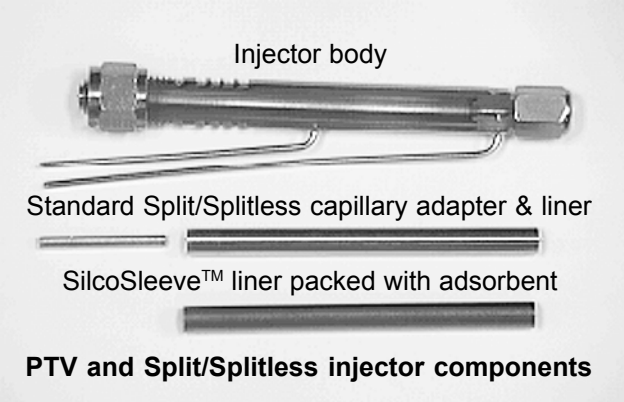
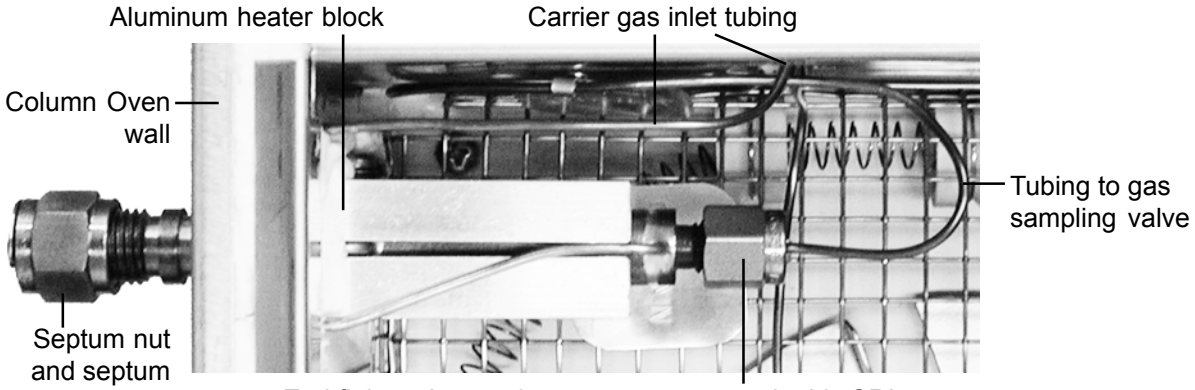
Overview

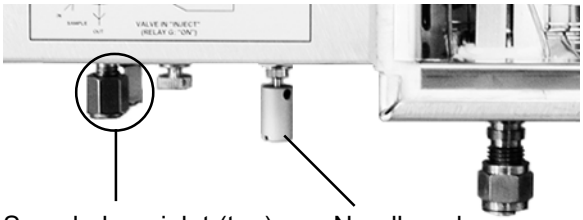


PTV and Split/Splitless injector components

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.



Front of Valve Oven and Column Oven - top view



INJECTORS
PTV - Programmed Temperature Vaporization Injector

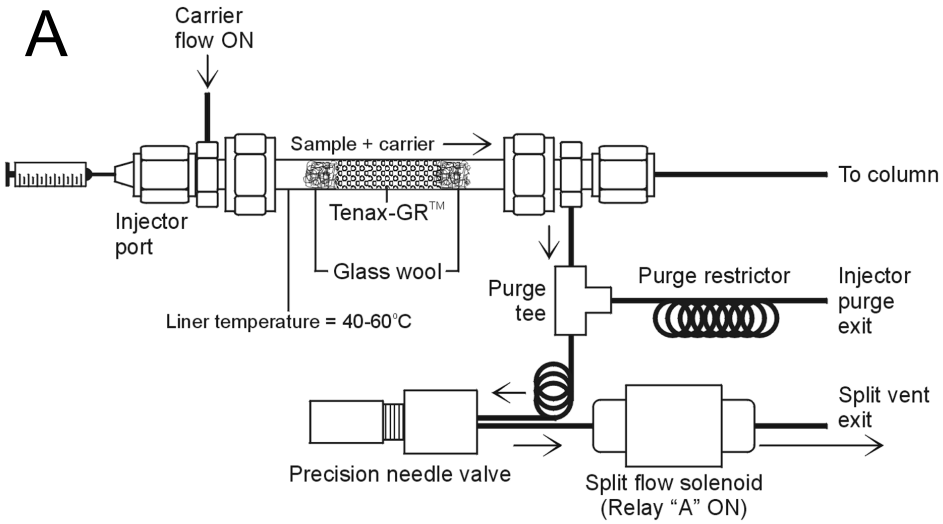
Theory of Operation

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.

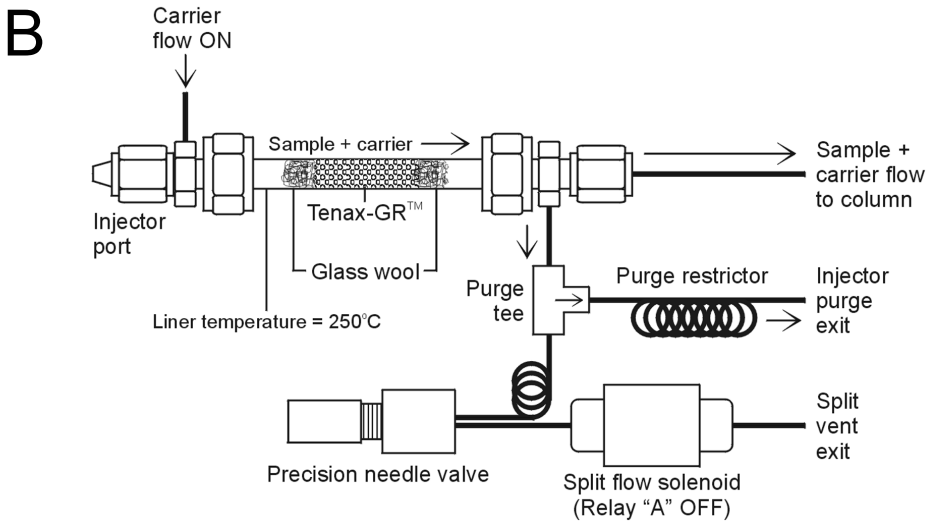
1) Large Volume Liquid Injector

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.



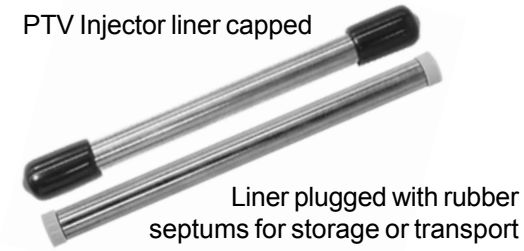
B. The split vent is then turned OFF, the PTV injector is ramped to 200-300°C, and the carrier flow transfers the analytes onto the column, which is still cool at this point. The cool column temperature promotes condensation and focusing of the analytes and helps prevent smearing and excessive tailing. 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.

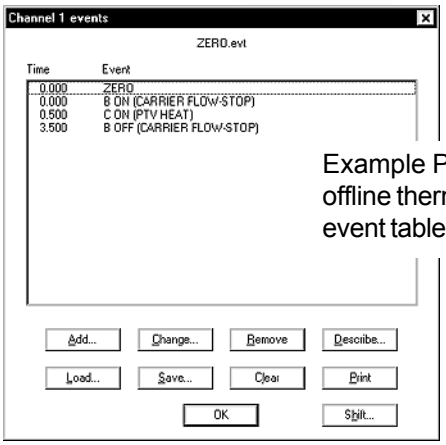


Theory of Operation continued

2) Offline Thermal Desorber

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.

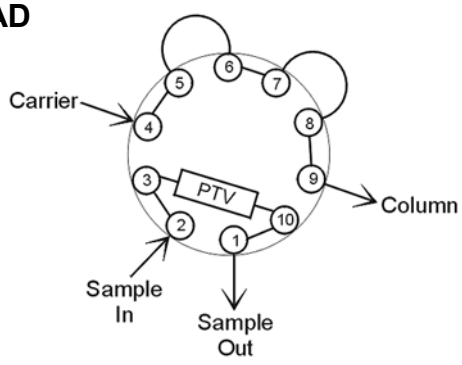
LOAD Position:
(Relay "G" OFF)

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.

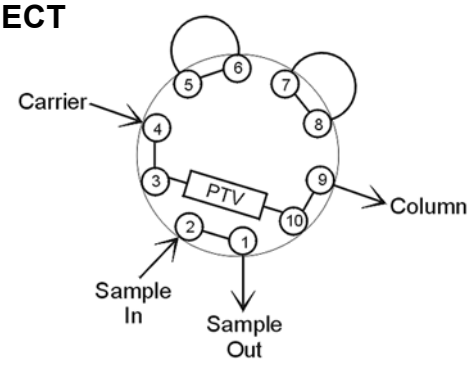
INJECT Position:
(Relay "G" ON)

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.

LOAD



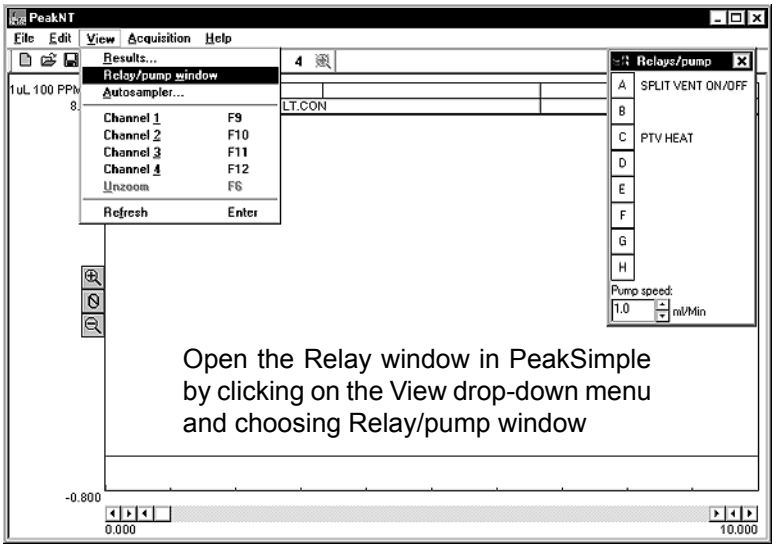
INJECT

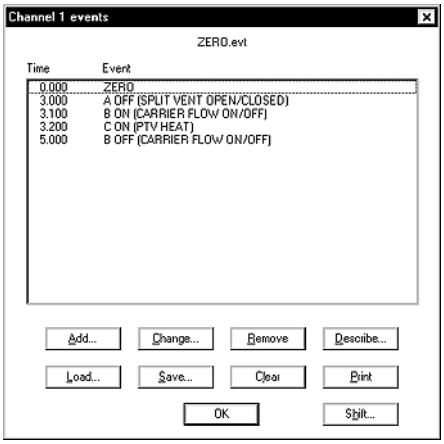


INJECTORS
PTV - Programmed Temperature Vaporization Injector

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:

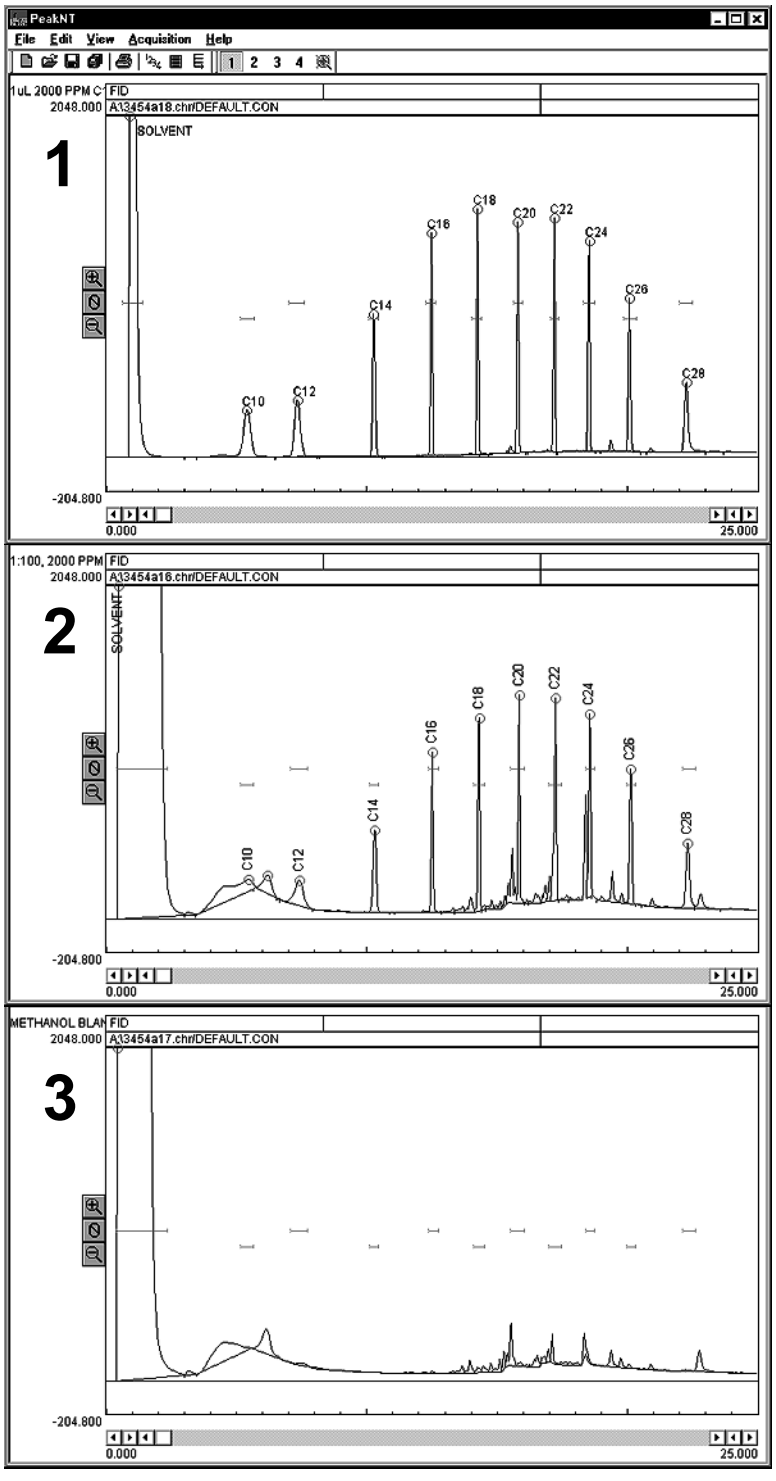
Time	Event
0.00	ZERO
3.00	A OFF (split vent closed; if you get too large a solvent peak, keep the split vent open longer)
3.10	B ON (carrier OFF)
3.20	C ON (PTV injector heat)
5.00	B OFF (carrier ON)

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 1µL 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