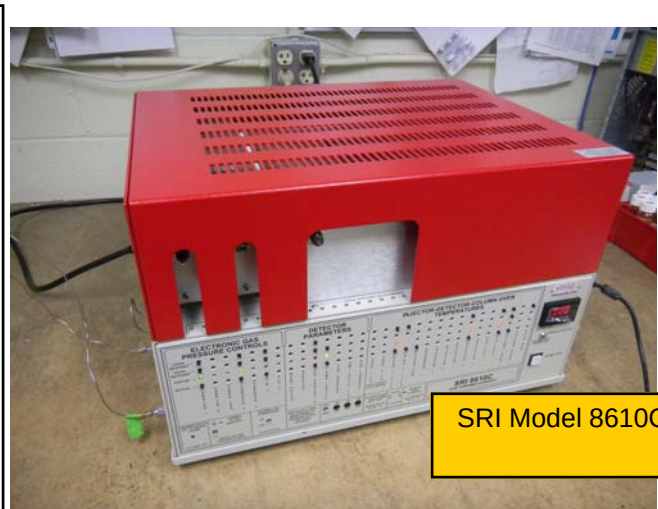
Multiple Gas#5 GC configuration
Jan 2016

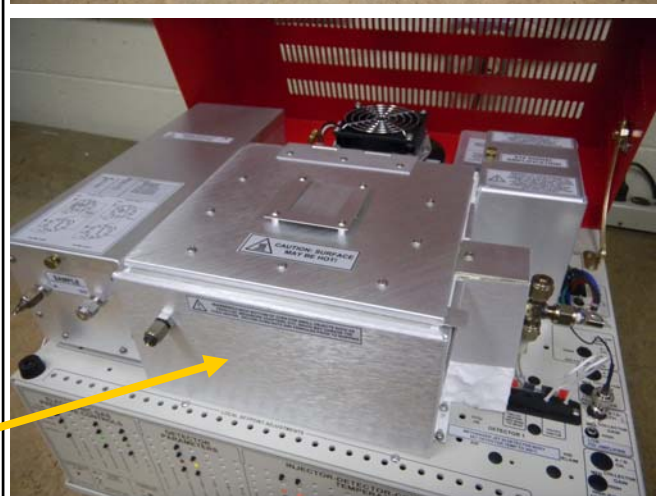
History:
Unfortunately there is no single column that can separate:
Hydrogen
Oxygen
Nitrogen
Methane
CO
CO2
Ethane
Water
Propane
Butane
Pentane

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



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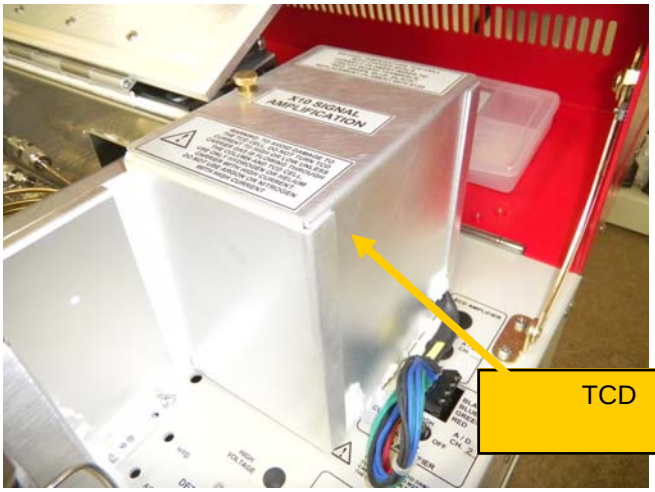
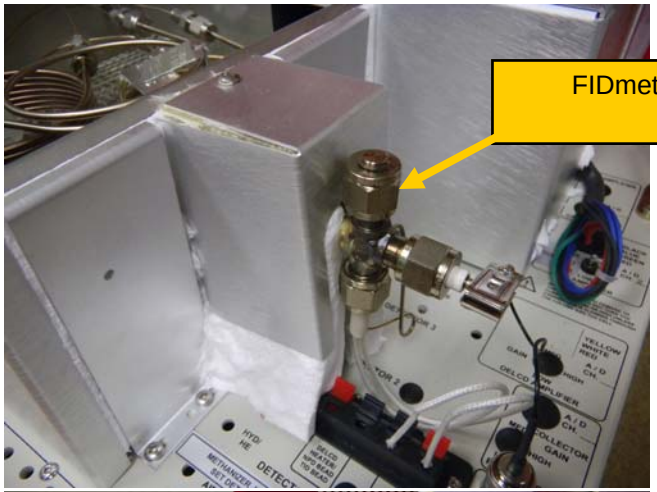
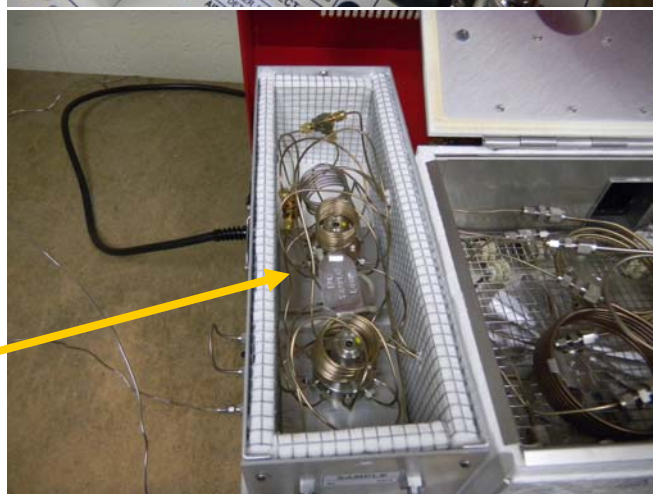
On the right side of the column oven is located the Thermal Conductivity Detector (TCD) which detects all the gases from 200ppm to 100% except hydrogen (see detailed explanation for this later).



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.



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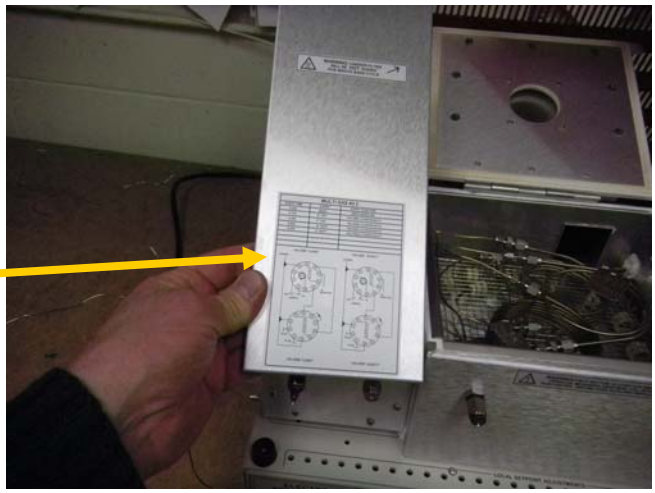
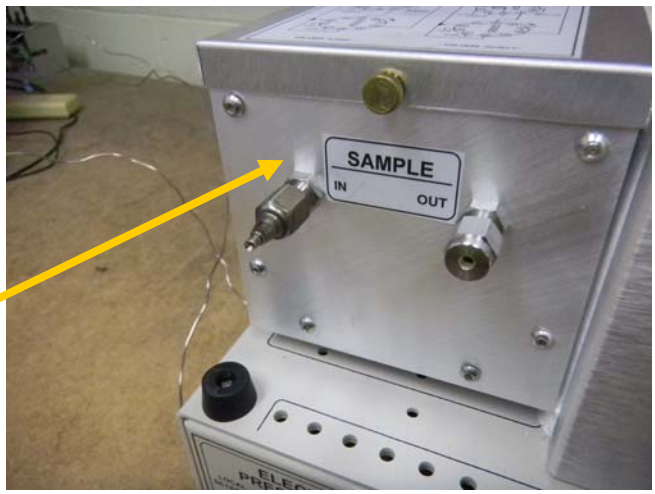
The Valco 10port gas sampling valve (GSV) looks like this. There is an electric motor inside the GC which turns a Teflon seal inside the valve at a specific time during the analysis to change the carrier gas flow path.

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.



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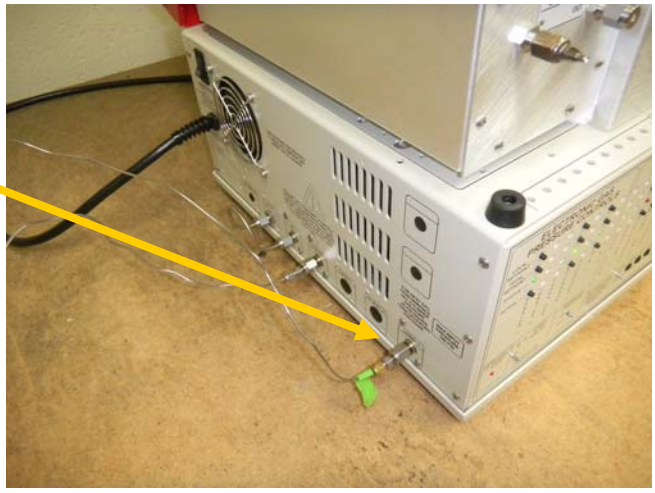
Jan 2016

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.

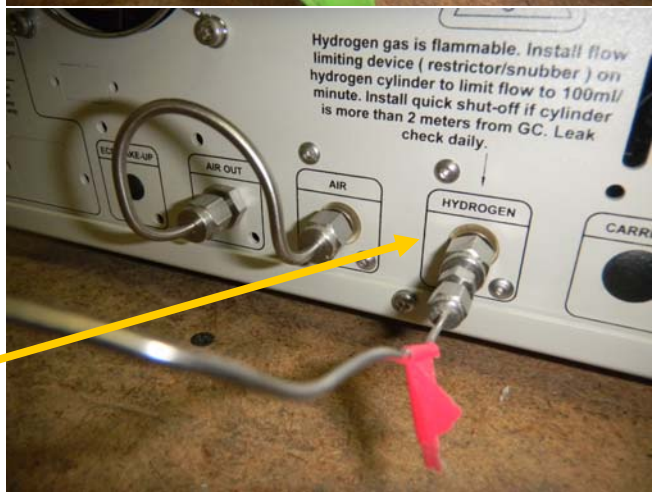


Helium is the most common choice because it gives the best overall results. However the sensitivity is not as good for hydrogen as it is for the other gases. This is because the TCD sensitivity depends on the difference of the "thermal conductivity" of the carrier gas relative to the sample molecule.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

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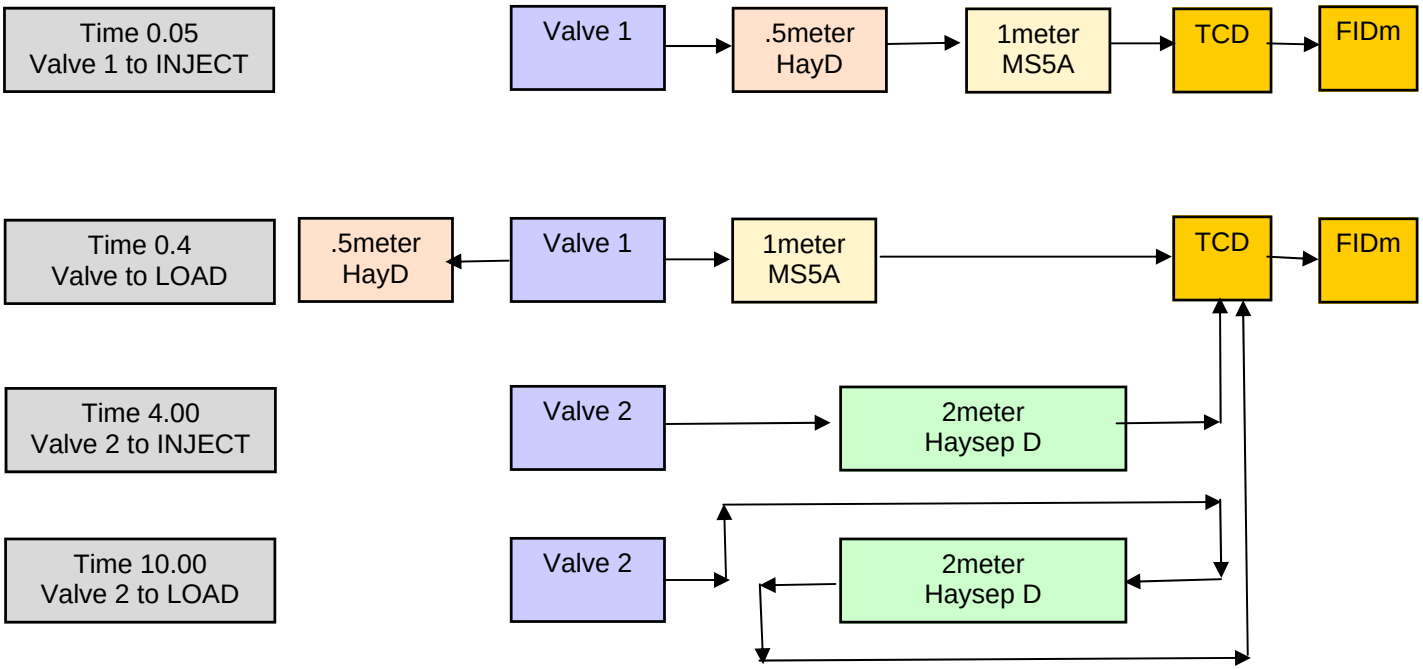

The schematic above shows the 4 steps in the MG5 analysis after the sample has been loaded into the loop of each valve.

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”.

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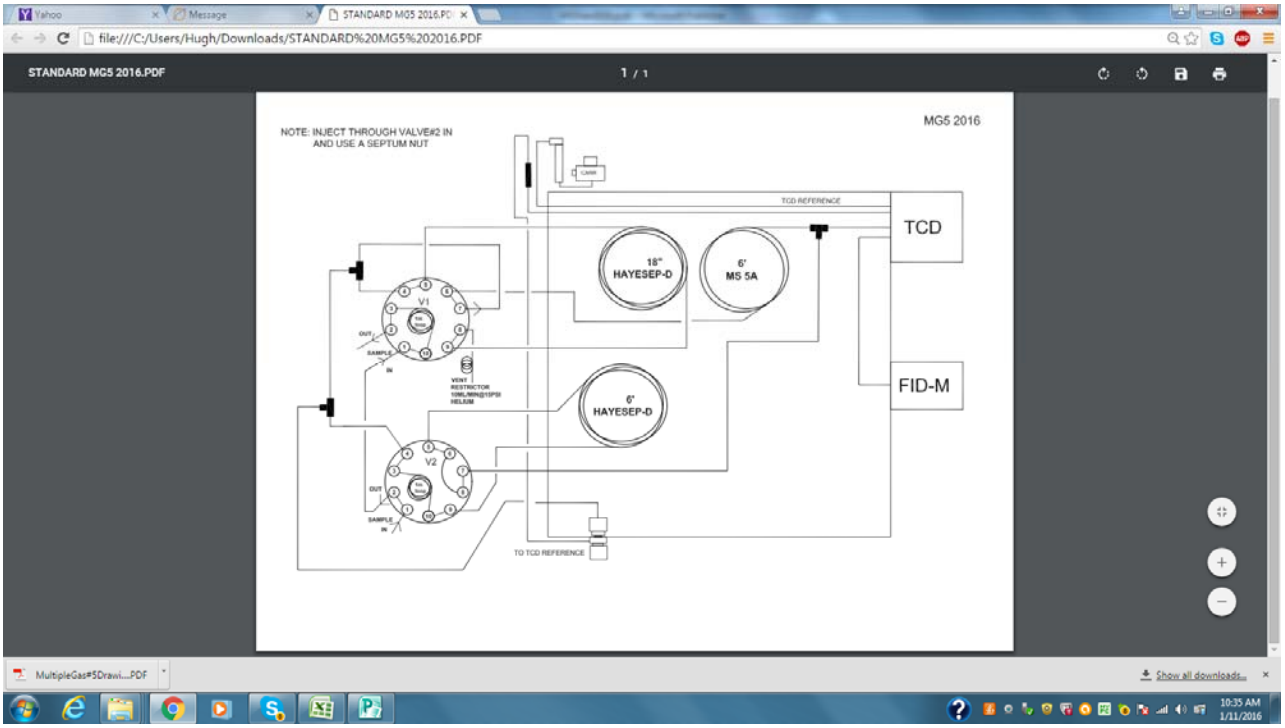
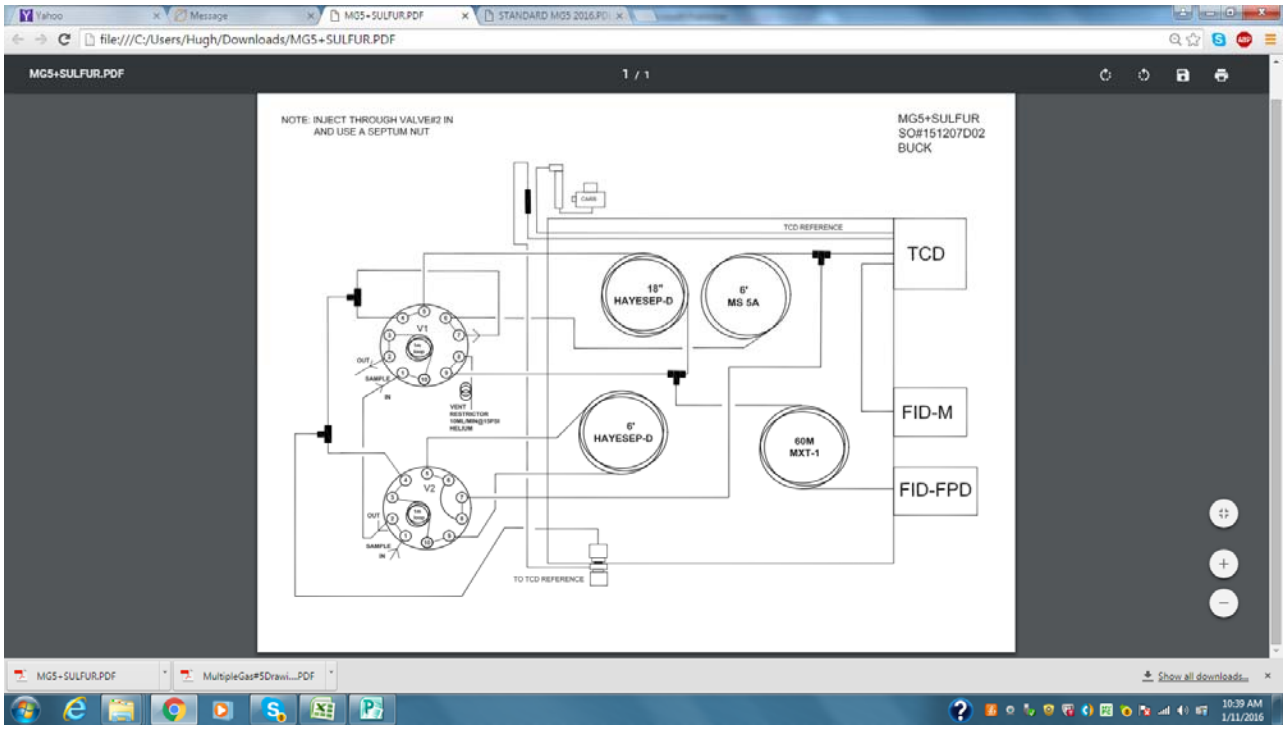


The diagram above shows a schematic of the “basic” MG#5 configuration with both valves in the INJECT position. A similar diagram below shows the “basic” MG#5 plus an additional column and FPD/FID detector to measure sulfur gases like H2S, CO2, SO2, mercaptans, DMS etc.



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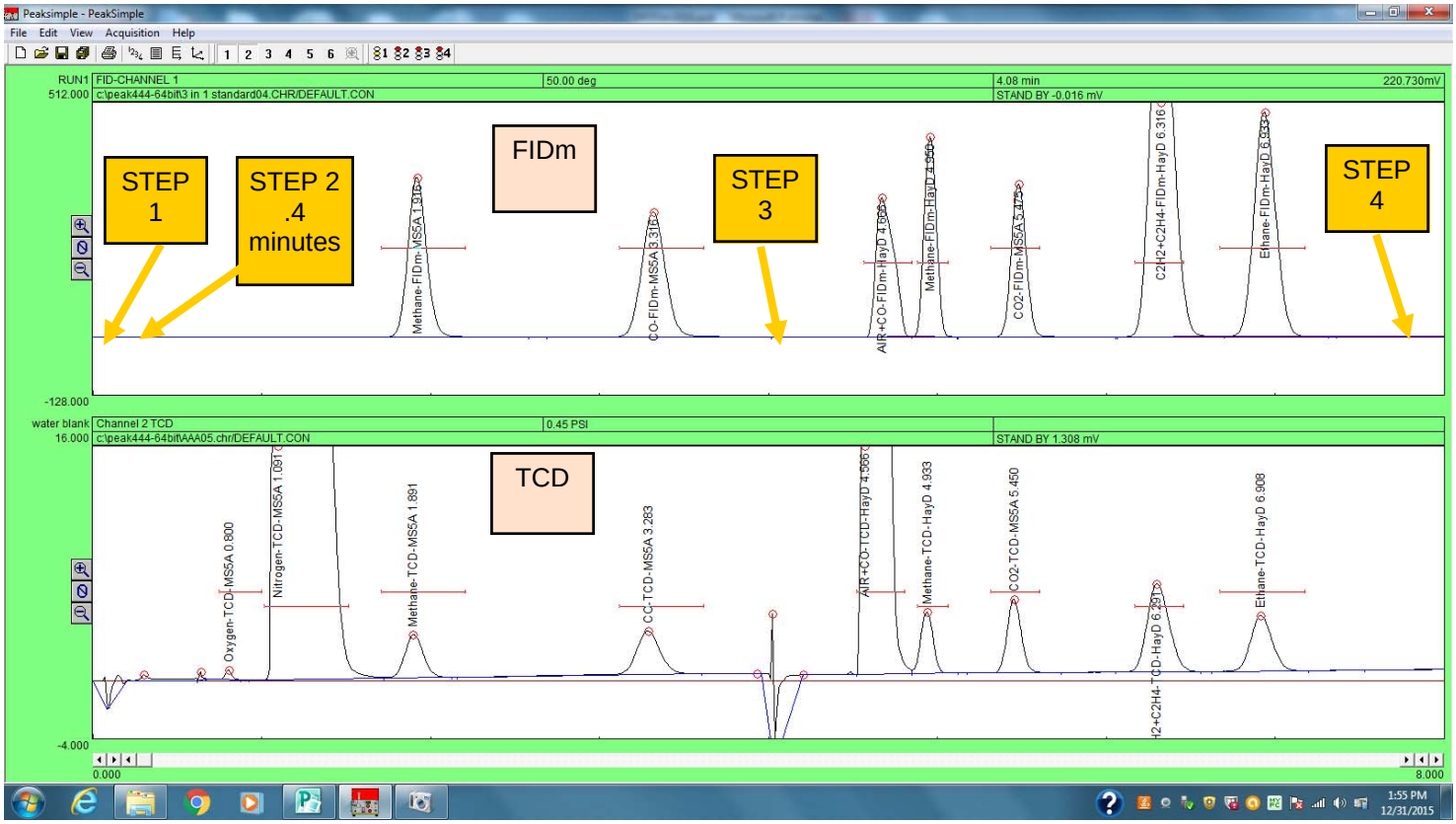

This is a typical chromatogram of gases at 1% in Nitrogen. The FIDmethanizer chromatogram is on the top and the TCD on the bottom.
STEP 1: Valve1 is turned to the INJECT position (Relay G on). The carrier gas pushes the sample out of the valve loop onto the 5.meter Haysep D column. H2, O2, N2 CH4 and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

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Multiple Gas#5 GC configuration
Jan 2016

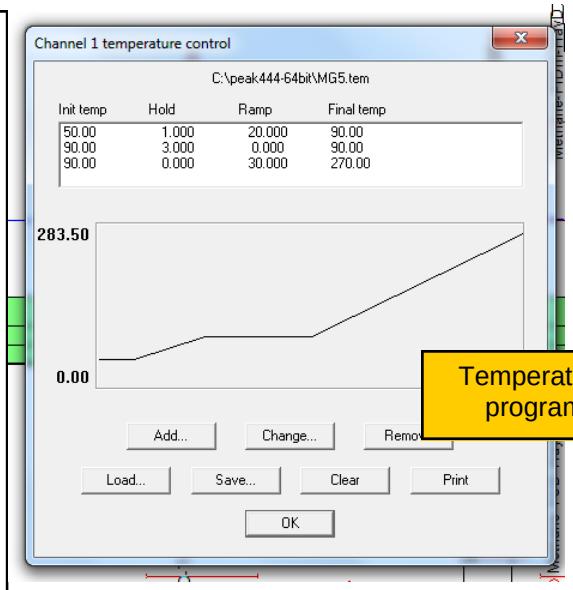
The screen at right shows the oven temperature program used.

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.

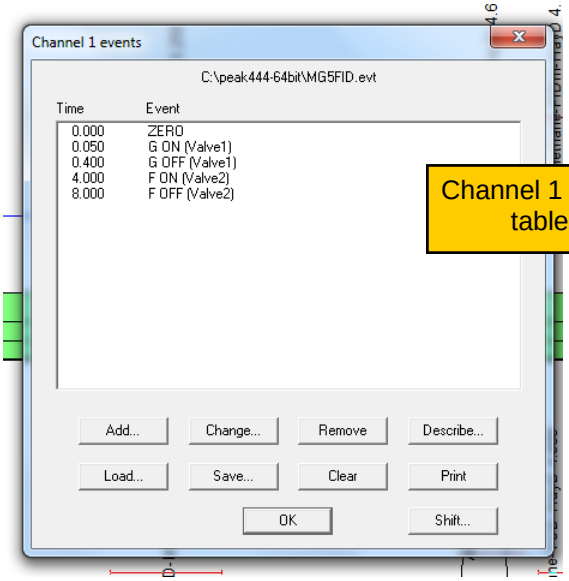
At some point while the column temperature increases, STEP 4 occurs, backflushing any un-eluted molecules.

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

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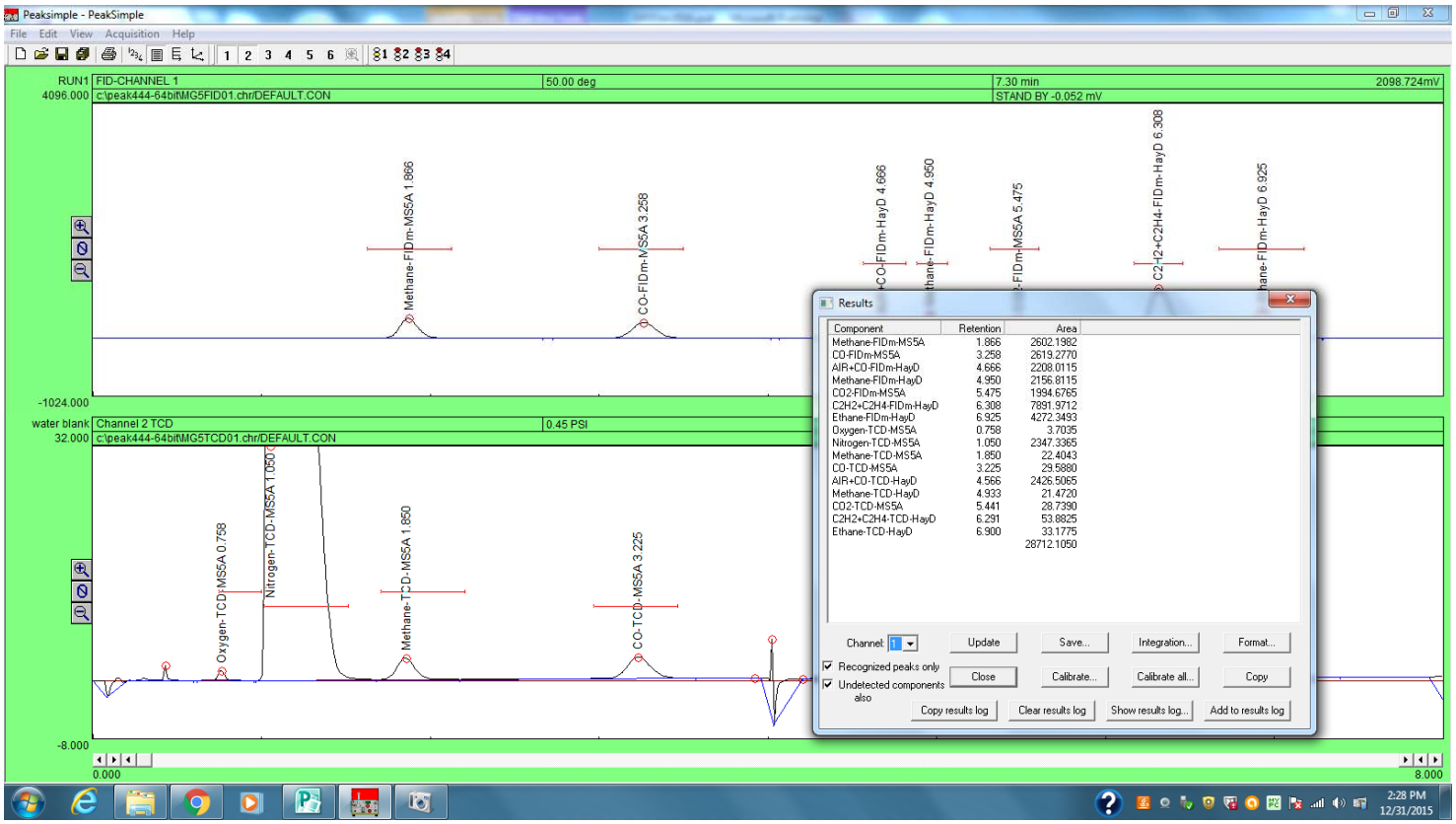

The sample above (helium carrier at 15psi) shows 1% each methane, CO, CO2, ethane, ethylene and acetylene with a little oxygen, and nitrogen balance. Note that the area of the methane, and CO peaks are about the same on the FIDm, and similar but not identical on the TCD.

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.

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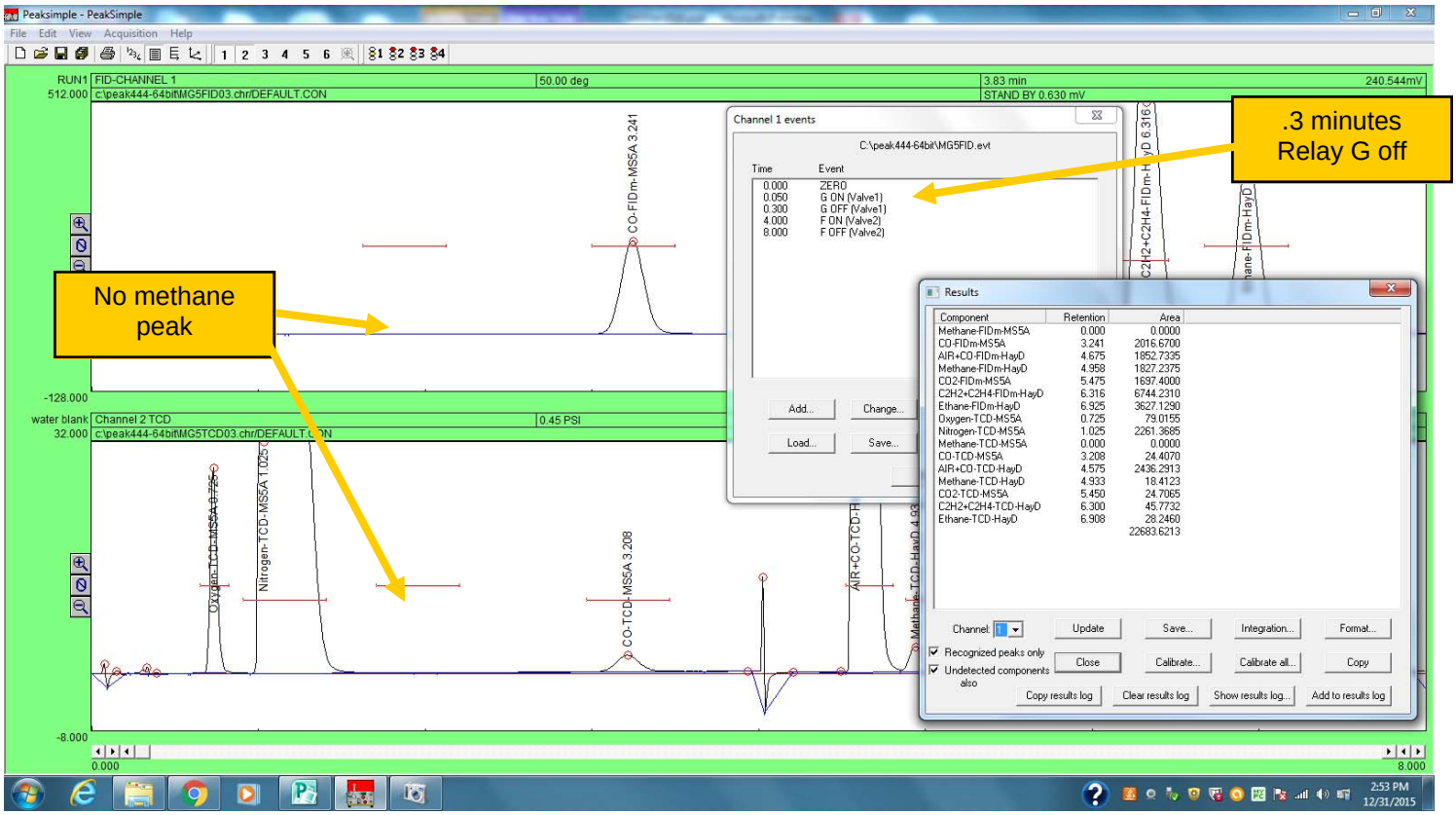

Compare the same sample but with Relay G off at .3 minutes instead of .4 minutes as in the previous page. The methane peak has disappeared because the time that Relay G turned off was too early, so the methane peak did not make it onto the MS5A column and was back-flushed off the pre-column (.5meter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

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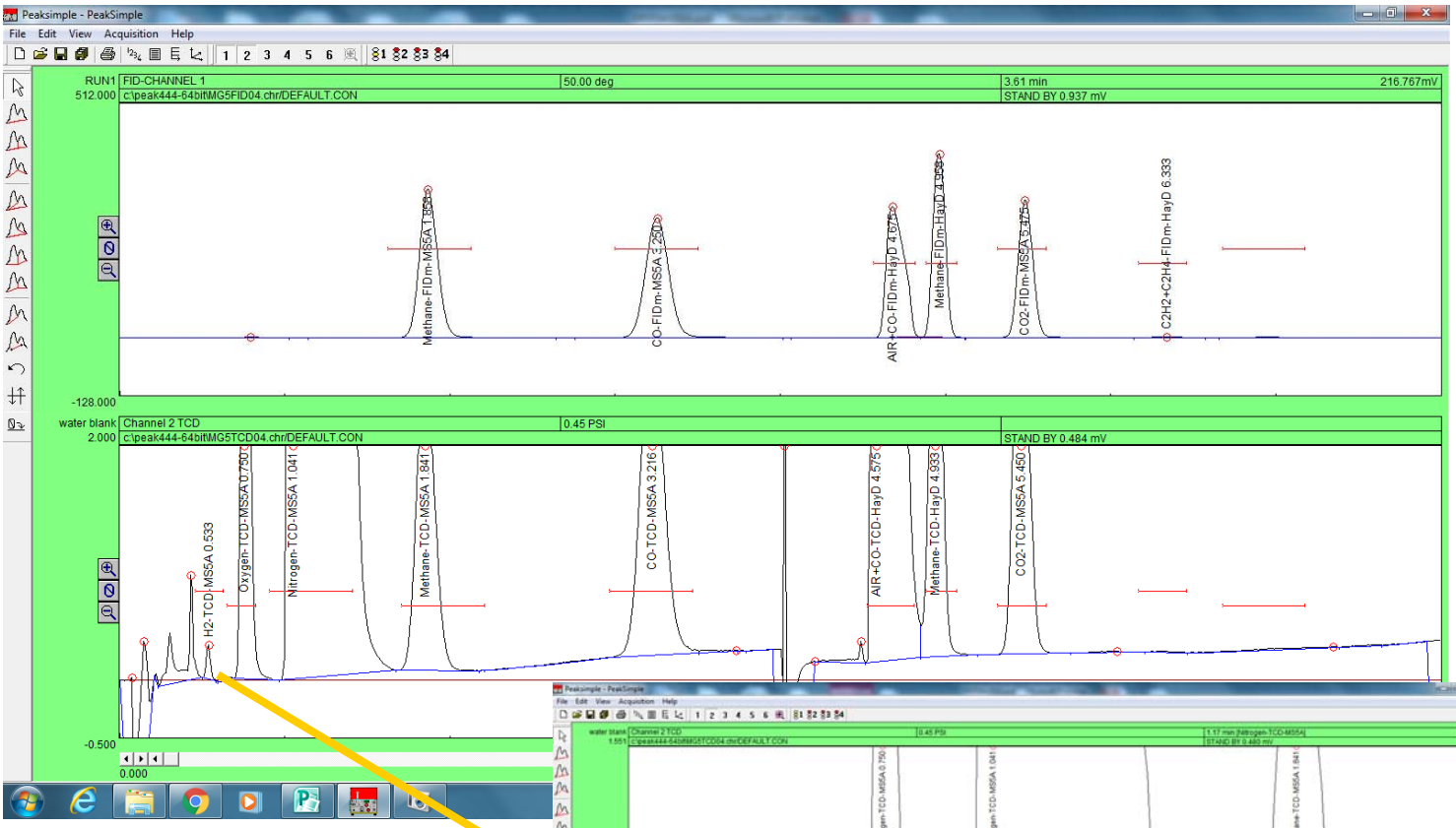

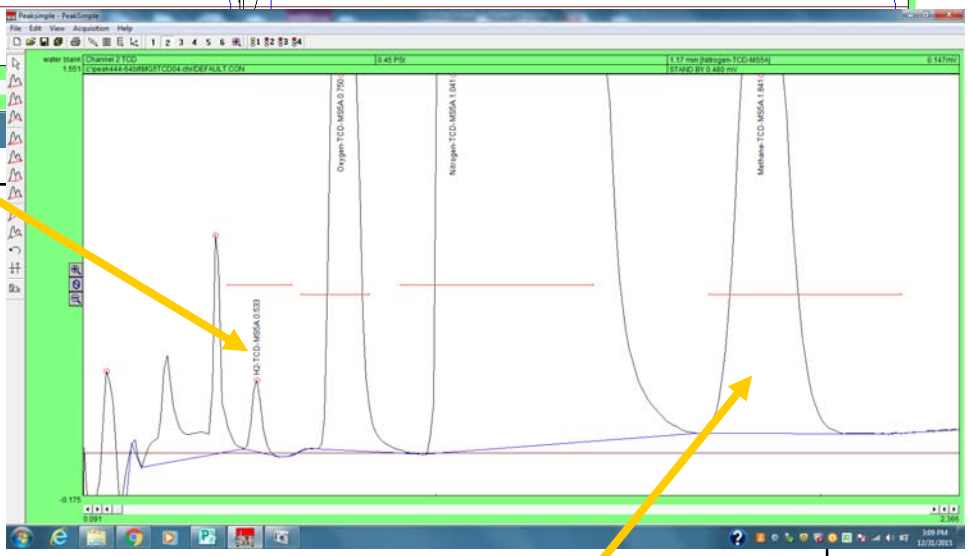

Here we show what 1% hydrogen looks like with helium carrier gas at 15psi and the same temperature program and event table.

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

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

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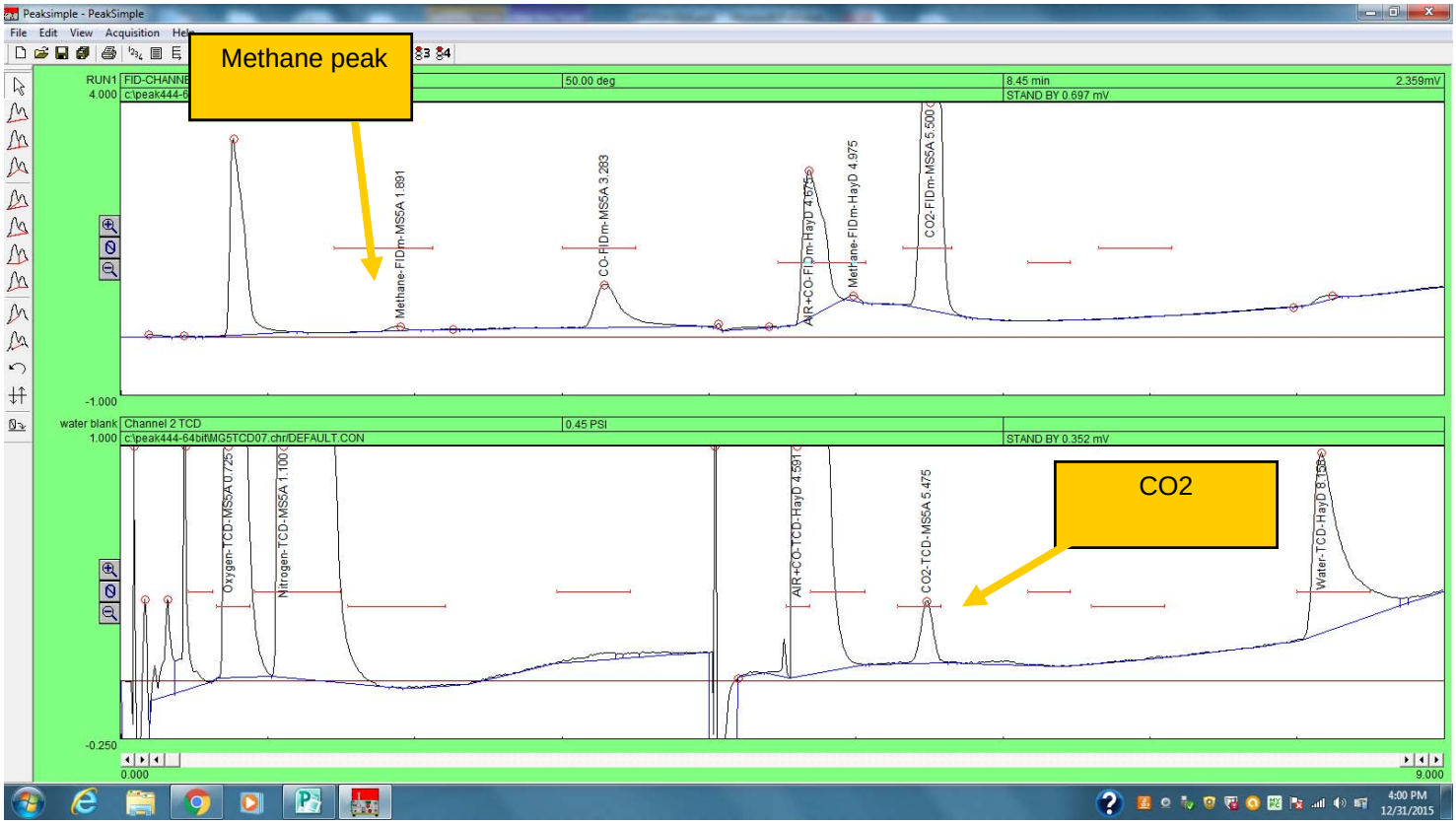
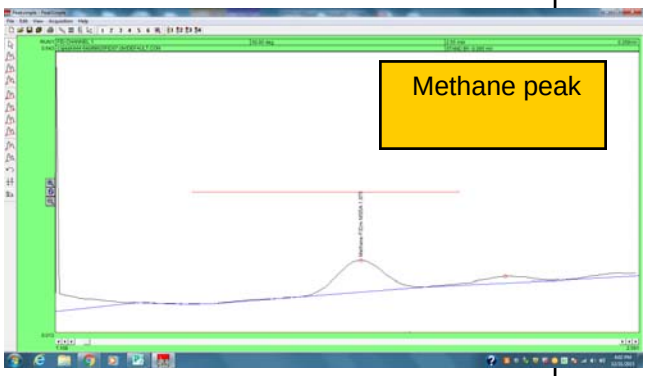


This is room air which has 2ppm of methane, 400ppm of CO2 and 10,000ppm of water.



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.

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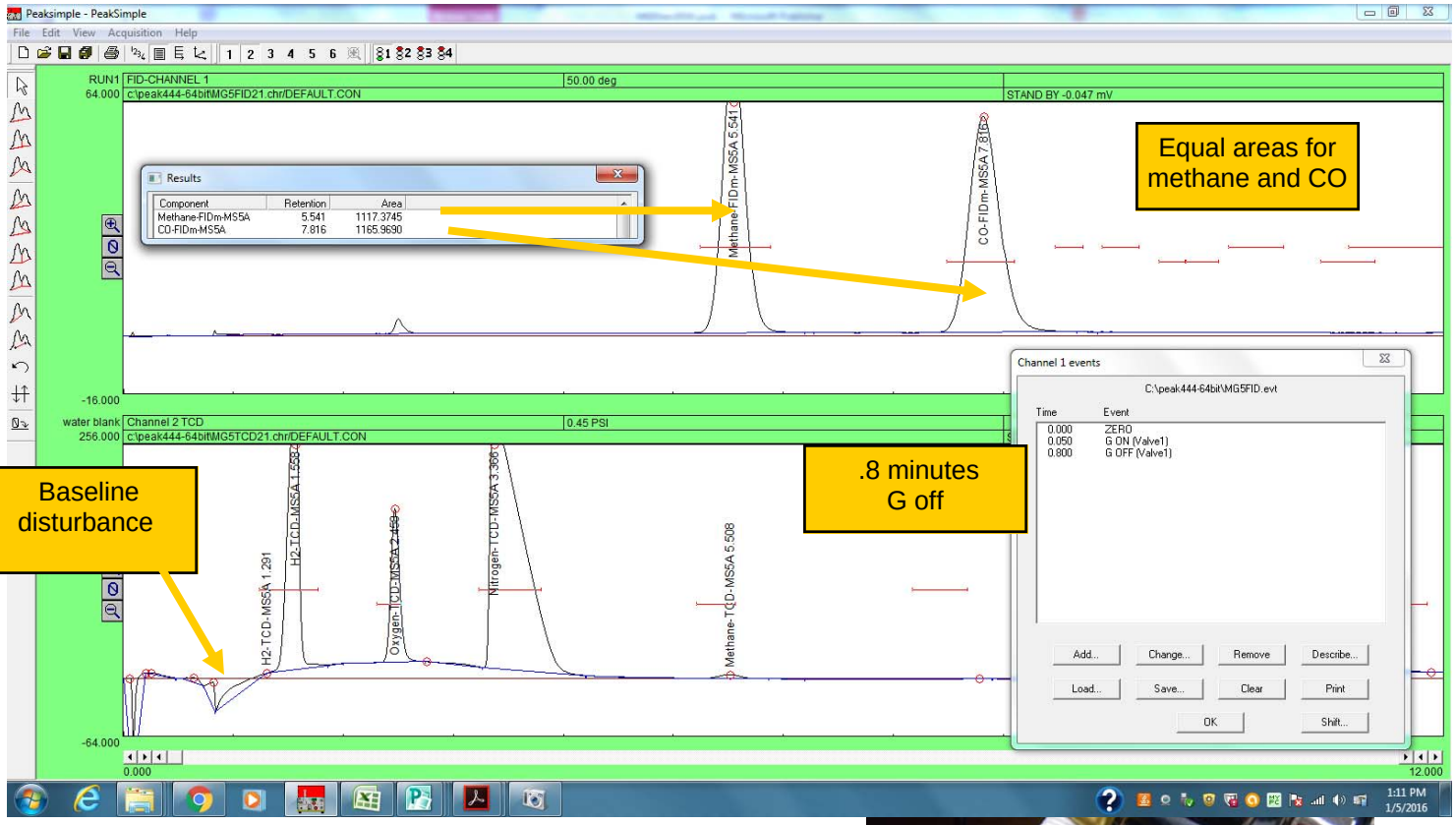

If hydrogen measurement is a priority its is best to use Argon carrier gas instead of helium. Because there is an un-avoidable baseline disturbance when Relay G turns off and backflushes the pre-column, it may make sense to use a 6 foot MS5A column instead of the 3 foot MS5A column. The 6' column makes 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

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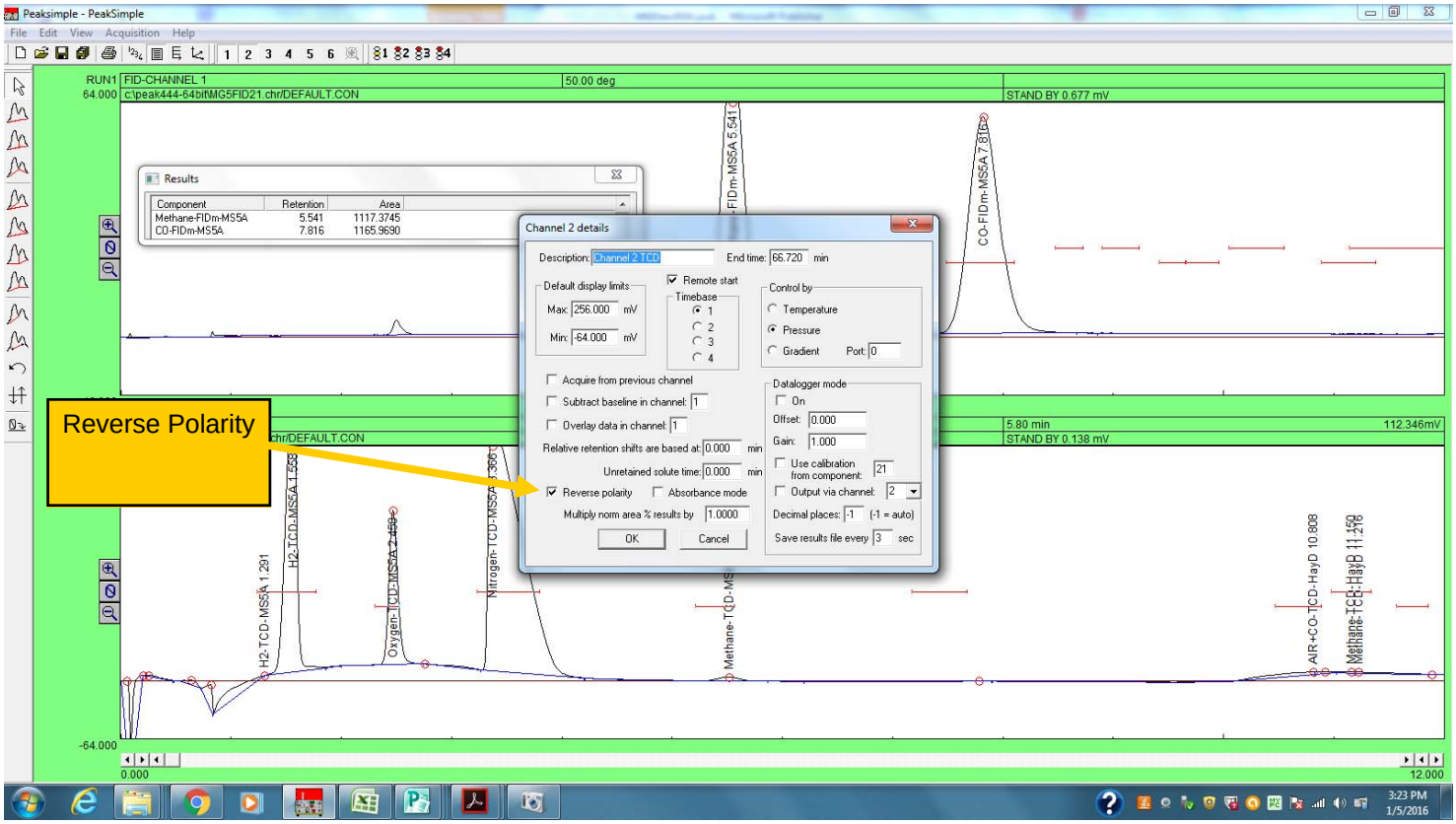

When using Argon or Nitrogen carrier gas the peaks come out upside down. In the channel 2 Details screen, click the box labelled “Reverse Polarity” so the peaks will come out in the positive direction.

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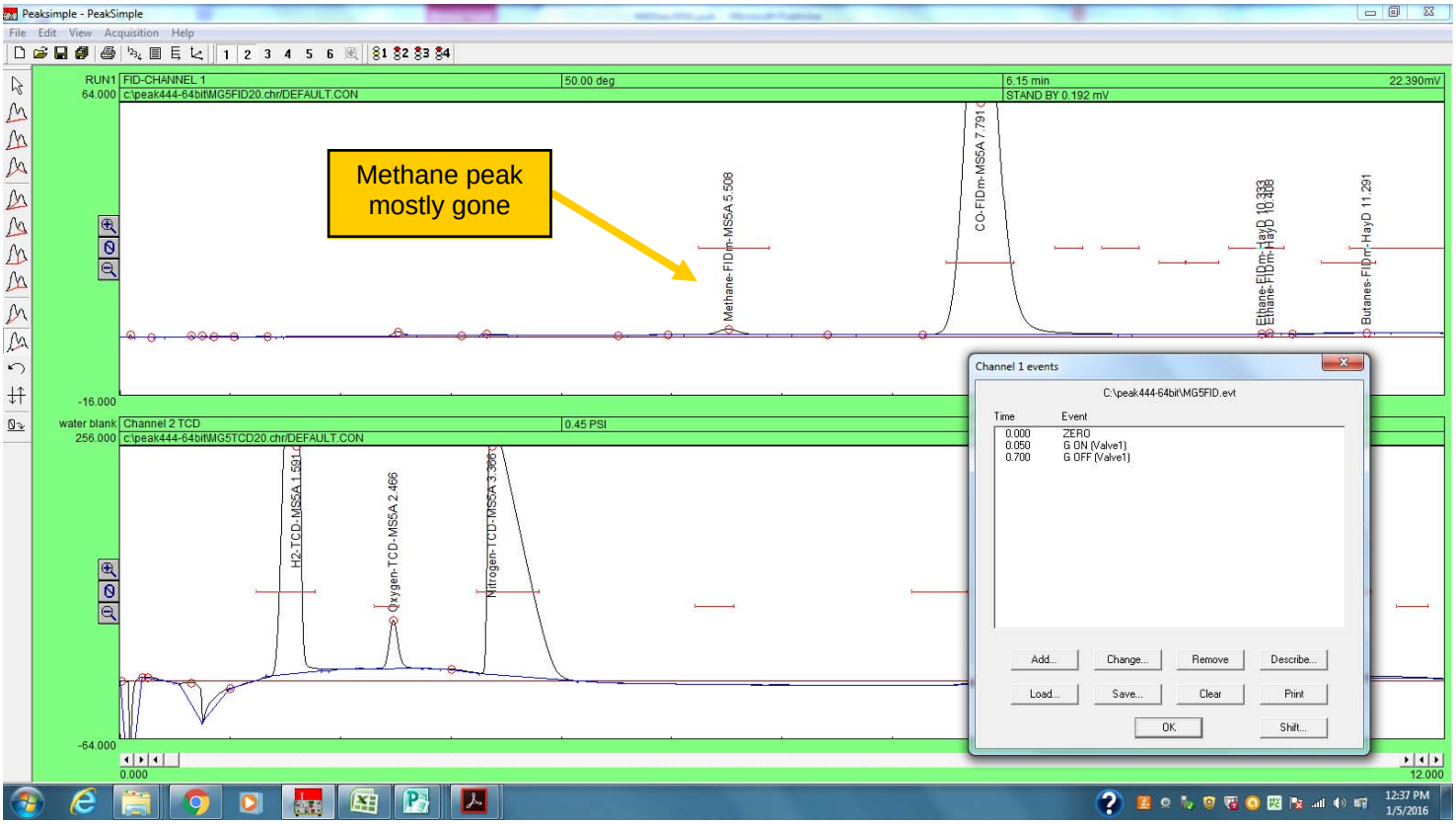

The exact time for Relay G off (backflush pre-column) will be different using Argon vs Helium. By trial and error move the Relay G off time earlier and earlier until you see the methane peak disappear.
Compare this analysis to the same analysis on page 13.
Both methane and CO are present in the sample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

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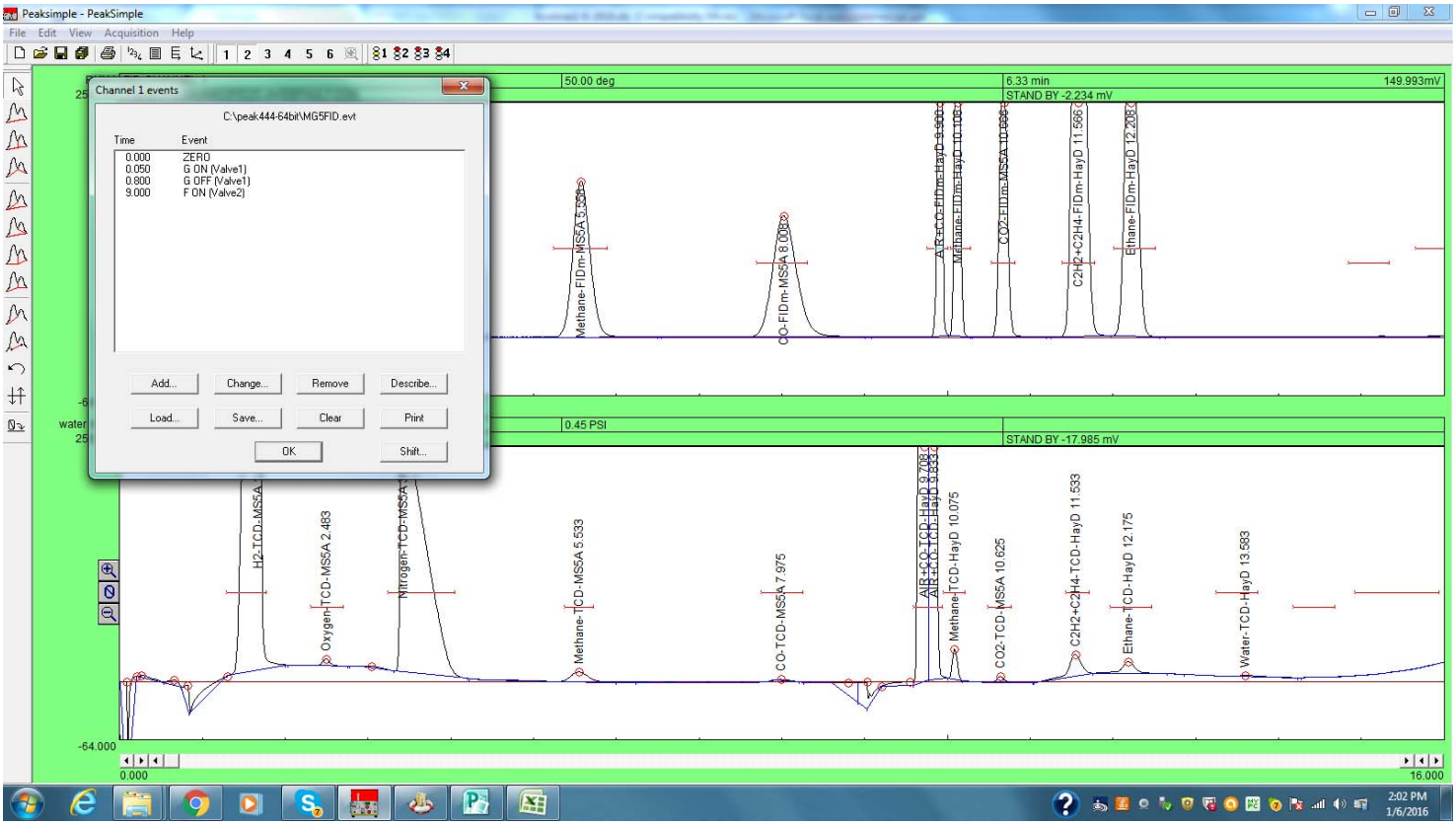

The chromatogram above shows the full analysis including the C2 peaks. Note that the Relay F on time (inject onto the Haysep dolumn) had to be delayed until 9 minutes to allow the CO to elute from the 6'MS5A column before making the injection onto the Haysep column.

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