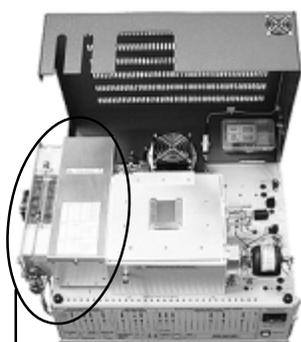


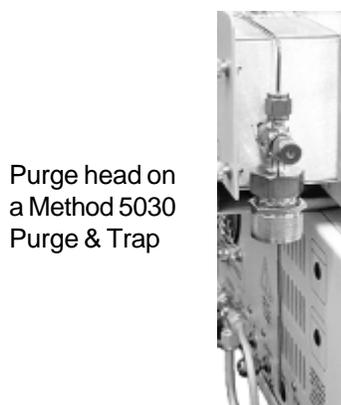
Overview

Built-in to the 8610C gas chromatograph, the SRI Purge & Trap is designed for compliance with EPA Methods 5030 and/or 5035 for the extraction of volatile organic compounds from water or soil samples. The purge and trap technique is applicable to a range of molecules from C₃ to C₁₂. The Purge & Trap hardware consists of a 10 port valve in a heated, ducted valve oven, two traps, a cooling fan, and the purge head(s). The unique dual trap design enables the simultaneous trapping of compounds with different boiling points. Each trap has its own heater, and the ends of the traps are enclosed in the valve oven ducts to prevent cold spots. The cooling fan maintains the adsorption temperature and rapidly lowers trap heat after desorption. The trap in the lower position (TRAP 1) is usually packed with Tenax™-GR at the factory, while the upper trap (TRAP 2) is left empty for the user to pack with the desired adsorbent. A Carbosieve™ packed trap is also shipped with the GC for optional installation in the TRAP 2 position. The Carbosieve trap is used only when the analysis includes light gaseous VOC's, the most common being vinyl chloride. The Method 5030 Purge & Trap is the standard model with a fixed purge head that uses disposable 16mm test tubes for ambient temperature purging. There is a built-in septum port on this purge head through which gas standards may be spiked. The

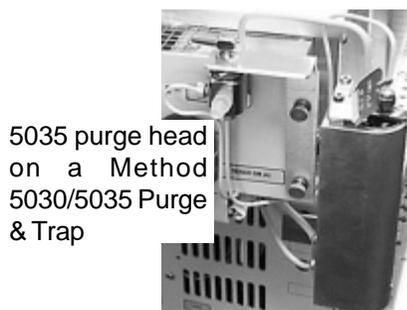


SRI GC equipped with Method 5030 Purge & Trap

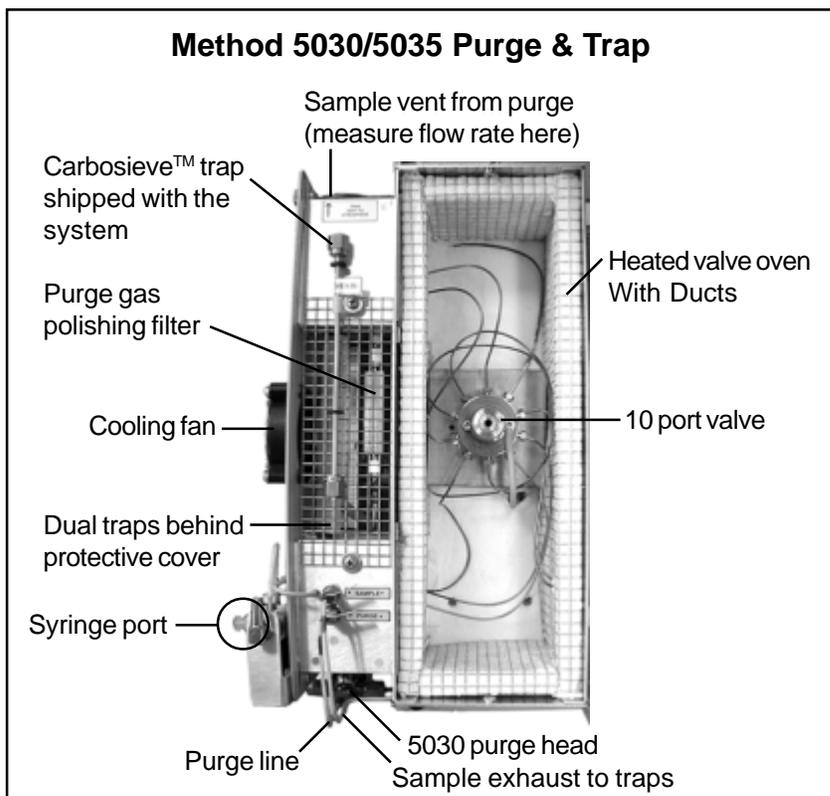
Method 5030/5035 Purge & Trap features interchangeable purge heads. The 5035 purge head is a thermostatted heater body (from ambient to 50°C) which accepts standard 40mL VOA vials. Inside the heater body are two needles which puncture the septum: the longer one bubbles helium purge gas through the sample, while the shorter needle exhausts sample-laden gas to the adsorbent traps. In compliance with EPA Method 5035, the purge head is mechanically agitated while the sample is being purged. There is a syringe port on the Method 5030/5035 Purge & Trap that allows water and internal standard to be added to the sample in the vial without puncturing the septum again. Operation of the Purge & Trap is automated by the PeakSimple data system.



Purge head on a Method 5030 Purge & Trap



5035 purge head on a Method 5030/5035 Purge & Trap

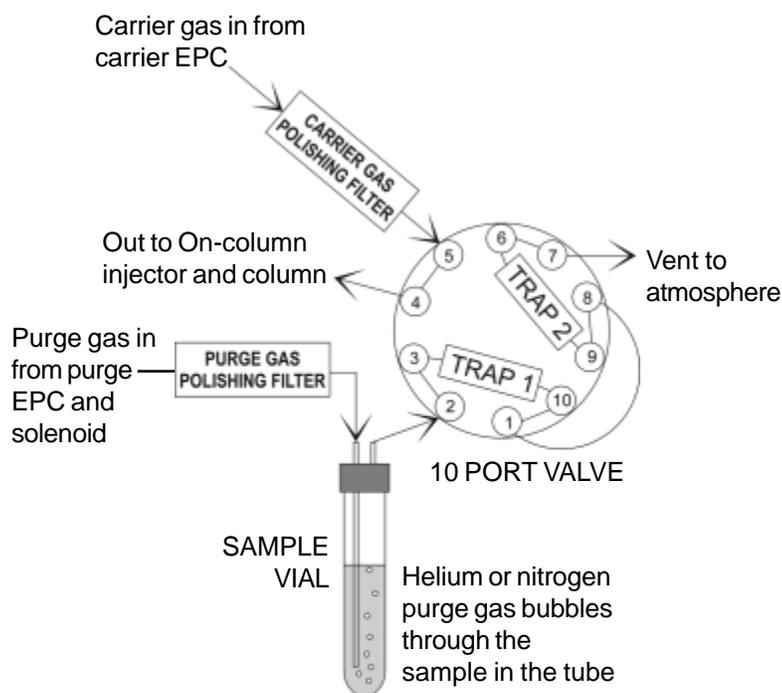


INJECTORS

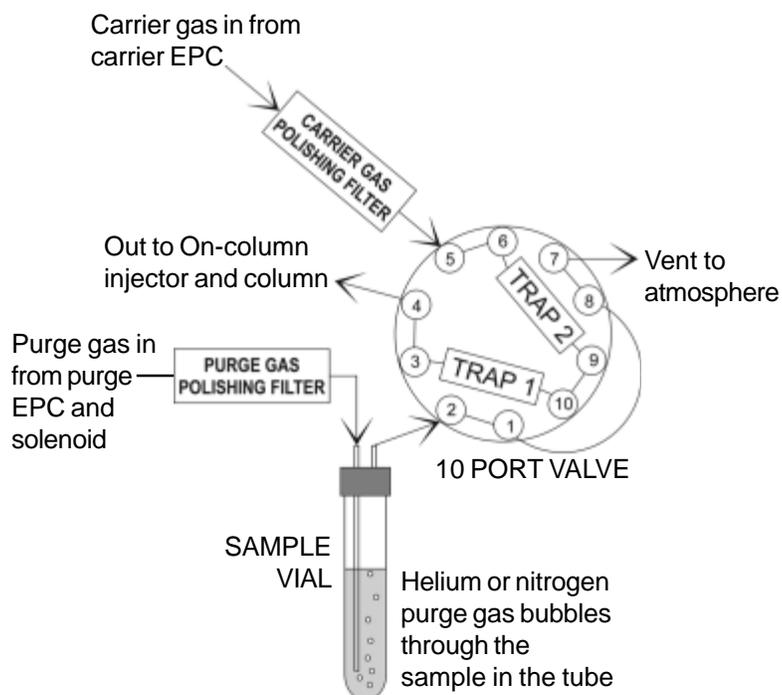
Purge & Trap

Theory of Operation

Purge & Trap Valve in the LOAD Position



Purge & Trap Valve in the INJECT Position



The SRI Purge & Trap uses a 10 port gas sampling valve and dual adsorbent traps. Each trap has independent adsorption and desorption setpoints to optimize the analyte trapping and releasing from each adsorbent.

When the valve is in the LOAD position, the sample-laden purge gas from the test tube or VOA vial is directed through the two traps, then out to vent, loading the traps with sample at the adsorption temperature (30-40°C). In this position, the carrier gas merely enters and exits the valve.

After a period of time sufficient for the traps to reach desorption temperature (200°C), the valve is actuated to the INJECT position. In the INJECT position, the carrier gas flows through the traps in the direction opposite to the sample-laden purge gas flow with which the traps were loaded. The carrier gas backflushes desorbed analytes into the column, while the purge gas flows out to vent.

The valve remains the INJECT position for the optional bake cycle, during which the respective desorption temperatures of both traps are raised an additional 50°C, and the purge gas polishing filter is reconditioned. A relatively high flow of purge gas sweeps through the hot polishing filter, which heats whenever TRAP 1 heats. This purge gas flow sweeps contaminants from the polishing filter and out to vent.

The valve is then actuated back into the LOAD position, TRAP 1 and the polishing filter heat are turned OFF, followed by TRAP 2, then the purge gas (see the Event Table on the **General Operating Procedures** page.)

Trap heating, valve rotation, and purge gas control are automated through the PeakSimple data system.

Sample Preparation

Sample preparation depends on the sample type, concentration, amount, etc. The third edition of SW-846 from the EPA is accessible on the Internet. Go to <http://www.epa.gov/epaoswer/hazwaste/test/main.htm> and click on the **5000 Series** link to download Methods 5030 and 5035.

Method 5030

Method 5030 style purge and trap is for the analysis of VOCs in aqueous samples. This purge and trap technique is limited to analytes that purge efficiently from water. 10mL of the sample is placed in a clean test tube. The test tube headspace will contain ambient air, so if your laboratory or work area is not free of solvent fumes, they will show up in your chromatogram.

For aqueous samples:

1. Insert a 10mL aliquot of the aqueous sample into a clean test tube.
2. Plug the test tube opening with your thumb and shake it until the contents are evenly dispersed.
3. Quickly slide the test tube over the purge gas tubing and into the purge head, and tighten it in place with the knurled retaining nut.
4. Immediately begin the analysis by pressing the RUN button on the front of the GC or by pressing the spacebar on your computer keyboard.

For medium concentration soil samples, do a quick methanol extraction:

1. Place 10g of sample into a clean glass container. Add 20mL of methanol and shake it for 1-3 minutes.
2. Allow the soil to settle, then pull 100 μ L of the liquid solution into a glass syringe and inject it into the test tube containing 10mL of organic free reagent water.
3. Plug the test tube opening with your thumb and shake it until the contents are evenly dispersed.
4. Begin the analysis. You may need to dilute the sample more or less, depending on the concentration.



Method 5035

Method 5035 style purge and trap is for the analysis of VOCs that are purgeable from soil at 40°C. This method does not allow the VOC's to escape the VOA vial until it is punctured by the 5035 purge head needles. Approximately 5g of soil, weighed in the field at the time of collection, is sealed in a pre-weighed, septum-sealed, screw-top VOA vial containing a preservative solution. There is no need to insert a magnetic stirring bar since the SRI purge and trap mechanically agitates the VOA vial during the analysis. Organic-free reagent water, surrogates, and internal standards (if applicable) are added through the syringe port immediately before beginning the analysis.

1. Insert the VOA vial containing 5g of soil and 5mL of reagent water into the Method 5035 purge head.
2. Using the syringe port, inject 5mL of organic free reagent water, internal standards, and surrogate compounds into the VOA vial.
3. Begin the analysis by pressing the RUN button or the computer keyboard spacebar.

INJECTORS

Purge & Trap

General Operating Procedures

The following are generalized operating guidelines for the SRI Purge & Trap system.

1. The purge gas flow is controlled with an Electronic Pressure Controller (EPC). Set the purge flow (measurable at the trap vent at the rear of the purge and trap system). 40mL/min is a typical purge flow. The pressure required for 40mL/min through a single Tenax trap is printed on the right panel of the GC. If you install the optional Carbosieve trap or another adsorbent trap in the TRAP 2 position, you will need to raise the pressure to maintain the flow. **NEVER use hydrogen as a purge gas.** SRI recommends helium purge gas.

2. TRAP 1 is in the lower position in the Purge & Trap, and TRAP 2 is in the upper position. The trap temperatures are factory set at 200°C for desorption and may be adjusted using the trimpot setpoints on the top edge of the GC's front control panel. For adsorption temperatures, trap 1 is set at 30°C and trap 2 is set at 35°C. Trap heating will be controlled by the timed Event Table during the run. **Note:** the actual trap temperatures typically run 5°C over the setpoint. See the information and instructions on the following 2 pages for adjusting the trap adsorption temperature settings.

EVENT TIME	EVENT	EVENT FUNCTION
0.000	ZERO	Zero signal
0.100	E "ON"	Purge "ON"
0.200	(D "ON")	Shaker "ON"
5.000	(D "OFF")	Shaker "OFF"
5.100	E "OFF"	Purge "OFF"
6.000	C "ON"	Trap 2 (heat) "ON"
6.050	F "ON"	Trap 1 (heat) "ON"
8.000	G "ON"	Valve in "INJECT"
12.000	E "ON"	Purge "ON"
12.900	B "ON"	Trap set "ON" (+50°C)
13.000	G "OFF"	Valve in "LOAD"
14.900	F "OFF"	Trap 1 "OFF"
15.100	C "OFF"	Trap 2 "OFF"
15.300	E "OFF"	Purge "OFF"
15.500	B "OFF"	Trap set "OFF" (+0)

3. Set the valve oven temperature to 100°C or higher to avoid water condensation. If you're using Method 5035, set the purge head heater body temperature to 40°C. It is factory set to 40°C but is user adjustable.

4. Load or create an event table that is appropriate to the sample to be analyzed, or that is designed for compliance with a particular EPA Method. The valve oven in your Purge & Trap system is labeled with a typical Purge & Trap event table for a single Tenax trap. The event table shown above is an example for both methods; the only difference is that Method 5030 does not use Relay D (the sample vial shaker).

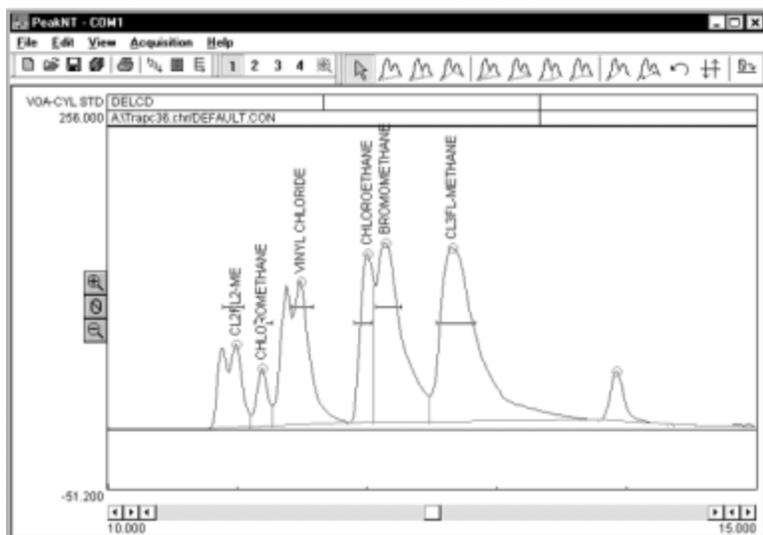
5. Load or create an appropriate temperature program for the column oven. **Epap&t.tem** is a typical Purge & Trap temperature program file provided with the PeakSimple software. As a basic rule for good separation, the column oven should be kept at 40°C for 10-12 minutes: 6 minutes while the sample is purging plus 4-6 more minutes after the valve actuates to the INJECT position.

6. Activate and energize the detectors as necessary. For instance, if you had an Environmental GC system, you would turn on the PID lamp current, light the FID flame, and set the DELCD reactor temperature. Choose the detector gain settings according to the analysis. Consult the manual sections for your particular detector(s) operating procedures.

7. When the system is at temperature and displaying a stable signal, insert the sample test tube or VOA vial into the purge head and begin the analysis.

General Operating Procedures Continued Using Two Traps

The SRI dual trap design gives the Purge & Trap user many options to effectively trap and release analytes from a particular adsorbent. Due to its low affinity for water, Tenax™-GR is especially useful for the purging of VOCs from aqueous samples, making it a good general purpose trap for EPA style purge and trap techniques. The Carbosieve™ packed trap is very retentive for light hydrocarbons, but since it tends to retain water and smear the other peaks, it should only be used when vinyl chloride is among the target analytes. This tendency to smear may be reduced by manipulating the desorption times for the two traps. If the Carbosieve™ trap (TRAP 2) is desorbed while the Tenax™-GR trap (TRAP 1) is still cold, the components will refocus on the Tenax™-GR. The Tenax™-GR trap is then heated to desorb all the components, which results in sharper peaks on the chromatogram. These two chromatograms are from an EPA style purge and trap analysis. The first one was made using Epa&t1c.evt for one trap. The peak separation is much better on the second chromatogram, which was made using Epa&t2c.evt for two traps.

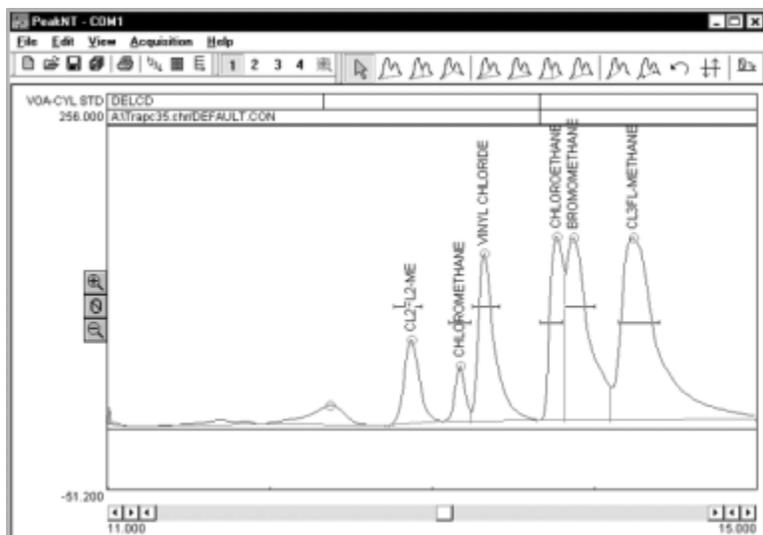


Sample: VOA-CYL standard
Method: 5030 P&T injection
Column: 60m MXT-1

DELCD gain: MED
DELCD temp: 260

Epa&t1c.evt		
EVENT TIME	EVENT	EVENT FUNCTION
0.100	E "ON"	Purge "ON"
5.100	E "OFF"	Purge "OFF"
6.000	C "ON"	Trap 2 (heat) "ON"
6.100	F "ON"	Trap 1 (heat) "ON"
8.000	G "ON"	Valve in "INJECT"
12.000	E "ON"	Purge "ON"
13.000	G "OFF"	Valve in "LOAD"
13.100	B "ON"	Trap set "ON" (+50°C)
14.900	F "OFF"	Trap 1 "OFF"
15.050	E "OFF"	Purge "OFF"
15.100	C "OFF"	Trap 2 "OFF"
15.200	B "OFF"	Trap set "OFF" (+0)

Temperature program:
Initial Hold Ramp Final
35°C 25.00 0.00 35°C



Dual Trap Event Table (Epa&t2c.evt)		
EVENT TIME	EVENT	EVENT FUNCTION
0.000	ZERO	Zero signal
0.100	E "ON"	Purge "ON"
5.100	E "OFF"	Purge "OFF"
6.000	C "ON"	Trap 2 (Carbosieve) heat "ON"
8.000	G "ON"	Valve in "INJECT"
8.100	F "ON"	Trap 1 (Tenax-GR) heat "ON"
8.500	G "OFF"	Valve in "LOAD"
10.000	G "ON"	Valve in "INJECT"
12.000	E "ON"	Purge "ON"
13.000	G "OFF"	Valve in "LOAD"
13.100	B "ON"	Trap set "ON" (+50°C)
14.900	F "OFF"	Trap 1 "OFF"
15.000	E "OFF"	Purge "OFF"
15.100	C "OFF"	Trap 2 "OFF"
15.200	B "OFF"	Trap set "OFF"