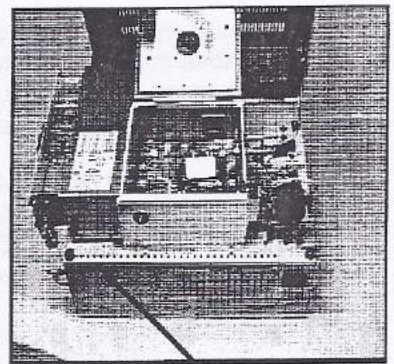
Chapter: Injectors

Topic: EPC (electronic pressure control) operation

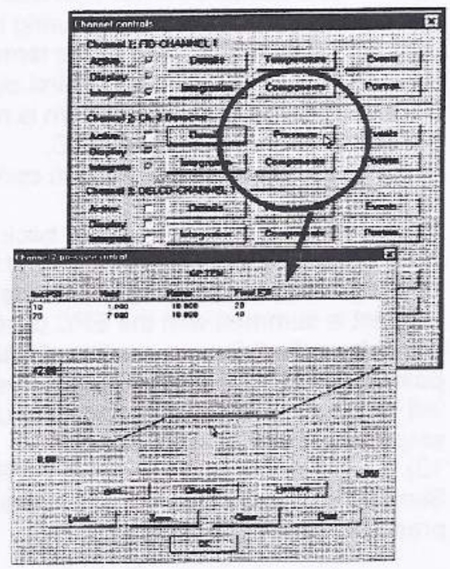


Local setpoint adjustments for temperatures and pressures using small screwdriver

SRI GCs are equipped with electronic pressure control of all system gases. Detector support gases such as hydrogen and air are controlled by the screwdriver adjustable local setpoint on the GC, and once set are seldom altered. The carrier gas pressure may be controlled by either the local setpoint screwdriver adjustment or by the channel two pressure program in the PeakSimple data system software. **The main benefit of carrier gas pressure programming (ramping) is to speed up the flow rate through the column at the end of the run in order to elute high boiling peaks more quickly.**

Most chromatographers choose to set the carrier gas pressure using the screwdriver local setpoint adjustment rather than the channel two pressure program for the following reasons:

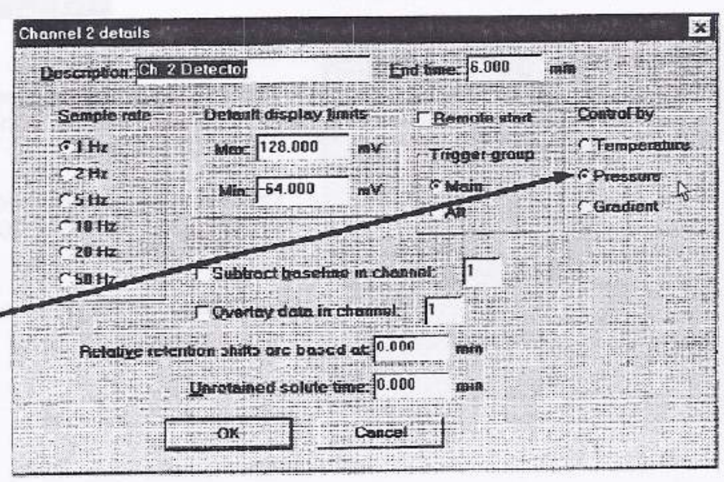
1) The screwdriver adjustment is simpler, and once set is not likely to be altered unintentionally.
2) The benefits of ramping the carrier gas pressure are often not worth the extra operational complexity.



Because very few users choose to utilize the pressure programming features, all SRI GCs are shipped with the EPC control disabled. Instructions for enabling the EPC are shown on the following page. Once the EPC is enabled, the carrier gas pressure will follow the pressure program loaded into channel two of the PeakSimple data system software. Channel two must be activated and a pressure program entered even if only a single detector signal is being acquired on channel one. The pressure program end time must be coordinated with the temperature program for the column oven which is loaded into channel one.

Once you make the changes, don't forget to save your control file (default.con) so PeakSimple will remember your changes the next time you boot up.

Set the "Control By" radio button in the Channel 2 Details screen to Pressure. Then enter the desired pressure program into Channel 2 by selecting the Pressure screen from the Edit/Channel menu



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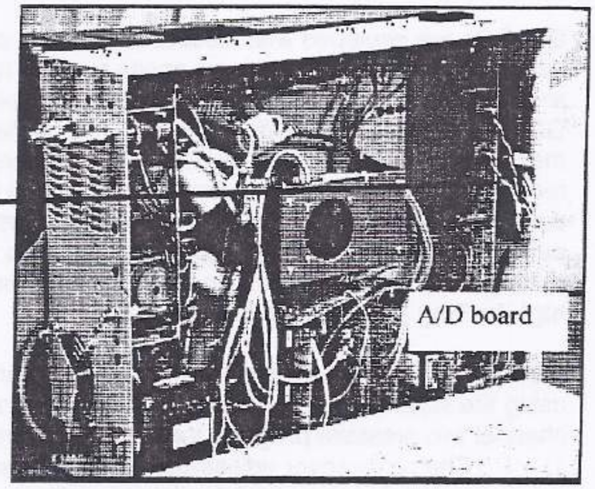
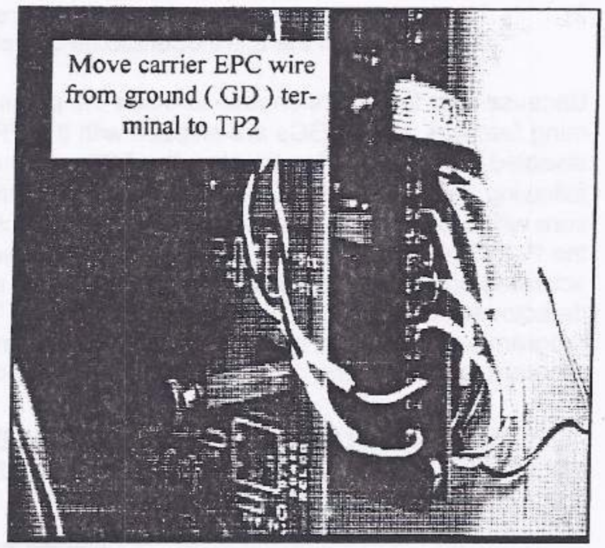
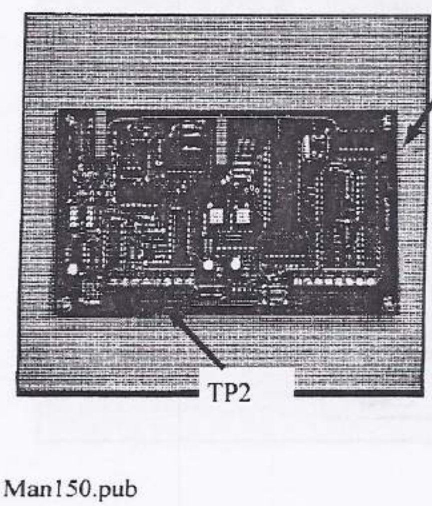
Chapter: Injectors

Topic: Enabling the carrier gas EPC

1) Un-plug the GC power cord.
2) Remove the six screw holding the bottom cover to the GC chassis.
3) Tilt the GC on its back and expose the interior.
4) Locate the A/D board which is mounted on the right hand interior wall.
5) Locate the carrier gas EPC wire (green with white stripe and labelled carrier EPC) This wire is attached to a Ground (GD) terminal on the A/D board before shipment from the SRI factory. Attaching this wire to Ground disables the computer control of the EPC.
6) Use the screwdriver provided with the GC to loosen the screw securing the wire and re-attach the wire to the terminal labelled TP2. The pressure control signal from the PeakSimple data system is now connected to the carrier gas EPC.
7) Re-assemble the botttom cover and screws.
8) Plug the GC power cord back in.
9) Use the screwdriver to adjust the carrier gas local setpoint to 0.00. The local setpoint is summed with the EPC control signal from PeakSimple, so if the local set-point is not set to 0.00, the carrier pressure will be the sum of the local and computer setpoints.
10) Enter a pressure program in Peak-Simple's channel 2, and verify that the GC pressure follows the program.



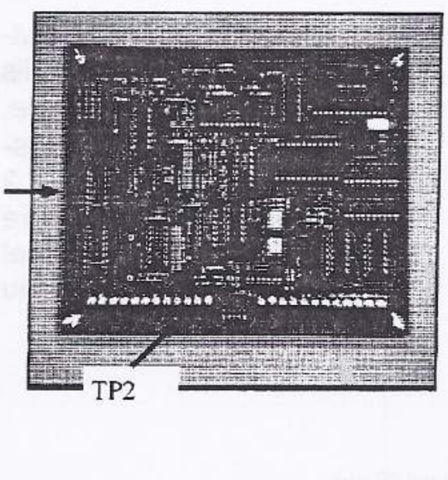




Some GCs will be equipped with the single channel Model 203 A/D board.

Other GCs will be equipped with the 4 channel Model 202 A/D board.

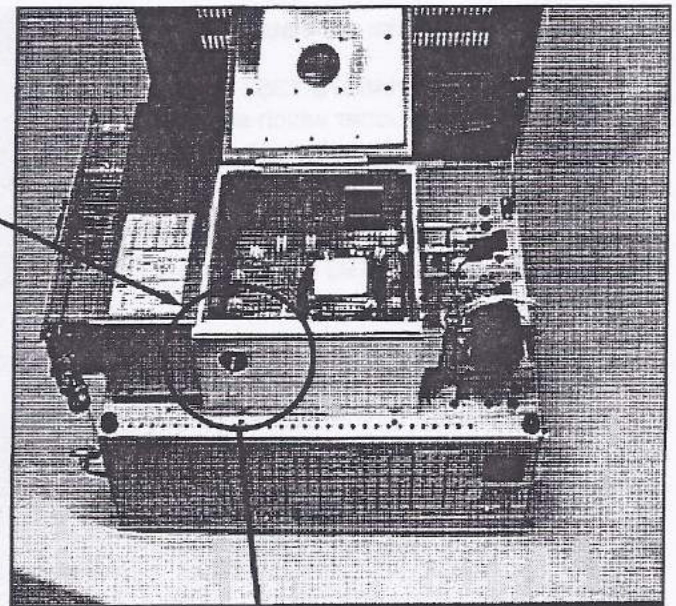
The procedure is identical on either board.



Chapter: Injectors

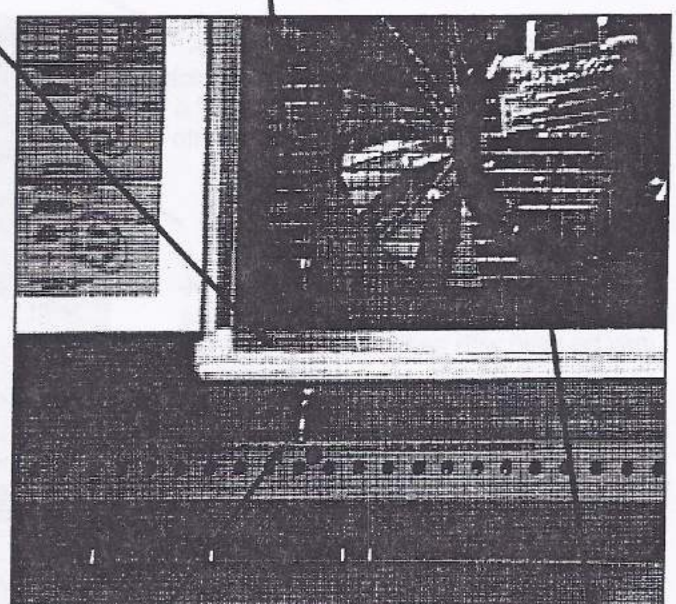
Topic: On-column Injector Operation

The On-column injector is designed for .53mm (wide-bore) capillary columns and 1/8" packed columns. One or two on-column injectors can be mounted on the 8610C GC, while a single on-column injector can fit on the Model 310 GC. The photo at right shows a single on-column injector mounted on the 8610C GC.



The on-column injector is not separately thermostatted because it closely follows the temperature of the column oven due to its low mass design and mounting location on the wall of the column oven.

Because the insulated oven wall on SRI GCs is only .75" thick, sample is injected onto the column well inside the column oven, so no cold spots can trap the sample, even if the sample consists of high boiling analytes.



Septum nut with silicone rubber septum seals carrier gas in, but allows syringe to penetrate into column

60 meter .53mm metal capillary column shown connected to on-column injector

For most applications, the on-column injector is the best way to inject a liquid sample because the syringe deposits the sample into the bore of the column itself. The column is usually the most inert surface available (more inert than glass injector liners), and unlike heated injectors, the sample does not undergo a flash vaporization which can broaden peaks and result in peak tailing. Also, because the entire sample is deposited on-column, boiling point discrimination can not occur as it can with split/splitless injection techniques.

Chapter: Injectors

Topic: On-column Injector Operation

The On-column injector consists of:

1) The septum, (part#8670-1353) which is a plug of silicone rubber which allows the syringe to penetrate but which prevents the carrier gas from escaping. The septum used on SRI GCs is sometimes called a "shimadzu plug" type septum and is widely available from GC supply catalogs

2) The special septum nut (part#8670-9090) for 26-27 gauge syringe needles. The extended snout on the septum nut helps guide the syringe needle straight onto the column.

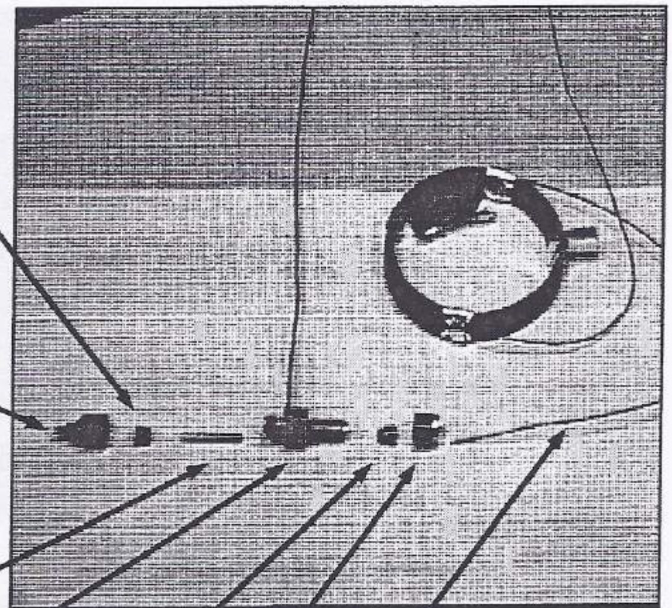
3) The wide-bore capillary column adapter (part#8690-9093) which aligns the syringe needle and the column inside the on-column injector body.

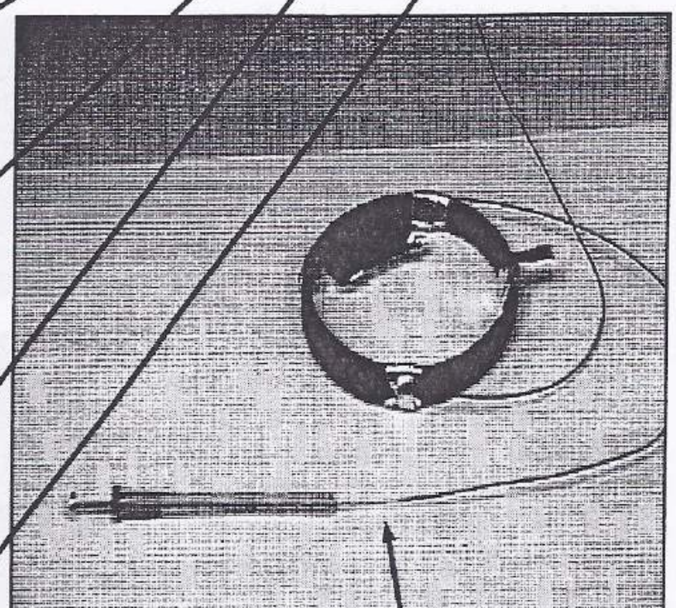
4) The injector body fitting (part#8670-9094). This is a stainless steel swagelok type fitting modified with the addition of a carrier gas inlet tube which is welded into the side.

5) A 1/8" to 0.8 mm graphite reducing ferrule secures the wide-bore (.53mm) capillary column into the injector body fitting. Either soft or hard graphite ferrules may be used with capillary columns.

6) A 1/8" swagelok type nut (stainless or brass) is used to compress the graphite ferrule around the column. Stainless is recommended for oven temperatures above 200°C.

7) A wide-bore capillary column (.53mm i.d.) of any length. The on-column injector is normally used with wide-bore capillary or 1/8" packed columns, not with columns whose inside diameter is less than .53mm since that is the smallest diameter into which a standard 26 gauge syringe will fit.

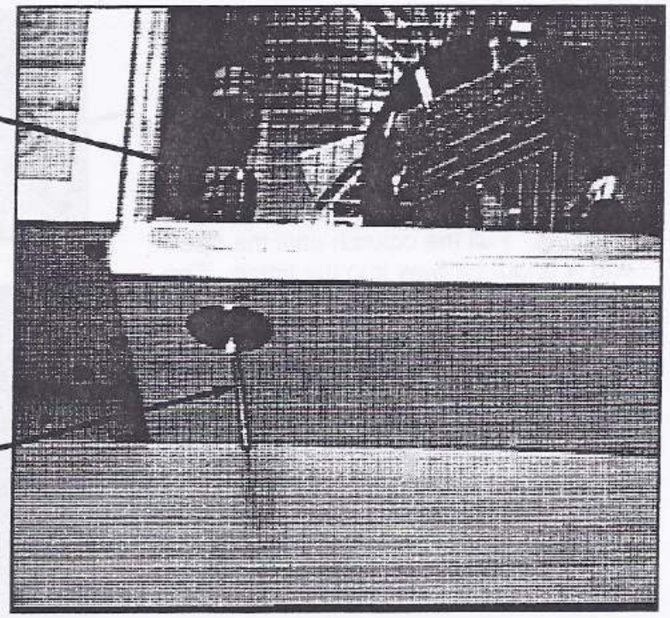




As shown above, the 26 gauge needle on the standard 10 ul GC syringe fits perfectly into the bore of a .53mm wide-bore capillary column

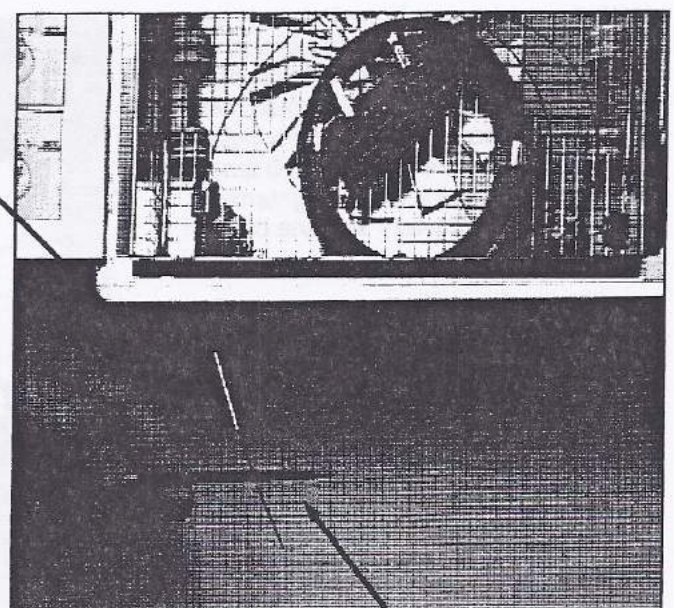
To install the column in the injector:

1) Feed the column end through the 1/8" swagelok type nut and graphite reducing ferrule. If the ferrule has been previously used, inspect it carefully to make sure it is still intact. Sometimes used ferrules will break inside the nut and a part of the ferrule will fall out. What's left inside the nut may not seal correctly. Try to avoid shaving bits of graphite from the ferrule into the bore of the column as this can cause peak tailing and absorption.



2) Push the column all the way through the injector fitting and out the front. Then slip the wide-bore adapter over the end of the column. Be sure that the conical end of the adapter is facing out towards the operator. The gash in the adapter allows carrier gas to enter the column even if the end of the adapter is plugged off.

3) If you are using a metal capillary as shown in the photo, use a sharp file make a score mark an inch or two from the end of the column. Holding your thumbnail under the score mark, snap the column end off to make a clean break. If you are using a polyimide coated fused silica capillary column, a razor or sharp knife edge is used to make the score mark. The end of the column is removed to ensure that no graphite particles or other debris which may have entered the column bore during the installation process remains in the column.

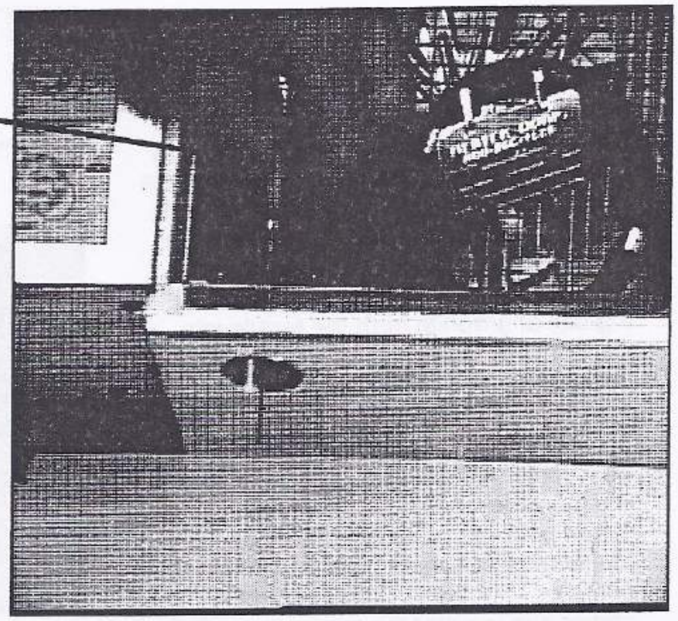


As shown above, a sharp triangle file is used to score the metal capillary column a few inches from the end which may have picked up graphite or other debris during the installation process.

HINT: Some chromatographers use a small reamer (Dremel tool bit) to clean up and smooth the end of the metal capillary column bore hole. The smoother hole allows the syringe to enter the column with less chance of snagging on the lip of the column. The syringe itself should be in good condition with no burrs or kinks. SRI supplies a syringe with a conical needle tip (part#8670-9550) in your choice of 5, 6, or 7 cm needle lengths

Chapter: Injectors

Topic: On-column Injector Operation

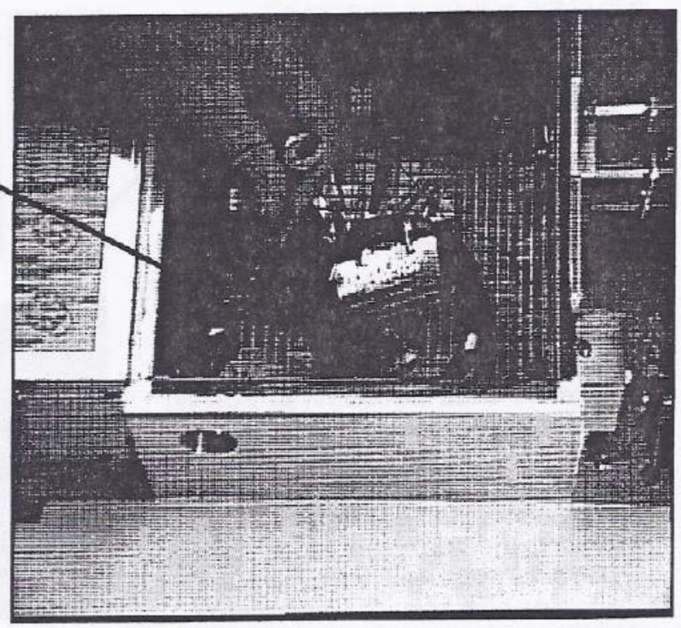


4) Pull the column and wide-bore adapter back into the injector fitting through the partially tightened nut and graphite ferrule. As you pull, the column will gradually disappear from view inside the injector fitting. Pull the column until the open end is about halfway into the fitting. The exact distance is not critical so long as the syringe needle ends up depositing the liquid sample in the bore of the column itself. If the column is pulled too far towards the oven, the syringe needle may deposit the sample in the adapter where it will gradually diffuse into the column causing wide or tailing peaks. If the column is positioned too far out towards the operator, the syringe needle may snag on the lip of the column as it is inserted.

With the column positioned, tighten the nut and graphite ferrule. You should feel the ferrule squish slightly as you tighten the ferrule, and the column should feel snug and immovable. A properly tightened ferrule can be re-used 5-10 times, while a ferrule which is over-tightened must be replaced every time the column is changed.

NOTE:

Metal capillary columns are easier to install than polyimide coated fused silica columns because as the syringe enters the column entrance it can chip away bits of the fused silica unless it is perfectly positioned. The metal columns are more forgiving since the column will not fracture when in contact with the syringe needle.

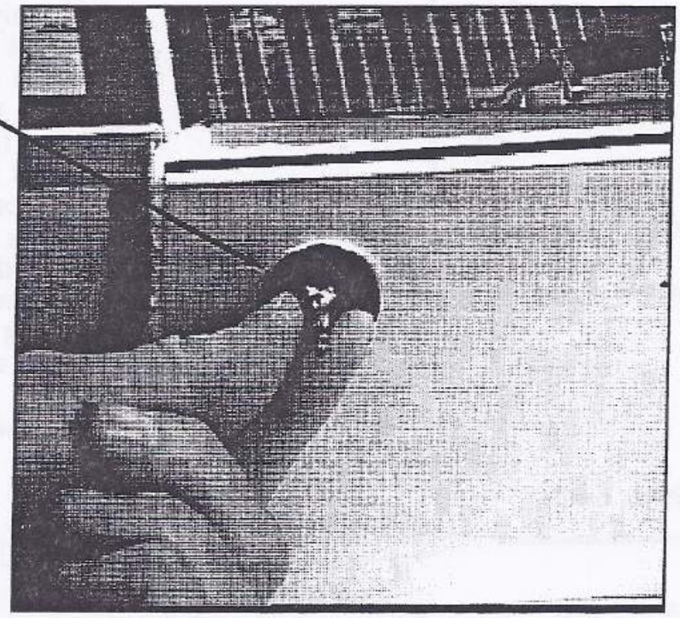


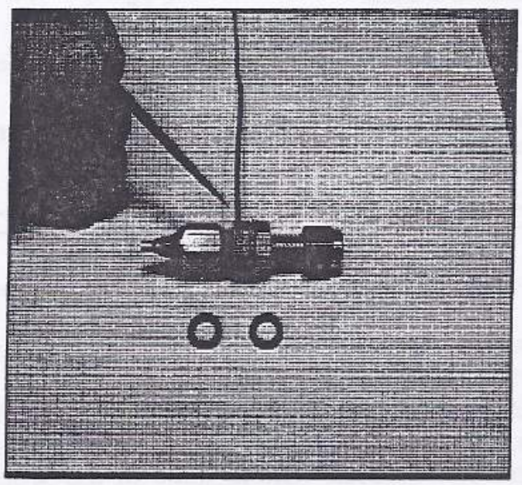
A 7/16" wrench is used to snug up the nut and graphite ferrule securing the column to the injector.

Chapter: Injectors

Topic: On-column Injector Operation

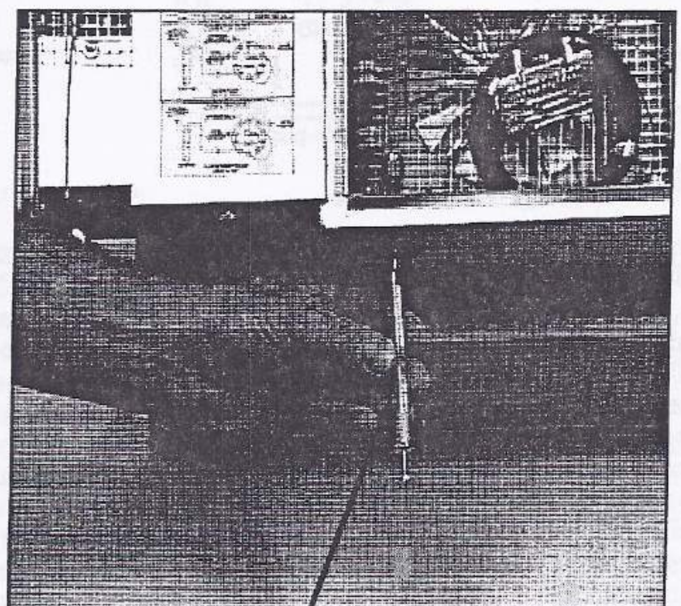
5) Tighten the septum nut until it contacts the one or two rubber o-rings on the injector body. The o-rings act as a helpful guide to avoid over-tightening the septum. When the soft silicone rubber of the septum is over-compressed, the syringe has to fight its way through often plugging with septum material in the process. A properly tightened septum cleaves easily to let the syringe needle pass, then self-heals itself when the syringe is withdrawn. Properly tightened, a plug type septum as used on the SRI GC will last up to 300 injections, while an over-tightened one will leak after 10-20 injections.



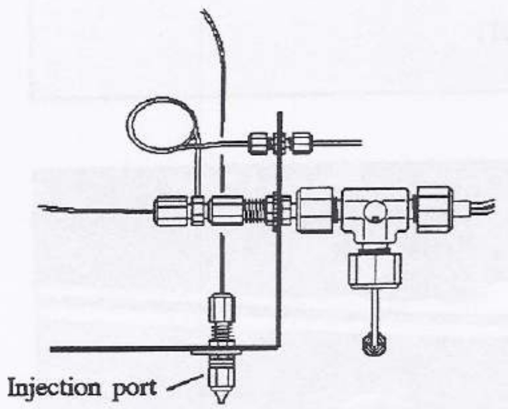


The photo above shows the injector fitting with two o-rings installed on it, and the septum nut tightened up so it just contacts the o-rings.

If the syringe snags on the edge of the column as it is inserted, loosen the swagelok nut and ferrule and pull the column another few millimeters further towards the inside of the column oven. Tighten the nut and retest by inserting the syringe.

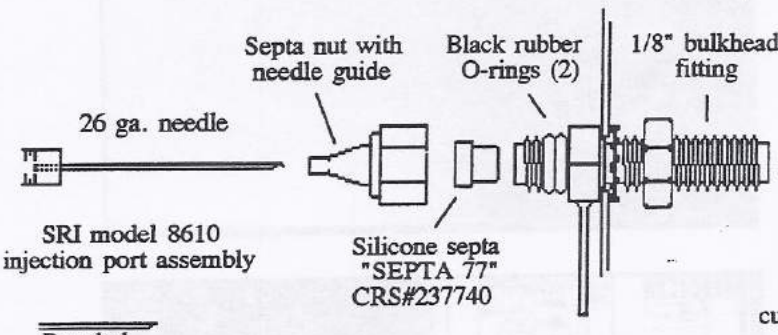


Test your installation by inserting a syringe into the column as far as it will go. The syringe should glide into the column bore smoothly without snagging or feeling rough.

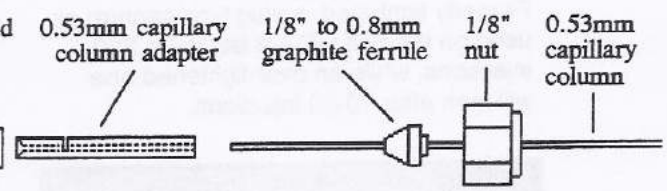


Location of injection port in typical FID system

The 8610 gas chromatograph is shipped equipped with a direct injection-type injection port. This port permits on-column manual injections with traditional chromatography syringes. The injection port is simple and highly efficient by design. Swagelok stainless steel hardware is used in the assembly of the injection port. Injection of gas and liquid samples is performed using standard syringes equipped with a 26 ga. needle. Beveled (medical-style), straight, and hooked tips are available from many suppliers in this needle size. For larger needles, such as a side-port, blunt-tipped needle, a 1/8" Swagelok stainless steel nut is used in place of the supplied septa nut. Although several needle tips are available, hooked-tip needles promote septa life by slicing through the septa without "coring" the silicone, as do medical and straight-tipped needles.

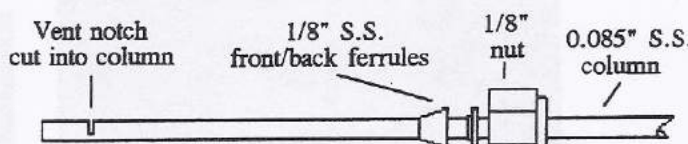


SRI model 8610 injection port assembly



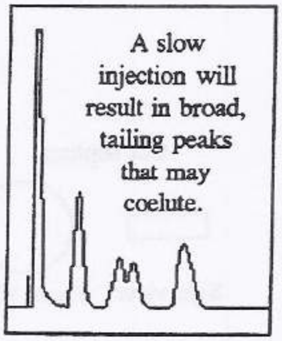
Connection of 0.53mm fused silica column to injection port

Syringe needle tip styles

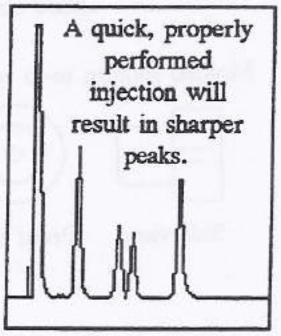


Connection of 0.085" I.D. stainless steel packed column to injection port

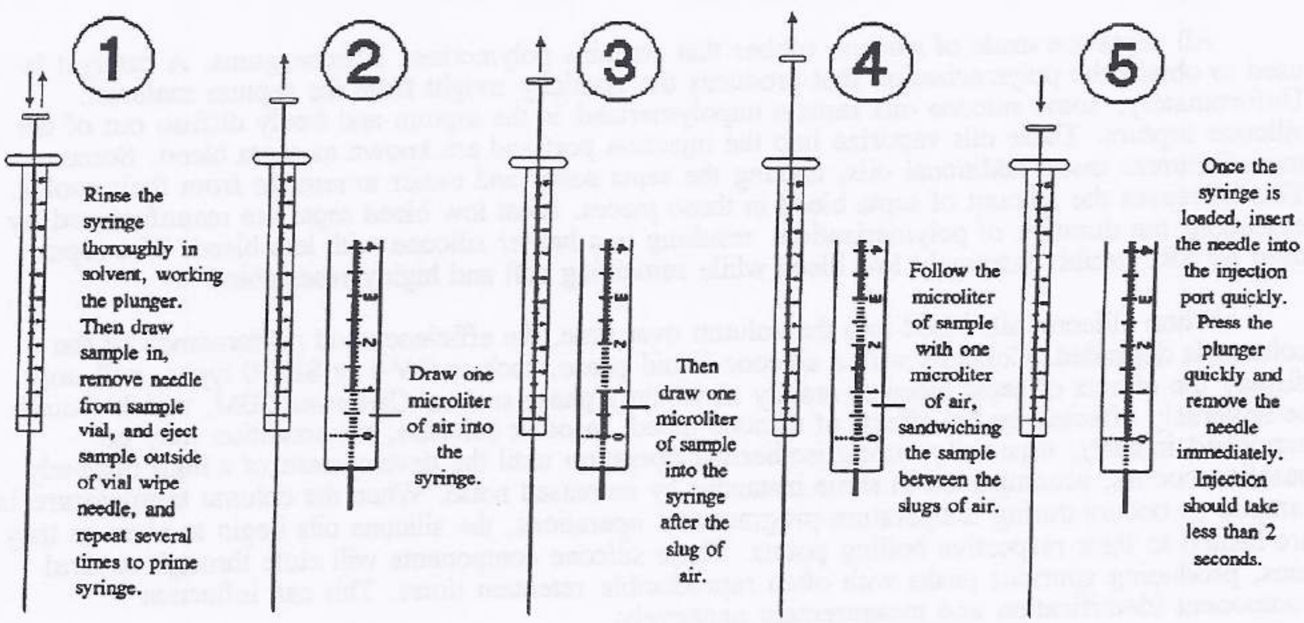
Therefore, they are recommended. "Coring" punches shards of septum into the injection port and may result in plugging of the syringe needle and failure to deliver sample. Over an extended period of time, these shards could migrate into the column. In a packed column, this accumulation of septum shards increases the exposed surface of silicone available to produce silicone or "septa" bleed. In a capillary column, these shards could plug the column completely. Routine maintenance of the septum prevents this from occuring. A bad septum may bleed excessively or permit carrier gas to leak out of the system, affecting retention times. It may visibly bulge or show numerous slices or shards of silicone protuding in toward the injection port. This usually occurs when the septum nut has been over-tightened and the physical characteristics of the septum have been altered due to compression of the silicone. If a septum is extremely bad, the user might see a puff of smoke blow out from the injection port after injection. This is the volatized sample blowing back out through the leak on a continuous stream of carrier gas. Septa may become tacky and unusable after extended service. The septa nut should be finger-tight. Once the user feels the septum seat snugly against the bulkhead fitting, the septa nut has been tightened sufficiently. Use the two black rubber O-rings on the injection port as a guide - the nut should barely make contact with the outer O-ring when the nut is properly tightened. NEVER use a wrench to tighten the septa nut. An over-tightened septum will have a markedly decreased lifetime. Larger side-delivery needles also tend to reduce septa life due to the size of the puncture created during injection. This requires more frequent servicing of the septum. Please note that when septum replacement is required during use of the thermal conductivity detector (TCD), the filament current should be turned off at the electrometer located on the right side control panel of the chromatograph, prior to removing the septa nut. Failure to do so could result in the destruction of the detector filaments due to lack of carrier gas flow through the column and into the detector.



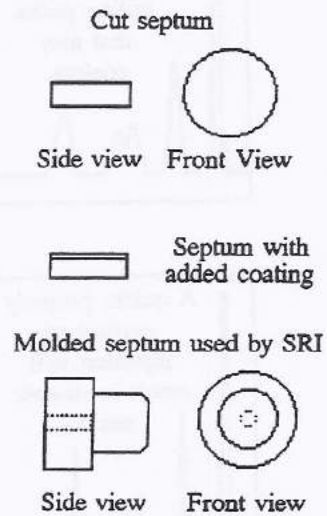
When performing analyses using manual direct injection, the method or technique used to prepare the syringe and perform the injection can mean the difference between obtaining chromatograms that are either poorly resolved or clean and sharp. Reproducibility can also be affected if the amount injected varies from injection to injection. This is why it is imperative that a consistent, reproducible method or technique of manual injection be used when performing direct injection.



Sample volume affects the quality of data produced by the gas chromatograph. If too much sample is injected, the column becomes overloaded and the peaks produced will be broad and tailing. Insufficient sample will likely result in quantitation inaccuracies. If the syringe is not properly primed and loaded (or the sample slug contains air bubbles) when injecting liquid samples, or the syringe has not been properly evacuated, purged and loaded when injecting gas samples, the sample amount actually injected will vary, as will the results obtained. The procedure indicated below is just one of many in use today by chromatographers performing direct injection of liquid samples. The syringe and plunger are cleaned. The plunger should not be bent. Then the syringe is flushed thoroughly, primed, and loaded with precision.



Properly prepared, the syringe needle is inserted completely into the injection port in one smooth, quick motion. Then the plunger is driven home immediately. As soon as the plunger tip hits the end of the sample chamber, the syringe needle is withdrawn from the injection port in a quick, smooth motion. This will prevent any sample remaining in the needle from having time to vaporize into the injection port before or during withdrawal (if this were allowed to occur, it would result in peak broadening and tailing). You may currently be using a different technique for direct injection. As long as the method being used is consistent and reproducible, you will obtain reliable, consistent reproducibility from your direct injection analyses of gases and/or liquids.



In order to place a sample into the column of a gas chromatograph without de-pressurizing the injection port and column or interrupting the carrier gas flow, some type of penetrable, resealable membrane must be used. The membrane must be penetrable to permit the introduction of the syringe needle into the injection port, but must also have the ability to re-seal itself. If it could not re-seal itself, each injection would leave a leak that would permit carrier gas to escape from the system. Each subsequent injection would worsen the condition, adversely affecting retention times and sensitivity. Silicone rubber is commonly used to produce injection port septa. Silicone, due to its formulation, is soft yet maintains the ability to seal puncture wounds created by syringe needles. Although septa differ in formulation, proper care will prolong the life of any septum. A silicone septa (CRS 800-327-3800, part number 237740) is installed in all SRI injection ports when shipped. This septum is very soft and resealable. It demonstrates low silicone bleed and does not affect sample component elution times. Additionally, this septum exhibits negligible "coring" for better durability and performance. This septum seals well in the tapered interior of the 1/8" modified Swagelok injection port. The example at right illustrates the difference in physical appearance between this septum and the standard cut septa machine-stamped from silicone sheets. Coated septa are manufactured this way. The coating is intended to reduce septum bleed and increase resealability.

All septa are made of silicone rubber that contains polymerized silicone gums. A catalyst is used to obtain the polymerization that produces the elasticity sought from the septum material. Unfortunately, some silicone oils remain unpolymerized in the septum and freely diffuse out of the silicone septum. These oils vaporize into the injection port and are known as septa bleed. Some manufacturers insert additional oils, making the septa softer and easier to remove from their molds. This increases the amount of septa bleed in those pieces. Most low bleed septa are manufactured by extending the duration of polymerization, resulting in a harder silicone with less bleed. The septa used by SRI exhibit extremely low bleed while remaining soft and highly resealable.

When silicone oils bleed into the column over time, the efficiency and performance of the column is degraded. Columns with a silicone liquid phase, such as OV-1 or SE-30 types, will not display the effects of septa bleed as readily as would a phase such as Carbowax 20M, which would be adversely affected by the effects of silicone bleed. In other columns, the condition may go unnoticed initially, especially during isothermal operation until the development of a high unsteady baseline occurs, accompanied in some instances by increased noise. When the column temperature is ramped as occurs during temperature-programmed operations, the silicone oils begin to elute as they are heated to their respective boiling points. These silicone components will elute through several runs, producing spurious peaks with often reproducible retention times. This can influence component identification and measurement negatively.

In some work, where sensitivity is not great, septa bleed is not a concern. To identify septa bleed, especially where temperature programming is employed, cool the unit to ambient temperature and hold for ten to fifteen minutes. Then ramp the temperature up to the maximum running temperature normally used, with the sensitivity set to high. Any peaks or baseline drift can be attributed to septa bleed. One method to minimize bleed is that of baking septa in an oven prior to insertion into the injection port in order to volatize the silicone oils. The septa may also be baked in the injection port overnight, as long as the column oven is maintained at the same temperature as the injection port to avoid the accumulation of bleed products. Regardless of septum type, septa should never be handled except with tools. Finger oils may appear on chromatograms as additional peaks.

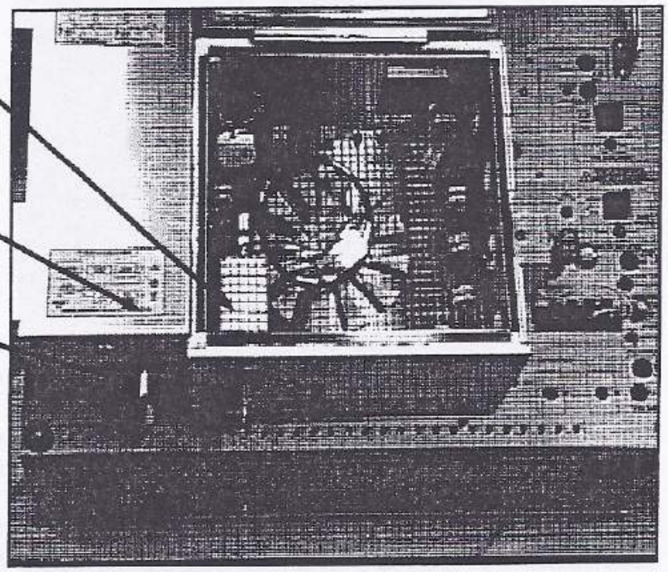
Chapter: Injectors

Topic: Heated Split/Splitless Injector

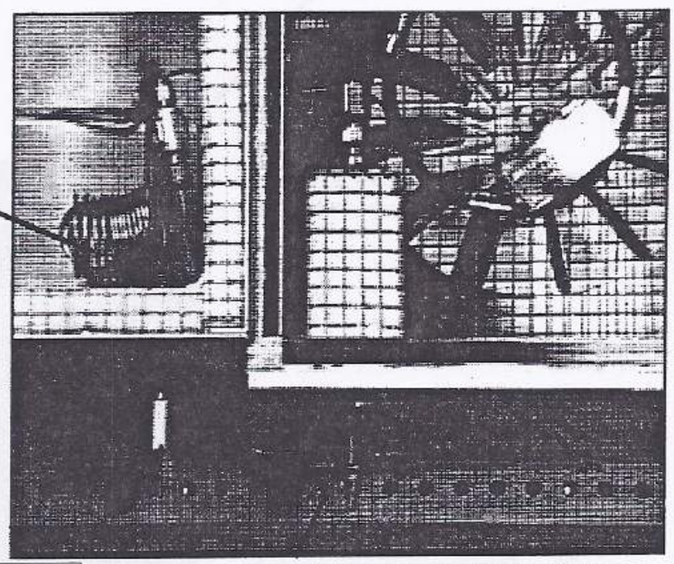
The Heated Split/Splitless Injector can be mounted on the 8610C or 310 GC. It is shown installed on the 8610C GC at right.

When mounted on the 8610C GC chassis, the precision needle valve which adjusts the split flow rate is mounted in the heated valve oven alongside the column oven.

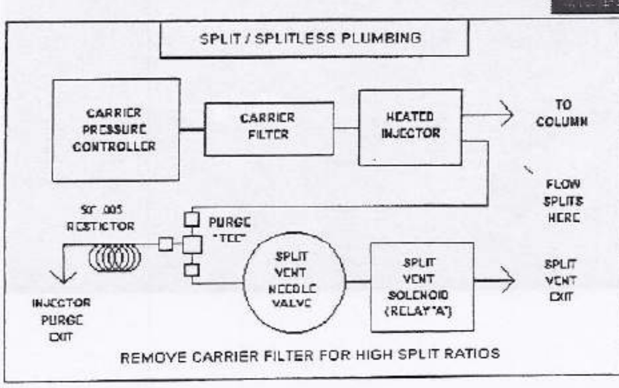
The split flow is adjusted by rotating this knob.



The lid on the valve oven has been removed to expose the Split/Splitless hardware which is installed in the valve oven.



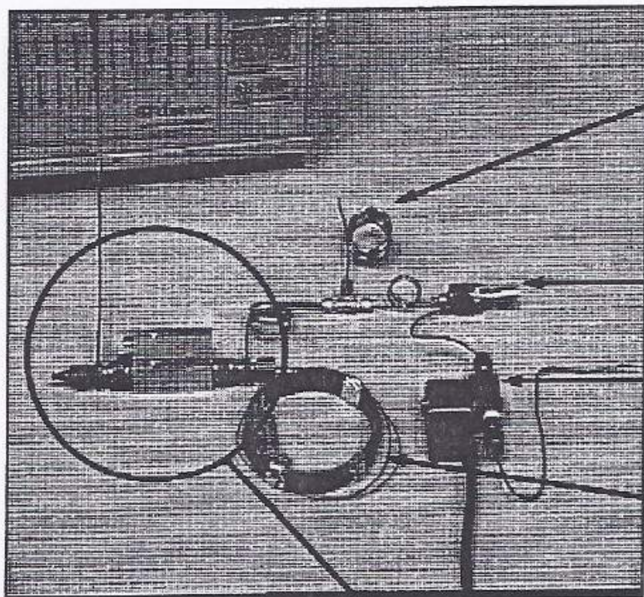
Septum nut mounted on front of Split/Splitless Injector.



The plumbing schematic shown at left illustrates the hardware comprising the Heated Split/Splitless Injector

Chapter: Injectors

Topic: Heated Split/Splitless Injector



The Heated Split/Splitless Injector parts are shown at left removed from the GC for clarity.

1) Injector purge restrictor. A few ml/min of carrier gas continuously exit the injector through this restrictor tubing to prevent high boiling point analytes from diffusing back into the injector.

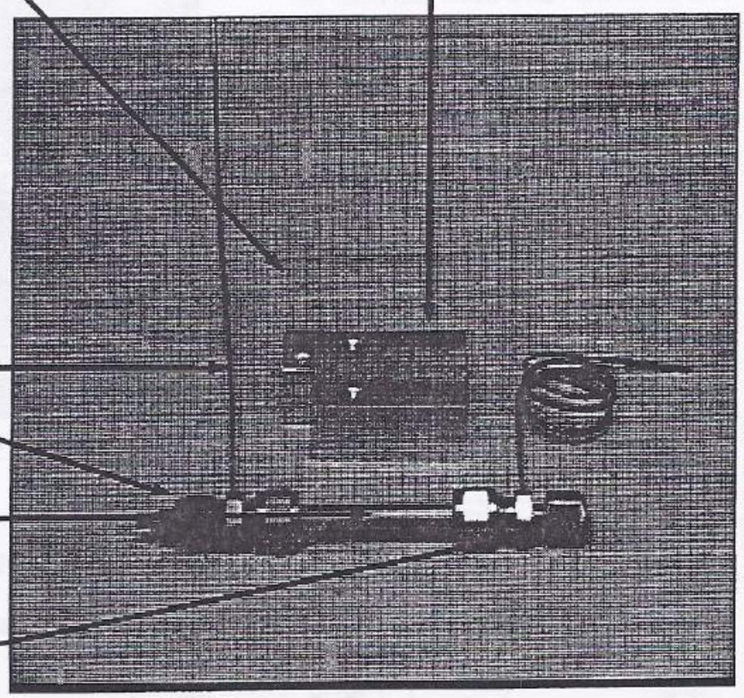
2) Precision needle valve for adjustment of split flow rate.

3) Split flow solenoid turns split on/off under control of the PeakSimple data system.

4) Column is secured into injector using nut and graphite ferrule.

5) Aluminum heater block contains heater cartridge and Type K thermocouple.

The injector liner is shown at right removed from the aluminum heater block for clarity.



1) Carrier gas inlet tubing .

2) Septum nut and septum.

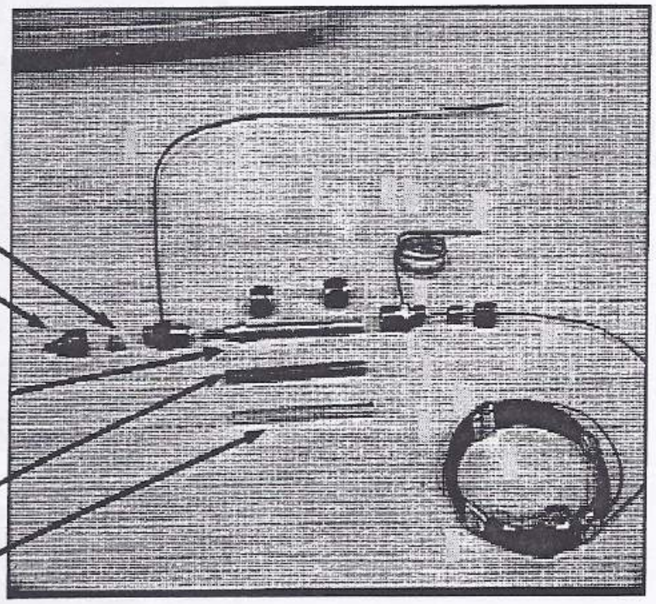
3) SRI stainless steel injector liner.

4) End fitting where column connects and split flow exits to purge vent and needle valve

Chapter: Injectors

Topic: Heated Split/Splitless Injector

A variety of injector liners can be used with the Split/ Splitless injector depending on the column and application.



Septum.

Septum nut.

SRI stainless liner with wide-bore column adapter.

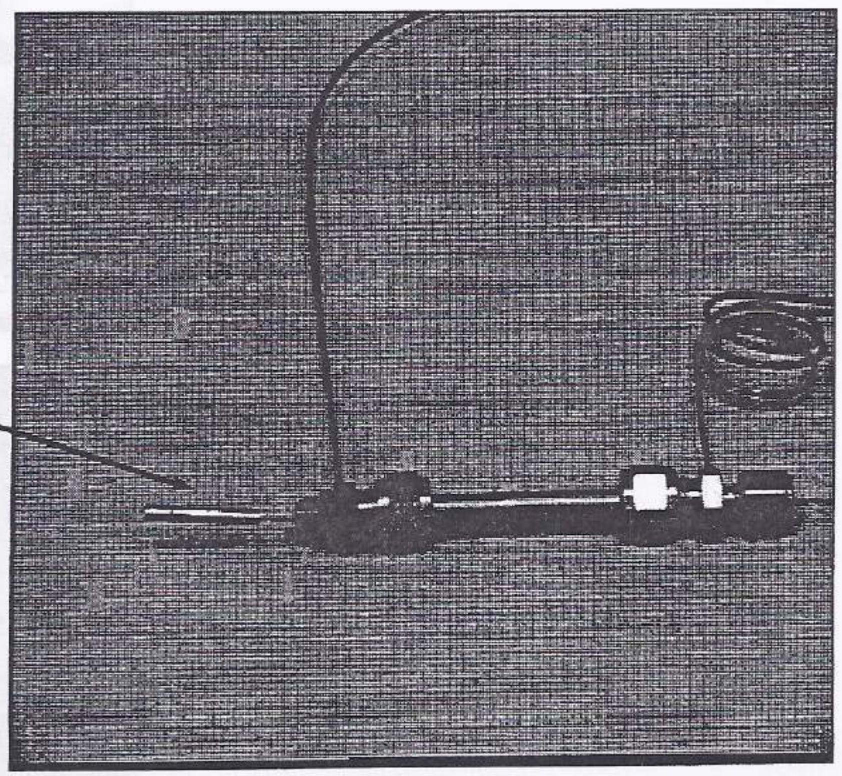
Restek Silco-Steel liner.

Supelco glass liner

SRI designed the Split/Splitless injector to use the same size liner as Hewlett-Packard 5890/6890 series GCs. A huge variety and selection of suitable injector liner types can be purchased from chromatography catalogs such as Alltech, Restek, Supelco and others. The liner supplied with the SRI GC is an unbreakable stainless steel type which also adapts for on-column injection onto wide-bore capillary columns.

The SRI stainless steel injector liner supplied with the GC as standard equipment is shown at right with the wide-bore column adapter slipped over the column in preparation for final adjustment for on-column injection (see on-column injector instructions).

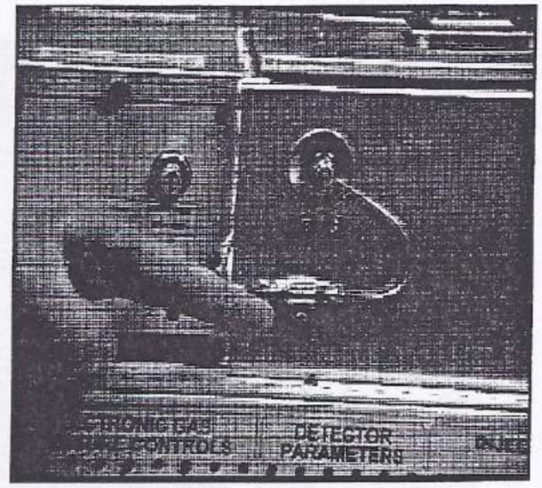
Wide-bore column (.53mm) adapter identical to that used in on-column injector fits perfectly into recess in stainless steel injector liner.



Chapter: Injectors

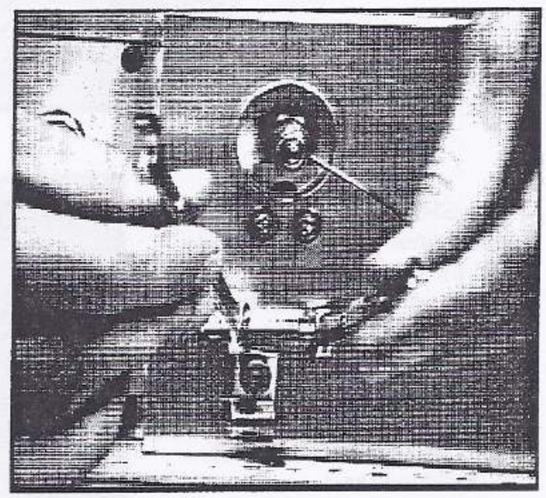
Topic: Heated Split/Splitless Injector

To remove the injector liner from the Split/Splitless injector:

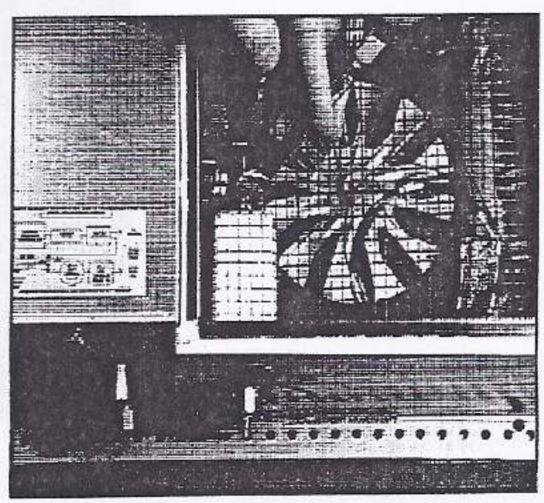


1) Loosen the brass thumbscrew holding the 1/16" stainless union in the carrier gas supply line.

2) Using two 1/4" wrenches, loosen the nut and ferrule on the downstream side of the union and disconnect the tubing leading to the injector.



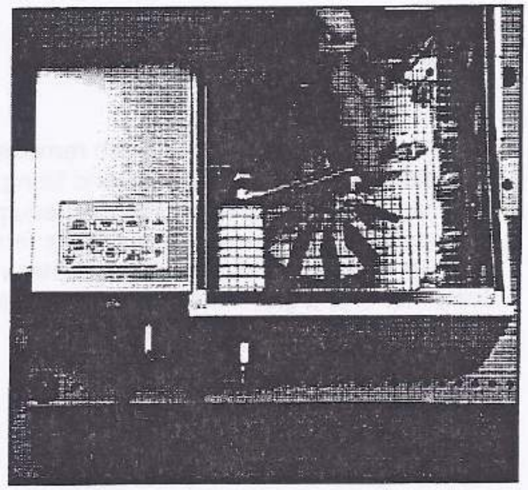
3) Using a 7/16" and 1/2" wrench, loosen the nut and graphite ferrule securing the column to the oven side of the injector.



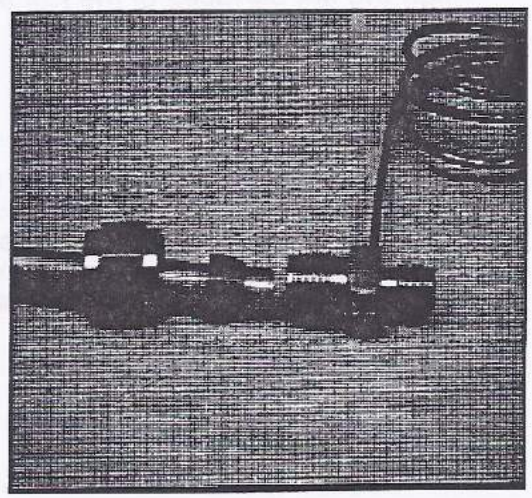
Chapter: Injectors

Topic: Heated Split/Splitless Injector

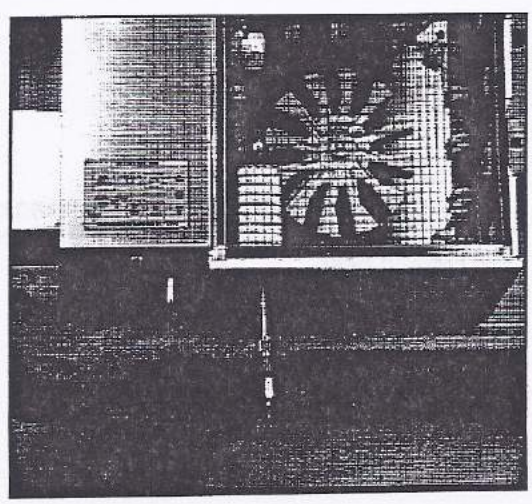
To remove the injector liner from the Split/Splitless injector:



4) Using a 1/2" and 9/16" wrench remove the swagelok type nut securing the end fitting.

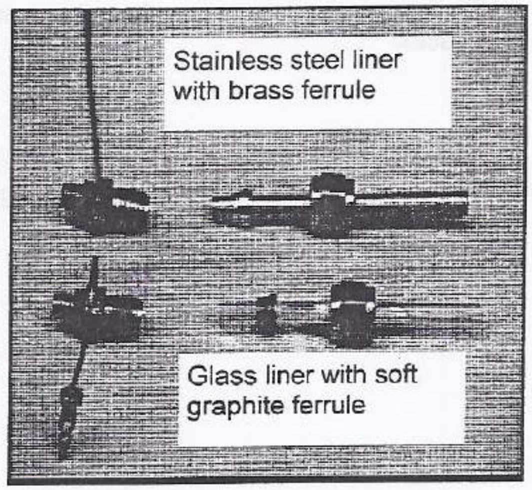


5) The end fitting is shown here removed from the GC for clarity. Notice the hard 1/4" hard graphite (mixture of graphite and vespel) ferrule on this end of the liner. If you are using a glass liner instead of stainless, a soft graphite (100% graphite) ferrule may be a better choice. A graphite ferrule is used on this end of the liner so the nut can slide off the liner.



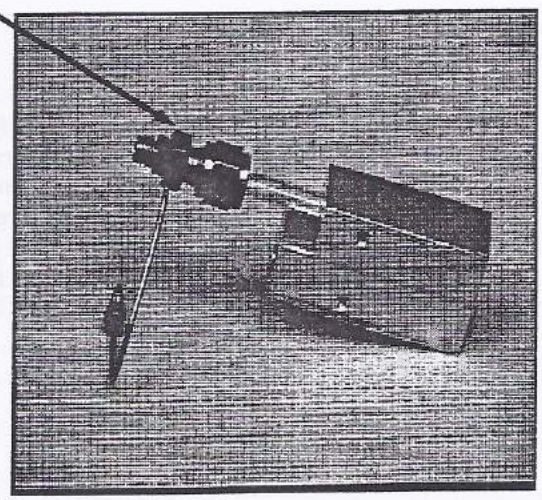
6) The injector liner and carrier inlet fitting can then be removed from the GC by pulling straight out towards the operator.

To replace the injection liner:

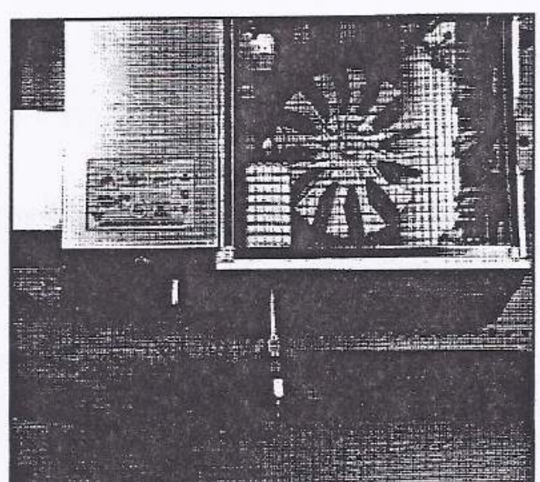


7) Using a 1/2" and 9/16" wrench remove the swagelok type nut securing the end fitting. The stainless steel liner provided as standard equipment with the split/splitless injector uses a brass ferrule on the septum end of the liner, but if you replace the stainless liner with a glass liner, you will need to use a 1/4" soft graphite ferrule instead.

Align the flat surfaces of the nut and fitting

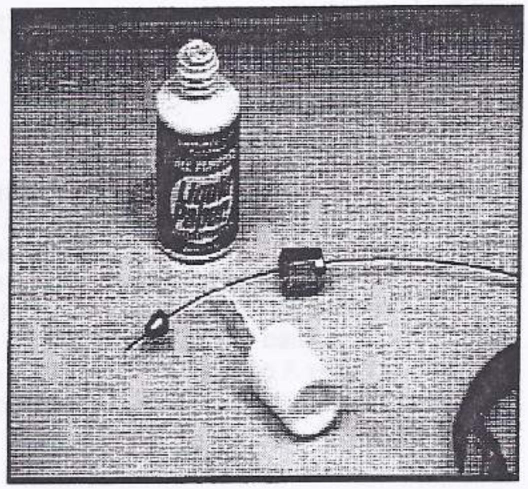


8) The glass liner and end fitting is shown here partially inserted into the heater block and removed from the GC for clarity. Be sure to align the flats on the nut and the fitting so that the carrier gas inlet tube will adopt the same orientation once the liner is fully inserted into the heater block.

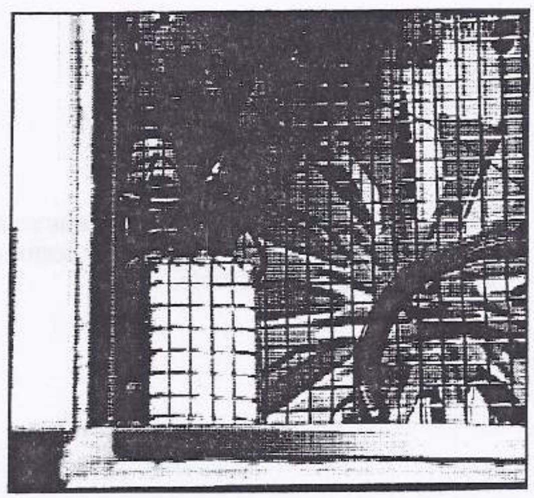


9) The injector liner and carrier inlet fitting can then be installed into the GC by sliding straight in towards the column oven

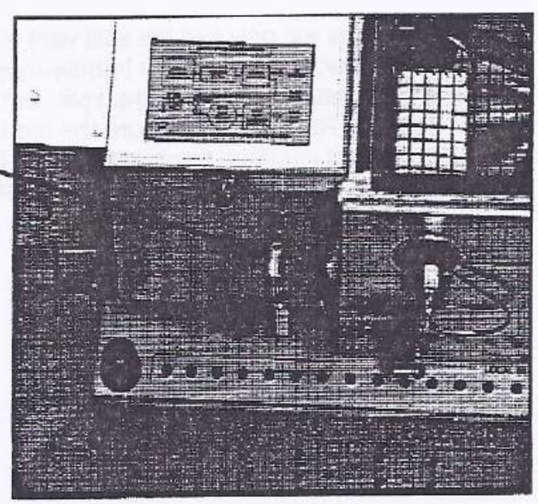
To install a narrow bore (.25mm) capillary column:



10) Use some white correction fluid to mark the column approximately 1.5" (4cm) from the end. Slip a 1/8" swagelok type nut and 1/8" to .5mm graphite reducing ferrule over the column. You can use soft or hard graphite ferrules.

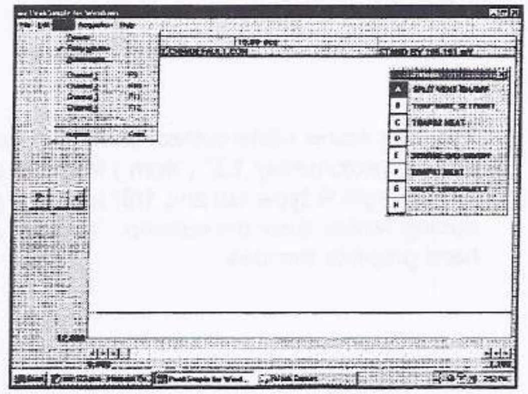


11) Using a 7/16" and 1/2" wrench secure the column into the injection liner so that the white mark on the column is just visible. The intent is to position the end of the column upstream of the split vent exit tube which is welded into the side of the end fitting.

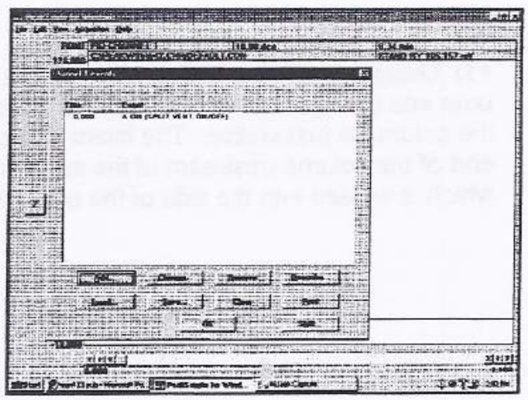


12) Adjust the split flow rate using the needle valve located on the front of the valve oven.

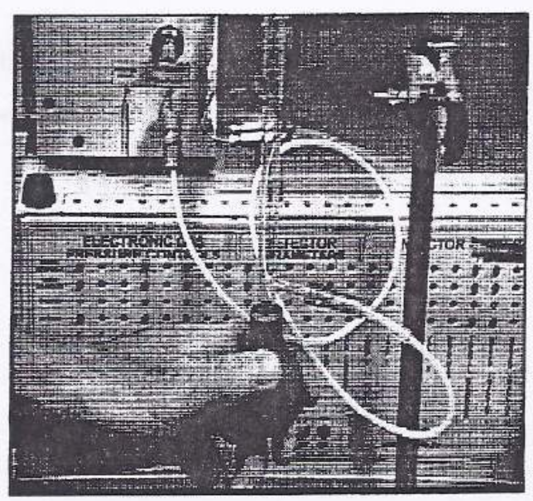
To install a narrow bore (.25mm) capillary column:



13) The split vent must be opened by activating one of the relay outputs from the PeakSimple data system. Typically Relay A is used to activate the split vent solenoid. If another relay has been allocated to this function, it will be annotated in the relay assignment chart located on the right hand side panel of the GC. Relay A can be turned on/off by displaying the relay window and then using the mouse to click on the letter A.



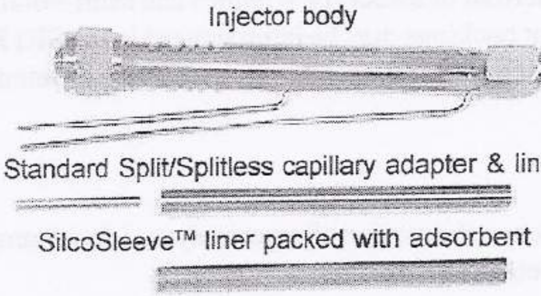
14) The relay can be turned on/off automatically during an analysis by entering the relay commands in the PeakSimple event table.



15) Carrier gas will only exit the split vent when Relay A is activated. Connect your bubble-meter or other flow measuring device to the split vent exit tube. Activate Relay A. Make sure the red lid of the GC is down (lid interlock disables solenoid function). Adjust the needle valve to obtain desired flow.

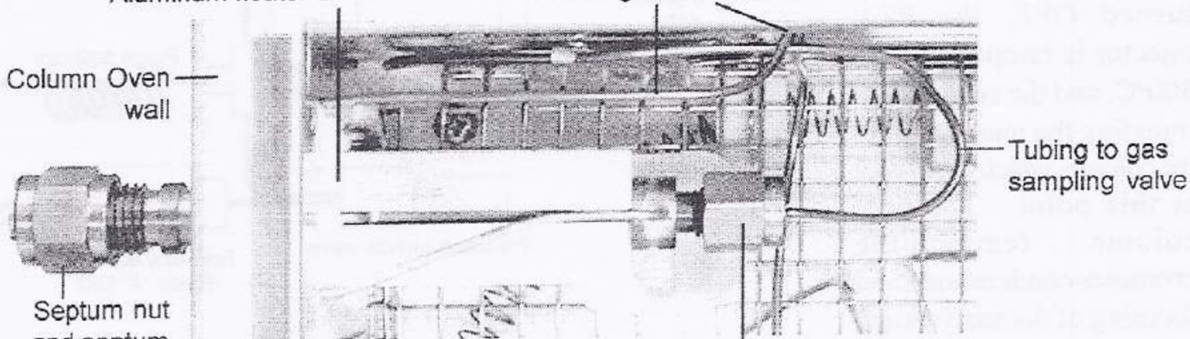
PTV - Programmed Temperature Vaporization Injector

Overview

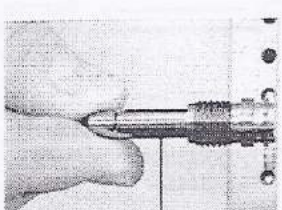


PTV and Split/Splitless injector components

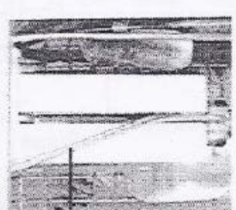
The Programmed Temperature Vaporization (PTV) injector is composed of the same parts as the Heated Split/Splitless injector: the injector body, a SilcoSleeve™ liner, an injector purge restrictor, a precision needle valve for adjustment of split flow rate, a split flow solenoid that turns on & off from the PeakSimple data system, and an aluminum heater block containing a heater cartridge and Type K thermocouple. Contrasted with the Split/Splitless injector, the PTV injector has a removable insulating sleeve, a larger (250 watts) heater cartridge with ballistic heating capability, and carrier flow ON/OFF control. The SilcoSleeve™ liner can be packed with a variety of optional adsorbents, depending on the application. The SRI PTV injector has three modes of operation: 1) large volume liquid injector, 2) an offline thermal desorber, or 3) an online thermal desorber in conjunction with a gas sampling valve.



End fitting where column connects or, as in this SRI Multiple Gas Analyzer #1, tubing to a gas sampling valve

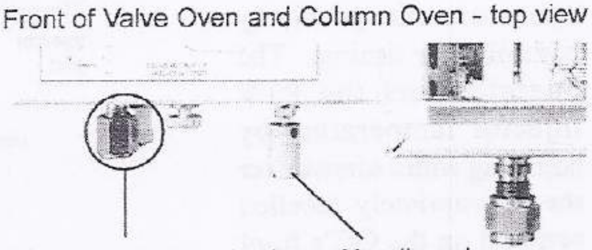


Tenax-GR™ packed SilcoSleeve™ liner, partially slid out for visibility



Split flow exits to purge vent and needle valve

Front of Valve Oven and Column Oven - top view



Sample loop inlet (top) and purge vent (bottom)

Needle valve precision control

INJECTORS
PTV - Programmed Temperature Vaporization Injector

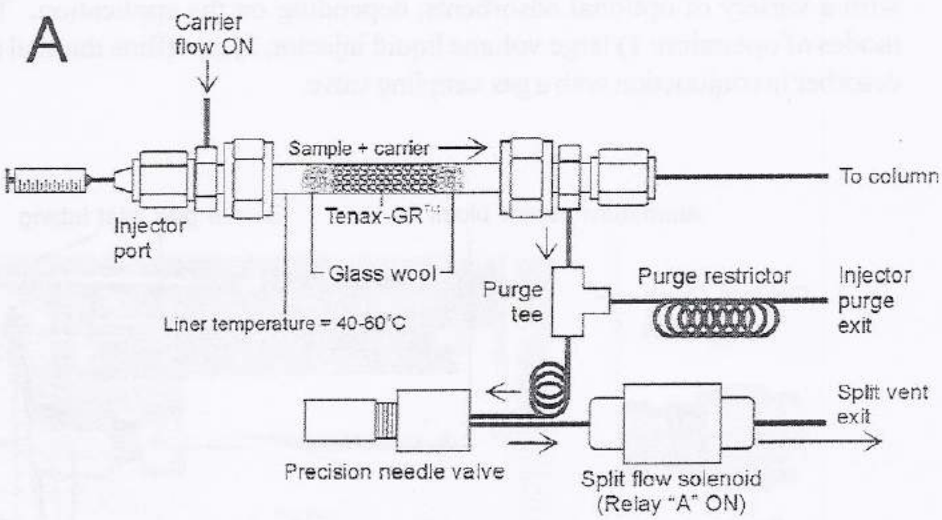
Theory of Operation

The Programmed Temperature Vaporization injector is basically a Heated Split/Splitless injector with the ability to rapidly heat to 300°C. This ballistic heating capability enables large volume liquid sample injections. The PTV injector can be used as a thermal desorber for volatiles and semi-volatiles, online or offline. Multiple liners with different adsorbent packings may be interchanged in the SRI PTV injector. The adsorbent used depends on the compounds of interest, as each has its own selective retention properties.

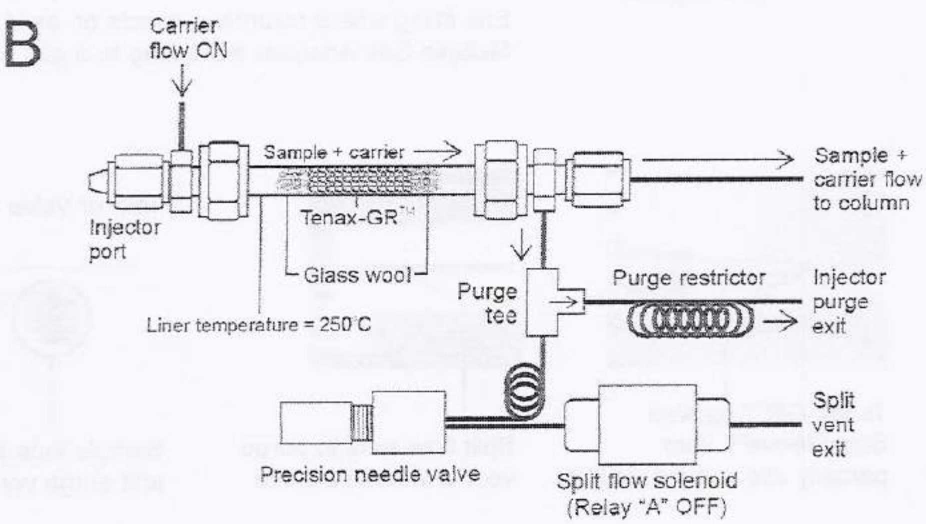
1) Large Volume Liquid Injector

Large volume injections allow analysis of samples with low concentration of target analytes. Liquid samples from 1µL to 200µL may be injected using the SRI PTV injector.

A. To begin, both the Column Oven and the PTV injector are held at 40-60°C. Prior to injection, the split vent is opened. Thus, the large volume liquid sample is injected into the PTV injector at 40-60°C with the split wide open. Introducing the sample at a low temperature allows the solvent to vent while the injector liner packing retains higher boiling point analytes.



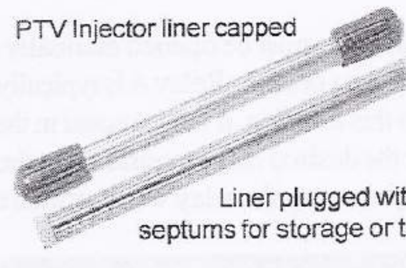
B. The split vent is then turned OFF, the PTV injector is ramped to 200-300°C, and the carrier flow transfers the analytes onto the column, which is still cool at this point. The cool column temperature promotes condensation and focusing of the analytes and helps prevent smearing and excessive tailing. Each of these events is automatically controlled through the PeakSimple data system, so operators can precisely control their timing. The operator sets the PTV injector temperature by adjusting with a screwdriver the appropriately labelled setpoint on the GC's front panel.



Theory of Operation continued

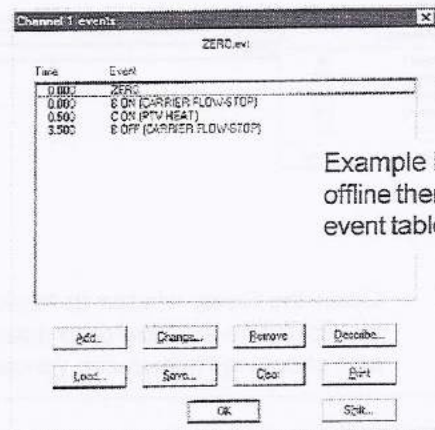
2) Offline Thermal Desorber

PTV Injector liner capped



Liner plugged with rubber septums for storage or transport

For offline thermal desorption, the SilcoSleeve liner packed with adsorbent such as Tenax-GR™ is loaded with sample outside of and separate from the GC. Although the best analysis is obtained from a fresh sample, the ends of the liner may be plugged after loading sample with rubber septa or capped with rubber end caps for storage or transportation. Turn off the flow before removing the injector liner by activating relay B, which stops the carrier gas flow. Leave the EPC flow off until the beginning of the analytical run (see the event table at right). To replace the liner, unscrew the septum nut and septum protruding from the front of the Column Oven wall. Remove the rubber septa or caps from the liner and slide it in with the gash toward the operator. Replace and close the septum and nut. With the carrier flow still turned OFF, start the run. When the PTV injector reaches temperature, the carrier flow is turned ON and the analytes are swept onto the column.



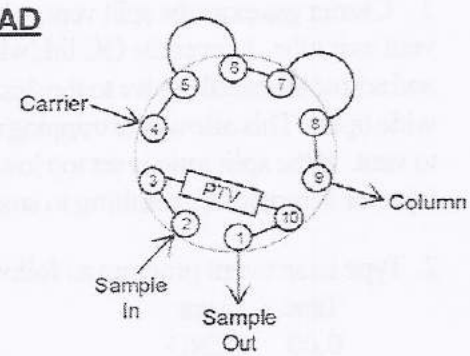
Example PTV as offline thermal desorber event table

3) Online Thermal Desorber

For online thermal desorption, the PTV can be plumbed with a gas sampling valve. In this mode of operation, the PTV functions as a sample loop, trapping and concentrating compounds for analysis.

LOAD Position:
(Relay "G" OFF)

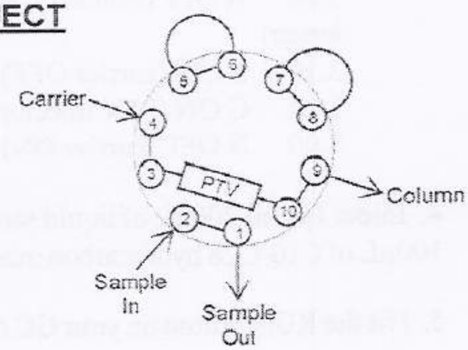
LOAD



When the gas sampling valve is in LOAD position, the PTV injector can be loaded with sample through the sample inlet and outlet. The PTV injector is at 40-60°C. Analytes are trapped in the injector's liner packing.

INJECT Position:
(Relay "G" ON)

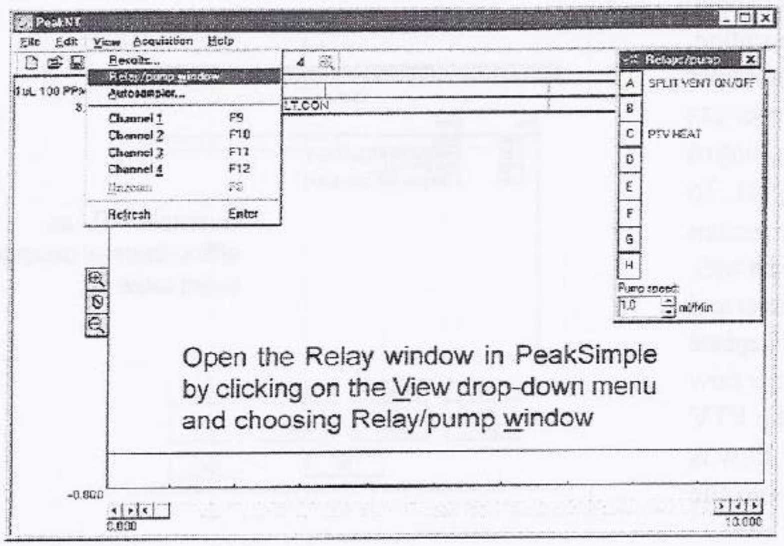
INJECT

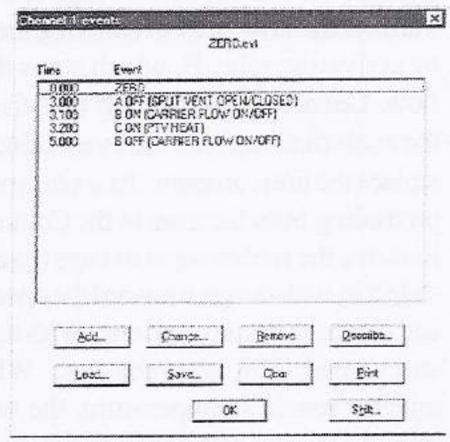


In the INJECT position, the PTV injector ramps to 300°C, vaporizing the sample. The carrier gas flow then flushes the desorbed components onto the column(s). The valve should be rotated back to the LOAD position after the components are transferred to the column to avoid smearing and peak tailing.

General Operating Procedure
Large Volume Liquid Injection Steps

1. The split vent must be opened manually prior to the run by activating one of the relay outputs from the PeakSimple data system. Relay A is typically used to activate the split vent solenoid. If another relay has been allocated to this function, it will be noted in the relay assignment chart located on the right hand side panel of the GC. Enter the desired relay commands in the PeakSimple Events table. The split vent can also be turned ON (or OFF) by opening the relay window then clicking on the letter A.





Example PeakSimple PTV event table

3. Carrier gas exits the split vent only when Relay A is activated. Connect a flow measuring device to the split vent exit tube. Lower the GC lid (when open, lid interlock disables the solenoid function), activate Relay A, and adjust the needle valve to the desired flow. For most liquid injections using a PTV, the split vent should be wide open. This allows the trapping material to retain the compounds of interest and quickly flush the solvent to vent. If the split ratio is set too low, some of the solvent and analytes may enter the column before the PTV injector is heated up, resulting in smeared or double peaks.

2. Type in an event program as follows:

Time	Event
0.00	ZERO
3.00	A OFF (split vent closed; if you get too large a solvent peak, keep the split vent open longer)
3.10	B ON (carrier OFF)
3.20	C ON (PTV injector heat)
5.00	B OFF (carrier ON)

4. Inject 1μL to 200μL of liquid sample into the PTV injector. In the "Expected Performance" example, 100μL of C10-C28 hydrocarbon mixture was injected.

5. Hit the RUN button on your GC or press the spacebar on your computer keyboard.

Expected Performance

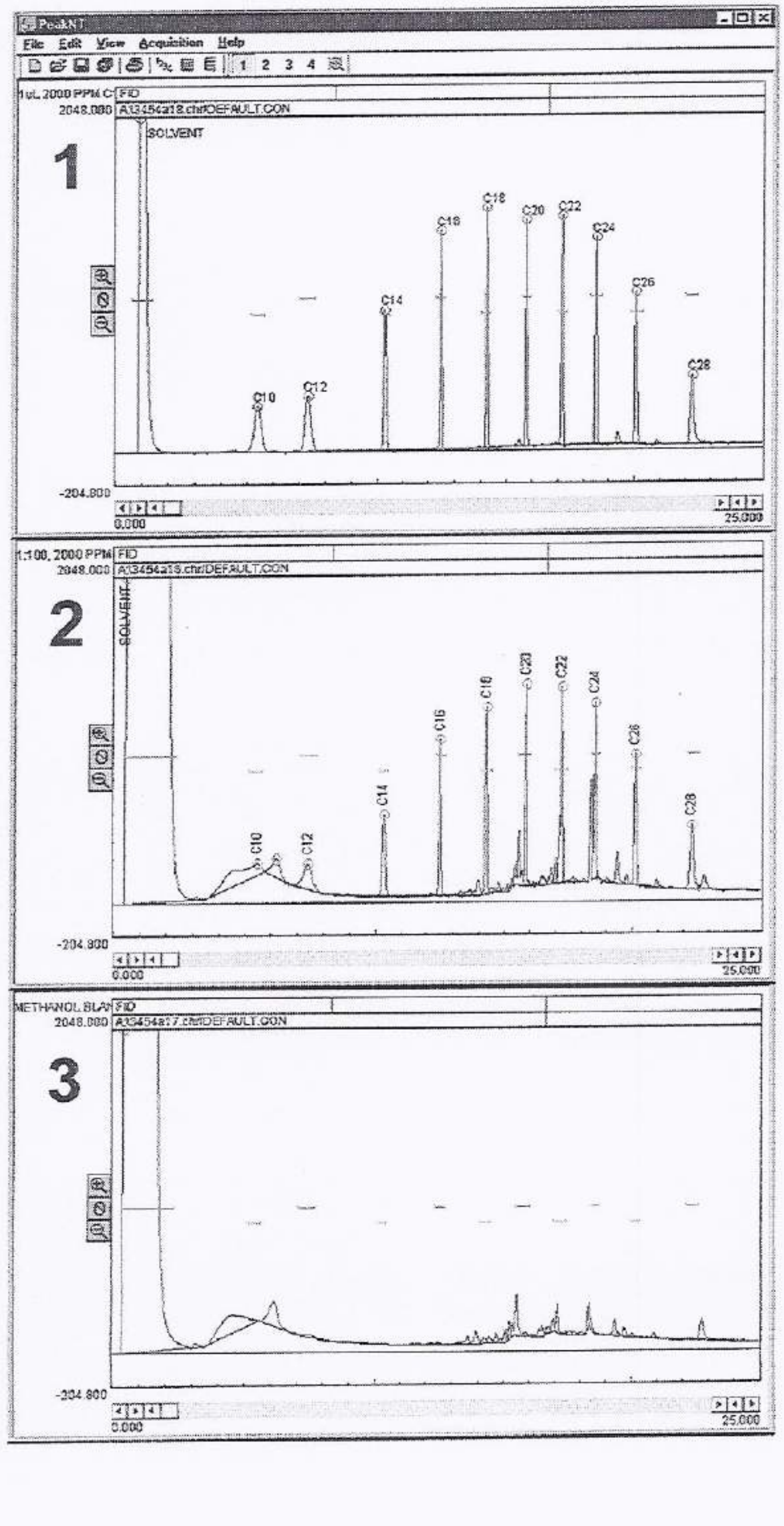
The following three chromatograms are from the FID in a SRI GC with a PTV injector upgrade. The liner was packed with 0.1 grams of Tenax-GR™ adsorbent. All three 25 minute runs utilized the same temperature and event programs. In the first one, a 1 µL 2000ppm C_{10}-C_{28} sample was injected through the PTV injector. In the second chromatogram, the same sample was diluted 1:100, then 100µl injected, achieving results consistent with the first run, and demonstrating the high volume liquid injection capability of the PTV injector. In the third chromatogram, 100µL of methanol was injected as a blank, resulting in a small hump between the 4 and 7 minute marks and miniscule peaks which correspond to contaminants in the methanol blank and bleed from the Tenax-GR™.

Chromatogram 1 Results:

Component	Retention	Area
Solvent	0.866	84953.1370
C10	5.366	5299.9150
C12	7.300	5034.0980
C14	10.233	4814.2000
C16	12.450	4600.0300
C18	14.216	4436.1780
C20	15.750	4528.2890
C22	17.150	4570.0975
C24	18.483	4778.9380
C26	20.033	4863.4290
C28	22.216	4135.4760
	Total	132013.7875

Chromatogram 2 Results:

Component	Retention	Area
Solvent	0.450	499472.8740
C10	5.433	2258.5340
C12	7.366	2614.0540
C14	10.266	3813.8985
C16	12.483	3924.8340
C18	14.266	3939.9080
C20	15.800	3933.0400
C22	17.200	4660.5860
C24	18.516	4737.3130
C26	20.083	4174.2920
C28	22.266	3260.1120
	Total	536789.4455



Temperature programs & events for all 3 runs:

PTV = 110°C (3min) to 275°C

Events: (A = split vent)

Time	Event
0.00	ZERO
3.00	A OFF
3.10	B ON
3.20	C ON
5.00	B OFF

Temperature program:

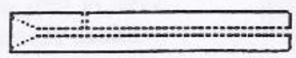
Initial	Hold	Ramp	Final
110°C	7.00	15.00	270°C
270°C	20.00	0.00	270°C

CONVERTING TO COLD ON-COLUMN MODE:

To set up the unit into a cold on-column mode, raise the red lid and adjust the injector temperature setpoint to 20° C. This will ensure that the injector does not heat itself but will be at the oven temperature. This temperature setpoint can be displayed on the digital display by turning the readout selector switch to INJECTOR SET.

When the injector and oven are cool, remove the oven lid. Remove the injector nut and 9.5mm septum (Alltech # 15428). Use a 7/16 inch wrench to loosen the nut that secures the column in place while holding the split vent fitting with a 1/2 inch wrench. Slide the column all the way through the injector until it is protruding out the front of the injector. Remove the 0.53mm I.D. capillary column adaptor from the holder in the oven lid and slip the column through the adaptor. Insert the 0.53mm I.D. capillary column adaptor all the way into the injector sleeve. The column should be inserted midway into the adapter. The adapter is then inserted into the injector so that the "funneled" end of the adaptor facing the needle is near the septum. The adaptor is vented so that carrier gas will flow to the column even if the adaptor is installed against the septum.

This end toward septum nut



0.53mm I.D. capillary column adaptor (part # 8670-9095)

Tighten the nut to secure the column in place while holding the split vent fitting with a 1/2 inch wrench. Replace the injector nut and 9.5mm septum making it finger tight.

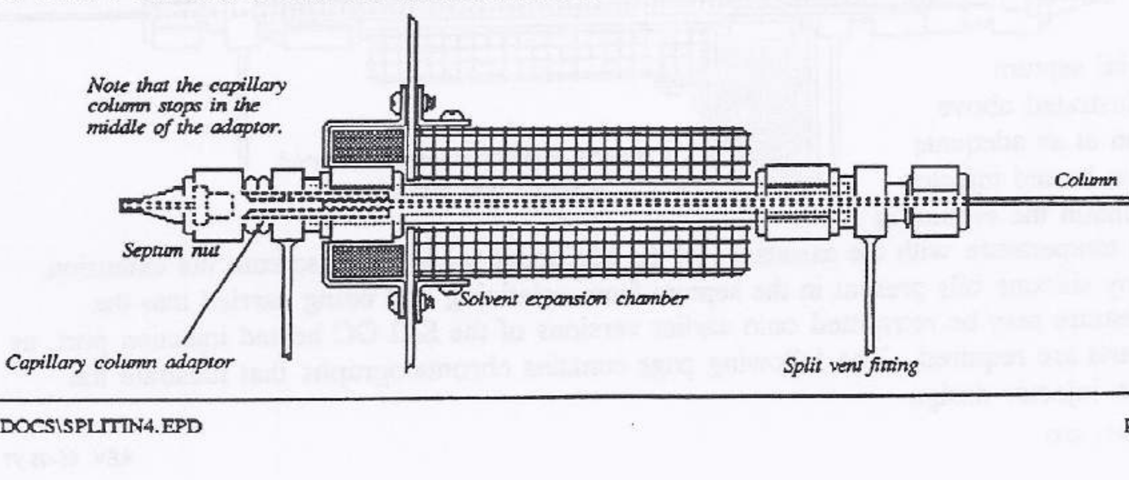
Use the toggle switch to select the flow controller to regulate carrier gas.

COLD ON-COLUMN OPERATION:

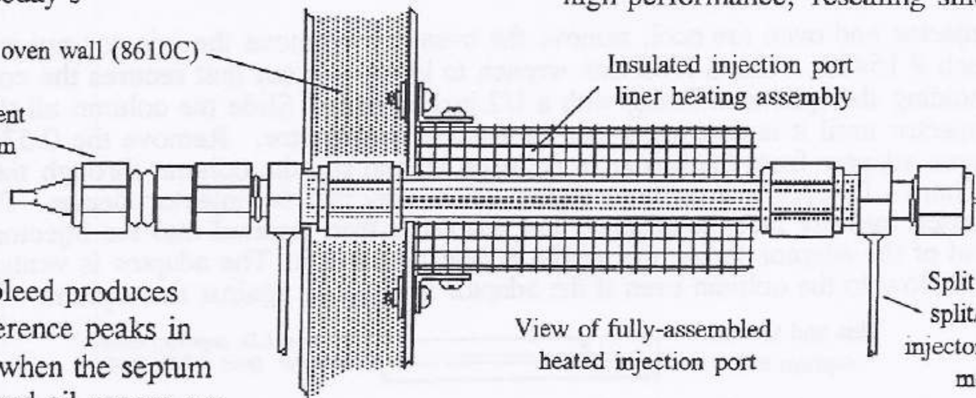
The GC is shipped configured in a cold on-column mode. This mode is the simplest to operate. The heated injector temperature setpoint is set to 20 degrees C. The flow controller is used to regulate the flow of carrier gas. A packed column can be directly connected to the back of the injector by removing the solvent expansion chamber. If a 0.53mm capillary column is used, it will be mounted in a capillary column adaptor (part # 8670-9095) which will allow direct injections onto the column. The adaptor is stored in a special holder in the back right corner of the red lid when not in use.

Unlike the split methods where much of the sample is lost, a cold on-column injection places all of the sample directly onto the column, therfore no sample is lost. Cold on-column injection method is ideal for samples of low concentration and gives the best sensitivity and sharpest peaks. The split vent is never opened for the cold on-column method.

CORRECT PLACEMENT OF COLUMN AND CAPILLARY ADAPTOR FOR COLD ON-COLUMN MODE:

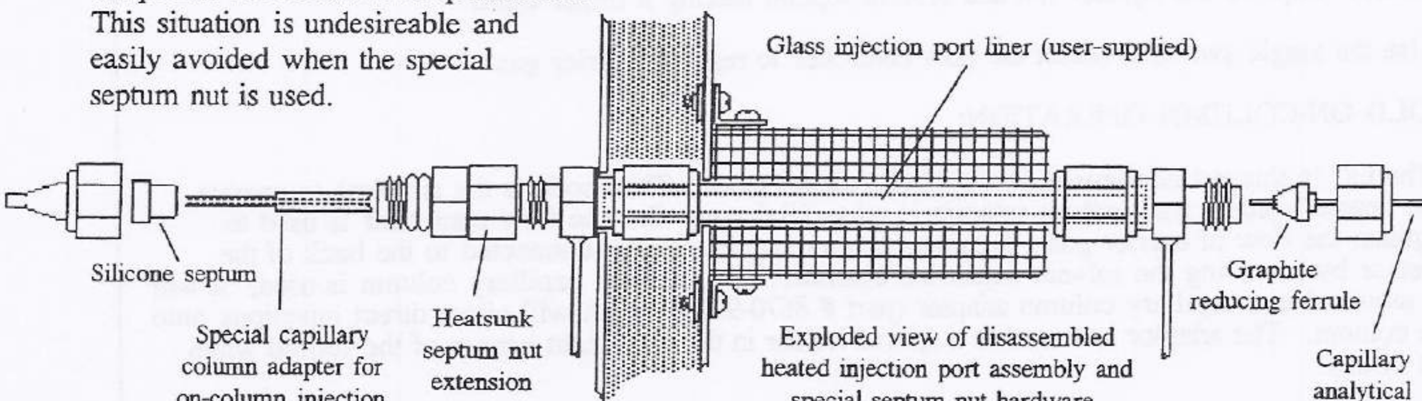


All SRI Instruments heated injection ports are equipped with a specially-designed septum nut which dissipates any heat that could be transfered from the heated injection port body (including split-splitless configurations), to the septum nut and septum by contact. Experience indicates that when injection ports are permitted to transfer heat to the silicone septum, that septum bleed can and does occur. Septum bleed is the volatization under heat of silicone oils used in the manufacture and formulation of today's high-performance, resealing silicone septa.

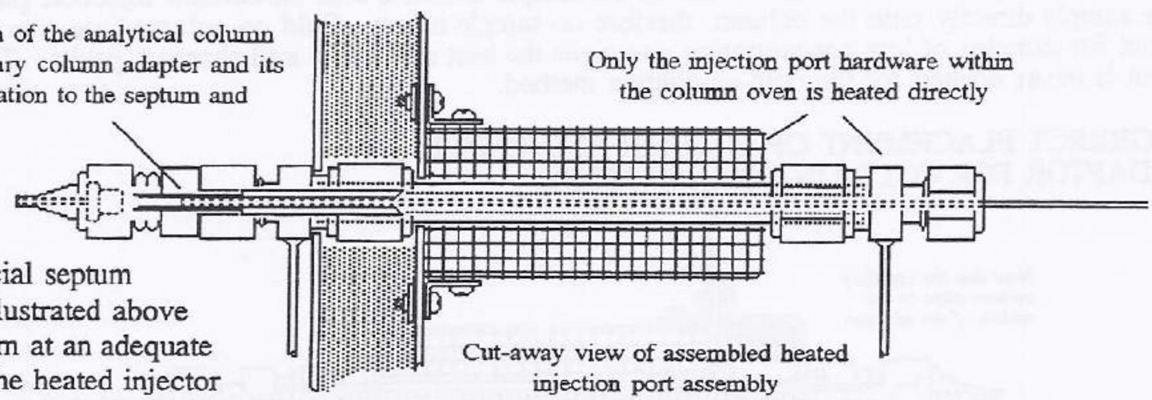


View of fully-assembled heated injection port

Silicone bleed produces numerous interference peaks in chromatograms when the septum exceeds 180°C and oil vapors are swept into the column and quantitated. This situation is undesireable and easily avoided when the special septum nut is used.



Exploded view of disassembled heated injection port assembly and special septum nut hardware



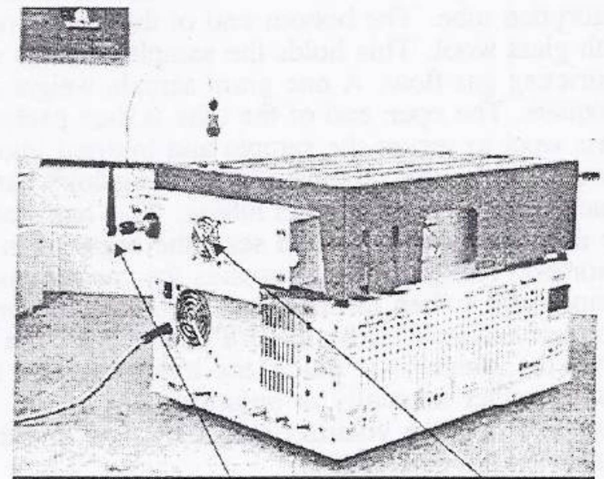
Cut-away view of assembled heated injection port assembly

The special septum nut assembly illustrated above keeps the septum at an adequate distance from the heated injector assembly to maintain the septum at or near ambient temperature with the assistance of the additional mass of the septum nut extension. This prevents any silicone oils present in the septum from volatizing and being carried into the column. This feature may be retrofitted onto earlier versions of the SRI GC heated injection port, as only two new parts are required. The following page contains chromatographs that illustrate the effect of this new injector design

Chapter: INJECTORS

Topic: THERMAL DESORBER OPERATION

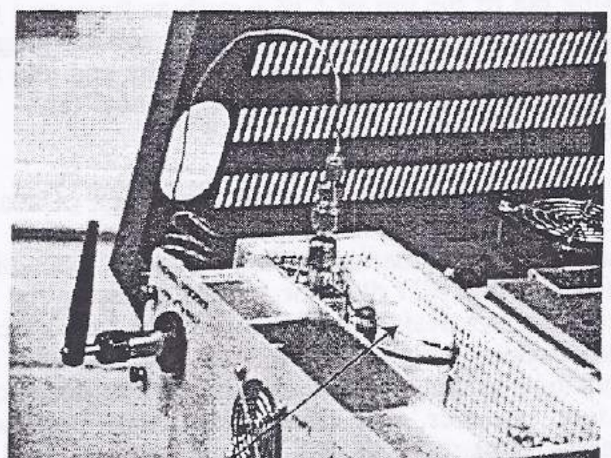
The SRI Thermal Soil Desorber accessory is useful for the analysis of volatile and especially semi-volatile compounds in soil or other granular solids. Because the analyte can be extracted from the soil by heat alone, with little or no sample preparation, field analyses can be performed without liquid solvent extraction. In addition, very high sensitivity for semi-volatile compounds such as diesel fuel can be obtained because essentially all the analyte is extracted from up to a gram of soil and deposited on column.



Valco Valve handle rotates to inject sample

Heated Valve Oven contains Thermal Desorber

The SRI Thermal Soil Desorber accessory is mounted in a heated valve oven on the left hand side of the 8610C Gas Chromatograph. The glass tube which contains up to a gram of soil is inserted into the hot (250 C) desorber cell through an opening in the top of the GC's red lid, and then secured by tightening the nut and 3/8" graphite ferrule. The handle of the manually operated Valco 10 port valve exits from the left rear of the heated valve oven, and is rotated to direct the carrier gas flow down and through the hot soil, transporting any hydrocarbons with boiling points below 300 C onto the GC column. The stainless steel tubing leading from the Valco valve to the column is routed and insulated to maintain a high temperature all along the path to the column oven to prevent high boiling compounds from condensing or tailing.



Transfer line from valve to column must be kept as hot as possible to avoid sample condensation. Arrange insulation to create "hot pocket" in this area.

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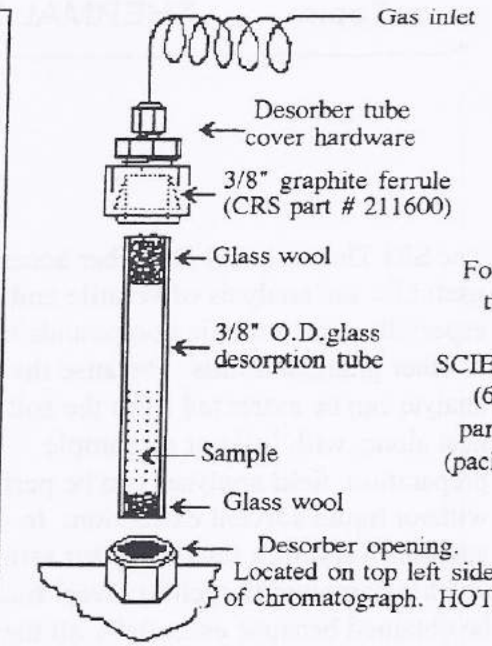
With the SRI 8610 Thermal Desorption unit, samples of soil or other solids can be analyzed for organic compounds without any extraction or other special sample preparation. The sample being tested is placed directly into the 3/8" O.D. machine glass desorption tube. The bottom end of the tube is plugged with glass wool. This holds the sample in place without restricting gas flow. A one gram sample weight is adequate. The open end of the tube is then packed with glass wool to secure the sample and inserted into the opening of the 3/8" stainless steel Swagelok® hardware attached to the pigtailed gas tubing. This hardware is the desorber tube cover and seals the organics in until desorbed. The gas tubing supplies the carrier gas. The sample tube is then inserted into the heated desorption chamber and secured by the 3/8" Swagelok® nut. When the sample is in place, the injection valve is rotated (either manually or automatically, if so equipped), and the volatized organics flow into the column on the carrier gas.

Historically, samples in soil have required solvent extraction with methlyene chloride, hexane, carbon disulfide or others prior to injection into a gas chromatograph. Unfortunately, solvent extraction often dilutes the sample and adversely affects detection limits. The detection limit for diesel fuel in soil by extraction is typically 10 ppm. When thermal desorption is employed, 10 ppb is attainable. With the phasing out of the use of CFCs such as freon and the ever-increasing scrutiny of laboratory solvent usage, the stripping of analytes from the soil by and into the column by thermal desorption is a practical (and sensible) alternative.

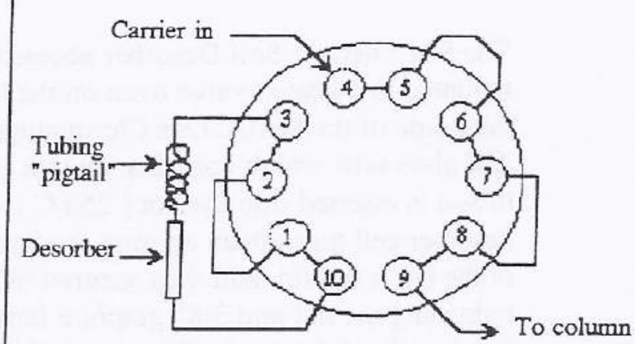
In the past, direct thermal desorption of average soil samples had been difficult due to the massive amounts of water liberated. This tended to extinguish the flame of the FID detector (typical detector for hydrocarbon analysis). Water elutes along with the early gasoline components and may interfere with the quantitation of benzene and toluene. Water does not interfere, however, with diesel quantitation because the diesel components elute well after the water.

The FID detector-equipped SRI 8610 gas chromatograph is supplied with an advanced design ceramic ignitor which can be run hot continuously, thus re-igniting the FID flame should it momentarily be affected by the passing water vapor. This minimizes the water interference and flame-out difficulties normally experienced with high moisture content samples analyzed with an FID detector.

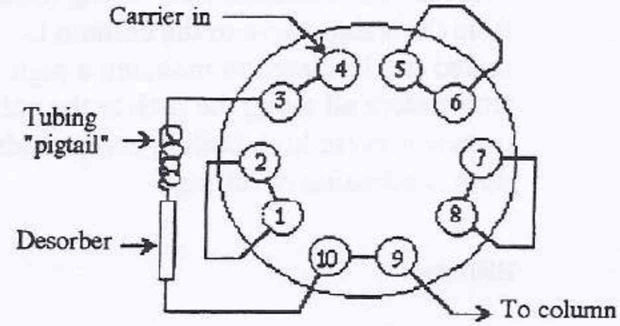


THERMAL DESORBER DETAIL

For replacement tubes, contact:
SANDFIRE SCIENTIFIC LTD.
(604) 826-5355
part no. S-9100
(pack of 10 tubes)



10 PORT VALVE DIAGRAM
"LOAD" POSITION



10 PORT VALVE DIAGRAM
"INJECT" POSITION

To operate the SRI model 8610 thermal desorption unit, the following steps are required:

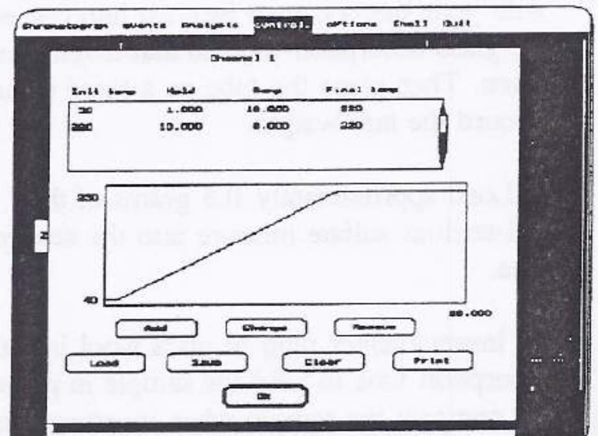
1. Place a clean desorption tube with a glass wool plug at one end on a scale of known accuracy. The tare weight is obtained. This is done by either weighing the clean, empty tube and recording the weight, or by placing the tube on the weighing platform and zeroing the balance.
2. Load the sample into the desorption tube and place the tube back on the balance. The gross or sample weight is recorded. The actual sample weight is obtained by subtracting the tare weight from the gross weight. A sample of solid weighing between 0.1 and 1.0 gm is recommended for best results. It is preferable to use a small sample due to the moisture that average samples contain. A small sample is less likely to interfere with the FID detector flame. A larger sample will permit the user to attain lower detection limits, but water content must be considered.
3. The tube containing the weighed sample is plugged with glass wool to hold the sample inside and the tube is inserted into the 3/8" opening of the Swagelok® hardware comprising the desortion tube cover. The end of the plugged tube is slid into the opening with the nut loosened. Once the tube has been inserted, the nut is tightened to seal the sample in the assembly.
4. Verify that the injection valve is in the "LOAD" position. Insert the sealed desorption tube assembly into the desorption chamber opening on top of the chromatograph and quickly secure it in place by tightening the Swagelok® nut at the opening. Care should be exercised when performing this step, as the desorption chamber is typically maintained at 350 degrees C and a burn potential exists.
5. Initiate the chromatogram either by keyboard or foot switch.
6. As soon as the desorption tube assembly has been secured into the desorption chamber, the injection valve is rotated to the "INJECT" position, and the sample is allowed to flow into the column. After the sample has desorbed completely, the valve is returned to the "LOAD" position. The tube may then be removed from the desorption chamber and cleaned. The contents of the tube should be removed and discarded. Once the tube has been thoroughly cleaned, it may be returned to service. If in doubt, a blank run should be used to verify that the tube has been cleaned adequately. Once the blank chromatogram is acceptable, the tube may be re-used for a subsequent sample.

Users may make their own tubes if so desired.

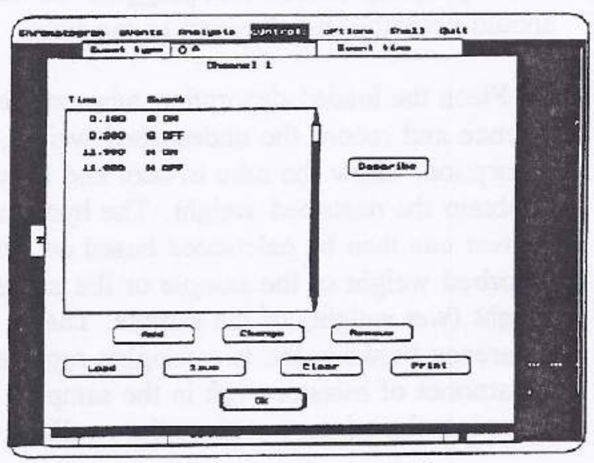
The column is connected to the injection valve inside the valve oven using a 1/16" to 1/8" adapter and 0.040" I.D. stainless steel tubing (1/16" O.D.). This ensures a uniform sample temperature while en route to the column and eliminates any possible cold spots.

The ignitor element may be set to 600°C (a dull red glow) for the duration of the run in order to avoid any possibility of FID flame-out should the sample have a high moisture content. The ignitor element can operate continuously at this high temperature without affecting its normal life expectancy.

Replacement desorber tubes may be ordered directly from Sandfire Scientific Ltd. in Mission, B.C., Canada at phone (604) 826-5355 (part no. S-9100).



EXAMPLE OF TEMPERATURE PROGRAM FOR DESORPTION



EXAMPLE OF TIMED EVENT TABLE FOR CONTROL OF AUTOMATED INJECTION

Chapter: INJECTORS & GAS VALVES

Topic: Thermal Desorber Soil Sample Preparation

1) To ensure that the soil sample analyzed is representative of the site sample, mix the soil in the sample container completely. Then weigh 10 grams of soil from the sample container into a 150 ml beaker.

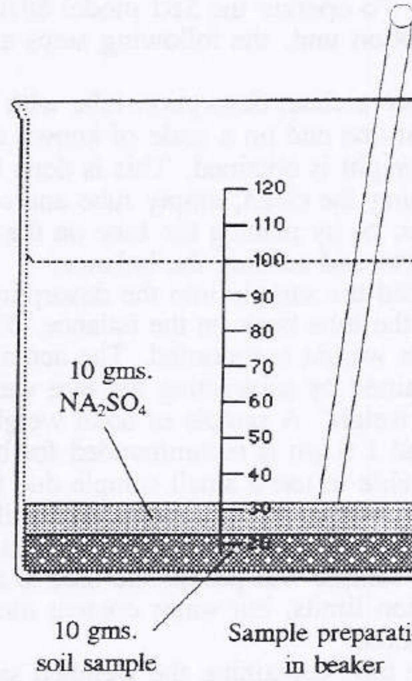
2) Add 10 grams of granular sodium sulfate (Na_2SO_4) to the beaker and mix with a stirring rod or spatula. The granular sodium sulfate, when mixed with the soil, absorbs most of the moisture from the soil, allowing clay soils to be ground into smaller particles. This is important because dense clay will not fully desorb. The mixture of soil and NA_2SO_4 should be of a granular consistency with small uniform particles.

3) Roll a small amount of glass wool into a ball with your fingers, then insert it into one end of the glass desorption tube so that it remains in place. Then place the tube on a tared balance. Record the tare weight.

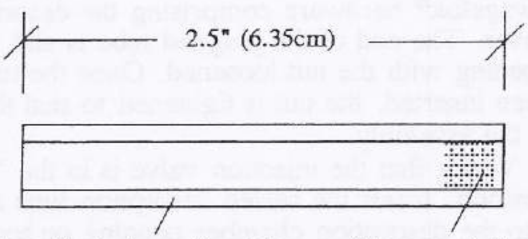
4) Load approximately 0.5 grams of the soil-sodium sulfate mixture into the desorption tube.

5) Insert another plug of glass wool into the desorption tube to hold the sample in place. Do not compact the sample when inserting the glass wool or the sample may not desorb thoroughly. When properly loaded and plugged, the tube should resemble the diagram to the right.

6) Place the loaded desorption tube on the balance and record the undesorbed weight. After desorption, allow the tube to cool and re-weigh to obtain the desorbed weight. The hydrocarbon content can then be calculated based on either the desorbed weight of the sample or the undesorbed weight (wet weight) of the sample. The difference between the two weights represents the amount of moisture left in the sample following the mixture with sodium sulfate.



Sample preparation in beaker



Placement of glass wool in desorption tube prior to sample insertion

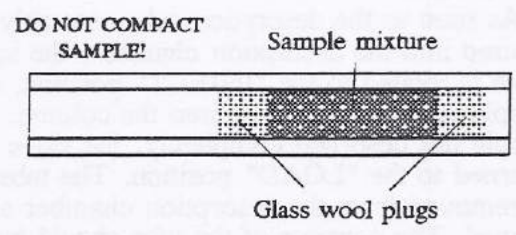


Diagram of assembled sample desorption tube containing 0.5 gms of soil - Na_2SO_4 sample mixture