. Some common GC maintenance procedures are changing septa and injector inserts, checking for leaks, conditioning columns and changing filters. In this section maintenance information is presented under four headings: general GC maintenance, injector maintenance, column maintenance and detector maintenance.

Note that many maintenance procedures, such as repair or replacement of electronic components, should be performed by a qualified Varian service representative. For maintenance problems where mechanical or electrical assemblies need to be repaired or replaced please call your local Varian service center.

There are certain maintenance tasks that should be carried out on a routine basis. These regular procedures are done to ensure that the 3800 Gas Chromatograph will continue to operate at optimum performance. The following is a brief summary of some common maintenance tasks and their typical frequency:

#### Maintenance Task

Change septa Check column nuts are tight Condition column Change injector inserts, if necessary Check gas supplies Leak check Change gas cylinders Condition system Clean detectors Replace gas purifiers Perform AutoCalibrate (EFC)

#### Frequency

Typically 50 - 100 injections Daily Daily or as required Weekly Weekly Monthly Quarterly or as required Semiannually When performance deteriorates Annually or as required Semi-annually

#### **General GC Maintenance**

The common general GC maintenance tasks are checking and changing gas supply cylinders, leak checking, and changing gas purifier filters. These should be performed at the frequency suggested above. The following procedures are critical to the successful long term operation of a Gas Chromatograph. It is very important to leak check the system on a routine basis and to ensure the quality of gas supplies, particularly the carrier gas.

Check and<br/>Renew GasThe pressure of the various GC gas supplies should be checked on a weekly<br/>basis and the following guidelines used for frequency of renewing supplies:Supplies

**Carrier gas:** The carrier gas supply cylinder should be changed when its pressure drops below 200 psi. This ensures that high purity carrier gas is always supplied to the instrument. With typical usage on one Gas Chromatograph an A-size cylinder of carrier gas should last for three to six months. Note that this usage includes also using the carrier gas as the supply of make-up to the detectors. When a new cylinder is installed the regulator and tubing should be purged with carrier gas before connecting to the GC. This will avoid introducing a large amount of air into the GC.

**Detector fuel gases:** The typical detector fuel gases are hydrogen and air. These should be changed when the supply pressure drops below 200 psi. Typically an A size cylinder of hydrogen used on one GC with one Flame Ionization Detector lasts about six months. However, due to the high flow rate of air used in a Flame Ionization Detector this supply will need renewing more frequently. For this reason it is worthwhile to consider a clean supply of compressed air for FID equipped instruments.

Leak Check Leak checking is one of the most important maintenance tasks that is carried out on a Gas Chromatograph. The following information refers to general leak checking procedures for the 3800 Gas Chromatograph. Specific information regarding individual 3800 components will be presented in the relevant part of this maintenance section.

Leaks in the GC system can lead to poor chromatographic performance or damage components such as the analytical column. The presence of oxygen in the GC carrier gas at elevated temperatures can lead to permanent column phase degradation. While the use of an oxygen filter on the carrier supply to the instrument can help, leaks downstream of the filter are generally more likely to be the problem. The use of soap-based leak detection fluids is not recommended for a high performance capillary Gas Chromatograph due to the danger of introducing contaminants into the system. If leak detection fluids are being used they should only be used outside the Gas Chromatograph, i.e., to verify there are no leaks in the plumbing to the Gas Chromatograph. If a leak detection fluid is being used inside the Gas Chromatograph, an alcohol such as isopropanol or a 50:50 mixture of isopropanol and water should be used. Use a dropper or syringe to place a few drops of leak detection fluid on the desired fitting and monitor the area for bubble formation.

The most important step in leak checking is to verify that the GC system can hold pressure. This is done by removing the column from the injector, sealing all exits from the injector and pressurizing the system. Use the following procedure to leak check 3800 injectors:

**Note:** When conducting leak check procedures it is important to completely seal all carrier gas exits, including septum purge and split vent outlets. If more than one injector is present, then the outlets of all the injectors must be sealed. The following procedure refers to one injector but should be duplicated for every injector on the 3800.

1	Set the column oven and the injector zone temperatures to 50°C and allow them reach this temperature. This allows safe handling of injector surfaces.
2	Remove injector septum nut and install a new septum. An older septum can often be the source of a substantial leak.
3	Remove the column from the injector. Use the appropriate nut and ferrules to seal the base of the injector. For packed column systems use a 1/4" Swagelok blank off plug (P/N 16-000154-00). For capillary systems use a capillary nut (P/N 03-949551-00) with a no hole ferrule (P/N 28-694590-01).
4	If the injector is a 1079, then the septum purge and split vent outlets must be sealed. The septum purge outlets are located on the top frame surrounding the column oven, behind the column oven door. The split vent outlets are located on the left side panel of the 3800. Seal the septum purge by removing the outlet fitting from the septum purge valve and replacing it with a blank off plug (P/N 16-000154-00). Seal the split vent outlet by installing a Swagelok union on the outlet tube and sealing it with a blank off plug.

5	With all outlet ports plugged, pressurize the system to 20 - 30 psi. This can be accomplished by adjustment of manual pneumatic controls or Electronic Flow Control. For information on setting carrier gas pressure see the Basic Operation section of this manual.
6	Shut off the carrier supply at the source and monitor the displayed pressure at the GC for 15 minutes. The pressure should not drop more than 0.5 psi in 15 minutes.

Leak checking ensures that there are no leaks in the GC system up to and including the injector assembly. If the indicated pressure on the GC drops by more than 0.5 psi during the 15 minute test period, this indicates that there is a positive leak in the system. Finding such a leak, particularly if it is a small leak, can be very difficult. In general the best approach is to systematically go through the pneumatic system from the source and tighten each fitting until the leak is eliminated. It is important to note that leaks are often found in the carrier gas supply to the GC.

Locate leaks with an electronic leak detector or leak detection fluid. An electronic helium leak detector is available from Varian (P/N 01-900153-01). This device can detect helium concentration in the air as low as 2 ppm and is very effective at identifying the precise location of a helium leak from a GC system.

**Gas Purifier Replacement** The most common gas purifiers used in gas chromatography are moisture traps for removing water vapor, hydrocarbon traps for removing low levels of hydrocarbon contaminants and oxygen traps for removing low levels of oxygen. In general the choice of filter depends on the application and detector used. Carrier gas should contain less than 1 ppm oxygen, moisture and trace hydrocarbon contaminants. A combined moisture / hydrocarbon filter is recommended for general applications to maintain detector background levels at their lowest possible level. If the detector used is an ECD or MS an oxygen filter should be employed as well.

Carrier gas filters are typically located on the rear panel of the 3800. Varian supplies several types of carrier gas purifiers. These include a combined moisture / hydrocarbon trap, indicating moisture trap, and indicating or non-indicating oxygen traps. While the choice of purifier depends on the individual configuration and performance expectations, the following configuration is recommended for maximum carrier gas purity and protection.

The carrier gas should first pass through a hydrocarbon trap, then an indicating moisture filter, then a high capacity oxygen trap and finally through an indicating oxygen trap. As moisture reduces the effectiveness of an oxygen trap, a moisture trap should always be placed in front of an oxygen trap. When the indicating oxygen trap begins to change color, the high capacity oxygen trap should be changed immediately. The moisture trap should also be changed immediately when it changes color to prevent breakthrough of moisture to the oxygen trap.

The minimum recommended carrier gas purifier configuration is a combined moisture / hydrocarbon trap. This configuration can be used with routine GC analyses using FID, TCD, TSD and PFPD detectors. If an ECD or mass spectrometer is being used, an indicating oxygen trap should also be added. For trace analysis the combination of hydrocarbon trap, moisture trap and oxygen traps should be used.

Use the following procedure to install or replace a carrier gas purifier on the 3800. Gas purifiers supplied by Varian and most other chromatography suppliers are equipped with 1/8" Swagelok fittings. This facilitates easy removal and replacement of purifiers. In addition gas purifiers are sealed at both ends for protection. Purifiers should only be uncapped when you are about to install them.

1	Turn off the carrier gas supply at the input regulator. Note the operating pressure to allow resetting to this value after the purifier is installed.
2	Carefully open the carrier gas fitting at the inlet bulkhead to the GC or the existing carrier gas purifier. Wait for the release of carrier gas pressure before removing the fitting.
3	Identify the recommended direction of carrier gas flow on the purifier. This is normally indicated by an arrow on the side of the purifier. Remove the inlet cap from the purifier and immediately connect the carrier gas inlet line to the purifier inlet.
4	Remove the outlet cap from the purifier, turn the carrier supply back on and allow the carrier gas to purge through the purifier for 0.5 - 1 minutes.
5	Connect the outlet of the gas purifier to the inlet of the GC or the inlet of the next purifier, if used. If a new connection is being made, use chromatography grade copper tubing and new Swagelok fittings.

The following are the gas purifiers available from Varian.

Purifier	Part Number
High capacity oxygen trap	03-949770-02
Indicating oxygen trap	88-501019-00
Combined hydrocarbon and moisture trap	03-949862-00
Indicating moisture trap	01-900007-00

- **Injector Maintenance The** injector is the component of the Gas Chromatograph that requires the most frequent maintenance. This is due to the fact that the sample is deposited in the injector, thus leading to potential contamination and build up of non volatile deposits. The most frequent injector maintenance is septum replacement. In addition insert replacement and injector cleaning are very common. As septum replacement is common to all liquid injectors on the 3800 this procedure is presented first, followed by specific maintenance procedures for the individual injectors.
- Septum Replacement The injector septum is an expendable part and must be replaced on a routine basis. The frequency of replacement depends on the number of injections and whether the injections are by hand or with an autosampler. In general the septum should be replaced every 50 - 100 injections or when symptoms of a septum leak are seen. These symptoms include changing retention times, reduced detector response or a drop in column head pressure. The latter symptom is not always valid as some injectors, such as the 1079, are pressure controlled. With a pressure controlled injector, the column head pressure will remain constant even if a leak is present.

Use the following procedure to change a septum on any Varian injector:

1	Cool the column oven and injector oven to 50°C. This ensures safe handling of injector parts and protects the column from elevated temperatures while it is exposed to air.	
2	Turn off the carrier gas supply to the injector.	
3	Remove the injector nut by turning it counterclockwise.	
4	Remove the old septum using tweezers or a septum pick (P/N 72-000084-00). It is best not to handle any internal parts of the injector.	
5	5 Install the new septum, again using tweezers to avoid contaminating the injector.	
6	Replace the injector nut and tighten it finger-tight until you feel resistance from the septum, then tighten an extra 1/4 turn. Turn on the carrier gas supply.	

On occasion to save time you may want to change a septum while the injector is hot. Use an injector wrench (P/N 03-908423-00) to remove and reinstall the injector nut. In all instances the column oven should be cool before removing the injector nut.

#### 1079 Injector

The 1079 is a universal capillary injector that can be operated in several modes. These modes include split, splitless, on-column and large volume injection. Typically, to change from one mode of operation to another involves changing the injector insert. The insert should also be cleaned or replaced on a routine basis. This is especially important when dirty samples are being analyzed.

After prolonged use, the 1079 Universal Capillary Injector glass insert may need to be removed either:

- To clean the current glass insert for reinstallation in the 1079 Injector, or
- To replace the current glass insert with a new insert.

Refer to the figure below when removing and/or replacing the glass insert.

- 1 Injector Nut
- 2 Septum
- 3 Septum Purge
- 4 Septum Support
- 5 Graphite Ferrule
- 6 Carrier Gas Inlet
- 7 Split Vent
- 8 Glass Insert 9 Fused Silica
- 9 Fused Silica Column
- 10 Capillary Column Nut
- 11 Vespel® Ferrule
- 12 Point of Injection

#### **Tools Required**

- Tweezers or septum pick (P/N 72-000084-00)
- Injector nut wrench (P/N 03-908423-00)
- Flat-blade screwdriver (short handle)
- Clean laboratory tissue
- Graphite ferrules (P/N 03-925342-02)
- Insert/ferrule positioning tool (P/N 03-925385-00)



Figure 3 1079 Injector - Cross-Sectional View

Remove the Glass Insert Follow these steps to remove the glass insert from the 1079 Injector.

**1** Use the injector nut wrench to remove the injector nut (Item 1). Place the nut on a clean surface (e.g., clean tissue).



# The injector nut may be hot. Lower the injector temperature to 50°C and permit the injector nut to cool before proceeding.

**Note:** If the GC is equipped with an 8200 AutoSampler, to access the injector nut, push the carrousel release button (back left) and swing the carrousel out.

2	With tweezers or septum pick, lift the edge of the septum (Item 2). Remove the septum.
	<b>Note:</b> Replace the injector septum each time the glass insert is replaced.
3	Use a clean flat-blade screwdriver to unscrew the septum support nut (Item 4) until it is loose.
4	Remove the septum support with the tweezers or septum pick.
	<b>Note:</b> Typically, when the septum support is removed, the insert and ferrule remain in the septum support. If the ferrule and insert are in the injector body after the septum support nut is removed, use the tweezers to grasp the top of the insert and lift it from the injector body.
5	Use a laboratory tissue to grasp the glass insert and remove it from the septum support nut.
6	To remove the graphite ferrule from the glass insert, use clean lab tissues to hold the graphite ferrule (Item 5) and the glass insert. Gently turn the glass insert while you pull off the graphite ferrule. <b>Note:</b> The glass inserts can be cleaned and reused. Unless the graphite ferrule is obviously damaged, it can be reused as well. However, replace the 5 mm graphite ferrule after the glass insert
	has been changed three or four times.



**Clean the Glass Insert** Glass inserts must be clean and free from sample residue and particulate matter (such as bits of septum rubber or graphite). Follow these steps to clean the glass insert in the 1079 Injector.

1 Remove the glass insert. It is safest to cool the injector and column ovens to 50°C before removing the insert.



WARNING:

**BURN HAZARD** 

Use care when removing inserts from the injector. Inserts can be at high temperatures and are likely hot. Place hot inserts on a clean glass or metal surface only.

- **2** To clean glass inserts, use one of the following procedures (the choice of cleaning procedure depends upon the nature of samples injected):
  - Rinse the inserts with solvent or soak the inserts in hot acid.
  - Heat them in a glass annealing oven (to 500°C) or pass the inserts through the flame of a Bunsen burner.
  - Wash in a 1:1:1 mixture of methanol:methylene chloride:hexane in an ultrasonic cleaner for 30-60 min, then dry the inserts in an oven.

**Note:** For the 2 mm glass wool packed glass insert, remove the glass wool by blowing compressed gas in the end of the insert. Clean the insert following one of the procedures listed above. Repack the insert with deactivated glass wool (10-20 mg). Leave ~1.5 cm of the bottom of the glass insert unpacked. The capillary column will be inserted in this empty space when re-installed in the injector body.

**Note:** Rinsing the glass inserts with strong acids or bases or heating to a high temperature will remove the deactivation coating on the glass inserts. Rinsing the glass inserts with solvents or mixed solvents will not remove the deactivation coating.

#### **Deactivate** Follow these steps to deactivate the glass insert in the 1079 Injector.

#### Deactivate the Glass Insert

**Note:** This procedure can be used on all glass inserts except inserts packed with 10% OV-101 on Chromosorb W-HP (P/N 03-918956-00). Up to three inserts can be deactivated at a time using this procedure.

1	In a 10 mL glass graduated cylinder, add 0.5-1 mL of dimethyldichlorosilane. Fill to 10 mL with isooctane, hexane or toluene.
2	Cover the top of the graduated cylinder with aluminum foil and place the cylinder in an ultrasonic bath. Sonicate for 30 sec to mix the solution.
3	Add up to 3 inserts to the solution.

*Note:* Deactivate glass inserts only after they have been thoroughly cleaned using the above procedure.

4	Sonicate the inserts in the cylinder for 10 min. Rinse the inserts 3 times with 10 mL isooctane, hexane or toluene. Each rinse should include 2-3 min of sonication.
5	Add 10 mL of methanol and sonicate for 2-3 min. Decant the methanol and repeat the methanol rinse step.
6	Decant the methanol. Transfer the deactivated inserts to a small clean glass beaker. Cover the beaker with aluminum foil and bake at 200°C for 1 hour.
7	After the inserts cool to room temperature, store them in a clean screw cap glass vial or the original packaging.

To clean the injector body, proceed as follows:

- Set the column oven temperature to 45°C and the 1079 injector temperature to 50°C. Wait for the zones to reach their set temperatures and then turn the column oven off by pressing the "TURN OVEN OFF" softkey located in the Column Oven section of the method.
- 2. Use the 5/16" open-end wrench to loosen the capillary column nut.
- 3. By hand, carefully withdraw the fused silica column and nut from the injector assembly. Set the column nut and column end on the floor of the GC oven.





Use proper eye and skin protection. Methanol and acetone are toxic and flammable chemicals. Exercise appropriate precautions when these chemicals are used.

- 4. Remove the injector nut, septum, and septum support, with ferrule and insert attached.
- 5. Moisten a cotton swab an a stick with methanol and gently swab center of the injector body.
- 6. Moisten a cotton swab on a stick with iso-octane and gently swab the center of the injector body.
- 7. Examine the end of the fused silica column to ensure that it has not been damaged. Slide the end of the fused silica column into the opening of the injector.
- 8. Gently twist the column nut in the opposite direction used to tighten the nut. Place the column nut on the end of the injector and tighten the nut. This procedure minimizes twisting the column.
- 9. Use the 5/16" open-end wrench and tighten with 1/6-turn to secure the column in place.
- 10. Replace the septum support, insert, ferrule, septum, and injector nut.
- 11. To check whether the injector is leak-tight, connect a flow meter to the split vent. Set the Relay to the split mode. If the flow meter indicates a flow less than previously measured, the injector has a leak.
- 12. Turn the column oven back on by pressing the "TURN OVEN ON" softkey in the column oven section of the method.

#### **1041 Injector**

Hardware<br/>Replacement<br/>and CleaningRoutine cleaning and maintenance of the 1041 Universal Injector system<br/>includes septum replacement and column conditioning. Pressure testing<br/>and leak checking procedures are included to assure proper functioning of<br/>the system.

Septum Replacement Septum replacement represents the major part of routine chromatographic maintenance. Septum damage from the needle penetrations can be avoided by injecting into the same hole and not using syringes with needles having burrs or bends at the tip which cut the septum.



The injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands. Allow sufficient time for the injector nut and surrounding assemblies to cool before continuing with this procedure.



Handling a septum with bare fingers may result in column contamination. Use tweezers, finger cots, or gloves when installing a new septum.

1	Be sure the injector nut is cool. Unscrew the injector nut and place on a clean, uncontaminated surface.		
2	2 Using tweezers, remove the septum, taking care not to scratch the internal surfaces of the injector.		
3	Using tweezers, place a new high temperature septum in the injector. If the septum is TFE or FEP coated put that side down.		
4	Replace the injector nut and tighten until resistance is felt, then tighten an extra 1/2 to full turn.		



Figure 4 1041 Injector Assembly

Needle/<br/>SyringeFor normal use, rinse the needle/syringe by slowly drawing up and quickly<br/>expelling solvent or the next sample to be used. Repeat the process several<br/>times.

**Note:** When rinsing with the next sample, DO NOT expel sample back into the sample container.

Leak	Leak checking methods used with the 1041 Universal Injector are presented
Checking	in order of overall sensitivity and performance. When leak checking the
•	injector only, be sure to use the appropriate procedure.

1	Remove the test column and plug the injector outlet with a 1/16" no- hole ferrule (P/N 28-694503-00). Remove the injector nut, install a new septum, then replace injector nut and turn nut clockwise until it comes to a stop.
2	Pressurize the injector to 60 psig with carrier gas. Turn off the carrier gas flow at the supply.
3	A pressure drop less than or equal to 1/2 psig in 15 minutes is acceptable. Locate leaks with an appropriate leak detector. <b>Note:</b> Any changes in temperature while performing this test may result in false readings due to expansion/contraction of the gas with temperature.



Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

4	An alternate way to leak check the injector only is to have the column
	installed in the injector end and the opposite column end sealed with
	a flame or appropriate fitting.

Gas Leak A convenient and non-contaminating way to leak test fittings and connections after installation or hardware replacement procedures is to direct a small jet of gas (butane from a disposable lighter recommended for flame ionization detectors) at the point to be tested, then use the detector, at maximum sensitivity, to detect leakage of gas into the system. Use normal column flow, a cool oven, and an operational detector. If a peak is detected in excess of the magnitude given in the following table, repair the leak.

Detector	Gas	Attenuation, Range	Maximum Peak Size
FID	Butane	16 x 10 <sup>-12</sup>	30%
ECD	Dichloromethane	2 x 1	50%
PFPD, Sulfur Mode	Natural Gas containing methanethiol	2 x 10 <sup>-10</sup>	50%
TSD	Dichloromethane	2 x 10 <sup>-12</sup>	2%

#### Visual Leak Checking

The least preferred method of leak checking is to place a drop of pure solvent (isopropyl alcohol recommended) on the suspected fitting or connection and look for bubbles.



Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

Syringe Leak In some instances, non-reproducible chromatographic responses can be attributed to a worn and/or leaky syringe. The syringe must be leak tight. To check the syringe, insert the needle in an injector operating at 20 to 30 psig with a no-hole ferrule. Place a drop of solvent at suspect leak locations and look for bubbles.

#### 1061 Injector

The 1061 is mechanically similar to the 1041 except that it uses a glass insert for flash vaporization injection. Follow the same maintenance procedures for the 1061 that are outlined above for the 1041, except in the case of changing or cleaning glass inserts.

Replacing the 1061 Glass Insert After prolonged use, the 1061 glass insert may need to be removed for cleaning purposes or to be replaced with a new insert. Note that there are two different glass inserts, one for use with large-bore (0.53 mm ID) capillary columns and one for use with packed columns. These inserts are not interchangeable.

- Large-bore glass insert, Part Number: 03-918339-00
- Packed column glass insert, Part Number: 37-000813-00

.3

Refer to the figure below when replacing the glass insert.

These tools are needed to replace the glass insert:

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

1 Septum Nut

- 2 Automatic Start Switch
- 3 Septum, 10 mm
- 4 Injector Insert
- 5 Injector Body



Figure 5 1061 Injector Assembly

**Remove the** Follow these steps to remove the glass insert from the 1061 Injector. **Glass Insert** 

**1** Use the injector nut wrench to remove the injector nut (Item 1). Place the nut on a clean surface (e.g., clean tissue).



#### The injector nut may be hot. Lower the injector temperature to 50°C and permit the injector nut to cool before proceeding.

**Note:** If the GC is equipped with an 8200 AutoSampler, push the carrousel release button and swing the carrousel out to access the injector nut.

2	With tweezers or a septum pick, lift the edge of the septum. Remove the septum. <b>Note:</b> Replace the injector septum each time the glass insert is replaced.
3	Use tweezers to grasp the glass insert and remove it from the injector. <b>Note:</b> The glass inserts can be cleaned and reused.

**Replace the** Follow these steps to replace the glass insert in the 1061 Injector. **Glass Insert** 

1	Pick up the new insert with tweezers and place it in the injector carefully.
2	Use tweezers to place a new septum in the injector. <b>Note:</b> If the septum has a Teflon face, place the Teflon face toward (down) the column.
3	<ul> <li>Place the injector nut on the injector and tighten by hand until you feel some resistance, then tighten an extra 1/2 to full turn using the injector nut wrench.</li> <li><b>Note:</b> After the injector nut has been replaced check the head pressure (EFC status on display or manual pressure gauge on pneumatics panel) increases to its normal value. If this does not happen there is an indication of a leak.</li> </ul>
4	Condition the insert by setting the 1061 Injector temperature to 300°C and allowing the system condition for 30 minutes.

**Clean the Glass inserts** must be clean and free from sample residue and particulate **matter** (such as bits of septum rubber or graphite). Follow these steps to clean the glass insert of the 1061 Injector.

1 Remove the glass insert. It is safest to cool the injector to 50°C before removing the insert.



#### Use care when removing inserts from the injector. Inserts can be at high temperatures and are likely hot. Place hot inserts on a clean glass or metal surface only.

- 2 To clean glass inserts, use one of the following procedures (the choice of cleaning procedure depends upon the nature of samples injected):
  - Rinsing the inserts with solvent or soaking the inserts in hot acid.
  - Heating them in a glass annealing oven (to 500°C) or passing the inserts through the flame of a Bunsen burner.
  - Wash in a 1:1:1 mixture of methanol:methylene chloride:hexane in an ultrasonic cleaner for 30-60 min, then dry the inserts in an oven.

Deactivate	Follow these steps to deactivate the glass insert in the 1061 Injector.
the glass	
insert	

1	In a 10 mL glass graduated cylinder, add 0.5-1 mL of dimethyldichlorosilane. Fill to 10 mL with isooctane, hexane or toluene.
2	Cover the top of the graduated cylinder with aluminum foil and place the cylinder in an ultrasonic bath. Sonicate for 30 sec to mix the solution.
3	Add up to 2 inserts to the solution.
	<b>Note:</b> Deactivate glass inserts only after they have been thoroughly cleaned using the above procedure.
4	Sonicate the inserts in the cylinder for 10 min. Rinse the inserts 3 times with 10 mL isooctane, hexane or toluene. Each rinse should include 2-3 min of sonication.
5	Add 10 mL of methanol and sonicate for 2-3 min. Decant the methanol and repeat the methanol rinse step.
6	Decant the methanol. Transfer the deactivated inserts to a small clean glass beaker. Cover the beaker with aluminum foil and bake at 200°C for 1 hour.
7	After the inserts cool to room temperature, store them in a clean screw cap glass vial or the original packaging.

#### **Flame Ionization Detector**

When the performance of the FID has degraded you may want to replace the flame tip ferrule, or clean the deposits from the internal parts, including the flame tip.

Clean theIf the FID noise increases with frequent spikes, the internal parts may beFlame Tipfouled and need to be cleaned.

Flame Tip and Internal Parts

To clean the flame tip and the internal parts of the FID, proceed as follows:

**Note:** Always handle detector components with tweezers to avoid contamination.





Use proper eye and skin protection. Methanol and acetone are toxic and flammable chemicals. Exercise appropriate care when you use and dispose of these substances.

1	Remove the detector and internal parts.
2	Scrape the deposits from the bore of the collector tube, the insulator, and the metal part of the flame tip with emery cloth (e.g., $SiO_2$ ).
3	If the flame tip is plugged, insert a wire through the flame tip orifice to clear it.
4	If you have access to an ultrasonic cleaner, clean the collector, insulator, and flame tip with distilled water.
5	Rinse the cleaned components with methanol or acetone and air dry.
6	Wipe the detector tower with acetone.
7	Clean the probe arms with methanol and air dry or dry in an air oven (maximum drying temperature of 150°C).
8	Reinstall the detector. To prevent leaks, use a NEW aluminum seal washer each time you install the detector tower.



#### Figure 6 Flame Ionization Detector Assembly

Replace theAfter a period of prolonged use, the flame-tip ferrule may deteriorate and<br/>crack. As a consequence, a leak may develop around the base of the flame<br/>tip assembly that causes unstable baseline noise and reduced sensitivity. In<br/>this situation, or when the detector is to be operated at temperatures above<br/>350°C, you must replace the ferrule. Be sure to select the proper ferrule for<br/>the application. The Vespel/graphite ferrule has a maximum temperature limit<br/>of 350°C.

#### **Thermionic Specific Detector**

Cleaning/ When the TSD exhibits low sensitivity or high background noise, Cleaning is indicated. Visual inspection may also indicate a need for cleaning. The ceramic bead, collector, or flame tip are all parts that are deposit prone, depending on the type of samples being used. Clean these parts as indicated in the following paragraphs.

**Note:** Always handle detector components with tweezers to avoid contamination.

**Bead Cleaning** Non-volatile deposits can be removed from the bead by gently scraping the bead surface with a sharp tool or abrasive paper. The bead should be supported while being cleaned to prevent bending the lead and cracking the ceramic coating on the leads.

Flame Tip and Internal Parts Cleaning Follow the steps below to clean the flame tip and internal parts.



Use proper eye and skin protection. Methanol and acetone are toxic and flammable chemicals. Exercise appropriate precautions when these chemicals are used.

1	Remove the detector and internal parts. Handle internal parts with tweezers and place on a clean, uncontaminated surface.
2	Clean deposits from the surface of the collector with water or abrasive paper.
3	Remove deposits which form on top of the flame tip by scraping. If flame tip is plugged, clear by inserting wire (approximately 0.012 inch diameter) into flame tip orifice.
4	Flush cleaned components with methanol and air dry.

#### Ferrule Replacement

If a leak develops around the base of the flame tip assembly due to a deteriorated ferrule or if the detector is to be operated at temperatures above 350°C, and results in a crack, the ferrule must be replaced. A leak at the ferrule may cause noise, instability, and loss of sensitivity. Be sure to select the proper ferrule for the application. The Vespel ferrule has a maximum temperature limit of 350°C.



Figure 7 Thermionic Specific Detector Assembly

#### **Electron Capture Detector**

Chemical contaminants may deposit on the components of the detector assembly as the ECD is operated. In many cases, you can remove contaminants deposited on the radioactive foil or cell ceramic insulators by heating.

Refer to the Radiation Safety Manual (P/N 03-913999-00) for detector repair, radioactive decontamination, foil replacement, and leak-test information.

**Note:** Although all heated zones on the 3800 have a potential upper temperature of 450°C, Varian recommends that you do not operate the ECD above 420°C. (The radioactive source temperature is less than 400°C even when the detector set temperature is 450°C.)

Thermal Use  $N_2$  or  $Ar/CH_4$  as the carrier gas to thermally clean the ECD. Refer to Table 2 on page 39 for flow rates. With Carrier

1 With carrier gas and makeup gas flowing through the ECD cell, increase the DETECTOR TEMP to 400°C.



Gas

Do not attempt to chemically clean the source. Submerging the cell in liquid or running a liquid through the cell will cause radioactivity to migrate, contaminate the exterior of the cell, and damage the insulators. The cleaning of the radioactive ECD source can by performed only by an NRC licensee. Cleaning of the ECD source by persons other than registered NRC licensees is strictly forbidden by law.



AVOID FOIL DAMAGE. The carrier gas must be flowing through the detector cell before setting the DETECTOR TEMP above room temperature. Failure to do so may damage the radioactive foil, which is not covered by warranty.

2	Monitor the output signal while the detector heats to its maximum temperature. The signal will increase initially, then gradually decrease as chemical contaminants vaporize from the detector surfaces.
3	Allow the DETECTOR TEMP to remain at 400°C until the signal reaches a stable level. A stable signal indicates that the foil has been cleaned as much as it can be by this method.
4	Set the detector at normal operating temperatures and flow carrier gas through the detector overnight. Check and readjust the Contact Potential setting if necessary.
5	If the performance of the detector is not restored by these cleaning methods, refer to the Radiation Safety Manual (P/N 03-913999-00) for information on replacement of the foil.

#### **Thermal Conductivity Detector**

The Thermal Conductivity Detector is not user serviceable and has no replacement parts. To condition the TCD, set the detector temperature to 350°C and allow it condition for 4 hours or overnight. The filament temperature should be set to zero during this procedure.

#### **Pulsed Flame Photometric Detector**

For PFPD maintenance procedures see the PFPD Operator's Manual, P/N 03-914649-00.

#### **Micro Thermal Conductivity Detector**

The Micro-TCD is not user serviceable and has no replacement parts.
# **Parts and Supplies**

To Order Parts and Supplies ...

- In the USA and Canada, call 1-800-926-3000.
- In Europe, call your local Country office.
- In the rest of the world, call your local Varian representative
- Or, contact Varian on the Internet at http://www.varian.com

## Injectors

Options

#### 1079 Injector

Description	Part Number
Ceramic scoring wafer (for cutting fused silica columns)	01-900158-00
Capillary column nut	03-949551-00
Ferrule, no hole (package of 10)	28-694503-01
Septum pick	72-000084-00
Graphite ferrule for insert, 5 mm (package of 2)	03-925342-02
Open 0.5 mm ID insert	03-925331-00
SPME open insert 0.8 mm ID	03-925330-00
Splitless insert, 2 mm ID	03-918466-00
Splitless insert, packed with deactivated glass wool, 2 mm ID	03-925350-00
Split insert, fritted, 3.4 mm ID	03-918464-01
Split insert, open, 3.4 mm ID	03-918464-00
Split insert, packed with 10% OV-101 on Chromosorb W-HP	03-918956-00
Septa (package of 25), 11 mm	00-997630-02
Septum support	03-918676-00
Insert/Ferrule positioning tool	03-925385-00

# Capillary Column Quick Connect Kit 03-925751-91 3800 Field kit, LCO2 Cryo Kit 03-925513-91 3800 Field kit, LN2 Cryo Kit 03-925506-91

#### 1041/1061 Injector

Packed Column Adapter Kit for 1041, 1/8" SS columns	03-925588-91
Packed Column Adapter Kit for 1061, 1/8" SS columns	03-925588-92
Note that these kits contain injector and detector adapters	

Packed Column Adapter Kit for 1/4" glass columns	03-925586-91
Packed Column Adapter Kit for 1/4" SS columns	03-925586-93
1061Glass insert for 0.53mm ID columns	03-918339-00
1061 Glass insert for Packed Columns	37-000813-00
Septa, package of 25, 10 mm	00-997628-02

### Detectors

#### **Flame Ionization Detector**

Flame Tip Assembly (0.02" ID)	02-001938-00
Flame Tip Assembly (0.01" ID)	02-001875-00
Aluminum Crunch Washer (25)	15-003347-01

# **Thermionic Specific Detector**

TSD Bead Probe	03-906074-00
TSD Bead Probe, preconditioned	03-906074-01
TSD Sulfate Bead*, preconditioned	03-925356-01
*Note that the sulfate bead is recommended for analysis of ester is used as the solvent.	s or when an ester

#### **Electron Capture Detector**

ECD Cell with <sup>63</sup> Ni Foil	02-001972-01
ECD Wrench	72-000081-00

Thermal Cor	nductivity Detector	
	TCD Make-up Gas Option	03-925605-01
Pulsed Flam	e Photometric Detector	
	Aluminum Crunch Washer	15-003347-00
	Combustor, Quartz (2mm ID)	03-925176-00
	Combustor, Quartz (3 mm ID)	03-925177-00
	Filter Assembly, Sulfur	03-925151-01
	Filter Assembly, Phosphorus	03-925151-02
	Ignitor Assembly	03-925160-00
	Combustor support, 2 mm	03-925178-00
	Combustor support, 3 mm	03-925179-00
	Combustor support seal	03-925138-00
Micro-TCD		
	Ferrule, 0.25 mm ID	CP85888

Ferrule, 0.25 mm ID	CP85888
Ferrule, 0.4 mm ID	CP85889
Ferrule, 0.5 mm ID	CP470100
Ferrule, 0.8 mm ID	CP470101