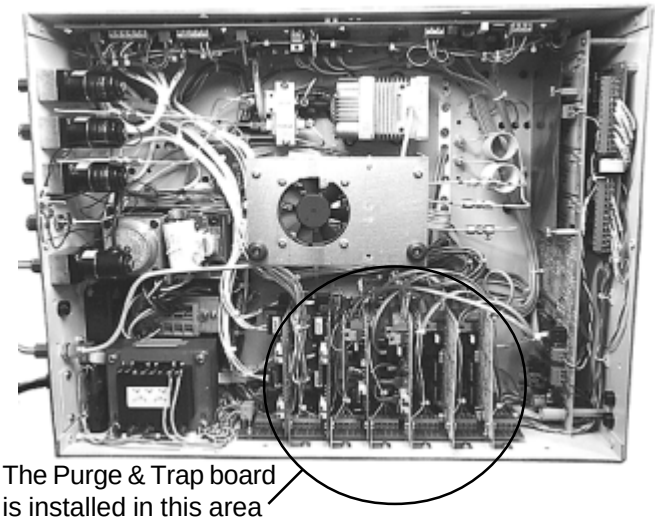
## *General Operating Procedures continued*

### Adjusting Trap Adsorption Temperatures

During the purge and trap process, the purge gas carries significant amounts of water into the traps. The Tenax™ trap is unaffected, due to its low affinity for water. The Carbosieve™ packing tends to retain the water, resulting in a large water peak at desorption. Adsorption settings can be adjusted by the user to set the Carbosieve trap at a high enough temperature to avoid water retention. However, this temperature may be too hot to trap target analytes. Therefore, experiment to find the adsorption temperatures that work best for your analyses. Once pinpointed, they usually require no further adjustment.

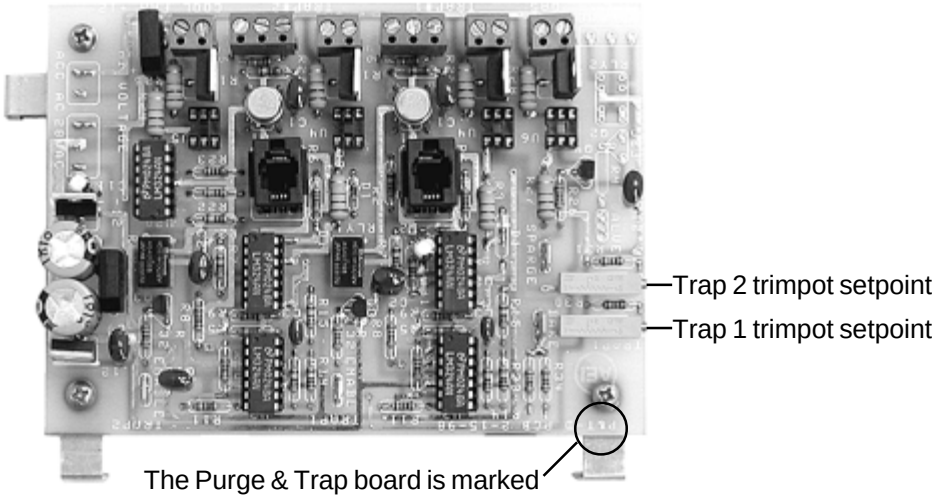
1. Remove the 6 screws that secure the bottom panel to the rest of the GC chassis. Support the panel while you gently rock the GC onto its back, then lower the panel to your working surface to access the chassis interior.

GC Chassis Interior




2. Locate the Purge & Trap board; it is one of a group of similar-looking boards installed along the back and top walls of the GC interior. The Purge & Trap board has two trimpots right next to each other, and it is marked with an upside-down "P&T" on the lower outer corner.

The Purge & Trap Board




3. The two trap trimpot setpoints are on the outer edge of the board. The trimpot for Trap 1 is on the bottom, and the top trimpot is for Trap 2. Turn the trimpot while pressing the TOTAL button and observing the bright red LED display to set the trap adsorption temperature.

4. When you are finished adjusting both trap adsorption temperatures, place the bottom panel on the GC chassis. Support the panel while you gently rock the GC onto its base. Secure the base with its 6 screws.

## *Switching / Replacing Traps*

Three traps are included with your SRI Purge & Trap: a Tenax™-GR trap and a Carbosieve™ trap, both permanently packed, and a blank trap. The blank trap may be packed with an adsorbent of the user's choice or left blank, depending on the analytical situation. Follow the instructions below to access the traps for switching or replacement.

1. With the red protective GC cover raised, remove the Purge & Trap cover plate by loosening the four brass thumbscrews at its corners.

2. Carefully remove the two squares of white insulation from each valve oven duct to expose the fittings that secure the traps ends to the 1/16" O.D. tubing leading to the 10 port valve.

3. Gently slide the trap assembly out of the slots in the valve oven ducts (there is enough slack in the heater and thermocouple wires to pull either trap about 1 inch outside the duct).

4. Use two wrenches to loosen the 1/8" Swagelok type nuts that secure the traps ends to stainless steel 1/8"-1/16" reducing unions.

5. The trap heater is a clamshell design, consisting of two halves. To remove the heater from the trap, loosen but do not remove the two securing hex head screws. The two halves of the clamshell heater will open enough to let the trap drop out.

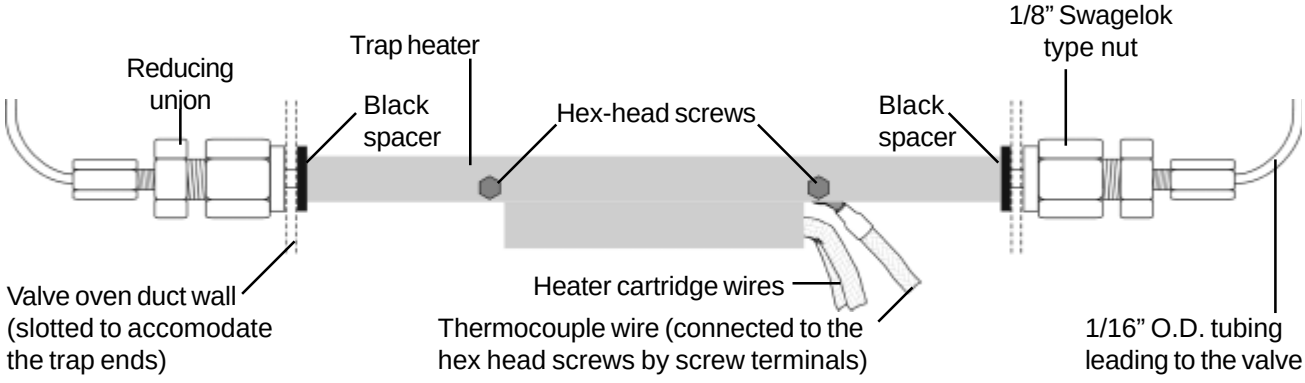
6. Attach the replacement trap to the reducing unions with the trap's two 1/8" nuts. Use stainless steel nuts and brass ferrules when replacing traps. DO NOT use graphite ferrules, as graphite has some adsorption properties and may interfere with your analysis.

7. Slip the trap into the clamshell heater and tighten the two hex head screws.

8. Gently push the trap ends back into the slots in the two interior duct walls, making sure that the black spacers are between the duct walls and the trap heater. TO AVOID DAMAGE, ARRANGE THE TRAPS SO THAT ONE TRAP'S HEATER WIRES DO NOT LAY ACROSS THE OTHER TRAP'S HEATER.

9. Repeat the process with the other trap if necessary. Replace the white duct insulation squares, then replace and secure the Purge & Trap cover plate.

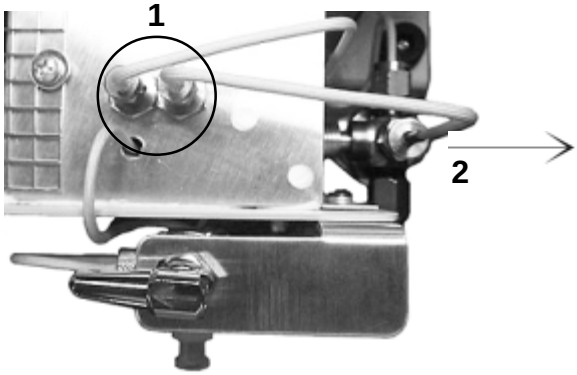
**SRI Trap Assembly**



## *Method 5030/5035 Purge & Trap:*
### Changing the Purge Heads

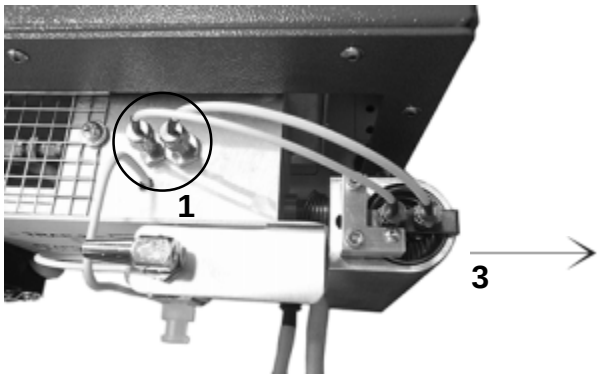
1. To change the purge heads, first disconnect the two purge gas lines at their fittings on the top of the front valve oven duct.

5030 Purge Head




2. If you are removing the 5030 purge head, pull it out toward the front of the GC, and unplug the 5-pin XLR dummy plug.

5035 Purge Head



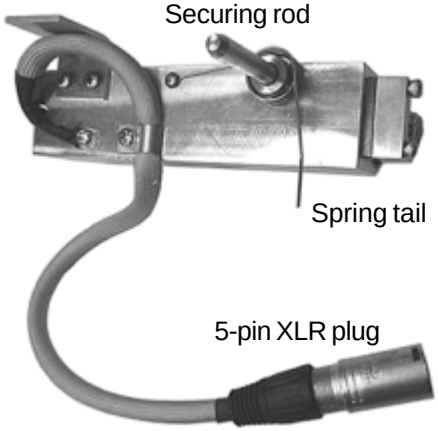

3. If you are removing the 5035 purge head, squeeze the protruding tail of the spring toward the heater body with your thumb as you pull the purge head out toward the front of the GC. Unplug the cord from the socket on the GC.

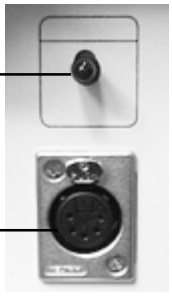
The spring tail (encircled in white) protrudes from the bottom of the front valve oven duct.

4. To install the 5030 purge head, line up the securing rod with the hole, and gently but firmly push it in until it locks into place. Connect the purge gas in and out lines to the fittings on the top of the front valve oven duct. Connect the dummy 5-pin XLR plug to the socket on the GC.

5. To install the 5035 purge head, hold the spring tail in a downward direction against the heater body as you slide the securing rod into the hole until it locks (the shaker will not work without the spring). Connect the purge gas in and out lines to the fittings on the top of the front valve oven duct. Connect the 5-pin XLR plug to the socket on the GC.

5035 Purge Head



## *Expected Performance*

The following two sets of chromatograms are from an Environmental GC system equipped with a Method 5030 compliant Purge & Trap, a PID detector, and a FID/DELCD combination detector. First, a 10ppb BTEX Plus sample was analyzed using the 5030 event table on the ***General Operating Procedures*** page and the **Epap&t.tem** temperature file. Second, a water blank was run through the system under identical conditions to show the component carry-over level of the Purge & Trap system. Toluene is used as a representative of the carryover in the Purge & Trap system; if the carryover level of Toluene is 0.5ppb or less on the PID chromatogram, then it will not affect subsequent analyses. NOTE: The TCE ghost peaks in the water blank chromatograms are augmented or caused by our factory test laboratory contamination.

Sample: 1µL 100ppm BTEX Plus dissolved in 10mL of water to yield 10ppb BTEX Plus

FID Results:

| Component | Retention | Area |
|---|---|---|
| Solvent | 10.616 | 921.0990 |
| Benzene | 15.033 | 1019.9260 |
| TCE | 15.883 | 441.8700 |
| Toluene | 17.683 | 1195.3320 |
| PCE | 18.700 | 383.3770 |
| Ethyl Benzene | 20.016 | 1247.3420 |
| Ortho Xylene | 20.800 | 1258.9260 |
| Bromoform | 21.166 | 78.9360 |
| | Total | 6546.8080 |

PID Results:

| Component | Retention | Area |
|---|---|---|
| Benzene | 15.016 | 311.1630 |
| TCE | 15.866 | 258.4360 |
| Toluene | 17.666 | 353.2160 |
| PCE | 18.683 | 233.4780 |
| Ethyl Benzene | 20.000 | 343.9640 |
| Ortho Xylene | 20.783 | 350.7040 |
| Bromoform | 21.133 | 32.3470 |
| | Total | 1883.3080 |

DELCD Results:

| Component | Retention | Area |
|---|---|---|
| TCE | 15.883 | 192.1020 |
| PCE | 18.683 | 209.2260 |
| Bromoform | 21.150 | 126.2820 |
| | Total | 527.6100 |

Sample: clean water blank (NOTE: the chromatograms are magnified for carryover visibility)

FID Results:

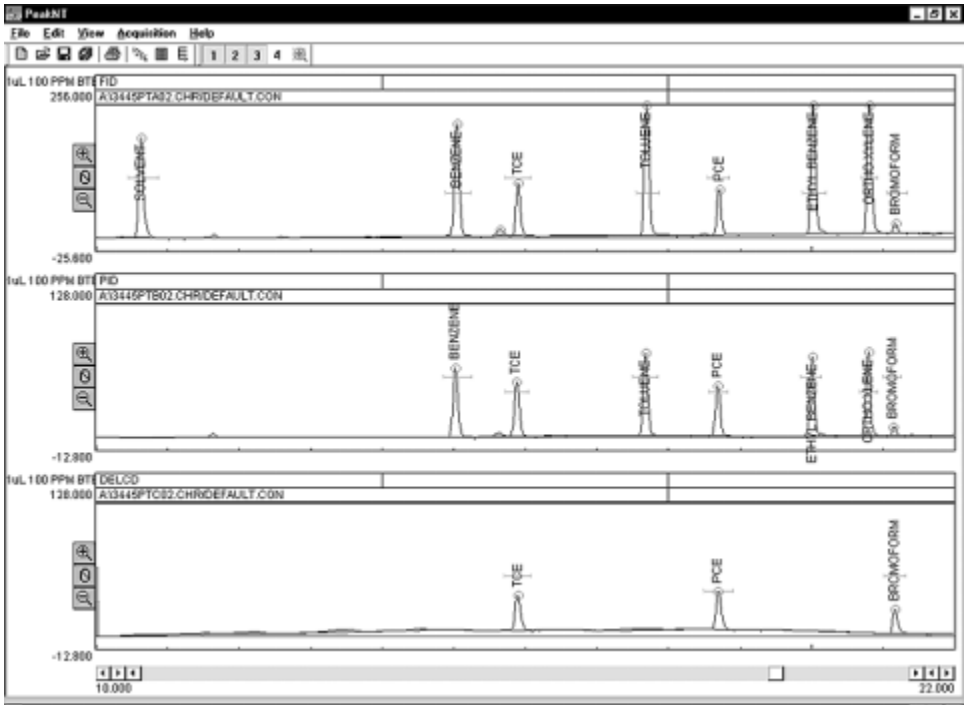
| Component | Retention | Area |
|---|---|---|
| TCE | 15.766 | 58.9100 |
| Toluene | 17.566 | 17.4000 |
| Ethyl Benzene | 20.033 | 51.9080 |
| Ortho Xylene | 20.833 | 91.5290 |
| | Total | 219.7470 |

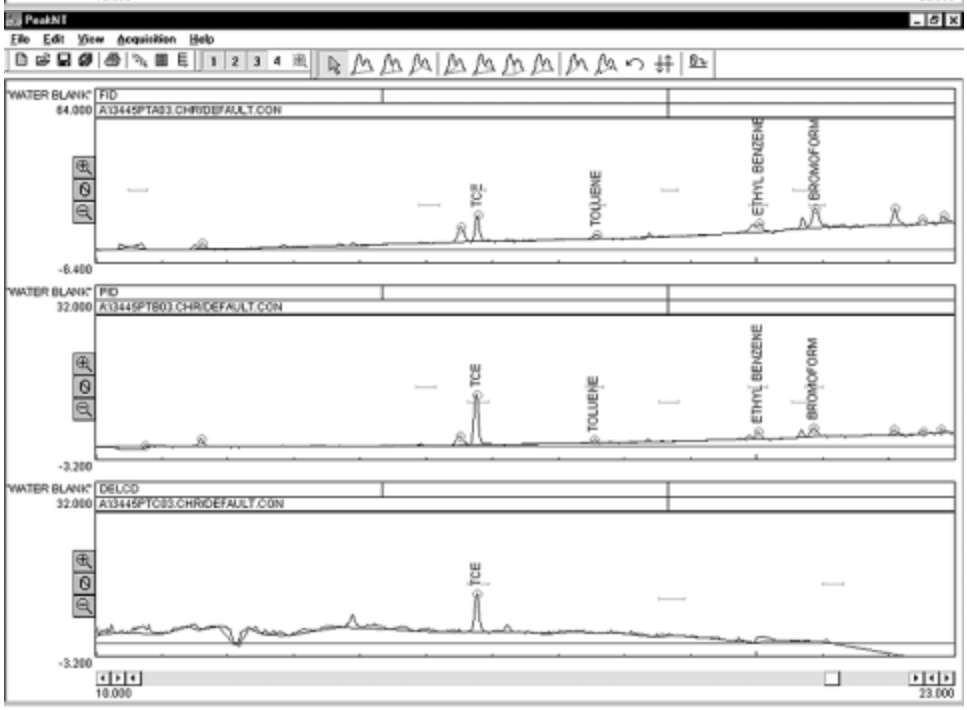
PID Results:

| Component | Retention | Area |
|---|---|---|
| TCE | 15.750 | 58.1920 |
| Toluene | 17.533 | 4.3400 |
| Ortho Xylene | 20.850 | 20.8720 |
| | Total | 609.1300 |

DELCD Results:

| Component | Retention | Area |
|---|---|---|
| TCE | 15.750 | 46.0340 |



## *Troubleshooting and Maintenance*
### Carryover

Carryover is a slight contamination of the purge and trap system by analytes (especially high boiling components), and is a normal condition of operation. All purge and trap systems exhibit some carryover. An organic free reagent water blank is analyzed after sample runs to determine the carryover level, as shown on the ***Expected performance*** page. Most regulatory Quality Control requirements allow carryover that is either less than the Minimum Detectable Limit (MDL) or less than 10% of the reported analyte concentration. For example, if the reported analyte concentration is 100ppb, then 10ppb is acceptable carryover. If the carryover is greater than an acceptable level, subsequent water blanks are run until the carryover is sufficiently low, or until the user has determined that there is system contamination that requires further cleaning.

The carryover level of the 10ppb BTEX sample on the ***Expected performance*** page was determined by comparing the areas of the resulting PID Toluene peaks, where **x** is the ppb concentration of the carryover:

$$\frac{4}{353} = \frac{x}{10ppb}$$

$$353x = 40ppb$$

$$x = 0.1133ppb$$

The 10ppb BTEX sample analysis resulted in a PID Toluene peak with an area count of approximately 353. The water blank analysis shows a PID Toluene peak with an area count of approximately 4. Since the carryover of Toluene is less than 10% or 0.5ppb, subsequent analyses may be resumed.

Most carryover problems occur while analyzing samples of unknown concentration. Because the user cannot assume there will be no carryover in this type of analytical situation, a clean water blank should be run between each sample analysis to ensure that carryover will not affect subsequent sample analyses. Avoid carryover contamination problems by screening your samples prior to purge and trap GC analysis. SW-846 contains two appropriate screening techniques:

- Method 5021, in which an automated headspace sampler is used with a PID and DELCD equipped GC
- Method 3820, in which a hexadecane extraction of the sample is analyzed by a FID and/or ECD equipped GC.

Segregate the screened samples according to concentration, then run the highly concentrated ones first. Clean the purge and trap system after the high concentration samples have been run, then analyze the low concentration samples.