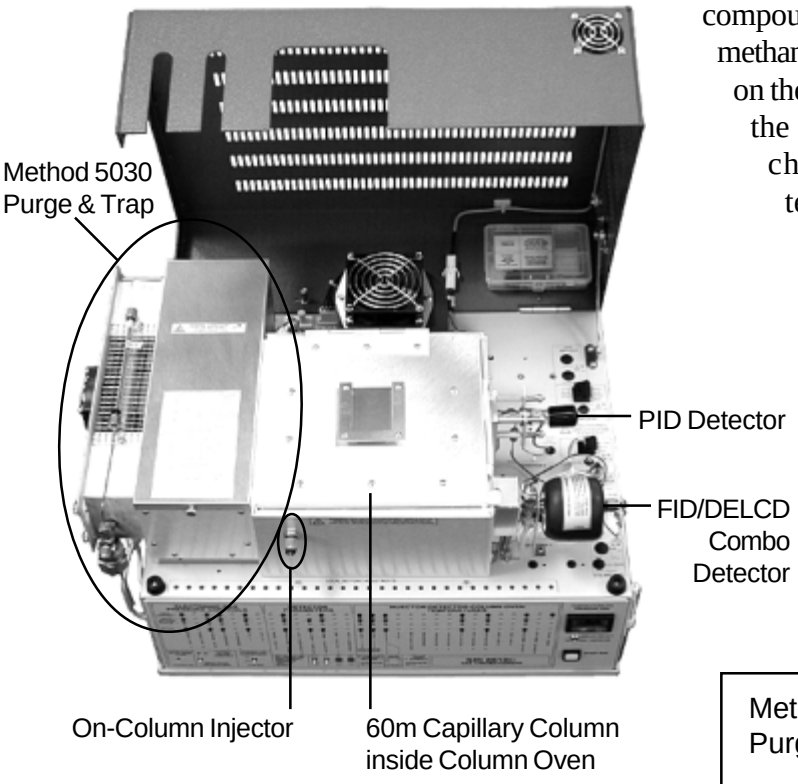
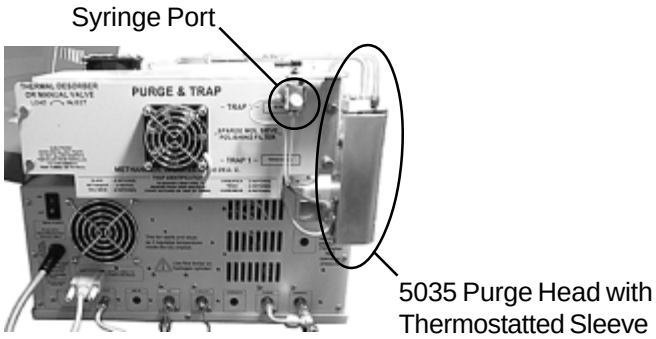
System Overview

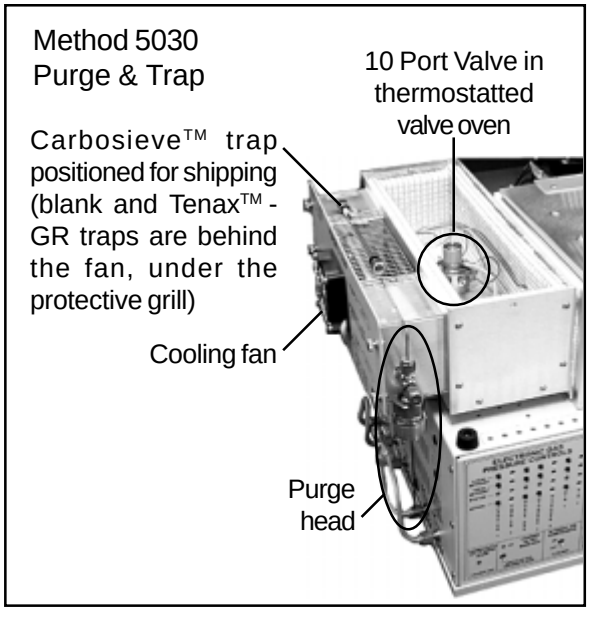
Your SRI Environmental GC is equipped with everything you need to generate certification quality data for EPA Methods 8010, 8015, 8021, and others. It is configured on the 8610C chassis, and includes a built-in Method 5030 or 5030/5035 compliant Purge & Trap for concentration of liquid and/or soil samples. Also included is an on-column injector for direct liquid injections. To detect commonly targeted pollutants, the Environmental GC uses a sensitive, non-destructive PID detector in series with a combination FID/DELCD detector. The PID detector responds to compounds whose ionization potential is below 10.6eV, including aromatics and chlorinated molecules with double carbon bonds. The FID detector responds to the hydrocarbons in the sample. The DELCD selectively detects the chlorinated and brominated compounds in the FID exhaust. Since the sample is pre-combusted in the FID flame, the DELCD is protected from contamination due to hydrocarbon overload. The PID is blind to certain compounds which can cause interference, such as methanol, and is recommended by the EPA. Peaks on the FID chromatogram that are obscured by the methanol peak are visible on the PID chromatogram. Benzene and carbon tetrachloride are common target analytes which co-elute. The FID responds to both. The PID responds only to benzene, while the DELCD responds only to carbon tetrachloride.

The BTEX GC is the same as the Environmental GC without the DELCD detector. Both systems have a "whisper quiet" internal air compressor and can be used with an H_2-50 hydrogen generator for tankless field operation.



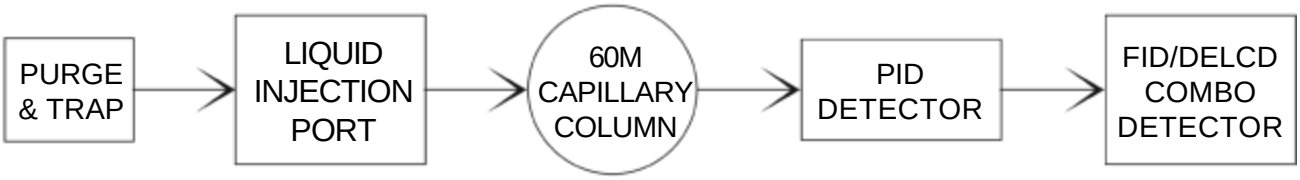
Method 5030/5035 Purge & Trap on an Environmental GC





POPULAR CONFIGURATION GCs
BTEX & Environmental

Theory of Operation

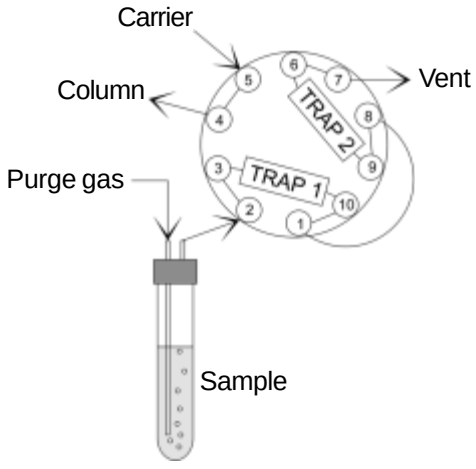


The versatile BTEX/Environmental GC systems can analyze gas, water, and soil samples. Four types of injection techniques can be used: purge and trap, direct liquid injection, TO-14 type gas sample concentration, and manual headspace injection. The Purge & Trap concentrator may be used for gas, liquid, and solid samples. For liquid samples up to 5µL and gas samples up to 1mL, direct injections can be made through the on-column liquid injection port. Larger gas samples can be injected through the syringe port on the 5030/5035 Purge & Trap concentrator or the septum port on the 5030 model.

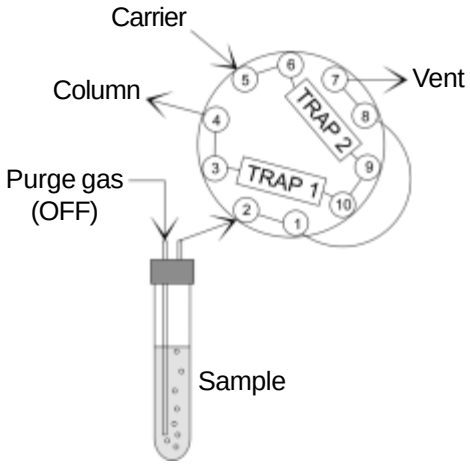
Purge & Trap Injection

Designed for compliance with EPA Methods 5030 and/or 5035, the Purge & Trap system extracts volatile organic compounds from the sample solution in the test tube or VOA vial. Using a dual trap design plumbed with a 10 port gas sampling valve, the Purge & Trap system enables the use of two separate adsorbents with different desorption temperatures for a wide range of target analytes. Each trap is heated independently.

10 Port Valve in LOAD



10 Port Valve in INJECT



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. The traps are heated to their respective desorption temperatures shortly after purging is stopped. When the traps reach desorption temperature, the valve is actuated to the INJECT position. In this position, the carrier gas backflushes through the traps in the direction opposite to the sample-laden purge gas flow with which the traps were loaded. The carrier gas flow sweeps desorbed analytes into the column, while flow from the purge vessel is stopped by the PeakSimple data system.

Theory of Operation continued

Direct Injection

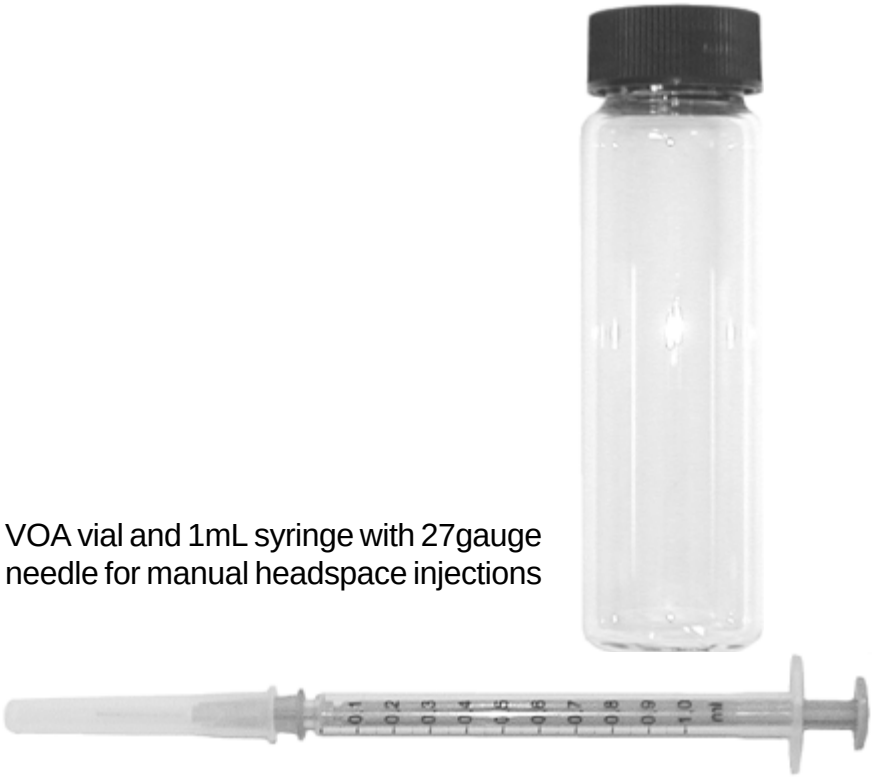
Direct injection with the BTEX or Environmental GC systems is simple and straightforward. This method uses the on-column injector to inject the sample directly into the column, bypassing the entire purge and trap injection system. Sample size for this technique is 1mL or less for gas, and 5µL or less for liquid. No event table is necessary, just a temperature program for the column oven.

Gas Sample Concentration

In this TO-14 type technique, a large volume of gas is pushed by syringe or pulled by vacuum pump through the dual traps. The trapped analytes are then desorbed and swept into the column. If the GC has the optional vacuum pump interface, the pump is plugged into it and may be controlled by the PeakSimple data system using an event table.

Room Temperature Manual Headspace Injection

When making headspace injections with the BTEX or Environmental GC systems, the sample is equilibrated offline at room temperature. It is then injected by syringe into the on-column injector. This technique is basically the direct injection of small gas samples.



VOA vial and 1mL syringe with 27gauge needle for manual headspace injections

General Operating Procedures

EPA Style Purge & Trap Injection

This technique is limited to volatile organic compounds that purge efficiently from water at ambient temperature and VOC's that are purgeable from soil at 40°C. 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. Also, please see the "***Sample Preparation***" page in the SRI Purge & Trap manual section (available online at www.srigc.com).

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. ***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. 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 temperatures typically run 5°C over the setpoint. See the instructions in the Purge & Trap section of the manual for adjusting the trap adsorption temperature settings.

3. 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 (such as **Epap&t1c.evt** for a single trap or **Epap&t2c.evt** for dual traps included in version 2.66 or higher of the PeakSimple software).

4. 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 for your convenience. As a basic rule for good separation using the purge and trap injection technique, 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 while the traps heat and the gas sampling valve (in the INJECT position) transfers the sample to the column.

Epap&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)

Epap&t1c.evt is designed for one trap, while **Epap&t2c.evt** is for two traps.

Dual Trap Event Table (Epap&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"

General Operating Procedures continued

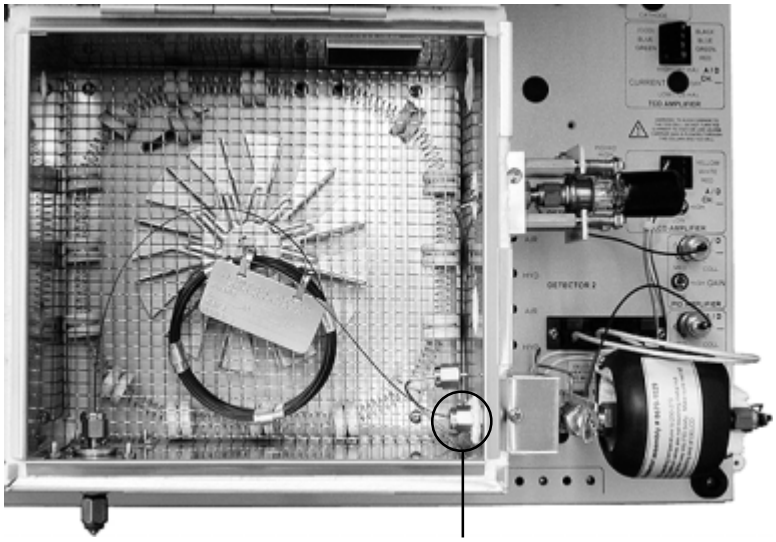
Direct Injection

This technique is useful for volatile and semi-volatile compounds, but is typically used for diesel and other compounds that don't purge well from aqueous or soil samples.

1. Perform **Detector Steps** 1-4, then proceed with step two below.

2. Load or create a Temperature Program for the column oven. You can create an isothermal or ramped temperature program; deciding which to use depends on the sample being analyzed, and the goals of the analysis. There are several preset **.tem** files included with version 2.66 and higher of the PeakSimple software. If the analysis requires the column to be hotter than 150°C, it is best to disconnect the column from the PID detector. The PID represents a cold spot in which higher boiling analytes will become trapped, never making it to the much hotter (300°C) FID for detection. Also, when the column is heated over 150°C, stationary phase bleed will adhere to the PID lamp window. The higher boiling analytes and the column bleed will create a coating on the PID lamp window that will interfere with the analysis. The PID lamp window may be cleaned in the event of contaminant condensation, but the resulting change in the PID response usually requires detector recalibration. To bypass the PID, turn its lamp current OFF, then disconnect the column from the detector by loosening the swagelok-type nut from the bulkhead fitting in the column oven wall. Remove the tubing that connects the PID exit to the FID/DELCD by loosening that nut. Place the end of the column into the FID/DELCD bulkhead fitting instead and tighten it in place.

PID detector bypass for column oven temperatures above 150°C

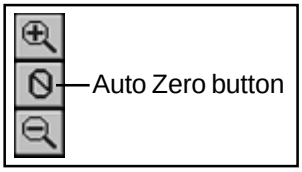


The column is inserted directly into the FID, bypassing the PID.

3. While the detectors are heating and stabilizing, prepare a diesel sample by shaking a known weight of the sample with a measured volume of methylene chloride for 1-3 minutes. Allow any particulates to settle before drawing the sample into the syringe.

4. Use a clean, standard glass 10µL GC syringe with a 26 gauge needle. Fill the syringe with sample, and work out any air bubbles. Depress the plunger until 1µL of sample remains in the syringe.

5. Zero the data system signal by clicking on the Auto Zero button on the left side of the chromatogram window. Or, make the first event ZERO (at time 0.00) in your event table.



6. Begin the analysis by pressing the RUN button on the GC or the computer keyboard spacebar.

7. Quickly and smoothly insert the syringe needle into the on-column injection port, and immediately depress the plunger.

General Operating Procedures continued

Gas Sample Concentration

This TO-14 type technique injects a gas or air sample using either a large syringe (60mL) or a Tedlar bag (1L). A vacuum pump may be used to pull the sample through the sorbent traps. The amount of sample that may be loaded onto the trap(s) is limited only by the capacity of the trap's adsorbent packings. The more gas that is loaded onto the traps, the lower the detection limit will be.

The volume and flow of sample and carrier gas that can be fed through the traps without adversely affecting the resulting chromatogram is known as the breakthrough volume. Different adsorbents have different breakthrough volumes. A breakthrough volume value is determined by the sample and target analytes, the adsorbent packing (pore size, natural affinities for certain compounds, etc.), the diameter of the trap, and the temperature at which the traps are loaded. Therefore, a given trap will have different breakthrough volumes in different analytical conditions.

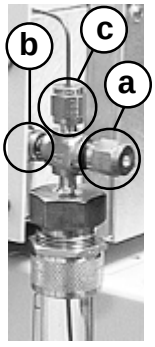
The SRI Purge & Trap concentrator is shipped with a blank trap and a Tenax™-GR trap installed, and a Carbosieve™ S-III packed trap for optional user installation. The Tenax-GR trap has a low affinity for water, making it a good adsorbent for the purge and trap technique. The Carbosieve has a high affinity for water, and is generally highly retentive; SRI recommends using it only when vinyl chloride is among the target analytes. The blank trap is provided for the user to pack with the adsorbent of choice.

Using a syringe:

1. Perform **Detector Steps** 1-4. While the detectors are heating and stabilizing, load or create an event table. The valve (Relay G) must be in the LOAD (G OFF) position while analytes are being adsorbed onto the traps. The valve is rotated to the INJECT (G ON) position during desorption. See the valve diagrams on the **EPA Style Purge & Trap Injection *Theory of Operation*** page. Relays C (trap 2) and F (trap 1) activate the traps' heat. The relays may also be activated by the operator during an analysis: open the Relay/pump window and click on the letter corresponding with the relay you want to turn ON or OFF.

2. Inject the sample into the 5030 septum nut or the 5030/5035 syringe port. Alternatively, the 5030 purge head may be removed by unscrewing nut **b,** allowing the sample to be injected directly into the bulkhead fitting on the front of the valve oven duct (see the photo, below right). Depending on the syringe you're using, you may have to make an adaptor for injection into the purge head.

3. Load or create a temperature program for the column oven. Once the detectors are activated and stabilized, begin the analysis.



Using a vacuum pump:

1. Connect the vacuum pump to the trap vent on the backside of the valve oven.

2. If your GC has the optional vacuum pump interface installed, plug the vacuum pump into that power socket on the left panel of the GC chassis. Enter events in the event table to turn the vacuum pump power ON and OFF as desired during the analysis. If your GC doesn't have the vacuum pump interface, plug the vacuum pump into a wall outlet instead, and control it's ON/OFF switch manually during the analysis.

3. Once the detectors are activated and stabilized, connect the Tedlar bag to the purge head septum nut (**a**), or remove the purge head and secure the Tedlar bag to the bulkhead fitting in the front valve oven duct. [To remove the purge head: loosen the nut (**b**) that secures the purge head to the bulkhead fitting in the valve oven duct wall. Loosen the nut (**c**) that secures the purge head to the purge gas tubing. Leave the second fitting (**c**) on the purge gas tubing and slide the purge head off of the tubing. See the photo, above right.] Load or create a temperature program. Begin the analysis.

General Operating Procedures continued

Room Temperature Manual Headspace Injection

1. In this technique, the sample is equilibrated offline. Transfer sample into a clean VOA vial until the vial is half full. Let it set at room temperature for 30 minutes to an hour to equilibrate.

2. Load or create a temperature program for the column oven.

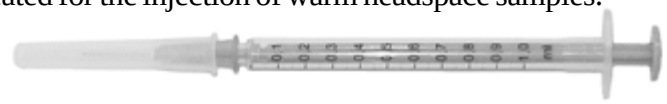
3. Perform **Detector Steps** 1-4, then proceed with the following steps.

4. Fill a plastic medical syringe with the vial headspace. Inject the sample into the GC injection port, bypassing the Purge & Trap concentrator.

5. Begin the analysis by pressing the RUN button on the GC or the computer keyboard spacebar.

Note: both the sample vial and the syringe may be heated for the injection of warm headspace samples.

40mL VOA vials are available from Eagle Picher under part number 140-40C/EP/ES.
1-800-331-7425



Disposable, sterile 1mL syringes are available in packages of 100 from Aldrich under catalog number Z23072-3. 27 gauge precision glide needles in packages of 100 are available under catalog number Z19237-6.
1-800-558-9260

Detector Steps

1. With the black plastic lamp hood in place on the PID lamp, turn ON the PID lamp current with the flip switch on the GC's front control panel. Set the PID current to 70 (= 0.70ma) by adjusting the appropriate trimpot setpoint on the top edge of the GC's front control panel. (Each detector zone is labeled on the front control panel under DETECTOR PARAMETERS, with the corresponding trimpot setpoint directly above it.) The lamp should emit a violet-colored light visible down the center of the tube. Set the PID temperature to 150°C. Set the PID gain to LOW.

2. Turn on the air compressor using the switch on the GC's front control panel. NOTE: since most ambient air will not cause interference with the DELCD, the built-in air compressor is appropriate for most analytical situations. However, if you are doing analyses in a lab environment with low levels of halogenated compounds in the ambient air, they can cause the DELCD to lose sensitivity, and fluctuations in the level of organics in ambient air may cause additional baseline noise. To avoid this, use clean, dry tank air.

3. Set the FID hydrogen flow to 25mL/min, and the FID air flow to 250mL/min. The pressure required for each flow is printed on the right hand side of the GC chassis. Ignite the FID by holding up the ignitor switch for a couple of seconds until you hear a small POP. Ensure that the flame is lit by holding the shiny surface of a chromed wrench to the tip of the collector electrode; when the flame is lit, you should be able to see condensation on the wrench. Set the FID gain to HIGH. If the peaks are more than 20 seconds wide at the base, use the HIGH FILTERED gain setting. If you wish to keep the ignitor ON to prevent flameout, set the ignitor voltage to -750 by adjusting the trimpot on the FLAME IGNITE zone.

4. If a DELCD detector is installed, set the DELCD reactor temperature setpoint to 260 (=1000°C) by adjusting the appropriate trimpot. The DELCD will heat to around 254 and stabilize; the protruding end of the ceramic tube will glow bright red in the heat. Set the DELCD gain to LOW.

5. When the system has reached temperature and each detector is displaying a stable signal, begin the analysis by pressing the RUN button on the front of the GC or the spacebar on the computer keyboard.

POPULAR CONFIGURATION GCs
BTEX & Environmental

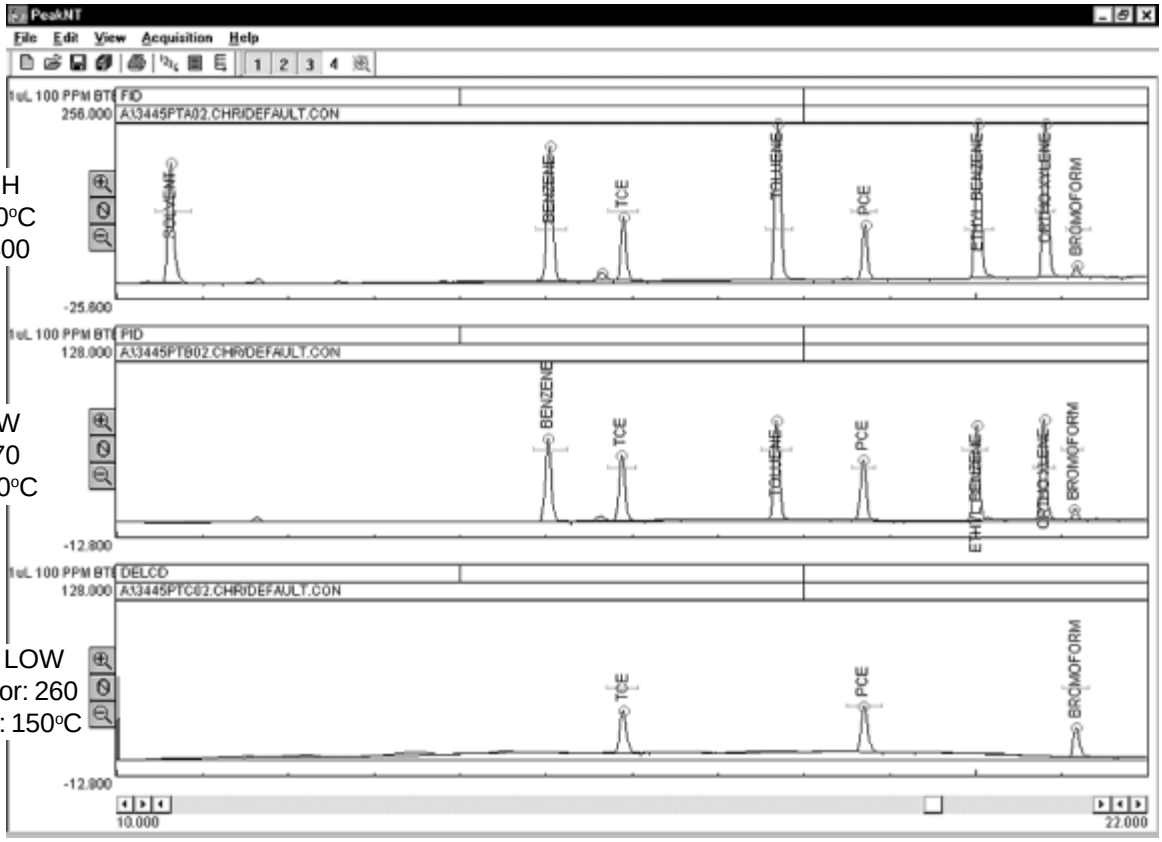
Expected Performance - Purge & Trap Concentrator

These chromatograms were produced from a 10ppb BTEX Plus standard analyzed in an Environmental GC equipped with a Method 5030 Purge & Trap injection system. The simultaneous display of all three detector channels illustrates their relative selectivity. The chromatogram on the next page shows the carry-over from the Purge & Trap concentrator on the subsequent analysis.

FID gain: HIGH
FID temp: 150°C
FID ignitor: -400

PID gain: LOW
PID current: 70
PID temp: 150°C

DELCD gain: LOW
DELCD reactor: 260
DELCD temp: 150°C



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

Events (5030.evt):

Time	Events
0.000	ZERO
0.100	E ON (PURGE GAS)
5.100	E OFF
6.000	C ON (TRAP 2 HEAT)
6.050	F ON (TRAP 1 HEAT)
8.000	G ON (VALVE INJECT)
12.000	E ON
12.900	B ON (BAKE)
13.000	G OFF (VALVE LOAD)
14.900	F OFF
15.100	C OFF
15.300	E OFF
15.500	B OFF

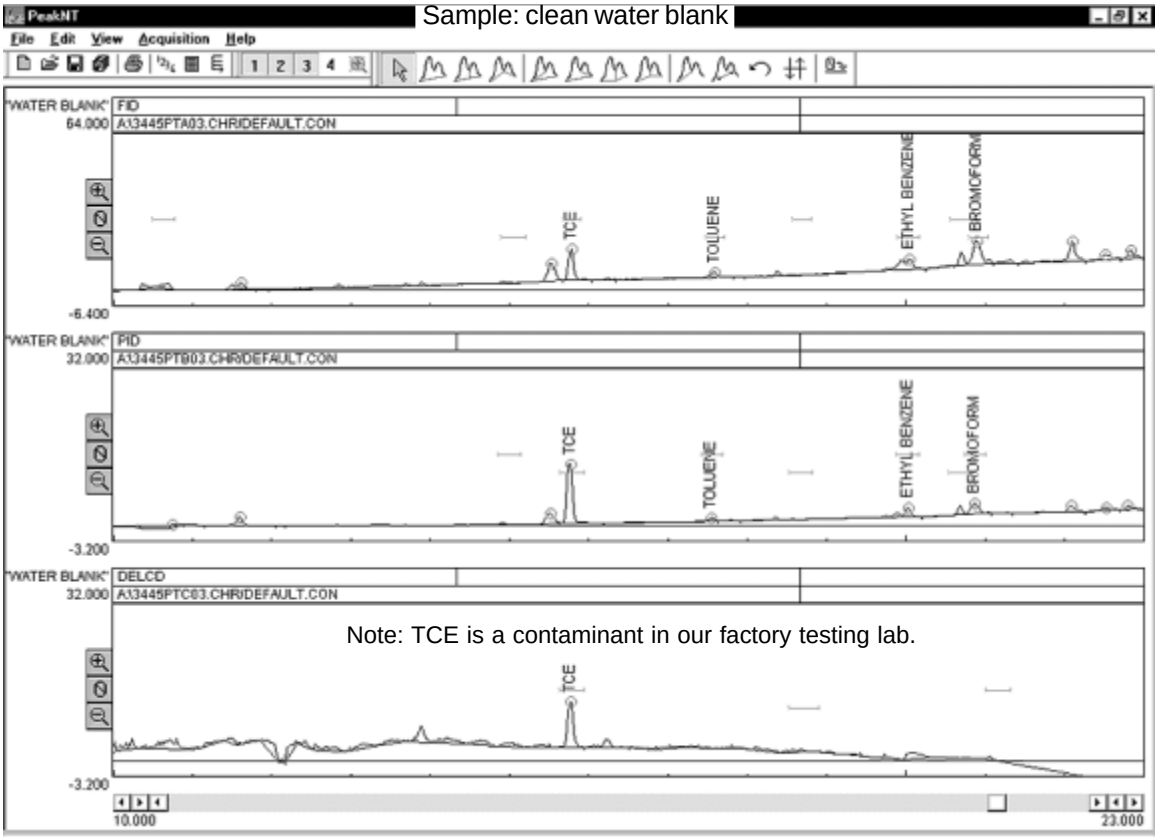
Sample: 1µL 100ppm BTEX Plus standard dissolved in 10mL of water to yield 10ppb of each analyte
Method: 5030 P&T injection
Column: 60m MXT-VOL
Carrier: Helium @ 10mL/min

Temperature Program:
(Epap&t.tem)

Initial	Hold	Ramp	Final
40°C	10.00	10.00	180°C

Expected Performance - Purge & Trap Concentrator

This chromatogram was produced from analyzing a water blank immediately after the analysis of the BTEX Plus standard to show the Purge & Trap carry-over. The blank was run under the same conditions (event table, temperature program, detector settings) as the sample. Acceptable carry-over is a contamination level of 1% or 0.5ppb—whichever is lower—of an analyte (especially high boiling components), and is a normal condition of operation. This 1% of contamination from preceding analyses should not be significant enough to affect quantitation unless a very high concentration sample is followed by a very low concentration sample. It is standard laboratory practice to run a blank after a high concentration sample. Toluene is used as a representative of the carryover in the Purge & Trap system; if the carryover level of Toluene is below 1% or 0.5ppb on the PID chromatogram, then it will not affect subsequent analyses. (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

Determine the carryover level by comparing the areas of the two PID Toluene peaks resulting from the sample and blank runs:

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

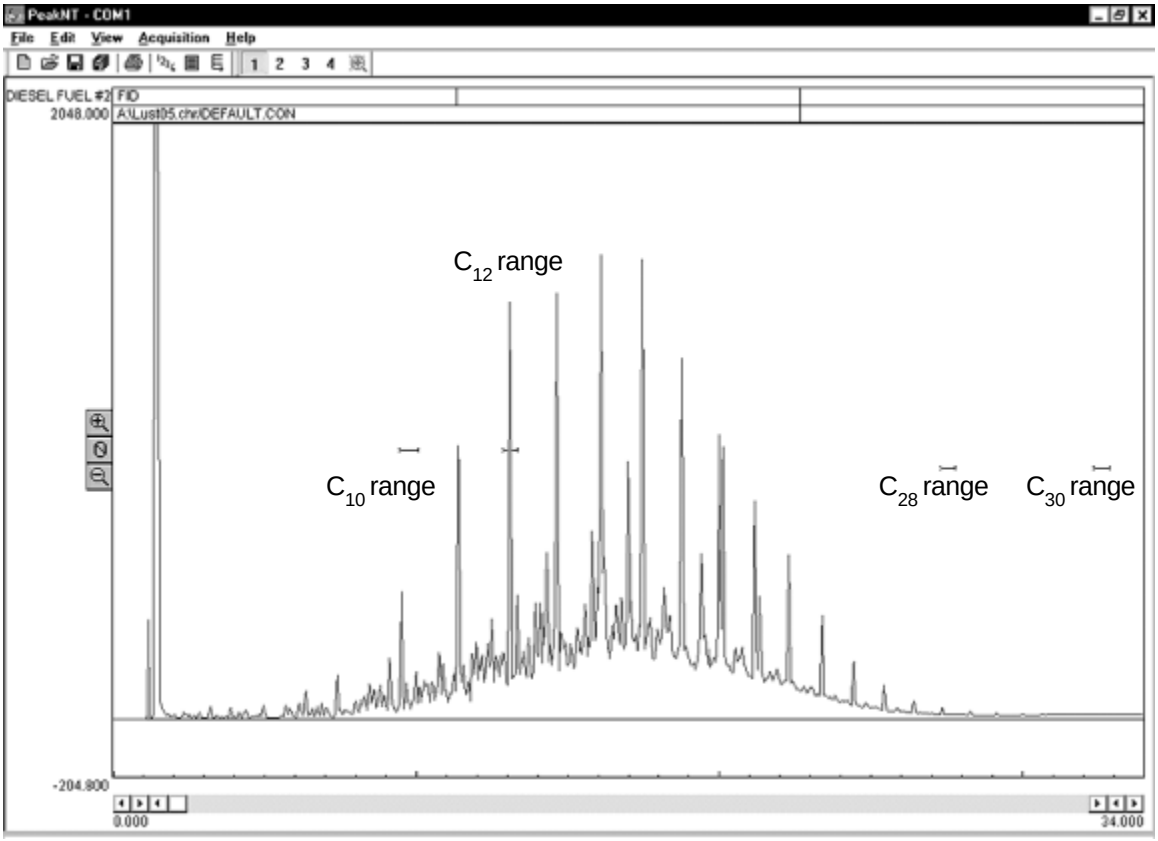
$$353x = 40ppb$$

$$x = 0.1133ppb$$

(**x** represents the ppb concentration of the carryover)

Expected Performance - Direct Injection

This chromatogram is from an analysis of a diesel sample. The PID detector was bypassed, and the column was connected directly to the FID detector inlet. The results are identifiable as diesel because it shows the range of hydrocarbons that compose this fuel. A few retention windows are placed in the chromatogram to show the approximate ranges of C_{10}, C_{12}, C_{28}, and C_{30}.



Sample: diesel fuel #2
Method: direct injection
Column: 60m MXT-VOL
Carrier: helium @ 10mL/min

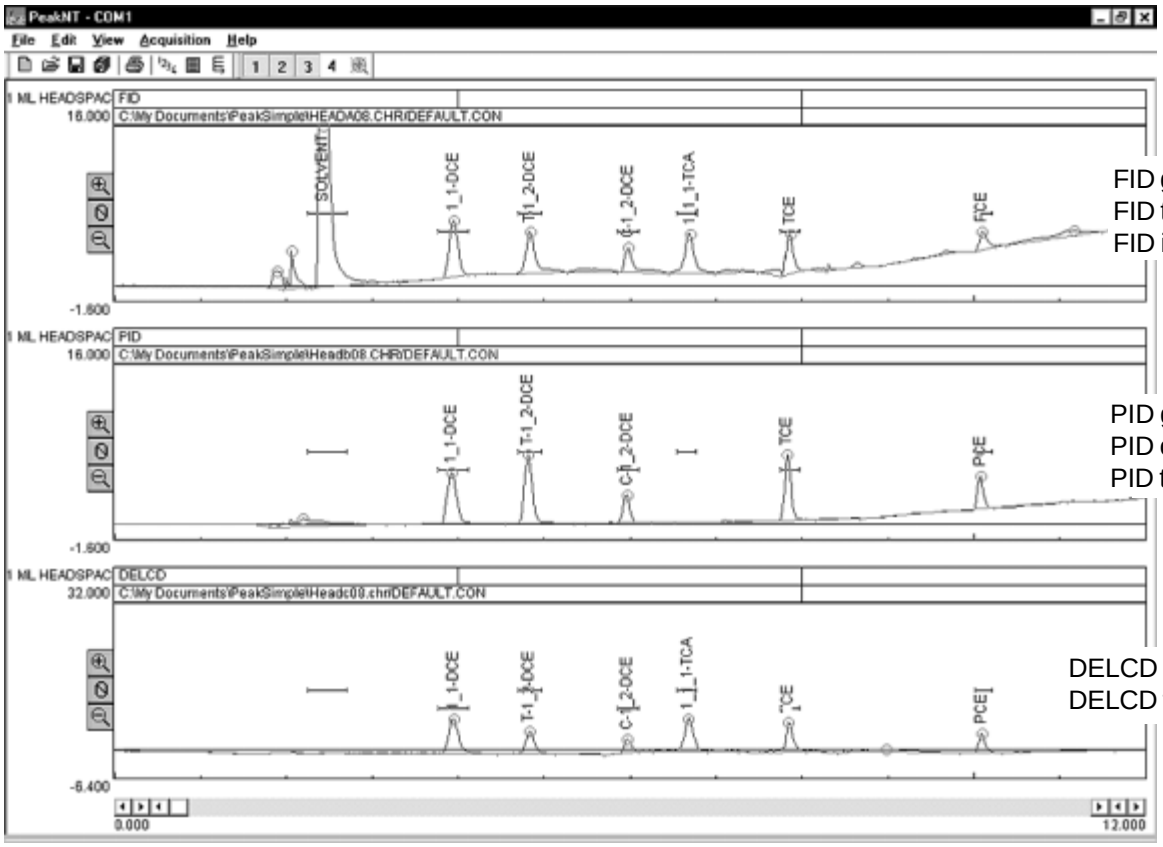
FID gain: HIGH
FID temp: 325°C
FID ignitor: -400

Temperature program:

Initial	Hold	Ramp	Final
50°C	3.000	10.000	320°C
320°C	30.00	0.000	320°C

Expected Performance - Manual Headspace Injection

To obtain the chromatograms below, 50ppb Japanese standard was placed into a VOA vial with water, and allowed to equilibrate at room temperature for 45 minutes. The FID (top) chromatogram shows all the components and the solvent. The PID (middle) does not detect the 1_1_1-TCA, while the DELCD (bottom) does not respond to the solvent.



FID gain: HIGH
FID temp: 150°C
FID ignitor: -400

PID gain: LOW
PID current: 80
PID temp: 150°C

DELCD gain: MED
DELCD temp: 260°C

Sample: 1mL headspace from 50ppb Japanese standard in water
Method: manual headspace injection
Column: 60m MXT-VOL
Carrier: helium @ 10mL/min

Temperature program:

Initial	Hold	Ramp	Final
40°C	2.000	15.000	220°C
220°C	10.00	0.000	220°C

FID Results:

Component	Retention	Area
Solvent	2.416	290.1100
1_1-DCE	3.933	39.6100
T-1_2-DCE	4.833	34.3780
C-1_2-DCE	5.966	18.6020
1_1_1-TCA	6.683	29.6320
TCE	7.850	23.4490
PCE	10.083	10.7560
	Total	446.5370

PID Results:

Component	Retention	Area
Solvent	2.183	22.7450
1_1-DCE	3.916	39.4070
T-1_2-DCE	4.800	45.0050
C-1_2-DCE	5.950	15.7380
TCE	7.816	33.7270
PCE	10.066	16.2780
	Total	172.9000

DELCD Results:

Component	Retention	Area
1_1-DCE	3.933	63.1790
T-1_2-DCE	4.816	38.0780
C-1_2-DCE	5.950	18.0560
1_1_1-TCA	6.666	53.2210
TCE	7.833	39.6900
PCE	10.083	20.8340
	Total	233.0580