CP-3800

GAS CHROMATOGRAPH

QUALITY SYSTEM
ISO 9001
CERTIFIED

P/N 03-914689-00
Rev 3

VARIAN

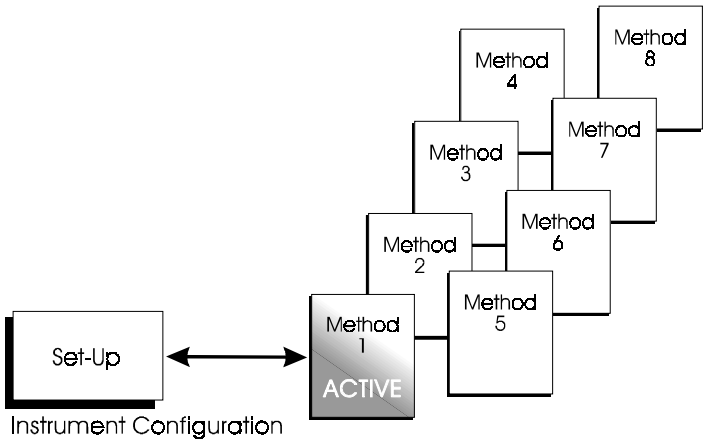
TABLE OF CONTENTS

This guide is intended for the novice and experienced user. You will find this to be an invaluable reference for setting up your GC, programming the system, and finding the most frequently needed part numbers.

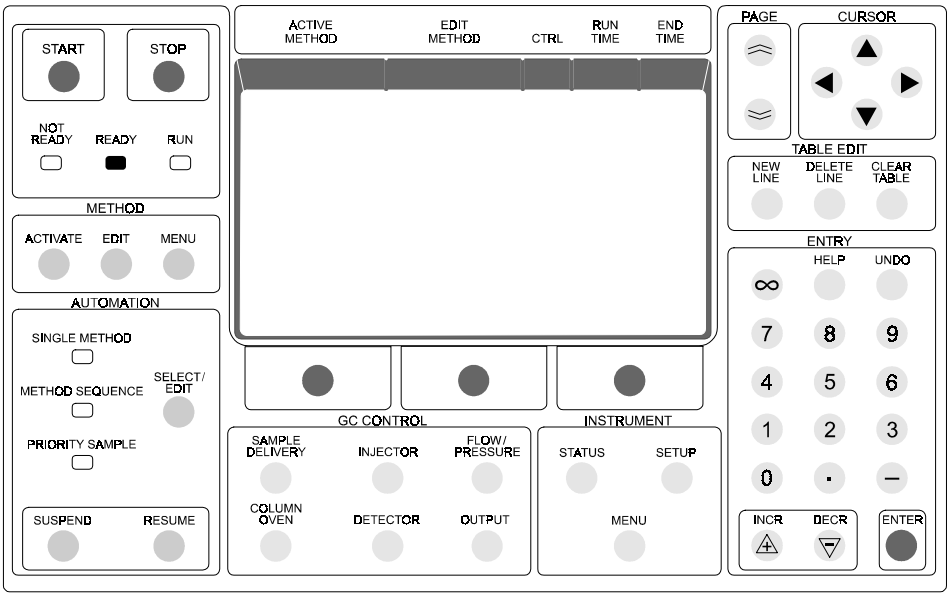
Keep this guide near your GC for convenient reference.

I. GC OPERATION

Overview



The CP-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. There are 8 available methods, one of which must be active at any given time. For more detailed information see the Operator's Manual, **CP-3800 Keyboard and Display**.

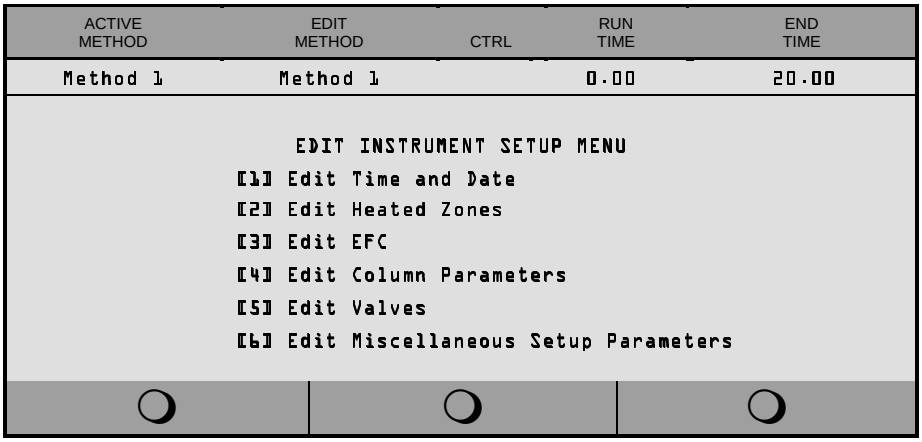


CP-3800 Keyboard

Instrument Setup

Action
1. Press **SETUP**.
2. Choose View Setup/Edit Setup using cursor keys.
3. Press **ENTER**.

"View Setup" allows you to check the current Instrument Configuration. Choose "Edit Setup" if you want to change the Instrument Configuration.



Use CURSOR keys to highlight section ...	**Press ENTER to access section and modify ...**
TIME AND DATE	■ Month / day / year ■ Time / hour: min: sec
HEATED ZONES	■ Device installed in each heated zone ■ Temperature limit ■ Coolant type (Column oven, zones 1-3 only)
EFC	■ Outlet pressure – atm/vacuum ■ Display units – psi, kPa, bar ■ Minimum flow – gas saver ■ Make-up gas type
COLUMN PARAMETERS	■ Length (m) ■ ID (μm) ■ Carrier gas type (He, H_2, N_2)
VALVES	■ Valve Numbers (1-7) ■ Valve Type (22 choices)
MISCELLANEOUS	■ Ready-in Contact State ■ FID Flame-out Enable ■ Micro-TCD Filament Resistance

Building a Method

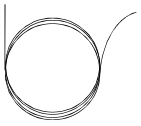
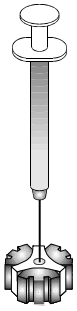
Follow these steps to build or edit a GC Method.

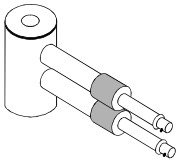
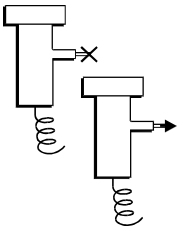
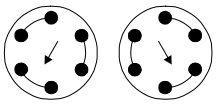
Action
1. Press **EDIT** from the Method section of the keypad.
2. Use the INCR or DECR keys to choose desired method.
3. Press **ENTER**.

Typical parameters for a GC method are:

- Column, Injector and Detector temperatures
- Flow or Pressure program, if EFC is installed
- Detector range and autozero
- Valve program, if performing a manual pneumatics splitless injection, or rotating a sampling or switching valve
- 8200 AutoSampler

Follow the method sections below to build your GC method. You can advance to any section by pressing the appropriate section key.

The Following Method Section ...	Will allow you to modify these method parameters ...
COLUMN OVEN 	■ Column temperature (isothermal or programmable) ■ Stabilization time ■ Coolant on/off ■ Enable coolant on/off temperature ■ Coolant timeout
INJECTOR 	■ Injector temperature (isothermal and programmable) ■ Coolant on/off ■ Enable coolant temperature ■ Coolant timeout ■ Split state (EFC only) ■ Split ratio (EFC only)
FLOW/PRESSURE (EFC Only)	■ Flow + Pressure (1079 + Valving) ■ Flow (1041/1061)

The Following Method Section ...	Will allow you to modify these method parameters ...
DETECTOR 	■ Detector temperature ■ Range, and autozero (time programmable) ■ Detector time constant ■ TCD filament temperature and polarity (time programmable) ■ PFPD → PMT voltage, gate width + delay, trigger level ■ TSD bead current and power (time programmable) ■ ECD contact potential and cell current ■ Detector flow (EFC only)
SAMPLE DELIVERY/ VALVE TABLE  	■ 8200 Sampling Parameters ■ Timed programmed events for splitless injections (manual 1079 only), or switching valves ■ For GCs with valves, consult your custom plumbing diagram for programming Events for a typical split and splitless injection are as follows: **Time** **Split Valve** *Split Mode* 0.00 min On (Split) *Splitless Mode* 0.00 min Off (Splitless) 1.00 min On (Split)

Automation

- Selects Automation Mode
- Method Automation Parameters
- Priority Sample

Activating the GC Method

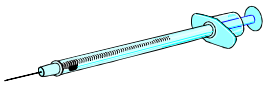
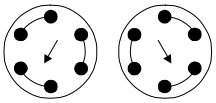
Action
1. Press **ACTIVATE** from the Method section.
2. Use the INCR or DECR keys to choose desired method.
3. Press the "ACTIVATE NOW" softkey.

NOTE: If you edit the active method, you must re-activate it before running an analysis.

Making a Single Injection

Use the following procedure if performing a single injection. If you are using the Star Chromatography Workstation, refer to the instrument's Operator's Manual.

Confirm that the GC is in the Ready state. The amber "Ready" light should be illuminated.

If you are making an injection with a ...	Then ...	Result
Syringe 	Inject the sample into the injector.	The GC method will automatically start.
Gas or Liquid Sampling Valve using an air actuator 	Confirm the sampling valve is in the fill position and the loop is loaded with sample. Press **START**	The gas or liquid sampling valve will rotate to the inject position and the GC method will start.

Do You Need HELP?

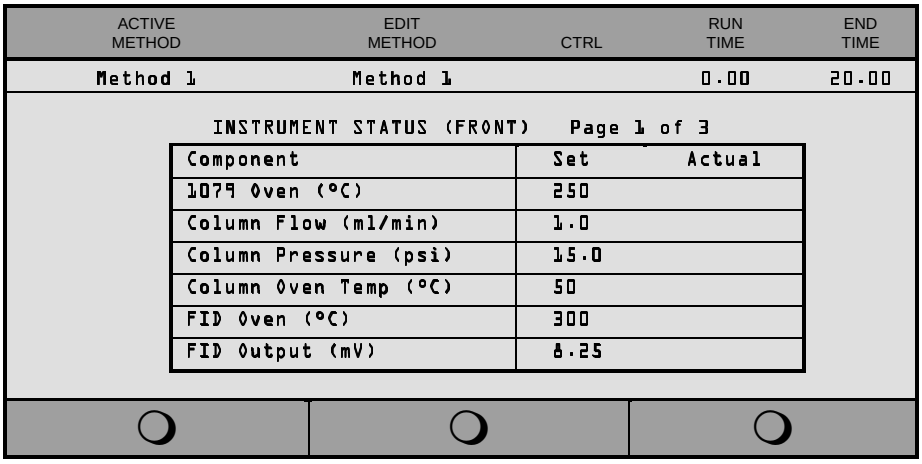
When the cursor moves from field to field on the display, the prompt line beneath the method indicates the parameter range.

Pressing HELP gives the user an explanation for the specific parameter.

GC Status

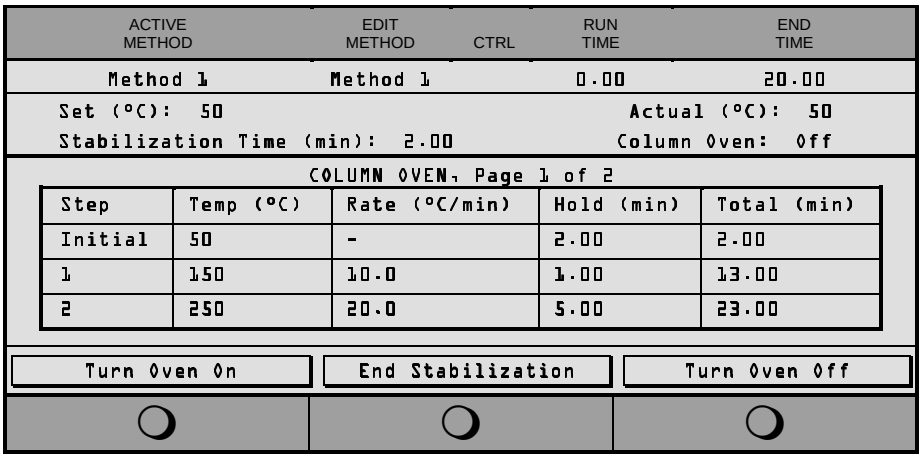
The Instrument STATUS key allows the user to view the current status of the various components of the CP-3800 instrument. Note that the current status of individual components can also be viewed by pressing the relevant key in the GC CONTROL keyboard section.

The primary status information provided 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 CP-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 as the values change during the run at a rate of once per second. If a component is not ready or faulted, this will be indicated on the STATUS screen. Note that the status screens are presented in location order, i.e., Front, Middle and Rear.



GC Control

When one of the six GC Control keys is pressed, the user is presented with a split display. The status information reflecting the current status of the CP-3800 appears above the bold line on the display. The information beneath the bold line is the method parameters of the EDIT METHOD. Note that the EDIT METHOD and the ACTIVE METHOD may be different.



II. HARDWARE INSTALLATION & SETUP

Column Installation

Follow these steps to install the capillary column and set gas flow rates:

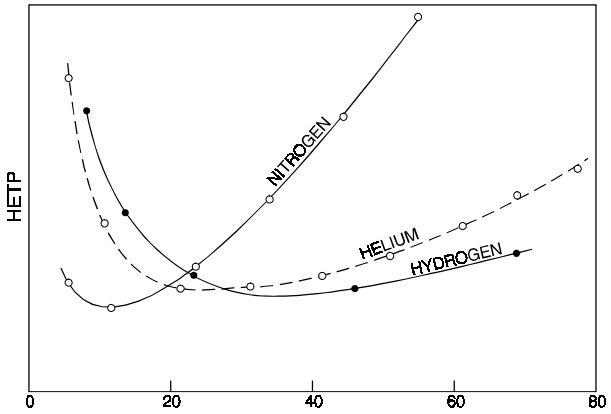
1. Cool all heated zones and replace depleted oxygen and moisture traps.
2. Replace critical injector inserts and septa.
3. Cut 2 cm from each column end.
4. Thread the nut and ferrule over the column on both ends. The ferrule should be installed with the taper end into the nut.
5. Cut 2 cm from each column end to remove ferrule fragments.
6. Mount the capillary column in the oven.
7. Install the column 7.5 cm into the 1079 injector, measured from the back of the nut. *The 1041 and 1061 do not require measuring. They are installed all the way up into the injector.*
8. Set the approximate column head pressure.
9. Set the split ratio and septum purge flows *(1079 only).*
10. Connect the column to the detector at the appropriate distance *(see Page 18).*
11. Check for leaks using a leak detector. *Do not use soaps or liquid-based leak detectors.*
12. Set make-up and detector gas flow rates *(see Page 10).*
13. Set injector and detector temperatures. *Do not exceed the column's* T_{max}.
14. Condition the column at its recommended conditioning temperature for two hours.
15. Run test mixtures to confirm proper installation and column performance.
16. Calibrate instrument and inject samples.

Optimum Capillary GC Carrier Flow Rate and Velocity

After conditioning your column, the carrier gas flow rate should be set for optimum separation of sample components. Since the flow rate is dependent on column temperature in a pressure regulated system, it is important to set the carrier gas at the same column temperature for a given analysis. For convenience, the carrier gas is often set at the initial temperature of the analysis. For a slightly faster analysis and improved separation, set the optimum flow rate at the maximum temperature of the analysis. For critical or hard to separate peak pairs in the chromatogram, set the optimum linear velocity at the oven temperature where they elute.

If you have Electronic Flow Control installed in your GC, setting the column flows is as simple as entering the column dimensions and carrier gas type in SETUP and building the appropriate flow or pressure program in the Method.

If you don't have EFC installed, inject 5 µL of a non-retained gaseous substance compatible with the detector *(Page 18)*. Calculate the column velocity, then flow rate and split ratio, if applicable, using the equations below.



Column Velocity, μ (cm/sec) =

$$\frac{\text{Column length (cm)}}{\text{Unretained peak time (sec)}}$$

Column Flow Rate (mL/min) =

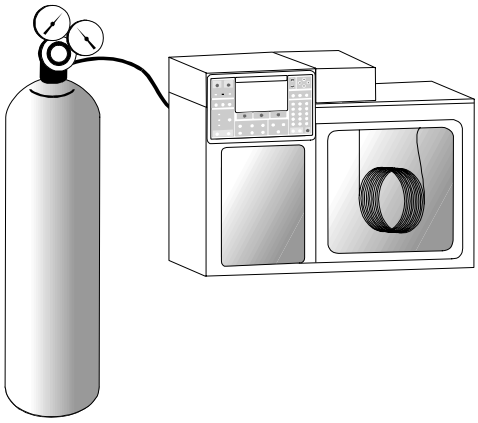
$$\mu\left(\frac{\text{cm}}{\text{sec}}\right) \times \pi \times r_{col}^{2}\left(\text{cm}^{2}\right) \times 60\left(\frac{\text{sec}}{\text{min}}\right)$$

Split Ratio =

$$\frac{\text{Split vent flow}\left(\text{mL/min}\right)}{\text{Column flow rate}\left(\text{mL/min}\right)}$$

Optimum Velocities and Flow Rates for Capillary Columns

	Column ID (microns)					
	250		320		530	
Carrier Gas	mL/min	cm/sec	mL/min	cm/sec	mL/min	cm/sec
He	1.3	45	1.7	35	2.8	21
H_2	1.6	55	2.1	43	3.4	26
N_2	0.4	14	0.5	11	0.9	7



Detector, Carrier, and Make-up Gas Flow Rates and Gas Types

Use the following guide for determining the appropriate gases for your GC system and setting detector, carrier gas, and make-up flow rates.

Detector	Flow Rates, mL/min				Gas Type	
	Carrier + Make-up	Hydrogen*	Air 1	Air 2	Carrier Gas	Make-up Gas
Micro-TCD	Max. ≤ 5 mL/min	–	–	–	H_2 or He	None
TCD	30/30 (reference gas)	–	–	–	He, N_2, H_2, Ar	Same as carrier
FID (0.020" flame tip)	30	30	300	–	He, H_2, N_2	He, N_2
ECD	30	–	–	–	N_2, Ar/CH_4 (He, H_2)**	N_2, Ar/CH_4
TSD	30	4.0	175	–	He, N_2	He, N_2
PFPD	Element specific – refer to PFPD Operator's Manual				He, H_2, N_2	–
Inlet Cylinder Pressure	80 psi ~560 kPa	40 psi ~280 kPa	60 psi ~420 kPa	60 psi ~420 kPa		
Purity (%)	99.999	99.999	Zero Grade	Zero Grade		

* Total H_2 flow including any used for carrier or makeup gas.

** He may be used only when capillary column flow rates are <10 mL/min.

III. RECOMMENDED PARTS & SUPPLIES

Gas Filters

Carrier and detector filters should always be installed to further clean high purity gases, reduce detector noise, and protect the chromatography system from potential contamination. The following filters are recommended for GC systems:

Filter	Description	Part Number
Moisture Gas Filter	■ Installed between gas tank and GC inlet ■ Molecular sieve ■ Removes water vapor ■ Filter should be changed when indicator shows filter is spent	CP17971
Charcoal Gas Filter	■ Installed between gas tank and GC inlet ■ Activated charcoal ■ Removes organic contaminants ■ Filter should be changed when indicator shows filter is spent	CP17972
Oxygen Gas Filter	■ Installed between carrier gas filter and GC inlet ■ Removes oxygen and water vapor ■ Recommended with capillary columns ■ Required for ECD ■ Filter should be changed when indicator shows filter is spent	CP17970

Column and Injector Ferrules for Capillary Columns

 Ferrule Size	Column ID (microns)	Ferrule		
		Polyimide (10/pk)	Graphite (10/pk)	Polyimide/Graphite (10/pk)
No hole 1/16" fitting	–	28-694503-01	–	28-694590-01
0.4 mm ID 1/16" Fitting	180-250	28-694586-01	28-694583-01	28-694580-01
0.5 mm ID 1/16" fitting	320	03-908361-01	28-694561-01	28-694581-01
0.8 mm ID 1/16" fitting	530	28-694552-01	28-694042-01	28-694582-01
5 mm ferrule for Split/Splitless Insert (1079)	–	–	03-925342-01	–

Column Ferrules for Micro-TCD

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

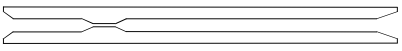
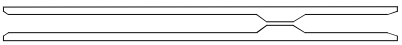
Column ID	Ferrule ID	Ferrule Part Number
0.1 mm	0.4 mm	CP85889
0.25 mm	0.4 mm	CP85889
0.32 mm	0.5 mm	CP470100
0.53 mm	0.8 mm	CP470101

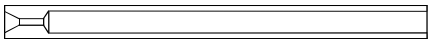
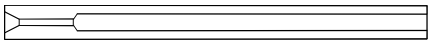
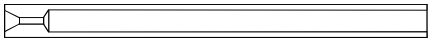
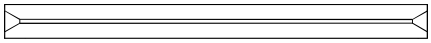
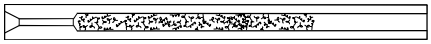
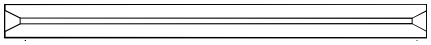
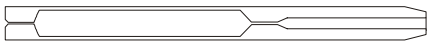
Glass Injector Inserts

Capillary injector inserts have a direct effect on analysis results. They are chosen depending on the injection mode (split, splitless, on-column, flash vaporization) and sample characteristics.

- The split and splitless inserts create a homogenous mixture of sample and carrier gas, transfer a representative sample into the column and minimizing molecular weight discrimination.
- The on-column temperature programmable inserts for the 1079 provide quantitative sample transfer and retain the liquid sample during cold sample introduction.
- The flash vaporization insert (1061) provides an expansion volume for large samples and minimizes non-volatile sample components from entering the column.

Insert variations for all injection modes are available for dirty sample matrices, labile compounds, and large volume injections. Inserts should be replaced as soon as a loss in chromatographic performance is seen. Note that all the 1079 inserts are deactivated.

	Description	Part Number
1079 DIRECT ON-COLUMN	*High Performance Insert* ■ 180 to 320 µm ID columns ■ Quantitative liquid sample transfer ■ Used for cold on-column injections	01-900109-06 
	On-Column Insert ■ 530 µm ID Columns ■ Quantitative liquid sample transfer ■ Used for cold on-column injections	01-900109-07 

	Description	Part Number
1079 SPLIT	*Frit Insert* ■ Linear split, adequate sample mixing, instantaneous sample vaporization. ■ 3.4 mm ID	03-918464-01 
	Unpacked Insert ■ Can be packed with quartz wool, glass beads, etc. (1079 is shipped with this insert installed) ■ 3.4 mm ID	03-918464-00 
	Packed Insert ■ 3.4 mm ID	03-918956-00 
1079 SPLITLESS	*Open Insert* ■ The narrow bore minimizes dead volume, ensuring efficient transfer of sample to the column. (One is included in the 1079 accessory kit.) ■ 2 mm ID	03-918466-00 
	Unpacked Insert ■ Can be packed with quartz wool, glass beads, etc. (1079 is shipped with this insert installed) ■ 3.4 mm ID	03-918464-00 
1079 SPLITLESS TEMP RAMP	*Open Insert* ■ Trace analysis. The low surface area makes it ideal for thermolabile and polar components. ■ 0.5 mm ID	03-925331-00 
	Packed Insert ■ Deactivated glass wool packing. Can be used in all three modes. For non-polar compounds above 1 ng level. ■ 2 mm ID	03-925350-00 
1079 SPME	*SPME Insert* ■ For SPME injections ■ 0.8 mm ID	03-925330-00 
1061 FLASH VAPORIZATION	■ 530 µm columns only	03-918339-00 
	■ Packed column insert	37-000813-00 

Septa

Septa allow the syringe needle to enter the GC injector, yet maintain a leak-free seal in the GC system. They are available in several different types of materials and sizes depending on injector model and analysis needs. The septum chosen for a GC analysis should exhibit low bleed, resist leaks, and be easy to pierce when performing injections.

Septa should be changed every 50 to 100 injections or when you note a change in peak retention time or ghost peaks. It is preferable to change septa routinely, rather than after leaks develop, thus minimizing instrument downtime and sample loss. Change the septum at the end of the workday, then keep the column oven temperature hot enough to prevent bleed from accumulating overnight. Using a needle guide, a syringe free of burrs, or an autosampler will prolong the septum life because a single hole will be repeatedly pierced allowing easier re-sealing.

Septa	Description	Dimensions	Qty	Reference
Ultrasep-R™	■ Red silicone rubber ■ T_{max}: 350°C ■ Low bleed ■ Long injection life ■ Easy sample injection	10 mm 10 mm	25 100	00-996881-01 00-996881-02
ThermoGreen™ LB-2	■ Green silicone rubber ■ T_{max}: 300°C ■ Low bleed ■ Recommended for all 1079, GC/MS and ECD applications	11.5 mm 11.5 mm	5 50	03-920357-01 03-920357-02
Standard	■ Beige silicone rubber with Teflon® face ■ T_{max}: 250°C ■ Standard septa for Varian GCs	10 mm 10 mm 11.5 mm 11.5 mm	25 100 25 100	00-997628-02 00-997628-03 00-997630-02 00-997630-03

Size your septa here

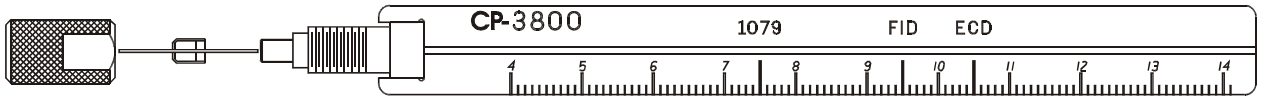
10 mm
1041 On-column
1061 Flash Vaporization

11.5 mm
1079 Injector

Column Quick Connect Kit

This kit simplifies the installation of capillary columns into Varian injectors and detectors. The kit contains split capillary column nuts, reusable jacketed graphite ferrules (for 250µ, 320µ, 530µ ID columns) and a column depth scale. No felt tip pen, typewriter correction fluid or tape is needed to mark column depth for any injector or detector. The split nut design allows you to remove the nut from the column when the column is stored without having to remove the ferrule. The split capillary column nuts are knurled so that all tightening can be done by hand. No tools are required.

Column Depth Scale



Included in This Kit *(P/N 03-925751-90)*

Description	Quantity
Column Depth Scale	1 each
Knurled Split Nut	2 each
Graphite Jacketed Ferrule (0.4 mm ID)	2 each
Graphite Jacketed Ferrule (0.5 mm ID)	2 each
Graphite Jacketed Ferrule (0.8 mm ID)	2 each

Spare Parts

Description	Quantity	Part Number
Graphite Jacketed Ferrule (0.4 mm ID)	10 each	03-925384-04
Graphite Jacketed Ferrule (0.5 mm ID)	10 each	03-925384-05
Graphite Jacketed Ferrule (0.8 mm ID)	10 each	03-925384-06

IV. METHOD DEVELOPMENT

Recommended Solvents for Capillary Columns

The choice of solvents for a chromatographic analysis depends on the component solubility, detector, and the polarity of the analytical column. Solvents should ideally match the polarity of the column, especially when injecting large volumes and performing on-column or splitless injections. Non-polar columns perform best with non-polar solvents. Polar columns perform best with polar solvents, however, they also perform well with non-polar solvents.

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

Column Phase	Recommended Solvent	Boiling Point (°C)
Non-Polar ■ 100% Methyl ■ 5% Phenyl, 95% Methyl	■ Pentane ■ n-Hexane ■ Cyclohexane ■ Isooctane ■ Benzene ■ Toluene ■ Ethyl Ether ■ Methyl tert-butyl ether ■ Methylene Chloride ■ Carbon Tetrachloride ■ Carbon Disulfide	36.1 69.0 80.7 99.3 80.1 110.6 34.6 55.2 39.8 76.7 46.5
Intermediate ■ 50% Phenyl, 50% Methyl	■ Ethyl Acetate ■ Acetone ■ Methyl iso-butyl ketone ■ Acetonitrile	77.0 56.5 127.0 81.6
Polar ■ Polyethylene Glycol	■ Methanol ■ Ethanol ■ n-Propanol ■ n-Butanol	64.7 78.5 97.2 117.7

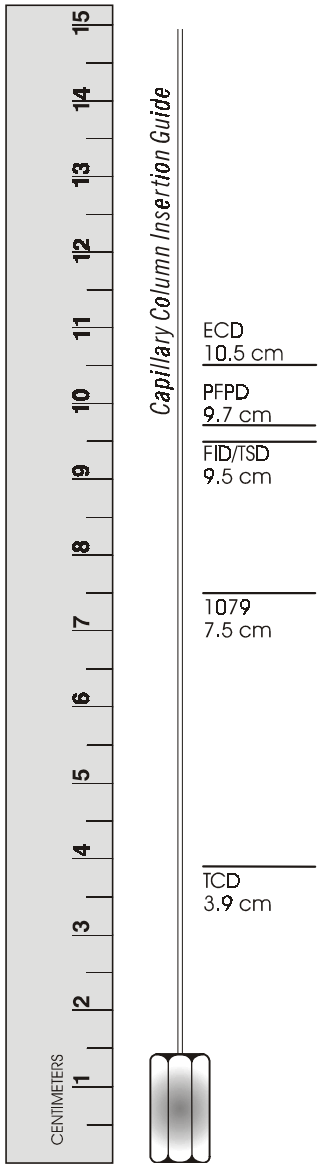
For optimum results with capillary columns:

- Use small injection volumes whenever possible (~1 μL) for on-column and splitless injections
- Small ID columns with thin film coatings accommodate smaller injection volumes
- Large ID columns with thicker coatings can accommodate larger injection volumes
- Replace injector inserts and septa frequently, as often as every 50 injections
- Use non-polar solvents whenever possible

Recommended Injector and GC Parameters

Injector	On-Column, 1041 Flash Vaporizing, 1061		1079 *Using* Split Insert	1079 *Using* Splitless Insert	1079 *Using* On-Column Insert	1079 *Using* High Performance Insert	
Column	*For* ■ Large Bore Capillary Columns (530 µm ID) ■ Packed Columns		*For* All Capillary Columns	*For* All Capillary Columns	*For* Large Bore Capillary Columns (530 µm ID)	*For* All Capillary Columns (180-320 µm ID)	
Sample Volume in µL	0.1 - 0.5	0.5 - 5	0.1 - 1	0.5 - 3	0.1 - 5	0.2 - 1	1 - 5
Injection Rate, in µL/sec	5	0.5 - 1	10	0.5 - 1	1 - 5	5	1
Injection Time (Needle Residence Time in min)	0.05 - 0.1	0.1 - 0.2	0	0.05 - 0.2	0	0	0
Hot Needle Time in min	0	0	0	0 - 0.1	0	0	0
Solvent Plug Size in µL	1	1	1	1	1	1	1
Column Oven	Isothermal or temp. program	Temp. program	Isothermal or temp. program	Temp. program; Initial temp. ≤20°C from the solvent boiling point	Temp. program	Temp. program; Initial temp. ≤20°C from the solvent boiling point. With larger sample volumes, solute focusing is possible with initial temp. 10-20°C above solvent boiling point.	

Column Installation Measurements for Injectors & Detectors



Not to Scale

Units of Measure

Name	Abbreviation	Weight/ weight	Weight/ volume	Volume/ volume
Parts per Thousand	‰	mg/g	µg/µL mg/mL g/L	mL/L
Parts per Million	ppm	µg/g mg/kg	ng/µL µg/mL mg/L	nL/mL µL/L
Parts per Billion	ppb	ng/g µg/kg	pg/µL ng/mL µg/L	nL/L
Parts per Trillion	ppt	pg/g ng/kg	fg/µL pg/mL ng/L	pL/L

Non-Retained Compounds for GC Detectors

Detector	Recommended Non-Retained Compounds
FID	Methane, propane, butane
TCD	Air, methane, butane
ECD	Methylene chloride headspace vapors
TSD	Acetonitrile headspace vapors, butane
PFPD	Sulfur hexafluoride, methane, propane, butane
MS	Carbon dioxide
Micro-TCD	Air, methane, butane

VARIAN

Varian Analytical Instruments
2700 Mitchell Drive
Walnut Creek, CA 94598/USA

Other Quick Reference Items for your GC