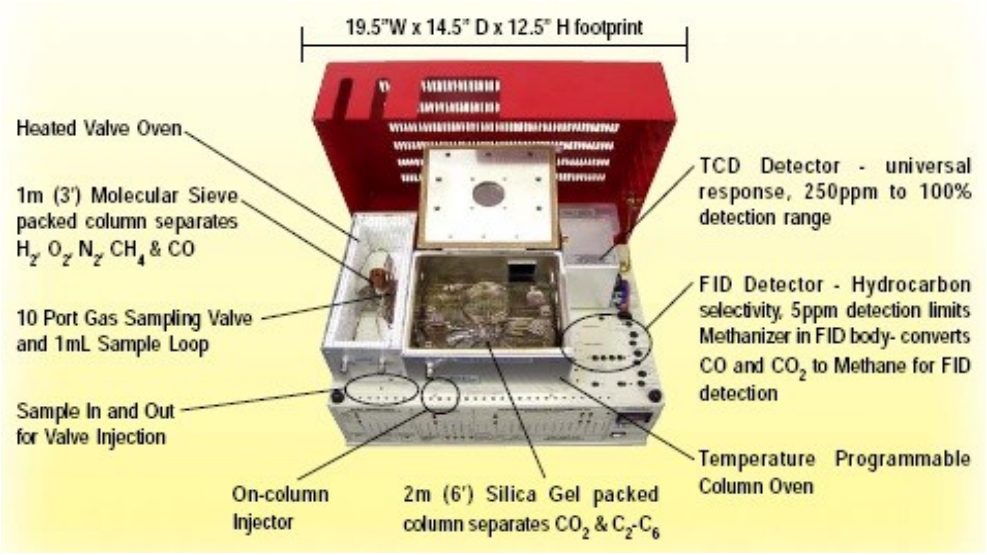
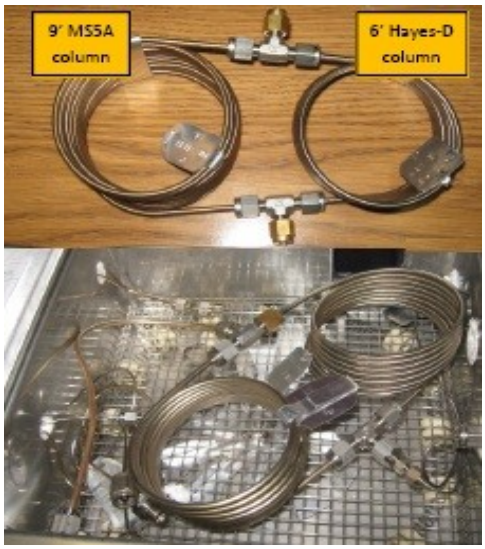
SRI Multi Gas Analyzer 2016

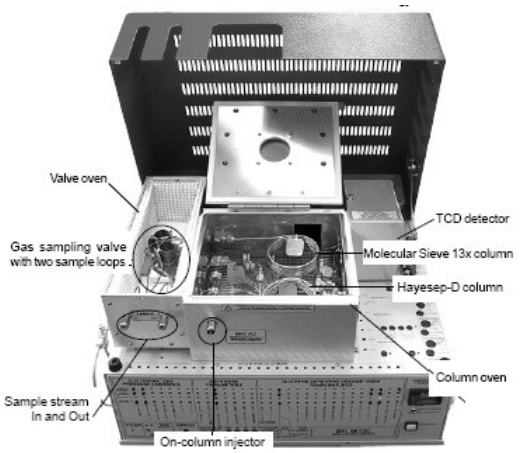
MG#1

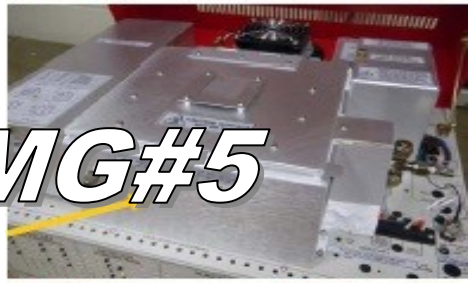


CTR1 dual Column



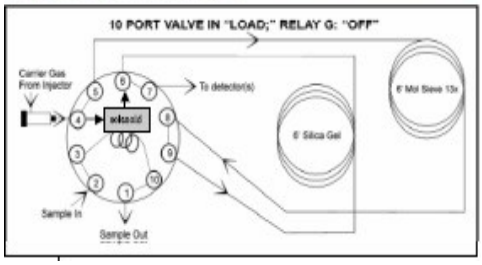
MG#2



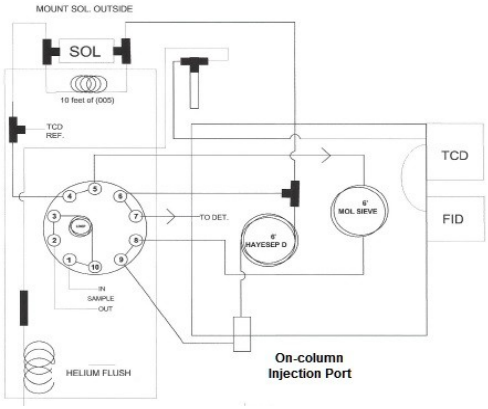


MG#5

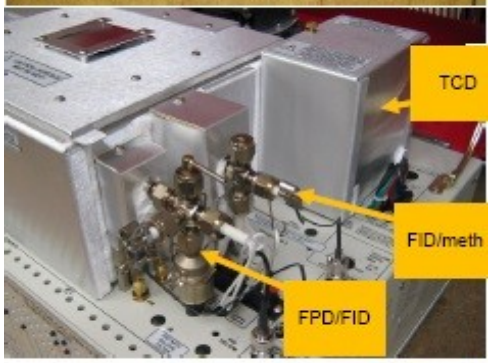
MG#3

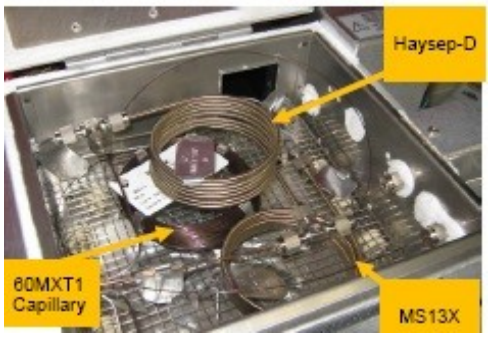


MG#3 + Injn Port



MG#3 plus Sulfur





- **TCD for 200ppM-50% Methaniser-FID to low ppM**
- **H2 analysis requires Ar Carrier**
- **Designs are cost compromised**
- **Multiple Columns require Technical Understanding and chromatogram interpretation due to some peak duplication**

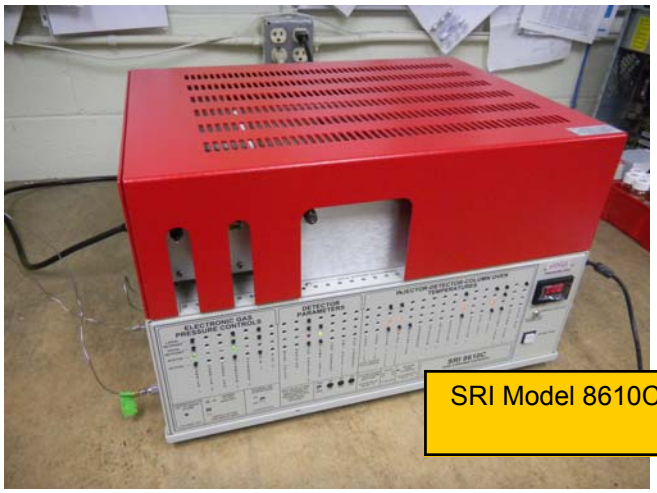
Multiple Gas#5 GC configuration
Jan 2016

History:
Unfortunately there is no single column that can separate:
Hydrogen
Oxygen
Nitrogen
Methane
CO
CO2
Ethane
Water
Propane
Butane
Pentane

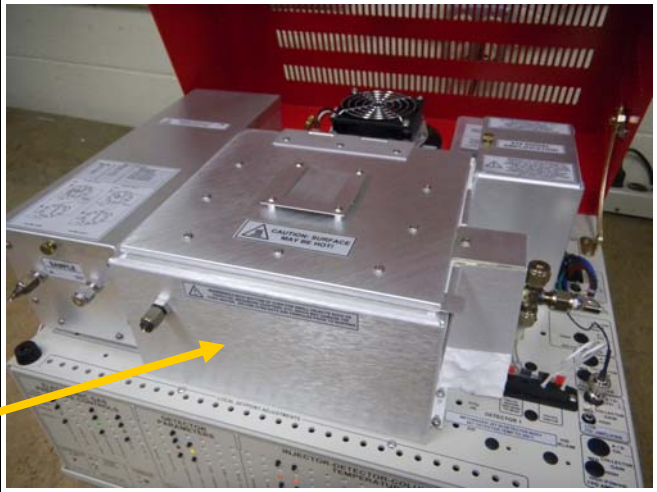
Over the years SRI Instruments has devised several solutions to this analytical problem, starting with the MultipleGas#1 configuration and evolving to the present MultipleGas#5 configuration.

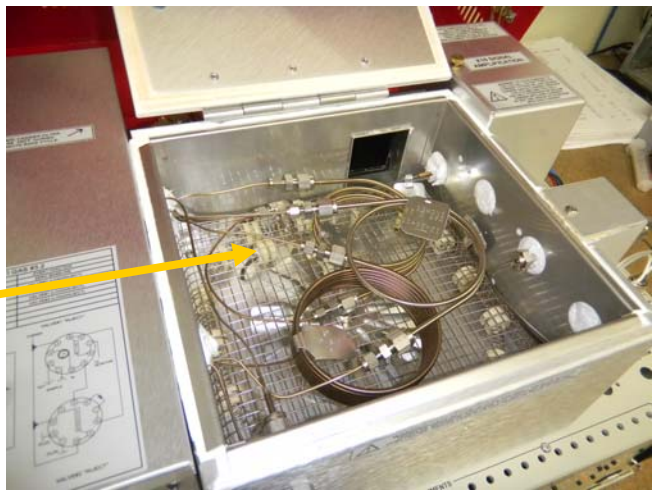
Like the earlier MG GCs the 8610C chassis includes an ambient to 400C programmable column oven.

Inside the column oven are three columns. There can be additional columns, but the basic MG5 includes:
.5 meter Haysep-D precolumn
2 meter MoleSieve 5A column
2 meter Haysep D column



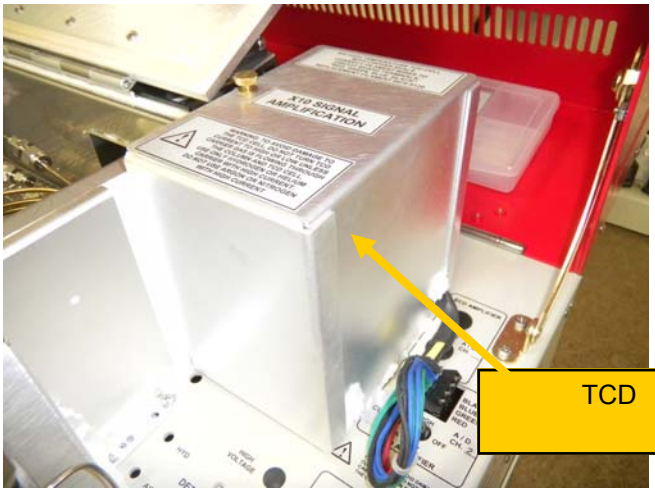
SRI Model 8610C



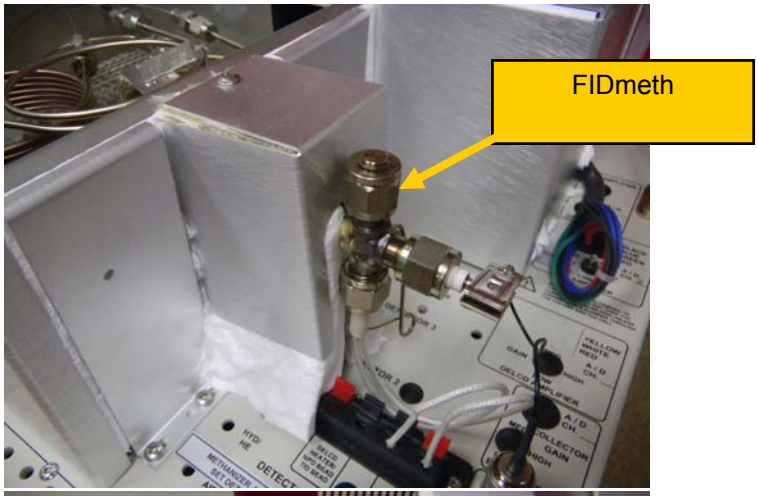


Multiple Gas#5 GC configuration
Jan 2016

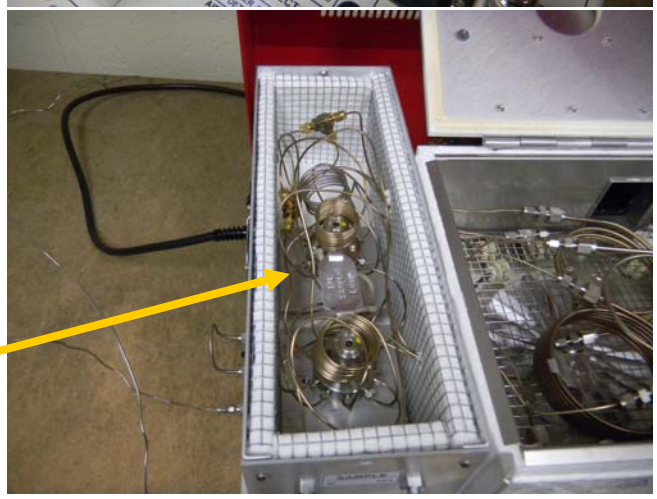
On the right side of the column oven is located the Thermal Conductivity Detector (TCD) which detects all the gases from 200ppm to 100% except hydrogen (see detailed explanation for this later).



Most MG5 configurations will also include a Flame Ionization Detector (FID) usually also including a Methanizer (FIDmeth) to enable the FID to also detect CO and CO2 from 1ppm to 50,000ppm. The FID can only detect hydrocarbons like methane and ethane, but when equipped with a methanizer, CO and CO2 are reacted to methane and thus detected at the same sensitivity as methane.



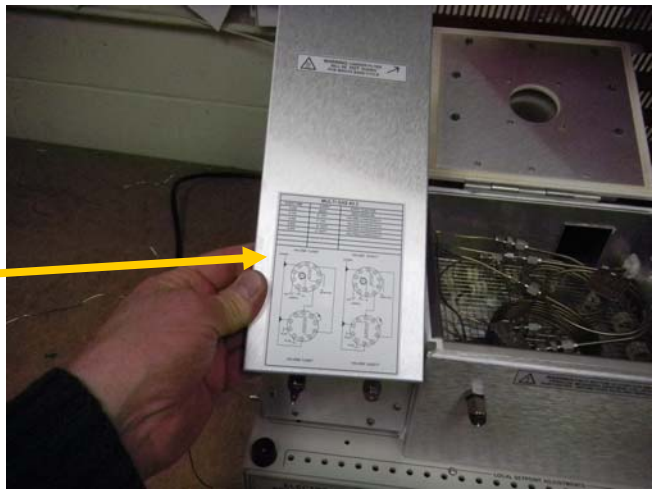
On the left side of the column oven is the valve oven, which contains two 10port Valco valves and lots of 1/16” stainless steel tubing.



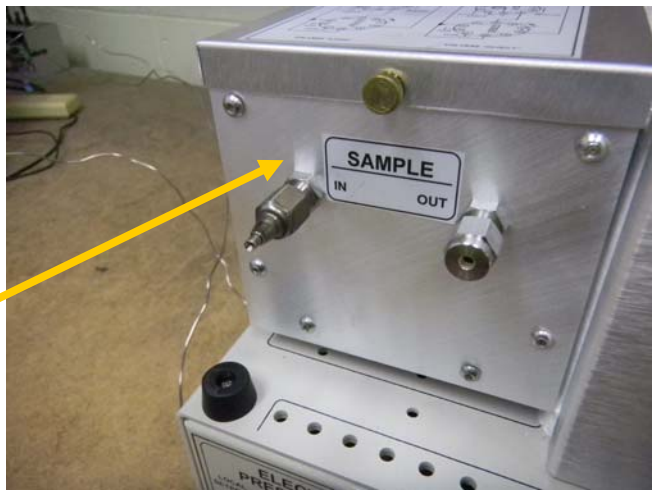
Multiple Gas#5 GC configuration
Jan 2016

The Valco 10port gas sampling valve (GSV) looks like this. There is an electric motor inside the GC which turns a Teflon seal inside the valve at a specific time during the analysis to change the carrier gas flow path.

We put a map on the cover of the valve oven so you can follow the carrier gas flow path in both positions (load and inject). It is important to understand the flow path to troubleshoot and optimize the analysis. It is also critical to understand that the valve does not "open" or "close". Rather, the carrier flows in one path or the other, but it always flows continuously.

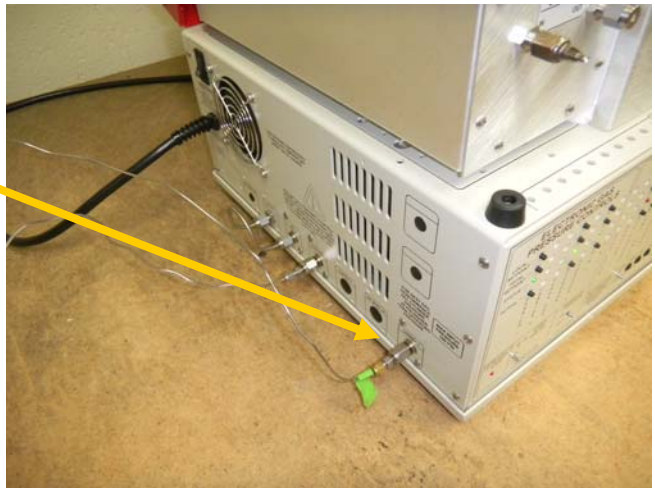


The sample to be analyzed is loaded at the front of the valve oven. The sample can flow from the "sample IN" through the "loop" and then out the "Sample OUT" continuously, or it can be flushed with new sample prior to starting an analysis. Normally it takes a minimum of 10ml of sample to flush the loop. There is no restriction or pressure to work against. You could blow through from "In" to "Out" with your mouth.



Multiple Gas#5 GC configuration
Jan 2016

The carrier gas is connected to the left side of the GC. The carrier gas can be helium, hydrogen, nitrogen or argon. Inside the GC there is a very precise pressure regulator called an " Electronic Pressure Controller" (EPC) which supplies the carrier gas at a stable pressure to the valves and columns.

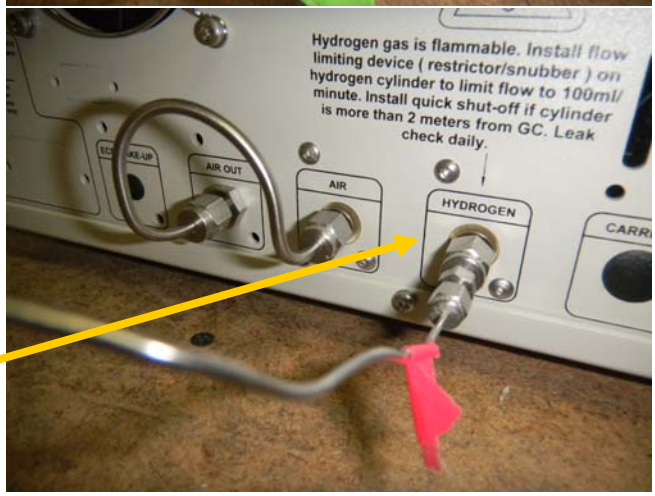


Helium is the most common choice because it gives the best overall results. However the sensitivity is not as good for hydrogen as it is for the other gases. This is because the TCD sensitivity depends on the difference of the "thermal conductivity" of the carrier gas relative to the sample molecule. The "thermal conductivity" difference between helium and hydrogen is very small

Hydrogen is sometimes used as carrier, but when it is, there is no sensitivity for hydrogen at all.

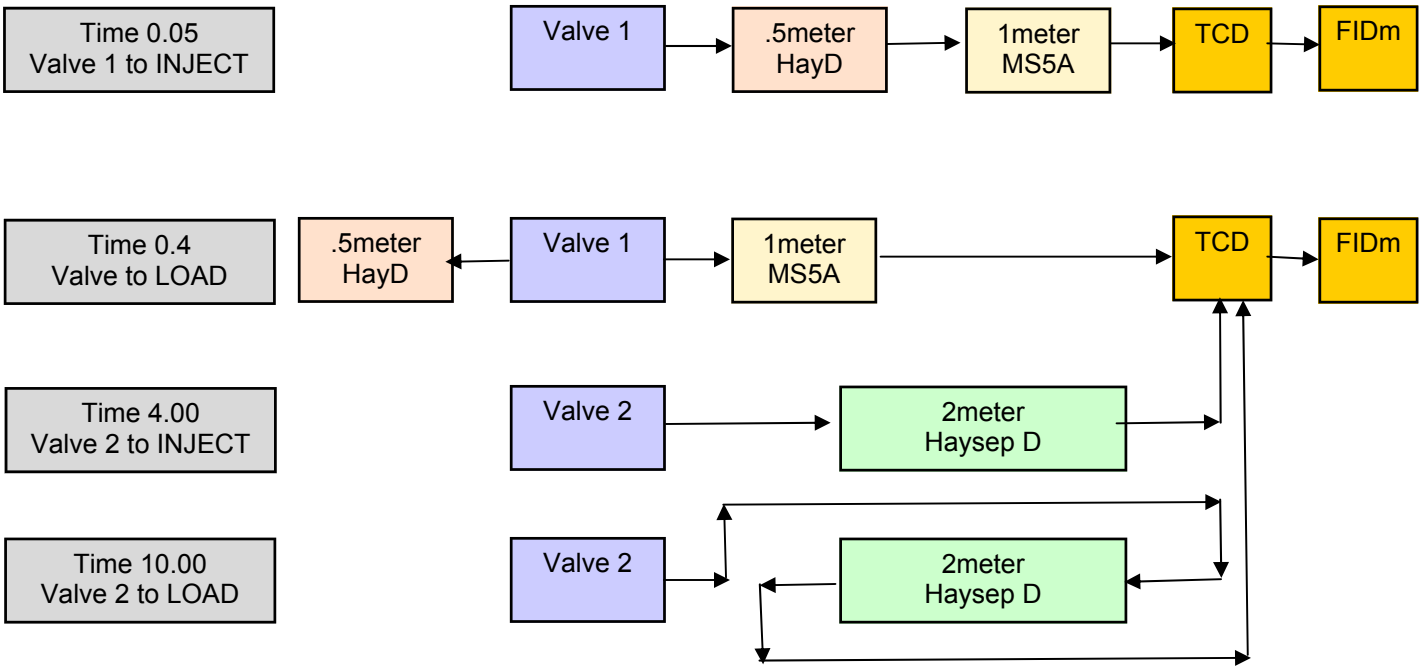
Nitrogen is sometimes used especially where it is important to measure hydrogen. Naturally, if nitrogen is used as carrier gas, there is no sensitivity to measure nitrogen.

Argon is used where it is important to measure hydrogen and also oxygen and nitrogen.



If an FIDmethanizer detector is also installed, then hydrogen is also connected on the left side of the GC. Air is typically supplied from the built-in air compressor, but can also be supplied from an external air cylinder. Both hydrogen and air are required for the FID flame.

Multiple Gas#5 GC configuration
Jan 2016



The schematic above shows the 4 steps in the MG5 analysis after the sample has been loaded into the loop of each valve.

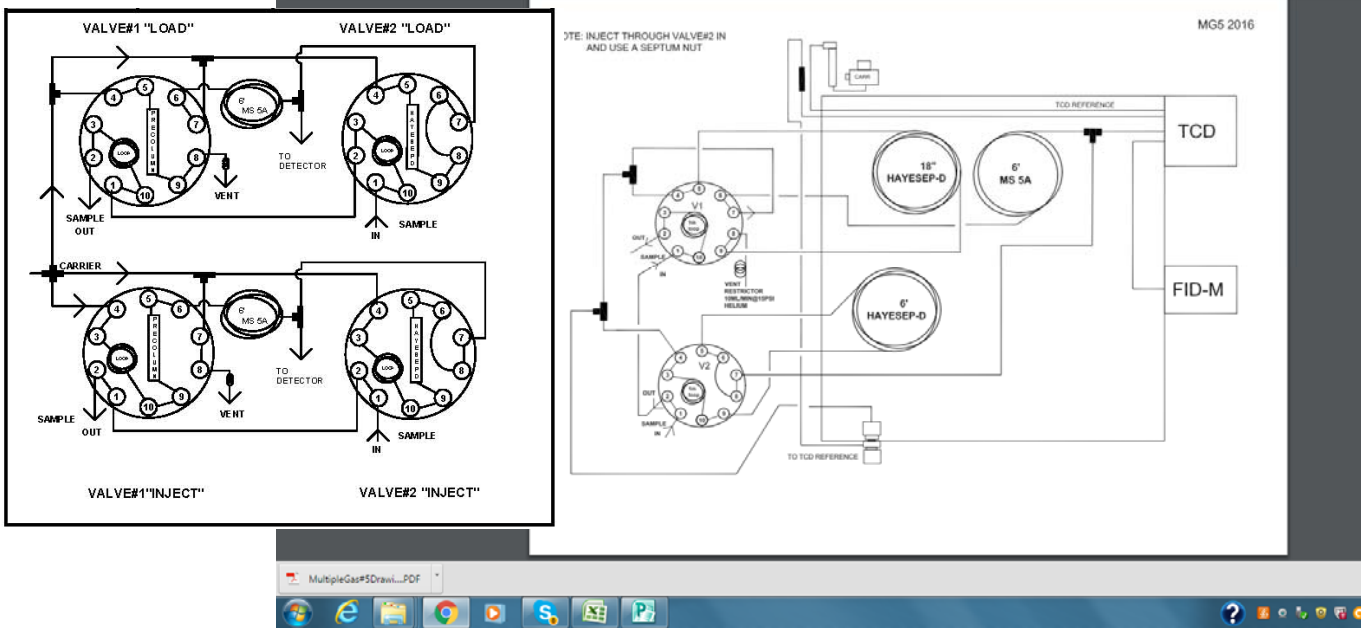
STEP 1: Valve1 is turned to the INJECT position (Relay G on). The carrier gas pushes the sample out of the valve loop onto the 5.meter Haysep D column. H2, O2, N2 CH4 and CO migrate through the .5meter HayD column very quickly and land on the 1meter MS5A column.

STEP 2: Valve1 is turned back to the LOAD position (Relay G off). Carrier gas continues to push the H2, O2, N2, CH4 and CO molecules through the MS5A column towards the detectors. Also carrier gas backflushes any remaining molecules backwards through the .5meter HayD column out to vent (not through the detectors). The molecules which remain on the .5meter column are CO2, Water, and C2 and higher hydrocarbons. These molecules would get stuck on the MS5A column if they were allowed onto the MS5A column. However, they easily backflush out of the HayD.

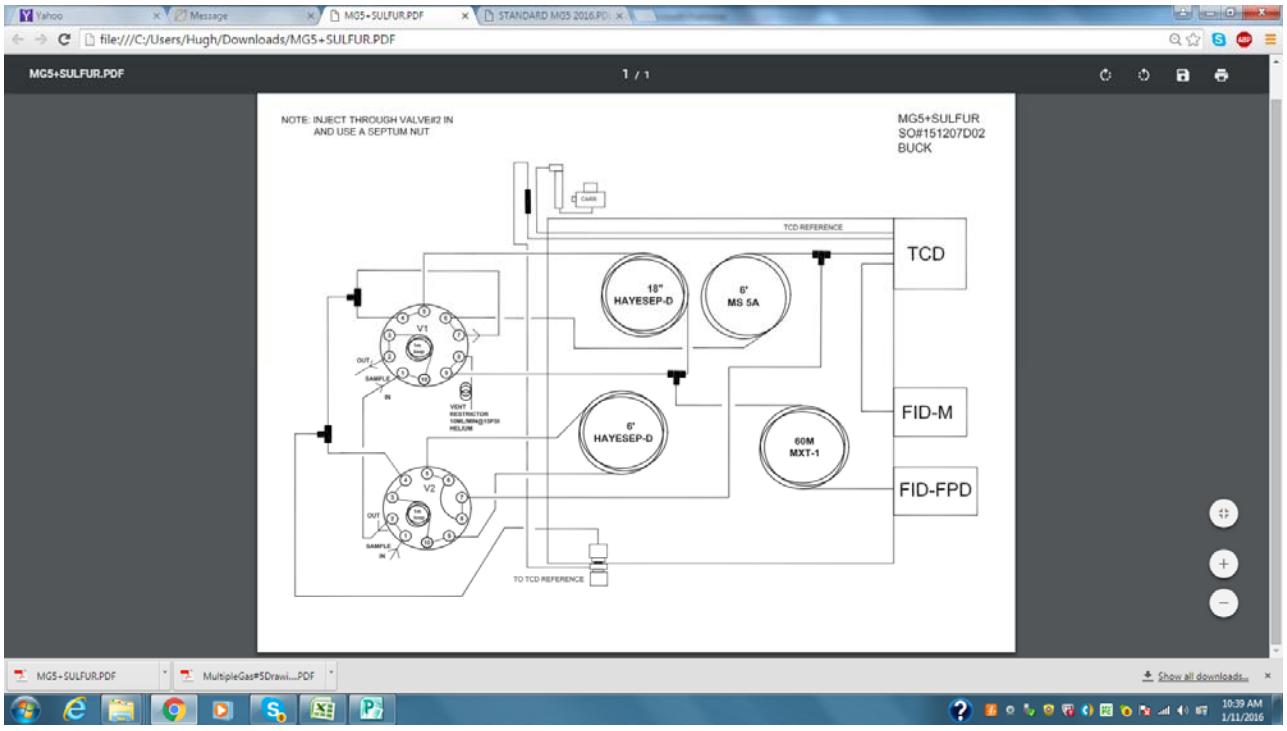
STEP 3: Valve2 is turned to the INJECT position (Relay F on). The carrier gas pushes the molecules in the loop of Valve2 onto the 2meter HayD column in the “forward” direction. H2, O2, N2 and CO elute from the column very quickly as one peak, followed by the CH4 peak, the CO2, water and the hydro-carbons from C2-C6.

STEP 4: At some point in the analysis Valve2 is returned to the LOAD position. This reverses (back-flushes) the flow direction in the HayD column. Any peaks which have not yet exited the HayD column now back out of the column and into the detector. If, for example the analysis had no peaks after CO2 (or you did not care about any peak after CO2), then you would backflush after the CO2 peak. Any peaks remaining in the HayD column would come out in a “lump”.

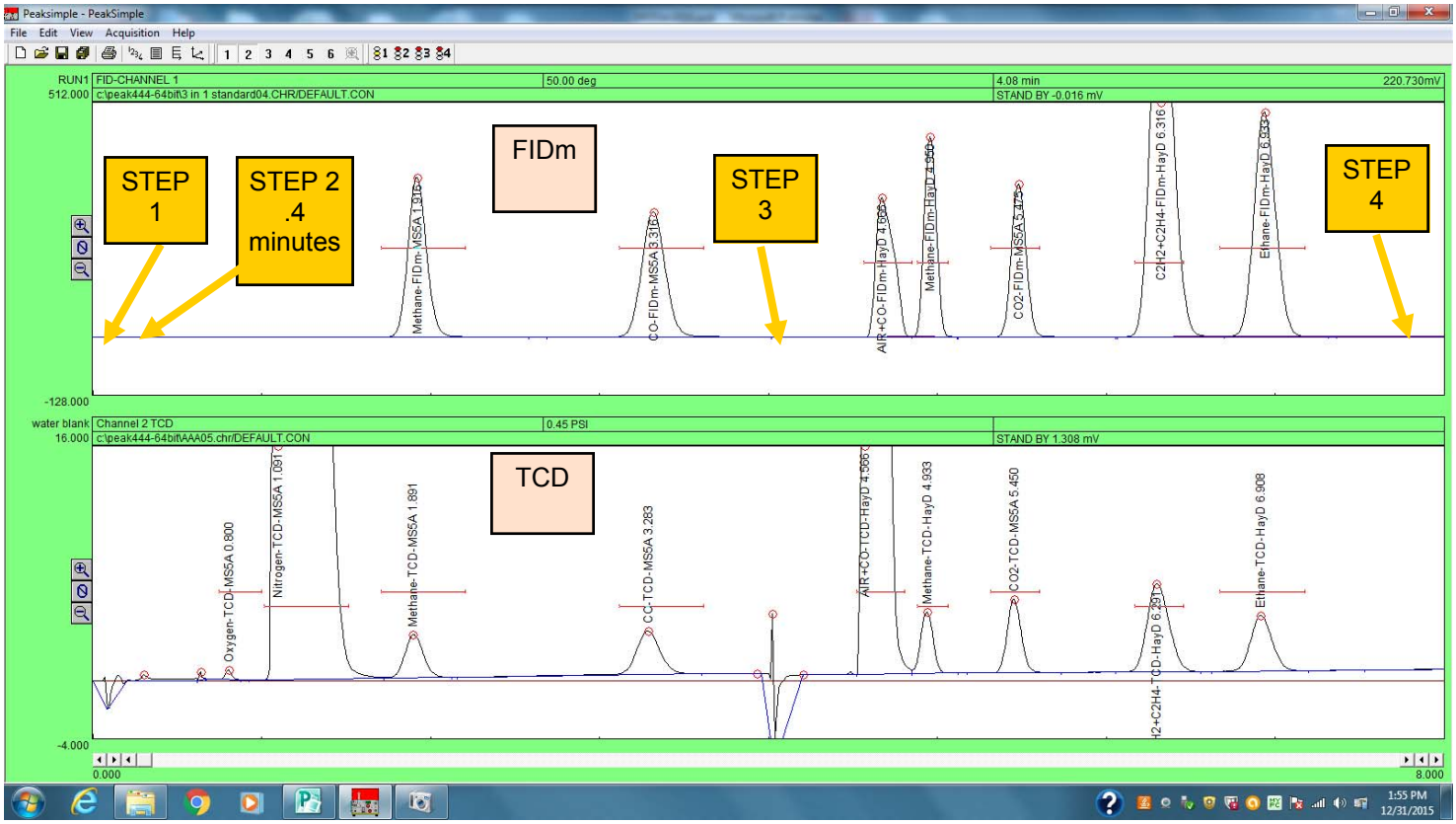
Multiple Gas#5 GC configuration
Jan 2016



The diagram above shows a schematic of the “basic” MG#5 configuration with both valves in the INJECT position. A similar diagram below shows the “basic” MG#5 plus an additional column and FPD/FID detector to measure sulfur gases like H2S, CO2, SO2, mercaptans, DMS etc.



Multiple Gas#5 GC configuration
Jan 2016



This is a typical chromatogram of gases at 1% in Nitrogen. The FIDmethanizer chromatogram is on the top and the TCD on the bottom.
STEP 1: Valve1 is turned to the INJECT position (Relay G on). The carrier gas pushes the sample out of the valve loop onto the 5.meter Haysep D column. H2, O2, N2 CH4 and CO migrate through the .5meter HayD column very quickly and land on the 1meter MS5A column.

STEP 2: Valve1 is turned back to the LOAD position (Relay G off) at .4 minutes. Carrier gas continues to push the H2, O2, N2, CH4 and CO molecules through the MS5A column towards the detectors. Also carrier gas backflushes any remaining molecules backwards through the .5meter HayD column out to vent (not through the detectors). The molecules which remain on the .5meter column are CO2, Water, and C2 and higher hydrocarbons. These molecules would get stuck on the MS5A column if they were allowed onto the MS5A column. However, they easily backflush out of the HayD.

STEP 3: Valve2 is turned to the INJECT position (Relay F on). The carrier gas pushes the molecules in the loop of Valve2 onto the 2meter HayD column in the “forward” direction. H2, O2, N2 and CO elute from the column very quickly as one peak, followed by the CH4 peak, the CO2, Water and the hydrocarbons from C2-C6.

STEP 4: At some point in the analysis Valve2 is returned to the LOAD position. This reverses (backflushes) the flow direction in the HayD column. Any peaks which have not yet exited the HayD column now back out of the column and into the detector. If, for example the analysis had no peaks after CO2 (or you did not care about any peak after CO2), then you would backflush after the CO2 peak. Any peaks remaining in the HayD column would come out in a “lump”.

Multiple Gas#5 GC configuration
Jan 2016

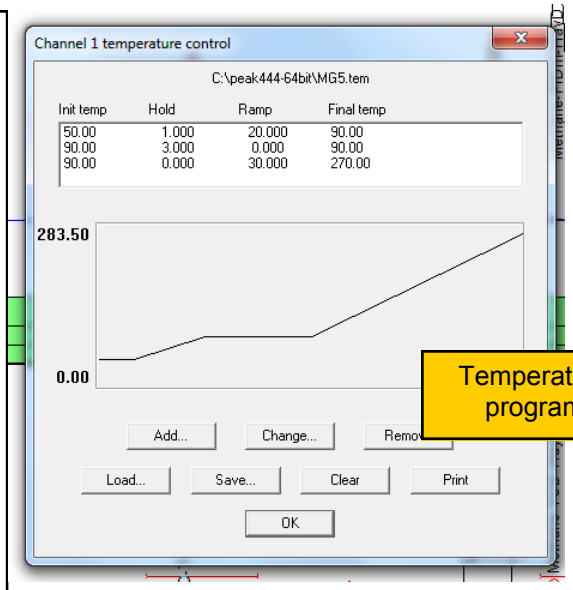
The screen at right shows the oven temperature program used.

STEPS 1 and 2 occur while the column oven is at 50C. After 1 minute, the column temperature increases to 90C and stays there until after STEP 3. Then the column temperature increases to 270C.

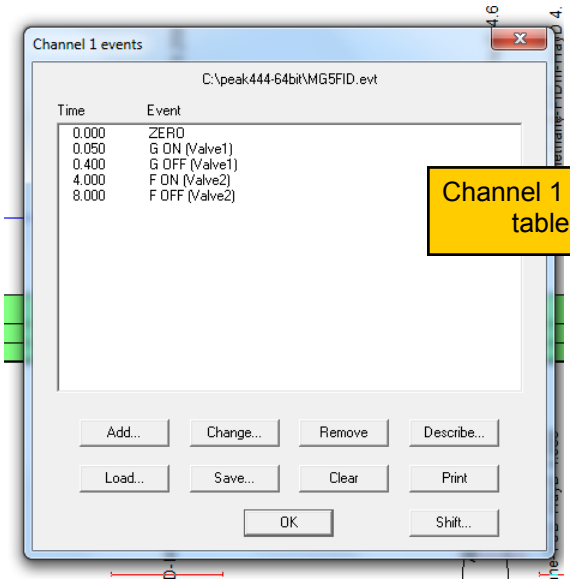
At some point while the column temperature increases, STEP 4 occurs, backflushing any un-eluted molecules.

The channel 1 Event table is shown at right.
At time 0.05 Relay G turns on. This intitiates STEP 1.
At time 0.4, Relay G turns off.
At time 4.00 Relay F turns on initiating STEP 3.
At time 8.0 Relay F turns off backflushing the Haysep D column to the detectors.

The exact times may change if the carrier flow rate changes or if a different carrier gas is used. The backflush time (STEP 4) especially may change depending on what molecules are in the sample.

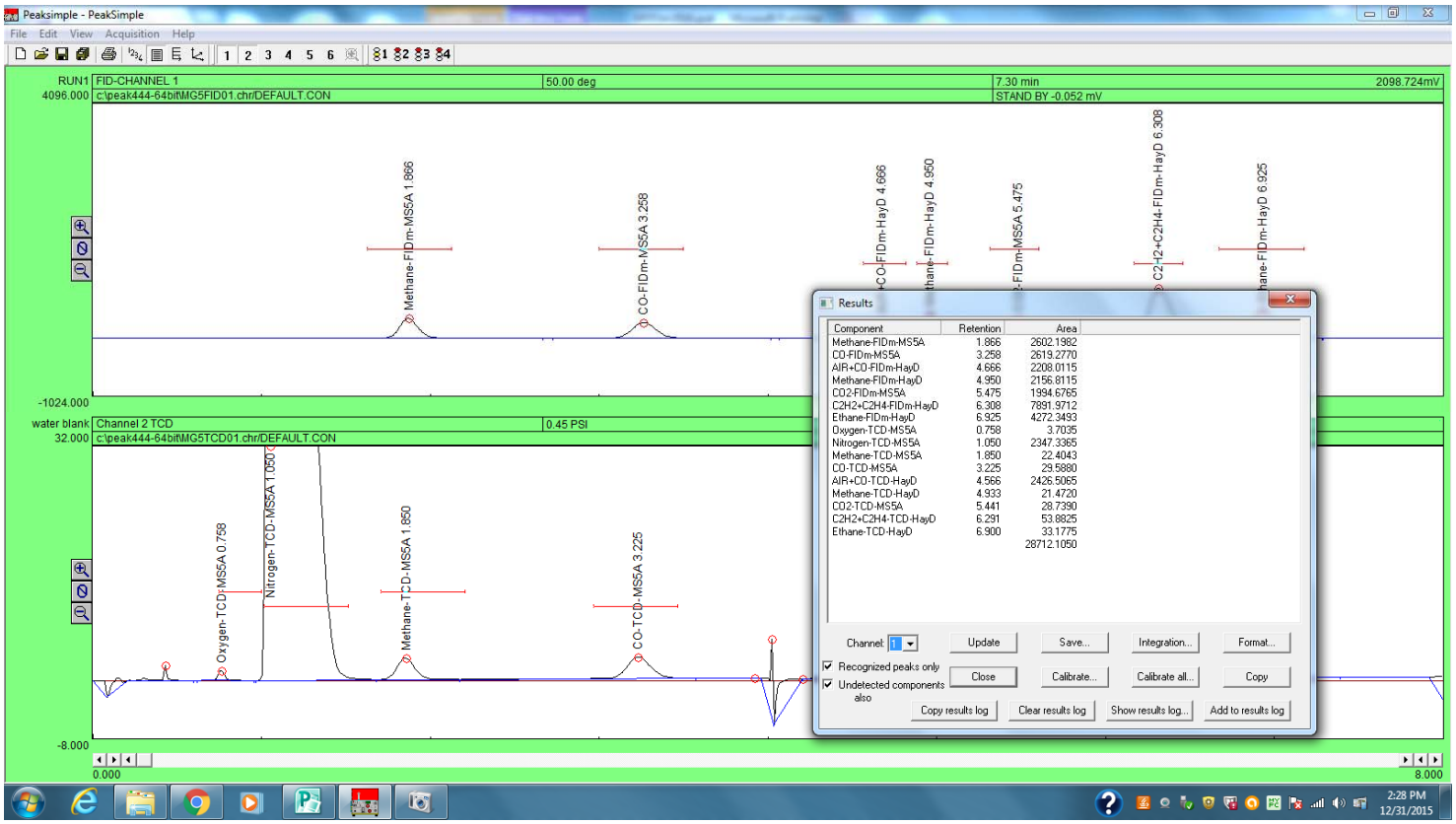


Temperature program



Channel 1 Event table

Multiple Gas#5 GC configuration
Jan 2016



The sample above (helium carrier at 15psi) shows 1% each methane, CO, CO2, ethane, ethylene and acetylene with a little oxygen, and nitrogen balance. Note that the area of the methane, and CO peaks are about the same on the FIDm, and similar but not identical on the TCD.

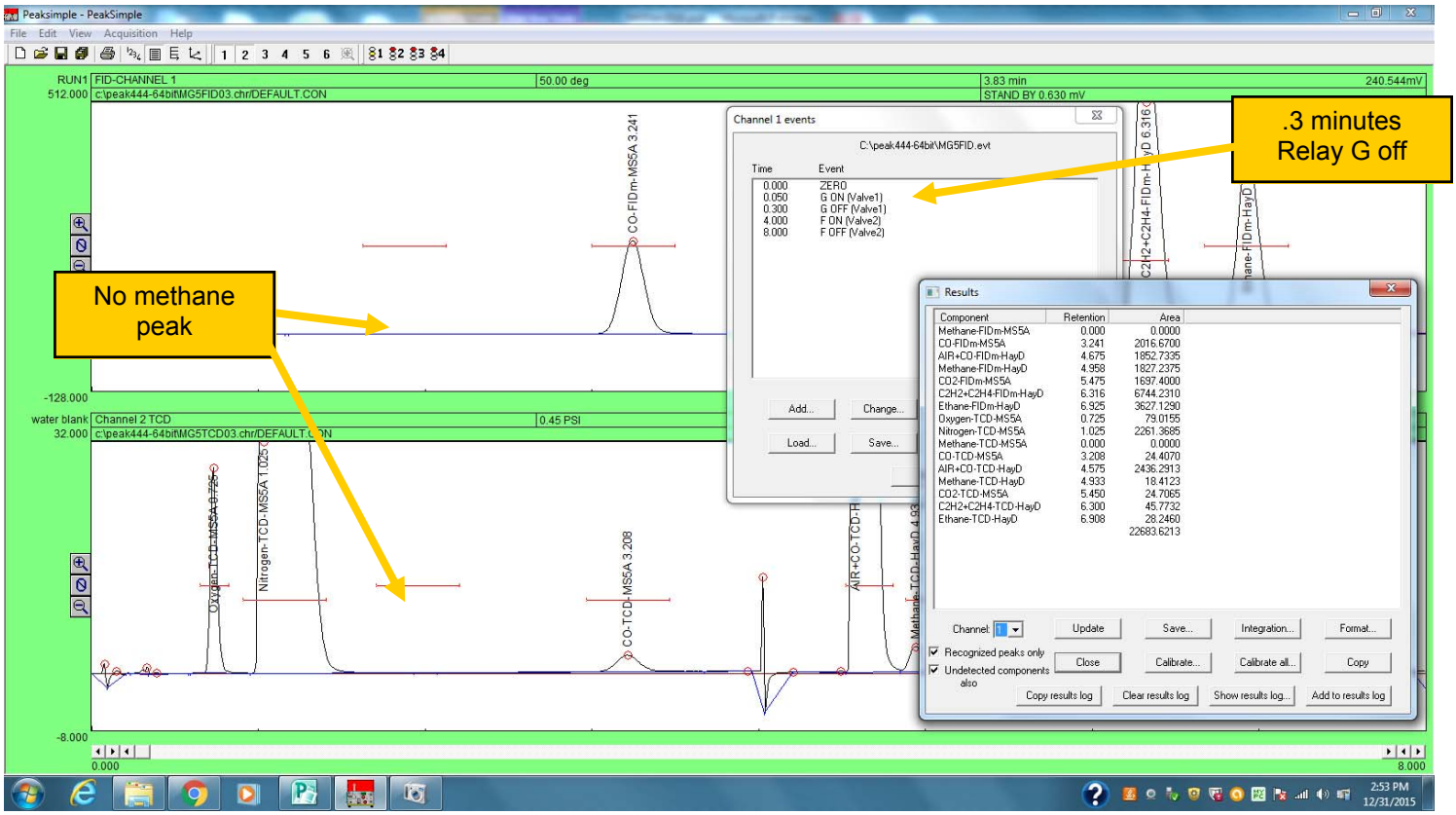
This shows that the methanizer is working 100% since every molecule of CO is converted to one molecule of methane.

It also shows that the Valve 1 timing (Relay G off at .4 minutes) is correct.

Methane and CO have different thermal conductivities so on the TCD the peak areas are slightly different from each other.

Note that on a HaysepD column, ethylene and acetylene co-elute. A different flavor of Haysep (Haysep N for example) can be substituted to separate these two molecules.

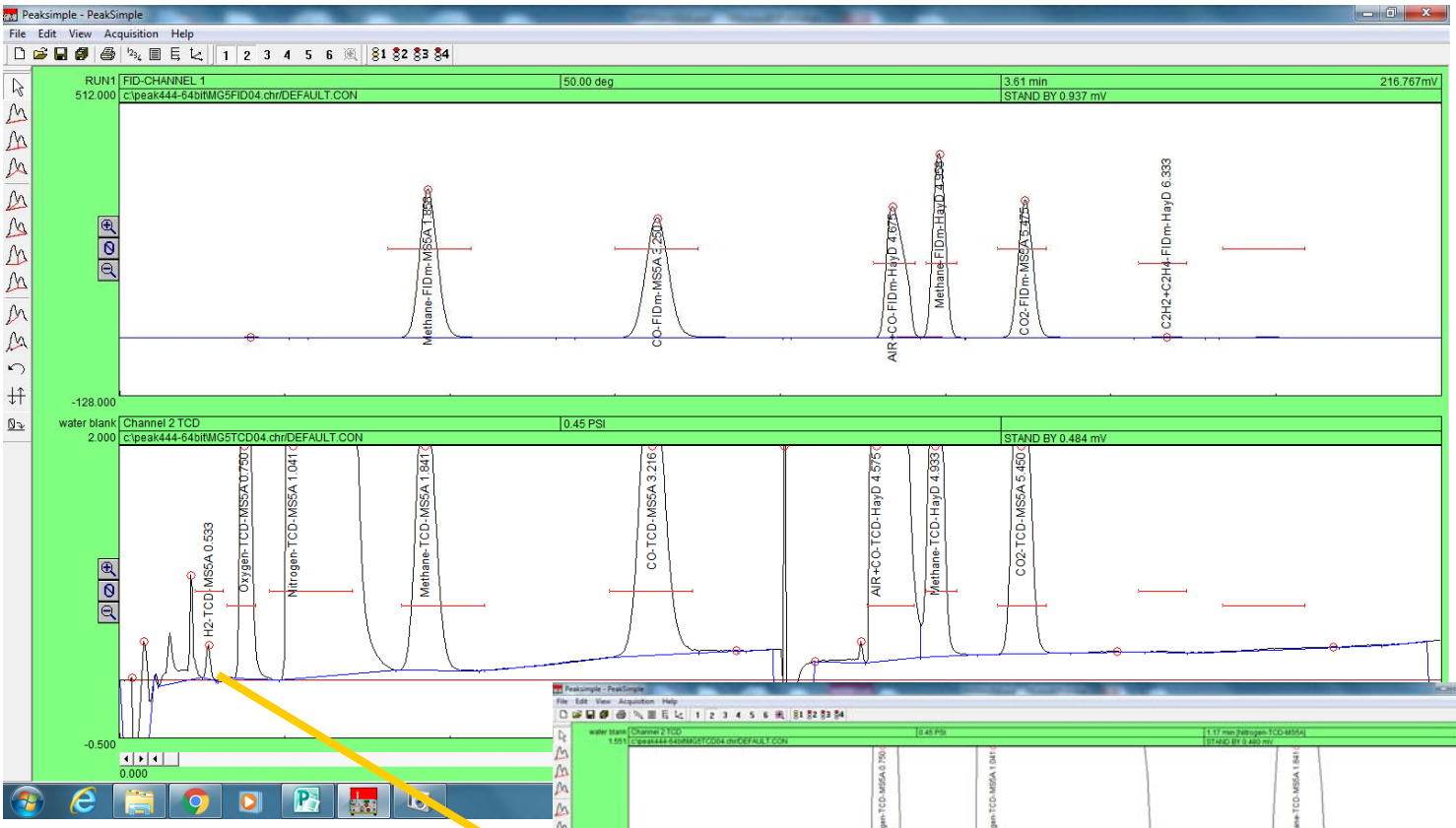
Multiple Gas#5 GC configuration
Jan 2016



Compare the same sample but with Relay G off at .3 minutes instead of .4 minutes as in the previous page. The methane peak has disappeared because the time that Relay G turned off was too early, so the methane peak did not make it onto the MS5A column and was backflushed off the pre-column (.5meter HayD) to vent.

If you change carrier gas types (argon instead of helium), carrier flow rates, column types (MS13X instead of MS5A), or column lengths, you will have to determine the correct timing by trial and error.

Multiple Gas#5 GC configuration
Jan 2016



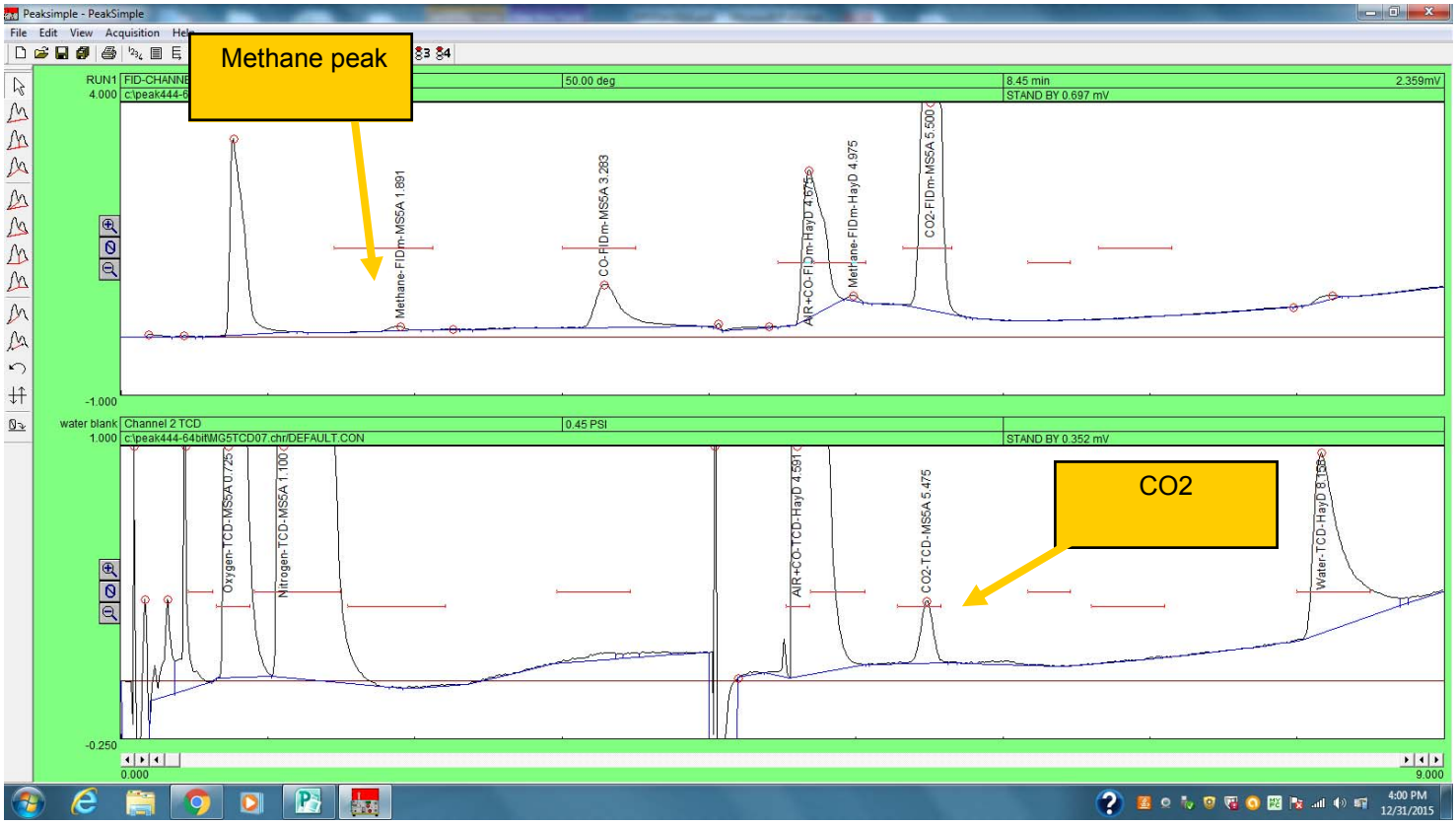
Here we show what 1% hydrogen looks like with helium carrier gas at 15psi and the same temperature program and event table.

Note how much smaller the 1% hydrogen peak is than the 1% methane peak.

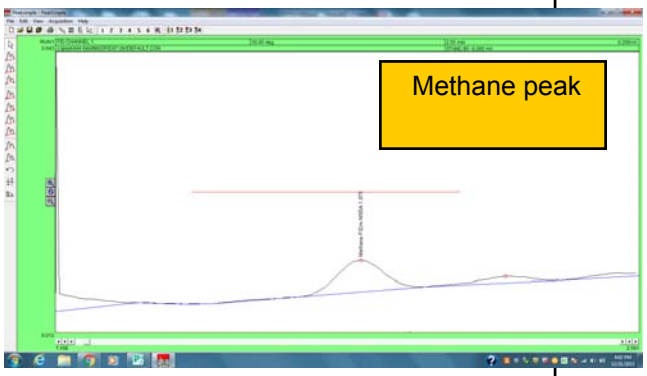
Multiple Gas#5 GC configuration
Jan 2016

This is natural gas. Notice there is no CO, but plenty of butanes and pentanes. There is also a water peak on the TCD.

Multiple Gas#5 GC configuration
Jan 2016



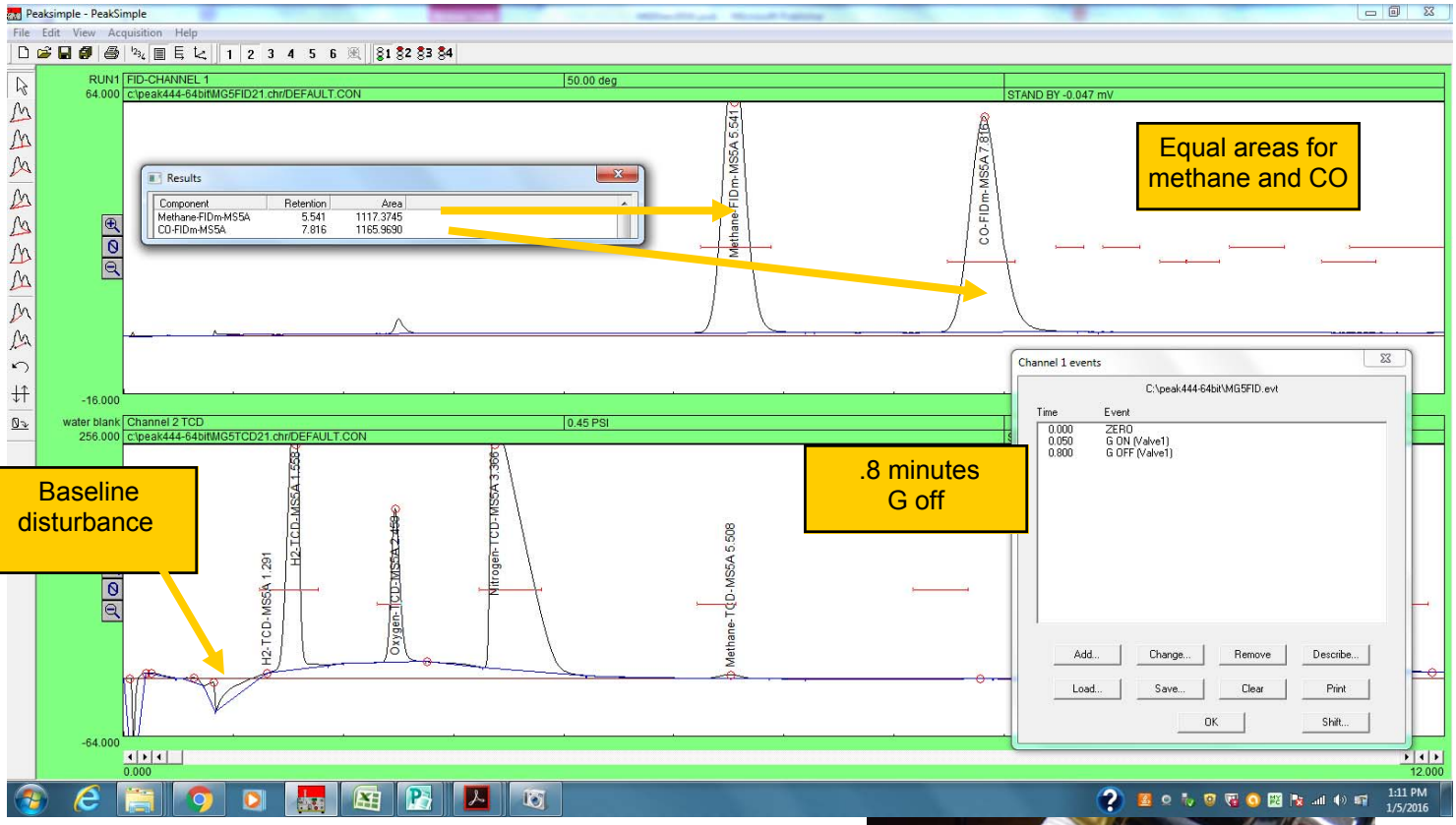
This is room air which has 2ppm of methane, 400ppm of CO2 and 10,000ppm of water.



Notice the 2ppm methane peak is easily detectable on the FIDmethanzier, and the 400ppm CO2 peak easily detectable on the TCD.

The room air in this case also apparently had some CO.

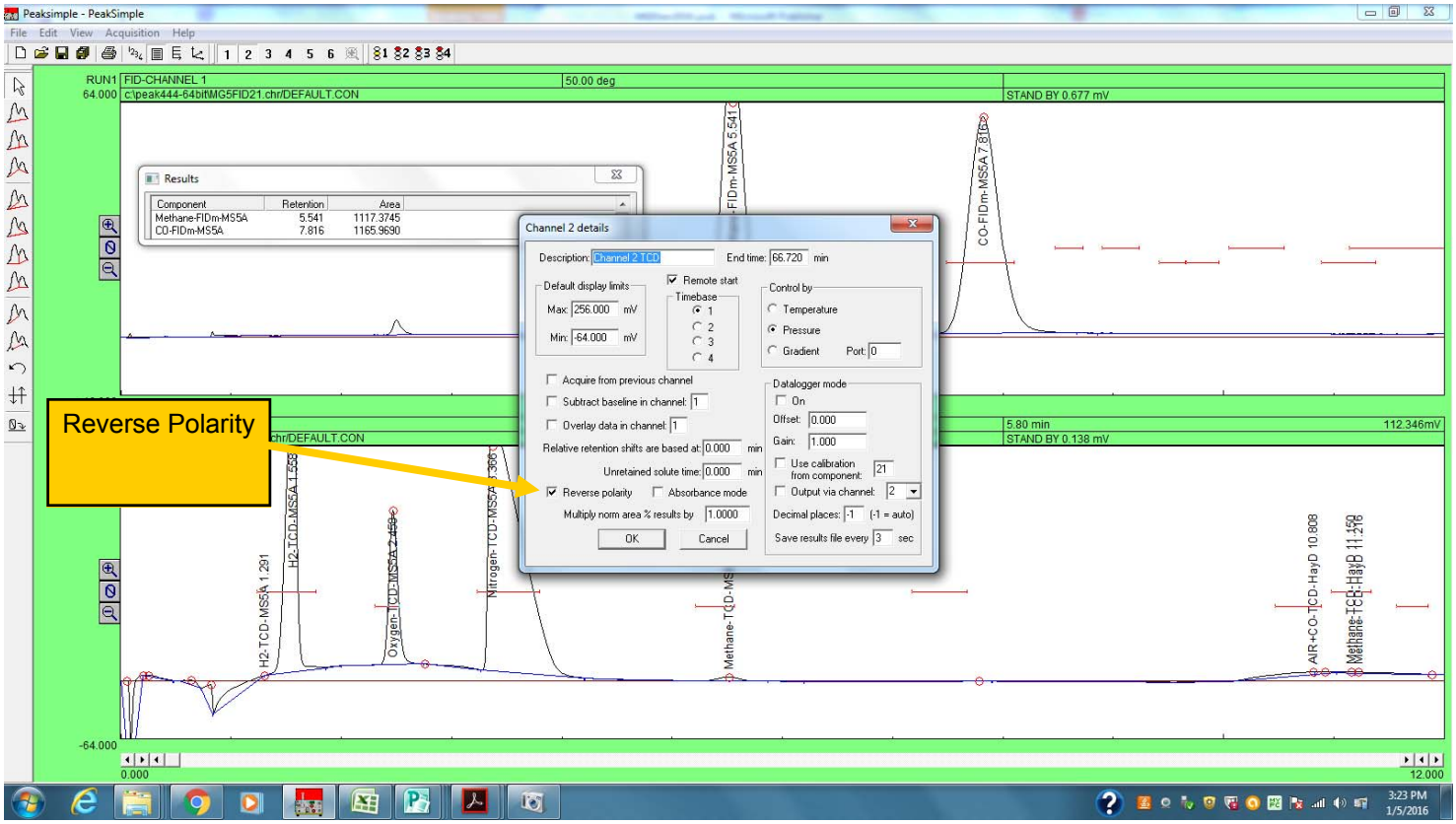
Multiple Gas#5 GC configuration
Jan 2016



If hydrogen measurement is a priority its is best to use Argon carrier gas instead of helium. Because there is an un-avoidable baseline disturbance when Relay G turns off and backflushes the pre-column, it may make sense to use a 6 foot MS5A column instead of the 3 foot MS5A column. The 6' column makes the Hydrogen peak come out a little later giving the baseline a chance to stabilize before the H2 peak begins.

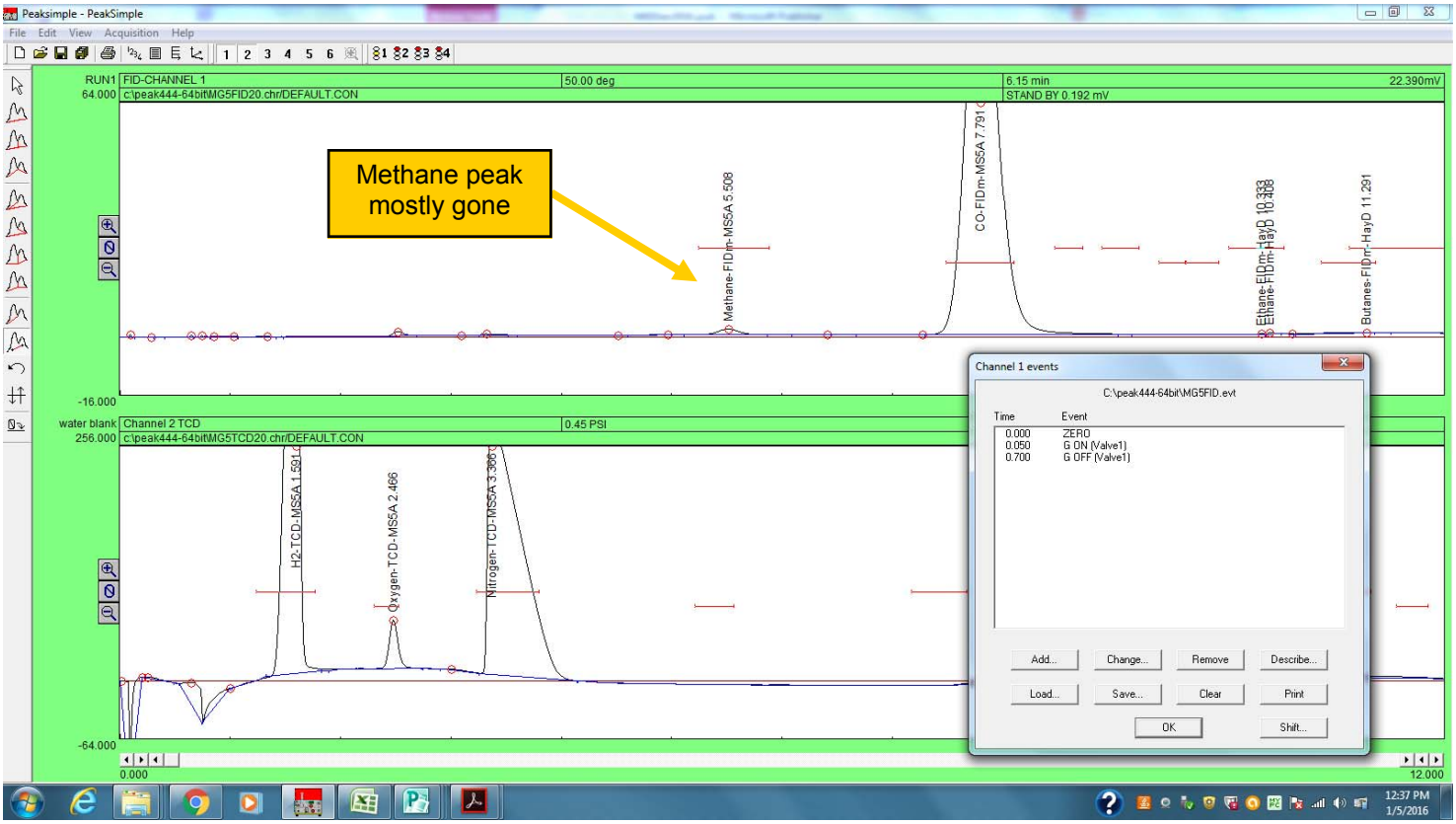
6' and 3' MS5A columns

Multiple Gas#5 GC configuration
Jan 2016



When using Argon or Nitrogen carrier gas the peaks come out upside down. In the channel 2 Details screen, click the box labelled “Reverse Polarity” so the peaks will come out in the positive direction.

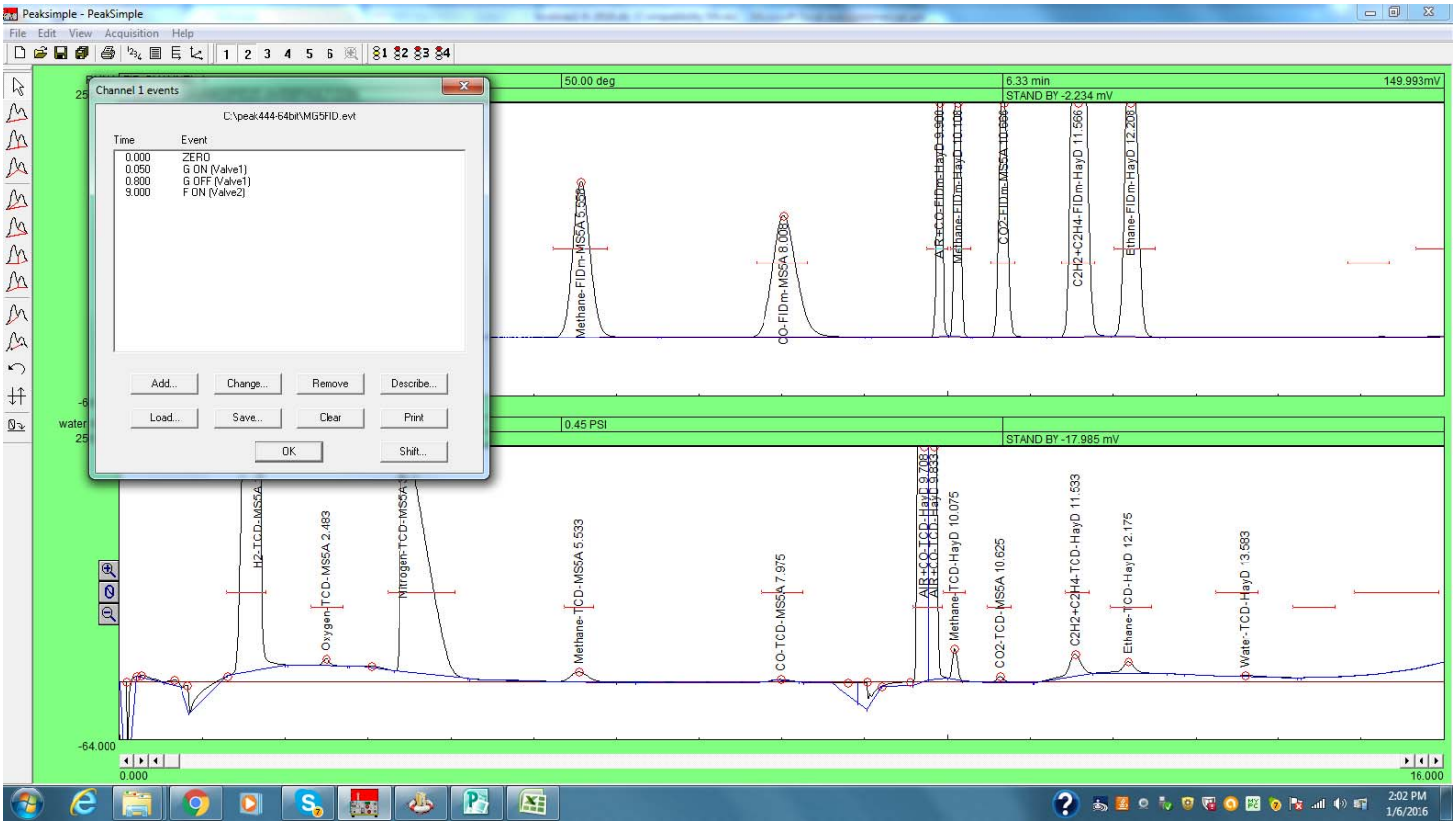
Multiple Gas#5 GC configuration
Jan 2016



The exact time for Relay G off (backflush pre-column) will be different using Argon vs Helium. By trial and error move the Relay G off time earlier and earlier until you see the methane peak disappear.
Compare this analysis to the same analysis on page 13.
Both methane and CO are present in the sample at 1% and have similar area count in the page 13 analysis with Relay G off set to .8 minutes.

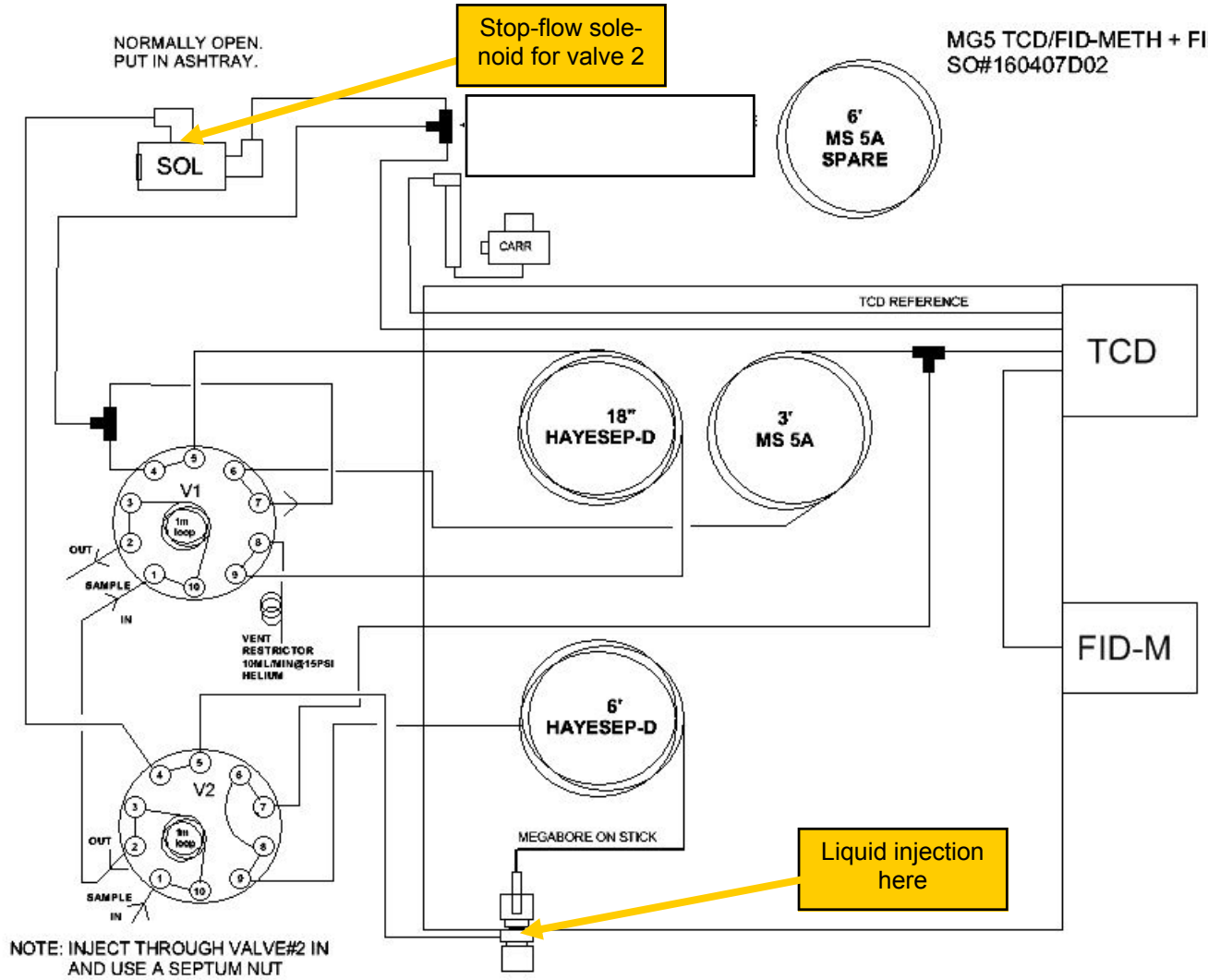
In the analysis above the Relay G off time is .7 minutes which was early enough to backflush the methane peak (which was still on the pre-column) , yet allow the CO peak to make it through onto the MS5A column.

Multiple Gas#5 GC configuration
Jan 2016



The chromatogram above shows the full analysis including the C2 peaks. Note that the Relay F on time (inject onto the Haysep dolumn) had to be delayed until 9 minutes to allow the CO to elute from the 6'MS5A column before making the injection onto the Haysep column.

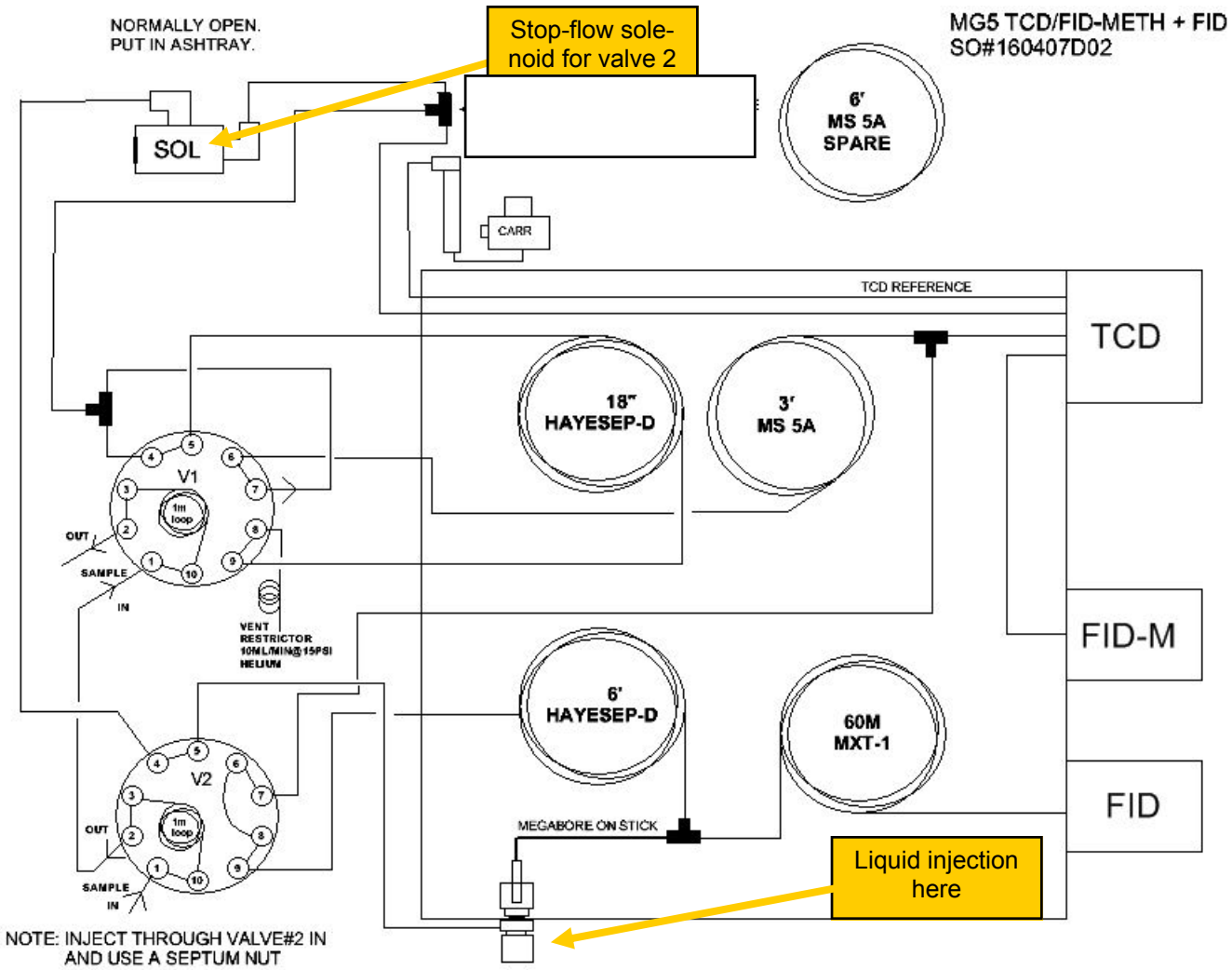
Multiple Gas#5 GC configuration
April 2016



Starting in March of 2016 all MG5 configurations were slightly modified:

1) The Haysep D column (and sometimes a capillary column) are connected to the injector port to make it easy to perform a liquid injection or small volume gas injection **without** using the gas sampling valves, and without having to reconnect columns inside the column oven. The low volume or liquid injection can only be made into the Haysep column, not the Molesieve.
2) A solenoid valve can interrupt the carrier gas to the Haysep valve and column. This allows both valves to inject at the same time if that is critical to the analysis (the normal MG5 valve sequence injects valve 1 immediately, and valve 2 some minutes later). When injecting both valves simultaneously, the solenoid is closed (Relay A ON usually) just after the valve injection to stop-flow the peaks in the Haysep D column until the MoleSieve peaks have eluted. An example of this is shown later in this document.

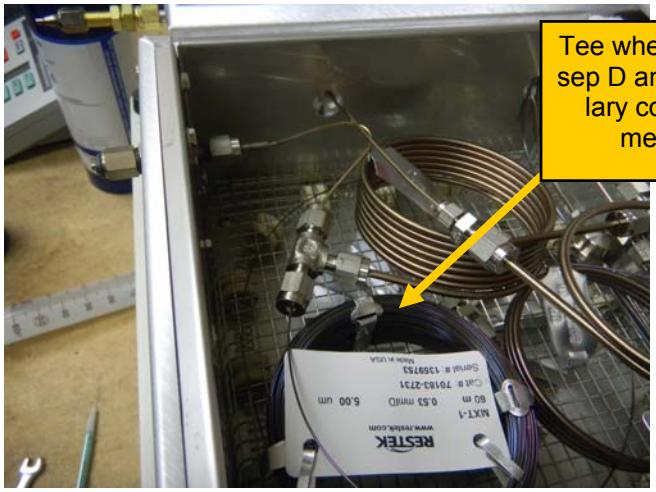
Multiple Gas#5 GC configuration
April 2016



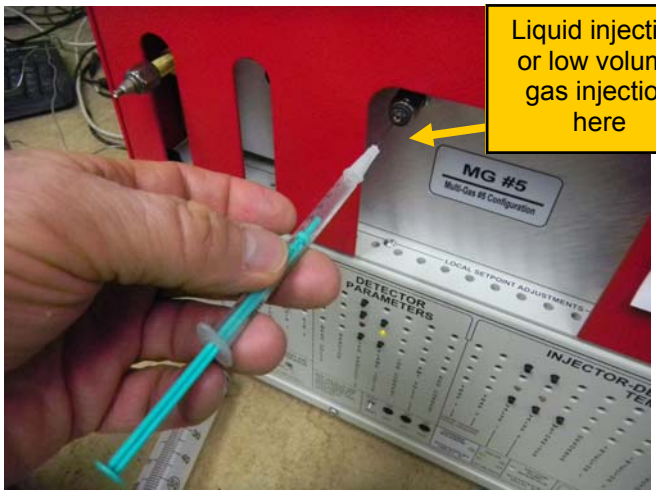
This drawing shows a capillary column and extra FID detector connected at a “tee” fitting so a liquid injection (or low volume gas injection) splits onto the capillary and the Haysep column.
In this configuration neither Valve 1 or Valve 2 is actuated at the beginning of the analysis. Valve 2 may optionally be rotated to the Inject position to backflush the Haysep column after the capillary peaks have eluted. See next page.

Multiple Gas#5 GC configuration
April 2016

When a capillary column is configured along with the Haysep column it can be connected at one of two places. Here it is shown connected to a "tee" fitting where one leg of the tee is the capillary column, the second leg is the Haysep column and the third leg is connected to the on-column injection port using a small adapter.

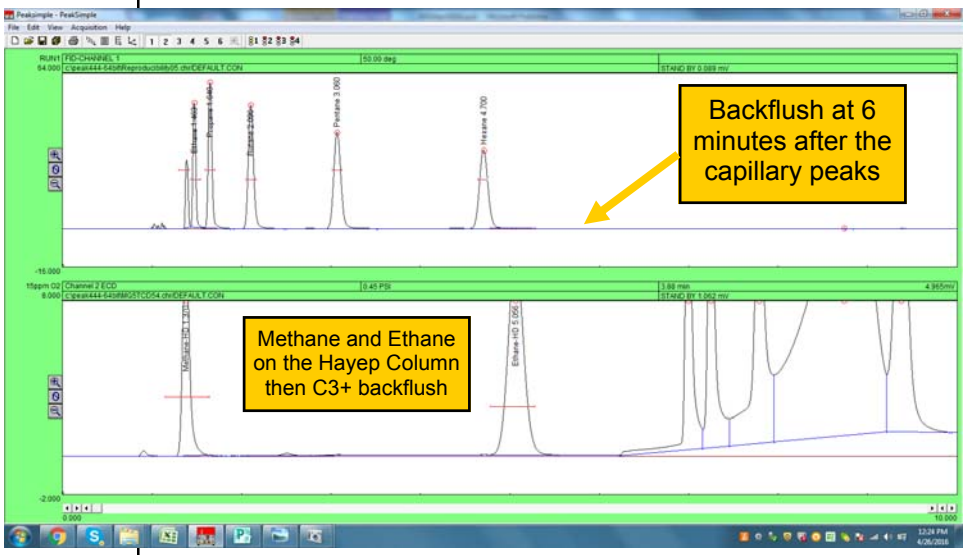


When making a low volume gas injection you do not need to use the gas sampling valves at all, unless you want to backflush the Haysep column after the capillary column peaks have eluted.

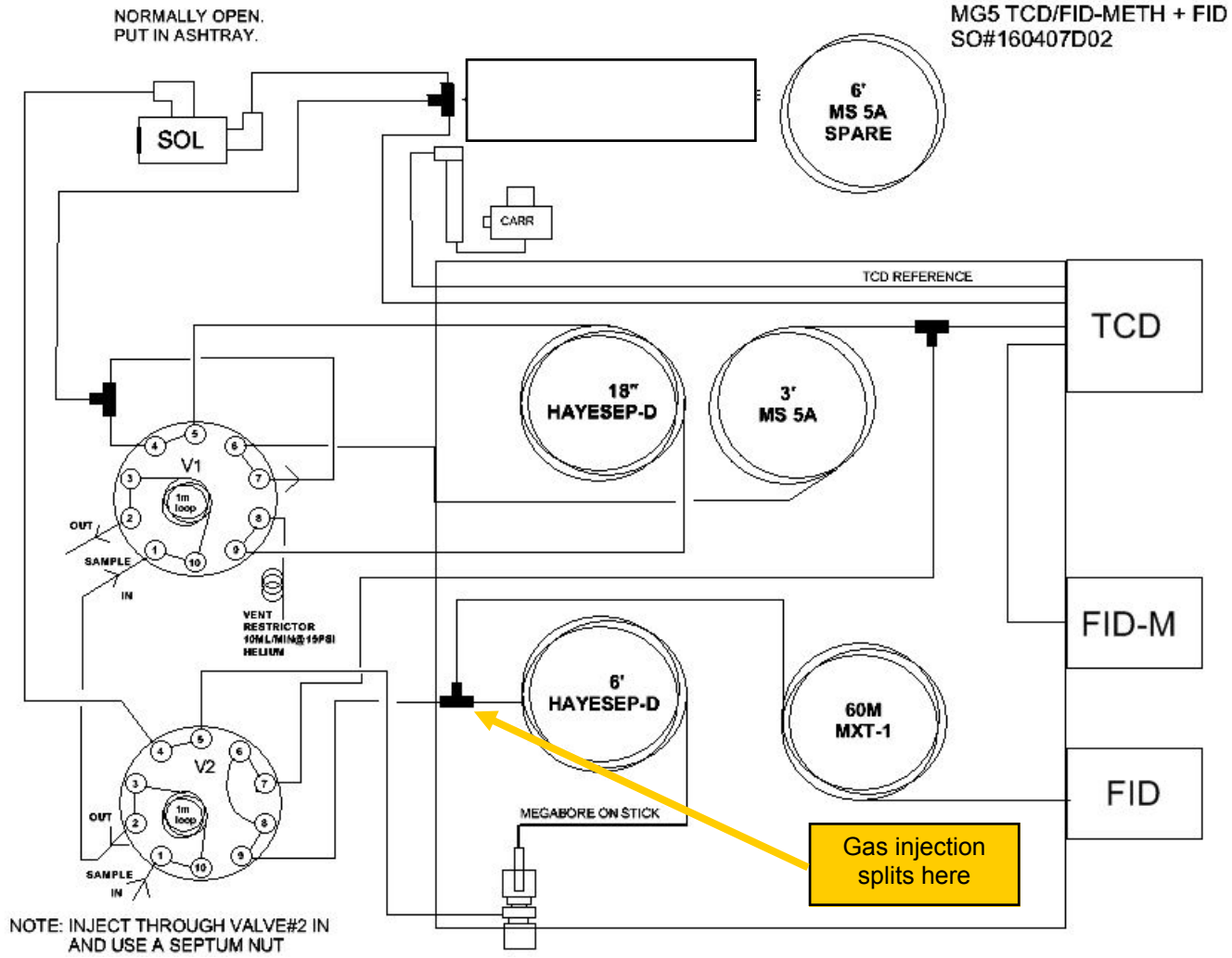


Here is a chromatogram of C1-C6 hydrocarbons injected via a gas tight syringe and backflushed at 6 minutes after all the peaks had eluted from the capillary column.

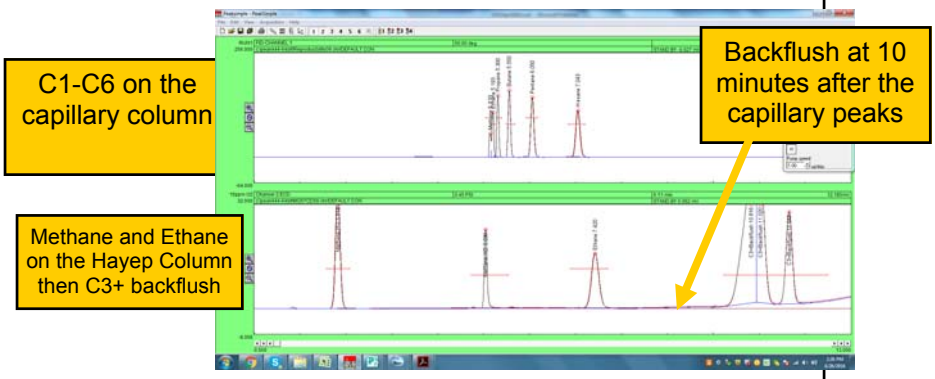
If you need to quantitate the backflush, the gas sampling valve loop must have a carrier gas purge to avoid injecting peaks which might be in the loop. See the diagram to understand.



Multiple Gas#5 GC configuration
April 2016



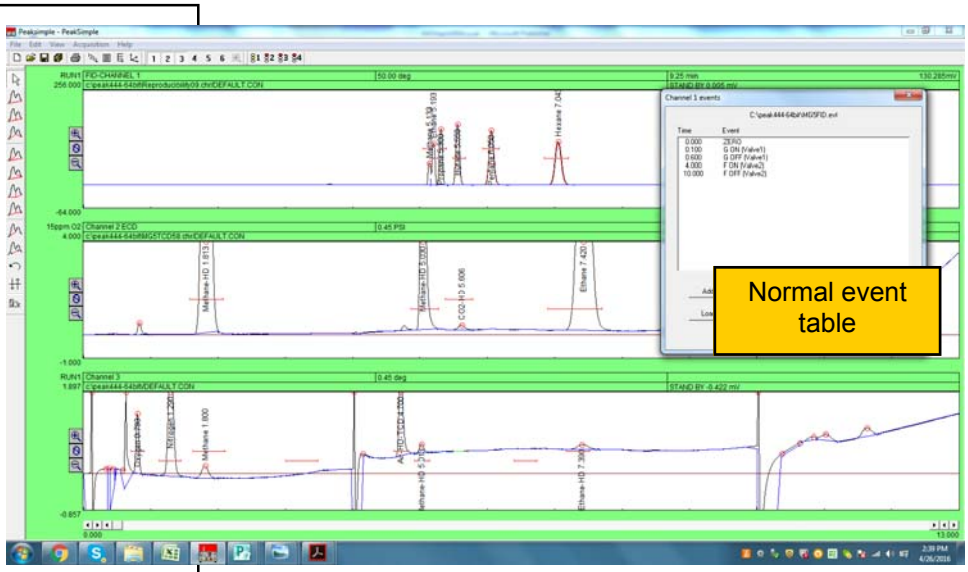
1) This drawing shows a capillary column set up for gas injection from the gas sampling valves onto both the Haysep and capillary column.



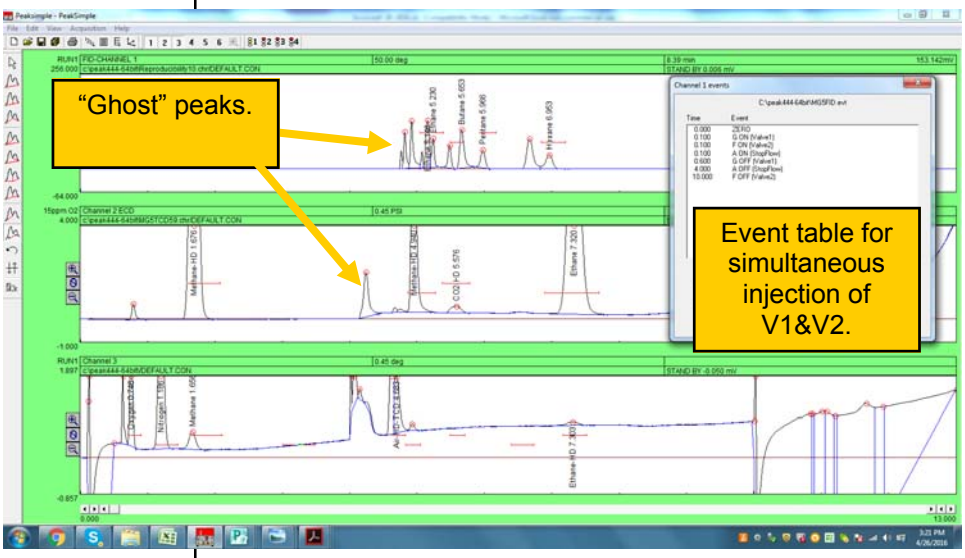
Multiple Gas#5 GC configuration
April 2016

There are some applications where the time delay between injecting the sample in Valve1 and Valve2 is important and un-desirable.

The top chromatogram shows the normal MG5 valve sequence/event table. The sample was 1000ppm C1-C6 aliphatic hydrocarbons. V1 injects at .1minutes. V2 injects at 4 minutes.



This chromatogram shows the same sample but with Valve 1 and 2 injected simultaneously. Relay A is turned ON at the same time (.1minute) which stops the carrier gas flow in the Haysep and capillary columns. This creates some extra “ghost” peaks but does not substantially affect the analysis. The stop-flow solenoid is turned OFF (re-establishing the flow) at 4minutes.



CTR1 Replacement Column for Fixed Gas analysis

May 2013

Separation of Oxygen, Nitrogen, Methane, CO and CO2 has been difficult since there is no single column material which is good for all these molecules at room temperature or above. In the past, one column vendor sold a “column in a column” called a CTR1 which is shown at right. This is a large bulky column consisting of an outer 1/4 od column and an inner 1/8 od column.

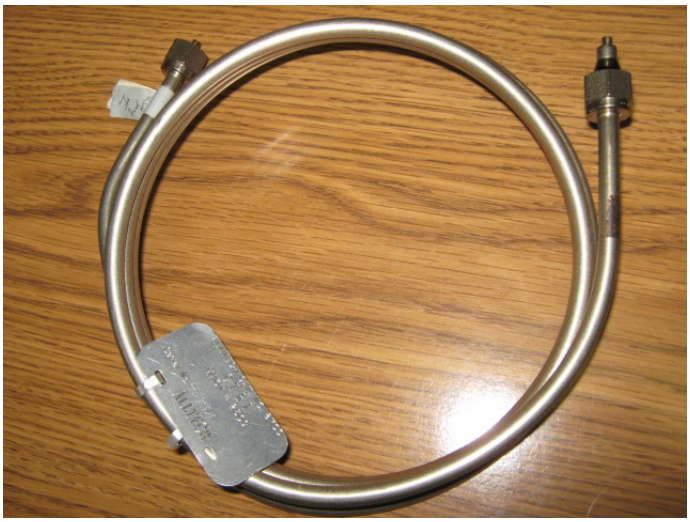
This column is no longer manufactured, or is not easily available, so SRI offers an equivalent or better column for the convenience of our customers.

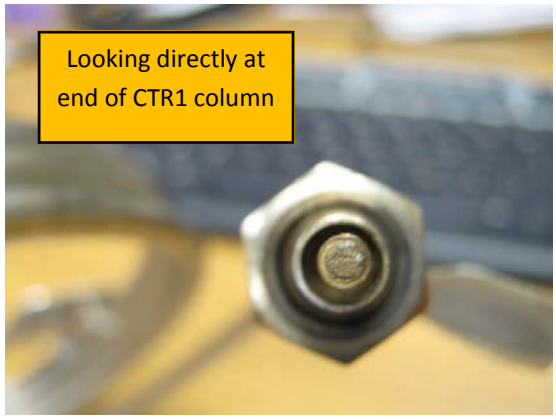
8600– PKC7 “Fixed Gas Column”

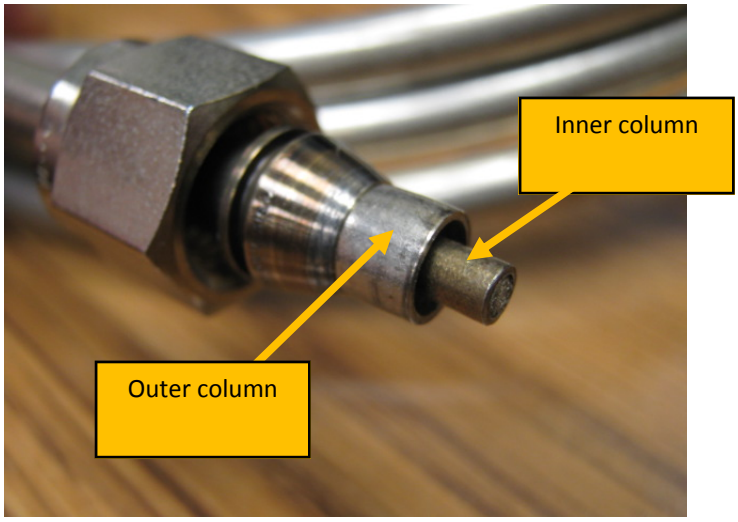
US$ 659.00 August 2013 (price may change)

For a more robust method of separating these molecules as well as others like propane, propylene, butanes, pentane etc. Please see the MultipleGas#3 document on www.srigc.com

http://www.srigc.com/MG3+SulfurFeb2013.pdf







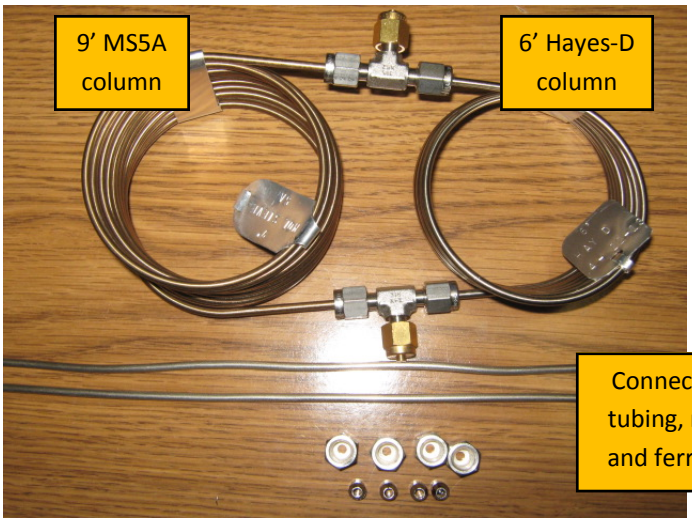
CTR1 Replacement Column for Fixed Gas analysis

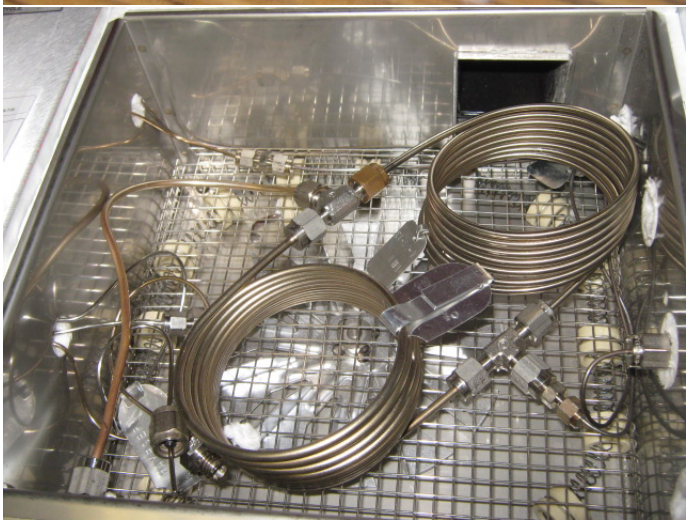
May 2013

The SRI " Fixed Gas Column" (FG) consists of two side by side columns rather than one column inside another. This arrangement is superior because the columns have different bake out temperatures and having them separable makes it easier to bake out the Molecular Sieve column (300C+)without damaging the lower temperature Hayesep-D (270C max) column.

The column is supplied with two 12" lengths of flexible 1/8"od nickel tubing to make connecting it easier and extra nuts and ferrules. We like the soft graphite ferrules for this application because they seal well and do not deteriorate at the 300C bake-out temperature. However metal ferrules can also be used.

The " Fixed Gas Column" is shown installed in an SRI 8610C GC column oven. There is still room for other columns.

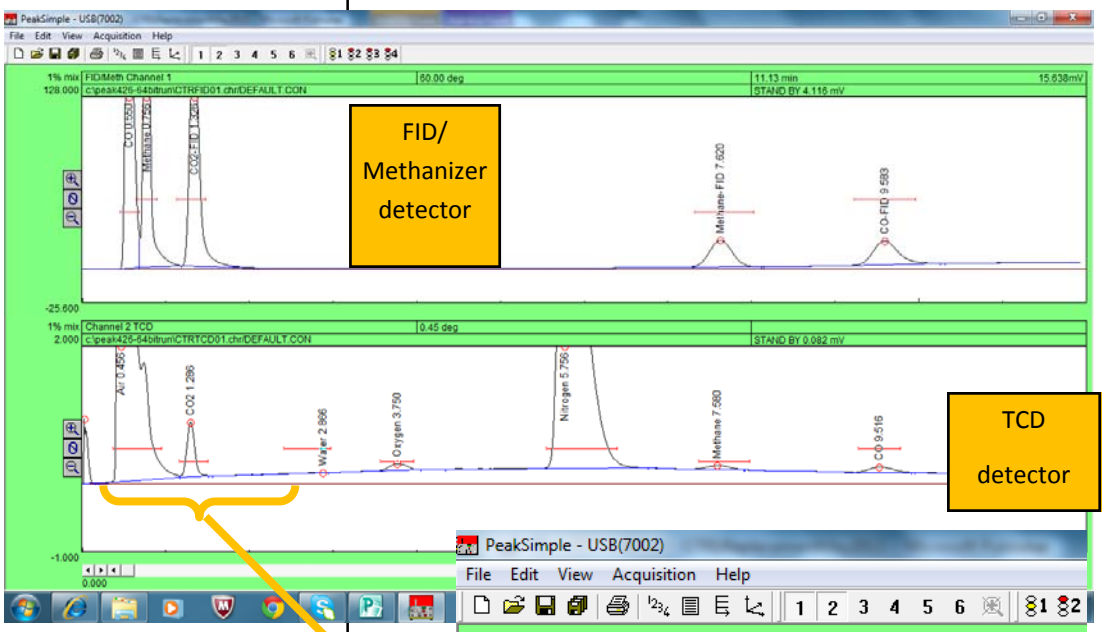


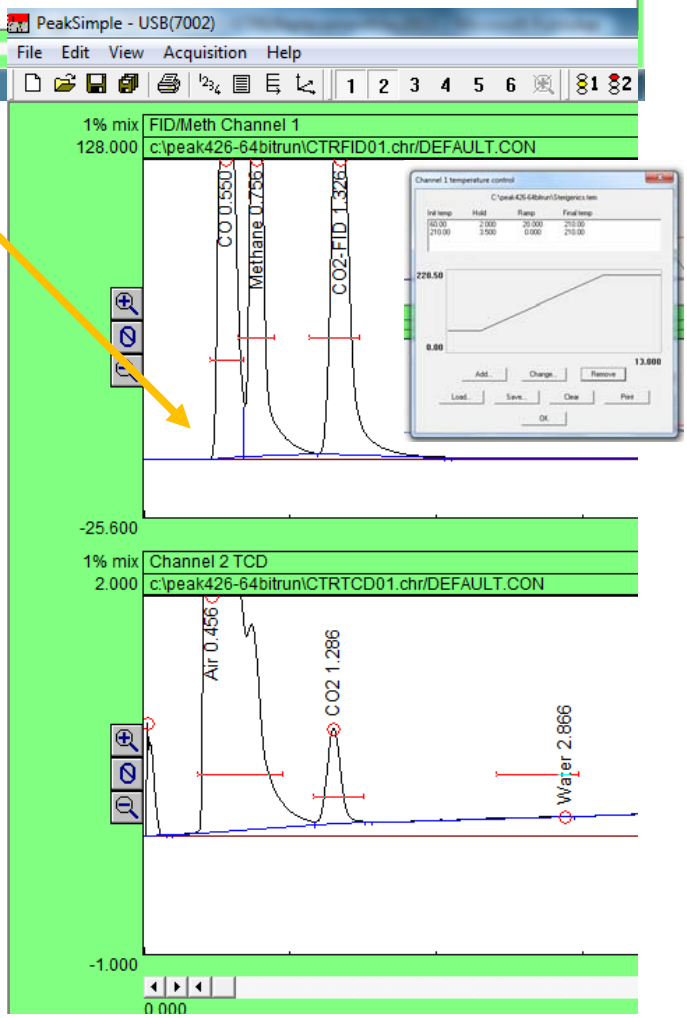


CTR1 Replacement Column for Fixed Gas analysis

May 2013

The FG column was installed in an SRI 8610C GC equipped with a TCD detector and FID/Methanizer (FIDM) detector. The two detectors were plumbed in series so some peaks are detected by both detectors. The methanizer part of the FID detector converts CO and CO2 to methane so the FID/Methanizer can detect those molecules with the same sensitivity as methane and other hydrocarbons. The TCD responds to all molecules including water.





All the peaks in the first few minutes of the chromatogram are separated by the 6' Hayesep-D column. As can be seen, the CO and Methane co-elute with the oxygen/ nitrogen, so these peaks are not detectable by the TCD, but are clearly seen on the FIDM.

In this case the helium carier gas was set to 20 PSI and the temperature program was set to start @60C hold 2minutes then ramp at 20degrees/minute to 210.

CTR1 Replacement Column for Fixed Gas analysis

May 2013

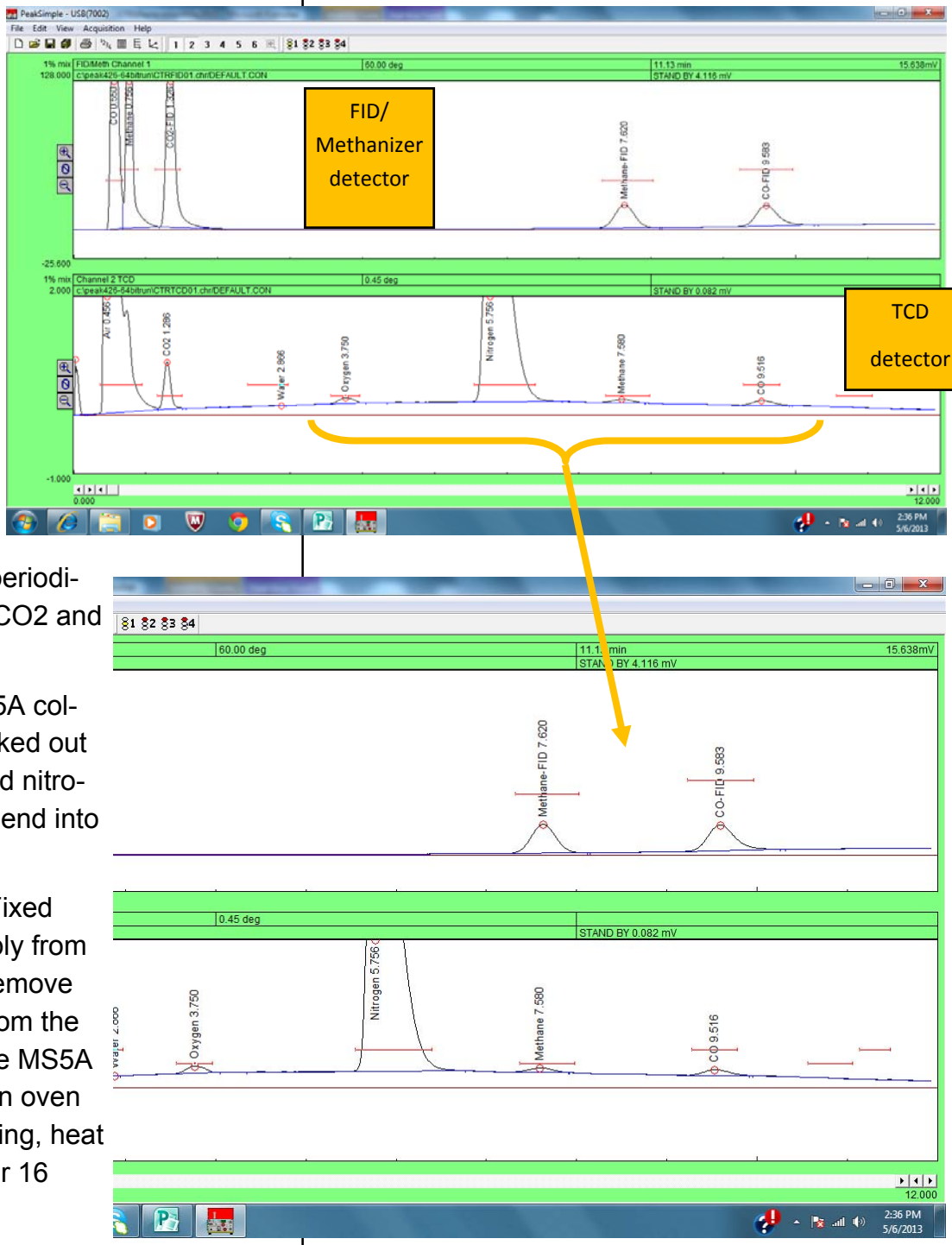
The peaks in the last minutes of the chromatogram are from the 9' Mole-Sieve 5A column.

This column separates oxygen from Nitrogen as well as methane and CO.

CO2 however and water are permanently absorbed by this column which must be baked out periodically to remove the CO2 and water.

You can tell the MS5A column needs to be baked out when the oxygen and nitrogen peaks start to blend into each other.

Remove the entire Fixed Gas column assembly from the oven and then remove the MS5A column from the assembly. Install the MS5A column in the column oven and with carrier flowing, heat the MS5A column for 16 hours at 300C.



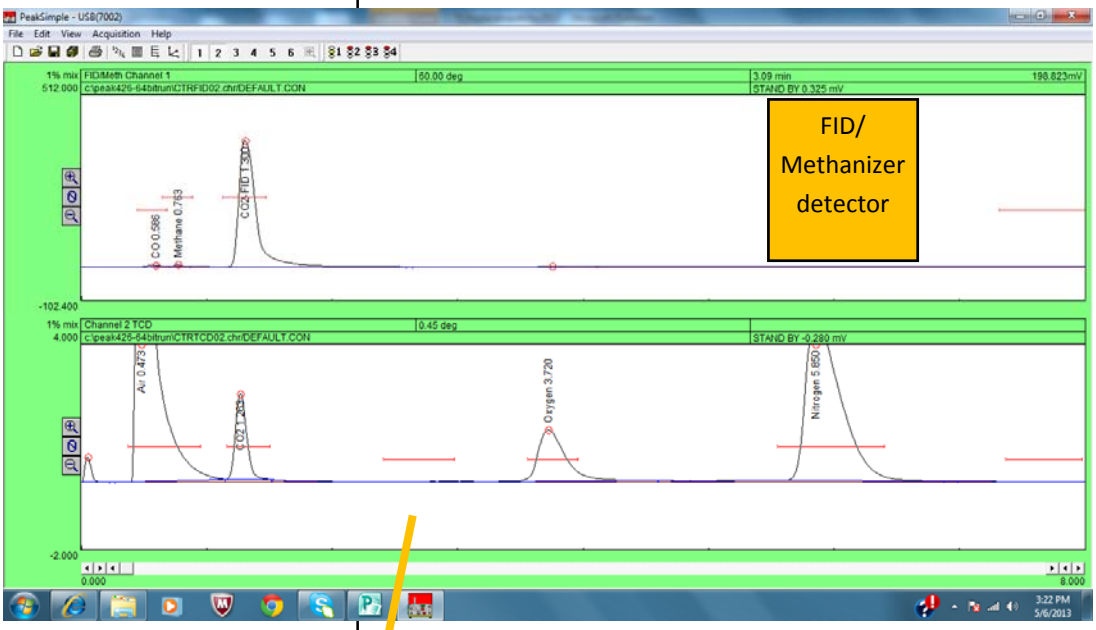
CTR1 Replacement Column for Fixed Gas analysis

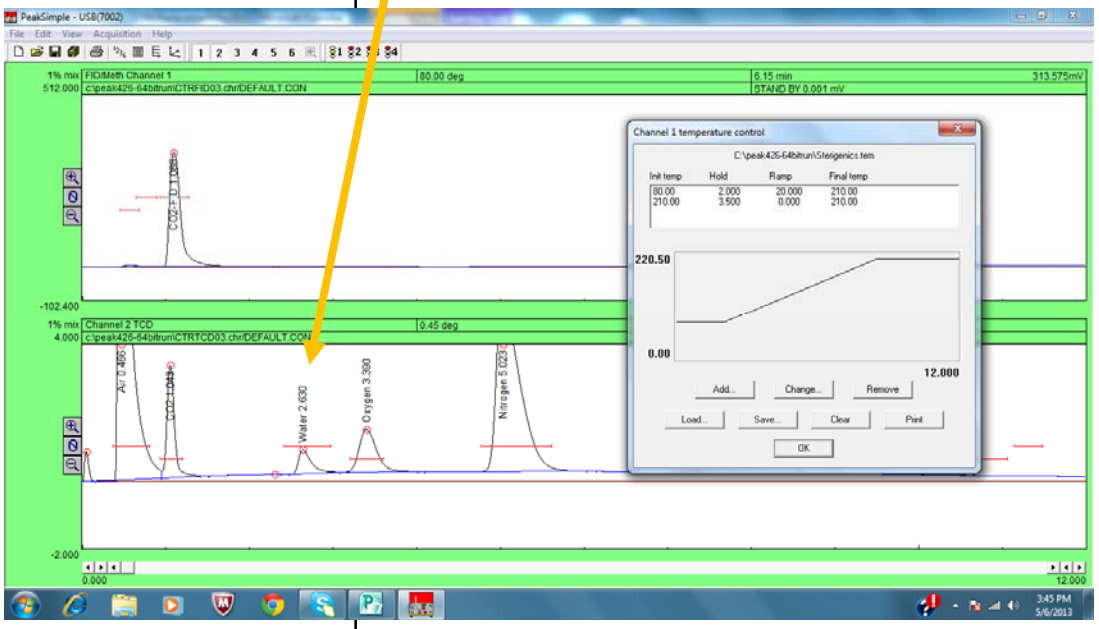
May 2013

Shown at right is a chromatogram of 1ml exhaled breath using the 60C starting temperature.

Compare this chromatogram to the one below at a 80C starting temperature. The water peak in the top chromatogram co-elutes with the oxygen while at the 80C Starting temperature, the lower chromatogram shows the water nicely separated from the oxygen.

There is less separation however between the CO2 and the air peak.

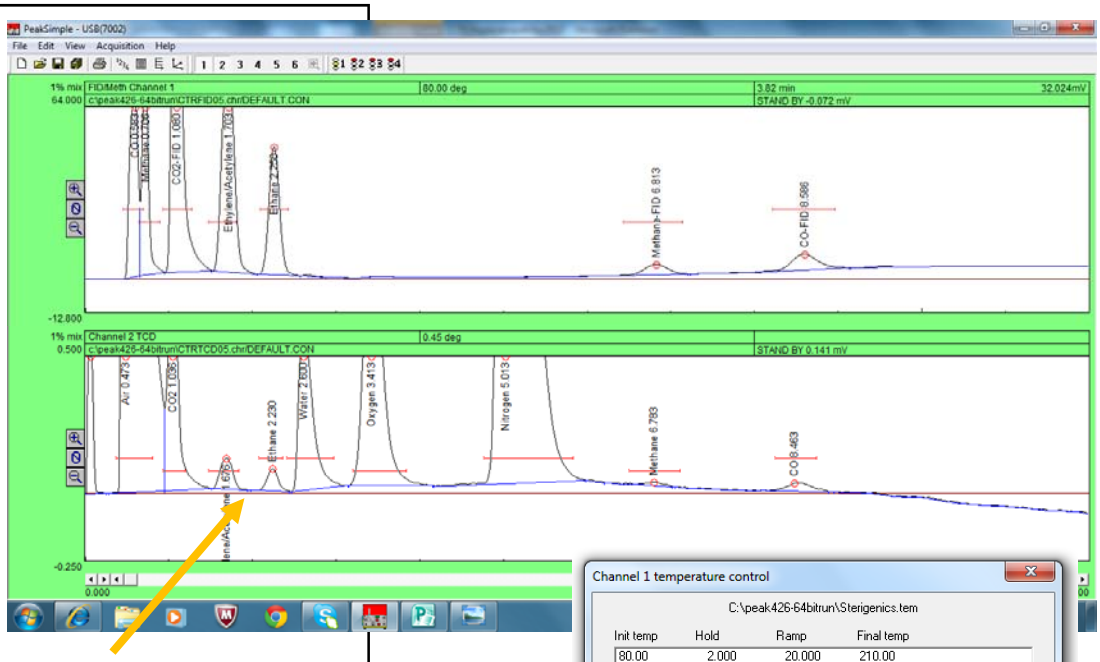




CTR1 Replacement Column for Fixed Gas analysis

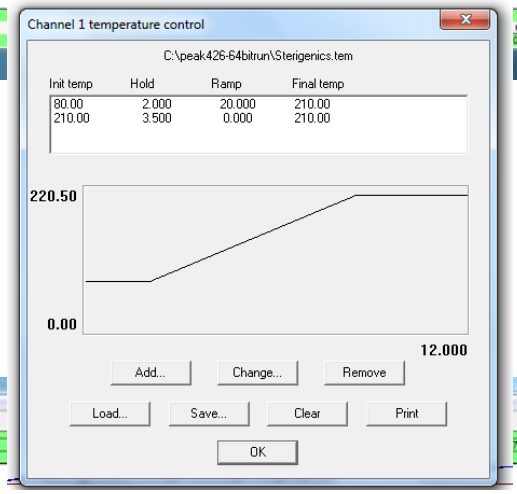
May 2013

Shown at right is a chromatogram of 1ml 1% gas mix including ethylene/ acetylene and ethane using the 80C starting temperature oven program.

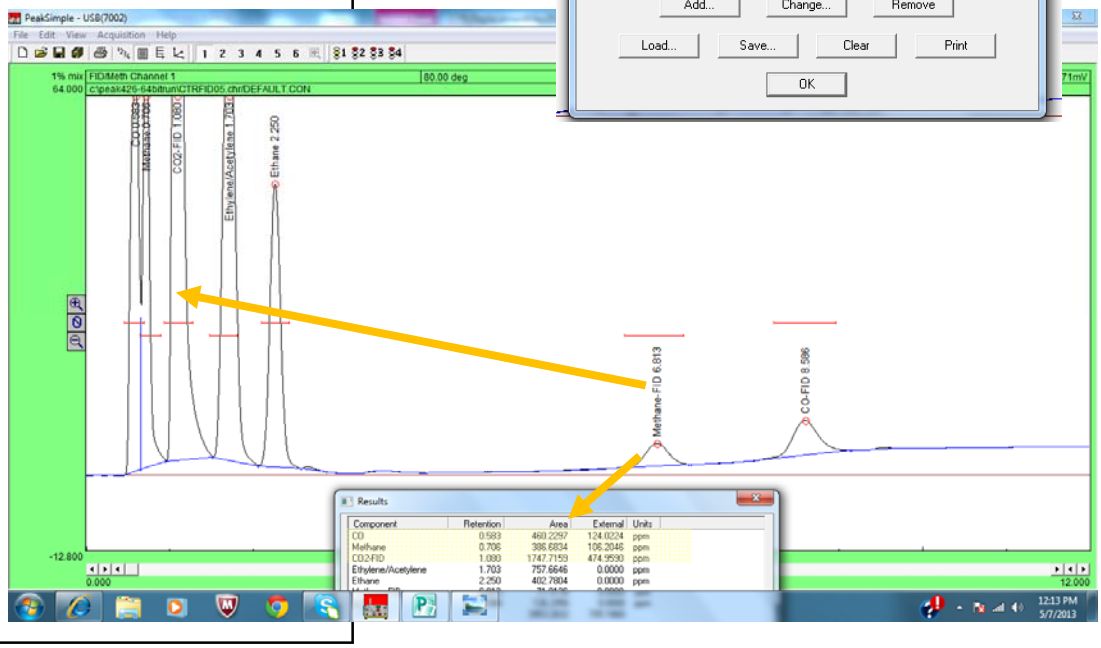


Between the TCD and FID/ Methanizer CO, CH4, CO, Ethane, Ethyelen/Acetylene, Water, Oxygen and Nitrogen are all resolved.

Ethylene and acetylene co-elute, but are separated from ethane and also water.



Note also that the split ratio between the columns is about 4:1 judging by the area counts of CO2 (which elutes from the 6'Hayesep-D column) and the methane and CO (which elute from the 9'MS5A column).

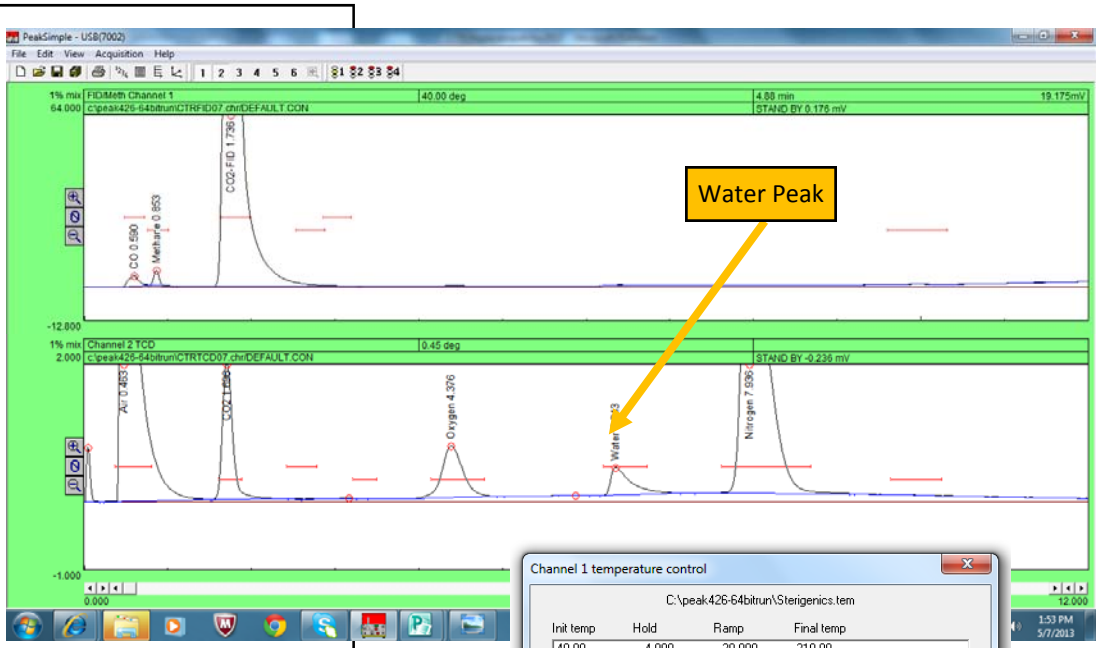


CTR1 Replacement Column for Fixed Gas analysis

May 2013

Shown at right is a chromatogram of exhaled breath starting at 40C.

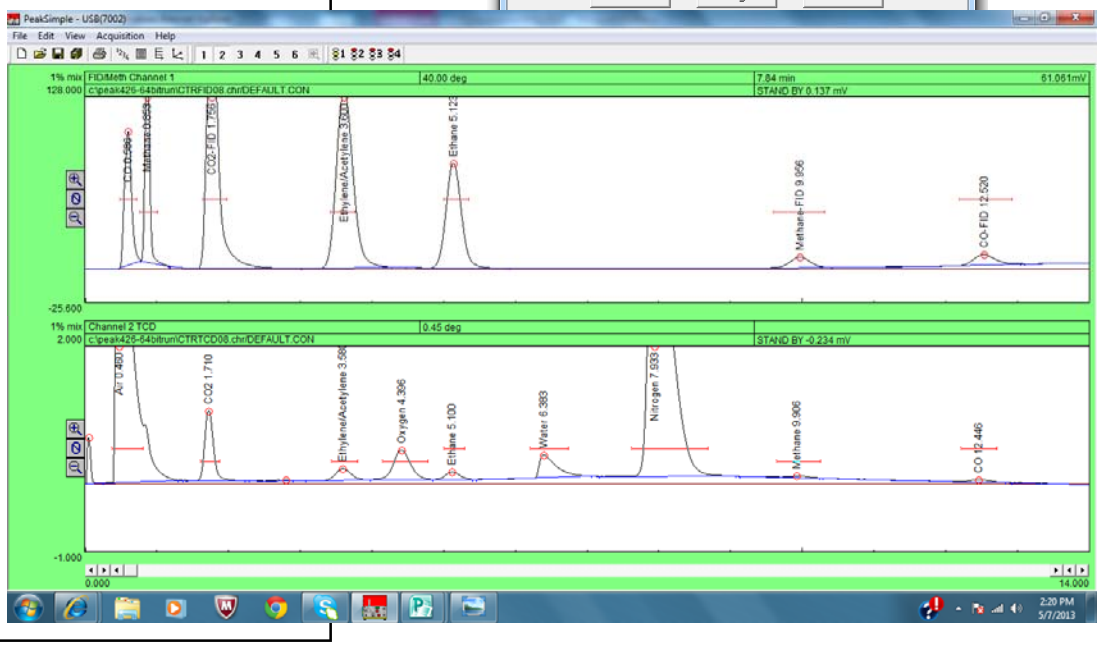
You can see the water peak has shifted to the right and elutes between oxygen and nitrogen.



A mix of:

O2, N2, CO, CO2, methane, ethane and ethylene/acetylene plus water is shown starting at 40C. You can see the peaks from the Hayesep-D column are interspersed with the peaks from the MS5A column.

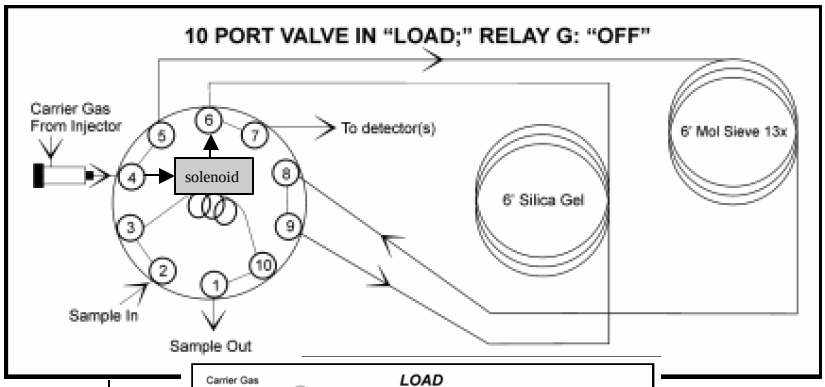
You can experiment with different temperature programs to best suit your particular mix of gases.



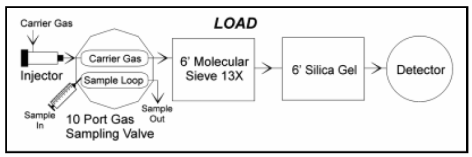
SRI 8610C Gas Chromatograph
Multiple Gas #3 GC configuration

The SRI 8610C Gas Chromatograph Multiple Gas #3 GC configuration is a versatile low cost way of analyzing many different kinds of gas samples. The GC pictured at right has two Multiple Gas #3 (MG#3) configurations implemented in a single GC chassis so there are two gas sampling valves and four columns as well as four detectors. This is why the column oven looks so crowded.

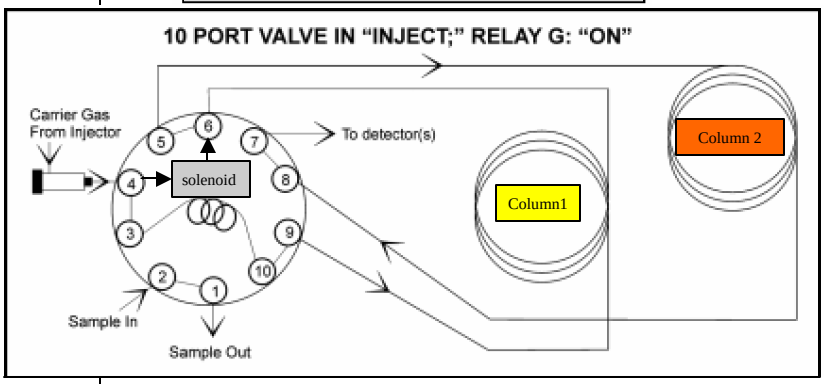
The MG#3 GC configuration is almost identical to the MG#1 GC configuration except there is an additional solenoid valve which when activated by the PeakSimple data system stops the flow of carrier gas in column 1.



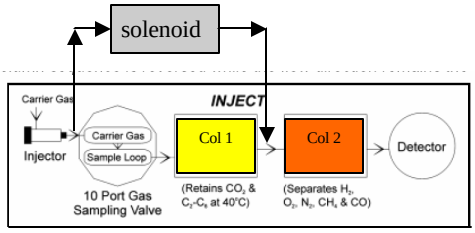
When the solenoid valve is actuated (typically while the gas sampling valve is in the INJECT position), column 1 has the same pressure applied to both its inlet and outlet. This stops the flow of carrier gas in column 1. The peaks which were in column 1 simply stop moving without broadening or distortion.



The flow of carrier gas in column 2 actually increases while the solenoid is actuated since the full carrier gas head pressure is now applied across a shorter restriction (one column instead of two in series).



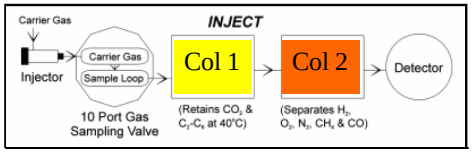
The MG#3 GC configuration is slightly more flexible than the MG#1 because the stop flow capability allows a wider selection of columns to be used, where the MG#1 only works with silica gel as Column 1 and MoleSieve 13X as Column 2.



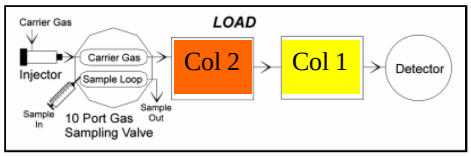
SRI 8610C Gas Chromatograph
Multiple Gas #3 GC configuration

The chromatograms shown on this page are a mix of natural gas and sulfur compounds. The top chromatogram shows the sulfur selective FPD response.
The middle chromatogram shows the FID response.
The two lower chromatograms show the FPD response (black) overlaid with the FID response (red).

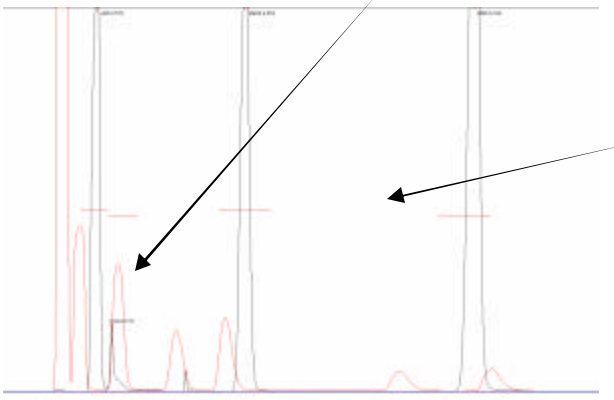
The PeakSimple event table shown at right rotates the valve from Load to Inject at .1 minutes and then back to Load at 1.00 minutes. Because even the first peak (methane) has not migrated from Column 1 though to Column 2 at this time, the equivalent effect is that the

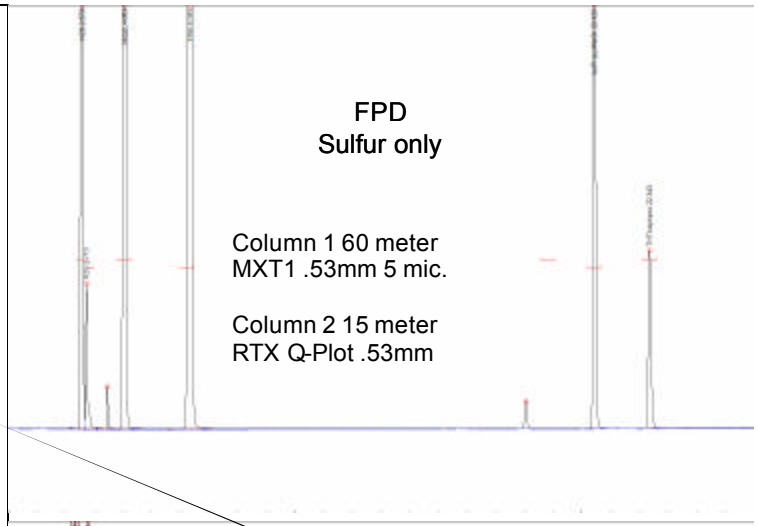


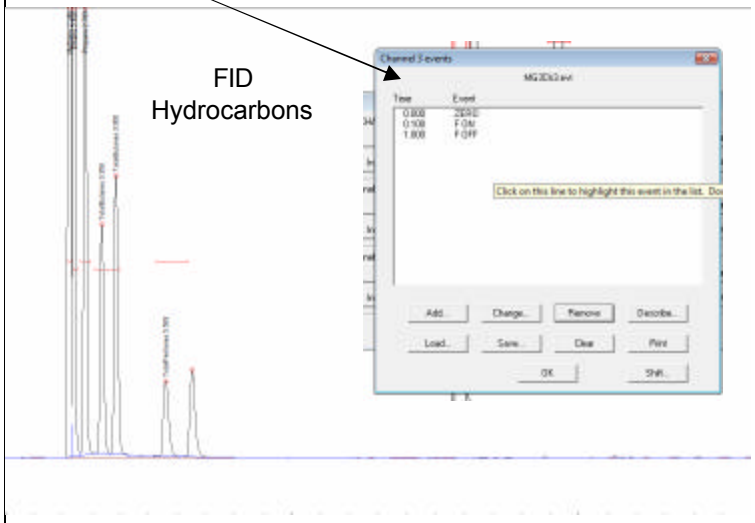
peaks are injected into and are separated by

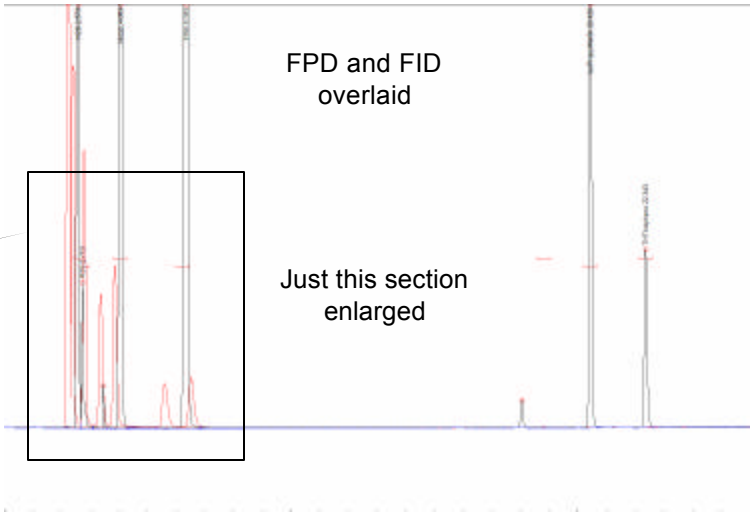


Column 1 only, as if Column 2 was not even connected. You can see by the overlaid chromatograms that COS co-elutes with Propane quenching its FPD response.



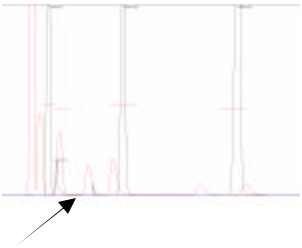


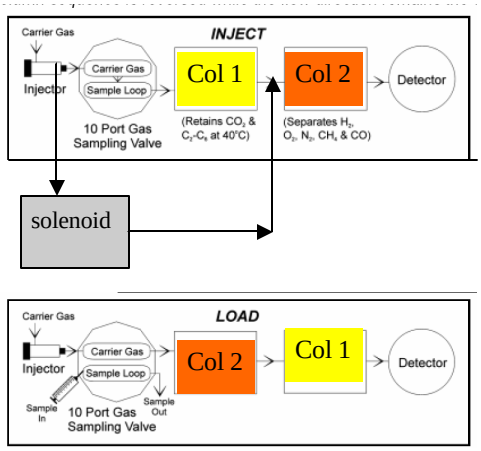




SRI 8610C Gas Chromatograph
Multiple Gas #3 GC configuration

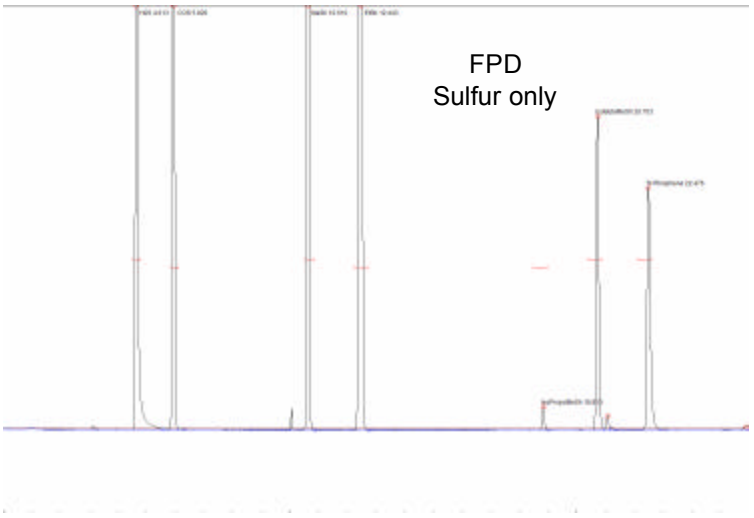
Instead, the MG#3 allows the Stop Flow solenoid to actuate at 3.5 minutes just after the Propane and COS migrate into Column 2 (15meter RTX QPlot .53mm).

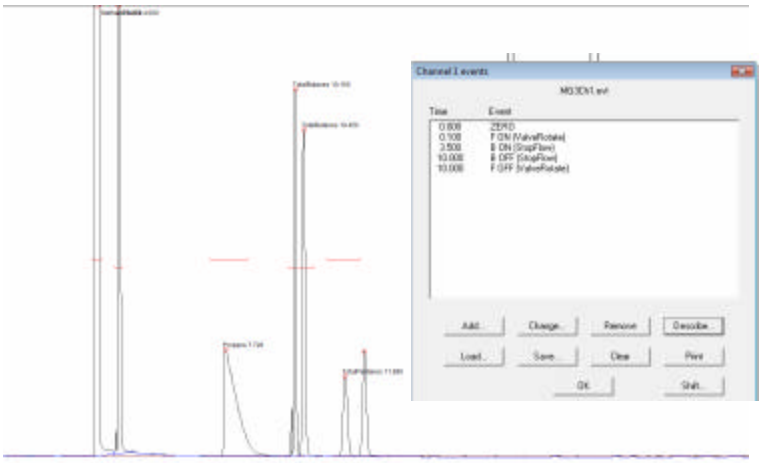




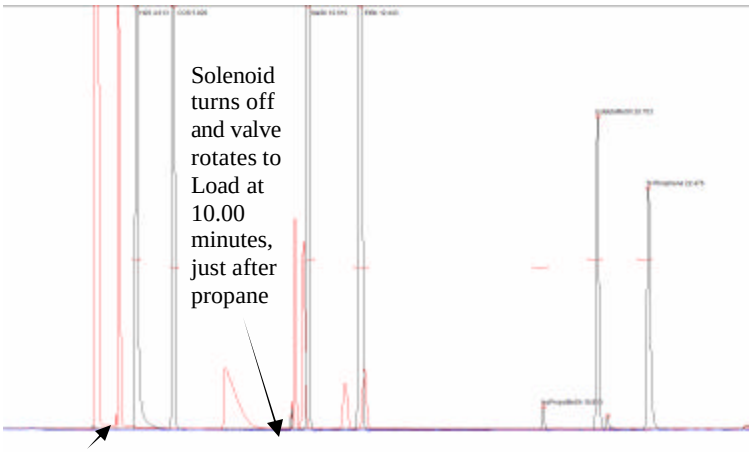
This traps the peaks after Propane in Column 1 while the peaks in Column 2 (Methane, Ethane, Propane, H2S and COS) separate and elute. Unlike column 1 which does not separate COS and Propane, the peaks are well separated on Column 2 so quenching does not occur.

Once Propane elutes from Column 2 (about 10 minutes) the valve rotates back to the Load position and the Stop Flow solenoid is de-energized. The peaks which were trapped on column 1 now elute to the detectors (Butanes, Pentanes, Mercaptans etc.)





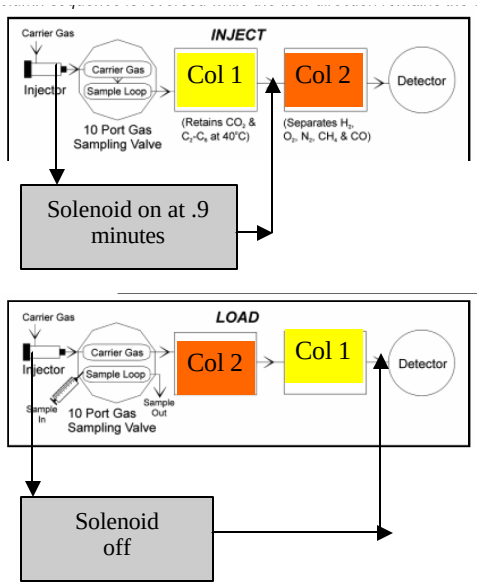
Oven temperature 40C for 10 minutes then 20C/min to 200C



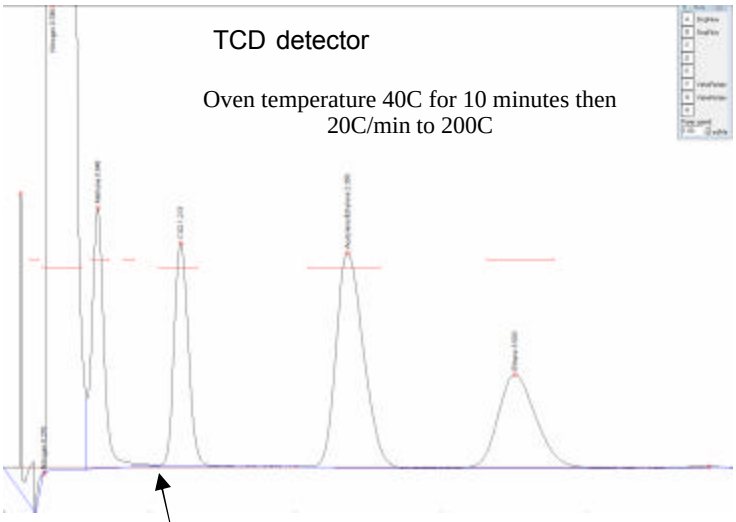
Solenoid turns on at 3.5 minutes, stopping flow in Col 1

SRI 8610C Gas Chromatograph
Multiple Gas #3 GC configuration

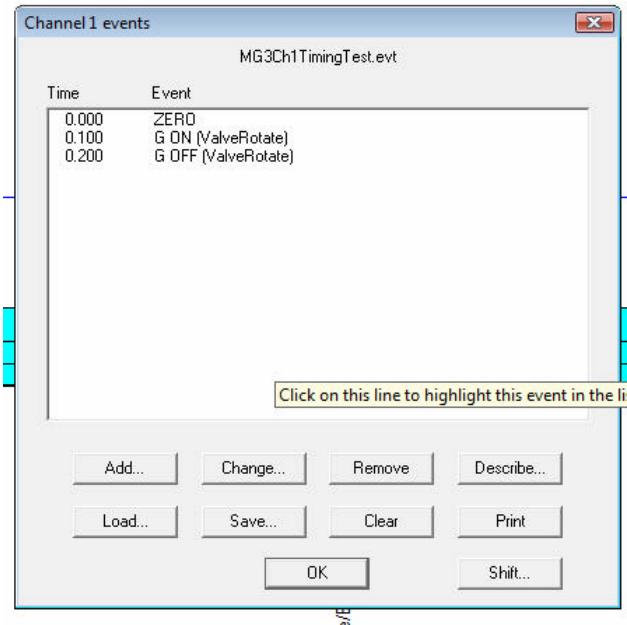
The MG#3 GC configuration is also useful with other column combinations. In this example, Column 1 is a 3' HaysepD and Column 2 is a 6' MS13X. The sample is first run on the 3' HaysepD using the event table shown at right. Because the valve is rotated back to the Load position almost immediately after injection



(.1 minutes) the separation occurs as if Column2 was not even connected. (no hardware changes are required to produce this effect). There is a convenient gap between Methane and CO2 where it would make sense to activate the stop-flow solenoid valve to immobilize the CO2 and heavier peaks in Column1 while the H2, O2, N2, Methane and CO peaks elute from Column1.

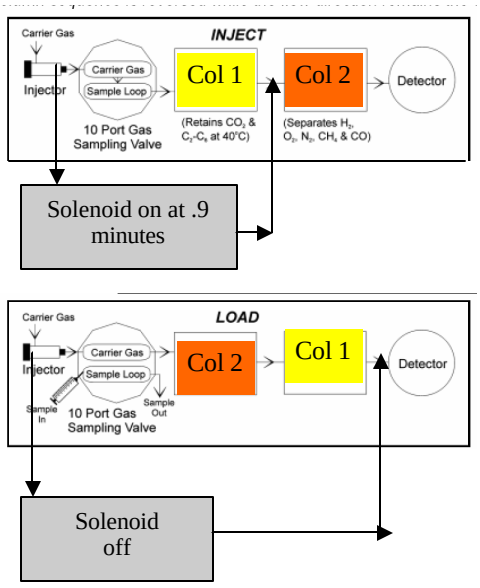


This would be a good time (.9 minutes) to activate the stop-flow solenoid. Just after the Methane migrates onto Column2 but before the CO2 and heavier peaks



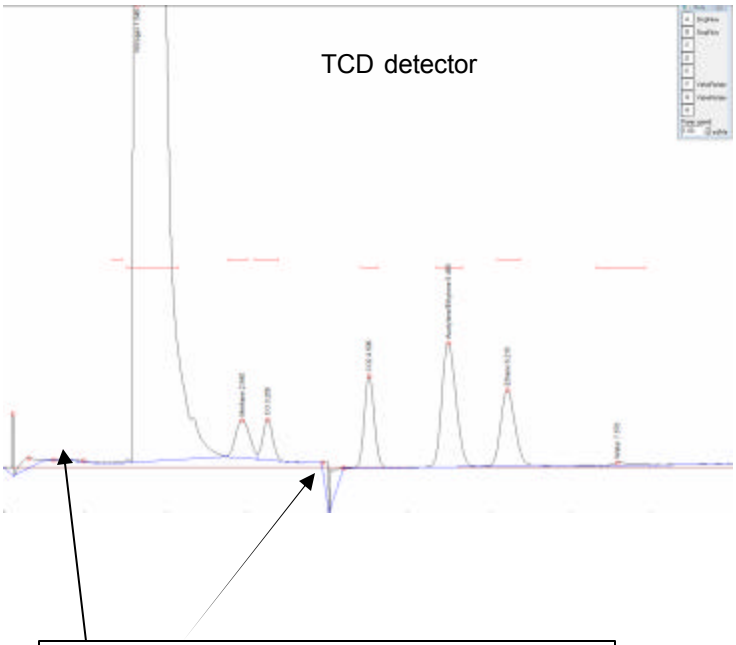
SRI 8610C Gas Chromatograph
Multiple Gas #3 GC configuration

The same sample is injected again using the Event table shown at right. The valve stays in the Load position until 4.00 minutes. The Stop-Flow solenoid is actuated at .9 minutes (determined from the chromatogram on the previous page) and de-activated at 4.00 mintes. This results in H2, O2, N2, CH4 and

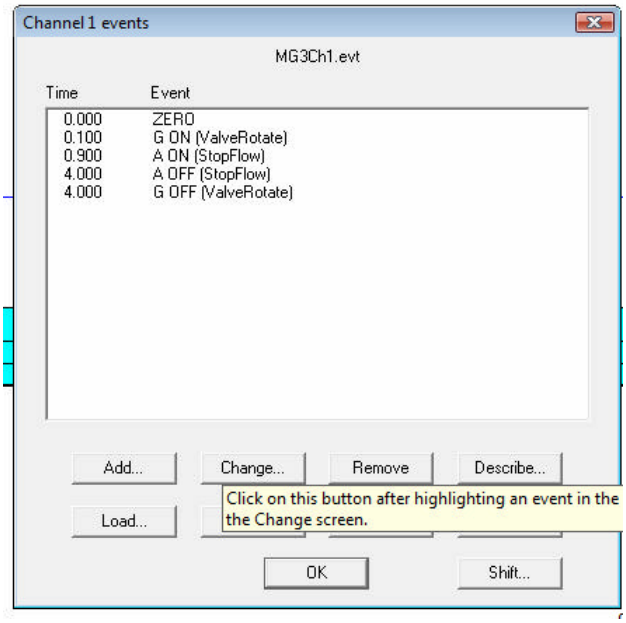


CO migrating onto Column2 (Mole-Sieve13X) where they separate and elute into the TCD detector. Once CO elutes (about 4.00 minutes), the valve is rotated back to the Load position and the Stop-Flow solenoid is de-energized.

The concept of immobilizing peaks by stopping the flow is applicable to many situations and many column combinations, not just the two examples presented here.

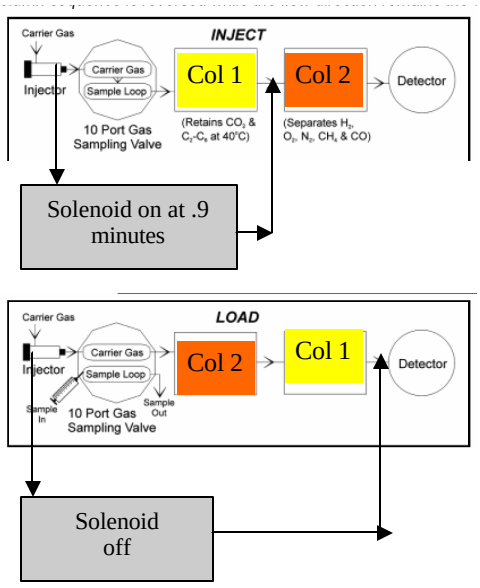


Stop-Flow solenoid on at .9 and off at 4.00 minutes.
Valve back to Load position at 4.00 minutes

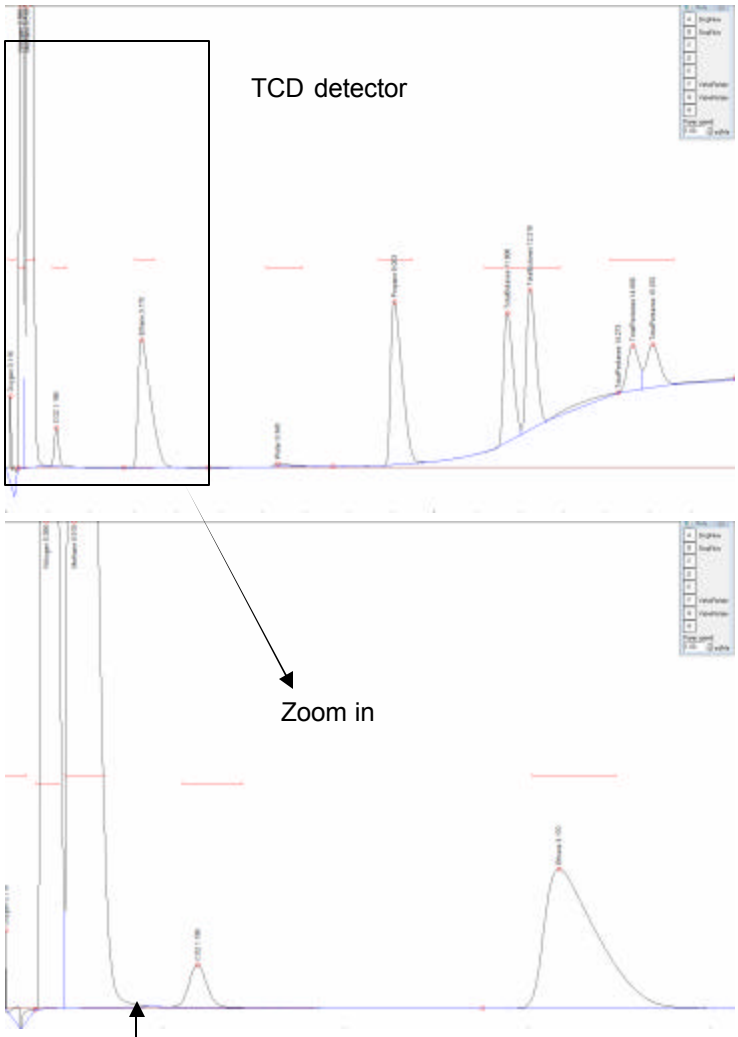


SRI 8610C Gas Chromatograph
Multiple Gas #3 GC configuration

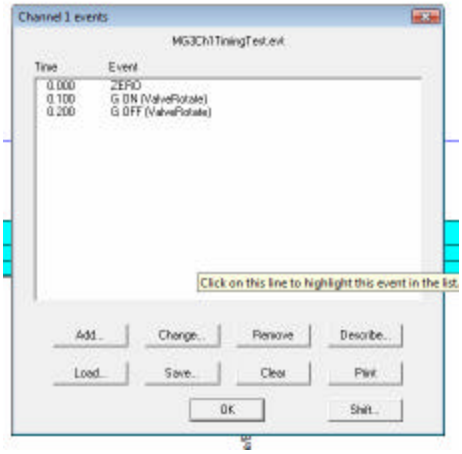
Another example is Natural Gas. Set the Event table up to inject and then immediately rotate the valve back to Load after .1 minutes in the Inject position. This has the effect of performing the analysis as if Column2 was not in the system.



Column 1 is a 3' Haysep D and Column 2 is a 6' MS13X. The Haysep D does not separate Oxygen and Nitrogen or CO. Set the Stop-Flow solenoid time by finding the gap between Methane and CO2, in this case about .9 minutes.

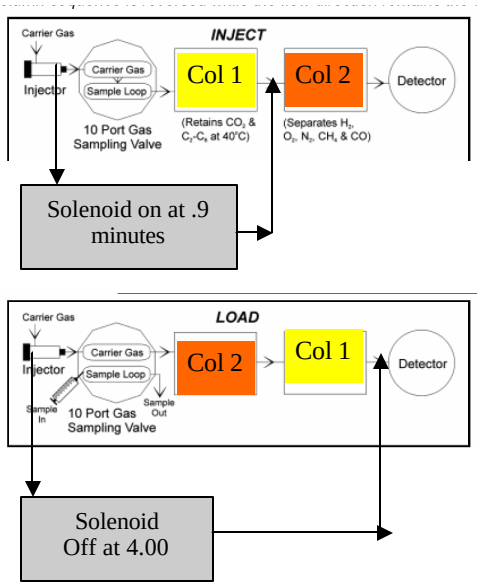


The gap between Methane and CO2 is a good time to actuate the Stop-Flow solenoid

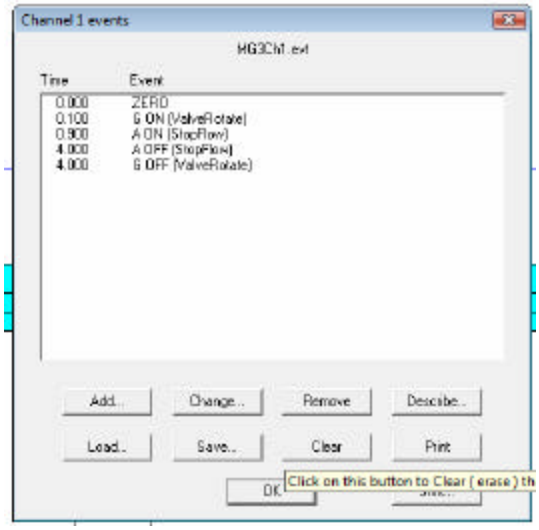


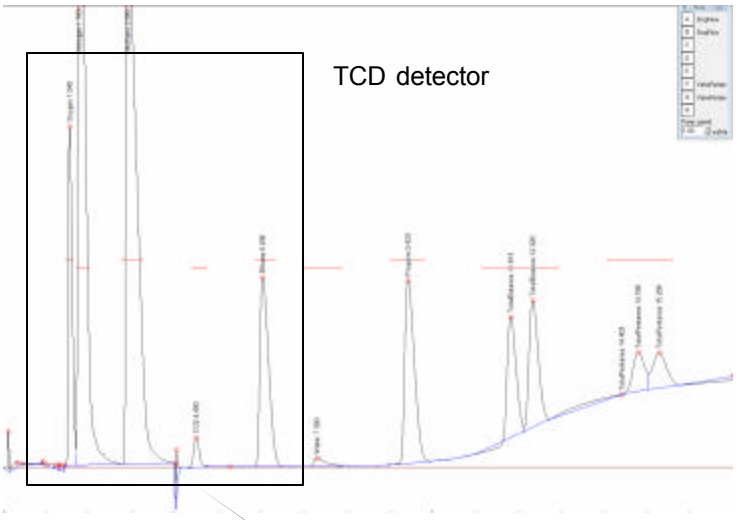
SRI 8610C Gas Chromatograph
Multiple Gas #3 GC configuration

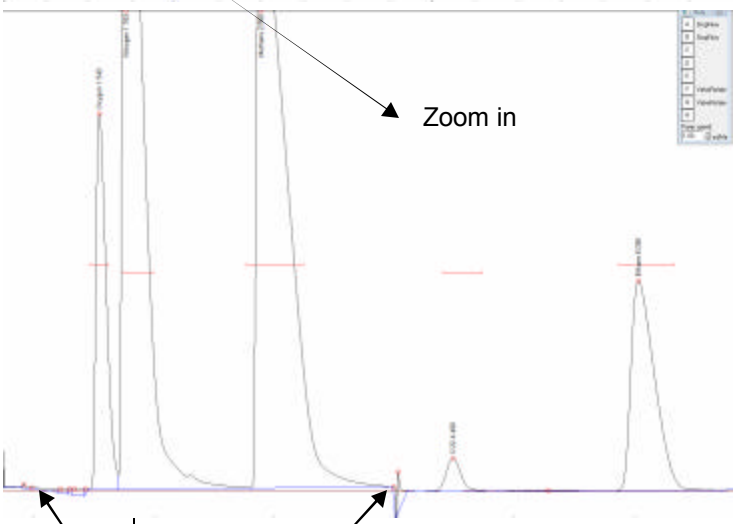
With the Event table modified, the Oxygen, Nitrogen and Methane separate on the MS13X. Then the Stop-Flow solenoid is de-energized and valve rotated back to Load position (both at 4.00 minutes) and the remaining peaks (Ethane, Propane, Water, Butanes, and Pentanes) which were immobilized on the



Haysep D (column 2) elute normally.



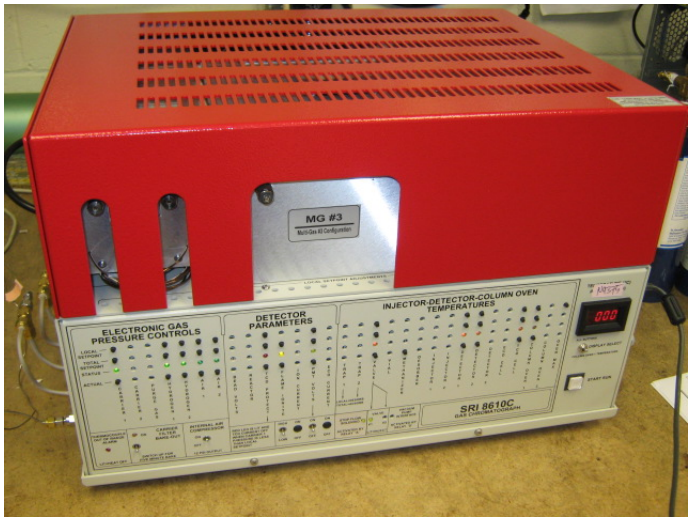




The Stop-Flow solenoid is actuated at .9 minutes and de-energized at 4.00 minutes. The valve is rotated back to Load also at 4.00 minutes

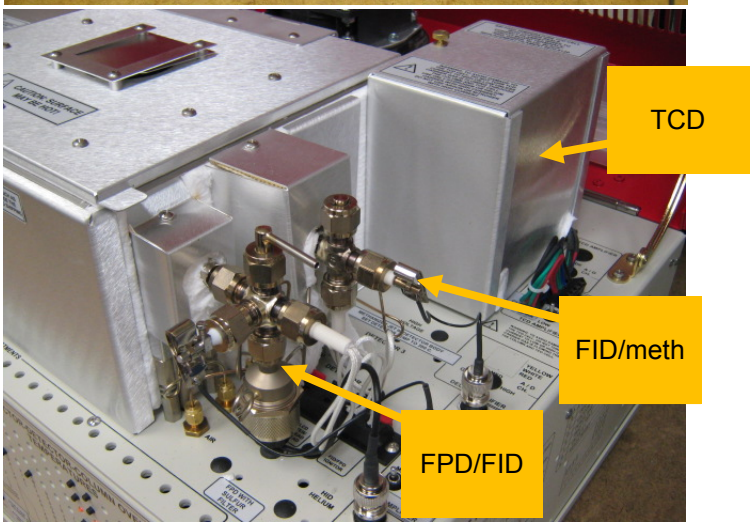
Muliple Gas#3 plus Sulfur
GC Configuration

The SRI Model 8610C Gas Chromatograph (GC) configured as a MultipleGas#3 plus Sulfur is designed to measure H2, O2, N2, CO, CO2, H2O, C1 through C5 hydrocarbons and also H2S, COS/SO2, and other sulfur molecules such as mercaptans, CS2, DMS, DMDS, Thiophenes and more in a single analysis.



The GC is equipped with three detectors:

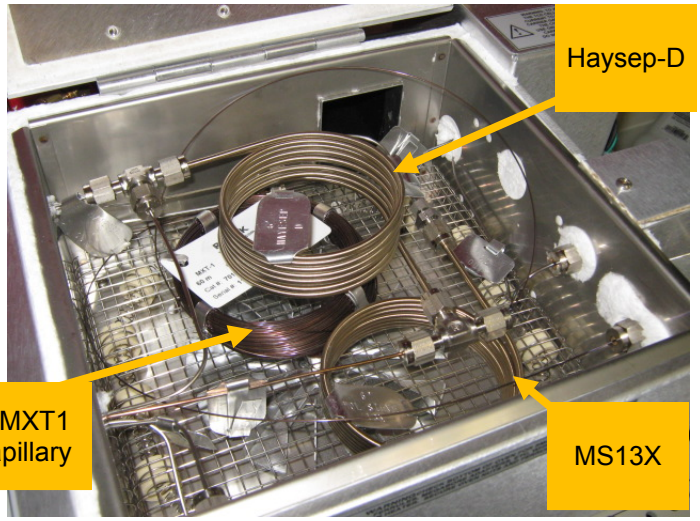
The Thermal Conductivity Detector (TCD) measures all non-sulfur molecules from 500ppm to 100%
The FID/Methanizer detector (FID/meth) measures hydrocarbons plus CO and CO2 from 1ppm to 50,000ppm
The Flame Photometric/FID combo detector (FPD/FID) measures all sulfur molecules plus hydrocarbons.



Inside the column oven are three columns.

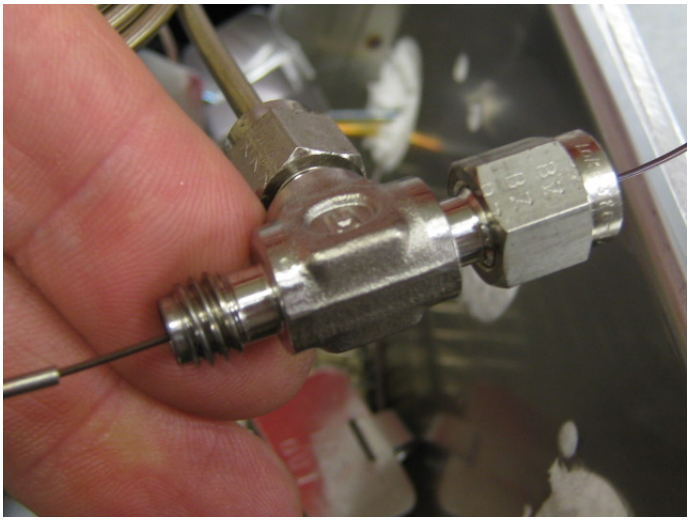
The Haysep-D (HD) and MoleSieve (MS13X) columns together separate H2, O2, N2, CO, CO2 and C1-C5 hydrocarbons as well as water.

The 60meter MXT1 capillary column separates the sulfur molecules and also hydrocarbons from C1-C10



Muliple Gas#3 plus Sulfur GC Configuration

The 60meter MXT1 capillary column is connected to the Haysep-D column using a “tee” fitting. When the sample is injected, it is split so about half the sample flows into the HD column and the other half flows into the capillary column. Notice how the cap column is inserted into the sample delivery tube in such a way that the sample splits cleanly. If the connection is not made in this way, the peaks will not be as sharp.

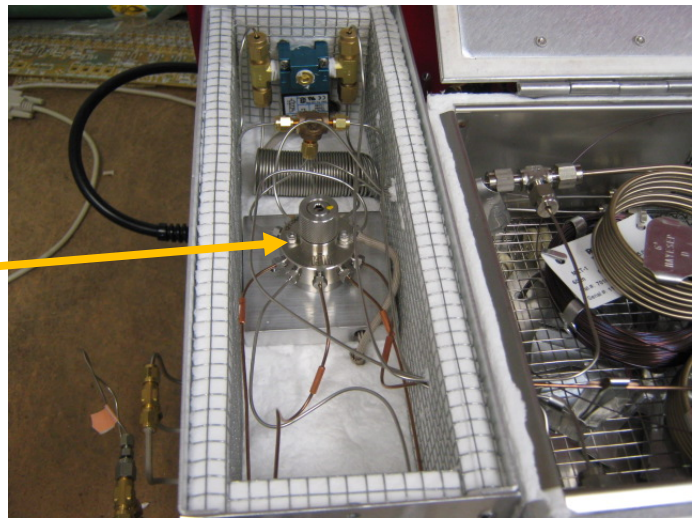


The other end of the capillary column is connected to the FPD/FID detector using a Swagelok nut and graphite ferrule.

The TCD and FID/methanizer detectors are connected in series, so all molecules exit the HD/MS13x columns and flow first through the TCD and then exit the TCD and flow into the FID/methanizer via a 1/16’ stainless steel tube.

Muliple Gas#3 plus Sulfur GC Configuration

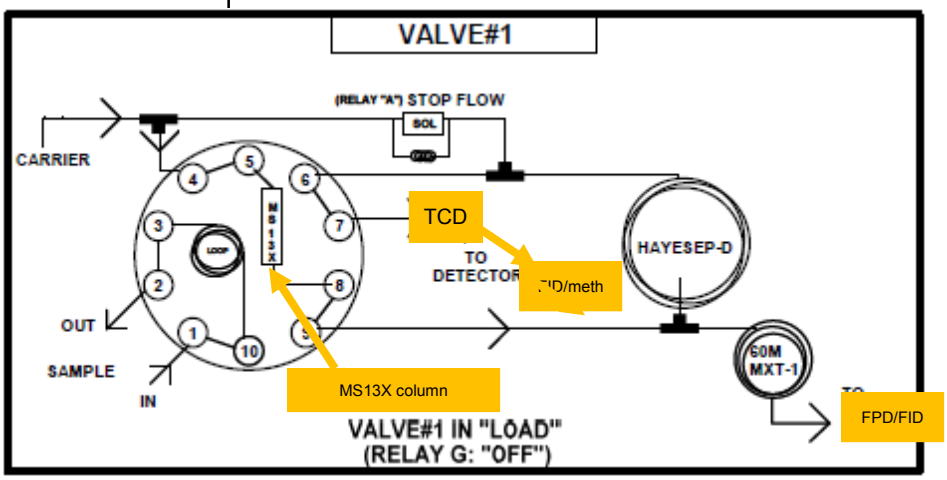
A 10 port Valco gas sampling valve is mounted in the heated valve oven.

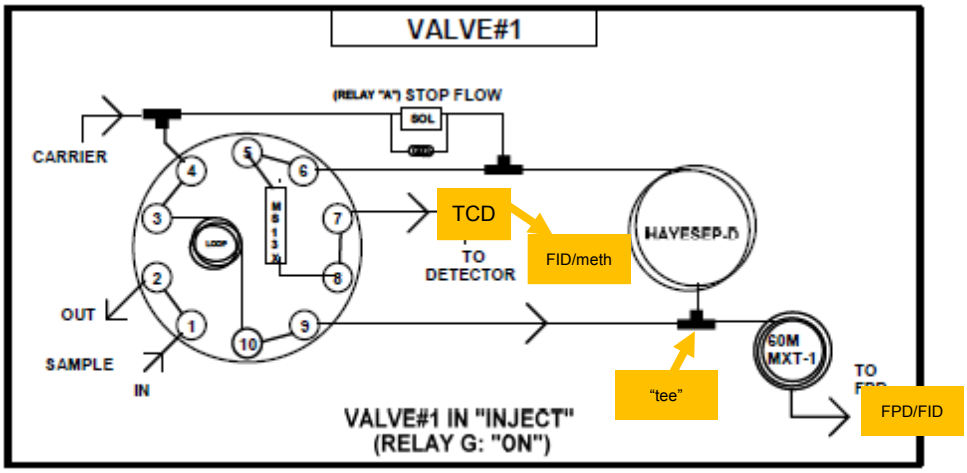


The valve is plumbed as shown in the diagram.

In the Load position the carrier gas flows through the three columns and into the detectors but the sample loop is isolated so sample can be loaded into the loop.

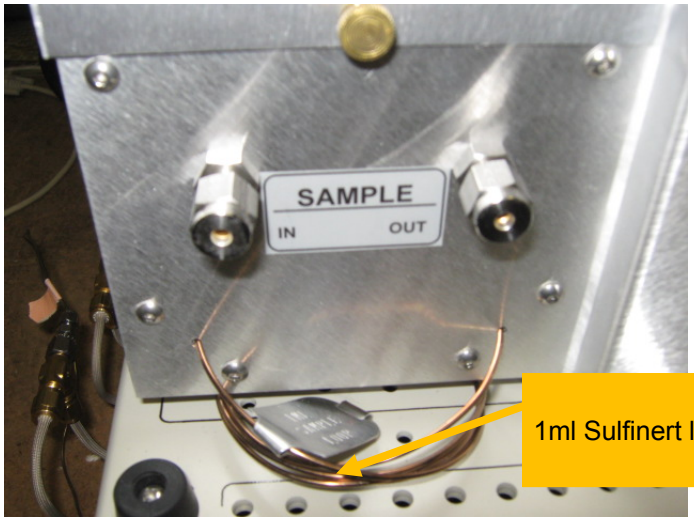
When the analysis is started, the valve rotates to the Inject position so the carrier gas now pushes the sample out of the loop, to the “tee” fitting where it splits into two paths.





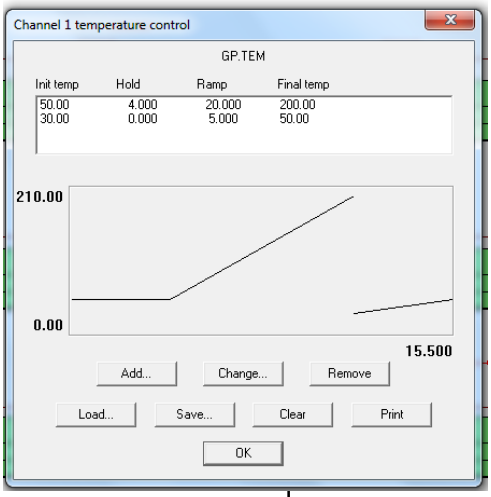
Muliple Gas#3 plus Sulfur
GC Configuration

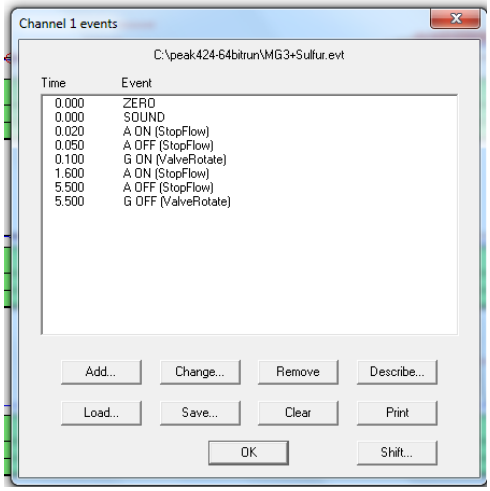
The sample loop is loaded with new sample by flushing the loop with 10ml or more of fresh sample. The loop itself is 1ml, but it takes 10ml to completely flush it. Sulfur compounds like H2S can be lost on active metal surfaces, so we use a special kind of tubing to make the loop called “Sulfinert”.



Sample can be pushed through the loop with a syringe, or it can flow continuously, or it can be pulled through with suction (vacuum pump). For sulfur compounds it is important to use Teflon tubing to avoid losses.

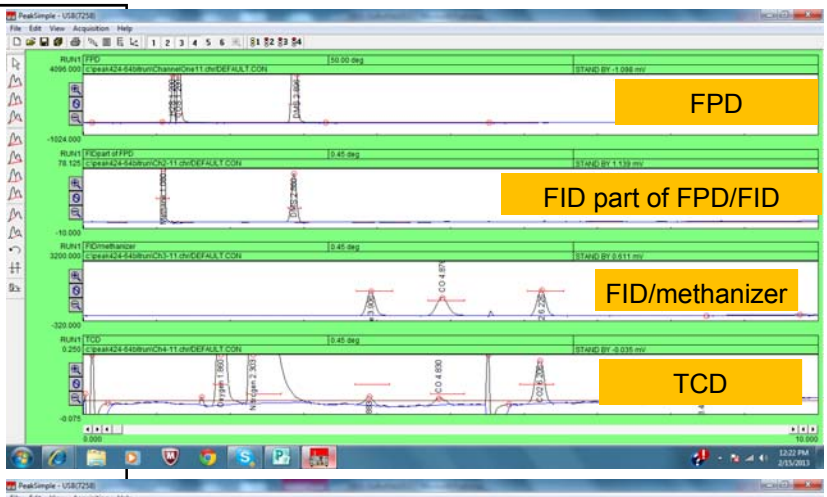
The GCs temperature program and event table is set up as shown. Users may expect to make modifications depending on the exact molecules being measured.



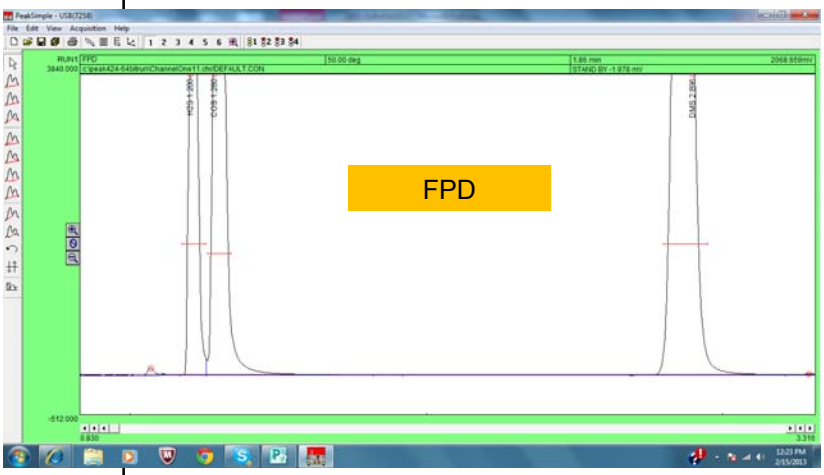


Muliple Gas#3 plus Sulfur GC Configuration

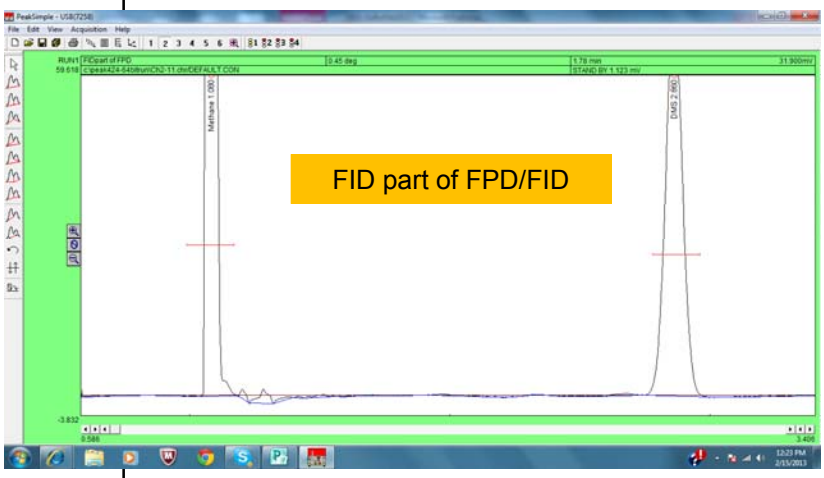
Since there are four detectors, the PeakSimple software will show four chromatograms simultanouosly on the computer monitor.



Zooming in on the FPD chromatogram you can see Hydrogen Sulfide (H2S), Carbonyl Sulfide (COS) and Di Methyl sulfide (DMS). The FPD is blind to hydrocarbons like methane.

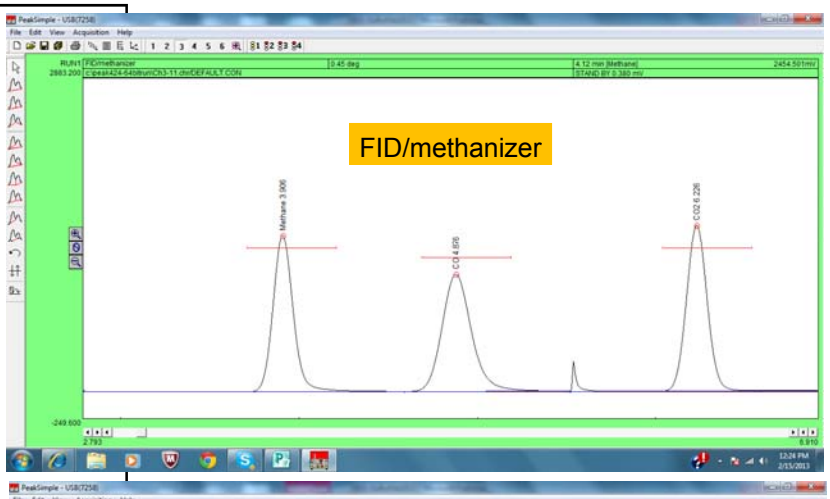


The FID part of the FPD/FID combo detector detects methane and DMS (C2H6S), but does not detect H2S and COS since the FID only detects molecules with carbon-hydrogen bonds. When the FPD/FID combo detector is optimized for best sulfur detection, the FID sensitivity and range is reduced to less that what a normal FID detector would deliver.

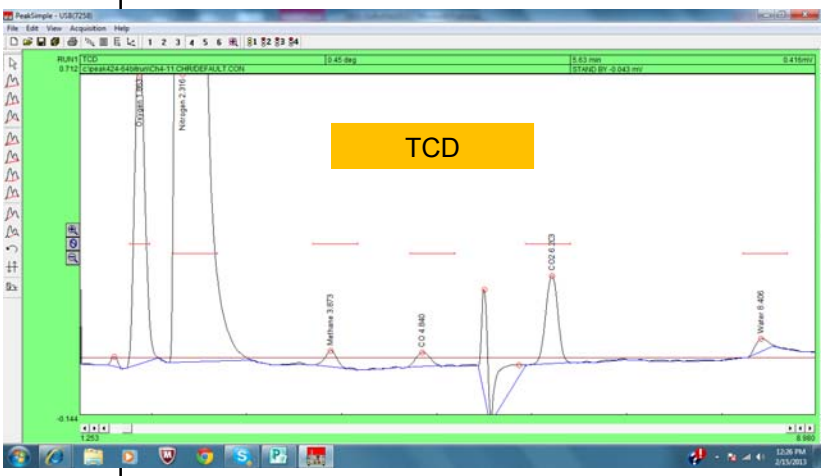


Muliple Gas#3 plus Sulfur
GC Configuration

The FID/methanizer detects all hydrocarbons from methane to hexane and also CO and CO2 which are converted to methane by the methanizer. Detection from 1ppm to 50,000ppm are possible. Shown are methane, CO and CO2 at 1000ppm.



The TCD detects Hydrogen, Oxygen, Nitrogen, Methane, CO, CO2, Water and all molcules with boiling points below hexane. Detection of most molecules is possible from 500ppm to 100%. Hydrogen can be detected from 10ppm to 100% if using Nitrogen or Argon carrier, but this increases detection limits for everything else from 500ppm to about 5000ppm. With helium carrier detection limit for hydrogen is about 10,000ppm to 100%.

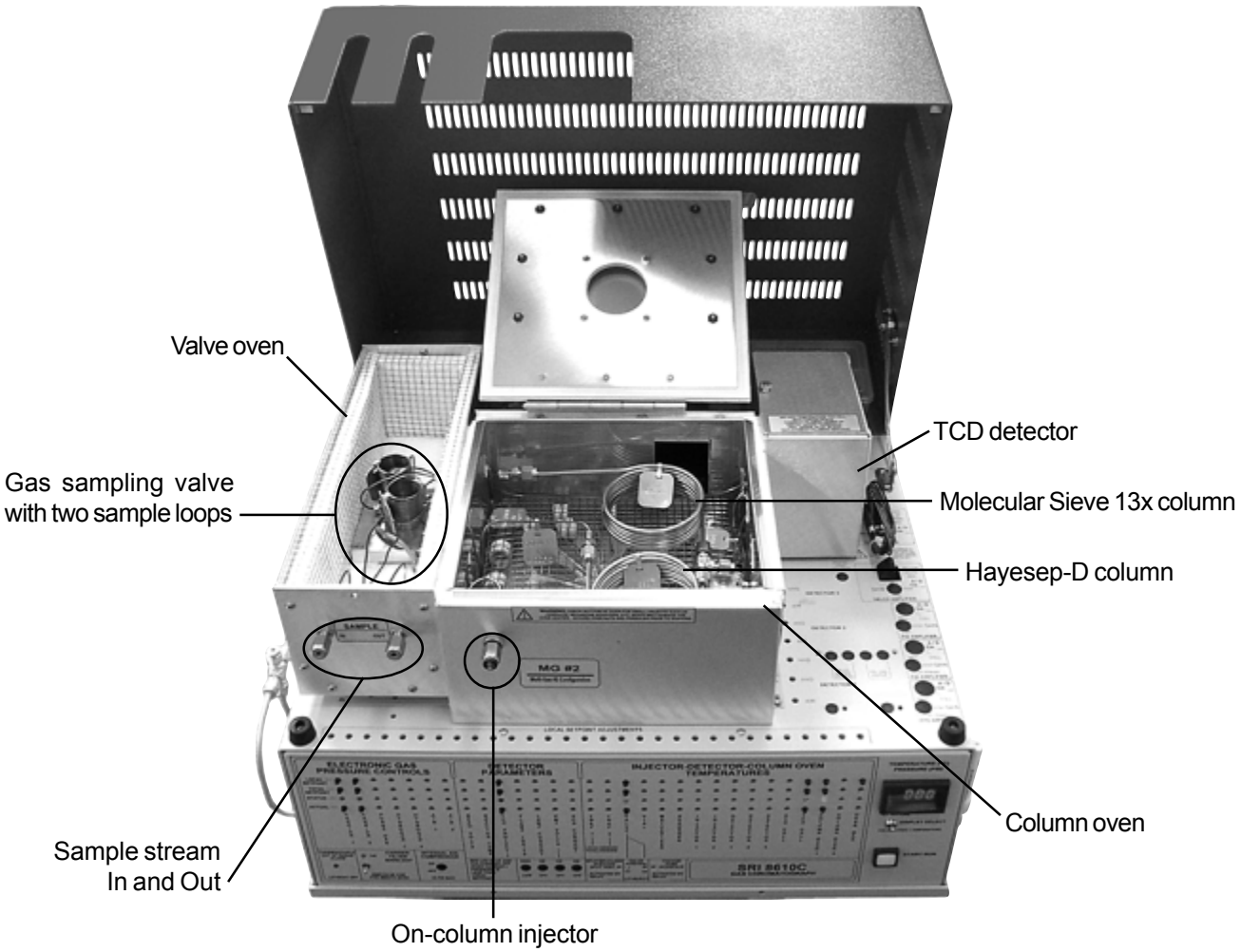


In some cases, the GC can be equipped with a second TCD detector , valve and column specifically to detect Hydrogen while helium carrier is used for the other molecules.

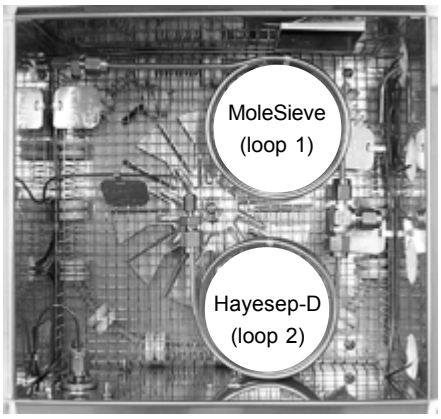
POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #2

System Overview

The SRI Multiple Gas Analyzer #2 (MG#2) is configured on the 8610C chassis. It is equipped with a gas sampling valve plumbed with dual sample loops in a heated valve oven, and two packed columns in the column oven. The basic model, shown below, comes with a TCD detector. The MG#2 may optionally be equipped with a FID/methanizer or HID detectors in addition to the TCD. A capillary column in parallel with the Hayesep-D column is an option for separating out hydrocarbons through C_{20}.



The MG#2 separates a wide variety of peaks without co-elution by turning the carrier gas flow to the two packed columns ON and OFF individually at different times during the run. The carrier to the Molecular Sieve 13x column (carrier #1) is turned ON first to complete the separation of H_2, O_2, N_2, CH_4 and CO. At this point, the MoleSieve carrier flow is turned OFF and the Hayesep-D carrier (carrier #2) is turned ON. All compounds in the C_1-C_6 range are then separated by the Hayesep-D column. The MoleSieve column is connected to sample loop 1, and the Hayesep-D to loop 2.

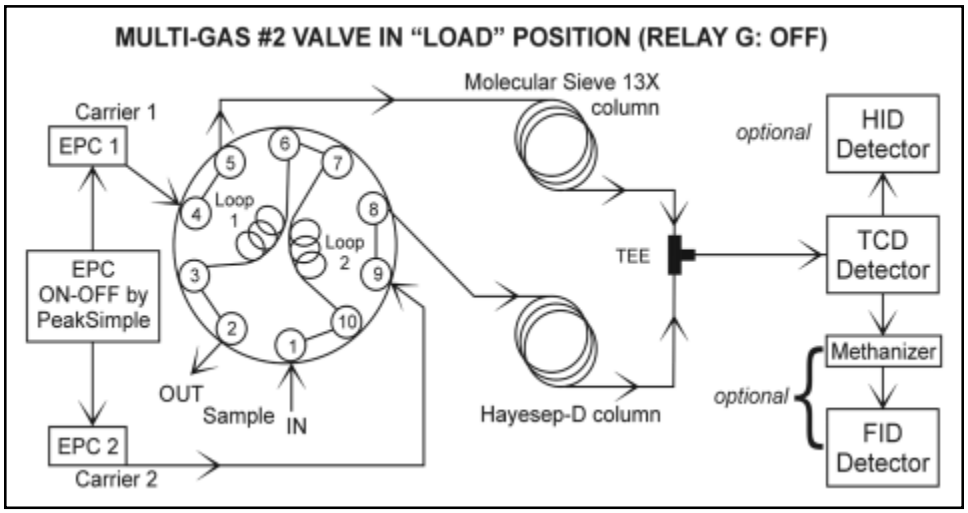


POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #2

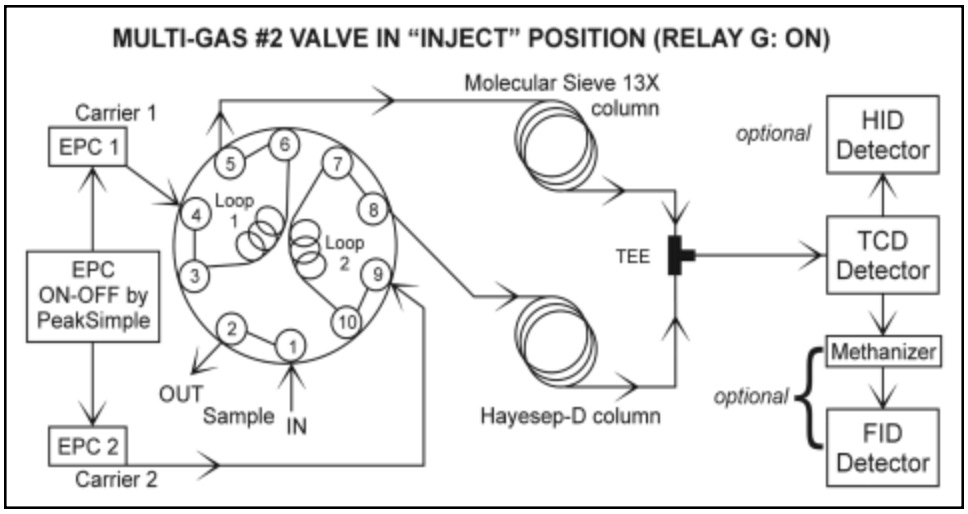
Theory of Operation

The MG#2 GC uses a single automated 10 port Gas Sampling Valve and multiple columns to separate a wide variety of compounds. It achieves this by turning the carrier gas flow to each column on at different times during the run. This procedure allows the Molecular Sieve 13x column to completely separate H_2, O_2, N_2, CH_4 and CO before the carrier flow to the Hayesep-D column is turned on. The Hayesep-D column then separates all compounds in the C_1-C_6 range. An optional 30-meter MXT-1 capillary column separates the remaining hydrocarbons through C_{20}, using the same carrier gas flow as the Hayesep-D column and an FID or HID detector.

The MG#2 is plumbed with two separate carrier gas flows, each regulated by Electronic Pressure Control (EPC) through the PeakSimple data system. Carrier 1 flows through sample loop #1 to the MoleSieve column, then on through the "Tee" to the TCD detector. Carrier 2 flows through sample loop #2 to the Hayesep-D column, then through the "Tee" to the TCD detector. Carrier #1 and #2 flows are turned ON and OFF by PeakSimple, controlled by the user with an Event table.



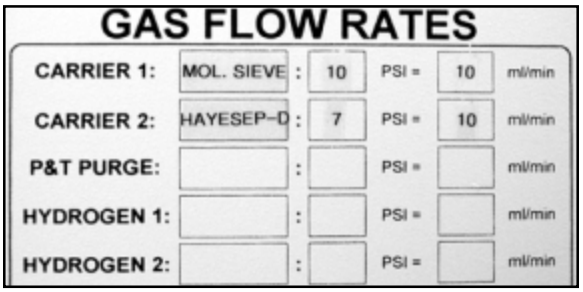
When the MG#2 valve is in the LOAD position, loops #1 and #2 are loaded with the sample gas stream while carrier flows #1 and #2 bypass the loops and travel on to the columns.



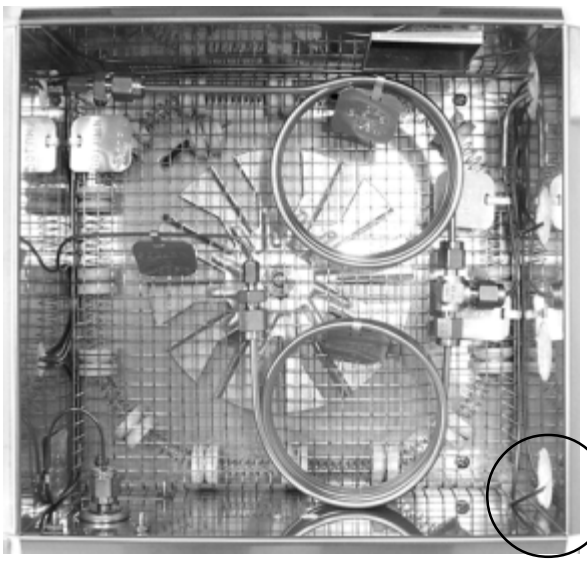
When the MG#2 valve is in the INJECT position, carriers #1 and #2 flow through the sample loops, sweeping their contents to the columns.

General Operating Procedure

1. Set the gas cylinder pressure 15-20psi higher than the head pressure (helium carrier). The carrier head pressure used to generate the test chromatograms at the factory is printed on the right-hand side of your GC. Verify that with carrier gas turned off at the cylinder, that the actual GC pressure reads ZERO.

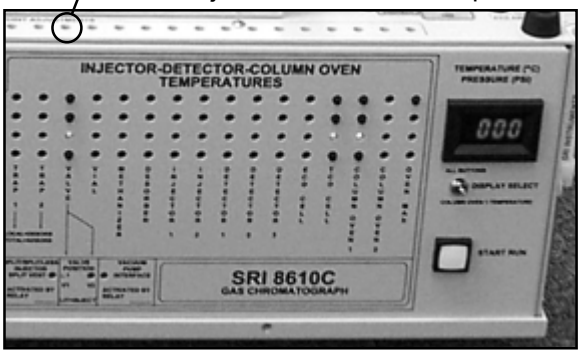


2. Damage or destruction of the TCD filaments will occur if current is applied in the absence of flowing carrier gas. ALWAYS verify that carrier gas can be detected exiting the TCD carrier gas outlet BEFORE turning ON the TCD current. Labelled for identification, the TCD carrier gas outlet tubing is located inside the column oven. Place the end of the tubing in liquid and observe. If there are no bubbles exiting the tube, there is a flow problem. DO NOT turn ON the TCD current if carrier gas flow is not detectable. A filament protection circuit prevents filament damage by shutting OFF the TCD current when the column head pressure is below 3psi. Because this protect circuit cannot prevent filament damage under all circumstances, any lack of carrier gas flow should be corrected before proceeding. NEVER turn both carrier #1 and carrier #2 OFF at the same time. Please see the TCD manual section for more information about the detector.



The TCD carrier gas outlet tubing is located inside the column oven. If there is also an FID detector on your MG#2, the TCD carrier gas outlet tubing is connected to the FID inlet bulkhead in the column oven wall. If your MG#2 has a TCD only, the end tubing will be on the outside of the column oven, on the detector side.

Use the trimpot directly above the "VALVE" zone to set or adjust the valve oven temperature.



3. Set the valve oven temperature to 90°C using the trimpot on the top edge of the GC front control panel.

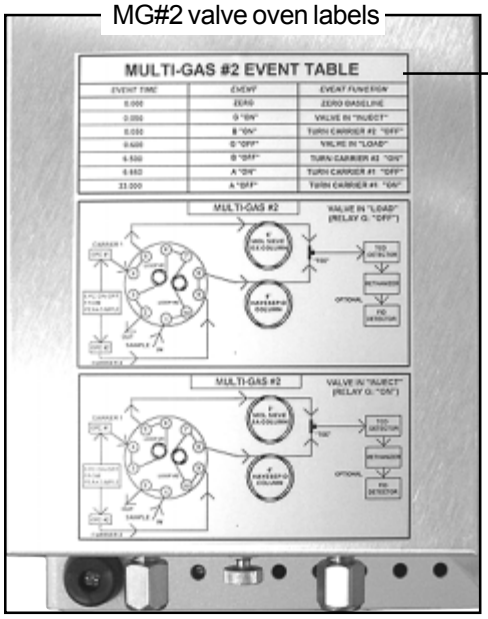
4. Turn the TCD current ON to LOW. If present, ignite the FID/methanizer and set the temperature to 380°C. If present, turn ON the HID current.

5. Set the column oven temperature program in PeakSimple as follows. (This is an example; your analysis may require a different temperature program.)

Initial	Hold	Ramp	Final
50.00	3.00	20.00	220.00
220.00	25.00	0.00	220.00

POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #2

General Operating Procedure continued

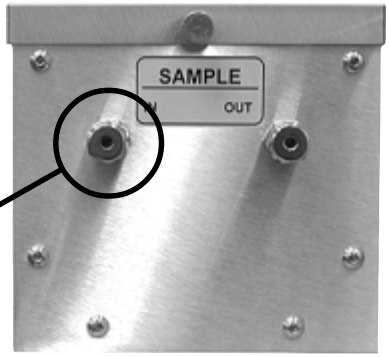


Example event table:

Time	Event
0.000	ZERO (zero data system signal)
0.050	B ON (carrier #2 OFF)
0.500	G ON (valve INJECT)
3.500	B OFF (carrier #2 ON)
3.600	A ON (carrier #1 OFF)
18.000	A OFF (carrier #1 ON- MoleSieve Bake Out phase)

6. Type in an Event table. The example shown is labeled on the MG#2 valve oven. The event table should allow for the elution of CO from the molecular sieve column before carrier #2 is turned back ON. The column oven temperature may be increased to speed the elution of the H_2, O_2, N_2, CH_4, and CO. Hydrocarbons like ethane and propane end up on the Molecular Sieve 13x column after its carrier is turned OFF and the Hayesep-D carrier is turned ON. The example Event table also turns Carrier #1 ON at the end of the run, while the column is still hot enough to bake the hydrocarbons out of the MoleSieve column. This Bake Out phase is required to get rid of any residual peaks, so that following analyses are not compromised. Keep Carrier #1 ON and the column oven hot long enough for any contamination peaks to elute. Click the Edit drop down menu in the main PeakSimple window, then choose Overall, then make sure that the "Reset relays at end of run" checkbox is selected. Otherwise, you will have to include G OFF at the end of the event table.

7. Load your sample gas stream by connecting the flow to the sample inlet port ("SAMPLE IN") on the front of the valve oven with the provided 1/8" swagelok nut and brass ferrule.

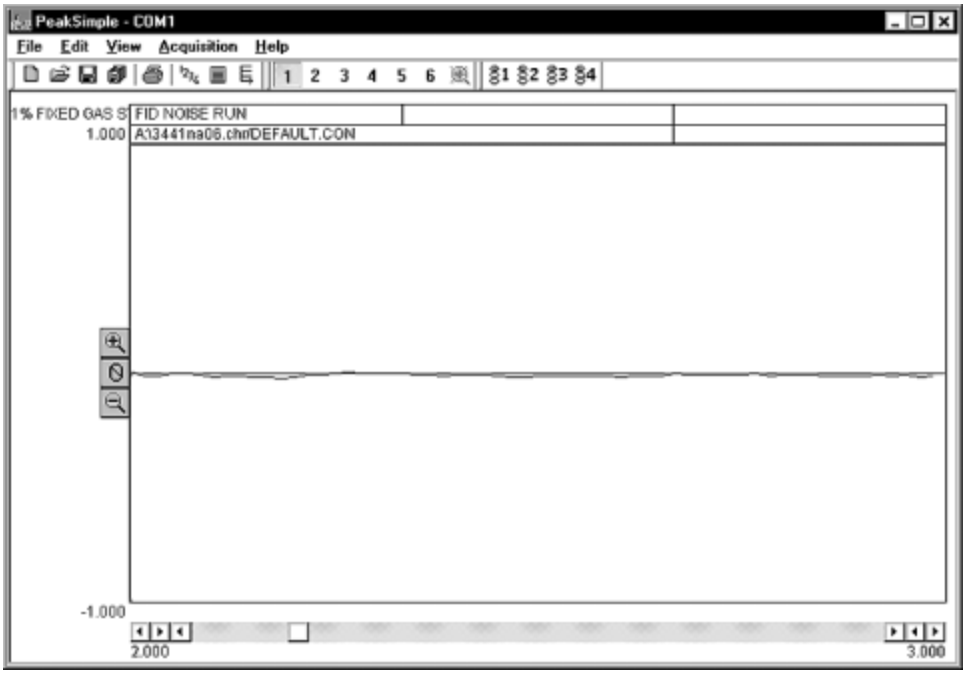


8. Start the analysis by pressing the START RUN button on the front of your GC, or by pressing your computer keyboard spacebar.

POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #2

Expected Performance

These two noise runs were made with identical parameters (carrier flow, columns, temperature program) on a Multiple Gas Analyzer #2 GC equipped with FID and TCD detectors. The only differences are the detector particulars, which are listed next to the appropriate chromatogram.



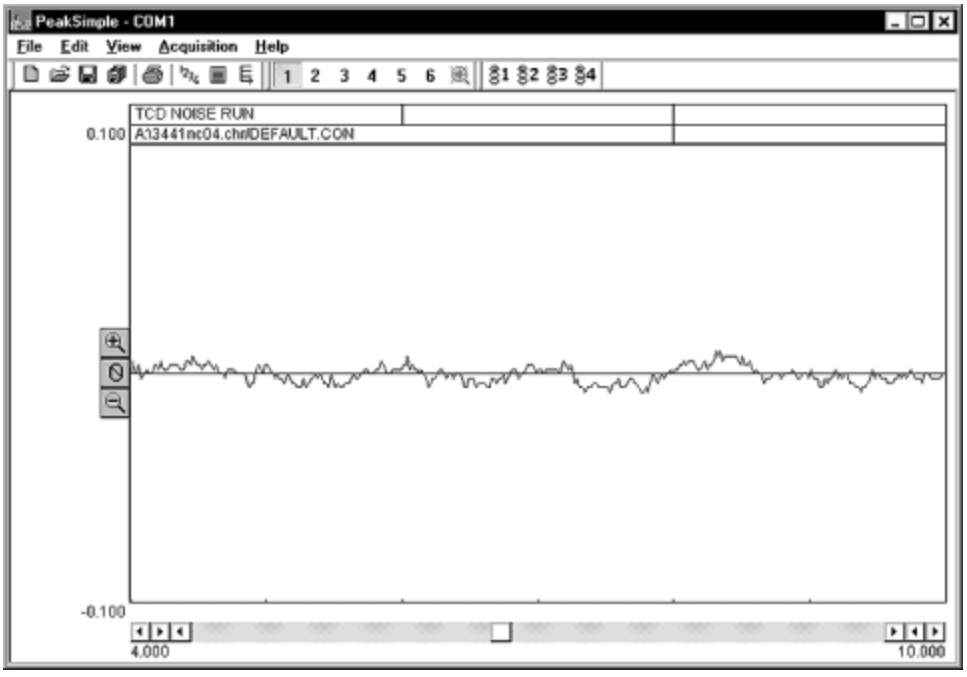
FID noise run

FID gain = HIGH
FID temp = 380°C
FID ignitor = -400
Methanizer in FID detector body

Valve temp = 90°C
Carrier #1 Mol. Sieve 13x = 20mL/min
Carrier #2 Hayesep-D = 20ml/min
Total carrier flow = 40mL/min

Temperature program:

Initial	Hold	Ramp	Final
80°C	20.00	0.00	80°C



TCD noise run

TCD current = LOW
TCD temp = 150°C

POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #2

Expected Performance

The first chromatogram shows the TCD response to a 1% Fixed Gas Standard sample. Using the same valve temperature, column oven temperature program, carrier flow and event table, the second chromatogram shows the TCD response to a Natural Gas Standard sample. The event table used is shown on the ***General Operating Procedure continued*** page.

Columns: 2-meter Hayesep-D, 2-meter Molecular Sieve 13x
TCD current = LOW; TCD temp = 150°C

Carrier: Helium at 40mL/minute combined (20mL/minute through each column)
Valve temp = 90°C

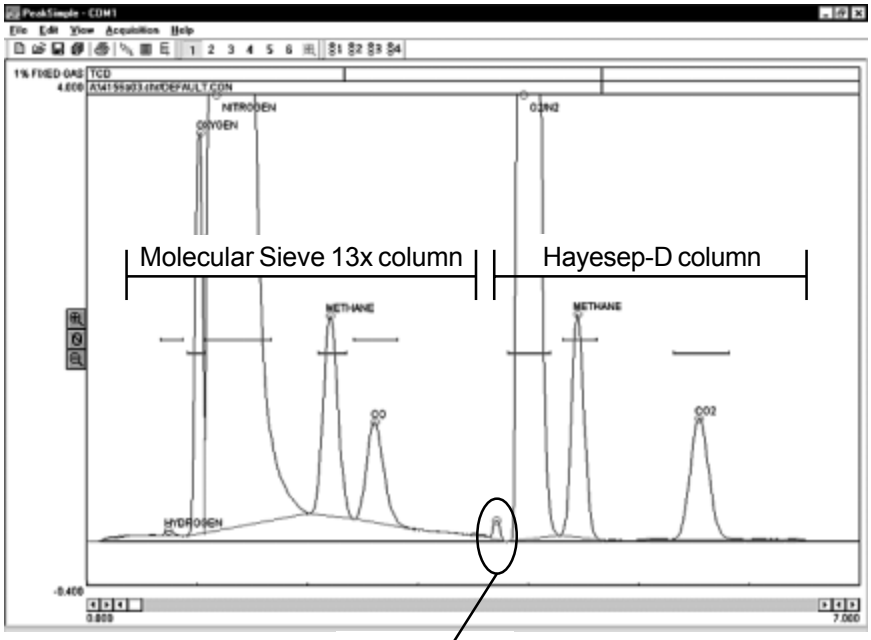
Column Oven Temperature program:

Initial	Hold	Ramp	Final
50°C	3.00	20.00	220°C
220°C	25.00	0.00	220°C

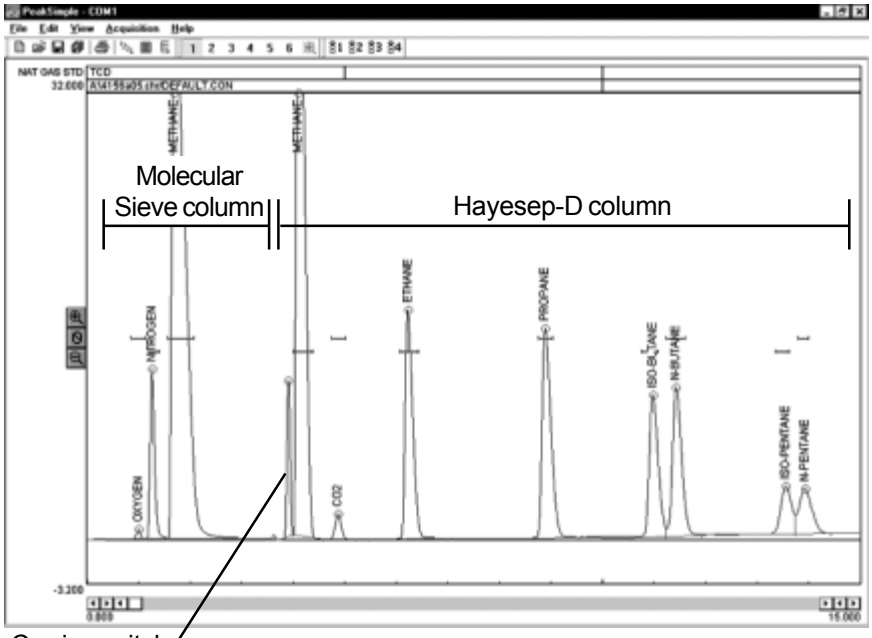
1% Fixed Gas Standard

RESULTS:

Component	Retention	Area
Hydrogen MS	0.733	00.2510
Oxygen MS	1.016	16.0495
Nitrogen MS	1.166	1108.7680
Methane MS	2.200	16.5050
CO MS	2.600	09.7370
O_2/N_2 Hay-D	3.950	863.6340
Methane Hay-D	4.433	15.7300
CO_2 Hay-D	5.533	12.9205
TOTAL		2043.5950



Carrier switch



Carrier switch

Natural Gas Standard

RESULTS:

Component	Retention	Area
Oxygen MS	0.983	3.4190
Nitrogen MS	1.250	72.5450
Methane MS	1.683	706.7920
Methane Hay-D	4.083	587.7140
CO_2 Hay-D	4.850	14.7710
Ethane Hay-D	6.216	169.1275
Propane Hay-D	8.866	180.2660
Iso-Butane Hay-D	10.966	126.6950
N-Butane Hay-D	11.400	134.1470
Iso-Pentane Hay-D	13.533	50.1540
N-Pentane Hay-D	13.916	54.4740
TOTAL		2099.1045

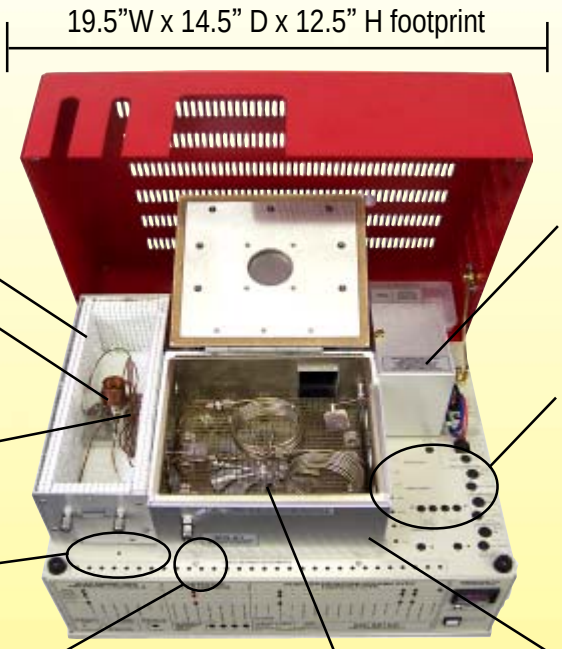
SRI GC Innovations

Multiple Gas Analyzer #1

Keep your gas products in spec! Monitor gas product purity, natural gas, and ambient air quality.

- ☆ Separates multiple gases with a single injection
- ☆ Very tolerant of user adjustments and timing variations
- ☆ Simpler than other multi-gas capable GC systems
- ☆ Multiple gas analysis in a compact unit

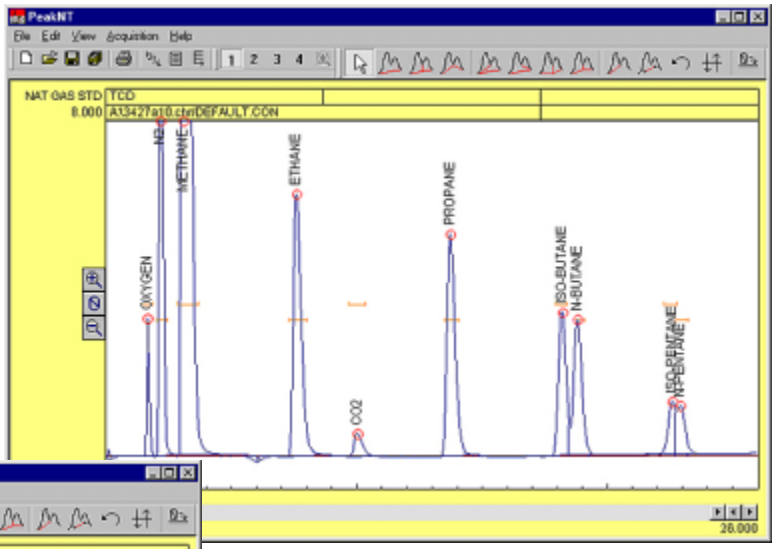
Sounds expensive and complicated to operate? Not from SRI! The SRI Multiple Gas Analyzer #1 uses just ONE gas sampling valve and TWO analytical columns to perform the same separations that require multiple valves and columns in other systems. Best of all, the Multiple Gas Analyzer #1 can achieve ppm to 100% concentrations with a single injection!

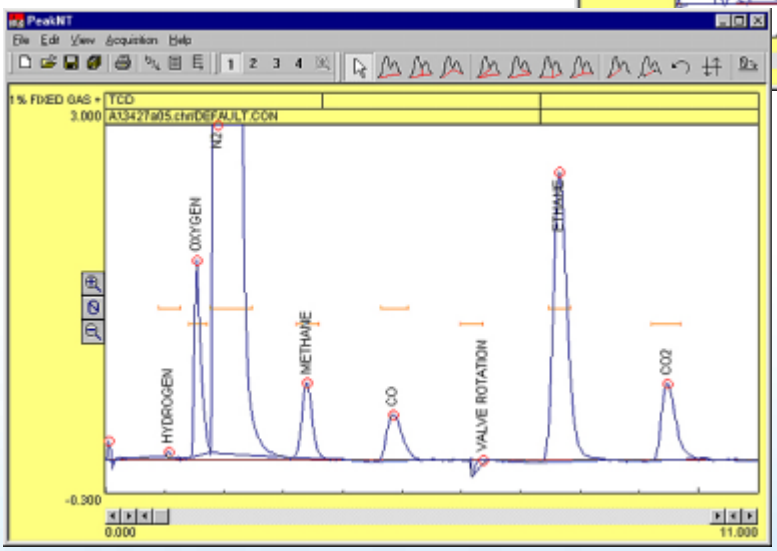


Specifically Designed for Separation of Whole Gas Components and Natural Gas Samples

Multiple Gas Analyzer #1

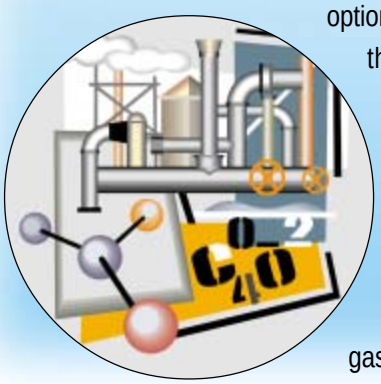
Separating out the hydrocarbon components of natural gas facilitates accurate BTU quantification. This compositional analysis of a natural gas standard by an SRI Multiple Gas Analyzer #1 shows good separation up to the pentanes. Performing compositional analyses of natural gas product before and after refining helps to maximize process efficiency and profit.

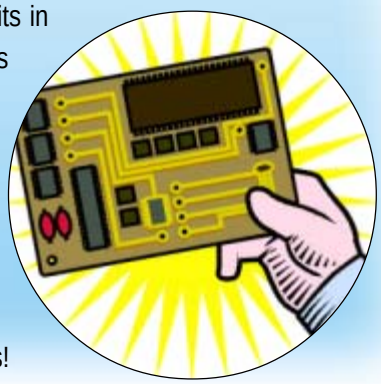




The same instrument produced this chromatogram, separating a sample mix of 1% fixed gas standard and ethane. With the built-in PeakSimple data system, the gas sampling valve was programmed to inject the sample loop contents into the carrier gas stream at 5 seconds, then rotate back at 6 minutes, after CO elution.

The basic Multiple Gas Analyzer #1 has a TCD detector only; this model provides analyses in the 250ppm to 100% range for fixed and natural gases. A second option is a TCD, Methanizer, and FID detector combination which adds 5ppm detection limits for CO, CO_2, and all hydrocarbon peaks; this model is useful for air quality monitoring and other applications. A third option is a TCD-HID detector combination, for detection limits in the 10ppm range for all analytes...the HID even sees hydrogen! Since we build each GC from the boards up, the Multiple Gas Analyzer #1 may be further customized to suit your application needs. With the optional built-in "whisper-quiet" air compressor, the Multiple Gas Analyzer #1 can be used with the SRI H_2-50 hydrogen generator to separate multiple gases anywhere, without using compressed gas cylinders!





8610-0070	Multiple Gas Analyzer #1 GC with TCD detector
8610-0071	Multiple Gas Analyzer #1 GC with TCD, Methanizer, FID & built-in Air Compressor
8610-0072	Multiple Gas Analyzer #1 GC with TCD & HID detectors
8690-0070	Built-in Air Compressor, 120 VAC
8690-2270	Built-in Air Compressor, 220 VAC

MG3 ONCOLUMN