System Overview

The SRI CryoSulfur GC comes with everything you need to detect low-level sulfur compounds in gas samples. Since some sulfur compounds do not trap well, the CryoSulfur GC uses the CryoCooler Peltier Trap Accessory to enrich the sample, providing lower detection limits. The CryoSulfur GC uses a vacuum pump (provided) to draw gas or air samples into the CryoCooler. You can sample ambient air, or use the provided adaptor to connect a Tedlar bag. The vacuum pump interface, which is an electrical outlet on the left-hand side of the GC, allows the vacuum pump to be turned ON/OFF by the PeakSimple data system to provide consistent sampling times.

Vacuum pump interface

CryoCooler

Vacuum pump
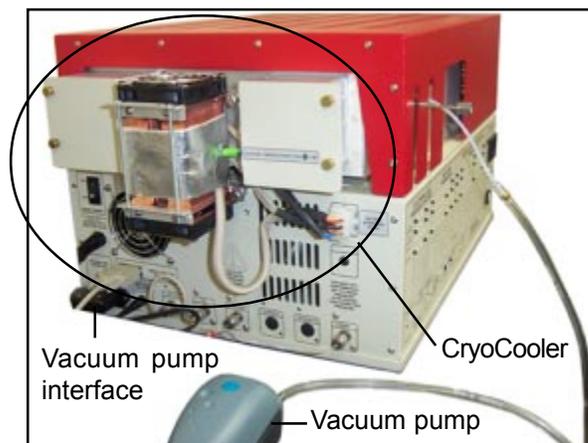
Like all SRI traps, the CryoCooler Peltier Trap Accessory is plumbed as the loop of a 10-port gas sampling valve. It has it's own power cord that must be plugged into a wall outlet, and an interface cord that plugs into the left-hand side of the GC. After enrichment, the valve injects the sample onto the 60-meter capillary column. Once the sample components are separated by the column, they will be detected by the Flame Photometric and Flame Ionization detectors.
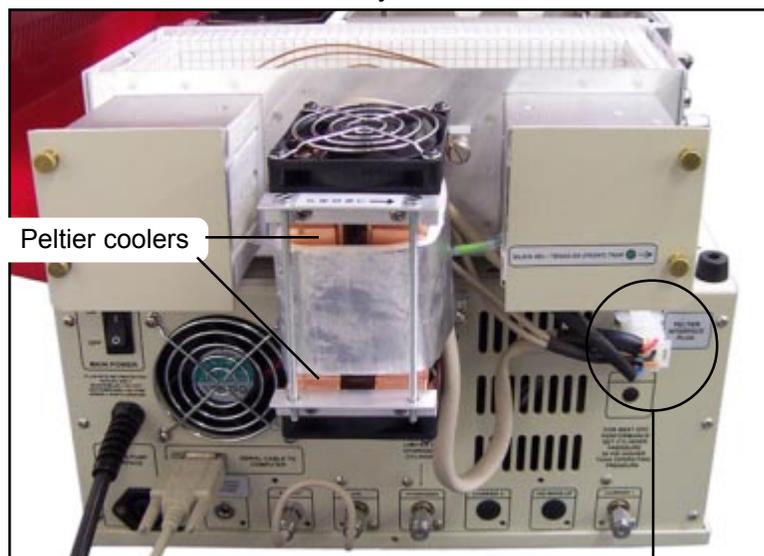

CryoCooler

60-meter x 0.53 MXT-1 capillary column, 5 micron coating

10-port gas sampling valve

FPD/FID combination detector

Tubing to vacuum pump, connected to the SAMPLE OUT fitting on the front of the valve oven

Connector for a Tedlar bag or other sample container (remove from the SAMPLE IN fitting to sample ambient air)
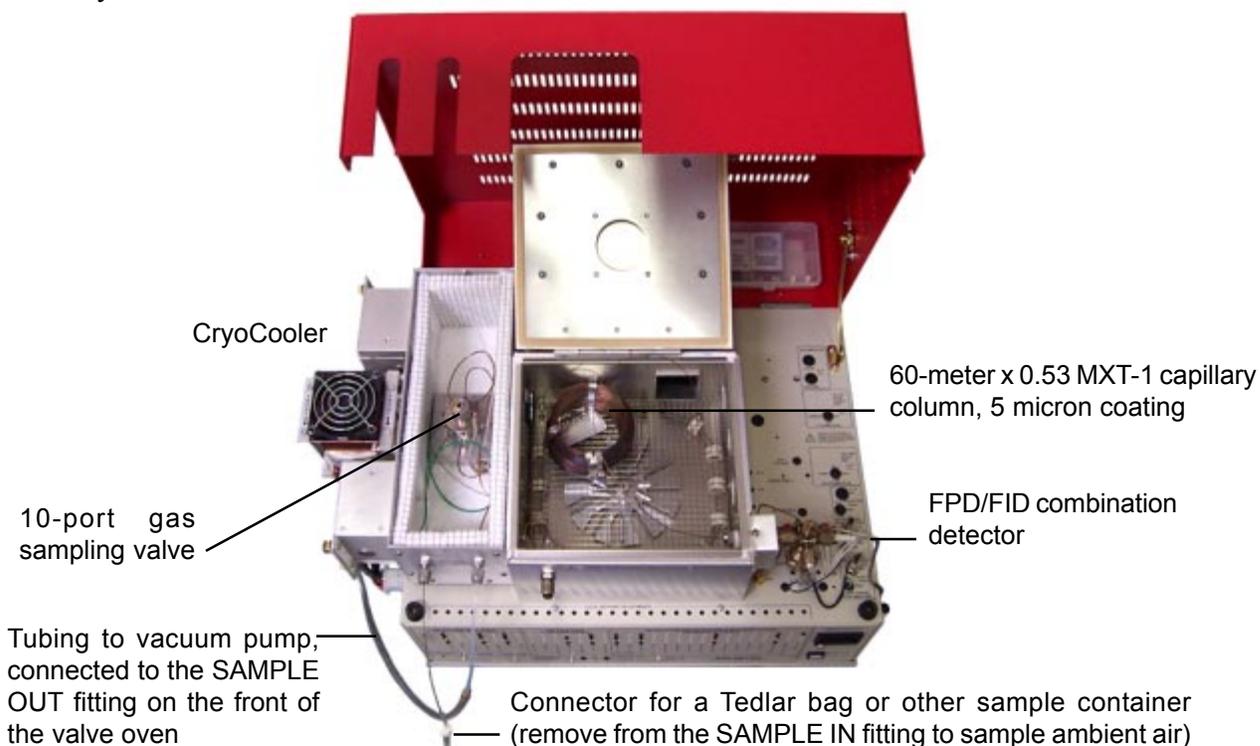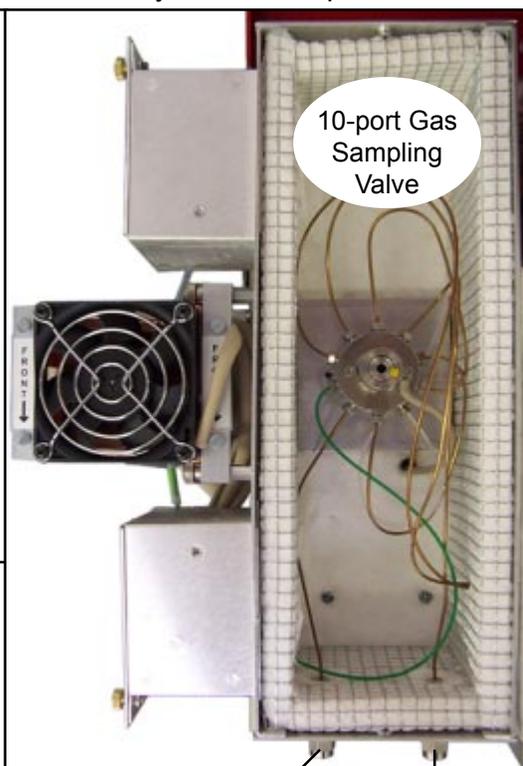
Theory of Operation

Some sulfur compounds do not trap well, but can be enriched for lower detection limits. The CryoCooler Peltier Trap Accessory is basically a heated trap sandwiched between two peltier coolers. The trap is filled with Tenax-GR (about 30%) and Silica Gel (about 70%) adsorbents. The vacuum pump draws sample through the CryoCooler from ambient air, or from a Tedlar bag or other sample container. The peltier coolers can cool the trap down to -15°C to enrich the sample.
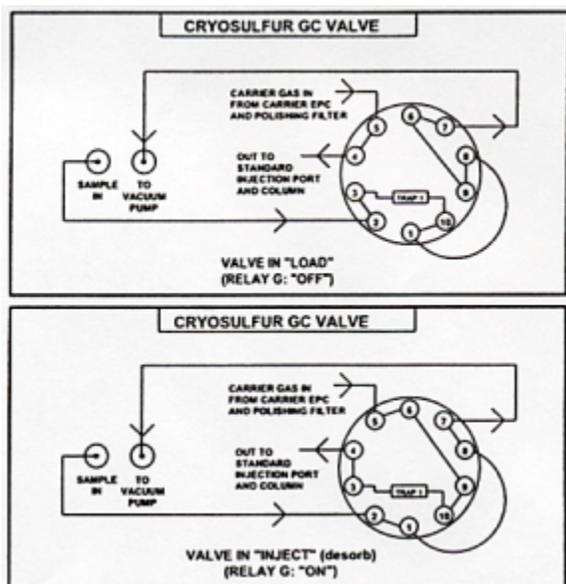
The CryoCooler

Peltier coolers

CryoCooler/GC interface

CryoCooler - Top View

10-port Gas Sampling Valve

SAMPLE IN

SAMPLE OUT to vacuum pump

Valve Diagram Label on Valve Oven Lid:

CRYOSULFUR GC VALVE

CARRIER GAS IN FROM CARRIER EPC AND POLISHING FILTER

OUT TO STANDARD INJECTION PORT AND COLUMN

SAMPLE IN   TO VACUUM PUMP

TRAP 1

VALVE IN "LOAD"
(RELAY G: "OFF")

CRYOSULFUR GC VALVE

CARRIER GAS IN FROM CARRIER EPC AND POLISHING FILTER

OUT TO STANDARD INJECTION PORT AND COLUMN

SAMPLE IN   TO VACUUM PUMP

TRAP 1

VALVE IN "INJECT" (desorb)
(RELAY G: "ON")

While the CryoCooler is enriching the sample, the 10-port gas sampling valve is in the LOAD position. At the conclusion of the sampling period, the heater heats the trap to 150-200°C and the valve is actuated to the INJECT position; this places the trap in the carrier gas stream and sweeps the enriched analytes onto the column.

Expected Performance

The following chromatograms were generated by an SRI CryoSulfur GC. The first chromatogram shows the FPD rsponse to 10ppb hydrogen sulfide (H_2S), as enriched by the CryoCooler at -10°C. The second chromatogram shows the FID response to 1000ppm C_1-C_6 hydrocarbons.

Sample: 10ppb H2S
Column: 60-meter MXT-1
Carrier: helium at 10mLs/minute
Vacuum pump: 20mLs/minute
Trap: -10°C
FPD gain: HIGH
FPD temperature: 150°C
FPD volts: 500
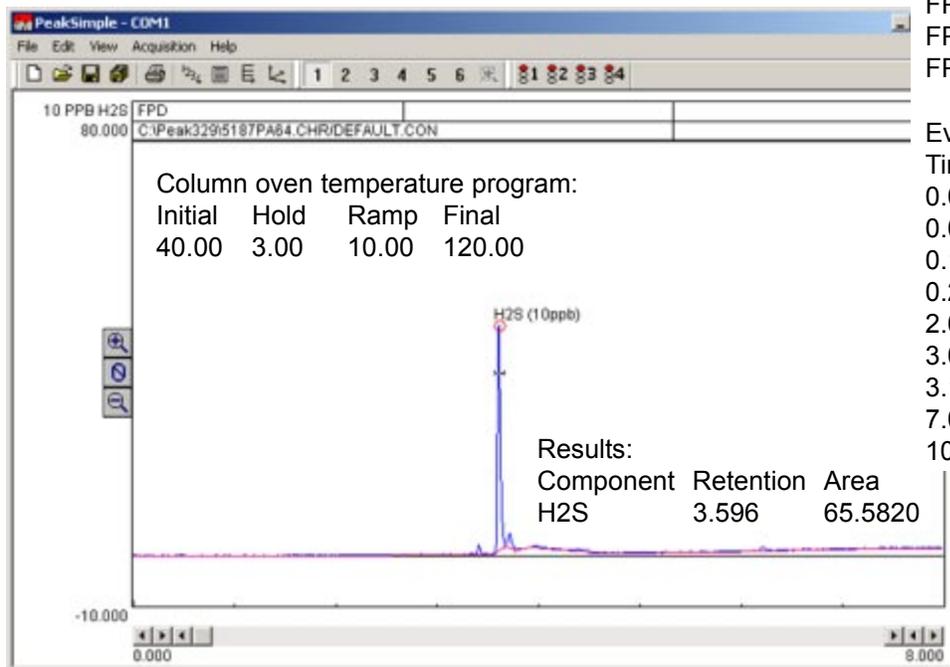
Event Table:

Time	Event
0.000	Zero baseline
0.050	D OFF (vacuum pump)
0.100	A OFF (CryoCooler)
0.200	F ON (trap heat)
2.000	G ON (valve INJECT)
3.000	F OFF (trap heat)
3.100	G OFF (valve LOAD)
7.000	A ON (CryoCooler)
10.000	D ON (vacuum pump)

Column oven temperature program:

Initial	Hold	Ramp	Final
40.00	3.00	10.00	120.00

Results:

Component	Retention	Area
H2S	3.596	65.5820

Sample: 1000ppm C1-C6
Column: 60-meter MXT-1
Carrier: helium at 10mLs/minute
FID gain: HIGH
FID temperature: 150°C
FID ignitor: -400

Column oven temperature program:

Initial	Hold	Ramp	Final
40.00	3.00	10.00	140.00

Results:

| Component	Retention	Area
Methane/ethane	2.116	2077.6740
Propane	2.383	1905.1160
Butane	3.183	2378.4140
Pentane	5.050	2326.9040
Hexane	8.050	1567.5950
Total		10255.7030
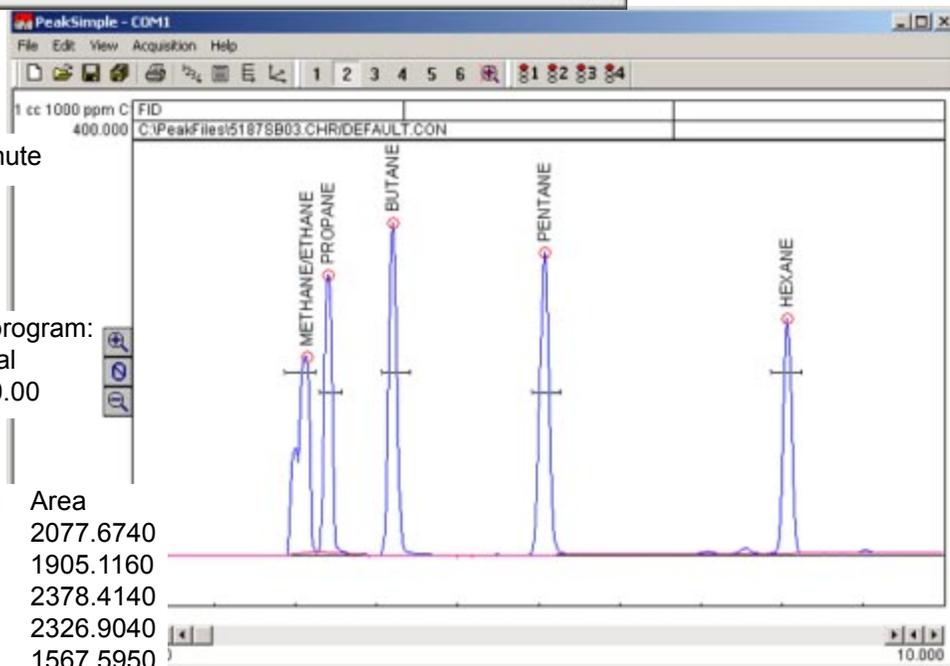
Expected Performance continued

The following two noise runs were generated by a CryoSulfur GC. Both used the same isothermal column oven temperature program, 60-meter MXT-1 column, and helium carrier at 10 milliliters per minute.
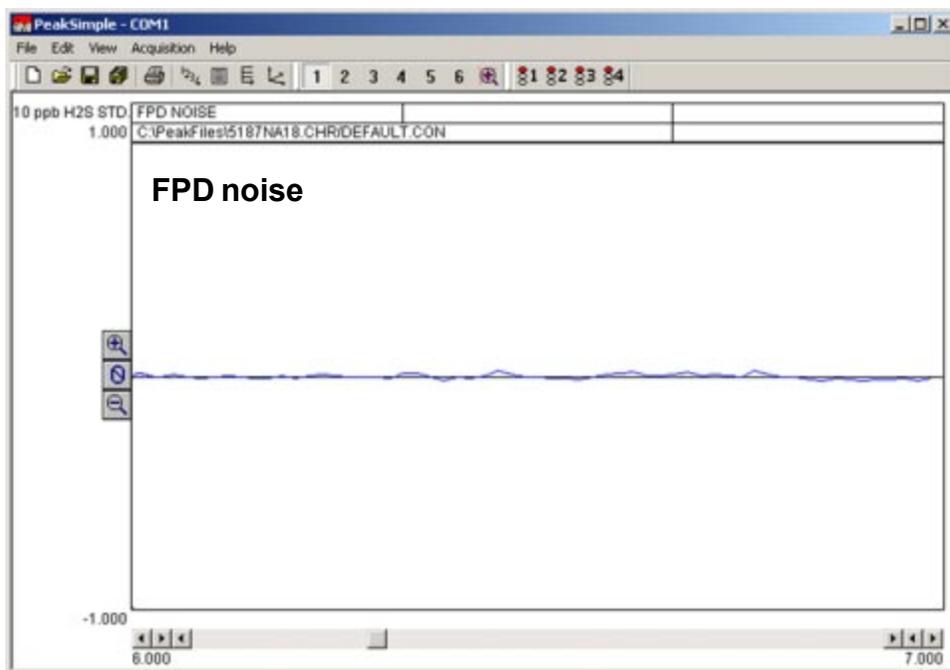
FPD gain: HIGH
FPD temperature: 150°C
FPD volts: 500

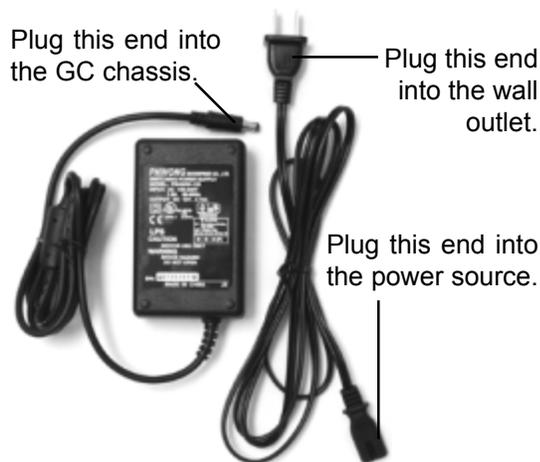Column oven temperature program:

Initial	Hold	Ramp	Final
80.00	24.00	0.00	80.00
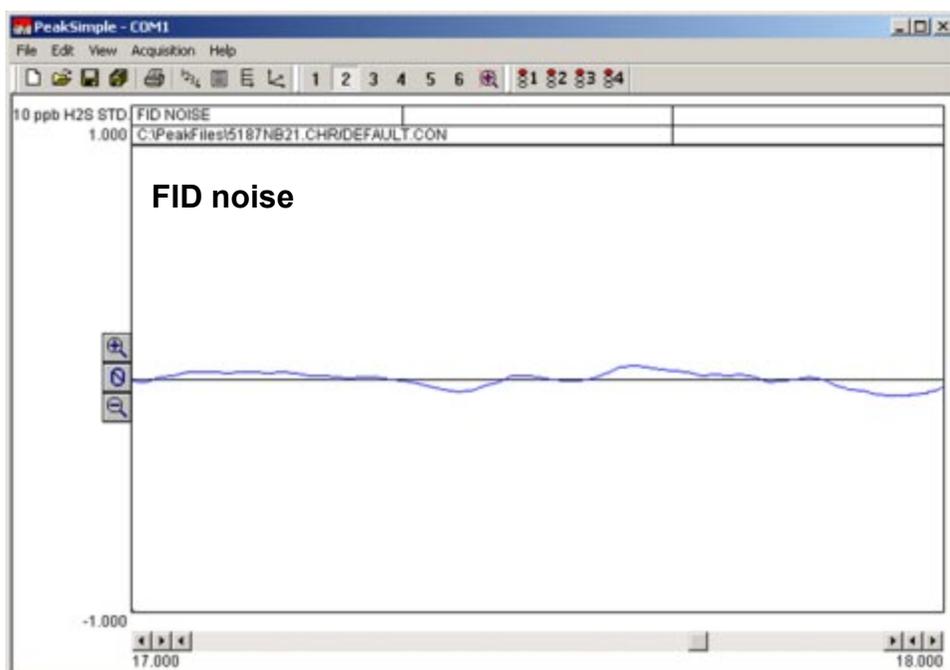
FID gain: HIGH
FID temperature: 150°C
FID ignitor: -400

General Operating Procedure

CryoCooler power source:

Plug this end into the GC chassis.

Plug this end into the wall outlet.

Plug this end into the power source.

1. Plug the GC power cord into a wall outlet and turn the main power ON. Plug the CryoCooler into it's power source, and plug the power source cord into the wall outlet. The CryoCooler power source plugs into the GC chassis between the vacuum pump interface and the Air Out fitting on the left-hand side of the GC as shown below.

Plug the CryoCooler's power source into the GC chassis here:
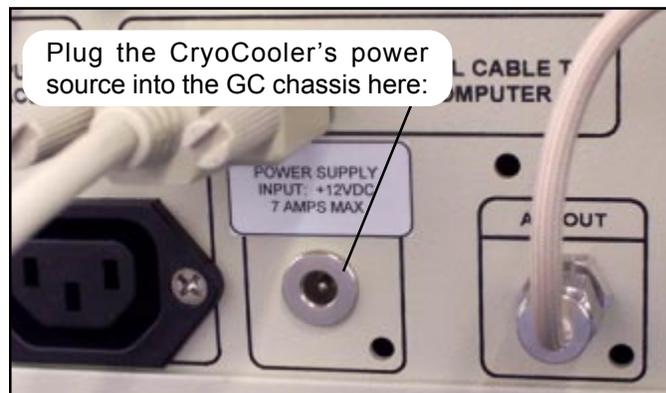
2. Connect your helium source to the carrier gas inlet on the left-hand side of the GC. Connect your hydrogen source to the hydrogen inlet. Leave the jumper in place if you plan to use the built-in air compressor to supply air for the detectors. Otherwise, remove the jumper and connect your air source to the air inlet on the left control panel.

3. Set the hydrogen flow to 60-80mLs/minute; this correlates to a flow of 30-40mLs/minute each for the primary and secondary hydrogen used by the FPD/FID combination detector. Set the air flow to 100mLs/minute. The detector air supply tubing is T'd inside the GC so that 10-30mLs/minute of air flows across the face of the photomultiplier (PMT). The gas pressures required to achieve these specific flows are printed on the right-hand side of the GC.

4. Use the switch on the GC front control panel to light the detector flame (vertically labeled "FLAME IGNITE" under "DETECTOR PARAMETERS"). Often the flame can be difficult to light because of the hydrogen-rich atmosphere inside the detector body. Make sure that the PMT voltage is OFF (that switch is also on the GC front control panel, vertically labeled "PMT VOLTS" under "DETECTOR PARAMETERS"), then remove the cap nut on the detector exhaust port. KEEP YOUR FACE AWAY FROM THE DETECTOR WHILE LIGHTING THE FLAME, and try the ignitor switch again. When the flame lights, there will be a loud noise like the backfiring of a car; this is normal and does not indicate a problem. The noise is accompanied by a flash of flame. Replace the exhaust cap nut after the flame is lit.

Exhaust port cap nut

Ignitor
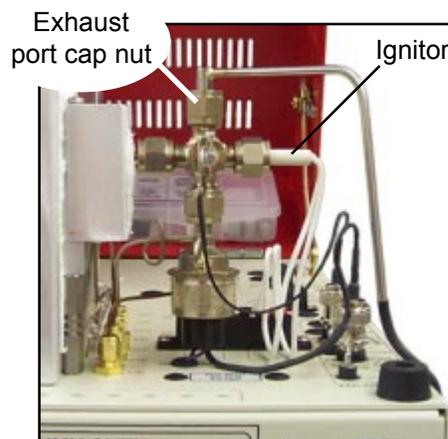
5. Switch ON the PMT voltage and set it at 400-500 by adjusting the appropriate trimpot ("PMT VOLTAGE" under "DETECTOR PARAMETERS"). The greater the voltage setting, the higher the FPD sensitivity. The PMT volts were set at 500 for the 10ppb H2S analysis shown on the Expected Performance page.

General Operating Procedure continued

6. Connect the provided vacuum pump to the SAMPLE OUT/VACUUM PUMP fitting on the front of the valve oven. Plug the vacuum pump power cord into the vacuum pump interface outlet on the left-hand side of the GC.

7. Connect your sample source to the SAMPLE IN fitting. It is critical to use as inert a sample pathway as possible to avoid absorption of sulfur compounds. You can also sample ambient air through the SAMPLE IN inlet, just remove the fitting.

8. In PeakSimple, type in the following event table:

Event Table:

Time	Event
0.000	Zero baseline
0.050	D OFF (vacuum pump)
0.100	A OFF (CryoCooler)
0.200	F ON (trap heat)
2.000	G ON (valve INJECT)
3.000	F OFF (trap heat)
3.100	G OFF (valve LOAD)
7.000	A ON (CryoCooler)
10.000	D ON (vacuum pump)

Then, in the Edit > Overall window, uncheck the box labeled "Reset relays at end of run."

9. The system works best when operated automatically every 20 minutes. The CryoCooler and vacuum pump are left ON between runs for sampling. The first run's results are ignored, as it takes multiple runs to equilibrate the sulfur compounds inside the GC system and achieve reproducibility. In PeakSimple, open the Channels window by clicking Edit > Channels. Click the Postrun button for channel 1. Click the "Save file as" checkbox, type a name for the chromatograms in the form field, and click the "Auto-increment" checkbox. For example, if you type in Sulfur, the second chromatogram will be saved as Sulfur1, followed by Sulfur2, and so on. Click the checkbox labeled "Restart run after" and type 20 minutes in the form field. Type the number of times you want to repeat the analysis into the "time total" form field.
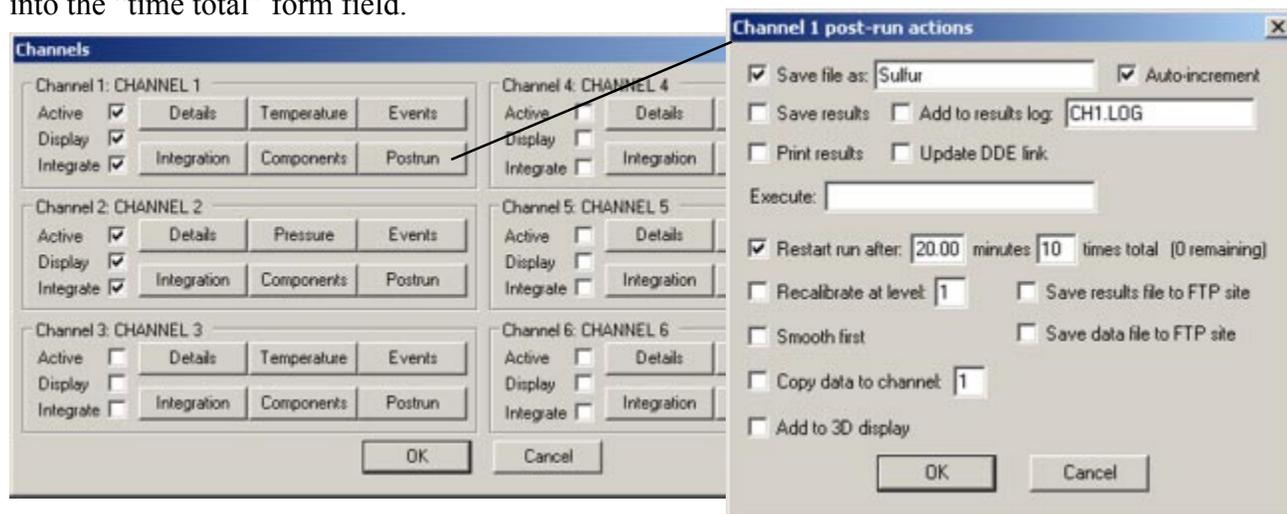
10. Press the START button on the front of the GC, or press your computer keyboard spacebar to start the run.