

SRI H2-100 H2



## SRI Multi Gas Analyzer 2016



## SRI MultiGas Analysers

SRI DataSystem #302 6#

PeakSimple Tutorials

PS OP HINTS

GC APPS > Details

MG#5 OP Hints

Previous 2019 Gas Analysis

## GC Troubleshooting



# SRI Model H2-100 Hydrogen Generator



Make GC quality hydrogen from distilled water.

100ml/minute flow 50 psi outlet pressure

22" x 14" x 20" Shipping weight 52 lbs.

115 or 230VAC power 100 watts

Quiet operation

One Year warranty

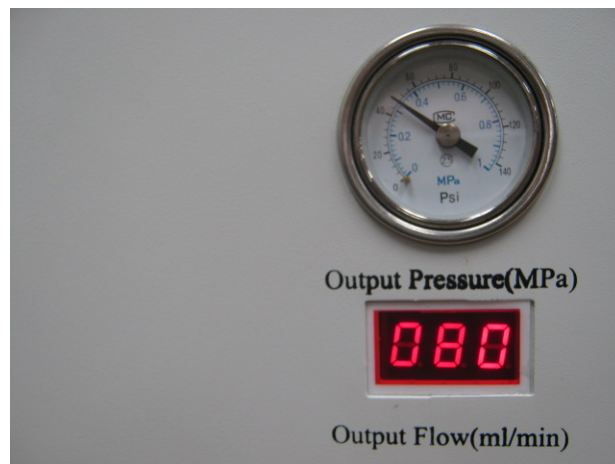
The SRI Model H2-100 is a perfect solution for labs which prefer not to have hydrogen cylinders. The H2-100 makes up to 100ml/minute of GC quality hydrogen from distilled water.

The outlet pressure is set to 50 psi but can be adjusted down if required. A quality 1/8" Swagelok style stainless steel outlet fitting makes connection to your GC easy, and a quick shut-off toggle valve provides instant on/off control.

A digital flow display shows H2 usage, and the handy pressure gage confirm proper operation.

## To order:

SRI part# 2014-H100 specify voltage ( 115/230 )  
100ml/minute hydrogen generator  
US\$ 3025. May 2014



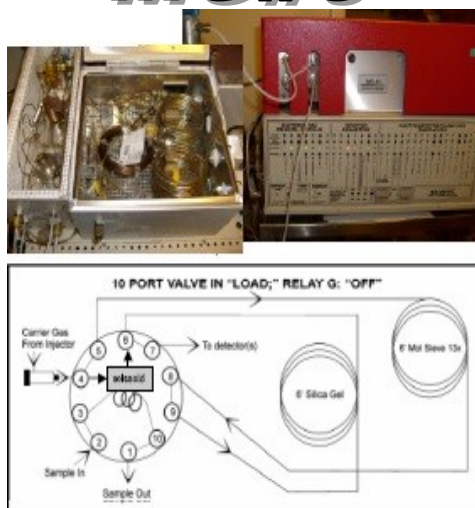
H2100FlyerMay2014



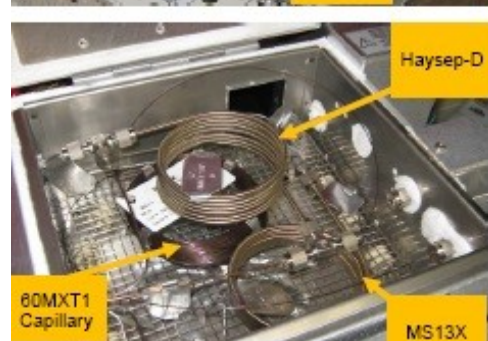
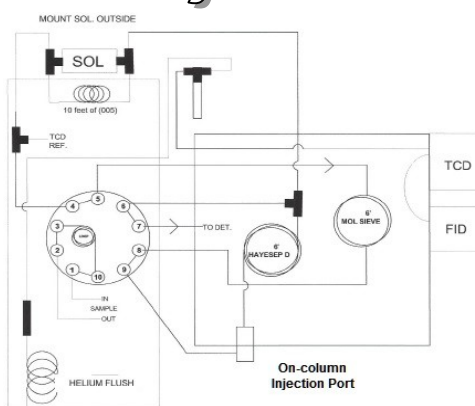
## CTR1 dual Column



# MG#3



# MG#3 + Injn Port



# MG#5



- 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:

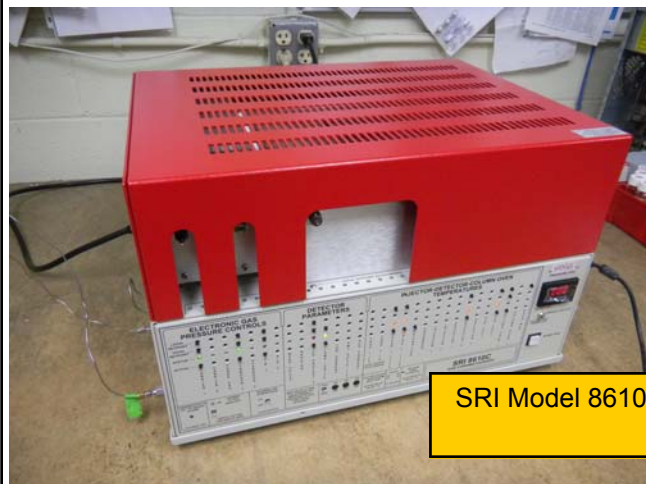
Hydrogen  
Oxygen  
Nitrogen  
Methane  
CO  
CO<sub>2</sub>  
Ethane  
Water  
Propane  
Butane  
Pentane

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.

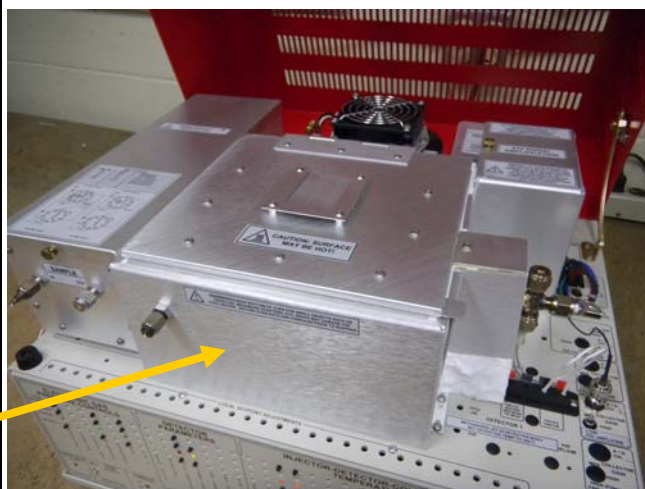
Like the earlier MG GCs the 8610C chassis includes an ambient to 400C programmable column oven.

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



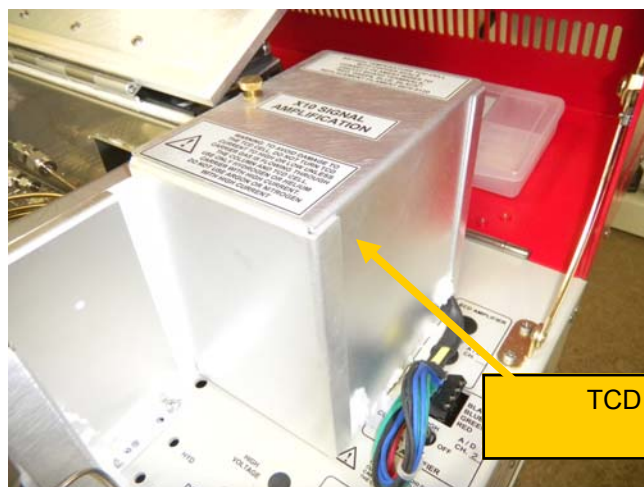


## Multiple Gas#5 GC configuration Jan 2016

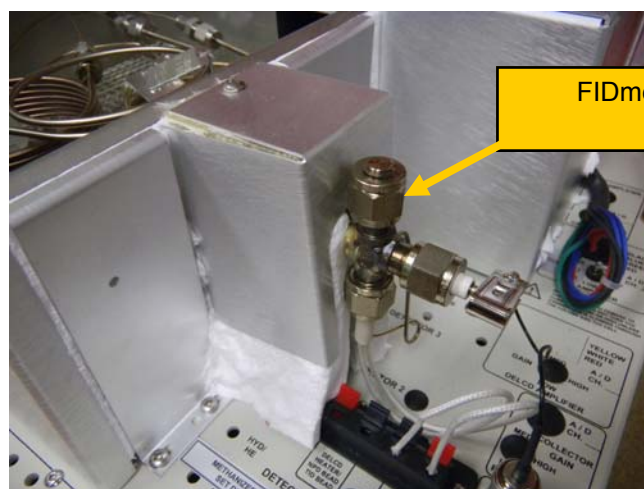
On the right side of the column oven is located the Thermal Conductivity Detector ( TCD ) which detects all the gases from 200ppm to 100% except hydrogen ( see detailed explanation for this later ).

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 CO<sub>2</sub> from 1ppm to 50,000ppm. The FID can only detect hydrocarbons like methane and ethane, but when equipped with a methanizer, CO and CO<sub>2</sub> are reacted to methane and thus detected at the same sensitivity as methane.

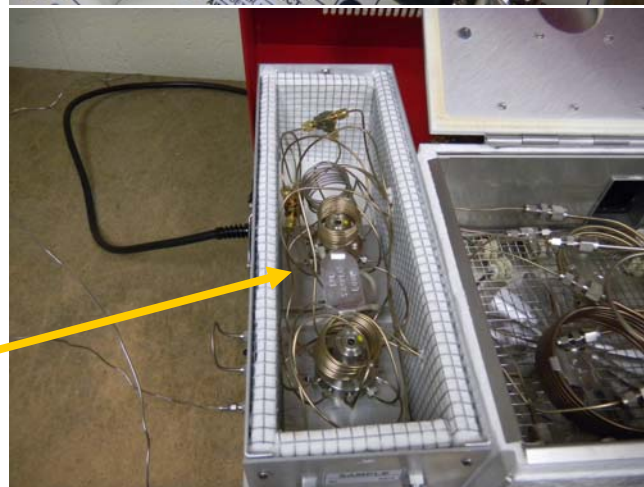
On the left side of the column oven is the valve oven, which contains two 10port Valco valves and lots of 1/16" stainless steel tubing.



TCD



FIDmeth

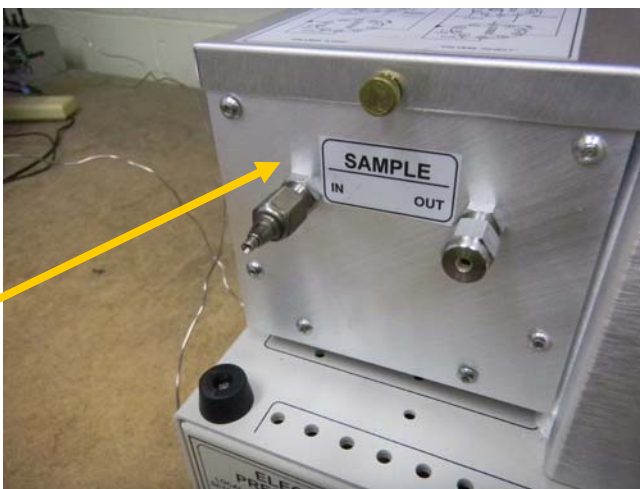
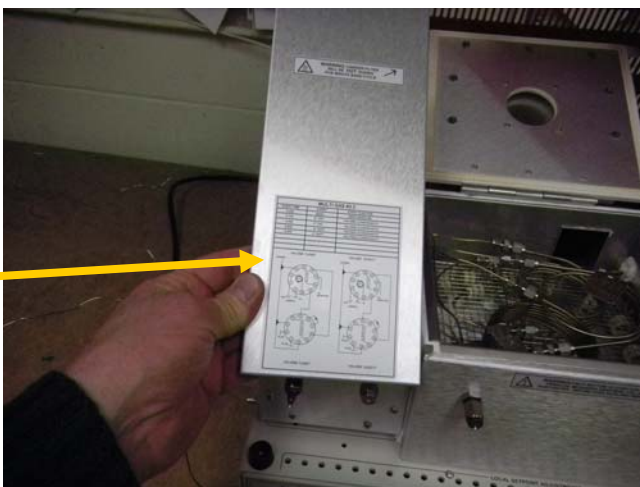


## Multiple Gas#5 GC configuration Jan 2016

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.

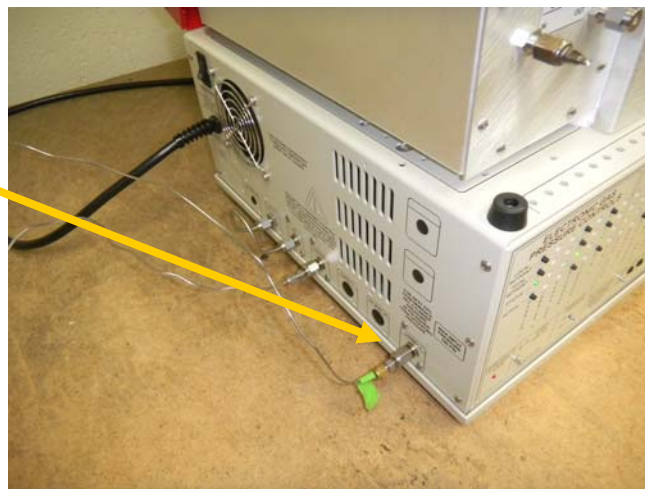
Helium is the most common choice because it gives the best overall results. However the sensitivity is not as good for hydrogen as it is for the other gases. This is because the TCD sensitivity depends on the difference of the “thermal conductivity” of the carrier gas relative to the sample molecule.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

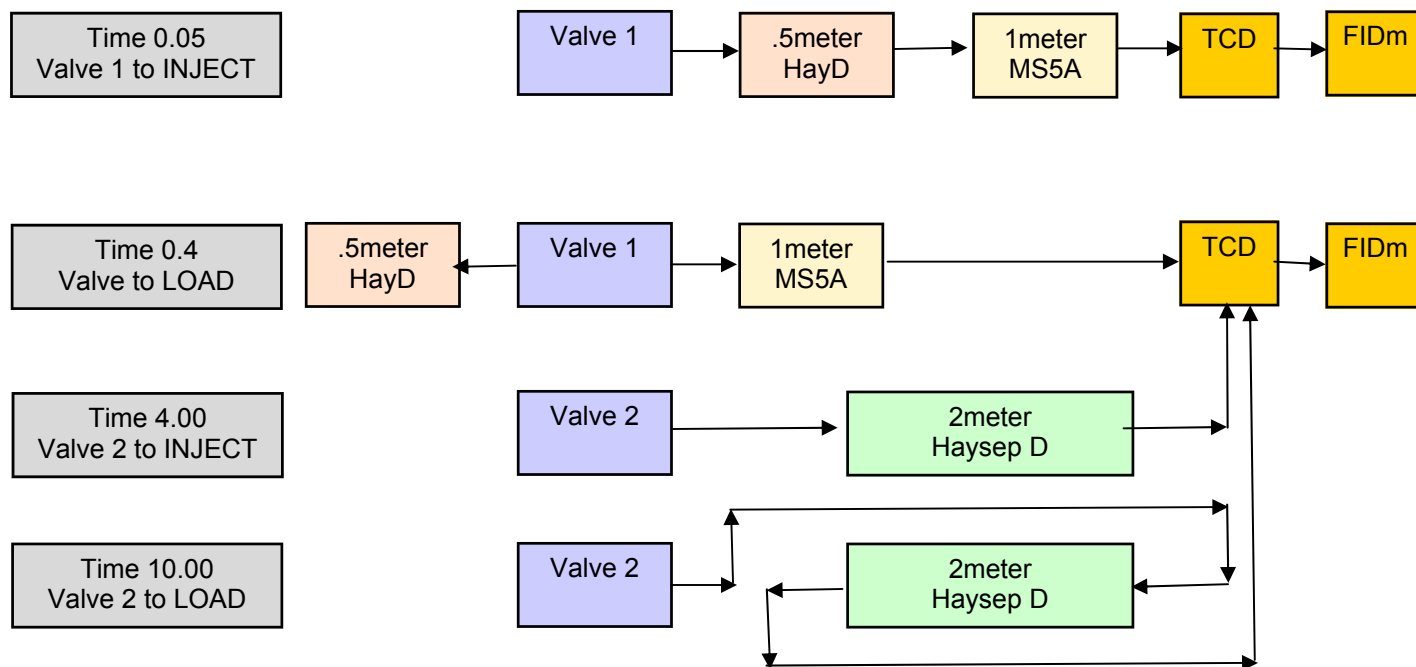
Argon is used where it is important to measure hydrogen and also oxygen and nitrogen.

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 .5meter Haysep D column. H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub> 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 H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub> 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 CO<sub>2</sub>, Water, and C<sub>2</sub> 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.

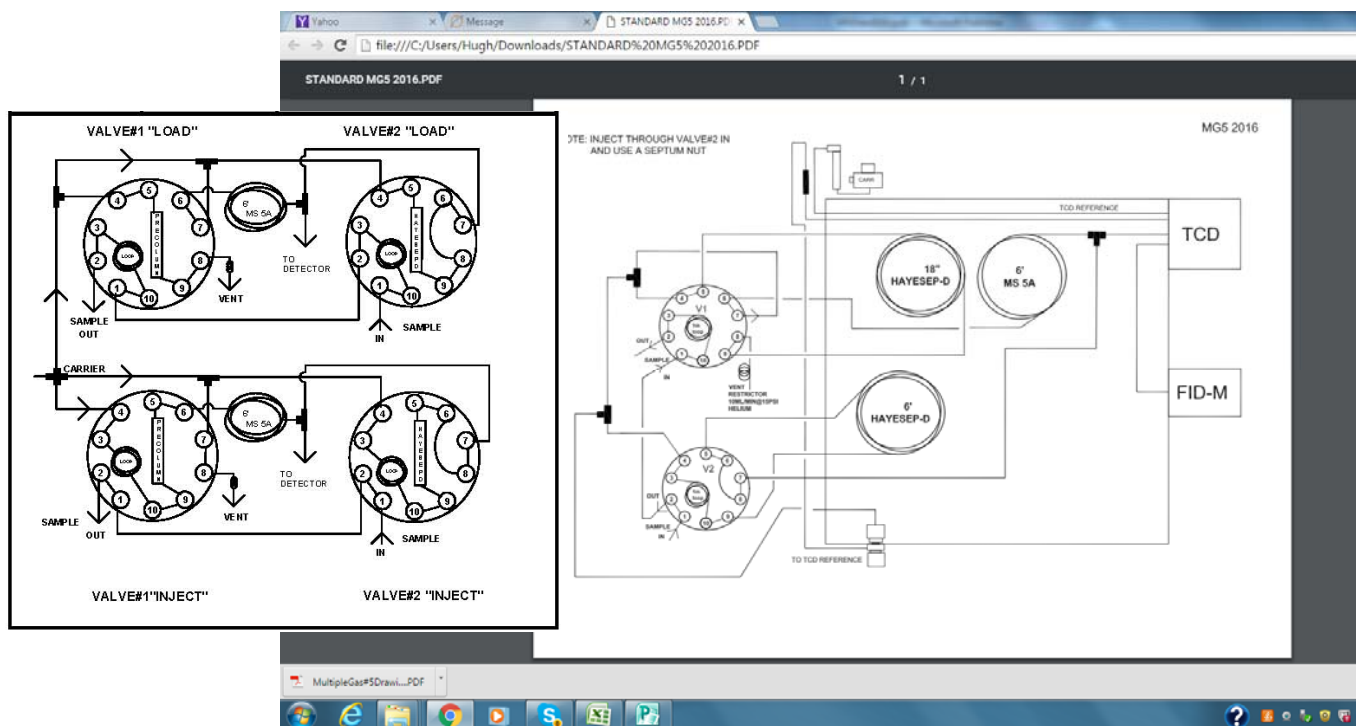
**STEP 3:** Valve2 is turned to the INJECT position ( Relay F on ). The carrier gas pushes the molecules in the loop of Valve2 onto the 2meter HayD column in the "forward" direction. H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub> and CO elute from the column very quickly as one peak, followed by the CH<sub>4</sub> peak, the CO<sub>2</sub>, water and the hydrocarbons from C<sub>2</sub>-C<sub>6</sub>.

**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 CO<sub>2</sub> ( or you did not care about any peak after CO<sub>2</sub> ), then you would backflush after the CO<sub>2</sub> peak. Any peaks remaining in the HayD column would come out in a "lump".

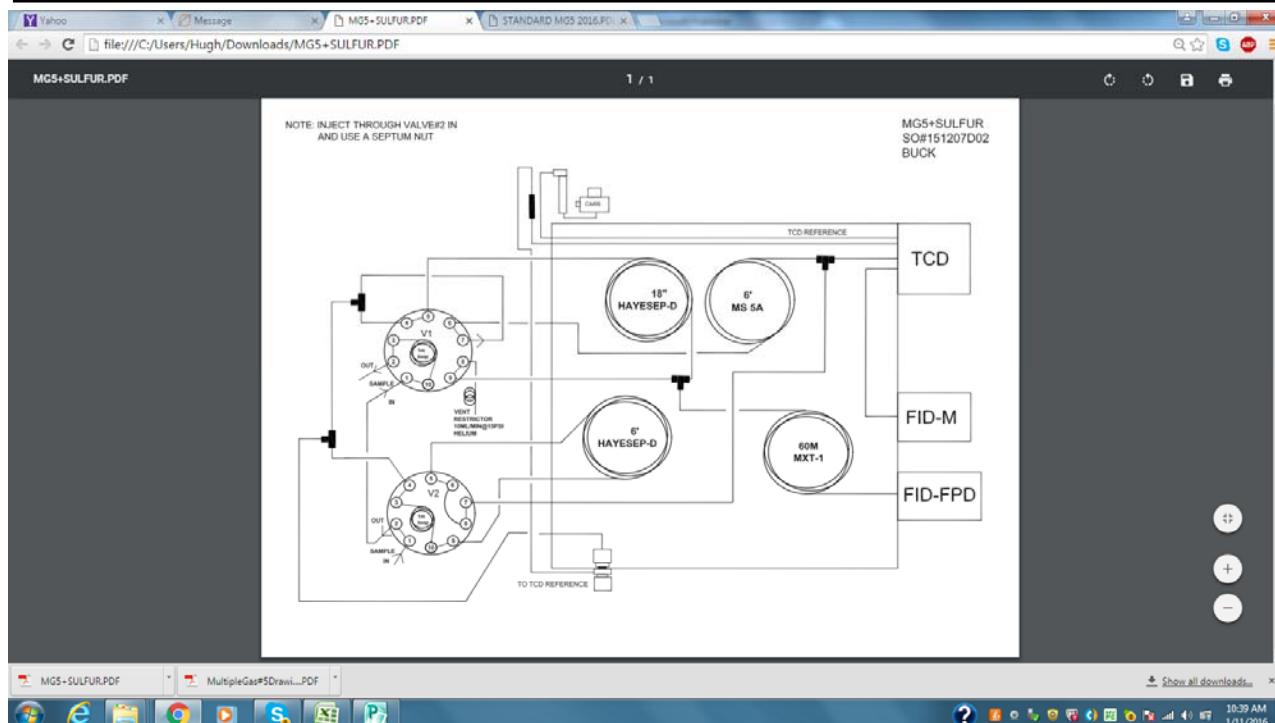


# Multiple Gas#5 GC configuration

## Jan 2016

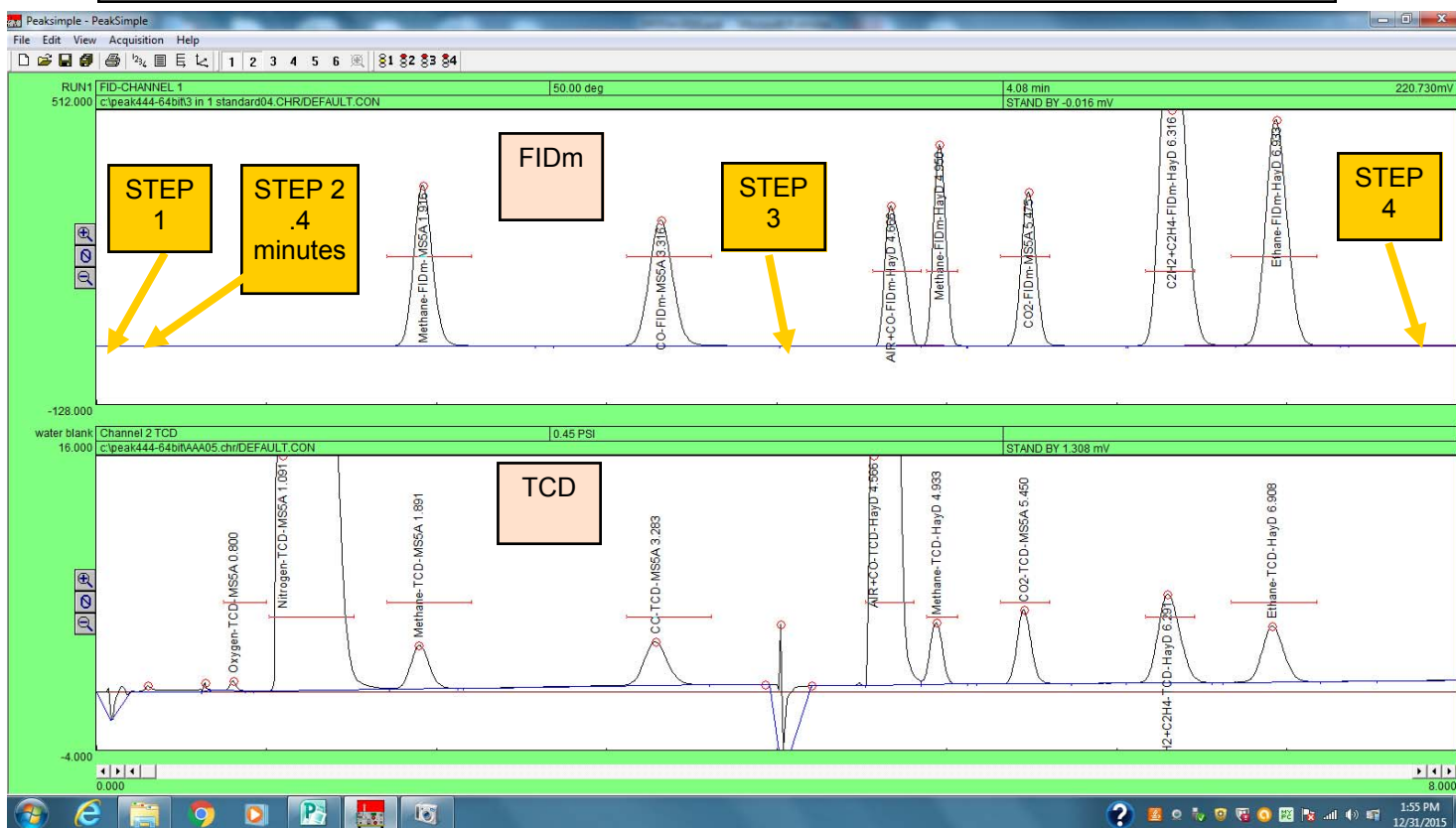


The diagram above shows a schematic of the "basic" MG#5 configuration with both valves in the INJECT position. A similar diagram below shows the "basic" MG#5 plus an additional column and FPD/FID detector to measure sulfur gases like H<sub>2</sub>S, CO<sub>2</sub>, SO<sub>2</sub>, mercaptans, DMS etc.



# 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. H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub> CH<sub>4</sub> 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 H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub> 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 CO<sub>2</sub>, Water, and C<sub>2</sub> 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. H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub> and CO elute from the column very quickly as one peak, followed by the CH<sub>4</sub> peak, the CO<sub>2</sub>, Water and the hydrocarbons from C<sub>2</sub>-C<sub>6</sub>.

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 CO<sub>2</sub> ( or you did not care about any peak after CO<sub>2</sub> ), then you would backflush after the CO<sub>2</sub> 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.

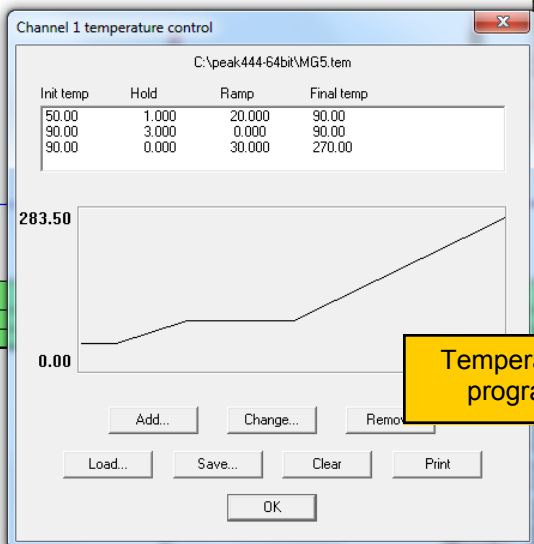
At time 0.05 Relay G turns on. This initiates STEP 1.

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 back-flushing 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 back-flush time ( STEP 4 ) especially may change depending on what molecules are in the sample.



The screenshot shows the 'Channel 1 events' window. It contains a table with the following data:

Time	Event
0.000	ZERO
0.050	G ON (Valve1)
0.400	G OFF (Valve1)
4.000	F ON (Valve2)
8.000	F OFF (Valve2)

A yellow callout box labeled 'Channel 1 Event table' points to the table.

# Multiple Gas#5 GC configuration

## Jan 2016



The sample above ( helium carrier at 15psi ) shows 1% each methane, CO, CO<sub>2</sub>, 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.

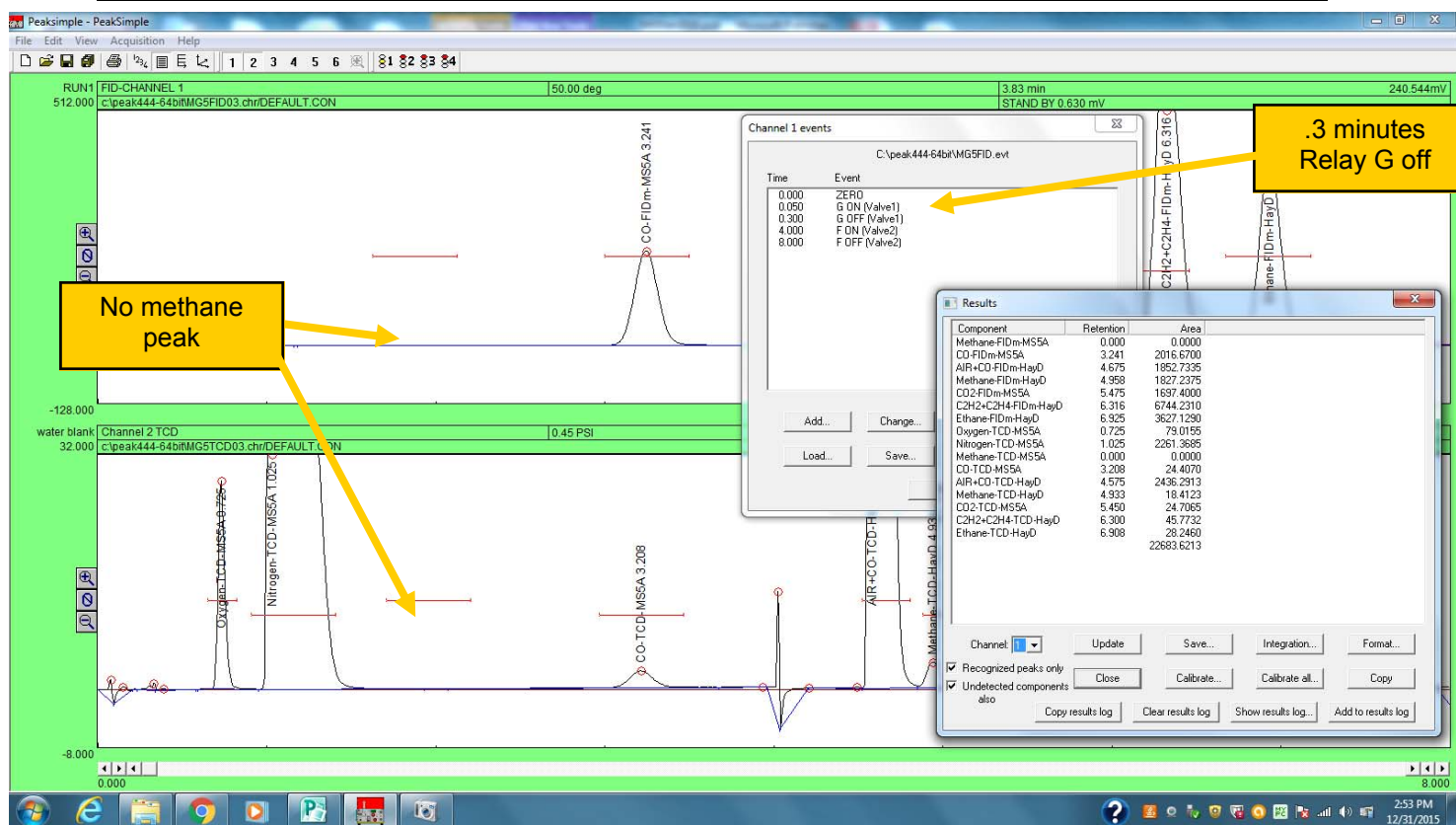
It also shows that the Valve 1 timing ( Relay G off at .4 minutes ) is correct.

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.



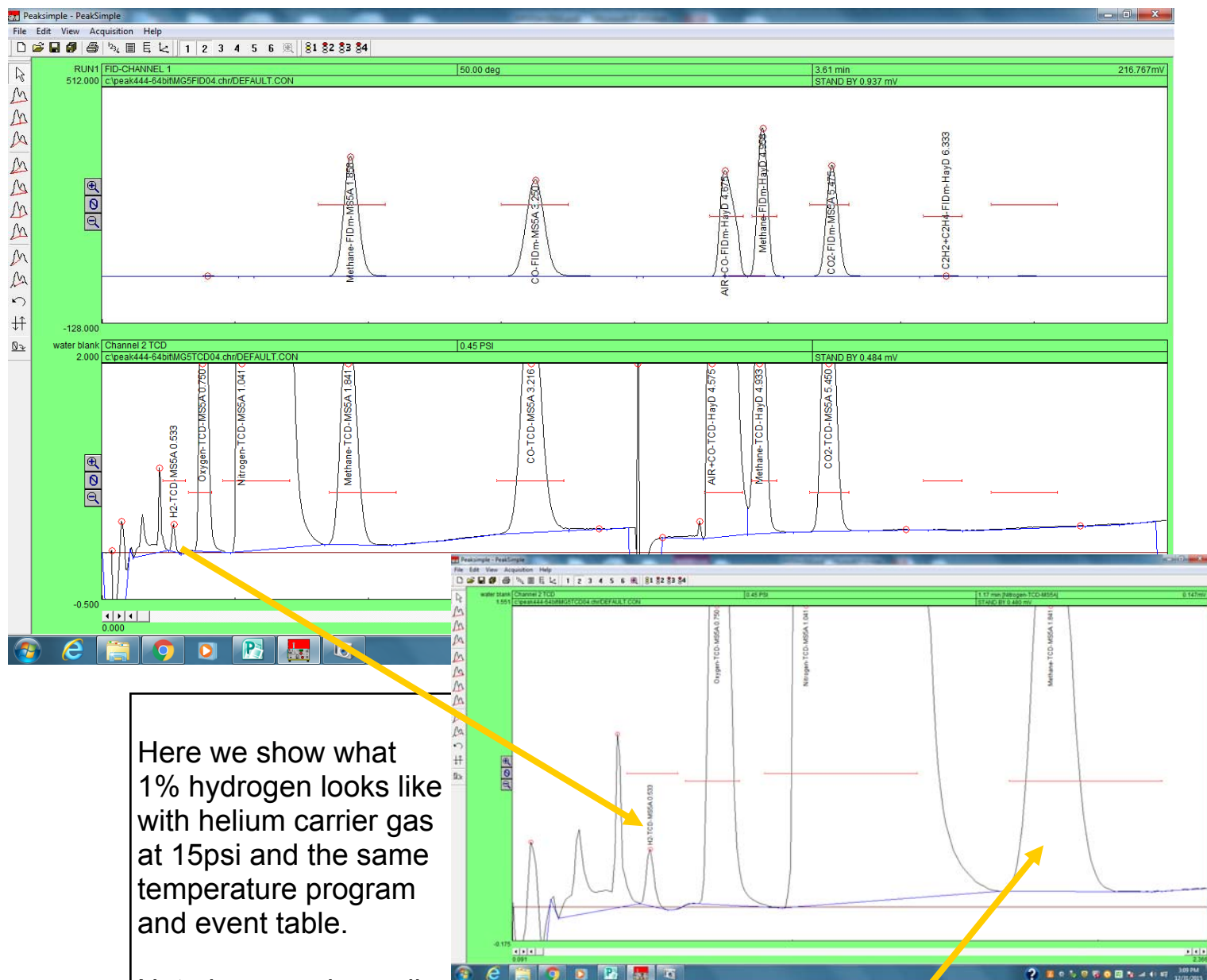
# Multiple Gas#5 GC configuration Jan 2016



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 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.

# Multiple Gas#5 GC configuration Jan 2016

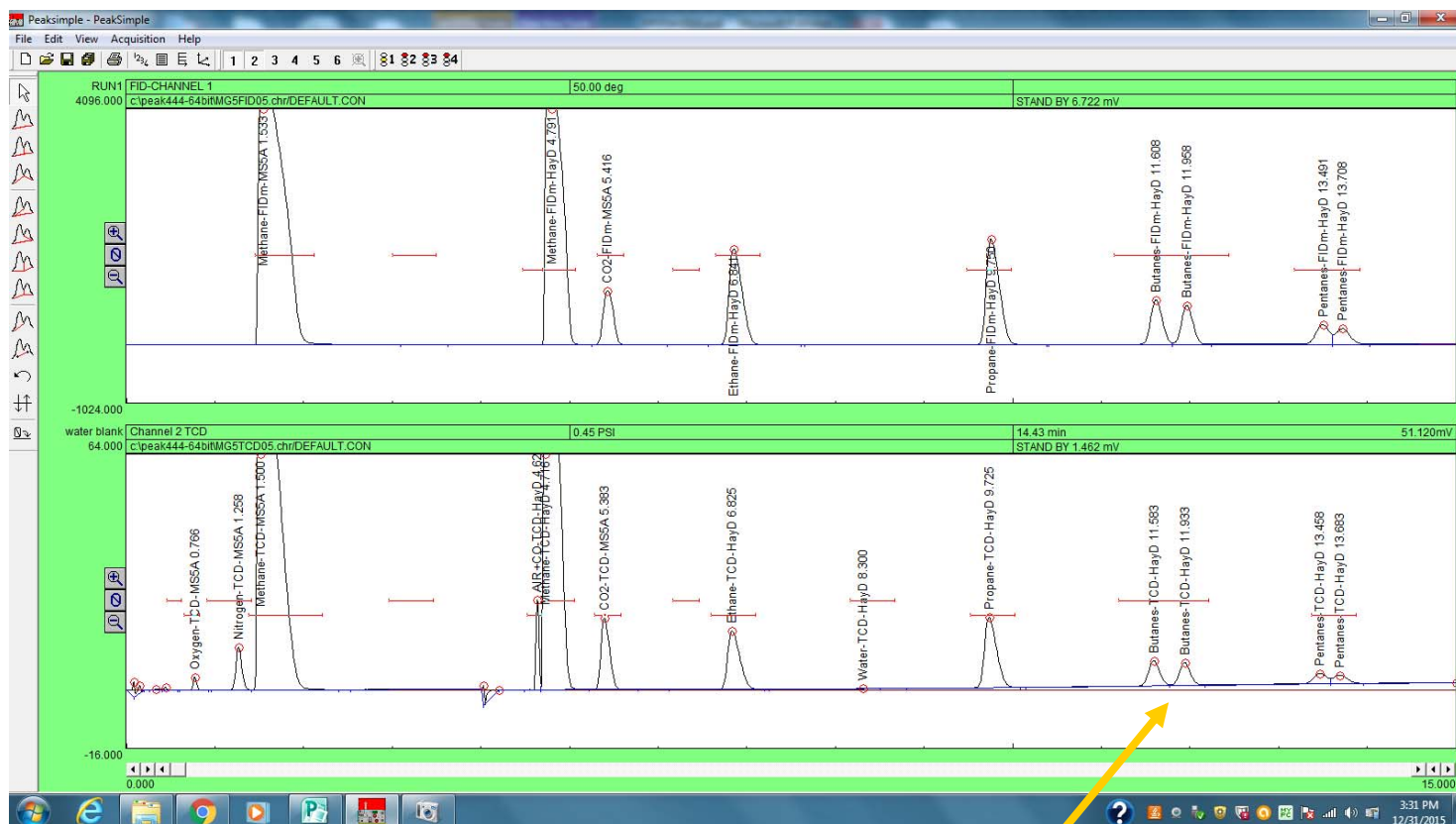


Here we show what 1% hydrogen looks like with helium carrier gas at 15psi and the same temperature program and event table.

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

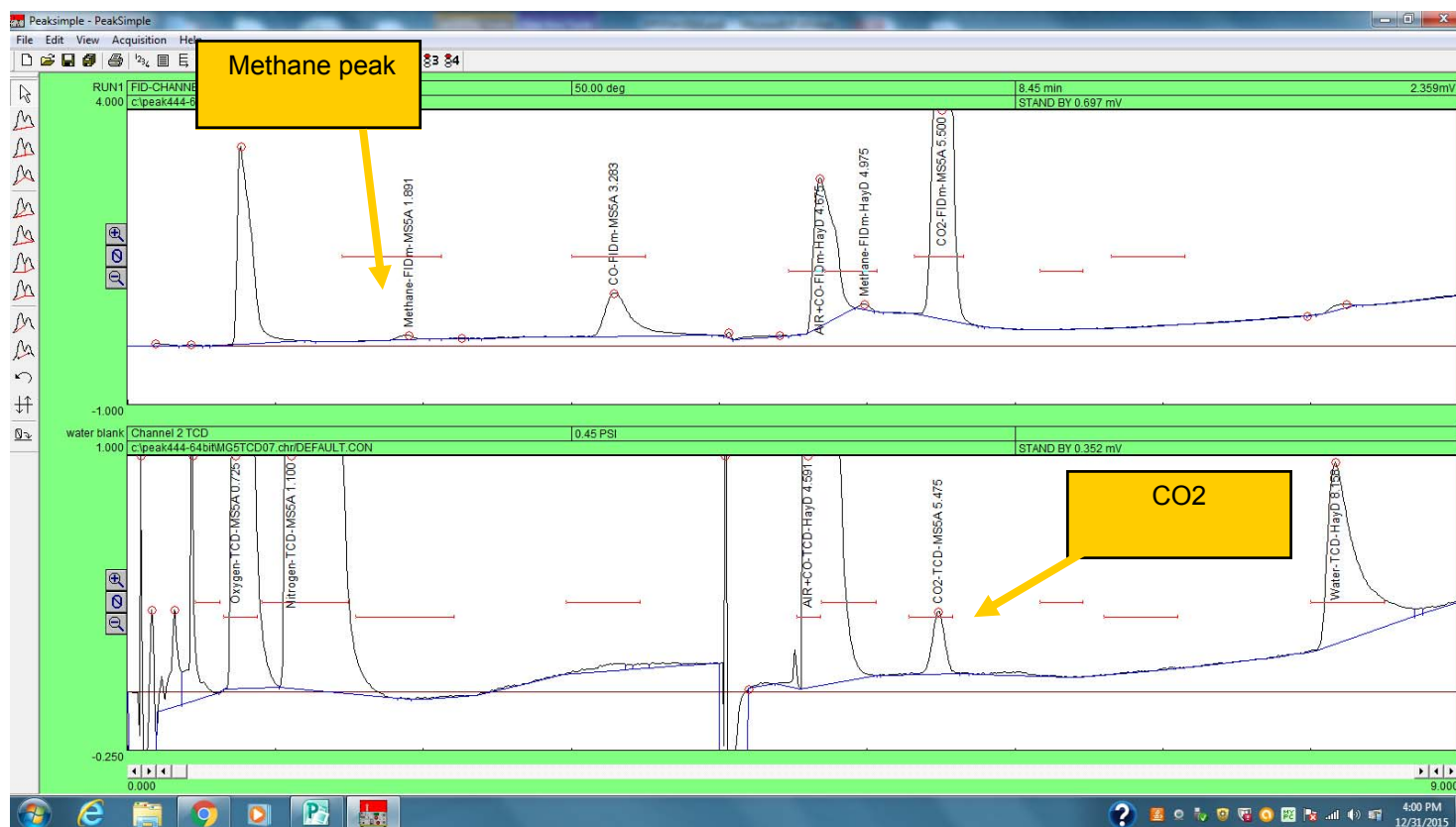


# Multiple Gas#5 GC configuration Jan 2016



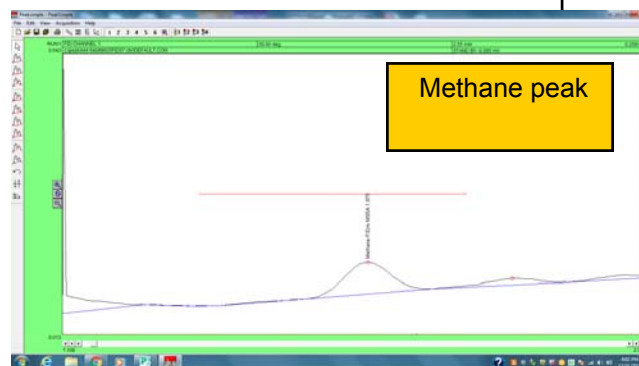
This is natural gas. Notice there is no CO, but plenty of butanes and pentanes. There is also a water peak on the TCD.

# Multiple Gas#5 GC configuration Jan 2016



This is room air which has 2ppm of methane, 400ppm of CO<sub>2</sub> and 10,000ppm of water.

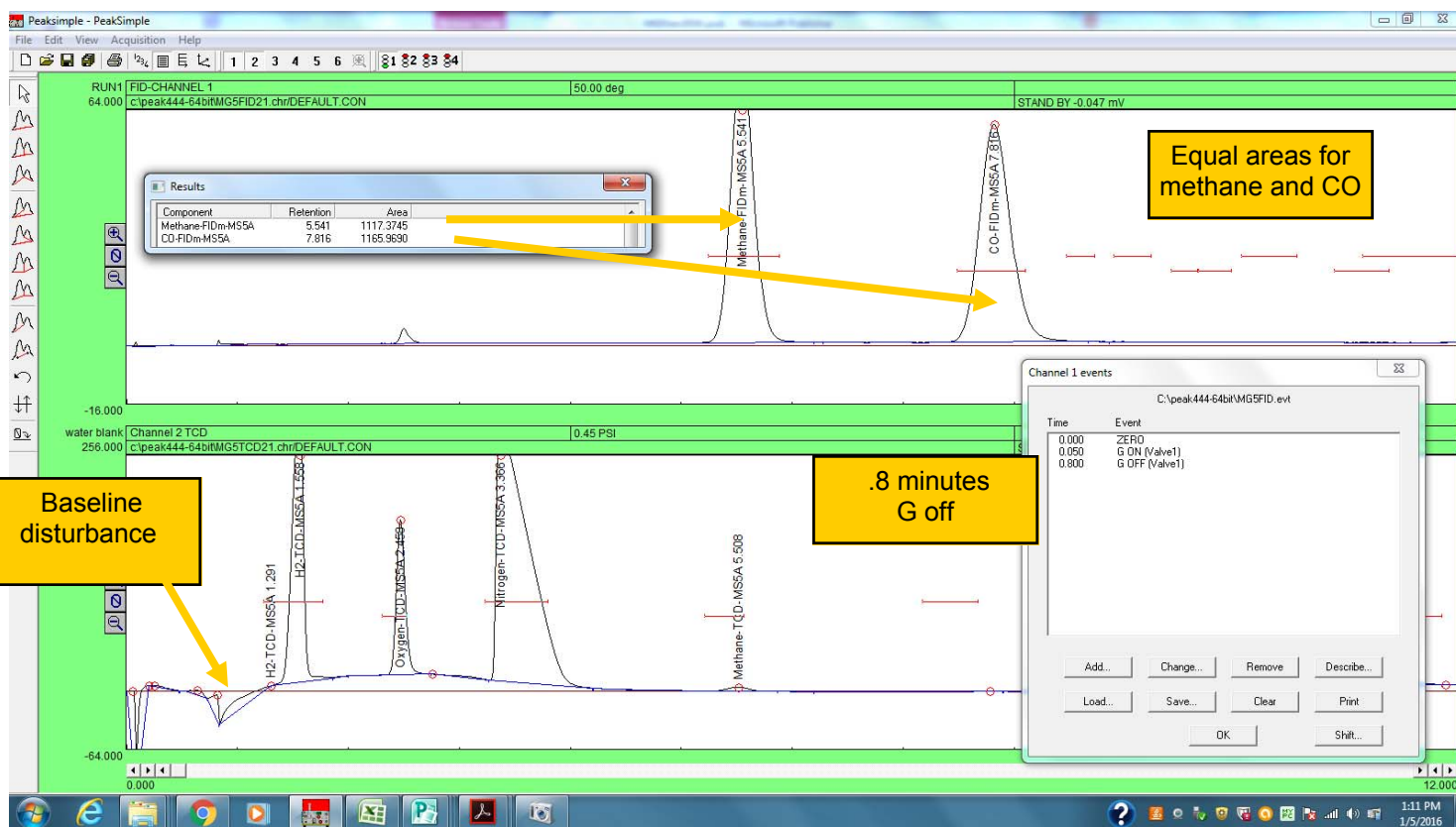
Notice the 2ppm methane peak is easily detectable on the FIDmethanizer, and the 400ppm CO<sub>2</sub> peak easily detectable on the TCD.



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



# Multiple Gas#5 GC configuration Jan 2016

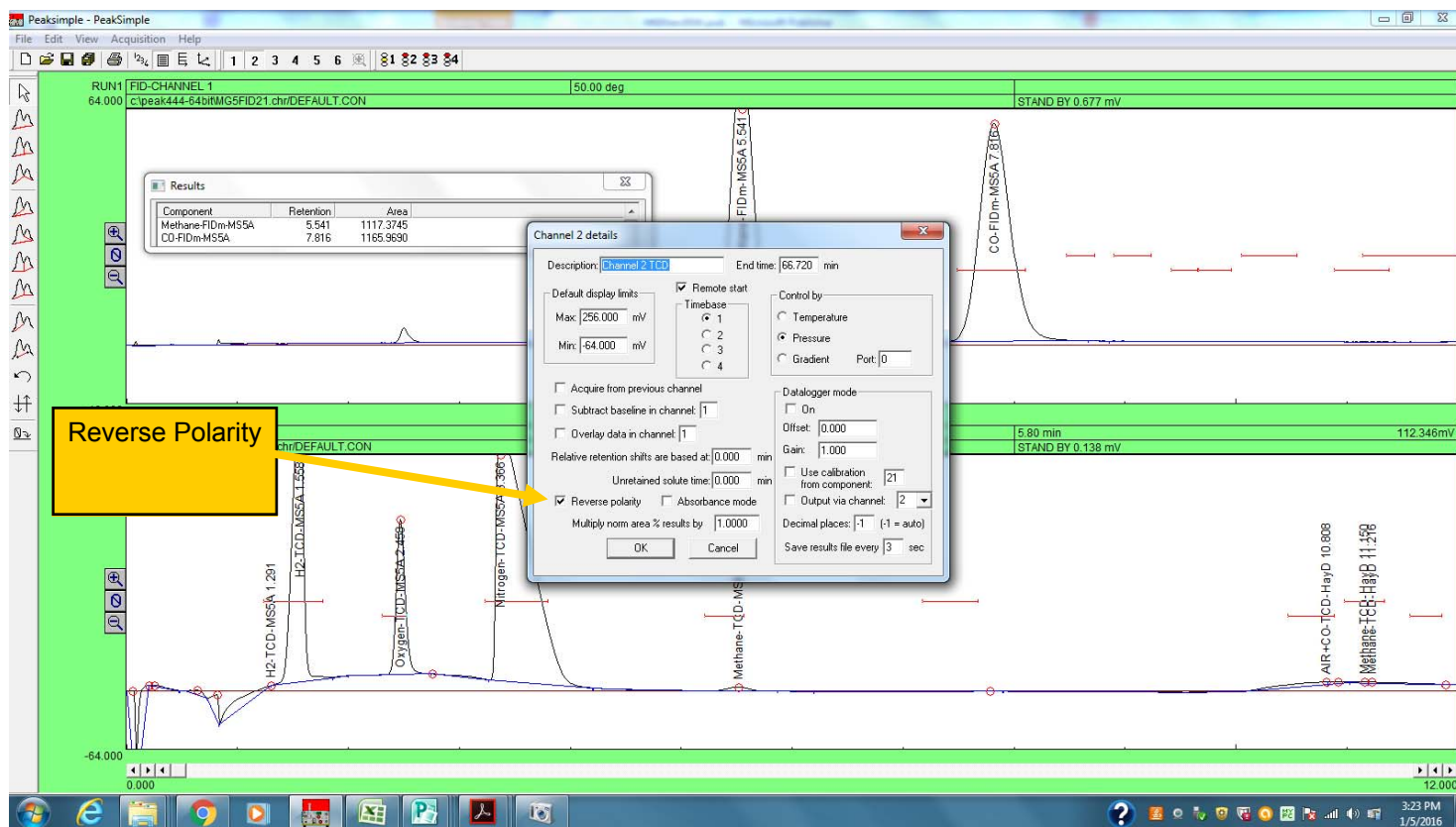


If hydrogen measurement is a priority its is best to use Argon carrier gas instead of helium. Because there is an un-avoidable baseline disturbance when Relay G turns off and backflushes the pre-column, it may make sense to use a 6 foot MS5A column instead of the 3 foot MS5A column. The 6' column makes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

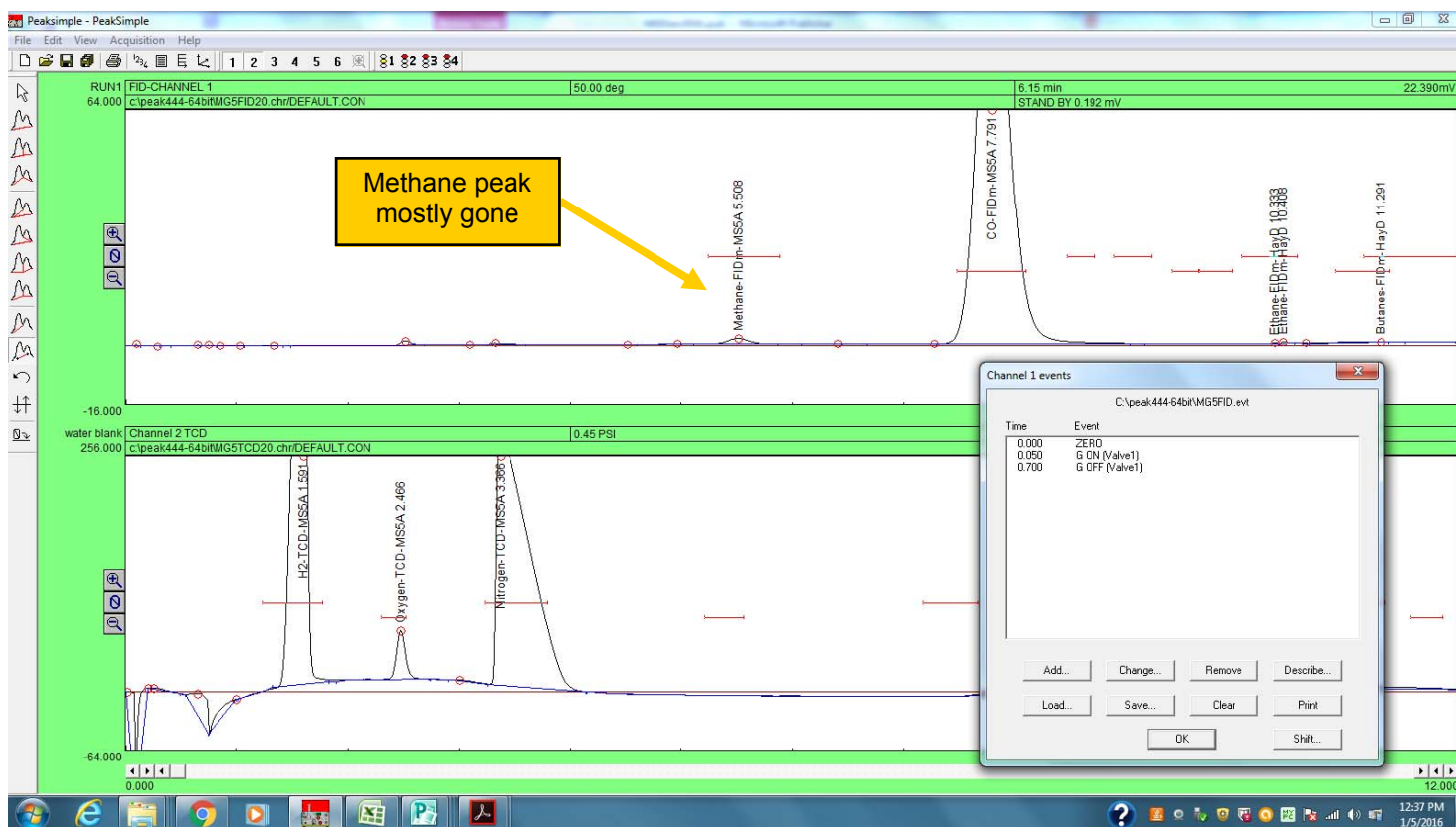
# Multiple Gas#5 GC configuration Jan 2016



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



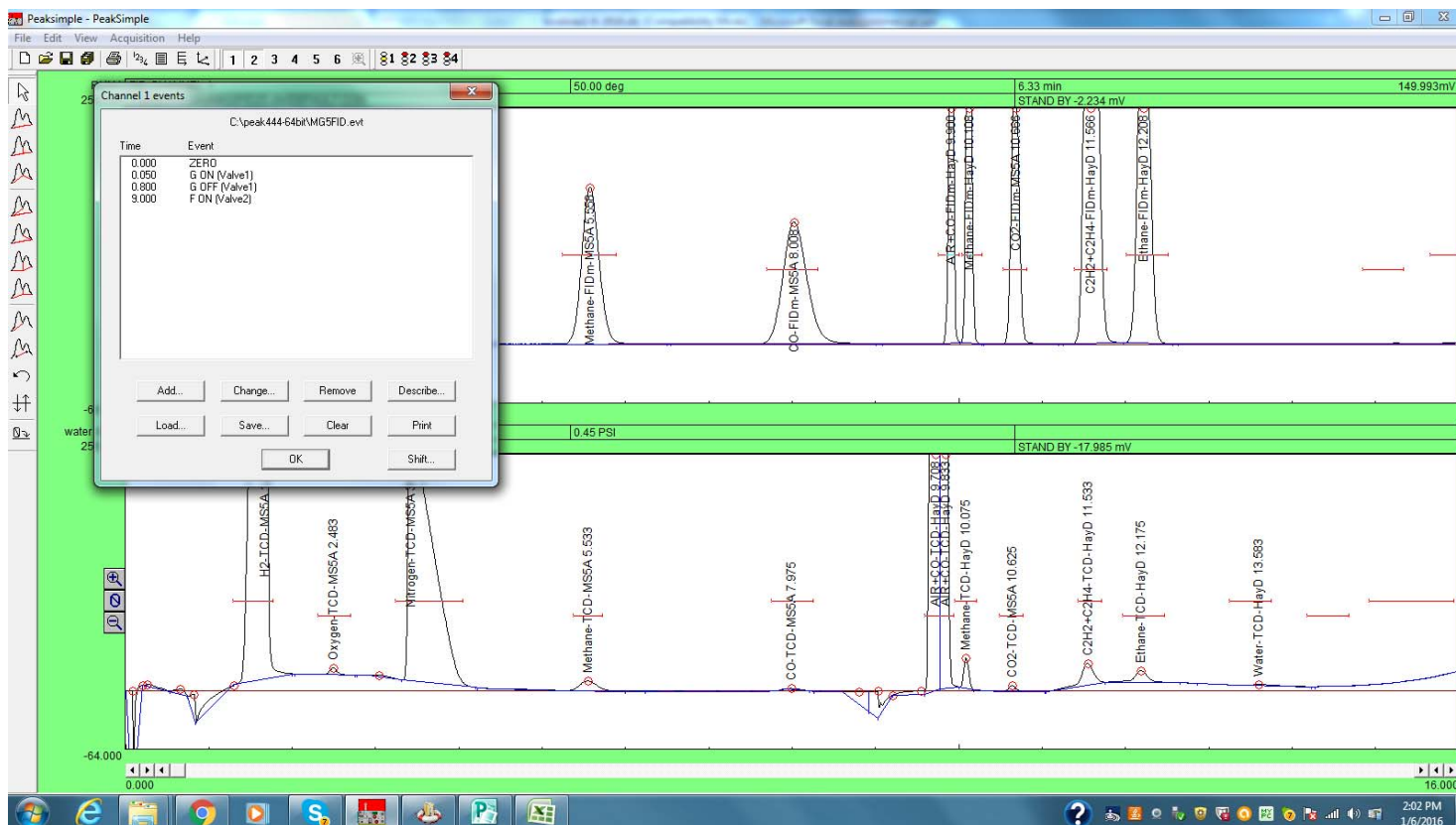
The exact time for Relay G off ( backflush pre-column ) will be different using Argon vs Helium. By trial and error move the Relay G off time earlier and earlier until you see the methane peak disappear.

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.

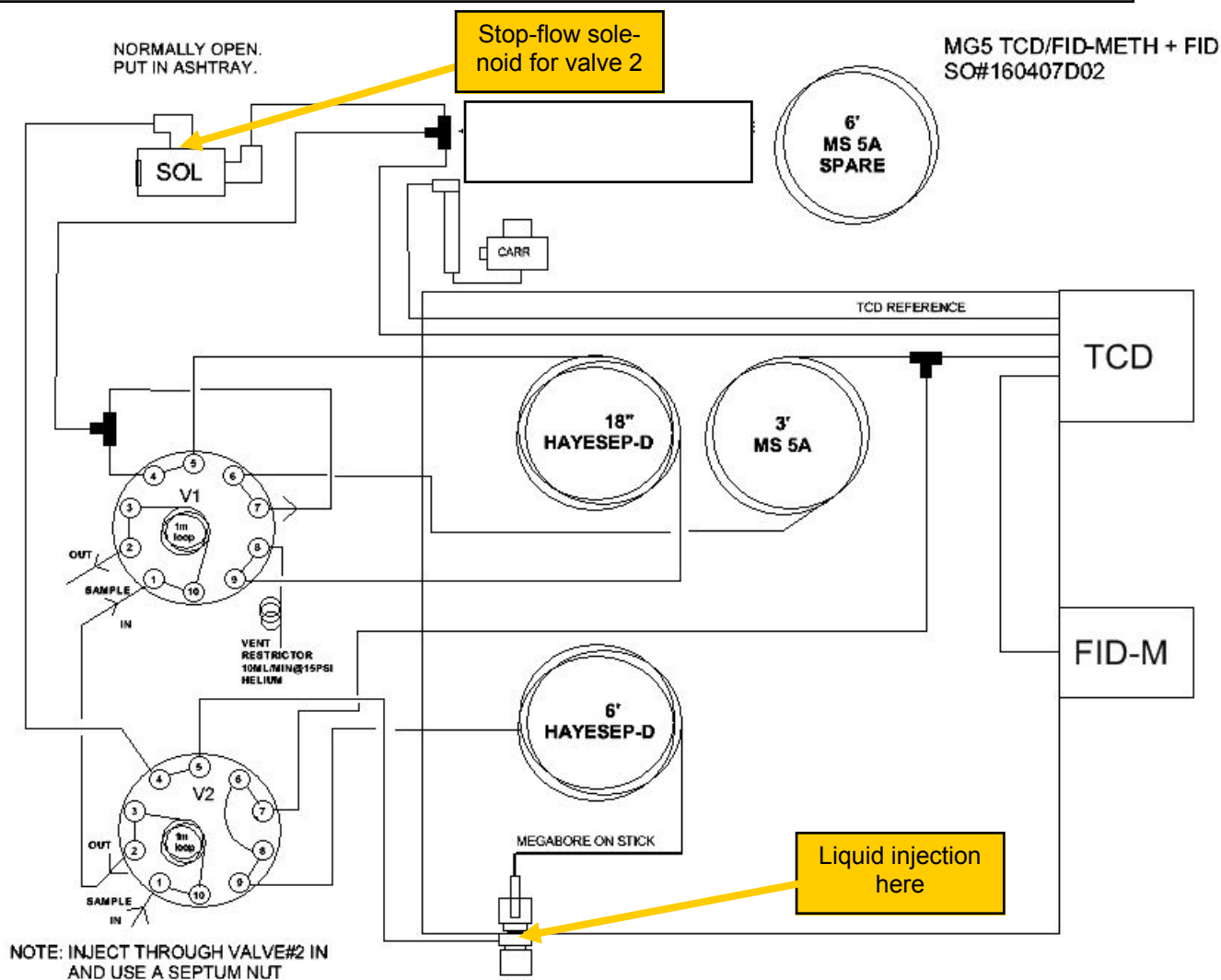
# Multiple Gas#5 GC configuration Jan 2016



The chromatogram above shows the full analysis including the C2 peaks. Note that the Relay F on time ( inject onto the Haysep column ) 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



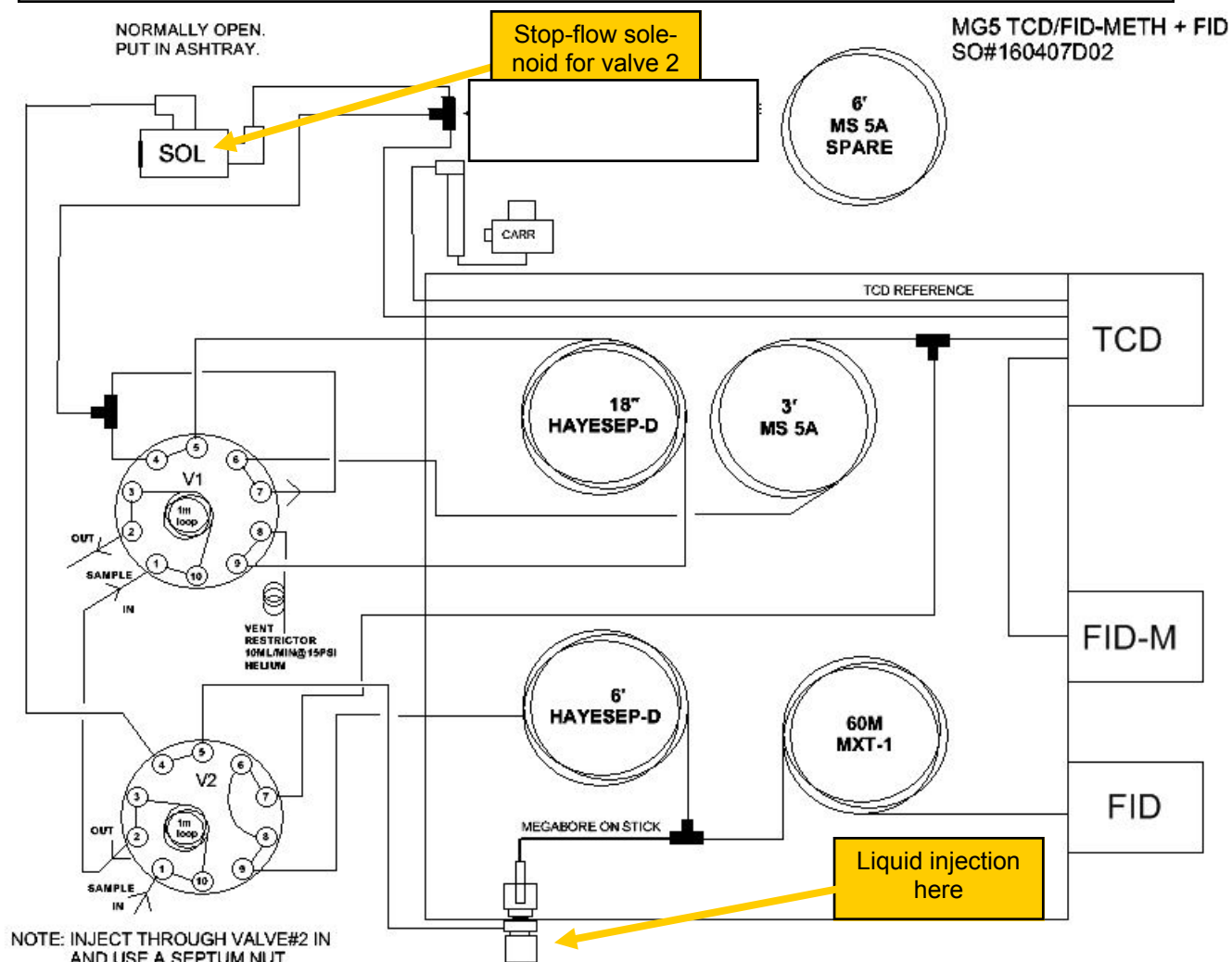
Starting in March of 2016 all MG5 configurations were slightly modified:

- 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 Mole sieve.
- 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

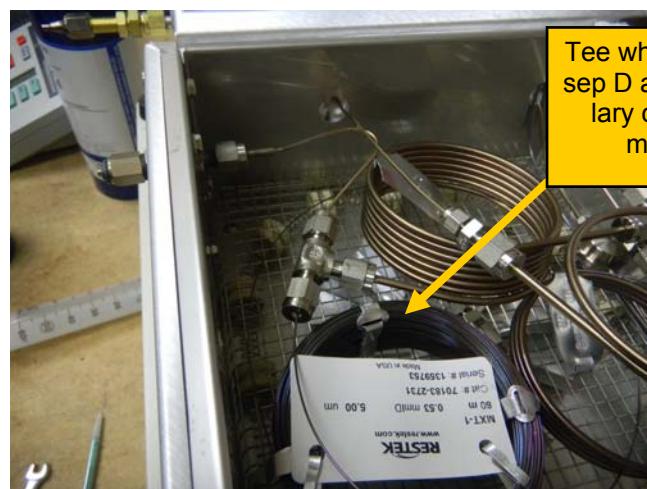


This drawing shows a capillary column and extra FID detector connected at a "tee" fitting so a liquid injection ( or low volume gas injection ) splits onto the capillary and the Haysep column.

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.

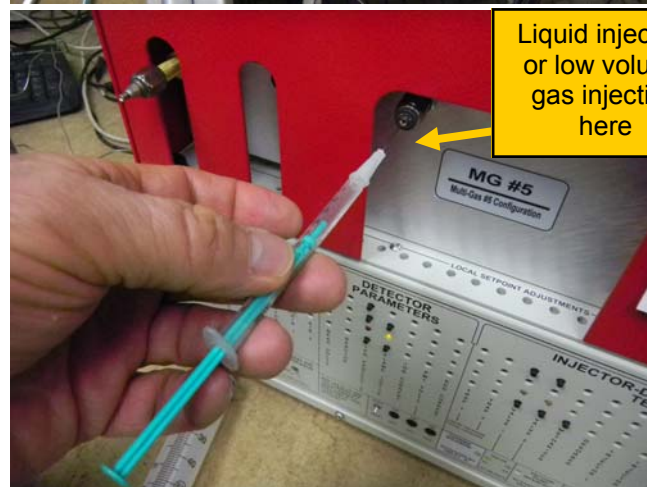
## Multiple Gas#5 GC configuration April 2016

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.



Tee where Haysep D and capillary column meet.

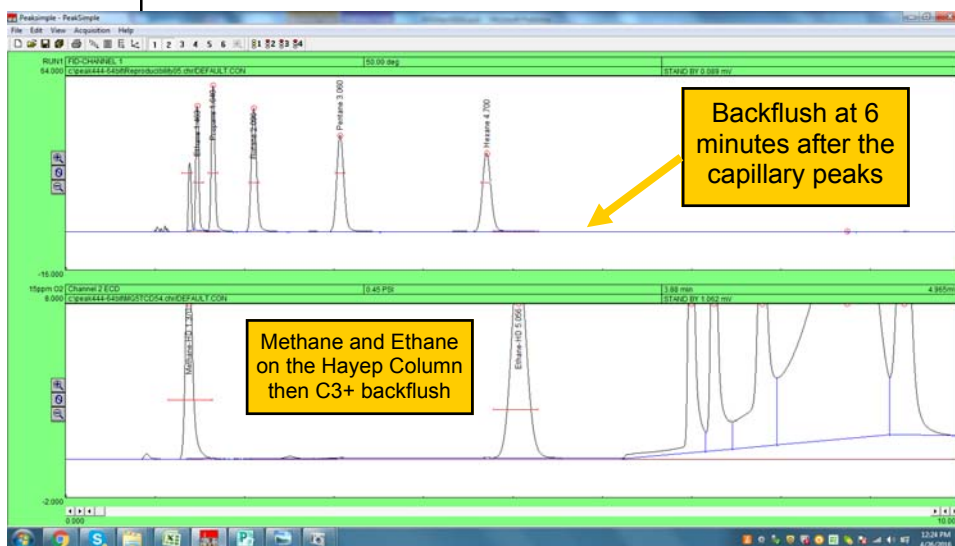
When making a low volume gas injection you do not need to use the gas sampling valves at all, unless you want to backflush the Haysep column after the capillary column peaks have eluted.



Liquid injection or low volume gas injection here

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.

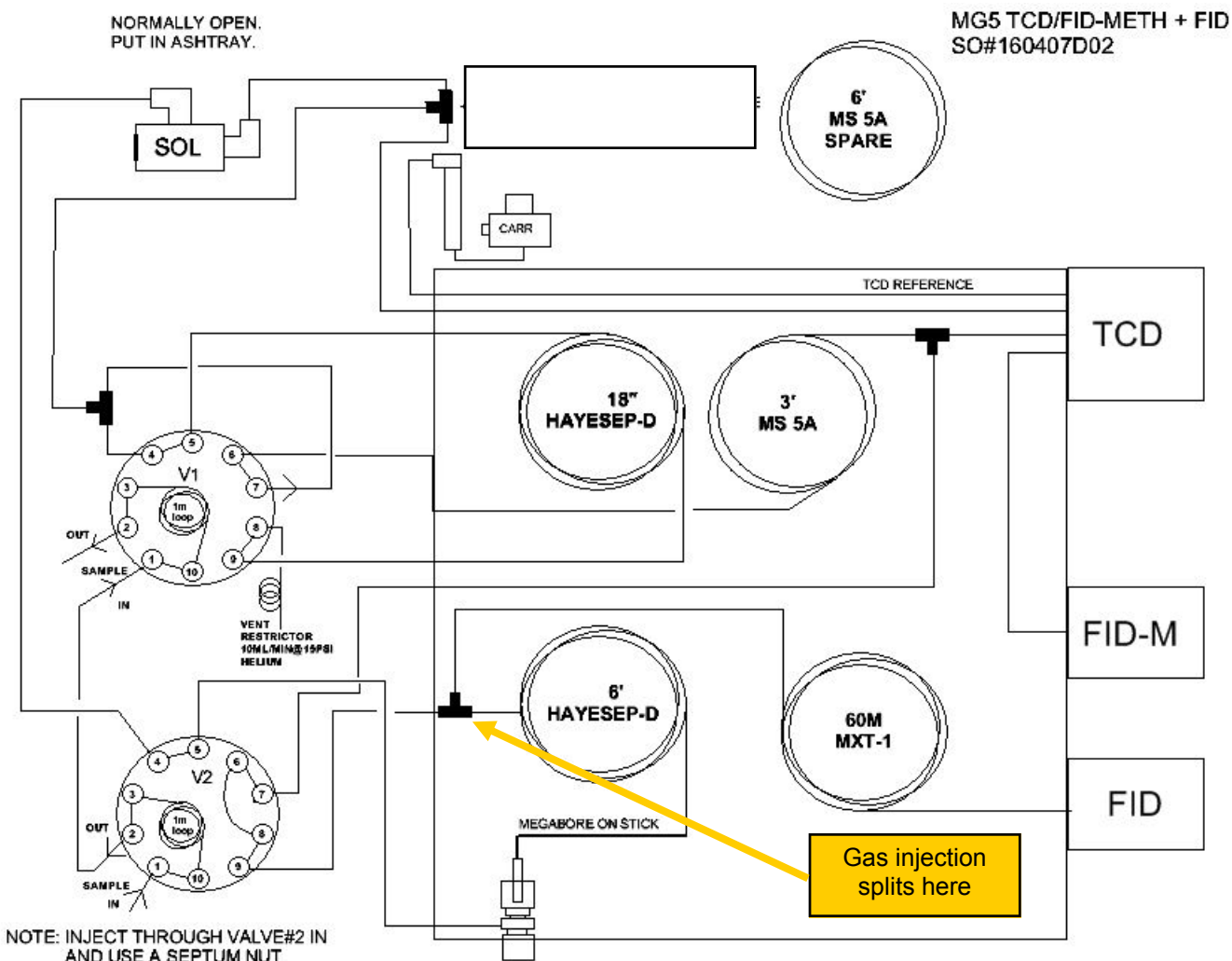
If you need to quantitate the backflush, the gas sampling valve loop must have a carrier gas purge to avoid injecting peaks which might be in the loop. See the diagram to understand.



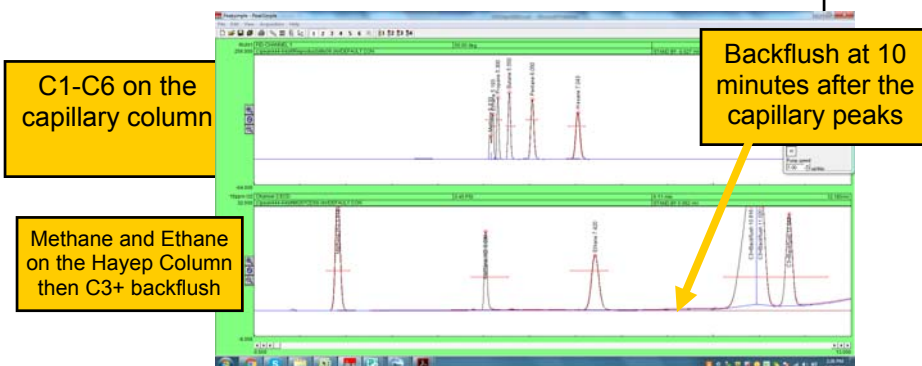
Backflush at 6 minutes after the capillary peaks

Methane and Ethane on the Haysep Column then C3+ backflush

# Multiple Gas#5 GC configuration April 2016

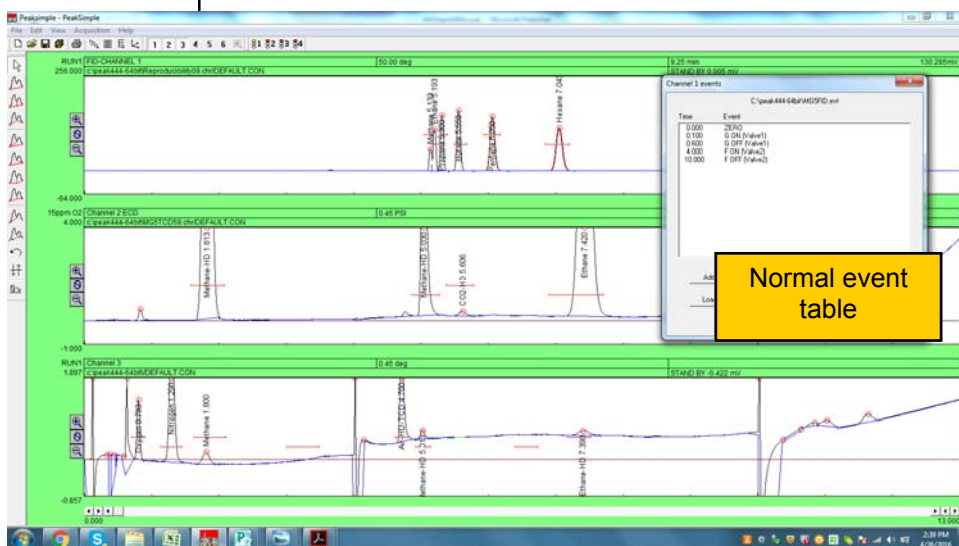


- 1) This drawing shows a capillary column set up for gas injection from the gas sampling valves onto both the Haysep and capillary column.

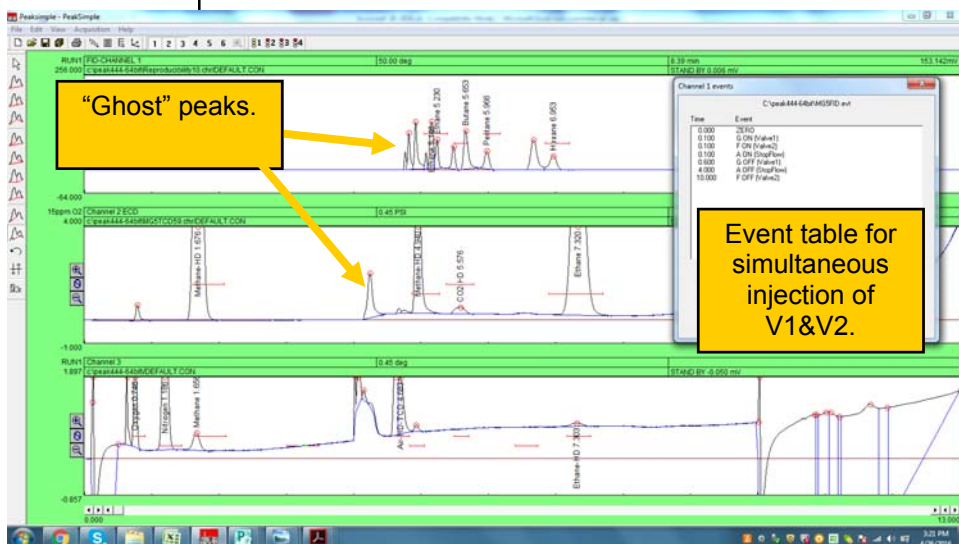




The top chromatogram shows the normal MG5 valve sequence/event table. The sample was 1000ppm C1-C6 aliphatic hydrocarbons. V1 injects at .1minutes. V2 injects at 4 minutes.



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 CO<sub>2</sub> 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.

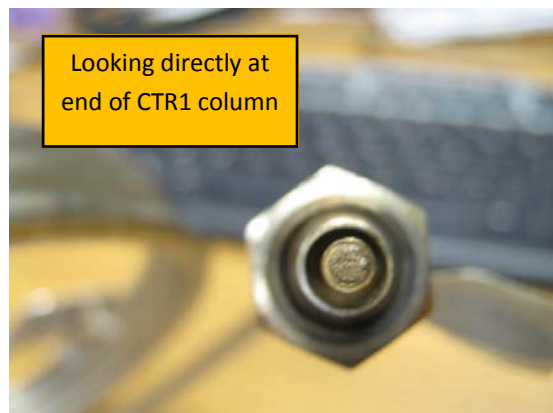
This column is no longer manufactured, or is not easily available, so SRI offers an equivalent or better column for the convenience of our customers.

8600- PKC7 "Fixed Gas Column"

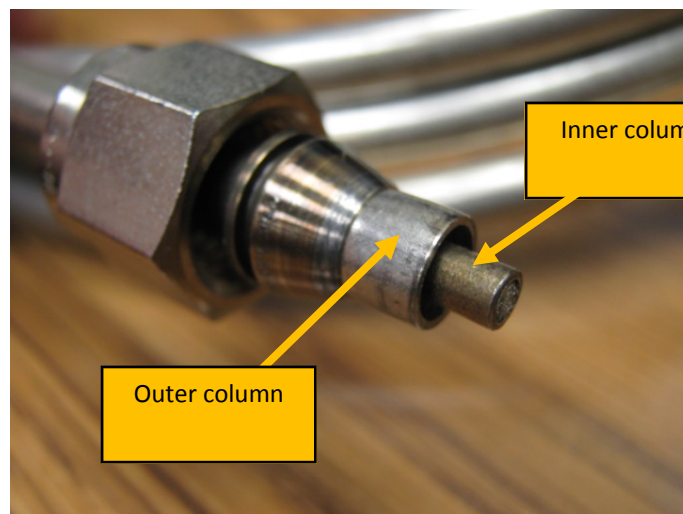
US\$ 659.00 August 2013 ( price may change )

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)

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



Looking directly at end of CTR1 column



Inner column

Outer column





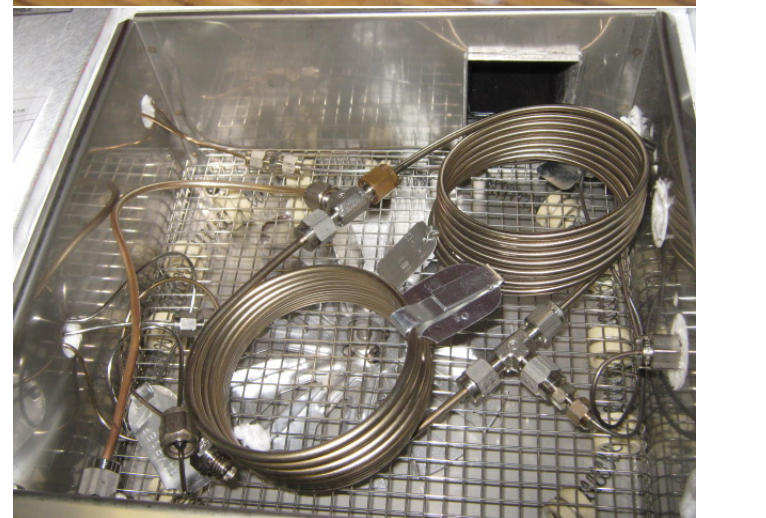
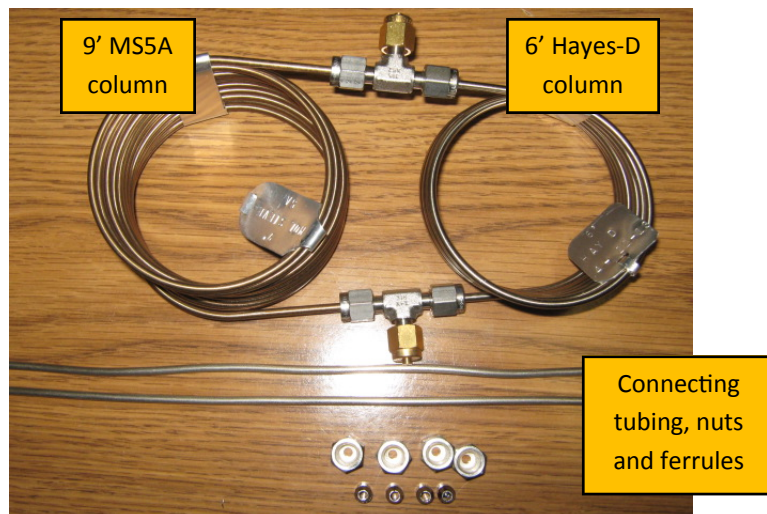
# CTR1 Replacement Column for Fixed Gas analysis

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.





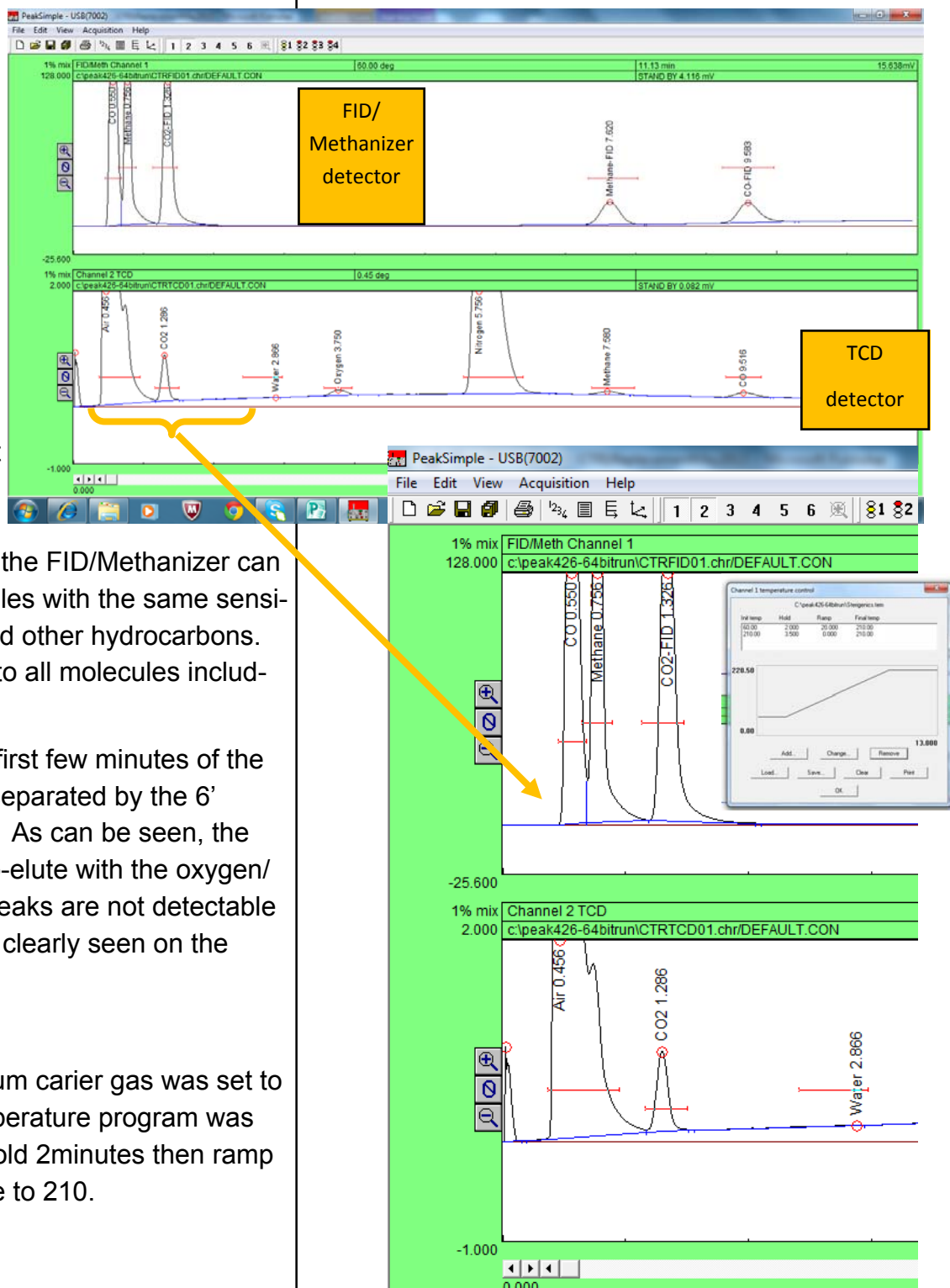
# CTR1 Replacement Column for Fixed Gas analysis

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 CO<sub>2</sub> 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 carrier 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

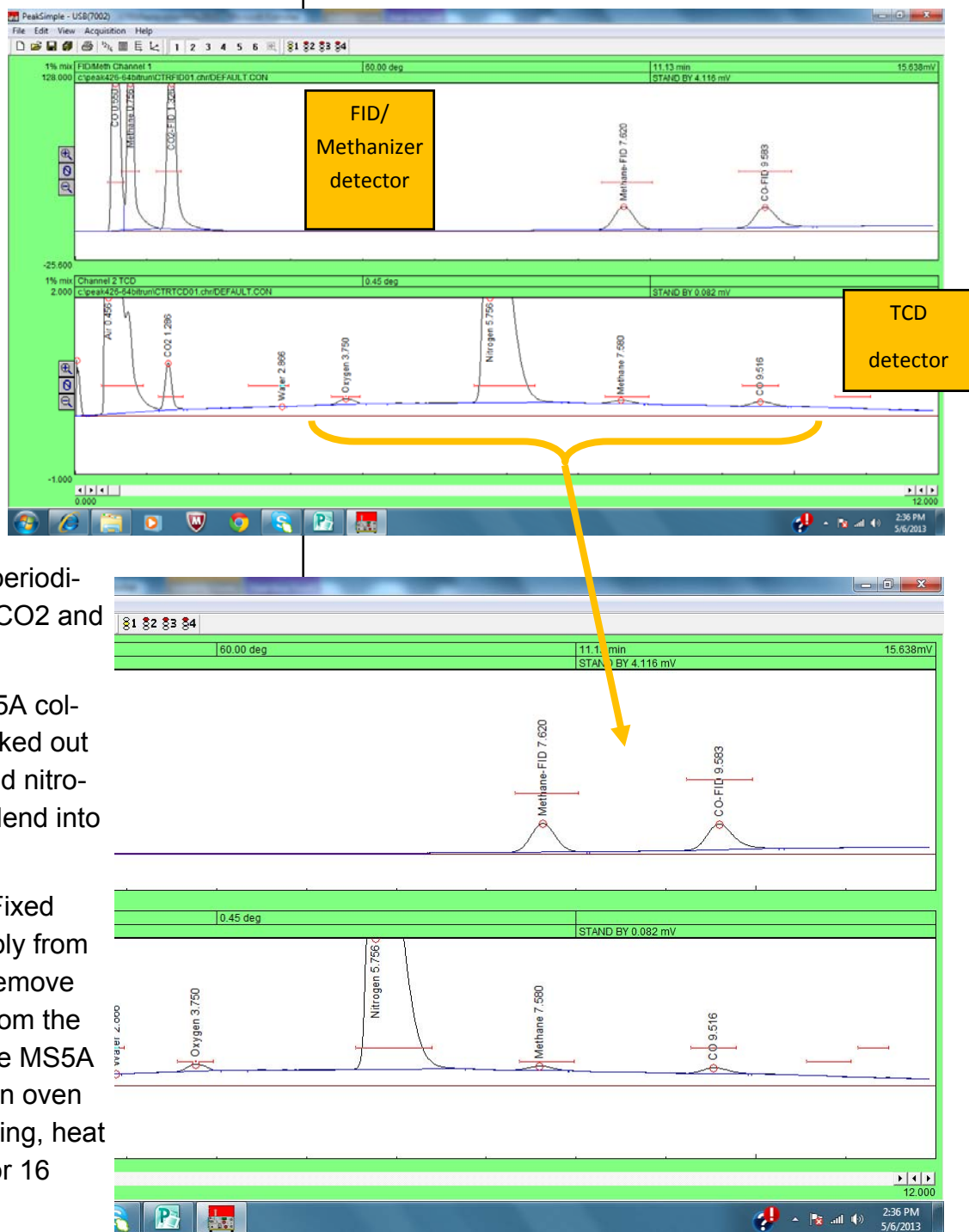
The peaks in the last minutes of the chromatogram are from the 9' Mole-Sieve 5A column.

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

CO<sub>2</sub> however and water are permanently absorbed by this column which must be baked out periodically to remove the CO<sub>2</sub> 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.



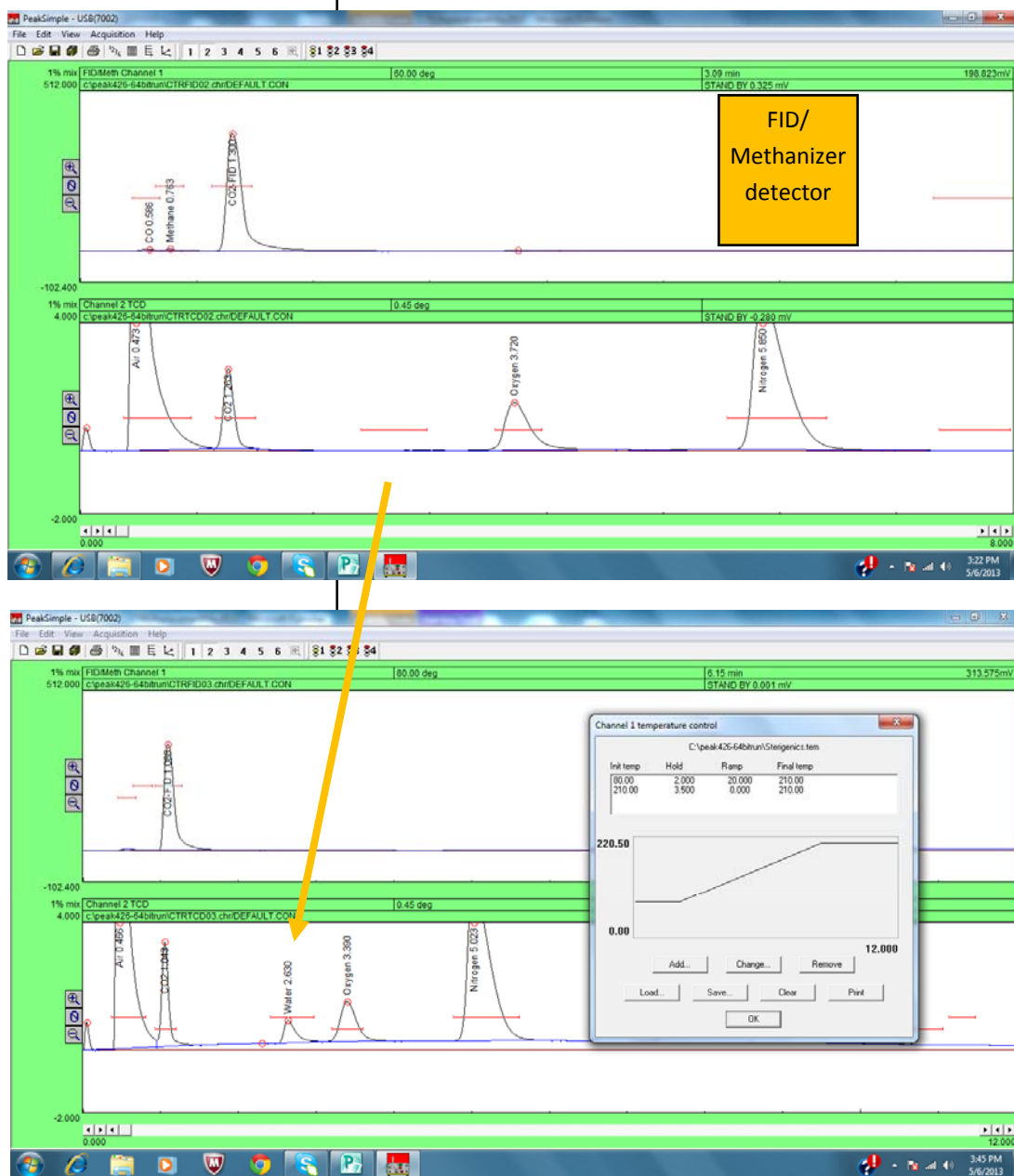
# CTR1 Replacement Column for Fixed Gas analysis

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

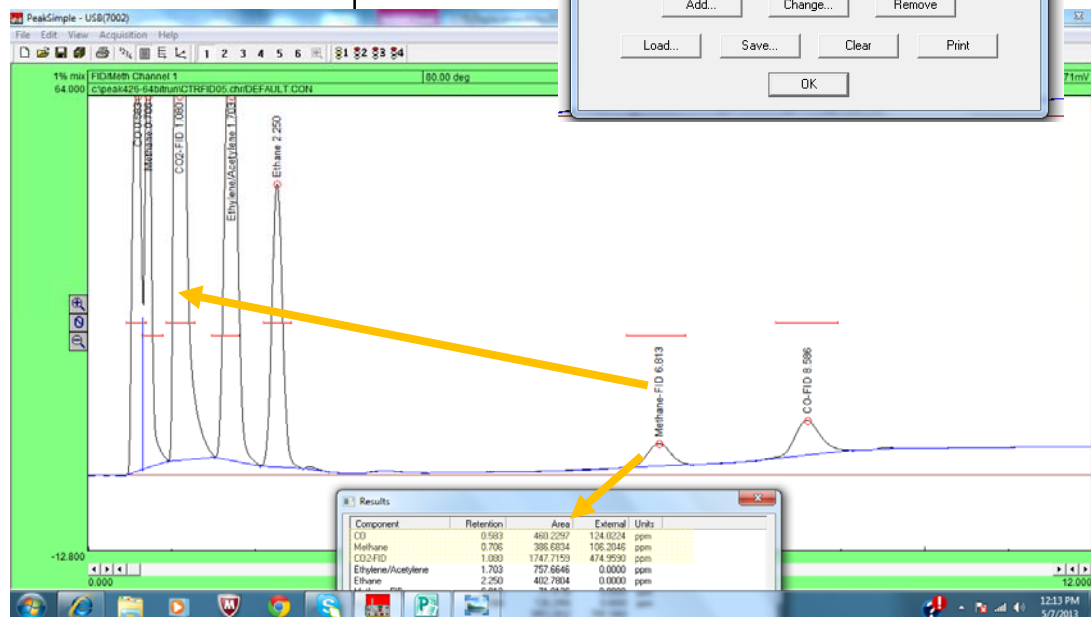
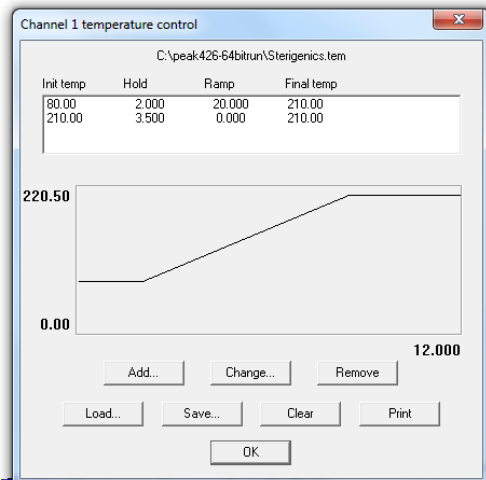
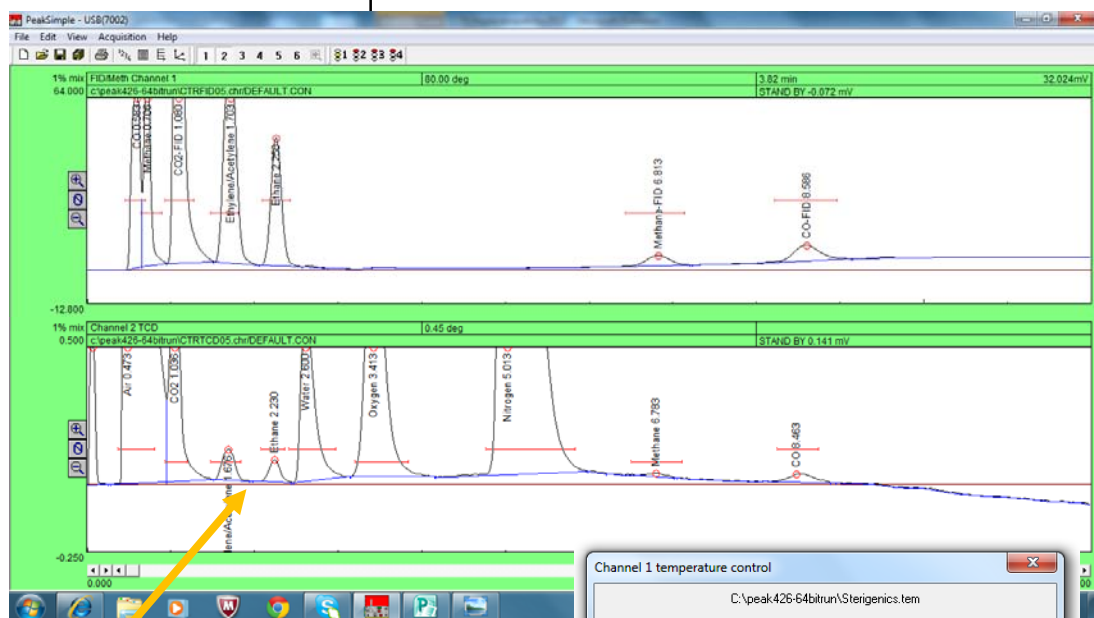
May 2013

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, CH<sub>4</sub>, CO, Ethane, Ethylene/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 CO<sub>2</sub> ( which elutes from the 6'Hayesep-D column ) and the methane and CO ( which elute from the 9'MS5A column ).



Component	Retention	Area	External	Units
CO	0.583	460.2297	124.0224	ppm
Methane	0.706	385.6334	106.2046	ppm
CO2/FID	1.000	1747.7155	474.9550	ppm
Ethylene/Acetylene	1.703	757.6646	0.0000	ppm
Ethane	2.250	402.7884	0.0000	ppm

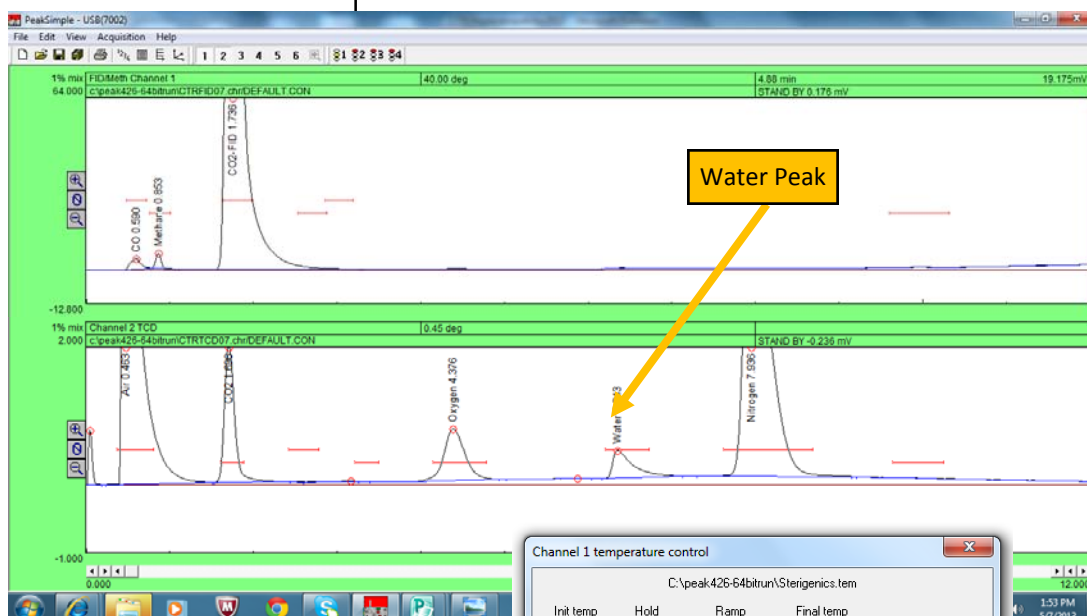


# CTR1 Replacement Column for Fixed Gas analysis

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.

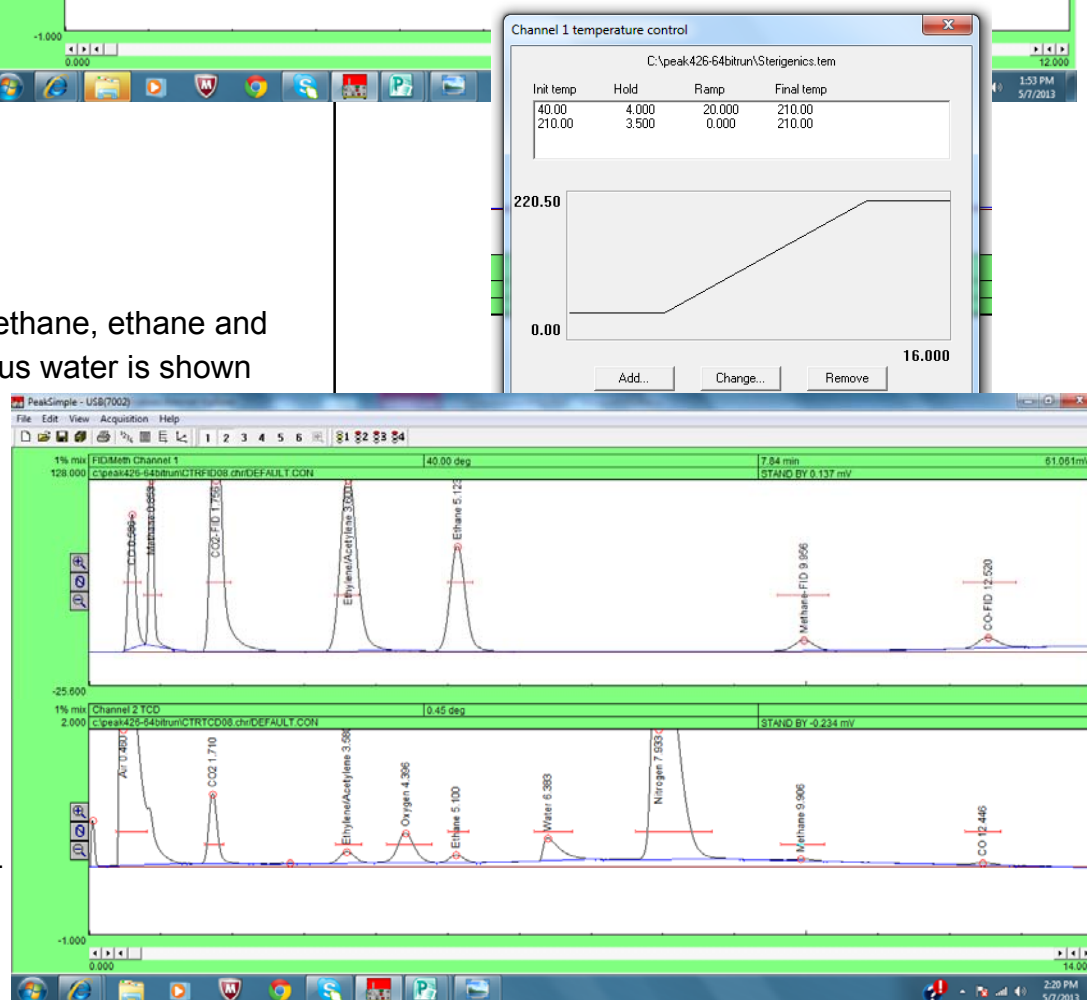


A mix of:

O<sub>2</sub>, N<sub>2</sub>, CO, CO<sub>2</sub>, methane, ethane and ethylene/acetylene plus water is shown starting at 40C. You

can see the peaks from the Haysep-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.

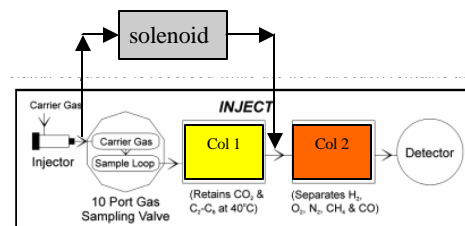
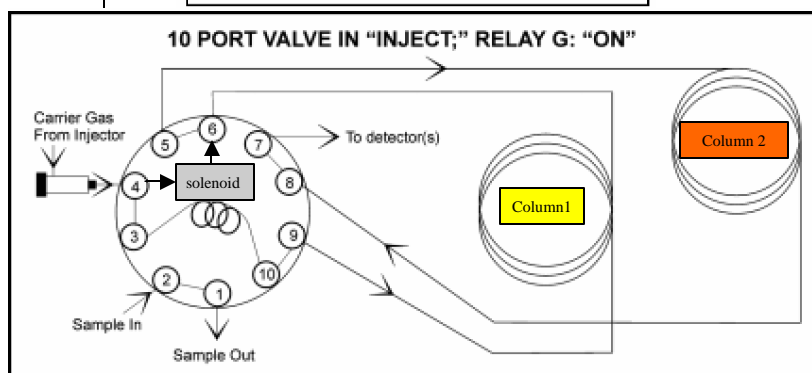
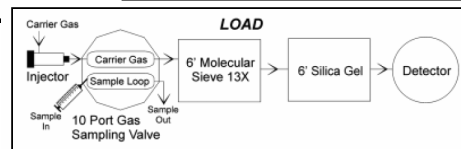
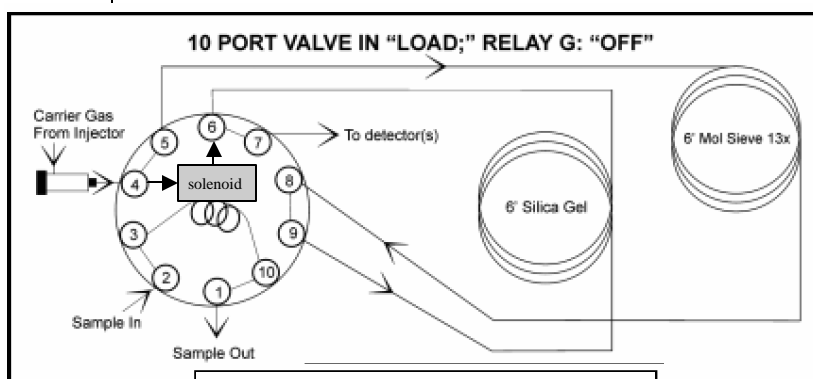


The MG#3 GC configuration is almost identical to the MG#1 GC configuration except there is an additional solenoid valve which when activated by the PeakSimple data system stops the flow of carrier gas in column 1.

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 Mole-Sieve 13X as Column 2.





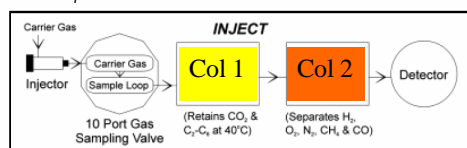
# SRI 8610C Gas Chromatograph Multiple Gas #3 GC configuration

The chromatograms shown on this page are a mix of natural gas and sulfur compounds. The top chromatogram shows the sulfur selective FPD response.

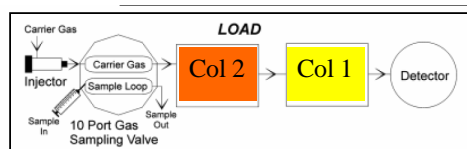
The middle chromatogram shows the FID response.

The two lower chromatograms show the FPD response (black) overlaid with the FID response (red).

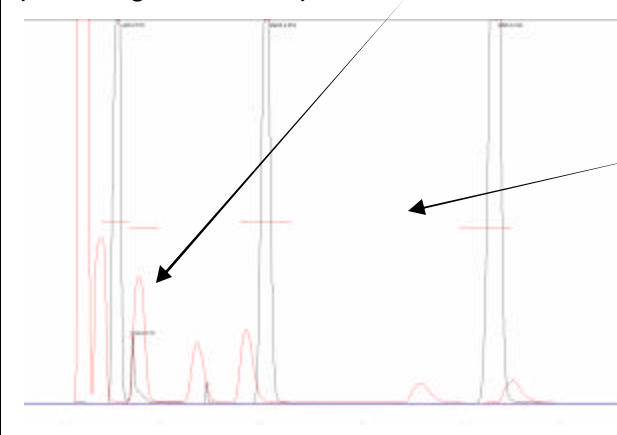
The PeakSimple event table shown at right rotates the valve from Load to Inject at .1 minutes and then back to Load at 1.00 minutes. Because even the first peak (methane) has not migrated from Column 1 though to Column 2 at this time, the equivalent effect is that the



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.

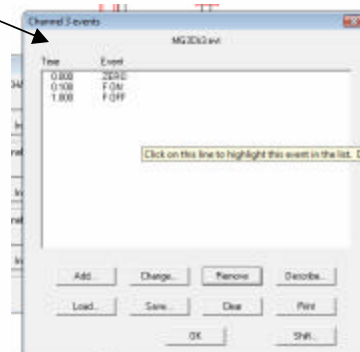


FPD  
Sulfur only

Column 1 60 meter  
MXT1 .53mm 5 mic.

Column 2 15 meter  
RTX Q-Plot .53mm

FID  
Hydrocarbons

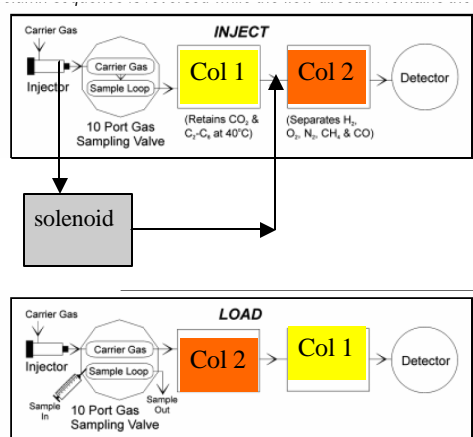


FPD and FID  
overlaid

Just this section  
enlarged

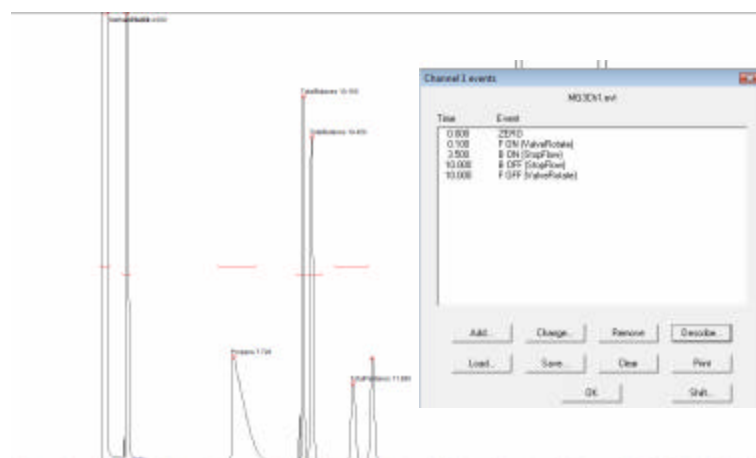
# SRI 8610C Gas Chromatograph Multiple Gas #3 GC configuration

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 ).

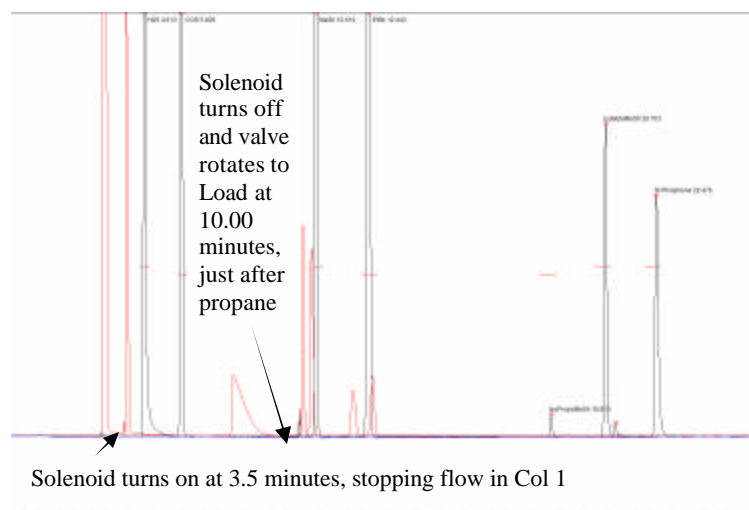


This traps the peaks after Propane in Column 1 while the peaks in Column 2 ( Methane, Ethane, Propane, H<sub>2</sub>S and COS ) separate and elute. Unlike column 1 which does not separate COS and Propane, the peaks are well separated on Column 2 so quenching does not occur.

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. )

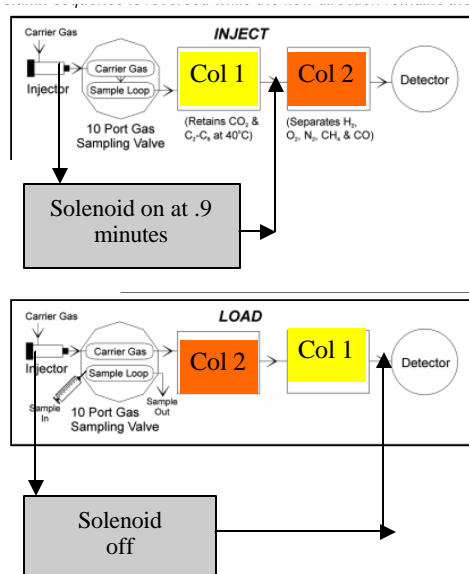


Oven temperature 40C for 10 minutes then 20C/min to 200C

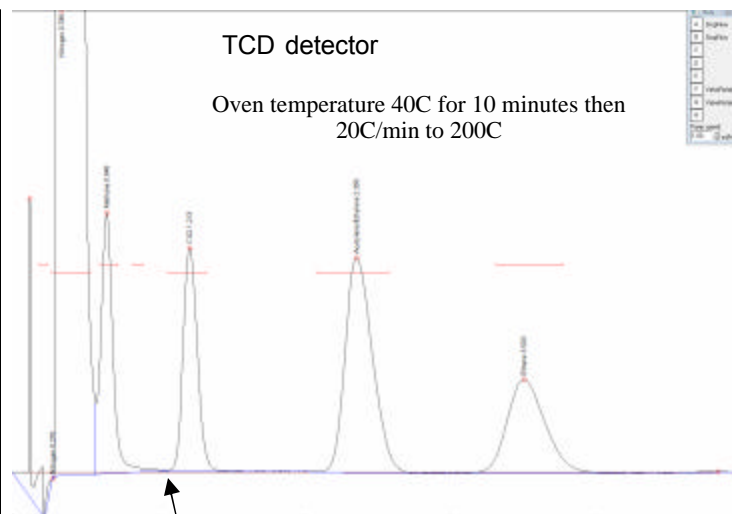


# SRI 8610C Gas Chromatograph Multiple Gas #3 GC configuration

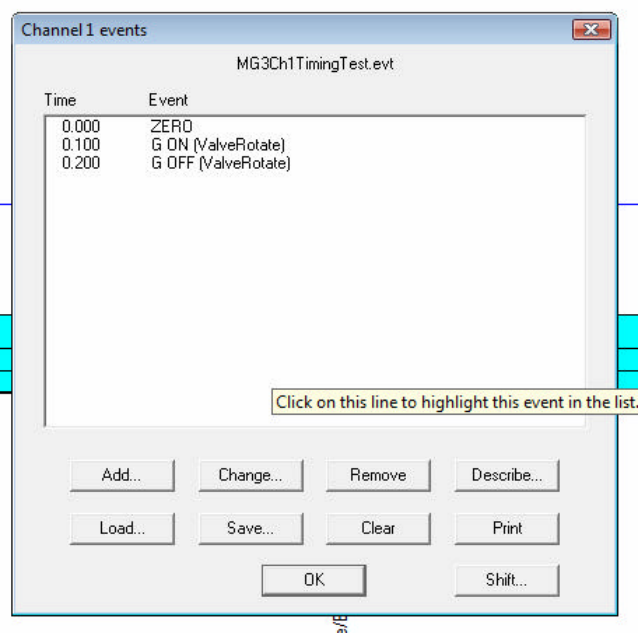
The MG#3 GC configuration is also useful with other column combinations. In this example, Column 1 is a 3' HaysepD and Column 2 is a 6' MS13X. The sample is first run on the 3' HaysepD using the event table shown at right. Because the valve is rotated back to the Load position almost immediately after 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 CO<sub>2</sub> where it would make sense to activate the stop-flow solenoid valve to immobilize the CO<sub>2</sub> and heavier peaks in Column1 while the H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, Methane and CO peaks elute from Column1.



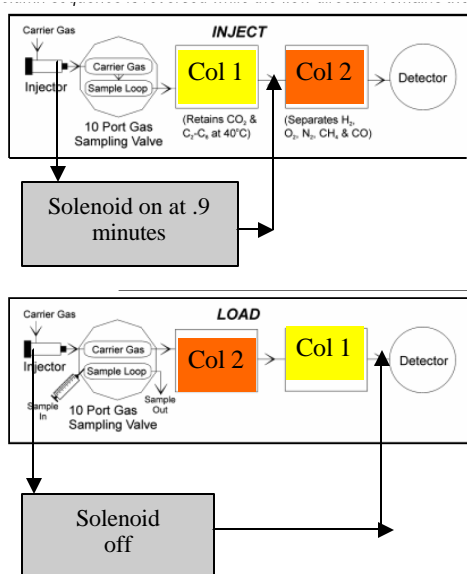
This would be a good time (.9 minutes) to activate the stop-flow solenoid. Just after the Methane migrates onto Column2 but before the CO<sub>2</sub> and heavier peaks





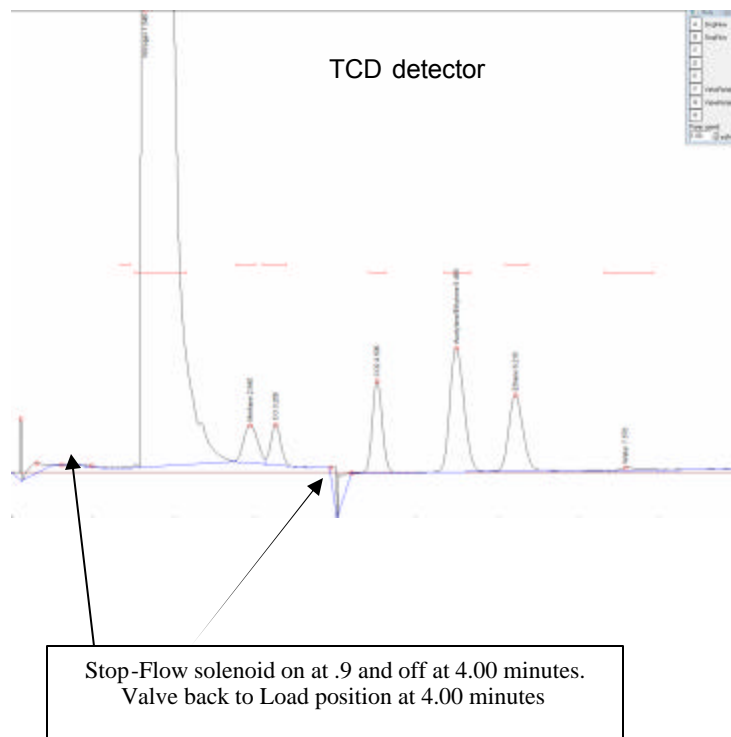
## SRI 8610C Gas Chromatograph Multiple Gas #3 GC configuration

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 minutes. This results in H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub> 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.



Channel 1 events

MG3Ch1.evt

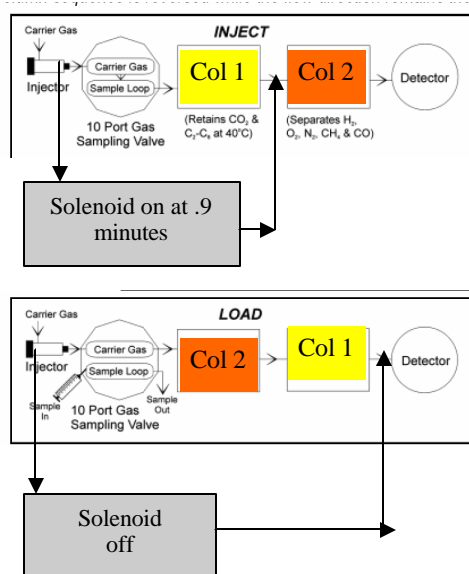
Time	Event
0.000	ZERO
0.100	G ON (ValveRotate)
0.900	A ON (StopFlow)
4.000	A OFF (StopFlow)
4.000	G OFF (ValveRotate)

Buttons: Add..., Change..., Remove, Describe..., Load..., OK, Shift...

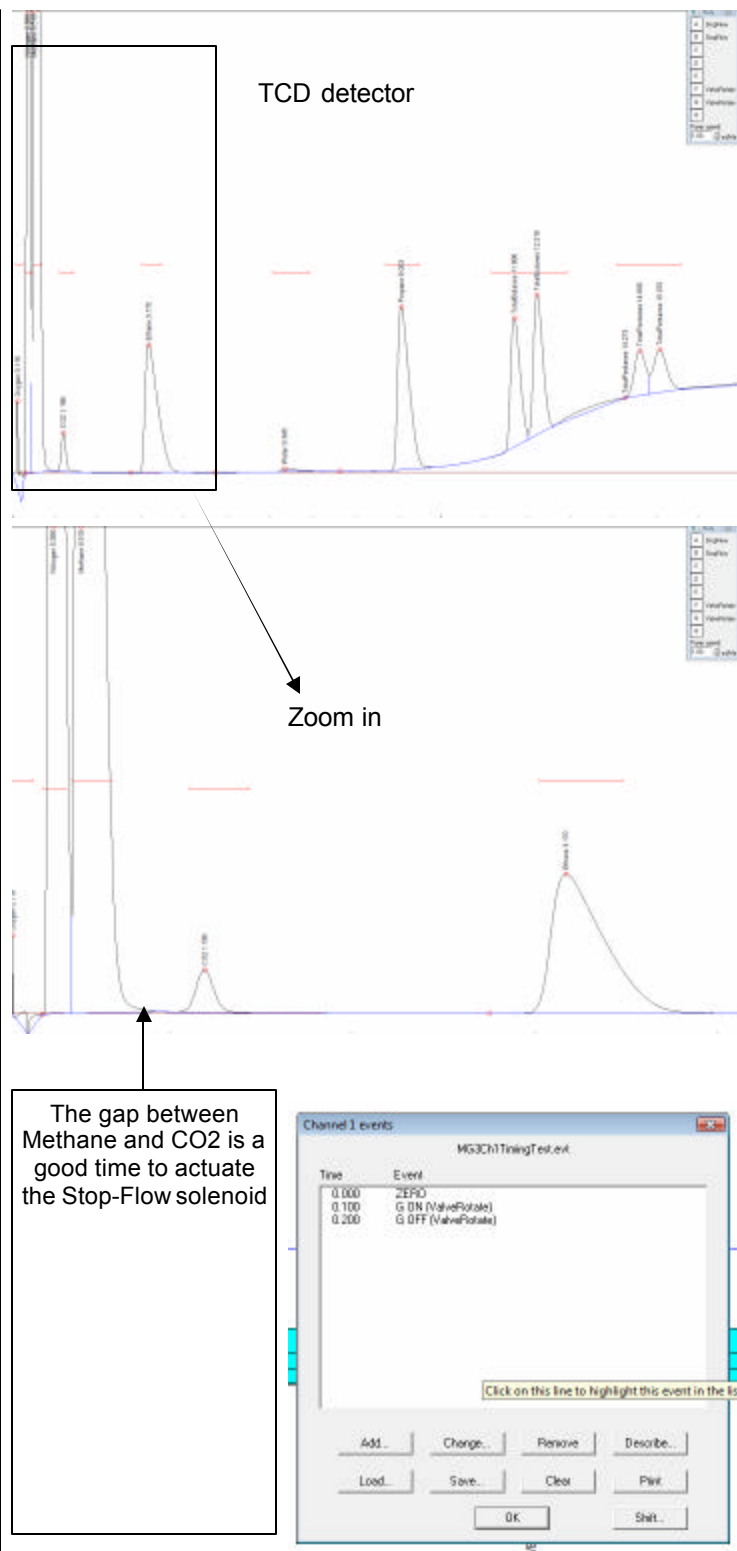
Click on this button after highlighting an event in the Change screen.

# 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.



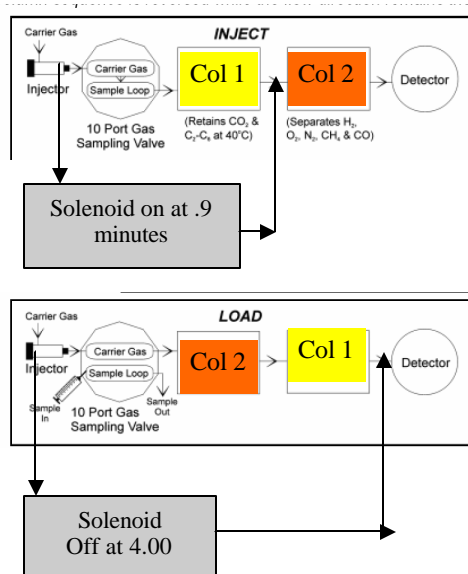
Column 1 is a 3' Haysep D and Column 2 is a 6' MS13X. The Haysep D does not separate Oxygen and Nitrogen or CO. Set the Stop-Flow solenoid time by finding the gap between Methane and CO<sub>2</sub>, in this case about .9 minutes.



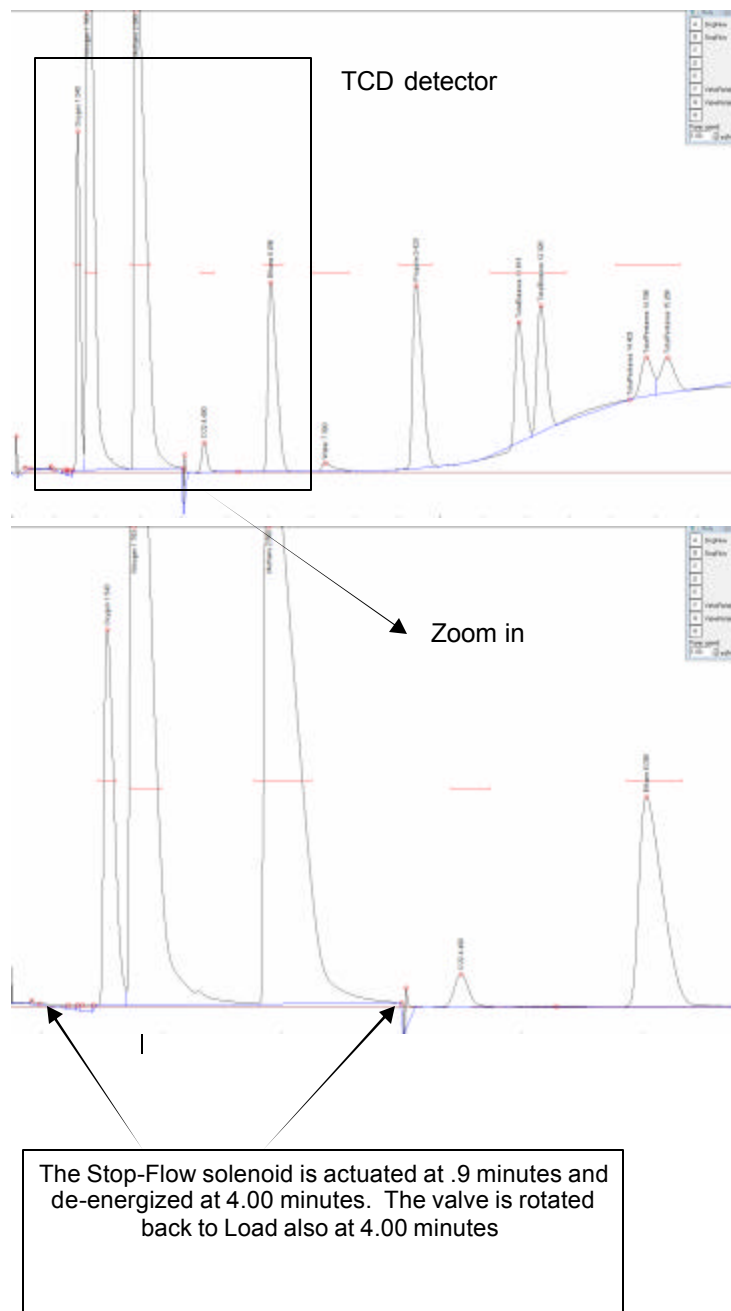
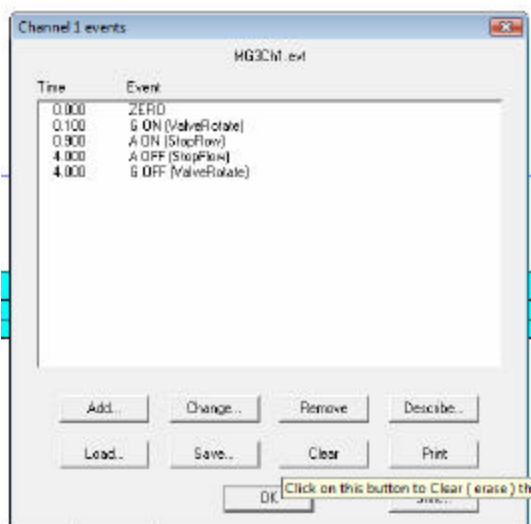
# SRI 8610C Gas Chromatograph

## 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.





## Multiple Gas#3 plus Sulfur GC Configuration

The SRI Model 8610C Gas Chromatograph ( GC ) configured as a MultipleGas#3 plus Sulfur is designed to measure H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CO, CO<sub>2</sub>, H<sub>2</sub>O, C<sub>1</sub> through C<sub>5</sub> hydrocarbons and also H<sub>2</sub>S, COS/SO<sub>2</sub>, and other sulfur molecules such as mercaptans, CS<sub>2</sub>, 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%

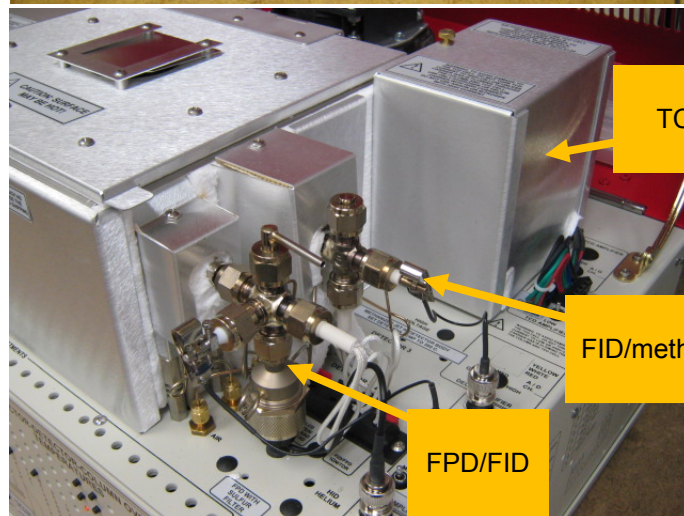
The FID/Methanizer detector ( FID/meth ) measures hydrocarbons plus CO and CO<sub>2</sub> from 1ppm to 50,000ppm

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 H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CO, CO<sub>2</sub> and C<sub>1</sub>-C<sub>5</sub> hydrocarbons as well as water.

The 60meter MXT1 capillary column separates the sulfur molecules and also hydrocarbons from C<sub>1</sub>-C<sub>10</sub>



## Multiple 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.

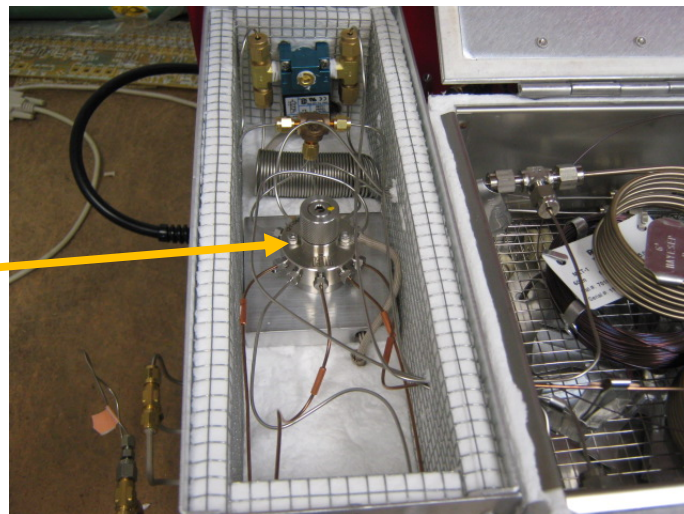




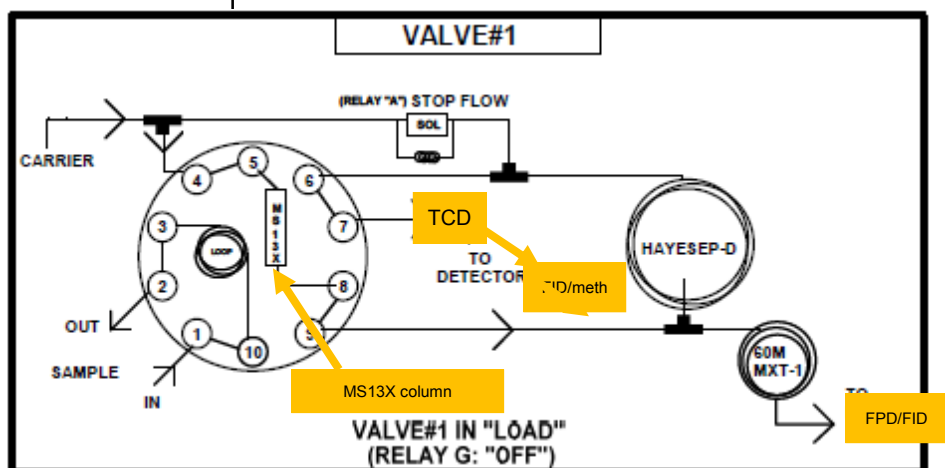
## Multiple Gas#3 plus Sulfur GC Configuration

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

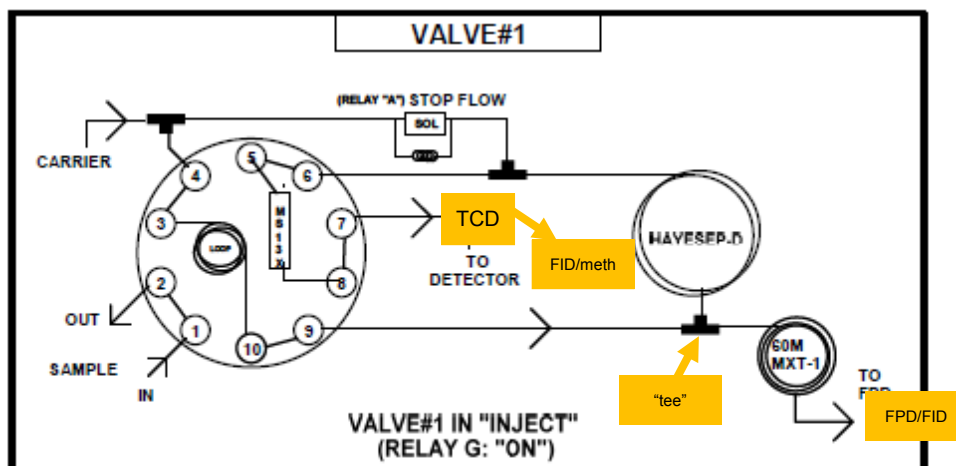
The valve is plumbed as shown in the diagram.



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.



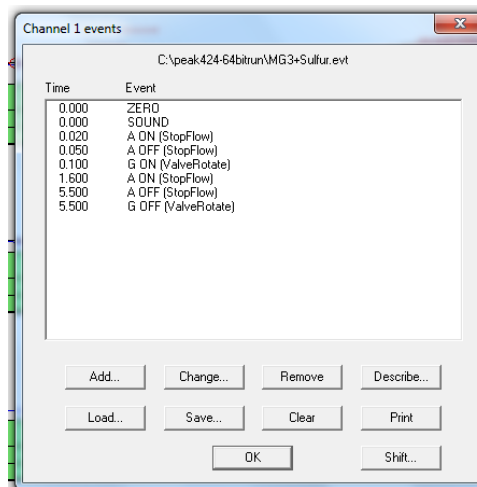
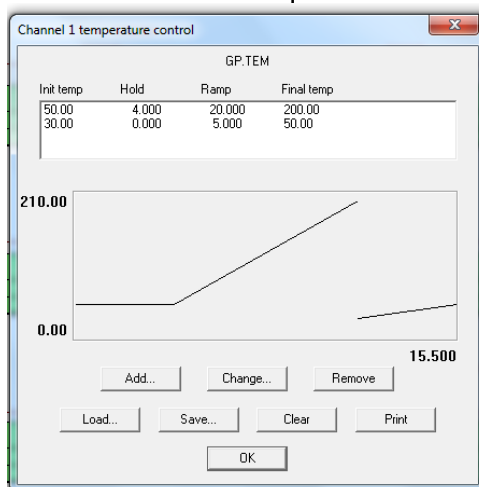
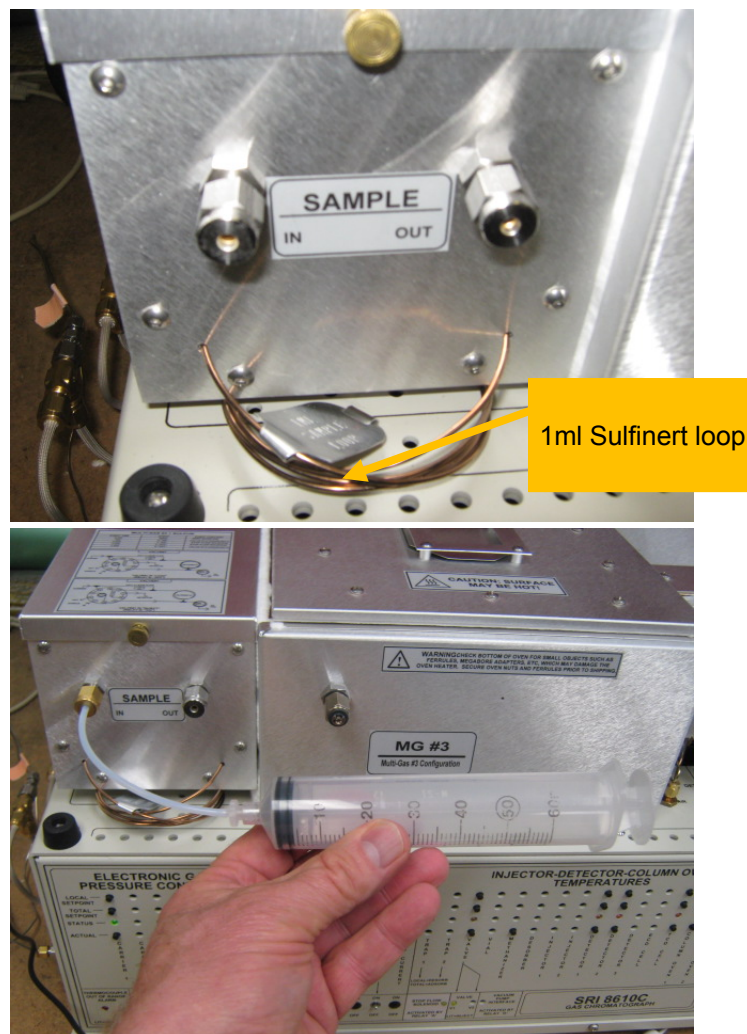


## Multiple Gas#3 plus Sulfur GC Configuration

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 H<sub>2</sub>S 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.

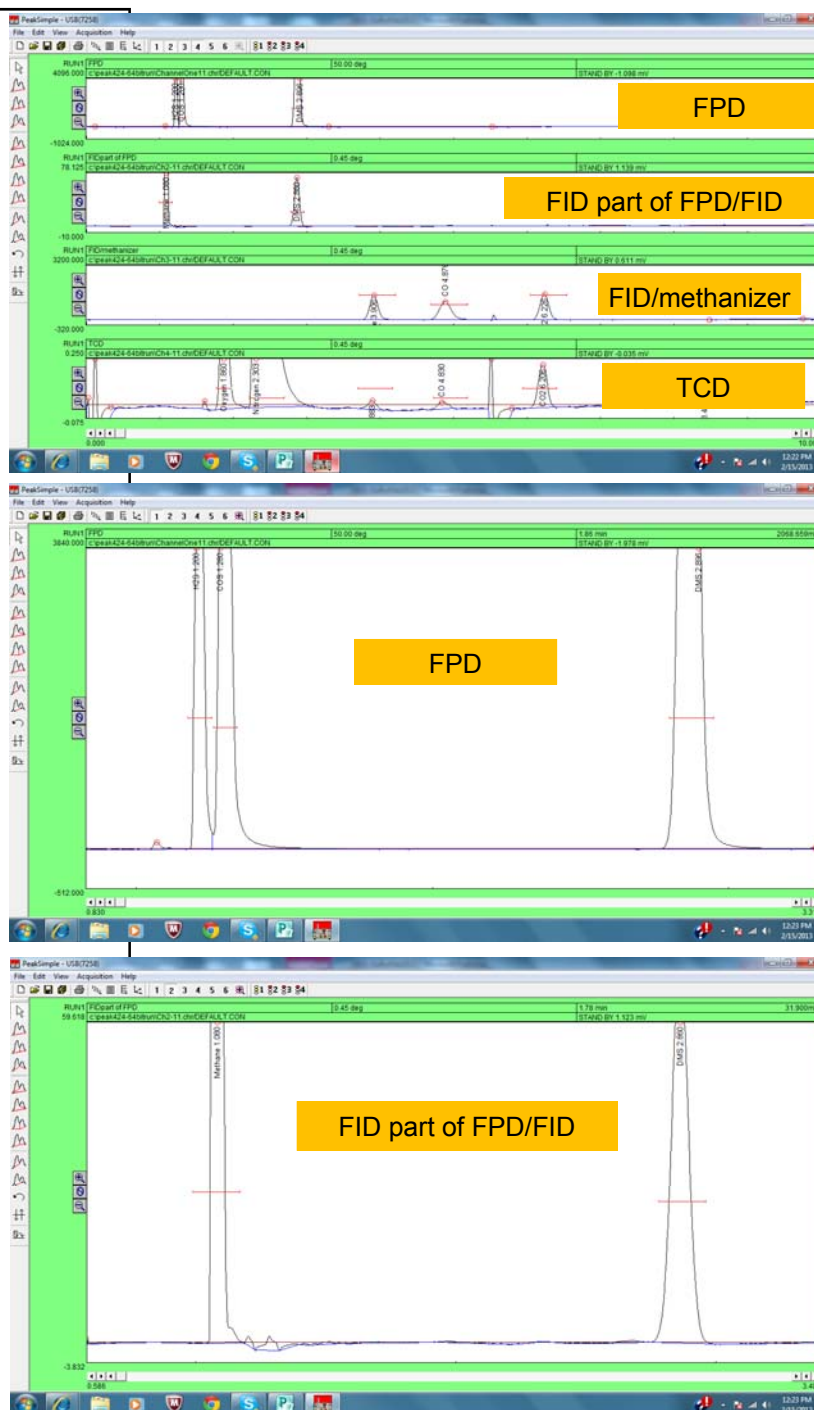


## Multiple Gas#3 plus Sulfur GC Configuration

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

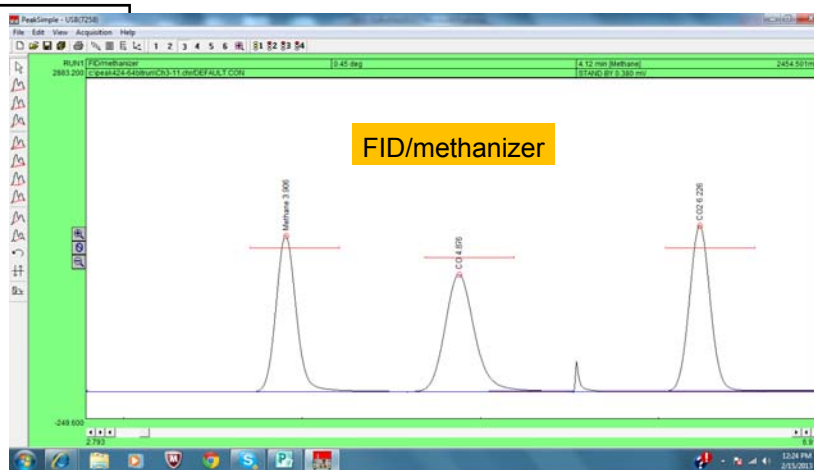
Zooming in on the FPD chromatogram you can see Hydrogen Sulfide (  $\text{H}_2\text{S}$  ), Carbonyl Sulfide (  $\text{COS}$  ) and Di Methyl sulfide (  $\text{DMS}$  ). The FPD is blind to hydrocarbons like methane.

The FID part of the FPD/FID combo detector detects methane and  $\text{DMS}$  (  $\text{C}_2\text{H}_6\text{S}$  ), but does not detect  $\text{H}_2\text{S}$  and  $\text{COS}$  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 than what a normal FID detector would deliver.

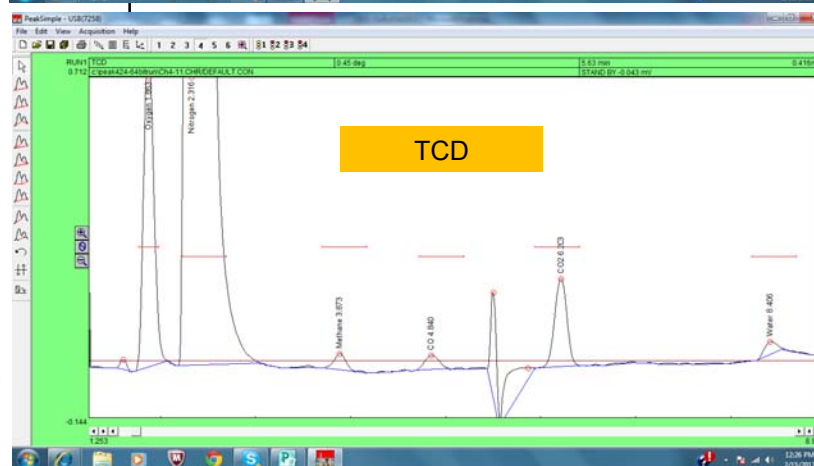


## Multiple Gas#3 plus Sulfur GC Configuration

The FID/methanizer detects all hydrocarbons from methane to hexane and also CO and CO<sub>2</sub> which are converted to methane by the methanizer. Detection from 1ppm to 50,000ppm are possible. Shown are methane, CO and CO<sub>2</sub> at 1000ppm.



The TCD detects Hydrogen, Oxygen, Nitrogen, Methane, CO, CO<sub>2</sub>, Water and all molecules 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.



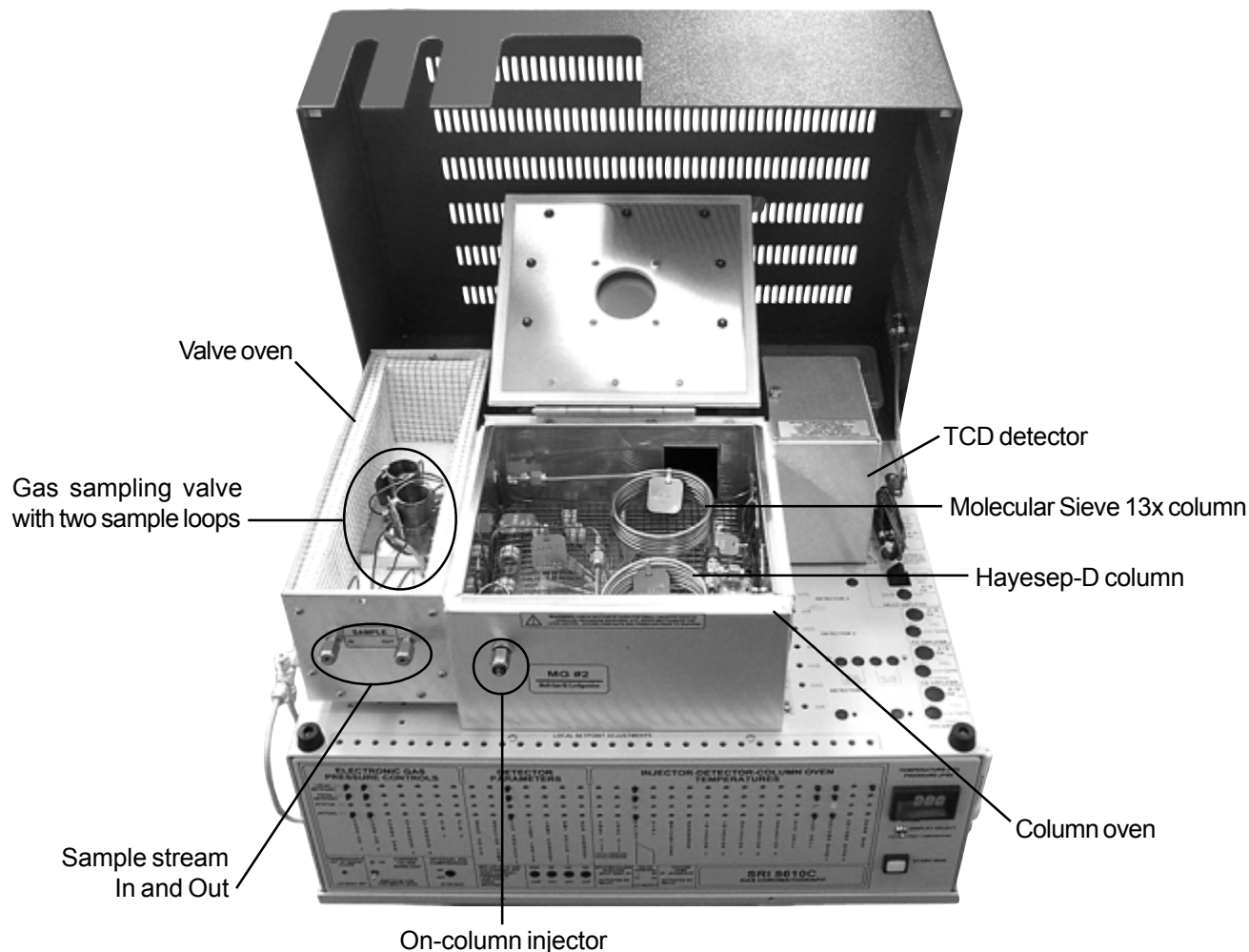


## POPULAR CONFIGURATION GCs

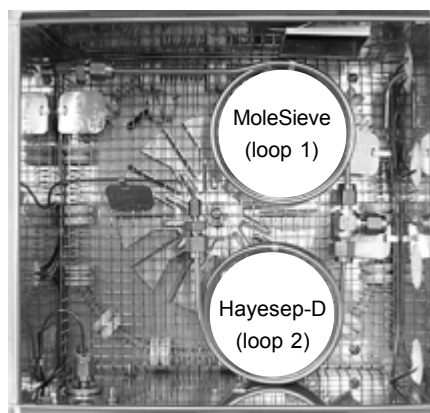
### Multiple Gas Analyzer #2

#### 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.



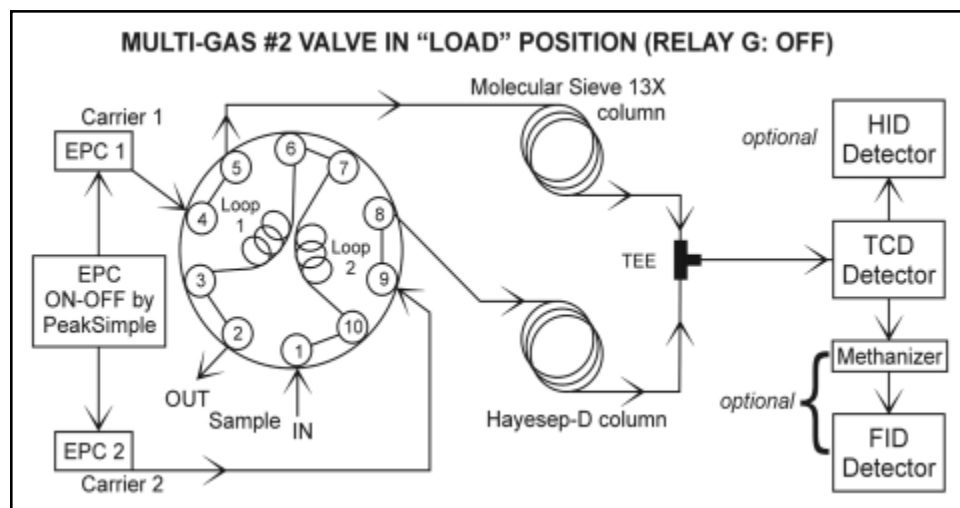
## POPULAR CONFIGURATION GCs

### Multiple Gas Analyzer #2

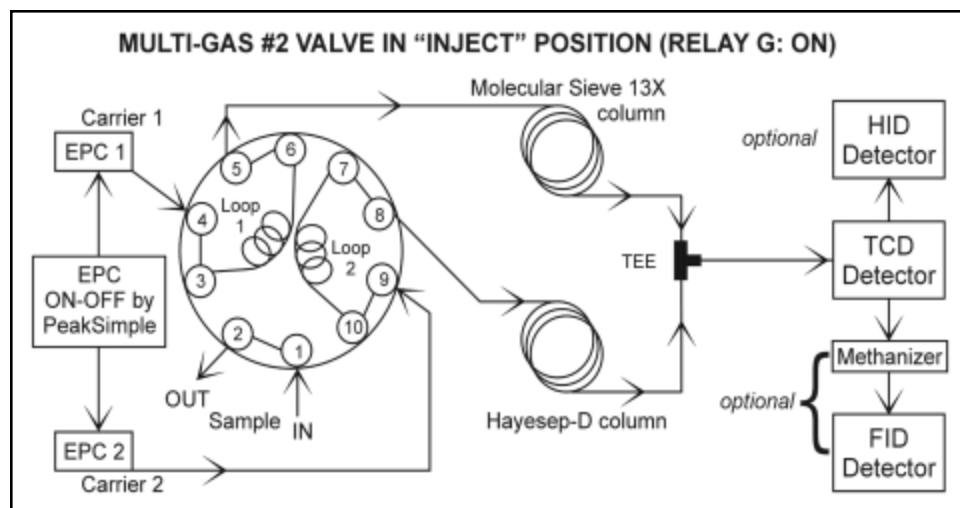
#### Theory of Operation

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.

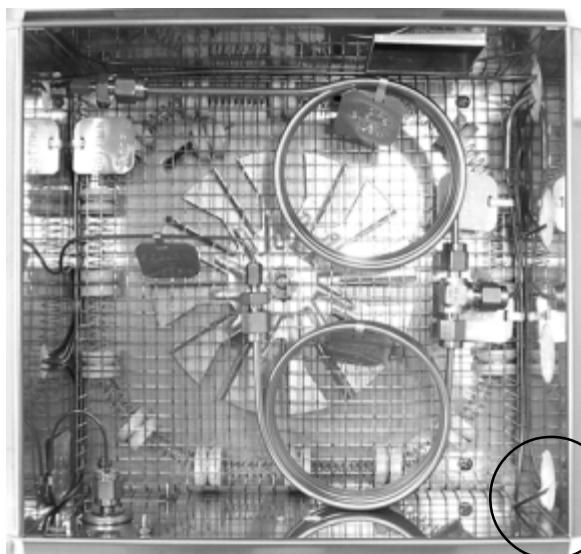
## POPULAR CONFIGURATION GCs Multiple Gas Analyzer #2

### 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.

GAS FLOW RATES					
CARRIER 1:	MOL. SIEVE :	10	PSI =	10	ml/min
CARRIER 2:	HAYESEP-D :	7	PSI =	10	ml/min
P&T PURGE:			PSI =		ml/min
HYDROGEN 1:			PSI =		ml/min
HYDROGEN 2:			PSI =		ml/min

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.)

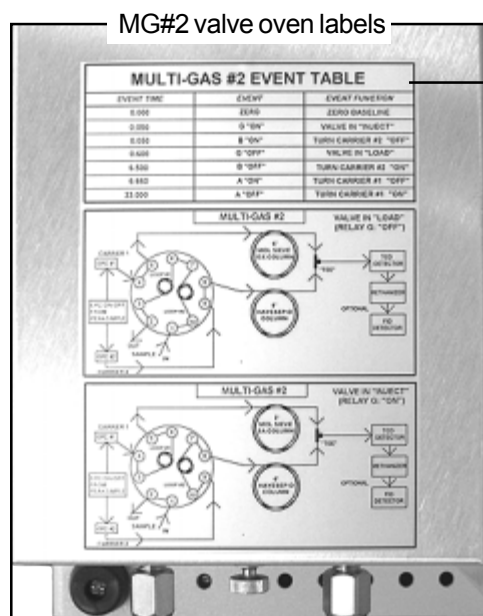
Initial	Hold	Ramp	Final
50.00	3.00	20.00	220.00
220.00	25.00	0.00	220.00



## POPULAR CONFIGURATION GCs

### Multiple Gas Analyzer #2

#### General Operating Procedure continued



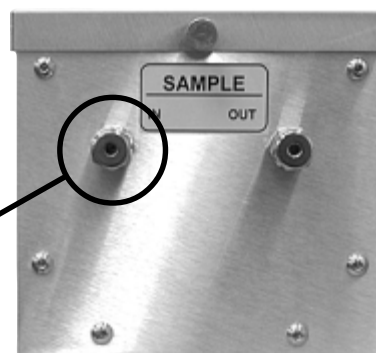
#### Example event table:

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)

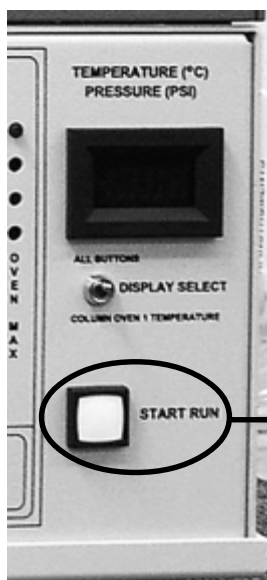
6. Type in an Event table. The example shown is labeled on the MG#2 valve oven. The event table should allow for the elution of CO from the molecular sieve column before carrier #2 is turned back ON. The column oven temperature may be increased to speed the elution of the H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>, and CO. Hydrocarbons like ethane and propane end up on the Molecular Sieve 13x column after its carrier is turned OFF and the Hayeseep-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.



Sample inlet port



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

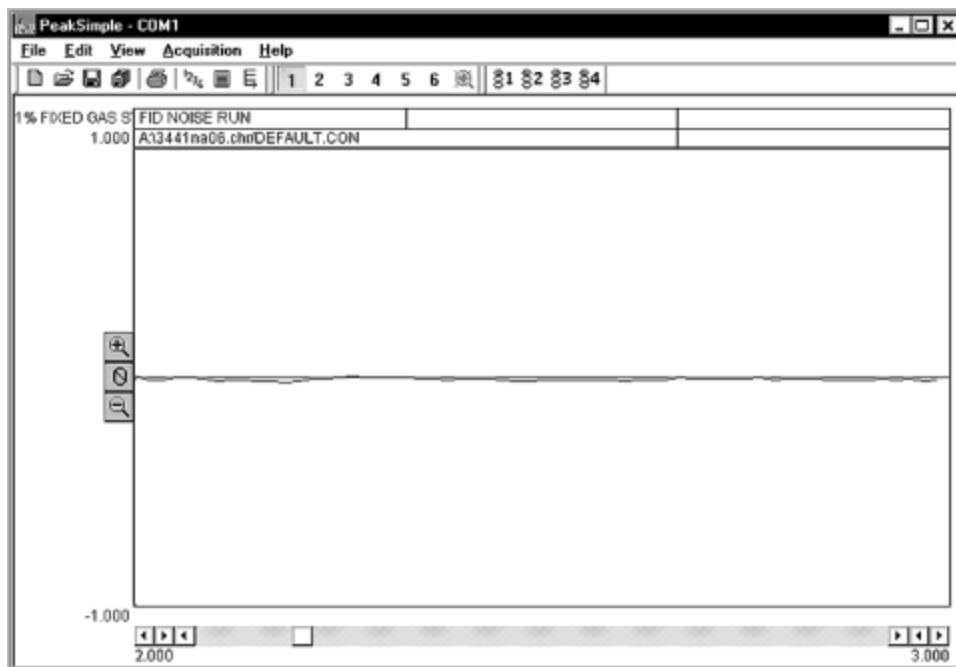
The START RUN button is on the lower right hand corner of the GC's front control panel.

## 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

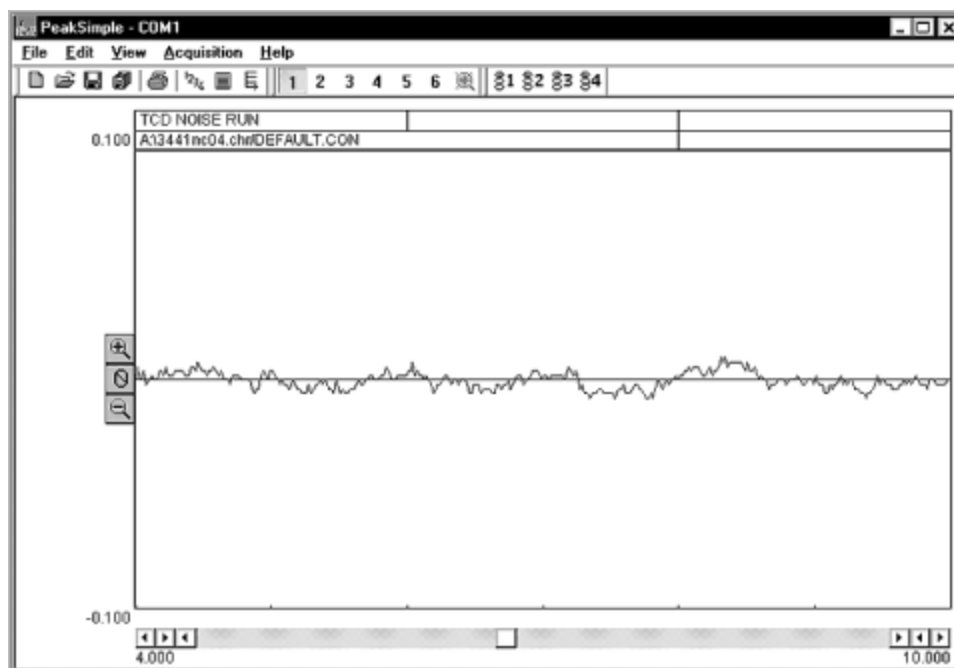
FID gain = HIGH  
FID temp = 380°C  
FID ignitor = -400  
Methanizer in FID  
detector body

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

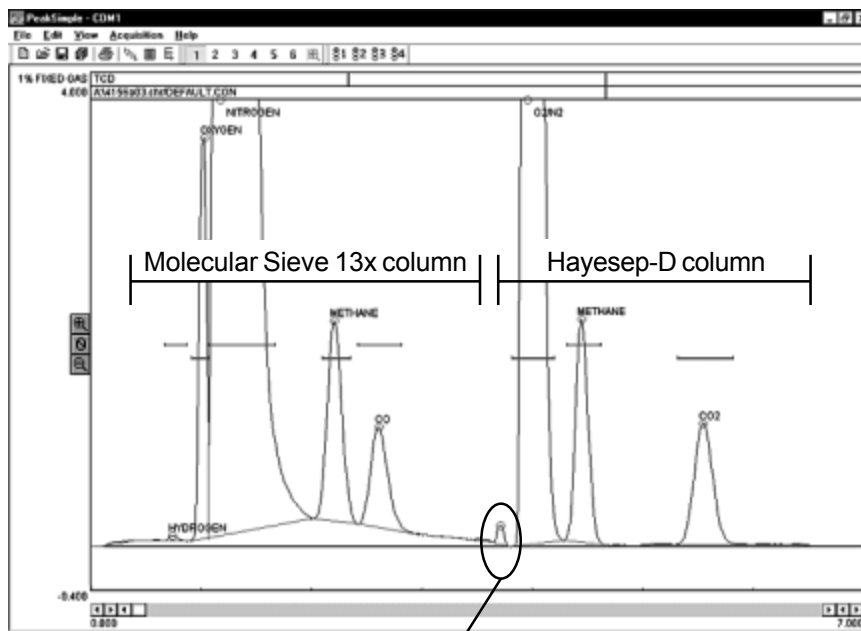
Temperature program:

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

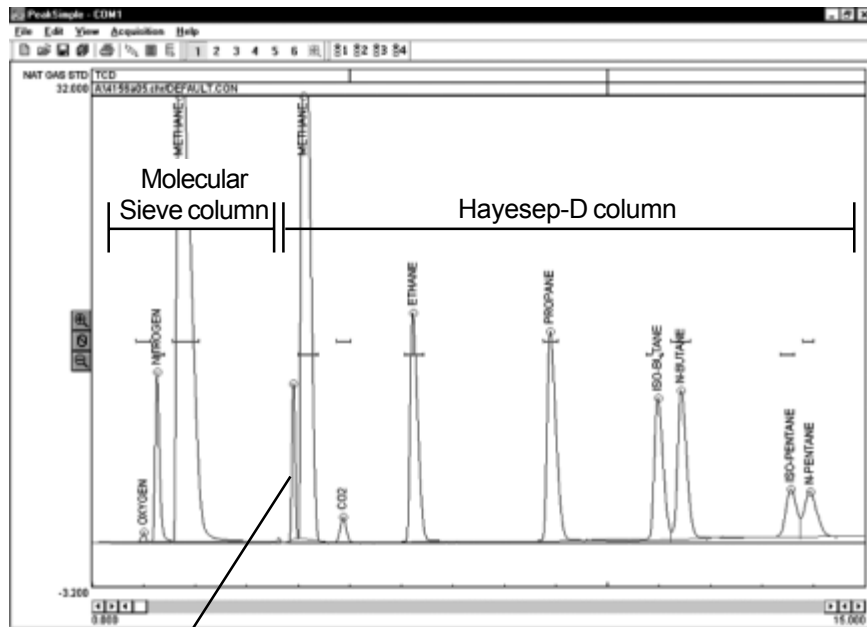
#### 1% Fixed Gas Standard

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 <sub>2</sub> /N <sub>2</sub> Hay-D	3.950	863.6340
Methane Hay-D	4.433	15.7300
CO <sub>2</sub> Hay-D	5.533	12.9205
<b>TOTAL</b>		<b>2043.5950</b>



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 <sub>2</sub> 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
<b>TOTAL</b>		<b>2099.1045</b>





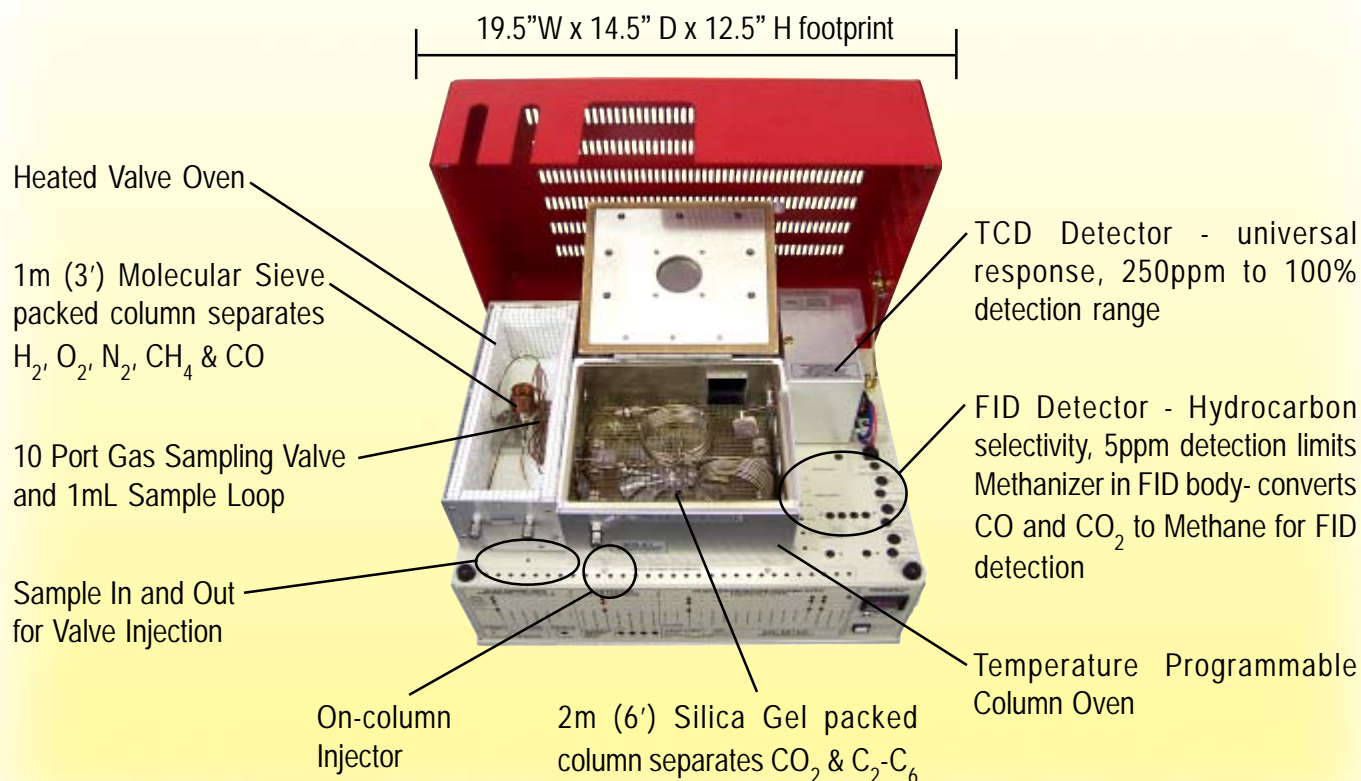
## Multiple Gas Analyzer #1

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

*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!*

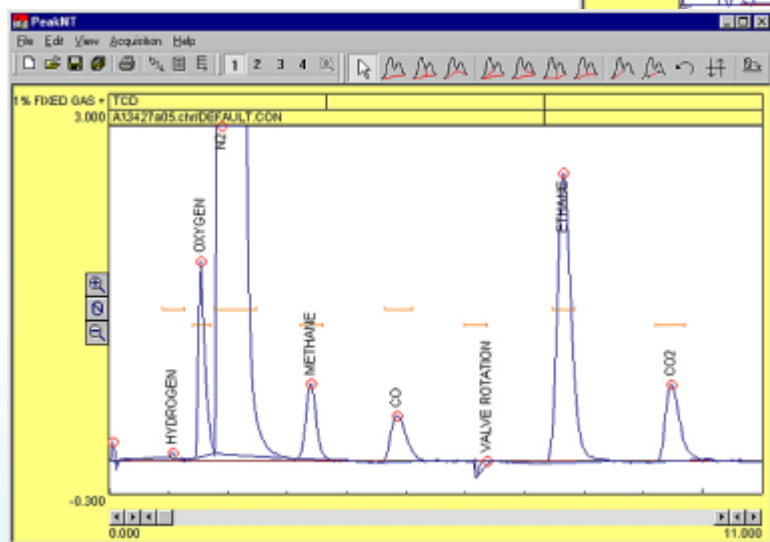
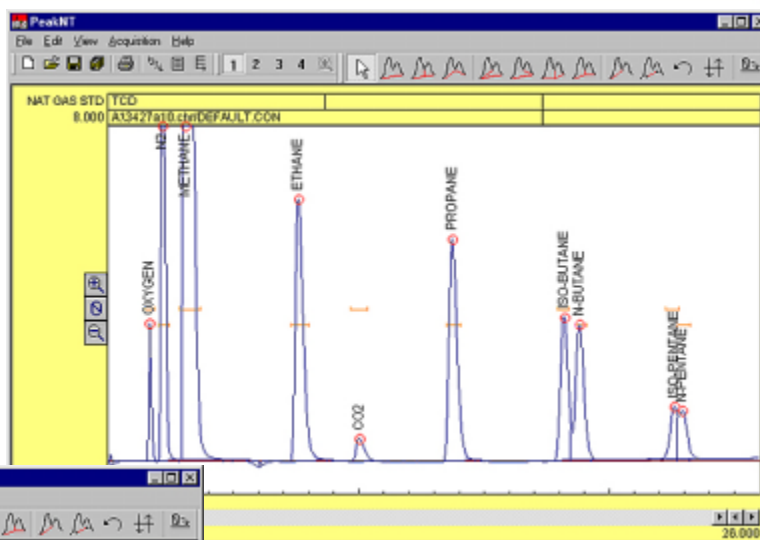
- ★ Separates multiple gases with a single injection
- ★ Very tolerant of user adjustments and timing variations
- ★ Simpler than other multi-gas capable GC systems
- ★ Multiple gas analysis in a compact unit



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

## Multiple Gas Analyzer #1

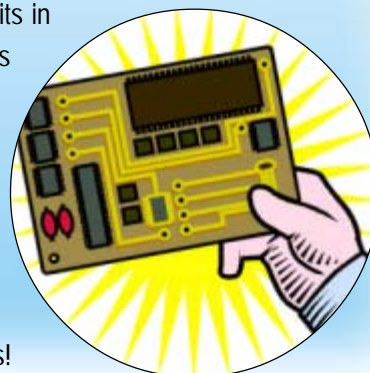
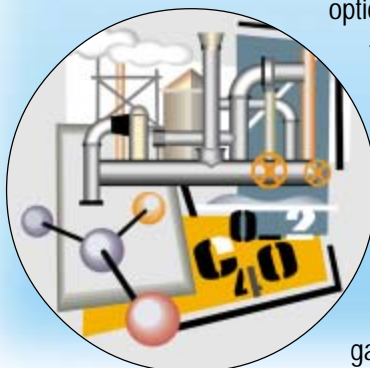
*Separating out the hydrocarbon components of natural gas facilitates accurate BTU quantification. This compositional analysis of a natural gas standard by an SRI Multiple Gas Analyzer #1 shows good separation up to the pentanes. Performing compositional analyses of natural gas product before and after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

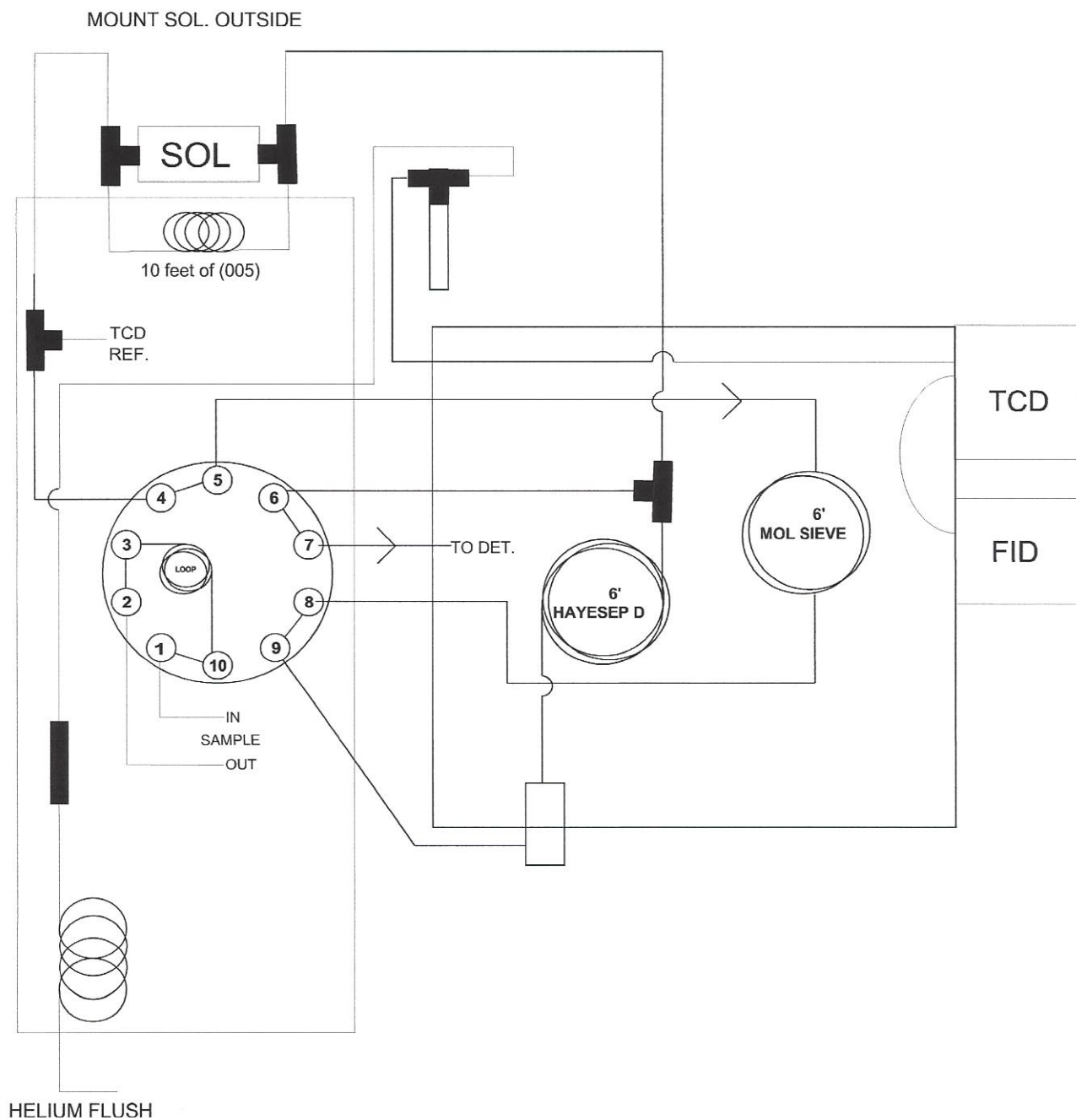
The basic Multiple Gas Analyzer #1 has a TCD detector only; this model provides analyses in the 250ppm to 100% range for fixed and natural gases. A second option is a TCD, Methanizer, and FID detector combination which adds 5ppm detection limits for CO, CO<sub>2</sub>, and all hydrocarbon peaks; this 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<sub>2</sub>-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





# SRI GCs H2-100 2020

## OPERATION MANUAL

QL-150/300/500 Hydrogen Generator

Shandong Saikesaisi Hydrogen Energy Co.,Ltd.

JINAN,SHANDONG PROVINCE, CHINA



Our Website(s) > [www.chromtech.net.au](http://www.chromtech.net.au)  
[www.chromtech-AUS.com](http://www.chromtech-AUS.com) > mobiles & responsive / autosize  
NEW [www.chromalytic.net.au](http://www.chromalytic.net.au) > still under development



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2. Operational principle and technological process.....	1
3. Electrical control.....	2
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5. Structure of the Generators.....	4
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**Dear Clients: Please read carefully the Operation Manual prior to operation.**

**Juveniles and those who do not understand the requirements of the manual cannot operate the generators .**

## **Operation Manual for QL Series Hydrogen Generators**

### **1. General description**

The data and operating requirements stipulated in this Operation Manual are applicable to all the QL series hydrogen generators .

QL series hydrogen generators are advanced patented products, which are light , highly effective , energy-saving and of environmental protection , producing extremely pure hydrogen through the electrolysis of pure water (without adding alkali).

The SPE electrodes, as the core of the product , are highly active catalytic electrode with nearly zero distance between the electrodes , which is formed by integrating composite catalyst with and ion membrane with high electrolytic efficiency. The other key parts are all produced by use of top-grade engineering plastics dies with superior quality . With perfect electric control system, designs of the generators are advanced with reliable quality, high automaticity, extremely pure generated hydrogen, huge output , the models and specifications of the generators are complete , and the generators are widely used . The small-sized generators are ideal equipment for all kinds of gas chromatographs and thin-film chromatographs, and the large-sized generators can be used in hydrogenation process of chemical industry and pharmaceutical industry, gas reduction protection of electronic industry, purification of semiconducting materials, metal welding, smelting and purification of heavy metals, surface protection of metals, water decomposition and composition in spacecrafts and submarine, and concentration of heavy hydrogen in atomic energy industry, etc. The products can absolutely take the place of hydrogen steel cylinders with safety and convenience in operation.

### **2. Working principles and technological process**

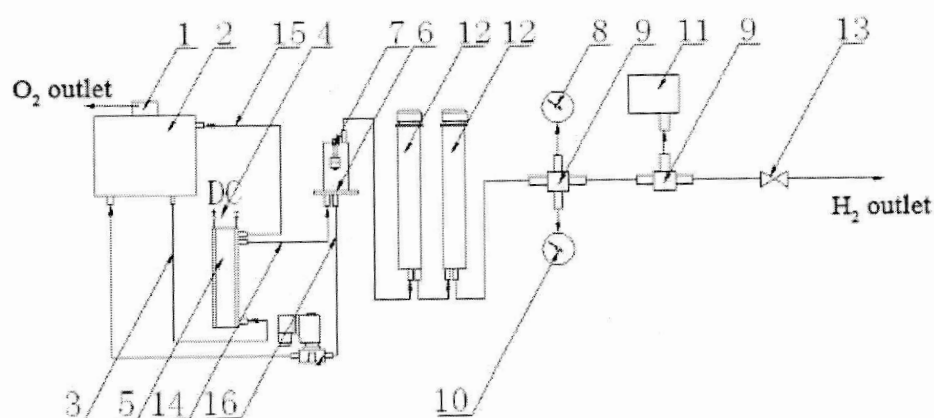
For the technological process, please refer to Fig.1.

Electrolytic water meeting the requirements (With electrical resistivity  $>1\text{M}\Omega / \text{cm}$ , and deionized or redistilled water in electronic and analysis industries can be used for this purpose.) , after being put into the anode chamber of electrolytic cell, when power is switched on ,will be decomposed at once at the anode :  $2\text{H}_2\text{O} = 4\text{H}^+ + 2\text{O}^{2-}$ . The decomposed Hydroxide ions ( $\text{OH}^-$ ) will immediately release electron to form oxygen ( $\text{O}_2$ ), which will then be discharged from the anode chamber, with some water, into the water tank. The water can be used circularly, and oxygen will be discharged from the small hole of the top cover of the water tank into the atmosphere. The hydrogen proton, in the form of aqua ion ( $\text{H}^+ \cdot \text{XH}_2\text{O}$ ), and under the action of electric field force ,through Proton Exchange





Membrane(PEM), will arrive in the cathode to absorb electron to form hydrogen ,which will then be discharged from the cathode chamber into the gas/water separator, where most of water it brought with from the electrolytic cell will be removed . The hydrogen with little water will be under moisture absorption of the desiccator, with its purity thus reaching 99.999 % or above. When the condensed water in the gas/water separator is accumulated to a certain quantity, it will raise the float and be discharged from the outlet at the bottom of the gas/water separator into the water tank for recycling. The float, after the discharging, will return immediately to its original position, and the water level of the gas/water separator thus remains constant.



**Fig. 1 Technological Process Schematic Diagram**

- |                                  |                                  |                   |
|----------------------------------|----------------------------------|-------------------|
| 1. Top Cover of Water Tank       | 2. Water Tank                    | 3. Feed Pipe      |
| 4. Power Supply for Electrolyzer | 5. Electrolysis cell             |                   |
| 6. Gas/water Separator           | 7. Float                         | 8. Pressure Gauge |
| 9. Cross Joint                   | 10. Pressure Controller          |                   |
| 11. Overpressure Protector       | 12. Desiccator                   |                   |
| 13. Gas Discharge Valve          | 14. Hydrogen/Water Outlet        |                   |
| 15. Oxygen/Water Outlet          | 16. Condensate Water Return Pipe |                   |

### 3. Electrical control

For the electrical principles of a complete set of generator, please refer to Fig. 2. The whole electrical system is mainly composed of four parts : a power supply system for electrolysis, main control subcontrol and a display panel. When the power switch SW1 is pressed , the generator will go into operation. In the course of electrolytic process, when air pressure reaches the preset value, the pressure transducer SEN will start to take control to

make electrolytic current decreasing along with rising of air pressure, thus enabling output of the generated hydrogen , under the stable pressure, to meet the demand of the consumption automatically .

In addition, the generators, for ensuring normal operation, are equipped with two alarming protection systems .

### 3.1 Overpressure Alarming

If the output pressure is out of control and rises to 0.46 MPa because of being strongly shaken or something is wrong with its certain parts in the course of operation , the generator will beep four times with an interval and automatically cut off the power supply for electrolysis and stop the electrolysis for realizing the overpressure protection . At that time the front board will show that hydrogen output is zero with pressure alarming light (red) on . End-users should release the pressure and restart up the generator after ensuring that power connection is good with no shaking . If the above-mentioned phenomenon still reoccurs , it can be considered as an failure, end-users should inform the manufacturer for maintenance.

### 3.2 Water-level Alarming .

If water level in water tank during operation drops down to the minimum limit or long-time operation of the generator under zero output pressure causes ponding in the gas/water separator to rise to the maximum limit , the generator will beep for alarming once every six seconds approximately and stopping electrolysis. End-users should switch off the generator to find out and clear the faults .If the water level in the water tank is normal and output pressure of the generator remains over 0.02 MPa , it can be considered the alarming is not caused by the above-mentioned two factors , and the manufacturer should be informed for maintenance .

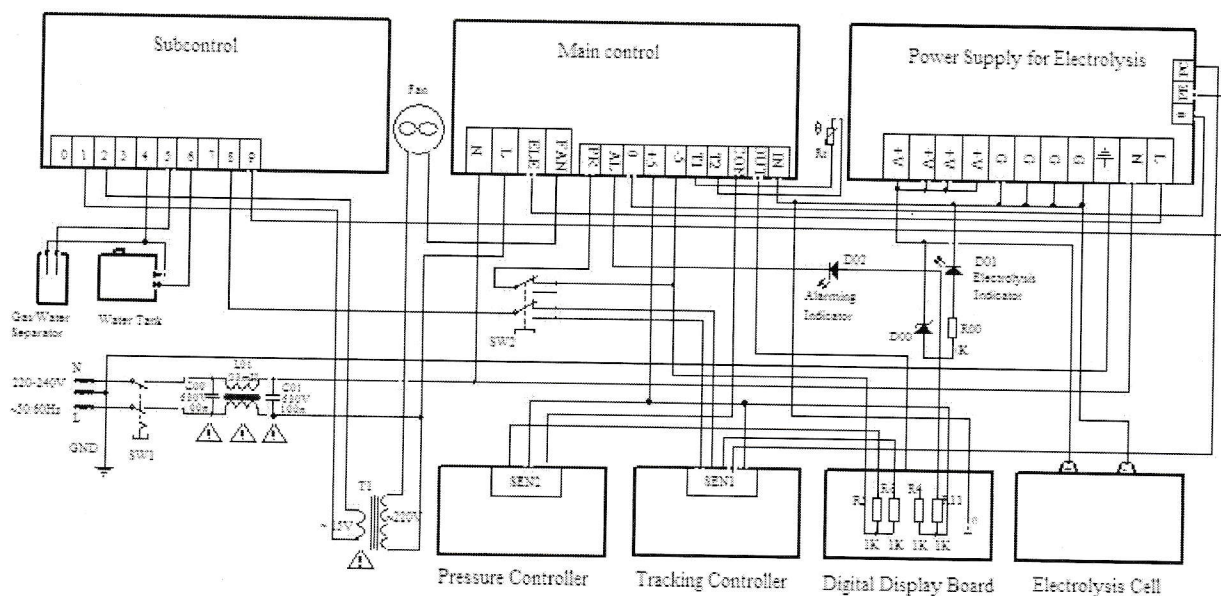


Fig. 2 Control Schematic Diagram

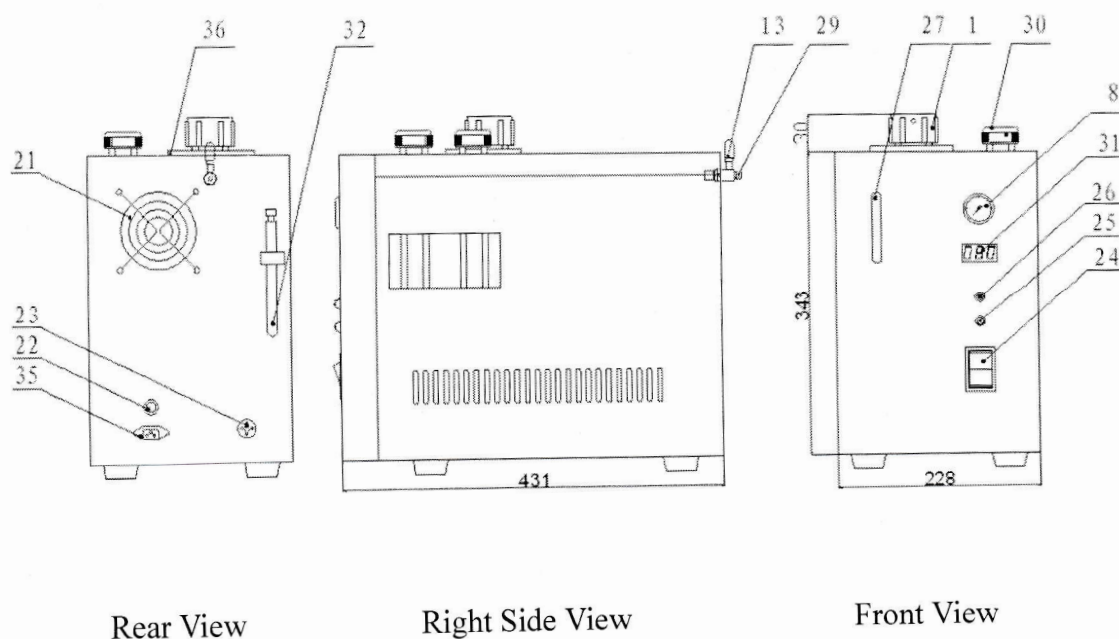
## 4. Technical parameters

Specifications Parameters	Models	QL-150	QL-300	QL-500
Output Flow Rate (ml/min)		0-150	0-310	0-510
Output Pressure (MPa)		0.02—0.4(Output under stable pressure )		
Purity of Hydrogen (%)		> 99.999		
Pressure Value for Overpressure Protection (MPa)		0.46		
Power Voltage (V)		110±15% ~ 50 ---60 Hz		
Input Power (W)		< 90	< 150	< 300
Net Weight of a Complete Set (Kg)		< 15	< 15	< 16

## 5. Structure of the Generators

### 5.1 Contour of Hydrogen Generator

For the contours and dimensions of hydrogen generators, please refer to the front view, side view and rear view in Fig. 3.



**Fig. 3 Contours and Dimensions Of Hydrogen Generators**

### 5.2 Internal Structure of Hydrogen Generators



For the internal structure of hydrogen generators, please refer to the following Fig. 4 and 5

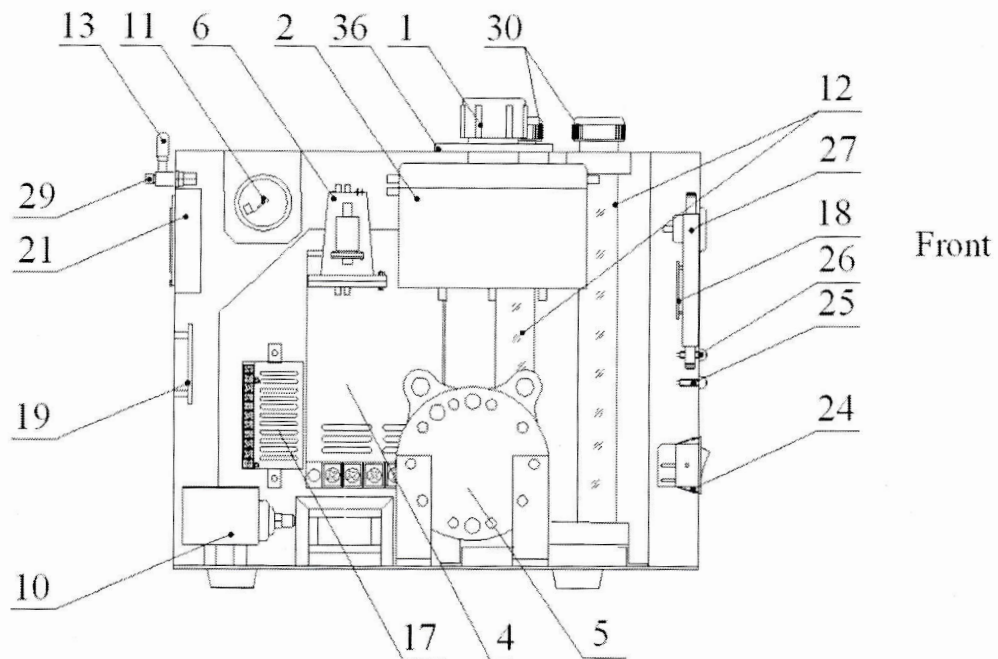


Fig. 4 Left Side View

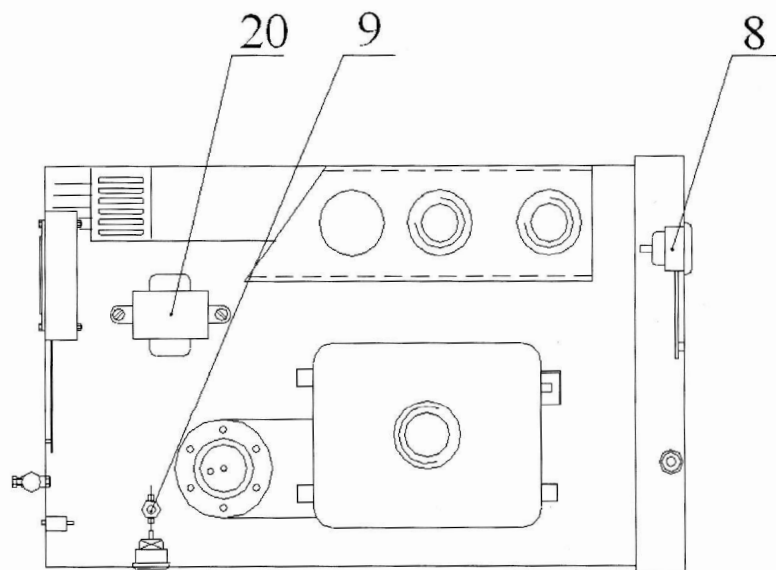
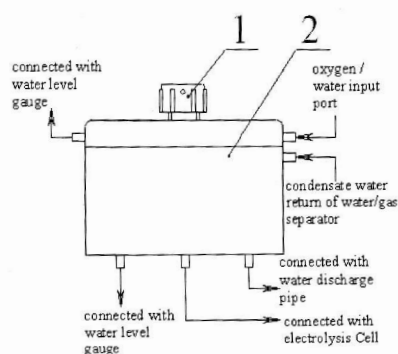


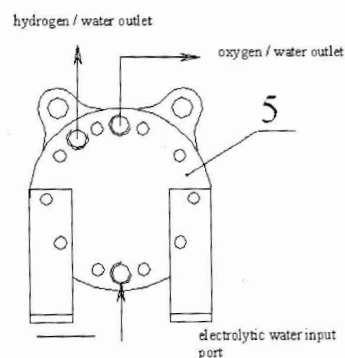
Fig. 5 Top View

### 5.3 Key Parts of Hydrogen Generators

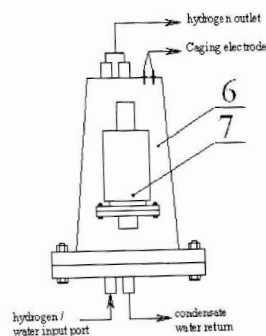
## 1. Water Tank



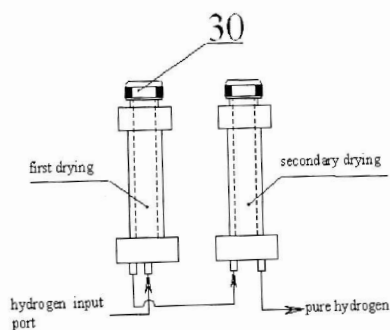
## 2. Electrolysis Cell



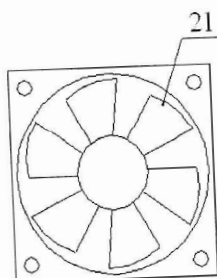
## 3. Gas/Water Separator



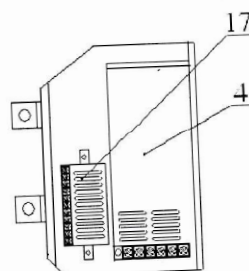
## 4. Desiccator



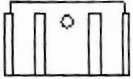
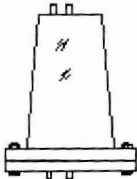
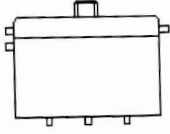
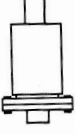

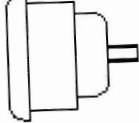
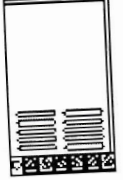
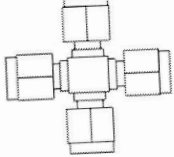
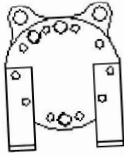
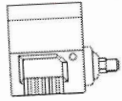
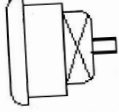

## 5. Electric Fan



## 6. Power Control Board



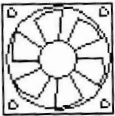
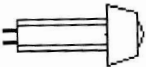


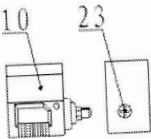



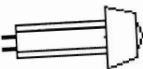
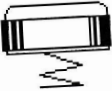
## 5.4 Main Parts General List for the Hydrogen Generators

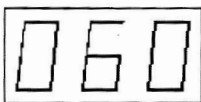

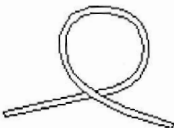

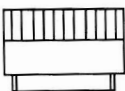
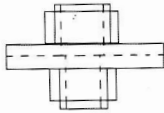
Serial No.	Description	Profile	Serial No.	Description	Profile
1	Top Cover of Water Tank		6	Gas/water Separator	
2	Water Tank		7	Float	
3	Feed Pipe		8	Pressure Gauge	
4	Power Supply For Electrolysis		9	Cross Joint	
5	Electrolysis Cell		10	Pressure Tracking Control	
Serial No.	Description	Profile	Serial No.	Description	Profile
11	Overpressure Protector		16	Condensed Water return pipe	



SHANDONG JIANGSAILI HYDROGEN



Serial No.	Description	Profile	Serial No.	Description	Profile
21	Electric fan		26	Electrolysis Indicator	
22	Fuse		27	Water Level Gauge	
	Cartridge Fuse	F3AL250V			
23	Select Switch For Pressures		28	Power Cord (Outside matches)	
24	Power Switch		29	Pure Hydrogen Outlet Joint	
	1-turn on 0-turn off				
25	Alarm Indicator Light		30	Nut for Desiccant Pipe	

Serial No.	Description	Profile	Serial No.	Description	Profile
31	Flow Rate Display Screen		34	Internal Pipe For Drying	
32	Drain Pipe		35	Socket for Power Supply	
33	Nut for Internal Desiccant Pipe		36	Overflow	

## 6. Operational Requirements ▲

**6.1** Juveniles and those who do not understand the requirements of the manual cannot operate the generators .

**6.2** The hydrogen generators are forbidden to be operated in a sealed room.

Require customers making use of hydrogen gas no more than 2/3 of the maximum output flow. If need long time working( more than 10 hours continually once), using output flow is much better no more than 1/2 of the maximum flow.

**6.3** Requirements for operational environments and conditions of the generators :

- Temperature : 4℃– 40℃ ;
- Humidity : <85% ;
- Power supply : 220v--240V~50-60Hz or 99-121v~50-60Hz;
- The generators should be put horizontally near hydrogen-applied instruments with their front boards facing operators for the convenience of operation ;
- There should be no obvious shaking and striking ;
- There should be no direct sunshine and open fire ;
- There should be no big dust , conducting particles , acid , alkali , and other corrosive gases ;

- h. Ventilation should be good ;
- i. Ground connection of power supply should be good .  $\perp$

**6.4 The water tank of a generator should at first be filled with deionized or redistilled water and then you should wait for five minutes prior to startup.**

**6.5 Requirements on pressure rising and how to deal with hydrogen produced when the set pressure is reached without connecting hydrogen-applied equipment .**

(1) During operation of the generators , operators are not allowed to look down at the sealed top caps of the two-stage desiccators from above in order to prevent eyes and faces from injuring .

(2) The generators cannot be operated when the output pressure is zero , and the minimum pressure for operation is 0.02 MPa . After start-up the pressure should be raised before it is too long , or it will lead to internal ponding without normal draining of gas/water separator, making the water level reach the maximum limit of alarming, resulting in stopping electrolysis . When output of hydrogen reaches the maximum value , time of the operation with zero of output pressure should not be over 10 minutes . ( Generally speaking , after the generators are delivered some internal resistance will be produced since the generators have been equipped with two-stage desiccators and brass pipelines connecting hydrogen output flow direction , and generally speaking , the internal resistance can reach the required limit of the minimum pressure .)

(3) After start-up a generator is not allowed to be operated for a long time when the set pressure is reached without connecting hydrogen-applied equipment , or it will damage the core component of electrolytic cell .

**6.6 Regulation of constant output pressure value**

Output pressure can be regulated only within the range of 0.02~0.4MPa by pressure regulator. If output pressure exceeds the range an mentioned about the pressure regulator can not work normally or will give an alarm. The pressure regulator locates at the right bottom of the backboard, and marked with the word "High/Low"

**6.6.1 Upward regulation of constant output pressure value**

On starting up, hydrogen output pressure will reach the factory-set value, and the output flow will be kept at about half of the rated value. Then, use a "+"-shaped screwdriver to insert into the "+" slot of the regulator to turn counterclockwise by a minute angle (less than 30 degree). Hydrogen output flow rises first, then it will drop gradually. When hydrogen output flow keeps stable, regulate it once again until obtaining a required value.

**6.6.2 Downward regulation of constant output pressure value**

Turn the pressure regulator clockwise by a minute angle (less than 30 degree). Hydrogen output flow drops first, then it will rise gradually. When hydrogen output flow keeps stable, regulate it once again until obtaining a required value.



**6.7 The pressure of a hydrogen generator should be released to zero after it is shut down .**

The pressure can be released by loosening the nut of venting valve in front of the hydrogen outlet on the backplate. The valve should be sealed again after the pressure is released .

**6.8 Requirements for Water Quality**

**6.8.1** As hard ions in unqualified water may cause sediment to block pores of electrodes, thus resulting in scrapping of the electrodes, the electrical resistivity of soft water (deionized or redistilled water ) in electrolysis should not be less than  $1\text{M}\Omega/\text{cm}$ . All end-users should keep it in mind, otherwise they should be responsible for all consequences.

**6.9 Water Level Requirements for Water Tank**

(1) The water level should be over two thirds of the volume of the water tank (volume of water tank is 3.2 L).

(2) Water should not be poured into the water tank violently and quickly to prevent water from spilling out of the nylon overflow , under which there is an O ring sealing the housing of the generator to prevent water from entering the generator to damage the electric components .

**6.10 Requirements for Changing Water and Cleaning Water Tank**

The water tank shall be kept clean. Even soft water will breed microorganisms and become turbid when it is used for a long time, influencing output of the hydrogen and the service life of electrolysis cell. The water tank, therefore, should be emptied through the drain pipe every two or three months, and then washed several times (fill the tank with a small quantity of new water and shake the generator lightly several times in every direction) until discharged water becomes transparent without cottony things.

The drain pipe of water tank is attached to the backplate of the generator and can be freely taken off and put in.

The small hole on the top cover of the water tank is used as oxygen discharging port, therefore do not block it , and the top cover of the tank should not be changed at random.

**6.11 During transportation of the generators, the water tanks should not store water so as to prevent the water from spilling out to cause damages of the electrical components.**

**6.12 The electrolytic cells should not be short of water .**

**6.13 Requirements and Methods for Replacing Desiccant**

The QL hydrogen generators manufactured by our company feature less internal resistance, high electrolytic efficiency, and extremely small consumption for transforming electric energy to heat energy , therefore the service life of desiccant (silica gel or molecular sieve) is the longest among all hydrogen generators in the market up to now. If the

phenomena contrary to the above-mentioned occur or the color of desiccant changes in a large proportion, it is possible that the generator has been in operation for a long time at full capacity and with a huge output . When you correct the above-mentioned improper operations and the color of desiccant still changes very fast , the manufacturer should be informed for maintenance .

### 6.13.1 Introduction on Replacing Desiccant

The desiccator is located inside the right plate ( facing the front board look backward it is on the right side ) equipped with a desiccant view port. The drying procedure is divided into two steps with blue silica gel as the first drying and incarnadine molecular sieve as the secondary drying respectively.

The top caps covering the two desiccant cartridges protrude outside the top cover of the generator, and arranged in a single row, with the same way for their replacements.

### 6.13.2 Requirements for replacing desiccant

(1) The desiccant should not be replaced during the operation of the generators to prevent high-pressure hydrogen from leaking and injuring people .

(2) Before desiccant is replaced , a generator must be shut down and the pressure must be released prior to unscrewing and opening top cap of the purifier .

(3) The desiccant ( discolored silica gel or molecular sieve) will lose effectiveness after water uptake to saturation, and should thus be replaced on time . When the height of discolored silica gel is over half of that of the view port, it must be replaced ! Or the water content in hydrogen will be more than the standard , influencing the purity of generated hydrogen . The molecular sieve for the secondary drying might be replaced once half a year or a year provided that the silica gel for the first drying is replaced in time

(4) The generator, after desiccant replacement, should be in idle operation for several minutes to wait for air to be emptied from the desiccant cartridges ,and hydrogen can only be used when purity of generated hydrogen reaches the standards .

### 6.13.3. Methods for Desiccant Replacements

(1) Turn off the generator and release the pressure, and unscrew manually the top cap covering the first desiccant cartridge (turn counterclockwise for opening it ). The top cap should be put there unpolluted, and the seal ring in the cap should not be discarded. The spring fixed in the top cap shall not be removed, nor polluted.

(2) With clean fingers nip the top cap of the internal cartridge with desiccant and lift it , and unscrew the top cap of internal cartridge (turn counterclockwise for opening it ) to empty the desiccant . Wash the cartridge with distilled water and dry the cartridge (by blowing or airing). Refill the new or regenerated desiccant into the cartridge, and screw the top cap and put the cartridge into its original place. **You should pay attention to the following two points during the procedures: a. The protruded head at the bottom of the internal cartridge should be seated in the cavity of the outer cartridge base! b. The O-rings in**

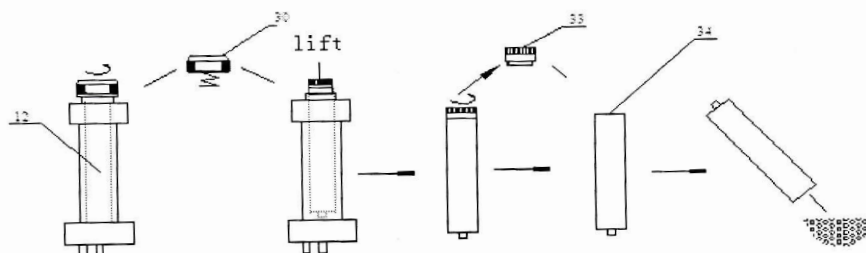




the protruded head at the bottom of the internal cartridge should not be lost ! If damaged, it should be replaced with the new one from the attached accessories. The purpose of attention to the two points is to guarantee that the hydrogen can go through the desiccator according to the required drying route , so as to guarantee purity of the hydrogen .

The methods for replacing the secondary desiccant are the same .

(3) Finally, securely screw and seal the top cover of the desiccator.



**Fig.6 Schematic Diagram on Desiccant Replacement**

#### (4) Desiccant Regeneration

- ① The silica gel should be baked under the temperature between 120 and 140 °C until its color changes into blue completely .
- ② The molecular sieve should be baked under the temperature between 150 and 180 °C for 2 hours.
- ③ The above-mentioned desiccant should be packed for use when it is dried and its temperature decreases to below 50° C , and it will scald skin if its temperature is too high . Too high temperature will scale the containers or skin of operators .

**6.14 A generator must be shut down before it is repaired . During operation of a generator , do not disassemble housings and other components of the generator in order to avoid an electric shock .**

**6.15 A generator must be shut down by power cord disconnect with power source before cartridge fuse is replaced . Model of cartridge fuse used in the generators of QL-150,QL-300 and QL-500 is F3AL250V , please do not make mistakes in using the cartridge fuse so as to avoid fires .**

## 7. Acceptance Check

### 7.1 Unpacking for the acceptance check

There are safe transportation marks on the surface of the packing boxes for the generators with some damp-proof and shockproof materials inside the boxes . The generators should remain intact if no accident occurs during the transportation , otherwise claims should be filed against carriers in accordance with the actual conditions .



**7.2 The attached accessories and technical documents should be checked according to the packing list.**

### **7.3 Operation for the acceptance check**

**7.3.1** The operational environment and conditions of the generators should satisfy the requirements stipulated in 6.3 .

**7.3.2** Open by hands the top cap of the water tank at the top of the generator, and remove the plastic film from the top cap . The water tank must be filled with deionized or redistilled water , and the other kinds of water will damage electrodes , resulting in damage of electrolytic cells . Water level in the tank should be between the minimum and maximum water level lines, and then screw the top cap . **Caution : The generators must be filled with water prior to startup ! Water must be guaranteed for electrolytic cells !**

**7.3.3** Connect source of power supply and the generator by using power cord along with the delivered generator , and insert the end of power cord in the socket ( Serial No. is 35 ) on the back panel of the generator , and then insert the plug of power cord in the socket of the power supply board . Ground connection of power supply must be good according to the requirements of 6.3 of the operation manual . After connecting the power cord , turn on the switch of power supply on the front panel . Both the power supply indicator and the electrolysis indicator (green) will be on, and the number indicating the output of hydrogen will be increasing all the way to the maximum output of the generator.

**7.3.4** When the nut for hydrogen outlet of the generator is sealed ( or screwed) securely, output pressure will rise, and when the pressure reaches the preset value, the pressure control system will take control to make the electrolytic current reduce to zero, and the number indicating output flow rate will show falling to zero. The number indicating output flow rate will show returning to the maximum of generated hydrogen when the above-mentioned nut is unscrewed ,which indicates the generator is in normal operation. After the acceptance check is completed , the generator can be operated in accordance to the Operational Manual.

### **7.3.5 Connecting hydrogen-applied equipment**

After acceptance check , at first shut down a generator when you are prepared to use it . Produce the pipe ( a brass coil pipe with outer diameter of 3 mm , wall thickness of 0.5 mm and length of 1,500 mm ) connecting hydrogen-applied equipment from an accessories bag along with the generator . When a generator is delivered , the two ends of the pipe have been equipped respectively with an international standard threaded nut of M8×1 and three O-rings for sealing . Distance between each end of O-rings and that end of the brass pipe is 6-8mm . The one end will connect outlet port of a generator , and another end will connect a set of hydrogen-applied equipment in the same way . If nozzles of hydrogen equipment are measured in the British system , for example , chromatographs made by Shimadzu of Japan , Agilent and Varian of USA , whose sizes of nozzles are measured in the British system , We can accessorize relevant nozzles according to different requirements of our customers and will mark out on the accessories bags of nozzles .

Insert the two ends of a connecting pipe into the relevant nozzles , and use a spanner



delivered along with the generator to seal the nuts and nozzles in clockwise sense . Do not overexert yourself in sealing the nuts prevent the O-rings losing elasticity , resulting in influencing the sealing effect .

After hydrogen-applied equipment is connected , use soap suds to check the sealing for leakage . If bubbles occur , the assemble should be readjusted until no leakage is found . The same method should be used for checking leakage in sealing nuts of the other parts and components .

## 8. Troubleshooting

**Warning:** The qualified person can go into the maintenance and repair of the generator only. Draw the power cord plug off from the power supply socket before any working of maintenance and repair, to avoid electric shock.

Breakdown	Causes	Guide for maintenance
1. When the power switch is turned on ,the power indicator light will not be on and the generator will not be in operation.	1. The power plug is in poor connection. 2. Blown fuse. 3. Power switch is damaged.	1.Recheck the plug and make it in good connection. 2.Take out and replace the damaged safety wire in the fuse. The replaced safety wire must be up to the type of the original safety wire. Do not change type of safety wire at will. 3.Repair or make replacement.
2. When the electrolysis indicator light is on with the maximum output generated , the pressure does not rise.	1. Leak in the pipe system. of hydrogen. 2. Poor sealing condition at the float of the gas/water separator with water outlet. 3. Sharply increasing output from the oxygen outlet means electrolysis cell has been damaged.	1.Use leak-hunting liquid to check sealing of all nuts, screw securely the fittings at the leaks. 2.Repair or replace the fittings. 3.Shut down the generator at once !The electrolysis cell, if damaged, must be returned to the manufacturer for replacement. Do not disassemble it by yourself, or you will be responsible for all the consequences arising thereby.



3. Beep four times with an interval , alarming light is red.	<p>Disconnected in the socket, and the contact is faulty.</p> <p>When power is turned off ,it is switched on immediately.</p> <p>The generator has been shaken.</p> <p>Mistakes made in selecting pressure.</p>	<p>1.Check,make current and restart the generator.</p> <p>2.Wait for 6 minutes after shutting down the generator prior to restart-up.</p> <p>3.Remove shaking and release the pressure prior to restart-up.</p> <p>4.Restart up after releasing the pressure.</p> <p>If the system still beeps after doing according to the above-mentioned , inform the manufacturer for maintenance, do not disassemble it by yourself.</p>
4. Beep once every six seconds approximately.	<p>Water tank is short of water.</p> <p>Ponding in the gas/water separator.</p>	<p>1.Add some water to the water tank.</p> <p>2.The generator has been operated for a long time with zero pressure or there are leaks in the pipe system .If the system gives an alarm when the pressure is over 0.012 MPa, it is a breakdown of the generators, inform the manufacturer for maintenance.</p>
5. If water is found to drain out of basal crack of the generator ( The problem is rarely seen ).	<p>1. Silicon rubber soft pipe and nylon ribbons are aging . The sealing O-rings between the metallic pipe and nuts are aging.</p> <p>2. The sealing pad of electrolytic cell is aging.</p>	<p>1. Shut down the generator and release the pressure of hydrogen . After draining off water from the draining pipe on the back board , it will be ok to replace the same accessories . ( Water used by the generators is not corrosive , so rub out the spilled water in the housing and use an air blower to blow it dry before restarting up the generator.</p> <p>2. The manufacturer will be responsible for repairing breakdown of electrolytic cells or making replacements.</p>

## 9. After-sales service

The warranty period of the generators is one year, and the maintenance will be lifelong . Maintenance and replacement of parts within the warranty period will be done free of charge, and beyond the warranty period, they will be done with only cost of the raw materials charged.

**If the following occurs , the maintenance will not be done free of charge:**

- users do not operate the generators according to the operational manual;
- users disassemble parts by themselves , which are forbidden by the manufacturer to be disassembled.

**Our company is able to undertake maintenance of all hydrogen generators with the SPE/PEM technology at home or abroad.**



# GC Tutorials : & U-Tube SRI 8610C

## Chromalytic > Tips for beginners

**NEVER use soap solution, Snoop etc for checking for leaks on fitting**

Check outlet for bubbles if possible by dipping detector pipe outlet in water

- use an electronic Leak Detector ( eg Restek – expensive)
  - Always start carrier gas wait >5 minutes at > eg 10ml/min, AND check for flow at column outlet.
    - BEFORE turning on the TCD Filaments
  - TCD may take 5 minutes to stabilise say at 150degC for gas analysis generally
    - SRI 8610C has a safety feature pre-set safety thresh-hold 1-2psi below which the TCD filament wont turn-on > oxidation / burnout of filaments WILL OCCUR instantly
- NOT fool-proof !- if column outlet leaks or is disconnected > NO FLOW thru TCD despite the positive pressure on the column inlet BUT pressure on the Inlet EPC could still be > pre-set 1-2psi and the TCD protection is then non function
- > a RED light WARNING on the GC Front Panel indicates This !

### To shut down the GC

Allow column oven to cool down to < 100degC

Then turn off GC TCD filaments

THEN You can turn off All gases

Otherwise column liquid phases deteriorate more quickly

### Column life is dependent >

- on average temperatures being used at but also on oxygen content of the column carrier gas
- TCD filaments can burn out > expensive to repair !

but also on water content and reactivity of sample components

#### • Column Separation

- Max separation efficiency at optimum gas velocity ( Column Length /air peak time ( cm/sec )  
: N2 15-20m/sec He 20-35, H2 30 to 50cm/sec.
  - increasing flow rate will decrease separation efficiency. Each Column Liquid Phase has its own unique Max Operating Temperature Packed Columns >> capillary columns.
- Use High Purity Carrier gas He or Hydrogen generally > 99.995% especially at High Temperatures eg 300degC .

but also any columns >70 degC recommended. A High drifting detector background signal near the Max Column Op temp expected often indicates liquid phase oxidation and bleed.

### Column Temperature Limits - Gas Analysis

**Silica Gel** 200degC

**Mol Sieve** 300degC - but CO2 DOES NOT elute

**HayeSep D** ~180degC ( Note other HS columns are of different polarity and limits ) bleed occurs if carrier gas is impure ( >99.99% is needed ! )

other phases ? > stick strictly to column OEMs recommendations . . . particularly for capillary columns !



### **Precaution >**

for "Trace Gas Analysis ( & <100ppM )

- use a High Capacity Oxytrap on the carrier gas inlet and close to the GC; NOT at any cylinder manifold except as a back-up
- Do NOT rely on a closed trap as they can & do expire quickly when changing cylinders for example
- use Isolation valves for the GC to prevent back diffusion up the GC Columns system when on GC / idle / standby etc ) AND on the trap once opened to air - a 'slug of air "will quickly deactivate the Oxytrap adsorbent
- Indicating Oxtrap - as an in-line back-up, this ensures there are no leaks to prevent back diffusion of Air from the atmosphere Any deep pockets / blind spots in the GC column plumbing does take time to diffuse out after the GC has been shut down, WAIT 5 to 10 minutes to diffuse this air out . . . On restarting !
- otherwise oxidation if inner tube surfaces and stationary phase will cause over time troublesome column bleed and drifting baselines . . . On ANY type of columns > limiting limits of detection

### **DON'T FORGET**

When the SRI RED GC Lid Is UP during minor maintenance > parameter resets ALL Hot Temperature Zones are TURNED OFF , but take time to cooldown and before restarting again

### **Injection**

Gas Samples inject via the auto Gas Sample Valve for consistency

But ensure you purge the GSV loops ( normally 1cc with at least 3x that volume

. . . & just before the injection

- Better still for convenience use the Vacuum Pump Interface device for doing multiple injections of the same sample or auto-injection of multiple field samples use the 10-Port Auto Gas Valve Injections Manifold as an option for the SRI 8610 MG#5 GC.

This device optionally can use a bank of gas sampling bags or alternatively low volume Sample Vials

- GC/ Peak Simple installation
- Set the device No in PS / option overall > 302 device for 6 channel GC - the USB Port Number must be set ( from value is on GC side panel )
- this also contains GC parameter data for the SRI Test sample
- a flow schematic is also stencilled to the cover of the GSV Oven for extra guidance.



## Software > Peak Simple

The SRI PS Data System has evolved over 30 - 40 years

It is the Operating System for ALL the SRI GCs

Now 1 or 6-Channel versions

- Also available as a stand-alone separate box system for other GCs and general lab use ) IT HAS TO BE RELIABLE > Proven - IT IS STABLE ! Only limited by PC H'ware and MicroSoft WIN10 ( WIN 7 is actually more stable ! But " sabotaged / upgraded to Win 8/9 > but don't use those version ) AND WIN10 with ALL it's "foibles" )

Software > Backup

ALL SRI Data is on their Website [www.srigc.com](http://www.srigc.com) AND on The GC installation CD-ROM supplied with each Purchased SRI GS An SRI Test Report is supplied with each GC

- relevant to the Application the GC may be customised for Take NOTE of ANY customised "EVENTS" Tables , TEMP profiles, "Peak Windows" settings RELEVANT TO YOUR APP and the SRI Test Report As a START UP as a Minimum you should try and duplicate the test conditions AND Chromatogram EXACTLY ( with relevance ? )

- with Capillary columns Theoretical Plates can be calculated from within "Results" .

For each new column you should actually measure this ON YOUR GC !

- in future use this as a "yard stick" to indicate ( with some common sense ) when column performance deteriorates and when to replace a column ( 1/2 the Plate No is of concern ! )

S'ware bugs > known Different versions of WIN10 ( with / without updates ) and PS V4.88 have proven to be problematic talking to each other

**Minimum PC or Laptop ?** > don't try and "skimp" too much on this > false economy !

• >get a decent size Laptop or PC RAM and Disk space > it's all becoming low(er) cost anyway ! My Lap Top > 2.4GHz, 8GB RAM and 100Gb on Internal Hard Drive ( SSB ??? > of Dubious merit ! )  
... Is perfect ! albeit > an overkill as PS is only 12Mb in size ... in theory !  
**But WIN10 and MS Office are both "dogs" in size ! )**

PS DDE Data Dump to MS Excel

- from PS Results and if checked in Post-Run then PS "auto-dumps"

- Currently the PS data is dumped to MS Notepad as a data block ( with Excel "open" )

> import each block into a single cell in Excel

BUT IT DOES / CAN develop into a REAL MESS and Quickly ! & particularly b4 you clean up trace peaks ( eg set Area "reject in PS) and try save Only your main peaks ... to avoid inevitable "clutter"

even copy raw data on to pen/paper scratc pad ... until you understand can sort it ALL out  
YOUR > "learning curve"

- **SRI PS Intro**

[https://www.youtube.com/watch?v=M5\\_jcgwth8Y](https://www.youtube.com/watch?v=M5_jcgwth8Y)

Peak Simple more detail -

GC Tutorial SRI 8610C Pt-2

[https://www.youtube.com/watch?v=cky1fvY\\_KjA](https://www.youtube.com/watch?v=cky1fvY_KjA) GC

Tutorial Pt-3 8610C Setup

<https://www.youtube.com/watch?v=kA6LRraq2IM>

Our Website(s) [www.chromtech.net.au](http://www.chromtech.net.au)





## **BEWARE :**

**PeakSimple** is designed as the dedicated Windows to **ALLSRI GCs** Operating System DO NOT COMPROMISE BY USING A NETWORK PC  
( controlled compromised by **IT Admin** rights etc ).

eg don't use a University / Corporate installed "secure" PC as part of the GC System 2020 There are still issues with the "notorious **WIN 10** and **Peak Simple ( v 4.88 )**

**IT MAY NOT EVEN CONNECT to the SRI GC !**

**> USE ONLY the Proven PS v 4.54 )**

Use a stand-alone PC ( Win 7 or Win 10 but with PS V4.54 ( 32 bit or 64 bit as appropriate ) IF access IS required to the Internet THEN use a separate IT Connected PC . . . **B'Ware !**

To AVOID CONFLICTS /VIRUS' AND POSSIBLE PS PROBLEMS PS may not even Connect properly

At least until you have become familiar with the PS File Structure

In a perfect World ( without WIN10 ) **anything is feasible !**

PS is designed to operate on a Network BUT ! . . .

In theory U can even run Multiple version of PS on the same computer and even control other SRI GCs across the Internet !

Install PS as directed by SRI

**Initially** keep ALL PS Files for a specific App in the same folder as the PS.exe file  
and it does get cluttered !

but THEN once satisfied ( **carefully** ) separate the data into other folders & Onlyafter ensuring you back up / save ALL data ( .chr, .con, .evt, .temp, .cal )

and store the "raw" data via the DDE Link into > NotePad > MS Excel Excel  
> Only a crude Field ( as per PS Results / Format vs Sample Run table

U need to restructure the Excel Data > the best U can !

BUT Test & Test again ! > Data / chromatograms can easily be Lost **FOREVER**  
**TAKE CARE / be systematic in naming ALL files!**

inc "Date", Channel, "GC Run #  
Save date into seperate "Detector".log

**> BEWARE ! . . . BE WARNED !**

**READ This Note Carefully ! AND All SRI Tech Notes ( [www.srigc.com](http://www.srigc.com) )**

**B 4 U get into technical "strife" !**

**NEVER play with real-life sample Data using PeakSimple**

- **get plenty of practice data on lab test samples FIRST !**
- **DON'T RISK REAL YOUR SAMPLE DATA !**

Our Website(s) > [www.chromtech.net.au](http://www.chromtech.net.au)

[www.chromtech-us.com](http://www.chromtech-us.com) ( mobile friendly > auto-scalable etc )

[www.chromalytic.net.au](http://www.chromalytic.net.au) ( still under development ! )



# Quick Start SRI GC Installation Guide

## I. Gas Installation & Connection

1. To connect your GC to a gas supply, we recommend the following:

- A 50 foot length of copper tubing
- A stainless steel gas line filter
- At least 2 sets of stainless steel Swagelok nuts and brass ferrules (it is a good idea to keep a few extras on hand)
- A cylinder pressure regulator with 0-100psi output

**NOTE:** each type of cylinder has a different CGA connection to the regulator (CGA = Compressed Gas Association). Air is typically CGA 590 or 346. Helium and nitrogen are CGA 580. Hydrogen and argon-methane are CGA 350.

Gas line installation kits that include everything you need are available from SRI:

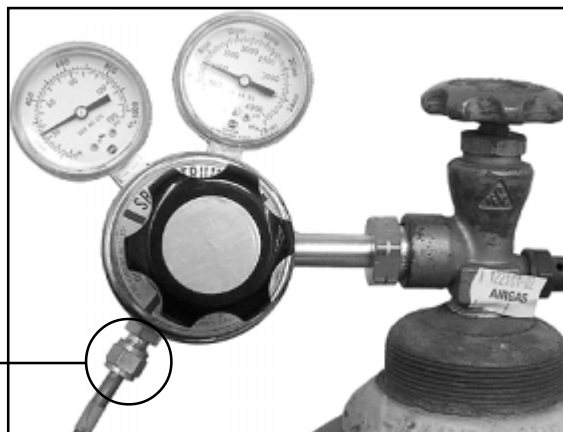
- |           |   |
|-----------|---|
| 8600-C590 | Air gas line kit (with both CGA 590 and 346 inlet fittings)   |
| 8600-C580 | Helium/nitrogen gas line kit  |
| 8600-C350 | Hydrogen/argon-methane gas line kit (the hydrogen CGA is equipped with a flow restrictor to limit the escape of gas in the event of a leak) |

These kits include everything in the list of recommended supplies above, plus a tubing cutter. Each regulator is supplied with a 1/8" Swagelok fitting for easy connection to the copper tubing.

2. Using the appropriate CGA connection as described above, attach the regulator securely to the gas cylinder.

3. Secure one end of the 1/8" copper tubing to the regulator with a Swagelok nut and ferrule. Cut the tubing to the desired length before connecting it to the GC. Make sure to leave it long enough to allow you to move your GC around your work area.

1/8"  
Swagelok  
fitting



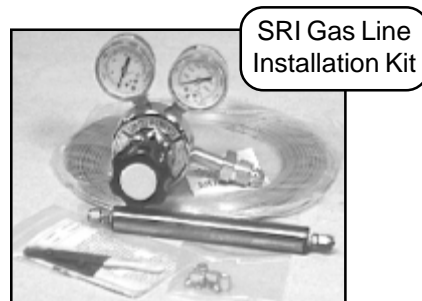
4. If you don't already filter your gas, install gas line filter(s) in the gas line(s) where it is convenient to replace when needed.



cylinder air, a jumper tube is secured to the air inlet and outlet. If you ordered your GC with an air compressor, it is shipped with the jumper tube in place as shown.

5. Connect the gas or gases to the inlets on the left-hand side of the GC as labeled.

**NOTE:** the GC shown here is equipped with a built-in air compressor. When using the internal air compressor instead of



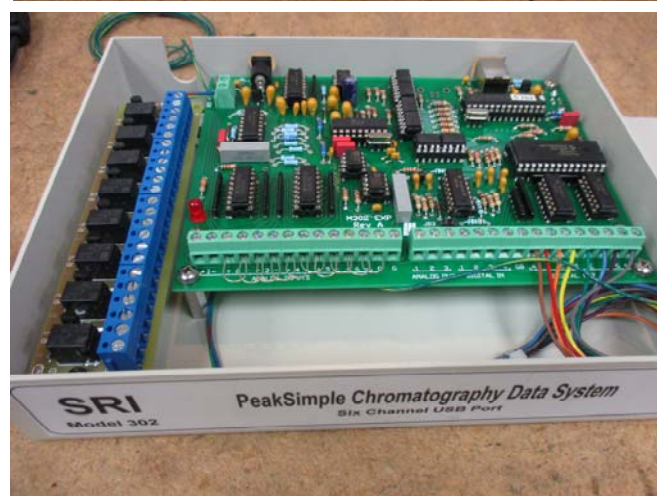
# Model 302 Data System

## August 2018

Starting August 2018 the PeakSimple Model 302 6 channel Chromatography Data System has been re-packaged into a smaller box.

The new box comes with a 2 meter USB cable and power supply which can operate on all worldwide voltages from 100-240 volts AC,

Inside the box is all the same hardware as in the previous much larger box.



**SRI Tech Support: [www.srigc.com](http://www.srigc.com)**

302DataSystemAugust2018

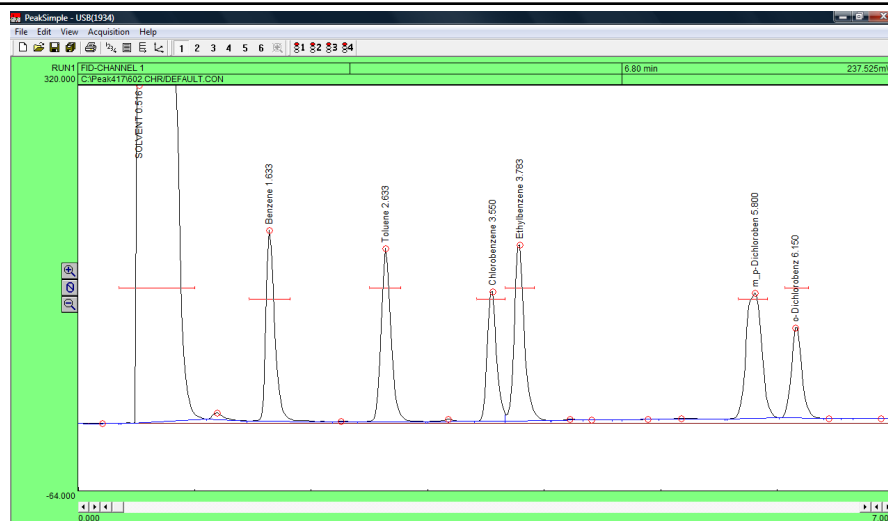
Page 1





# PeakSimple Basic Tutorial

## Version 4.17, September 2012



Installing PeakSimple from the CD or USB thumb drive:

- Start the Windows operating system in use on your computer. (Windows XP, Vista, or 7)
- Insert the CD or USB thumb drive into the computer.
- Open **My Computer** and open either the CD or thumb drive.
- Double-click on the **Setup.exe** file. Make sure to select the right version of PeakSimple to install (32- or 64-bit). Windows XP and some Vista computers need to install the 32-bit version, other Vista computers and Windows 7 need to install the 64-bit version. If you are unsure, right-click on **My Computer** then select **Properties** in order to determine what bit operating system you are using.
- To complete installation follow the onscreen instructions provided by the installation wizard.
- For instructions on loading the driver, please refer to the Quick Start Documents located in the PeakSimple folder or on the [www.srigc.com](http://www.srigc.com) website.

Installing PeakSimple from software download:

- Start the Windows operating system and use an online browser to access [www.srigc.com](http://www.srigc.com).
- From the menu on the left hand side of the screen select **Download PeakSimple** and then download the latest version. Windows XP and some Vista computers need to download the 32-bit version, other Vista computers and Windows 7 need to download the 64-bit version. If you are unsure, right-click on **My Computer** then select **Properties** in order to determine what bit operating system you are using.
- Save the file to a temporary folder and double-click on the setup file when it is finished downloading, or, just click **Run** to install PeakSimple without saving the setup file.
- Follow the onscreen instructions provided by the installation wizard.
- For instructions on loading the driver, please refer to the Quick Start Document located in the PeakSimple folder or on the [www.srigc.com](http://www.srigc.com) website.

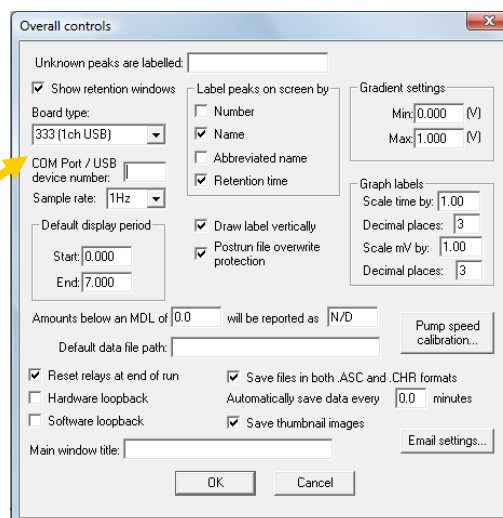
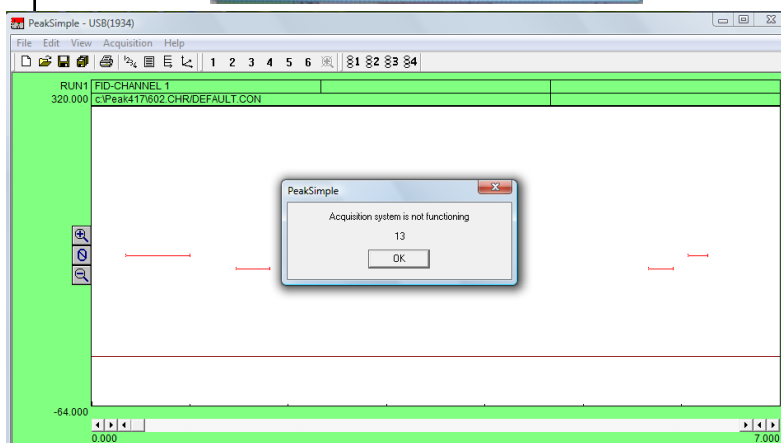
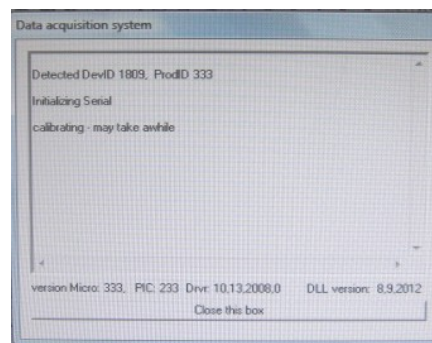


# PeakSimple Basic Tutorial

## Version 4.17, September 2012

### Launching PeakSimple

1. Double-click on the Desktop PeakSimple icon to launch PeakSimple.
2. The data acquisition system will attempt to initiate communications between the computer and the data system.
3. If PeakSimple comes up with an error message stating "Acquisition system is not functioning" with a countdown timer, it is indicating that there is a communication problem between the computer and the data system or that the data system and the hardware is not connected. Click OK to continue working with PeakSimple.
4. The first time PeakSimple connects to a GC or data system open the **Edit** menu and select **Overall** to get to the **Overall Controls** Screen.
5. Enter the proper **Board type** (202, 203, 302, or 333) and **COM Port/USB device number** (Found on data system or GC). Select OK and PeakSimple will establish communications with the data system.
6. Most of the commands and options in PeakSimple are equipped with tool tips that will automatically pop up to display useful information when the mouse cursor is held over a command. To turn off the tool tips deselect the tool tips option in the Help menu.



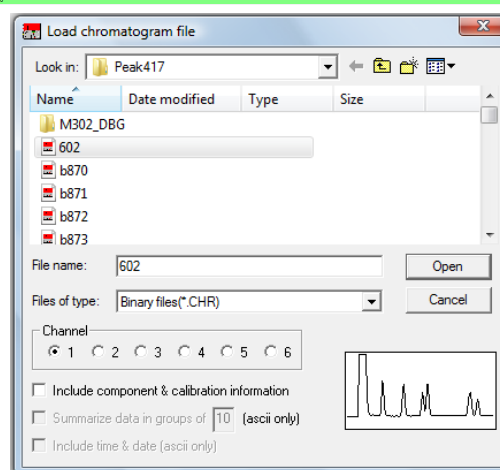
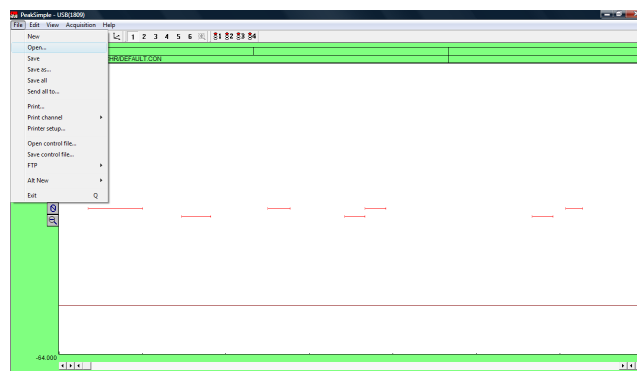
Enter the A/D board type. Your choices are Model 203 single channel serial connection, Model 202 4 channel serial connection, Model 333 single channel USB and Model 302 6 channel USB.

# PeakSimple Basic Tutorial

## Version 4.17, September 2012

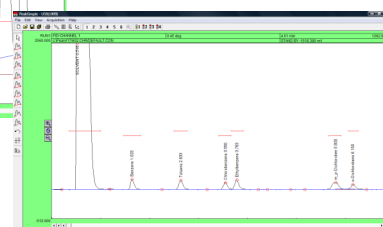
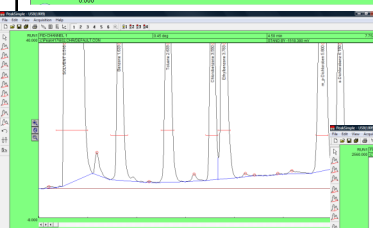
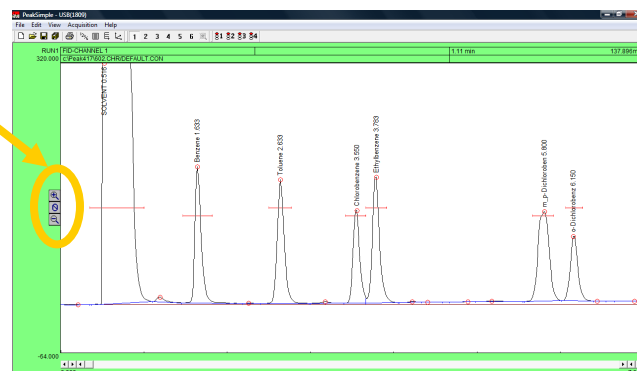
### Opening a PeakSimple Data File

1. To open a PeakSimple data file or chromatogram, begin by selecting **File** in the PeakSimple menu bar and then choose **Open...** from the set of options.
2. The Load Chromatogram File window is now open. The PeakSimple software includes a number of sample chromatogram data files that can be opened, displayed, and manipulated. One file, 602.CHR, will be used throughout the rest of the tutorial. Select file **602.CHR** from the PeakSimple directory, choose **Channel 1** as a destination channel, and then select **Open** to load the file.



### Adjusting Display Limits

1. To adjust the display limits of a chromatogram click on either the **+** magnifying glass icon or the **-** magnifying glass icon to the left of the chromatogram. This will increase or decrease the limits by a factor of two each time you click on the icons.
2. After opening chromatogram 602.CHR, practice making the display limits smaller but the peaks larger by clicking the **+** magnifying glass icon.
3. Practice making the display limits larger but the peaks smaller by clicking on the **-** magnifying glass icon.



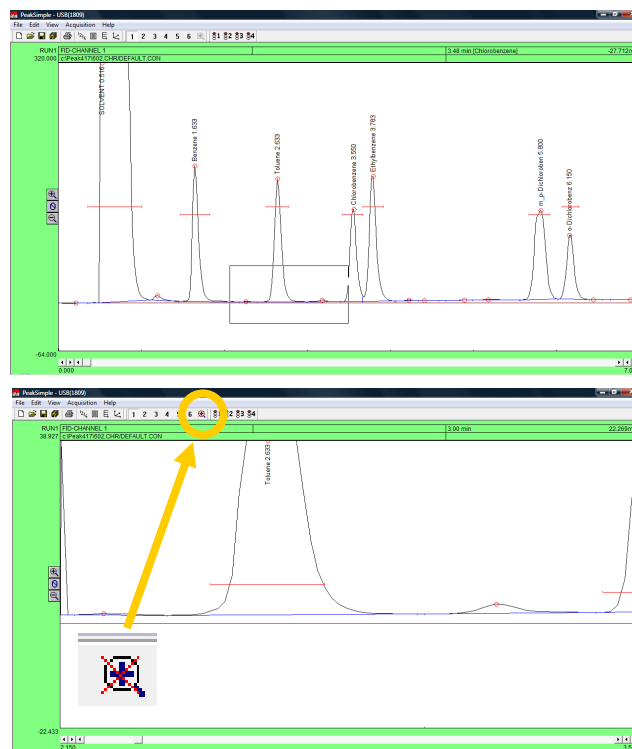


# PeakSimple Basic Tutorial

## Version 4.17, September 2012

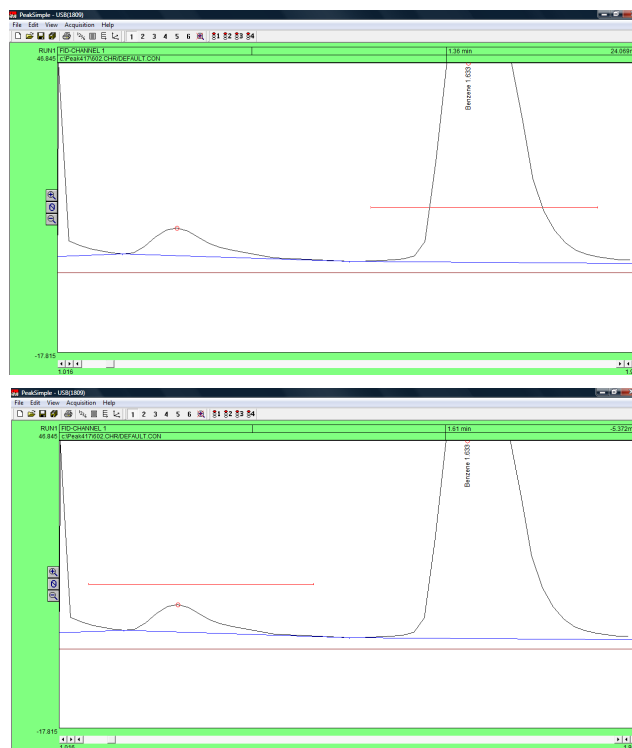
### Zooming

1. To zoom in on a specific part of a PeakSimple chromatogram, click and hold the left mouse button and drag it over the desired area.
2. After opening chromatogram 602.CHR hold the left mouse button and drag it over the base of the toluene peak. Let go of the mouse button and there will be a larger view of the area that was selected.
3. To return to the original display limits of the chromatogram and unzoom the area selected press **F6** or select the unzoom icon located in the PeakSimple toolbar at the top of the screen or right-click and select **Unzoom**.



### Dragging Retention Windows

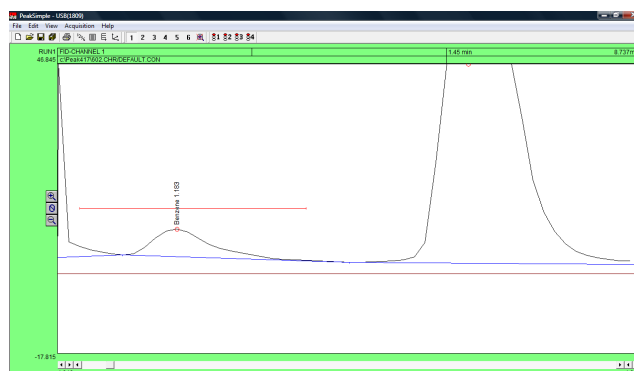
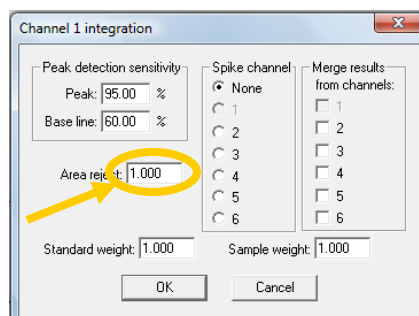
1. To drag a retention window bar place the mouse cursor on the bar until a double sided arrow pops up. Click on the left mouse button and hold and then drag the retention window bar to its desired place.
2. After opening the chromatogram 602.CHR zoom in on the benzene peak and the smaller peak to its left. Locate the benzene retention window bar and drag it over to the smaller unnamed peak to the left of the benzene. Because this is a small peak it is not immediately recognized.



# PeakSimple Basic Tutorial

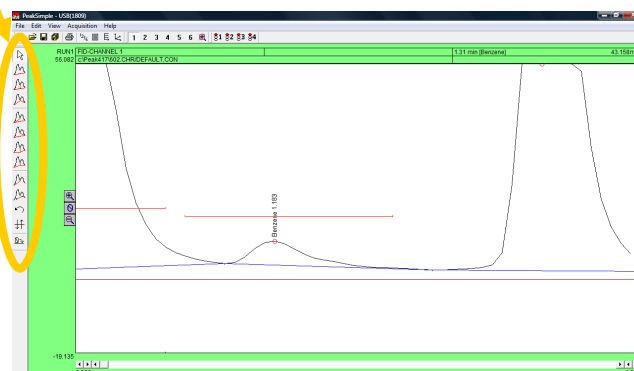
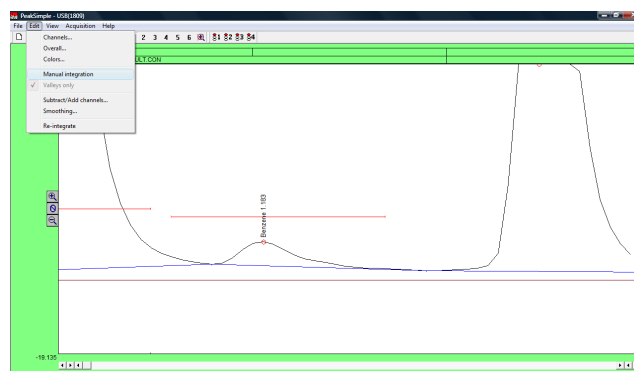
## Version 4.17, September 2012

- Right click on the chromatogram over the unnamed peak and select **Integration** from the resulting menu.
- From the integration window locate the **Area Reject** dialogue box, erase the 100.0 in the box, and add the number **1.0** to the dialogue box. Click **OK** and the integration window will exit.
- Press the **Enter** or **Return** key on your keyboard and the smaller peak will now be recognized as Benzene.



### Manual Integration

- To manually adjust the integration baseline and peak separation in a chromatogram use the manual integration toolbar provided by PeakSimple. To open up the manual integration toolbar select **Edit** in the PeakSimple menu bar and then click on the **Manual Integration** option. The manual integration toolbar will now appear to the left of the chromatogram.
- The manual integration toolbar contains nine types of manual integration options. Four of the most commonly used options are **None** integration, **Drop** integration, **Based** integration, and **Rubber Band** integration.



# PeakSimple Basic Tutorial

## Version 4.17, September 2012

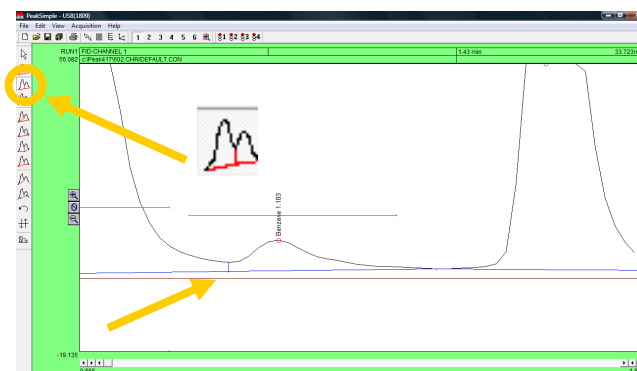
3. To make a baseline “ignore” a peak use the **None** integration tool. After opening chromatogram 602.CHR and the manual integration toolbar, zoom in on the baseline of the solvent peak and the smaller unrecognized peak immediately to its right. Click on the **None** integration tool in the manual integration toolbar with the mouse cursor and then click on the valley between the two peaks where they meet the baseline. The area of the small peak is now added to the solvent peak.



4. To undo the changes made to a chromatogram at any time simply click on the **Undo** integration tool in the manual integration toolbar. After selecting this tool all integration changes made to the chromatogram will be undone.



5. Click on the **Undo** tool with your mouse cursor and select the **Drop** integration tool to enable the dropping of the baseline below the two peaks. After selecting the Drop tool click where the valley of the peaks meet the baseline with the cursor. The baseline should now be dropped below the base of the peaks and a line should extend from it to the baseline.

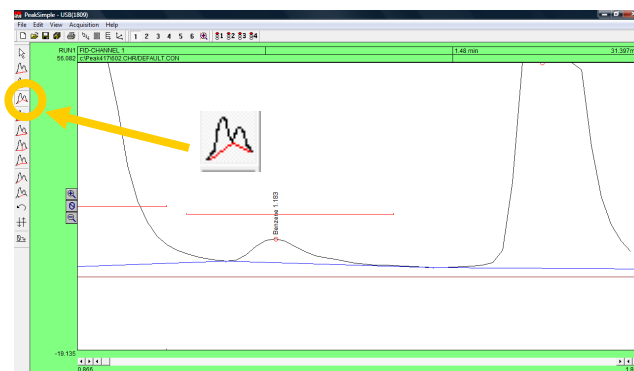




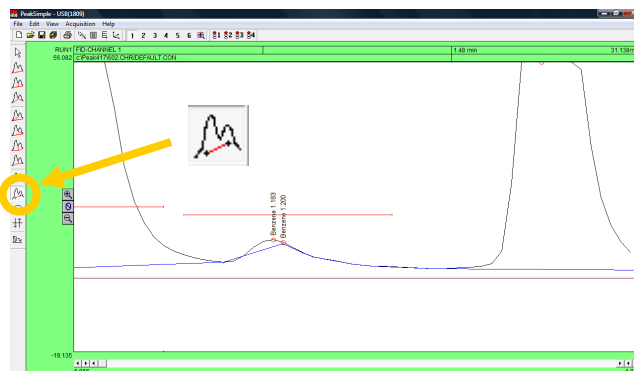
# PeakSimple Basic Tutorial

## Version 4.17, September 2012

6. After the manual integration between the two peaks is dropped use the **Based** integration tool to raise the baseline to the valley between the peaks. Once the Based integration tool is selected, click on the valley between the solvent peak and the smaller peak to its right with the mouse cursor. The baseline will now extend up to meet the valley of the two peaks.

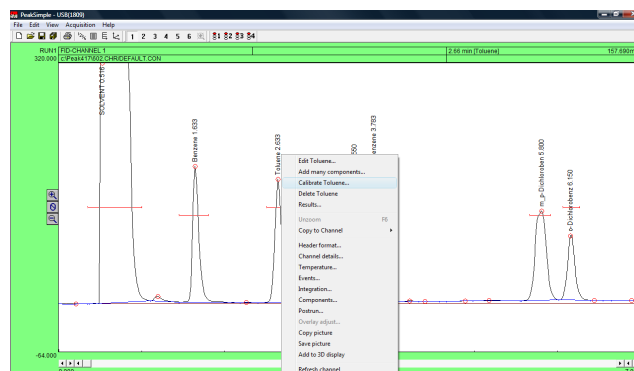


7. Once again click on the **Undo** tool in the manual integration toolbar to remove all changes done to the chromatogram. Select the **Rubber Band** integration tool to manually draw a baseline. Once the Rubber Band tool is selected take the mouse cursor and click on a part of the baseline. While holding down the left mouse button extend the line to another part of the baseline further to the right of the starting point and let go of the mouse button. The base line will now be drawn according to the line that was drawn using the Rubber Band integration tool.



### Calibration

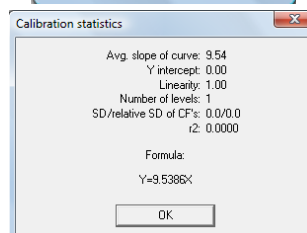
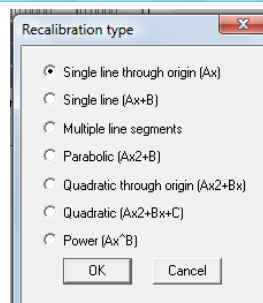
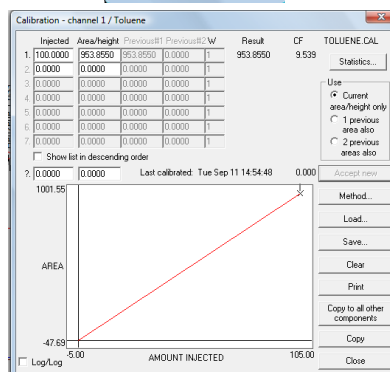
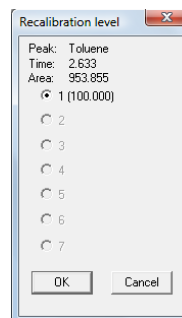
1. To turn the raw area of a peak into a real-world number the peak first needs to be calibrated. To calibrate the Toluene peak in chromatogram 602.CHR, open up the file and then right click using the mouse on the Toluene peak. After right clicking on Toluene select **Calibrate Toluene** from the resulting menu.



# PeakSimple Basic Tutorial

## Version 4.17, September 2012

- From the Recalibration level window click on the first level radio button **1 (100.000)** and then select **OK** with your mouse cursor.
- After selecting OK from the Recalibration level menu the Calibration menu for Toluene will pop up. Check to make sure the flashing asterisk on the calibration curve is on level 1 and then click on the **Accept New** button to the right of the window.
- Once the new data is accepted, click on the **Method** button immediately below the Accept New button. The Recalibration type window will now open allowing the user to select a method of calibration. By default the calibration type is set at Multiple Line Segments. Select the **Single line through origin (Ax)** radio button and then click on **OK** with the mouse cursor.
- After changing the method of calibration click on **Statistics** in the upper right hand corner of the Calibration level window. The Calibration statistics window will pop up revealing the statistics for the calibration of Toluene. Click **OK** with the mouse cursor to close the Calibration statistics window and then select **Close** from the Calibration window to finish calibrating Toluene.
- View the calibrated results in the Results screen by right-clicking on the chromatogram and selecting **Results**.



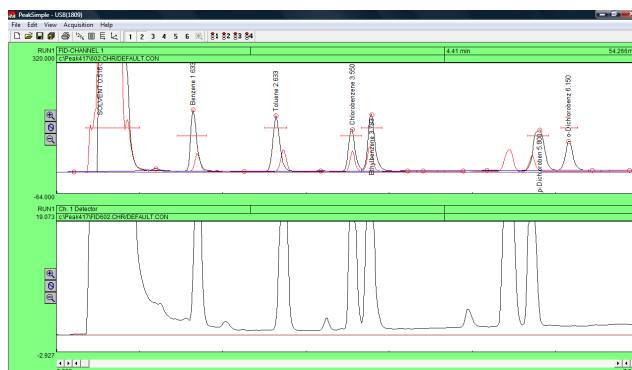
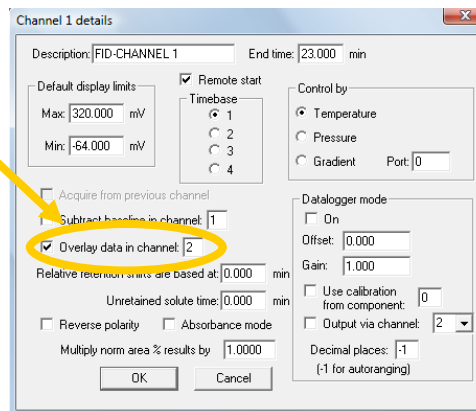
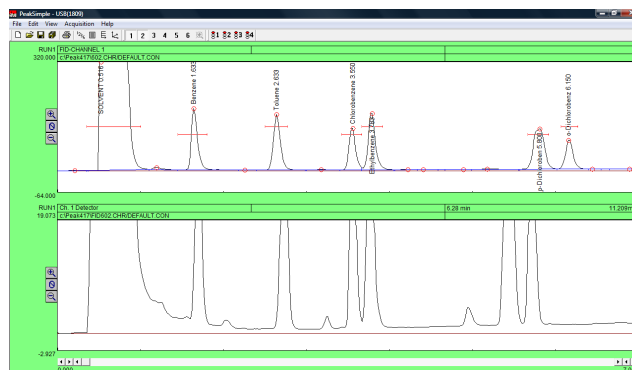
Component	Retention	Area	Height	External	Units
SOLVENT	0.916	71603.5420	888.161	0.0000	%
Benzene	1.633	538.6270	180.764	100.0000	ppm
Toluene	2.633	953.8550	163.086	100.0000	ppm
Chlorobenzene	3.950	676.9750	122.832	72.1215	ppm
Ethylbenzene	3.763	986.4475	166.671	112.3559	ppm
m,p-Dichlorobenz	5.800	1093.8760	119.019	124.2345	ppm
o-Dichlorobenz	6.150	537.4520	95.167	54.6115	ppm
		76603.7715		953.3434	

# PeakSimple Basic Tutorial

## Version 4.17, September 2012

### Overlay

1. To compare two or more chromatograms overlay them using PeakSimple. To overlay two chromatograms first open chromatogram 602.CHR and then click on the **2** button in the PeakSimple toolbar. A second chromatogram channel is now open in the PeakSimple window.
2. Once the second channel is open select **File** from the PeakSimple menu bar and then click on **Open**. The Load chromatogram file window will open up displaying a list of files to load. Select chromatogram **FID602.CHR** to load and then select the **2** channel radio button to load the chromatogram in the second channel.
3. Once FID602.CHR is open in the second channel right click using the mouse on the first channel and select **Channel Details** from the list of options.
4. After the Channel 1 details window appears on the screen locate the **Overlay data in channel** check box and select it. Look to the dialogue box to the right of the Overlay data in channel check box and insert the number **2** in place of the 1. Click on **OK** with the mouse cursor to exit the Channel 1 details window.
5. The chromatogram FID602.CHR is now in place overlaid on top of chromatogram 602.CHR in channel 1. Chromatogram 602.CHR is in black while FID602.CHR is in red.



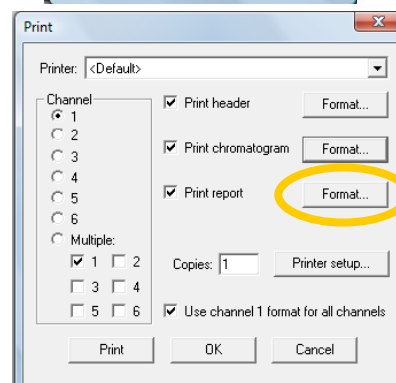
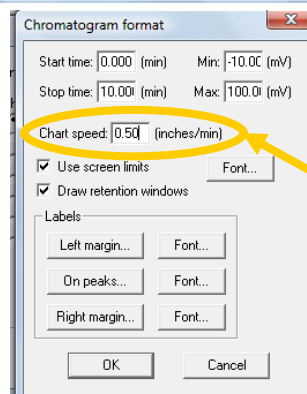
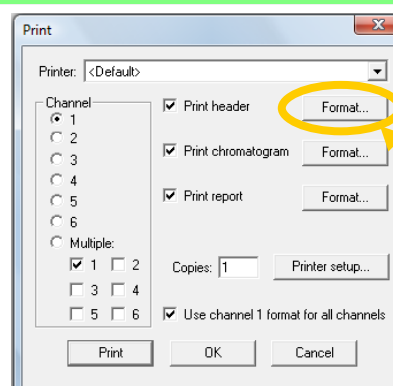
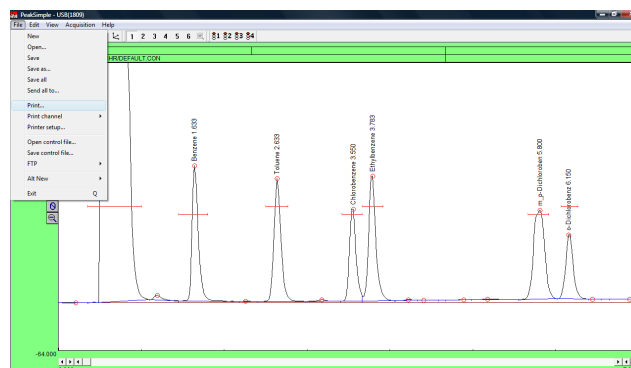


# PeakSimple Basic Tutorial

## Version 4.17, September 2012

### Printing a Chromatogram

1. To print a chromatogram first open chromatogram 602.CHR. Once the chromatogram is open select **File** from the PeakSimple menu bar and then select **Print** from the drop-down menu.
2. The Print window will open and will allow the user to customize the printing of a chromatogram. Click on the **Format** button for the Print header to open up the Header format window. Add or delete any information in the window by clicking on the fields and inserting the desired information. Click on the **OK** button when all the desired information is inputted to close the window.
3. In the Print window click on the **Format** button for Print chromatogram to open up the Chromatogram format window. Locate the **Chart speed** dialogue box and insert the number of inches each minute on the chromatogram will take up when printed (for a nine minute run try **0.50** inches per minute). After the Chart speed is entered click on **OK** to exit the window.
4. In the Print window locate the Print report check box and click on the **Format** button to its right.

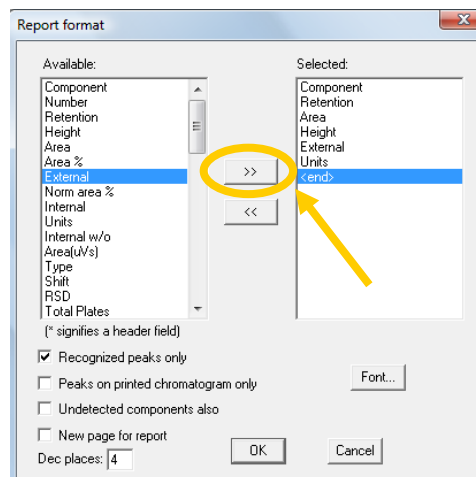


# PeakSimple Basic Tutorial

## Version 4.17, September 2012

5. Once the Report format window is open click on **External** in the Available dialogue menu (on the left) and then click with the mouse cursor on the right facing arrow button to add External to the Selected dialogue box (on the right). After External is added to the Selected dialogue box click on **Units** with the mouse cursor and click on the right facing arrow button to add Units to the Selected dialogue box. Click on **OK** with the mouse cursor to exit out of the Report format window.

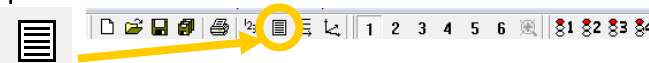
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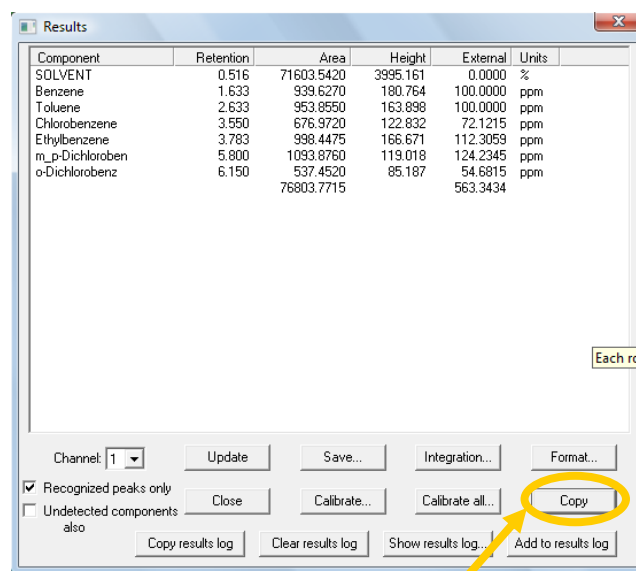
6. Select **Print** in the Print window to print the chromatogram or click on **OK** in the Print window to exit the window.

### Exporting to Excel

1. In the PeakSimple toolbar click on the **Results** window button to open up the Results window. Once the Results window is open click on the **Copy** button to copy the results data to the Windows clipboard.



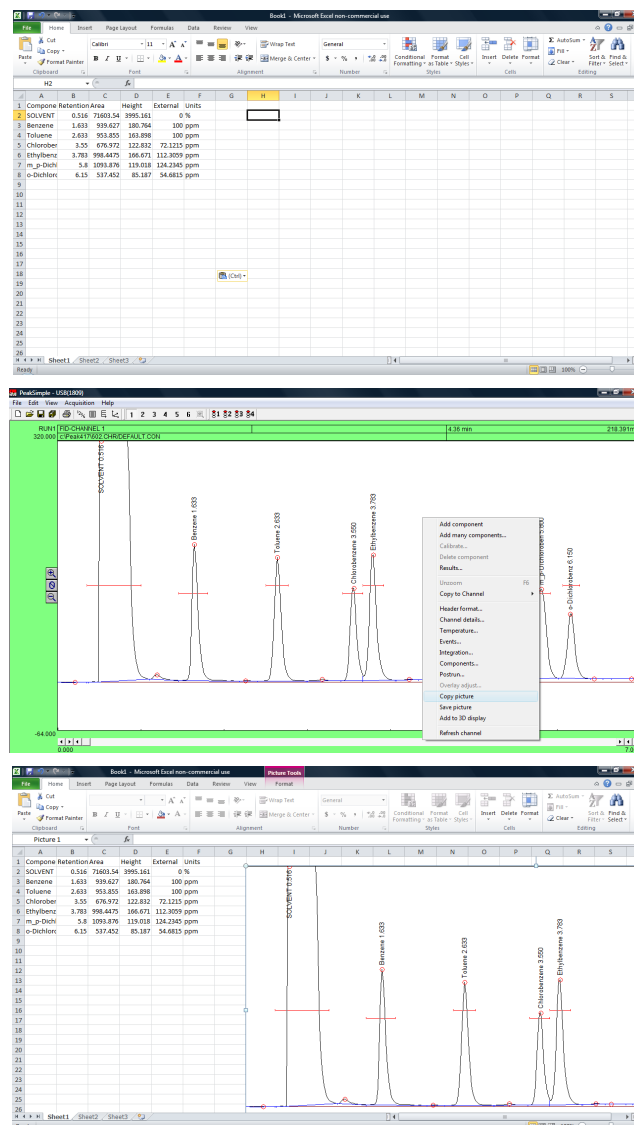
2. Make sure Microsoft Excel is loaded on the computer. If Excel is not loaded you can copy results data and chromatograms to Microsoft Word or PowerPoint. Open up Microsoft Excel by clicking with the mouse cursor on the **Start** button in the bottom left of the Windows screen and then **Programs** and then **Microsoft Excel** in the Windows Program menu.



# PeakSimple Basic Tutorial

## Version 4.17, September 2012

- Once Excel is opened select **Edit** from the Excel menu bar and then **Paste** from the drop down menu. The results data is now placed into the columns and rows of Excel. Using the mouse cursor, select a box to the right of the results data in the Excel spreadsheet. Go back into the PeakSimple program and hit **Close** to exit the Results window.
- Right click with the mouse cursor anywhere on chromatogram 602.CHR and select **Copy picture** from the resulting menu. Go back into Excel and select **Edit** from the Excel menu bar and then **Paste** from the drop down menu. The PeakSimple chromatogram will now be displayed next to its results data in the rows and columns of Microsoft Excel.



This concludes the PeakSimple Basic Tutorial.

An Advanced Tutorial can be obtained by going to:  
[www.srigc.com](http://www.srigc.com)

If you have questions or would like to place an order, call:  
(310) 214-5092





# Model 302 Data System

August 2018

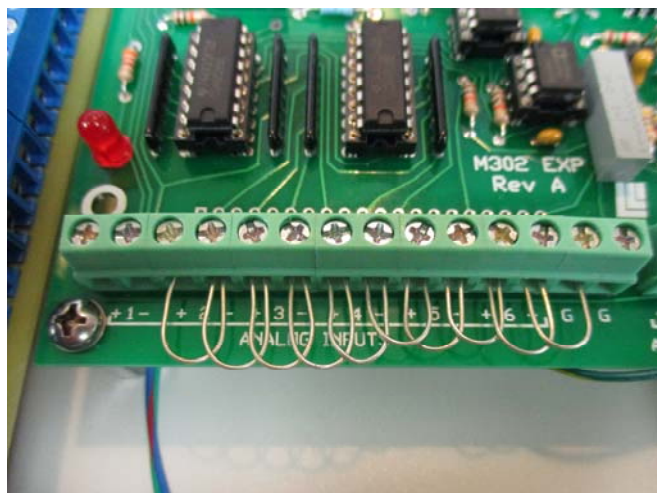
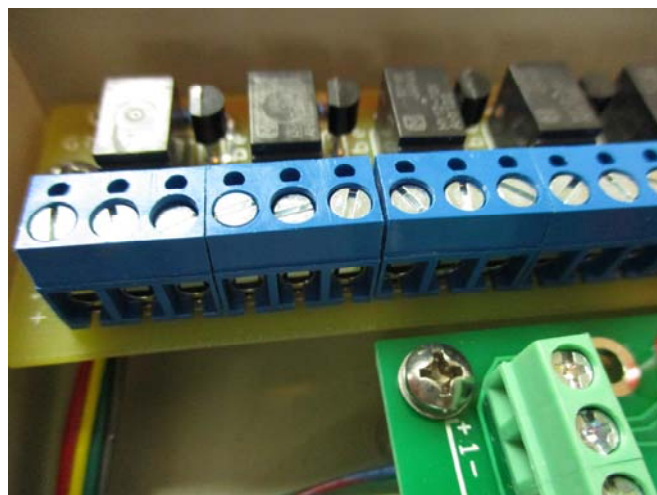
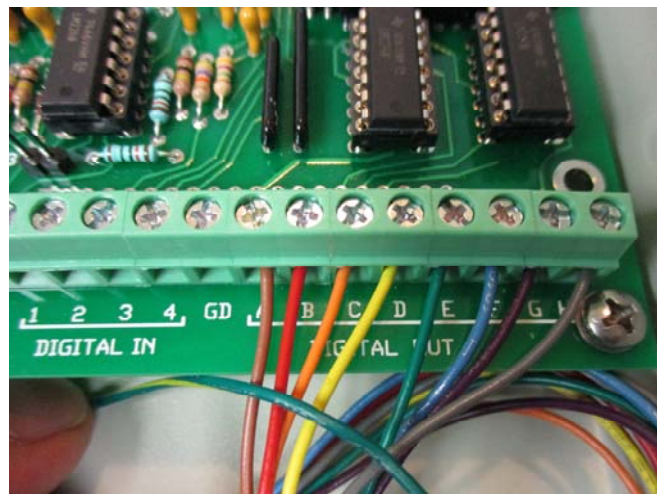
There are 8 TTL outputs labelled A-H.

Each TTL output is connected to a mechanical single pole, dual throw relay to make it easier to interface Valco Valves, solenoids etc.

Each relay is rated at 24 volts DC 2 amps max.

**They are not to be used for switching line voltage 115VAC.**

There are six signal inputs. Each input requires a plus and minus voltage between  $-5$  and  $+5$  volts. If you do not connect a signal it is best to connect all unused inputs to ground as shown by the daisy chained jumper wires in the photo.



SRI Tech Support: [www.srigc.com](http://www.srigc.com)

302DataSystemAugust2018

Page 2



# Quick Start GC Installation Guide

("Gas Installation & Connection" *continued*)

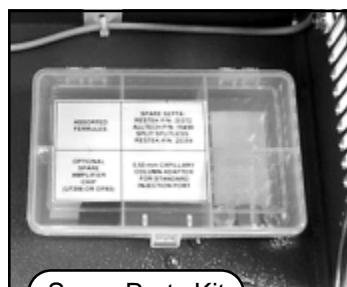
GAS FLOW RATES					
CARRIER 1:		:	7	PSI =	10 ml/min
CARRIER 2:		:		PSI =	ml/min
P&T PURGE:		:		PSI =	ml/min
HYDROGEN 1:	FID	:	21	PSI =	25 ml/min
HYDROGEN 2:		:		PSI =	ml/min
AIR 1:	FID	:	9	PSI =	250 ml/min

6. The pressure that correlates with the flow rate for the column, make-up gases, and detector supplies is labeled on the right-hand side of the GC. For best EPC performance, set the incoming gas pressure(s) 15-20psi higher than the operating pressure listed on the right-hand side of the GC.

## II. Column Installation

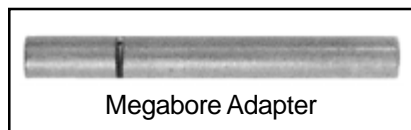
1. If you ordered a column with your GC, it is shipped installed in the column oven and you can skip this section. Otherwise, open the GC lid and the column oven lid.

2. These instructions will cover the installation of a 0.53mm capillary column into an on-column injector. The SRI on-column injector is designed for a 26 gauge syringe needle; a 10 $\mu$ L liquid injection syringe with a 26 gauge needle is included in the Accessories Kit shipped with your GC.



Spare Parts Kit

A megabore adapter for syringe injection onto 0.53mm capillary columns is included in the Spare Parts Kit affixed to the inside of the GC lid on the right-hand rear corner.



Megabore Adapter



Accessories Kit

-OR-

Accessories Kit contents:

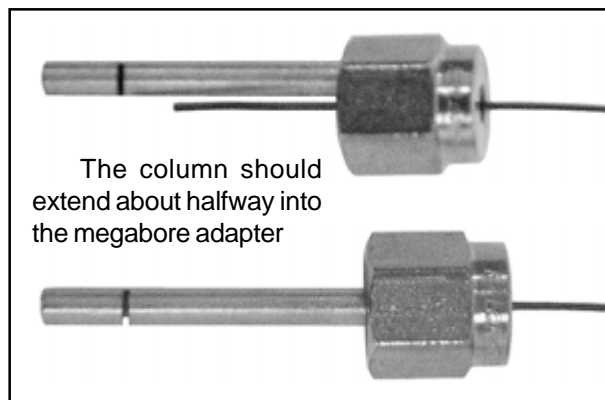
- 6' Serial **or** USB cable
- Tubing cutter
- 10 $\mu$ L liquid injection syringe
- 1mL gas injection syringe & needle
- 3mL leak check syringe

3. The megabore adapter is a 1" x 1/8"OD stainless steel tube with a perpendicular gash cut into it, and a conical entry to guide the syringe needle into the column. A 0.53mm capillary column connects to the SRI on-column injector with a graphite reducing ferrule and a 1/8" Swagelok nut. Insert one end of the column through the nut, then through the graphite ferrule. It is a good idea to trim off about one inch of the column to avoid possible peak tailing from any graphite shavings left behind after inserting the column through the ferrule; make sure the cut is clean, with no jagged edges.

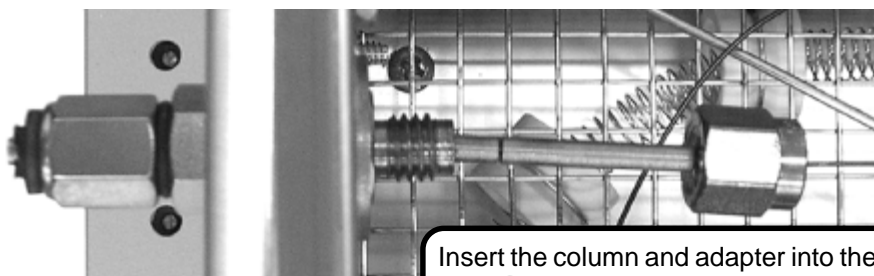
# Quick Start GC Installation Guide

("Column Installation" *continued*)

4. Insert the column end with the graphite ferrule and Swagelok nut about halfway into the megabore adapter and tighten it with the nut and ferrule.



5. After inserting the column into the adapter, insert the column and adapter together into the injection port. Tighten the Swagelok nut with a 7/16" wrench. You should feel a little give from the ferrule, but do not overtighten it. You want it tight enough to prevent leakage, but do not



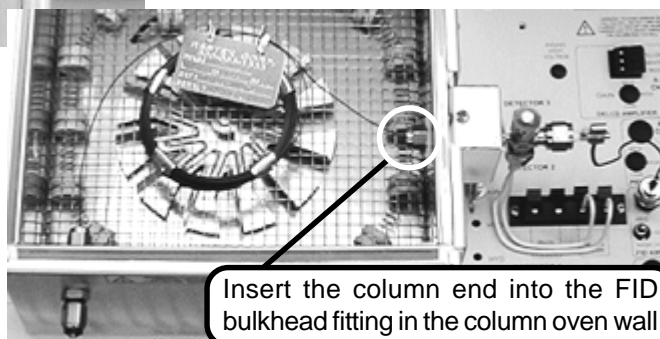
Connect the column to the TCD IN tubing



6. Slide another 1/8" Swagelok nut and graphite ferrule over the other end of the column. For a TCD detector, connect the nut to the fitting labeled "TCD IN" in the column oven.

For an FID detector, leave about 1" of the column protruding through the nut and ferrule. Insert the column into the FID bulkhead fitting in the column oven wall and tighten the Swagelok nut.

Please see "Analytical Column Installation" in the INSTALLATION section of your manual for more detailed information on column installation.





# Quick Start GC Installation Guide

## III. Software Installation

NOTE: There are tutorials in the manual and online at [www.srigc.com](http://www.srigc.com) (click on the “Download Our Documents” button) that will acquaint you with the basic functions of the PeakSimple chromatography software included with your GC.

1. Connect the serial or USB cable to your computer and the GC. The serial port connection is on the left-hand side of the GC, and the USB connection is on the right-hand side.

2. Locate your copy of the PeakSimple software just inside the front cover of your SRI manual. Insert the CD or floppy disks into your computer's appropriate drive.



SRI Manual



3. Double click on “My Computer,” then on the appropriate drive to open it. Double click on the “setup.exe” icon, and follow the instructions.

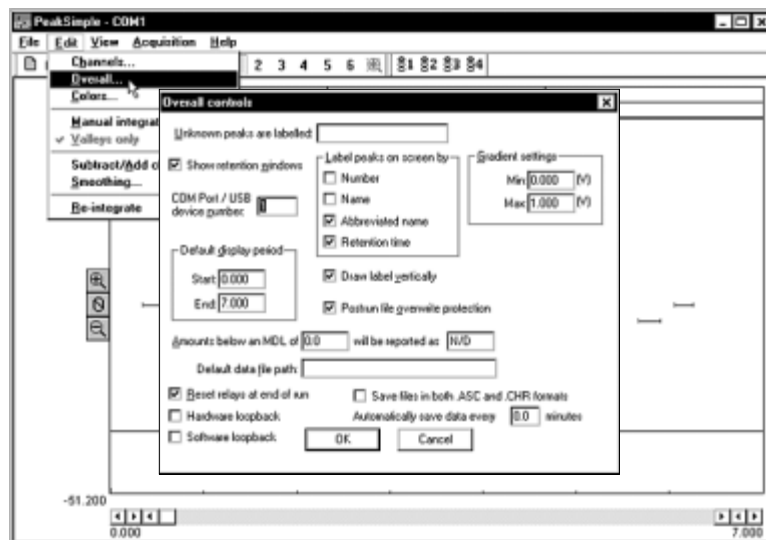


4. For USB, refer to “Installing the USB Drivers for Model 302 USB PeakSimple Data System” which you will find immediately behind these instructions in your manual, or online at [www.srigc.com](http://www.srigc.com). Return to step #5 below when you are finished installing the USB drivers. For serial port, proceed to the next step.



5. Double-click on the PeakSimple icon to launch the program. Verify that communication has been established between the computer and the GC. An error message will appear if communication is not established.

6. Open the Edit menu and choose Overall. In the dialog box that pops up, enter the number of the COM port to which you have connected the GC. For USB, enter the unique USB device number that is printed on your PeakSimple disk(s), and on the back of the GC. It is a 4-digit number that always begins with “5” (5093, 5276, etc.).



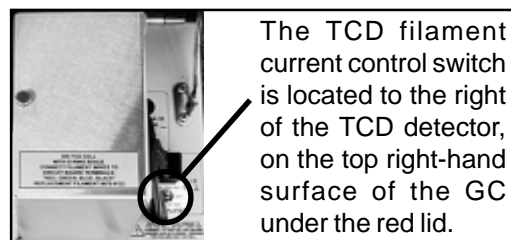
# Quick Start GC Installation Guide

## IV. Detector Activation

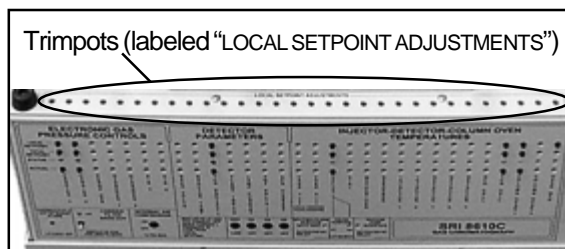
**IMPORTANT:** If you have a pre-configured GC system, please see the manual section for instructions on operating procedures. The manual is organized into sections with labeled tabs. In addition to preconfigured GCs, there are sections on detectors, injectors, autosamplers, valves, and more.

### A. TCD Detector

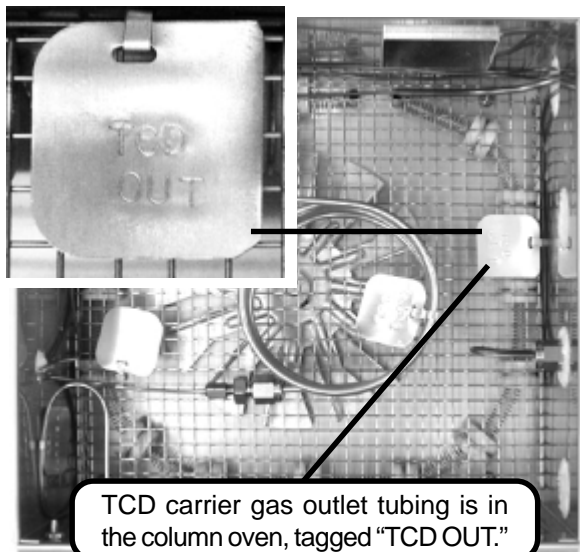
1. Your GC power should still be ON, and the filaments should still be OFF. The TCD oven is set to 150°C at the factory. It is adjustable by turning the trimpot while observing the TCD CELL LOCAL SETPOINT temperature on the LED display. The trimpots are located on the top edge of the GC front control panel. Allow the TCD to reach desired operating temperature and stabilize.



The TCD filament current control switch is located to the right of the TCD detector, on the top right-hand surface of the GC under the red lid.



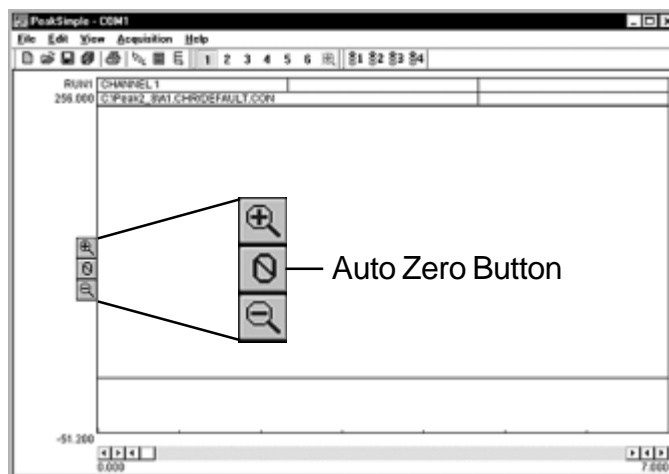
Trimpots (labeled "LOCAL SETPOINT ADJUSTMENTS")



TCD carrier gas outlet tubing is in the column oven, tagged "TCD OUT."

2. TCD filaments will be damaged or destroyed if current is applied in the absence of flowing carrier gas. Therefore, always verify that carrier gas is exiting the TCD carrier gas outlet before energizing the TCD filaments. The TCD carrier gas outlet tubing is in the column oven, labeled "TCD OUT." Place the end of the tubing in some liquid; if no bubbles are exiting the tube, there is a flow problem. **DO NOT** turn the TCD current ON if you cannot detect carrier gas flow. A filament protection circuit prevents filament damage if carrier gas pressure is not detected at the GC, but it cannot prevent filament damage under all circumstances. Correct any lack of carrier gas flow before proceeding.

3. With the TCD filaments still OFF, zero the data system signal by clicking on the Auto Zero icon on the left side of the chromatogram. Switch the TCD current to LOW. The data system signal's deflection should not be more than 5-20mV for a brand-new TCD detector. There is also a HIGH current TCD filament setting, but to avoid filament damage, we recommend you use only the LOW setting until you are familiar with your GC and TCD detector.

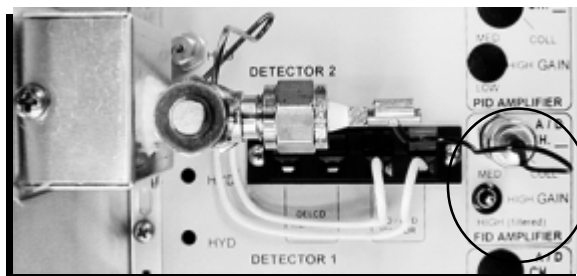


Auto Zero Button

# Quick Start GC Installation Guide

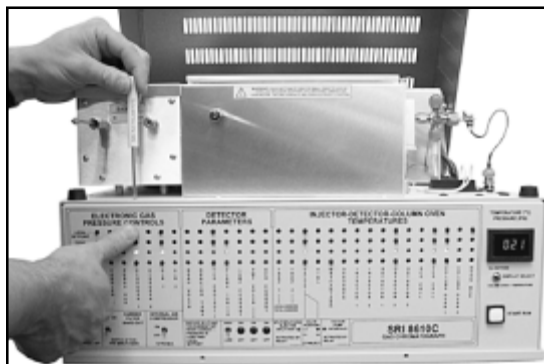
## B. FID Detector

1. Set the FID amplifier gain switch to HIGH for most applications. If peaks of interest go off the scale (greater than 5000mV), set the gain to MEDIUM.

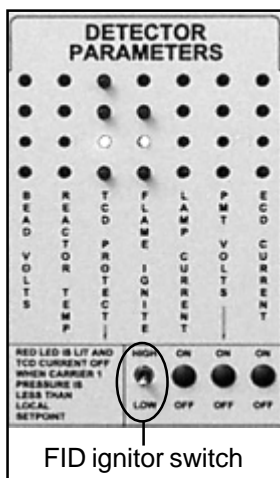
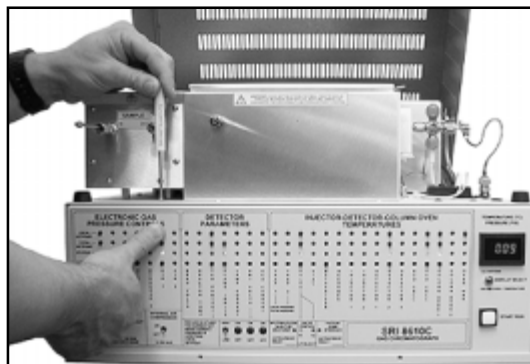


FID amplifier gain switch

2. Set the FID hydrogen flow to 25mL/minute, and the FID air to 250mL/minute. The approximate pressures required for this flow through your GC are labeled on the right-hand side of the GC chassis. In most cases, the pressure will have been set correctly at the factory. Check the hydrogen and air flow settings by pressing the LOCAL SETPOINT button while observing the LED display. The gas flow settings are adjusted using the trimpots on the top edge of the GC front control panel.

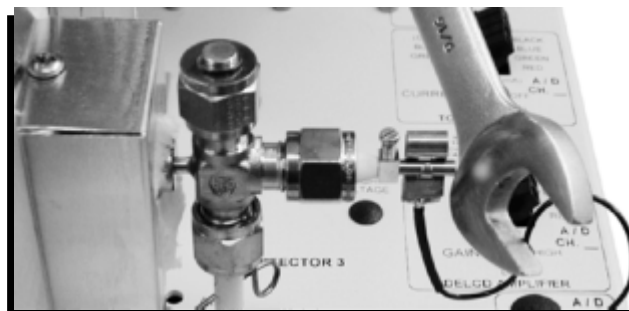


Turn the trimpot while holding down the "LOCAL SETPOINT" button until you read your desired setting in the LED display.



FID ignitor switch

3. Ignite the FID by holding the ignitor switch up for a couple of seconds, until you hear a small POP. The ignitor switch is located on the front panel of your GC under the "DETECTOR PARAMETERS" heading, with a vertical label reading "FLAME IGNITE." Verify that the flame is lit by holding the shiny side of a wrench directly in front of the collector outlet/FID exhaust vent. If water condensation becomes visible on the wrench surface, the flame is lit.



C. For all other detectors, and for more information on the TCD and FID, please see the corresponding manual sections.





# Quick Start GC Installation Guide

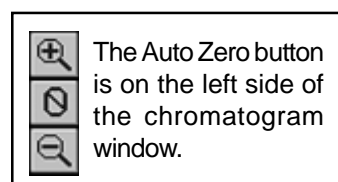
## V. Inject Your Sample

NOTE: If you are injecting with a Purge & Trap, TO-14, or Headspace concentrator, a thermal desorber, an autosampler, or any of the heated on-column injectors (PTV, Split/Splitless, etc.), please see the corresponding manual section for operating procedures.

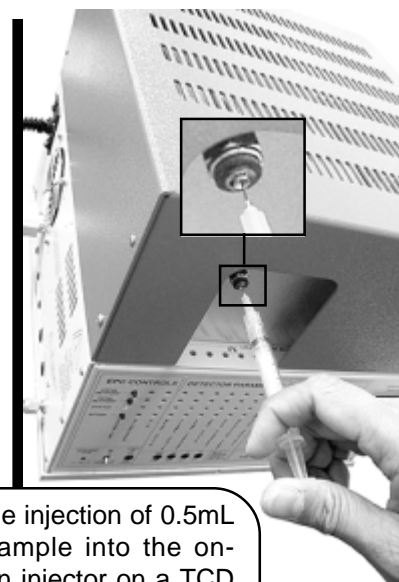
### A. Syringe Injection

1. Enter a temperature program for the column oven. The temperature program is determined by the sample and the goals of the analysis.

2. For gas samples, fill the 1mL gas syringe with 0.5-1mL. For liquid samples, fill the 10µL liquid syringe with 1µL, removing the bubbles before injecting.



3. Click on the Auto Zero button to zero the data system signal. Hit the computer keyboard spacebar.



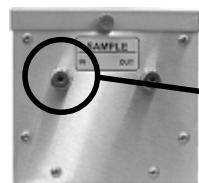
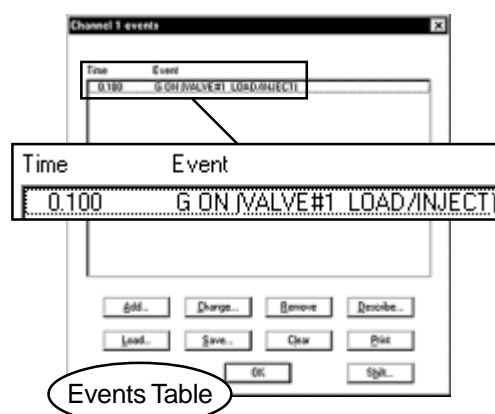
Syringe injection of 0.5mL gas sample into the on-column injector on a TCD equipped Model 310 GC

4. Pierce the septum in the on-column injector with the syringe needle. Insert the needle straight into the on-column injector port; avoid bending the needle. Depress the syringe plunger to inject the sample, then withdraw the syringe. For the best and most consistent results, use an easily reproducible injection technique with quick, smooth movements.

### B. Valve Injection

1. Set the valve oven temperature between ambient and 175°C using the trimpot on the top edge of the front control panel. Enter a temperature program for the column oven.

2. Enter an event program to automatically inject the contents of the valve sample loop. The valve is usually in the LOAD position (default), during which Relay G is OFF. When relay G is activated, the valve is rotated to the INJECT position, in which the carrier gas stream sweeps the contents of the sample loop onto the column(s). Set the valve to INJECT (Relay G ON) 0.1 minutes into the run unless you have specific run parameters that require different timing.



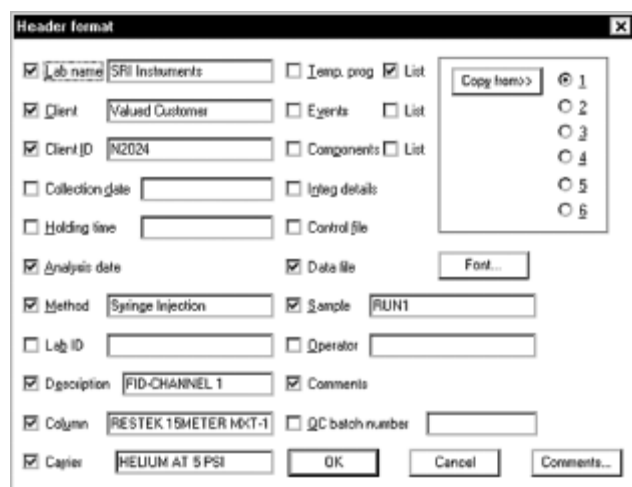
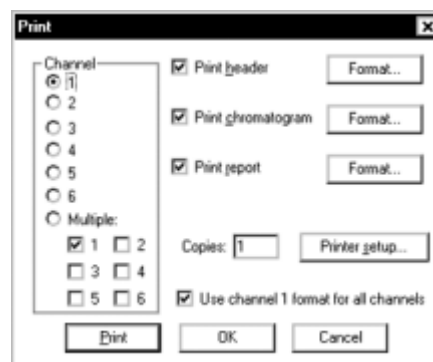
3. Sample is injected into the bulkhead fitting labeled "SAMPLE IN" on the front of the valve oven. The fitting is equipped with a 1/8" Swagelok nut for easy connection of sample streams.

4. Press the computer keyboard spacebar to initiate the run. The valve will automatically rotate to the INJECT position at 0.1 minutes (or whatever time you entered in the Events Table).

# Quick Start GC Installation Guide

## VI. Print Your Chromatogram

1. Choose File / Print from the main menu bar.
2. In the Print screen, designate which channel(s) you want printed. Use the radio buttons to pick a single channel, or select "Multiple:" and click the checkboxes to select the channels you want to print.



3. Click the checkbox to select "Print header," then click on the "Format..." button to set up the Header. The Header is printed above the chromatogram on the page, and can contain such information as the analysis date, the sample and injection type, column and carrier gas used, client and lab names, and any special comments about the analysis that you want printed with the chromatogram. Click "OK" when finished formatting your header. The Print screen is still open.

4. In the Print screen, click the checkbox to select "Print chromatogram," then click on the "Format..." button. Choose "Use screen limits" to print the chromatogram as you see it onscreen. You can also choose the chart speed, which determines the number of inches per minute displayed in the chromatogram timeline. For example, if your chromatogram is 10 minutes long and you want it to occupy 5 inches on the paper, choose 0.5 inches/minute. Click "OK" when finished.

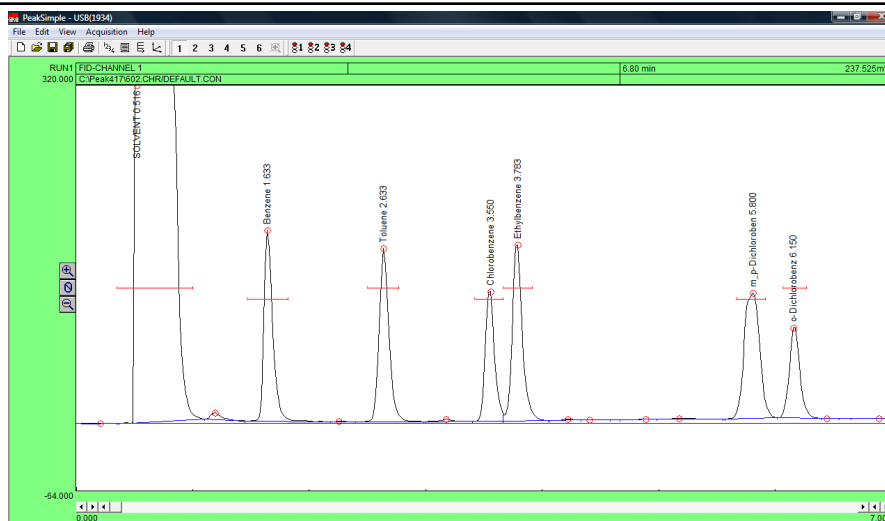


5. In the Print screen, click the checkbox to select "Print report," then click on the "Format..." button to choose the data that will be included in the report at the bottom, such as the component name, retention time, peak area and height, etc. Click "OK" when finished.

6. Now that your chromatogram is ready to print, click on the Print button in the Print screen.

# PeakSimple Advanced Tutorial

## Version 4.17, September 2012



Installing PeakSimple from the CD or USB thumb drive:

- Start the Windows operating system in use on your computer. (Windows XP, Vista, or 7)
- Insert the CD or USB thumb drive into the computer.
- Open **My Computer** and open either the CD or thumb drive.
- Double-click on the **Setup.exe** file. Make sure to select the right version of PeakSimple to install (32- or 64-bit). Windows XP and some Vista computers need to install the 32-bit version, other Vista computers and Windows 7 need to install the 64-bit version. If you are unsure, right-click on **My Computer** then select **Properties** in order to determine what bit operating system you are using.
- To complete installation follow the onscreen instructions provided by the installation wizard.
- For instructions on loading the driver, please refer to the Quick Start Documents located in the PeakSimple folder or on the [www.srigc.com](http://www.srigc.com) website.

Installing PeakSimple from software download:

- Start the Windows operating system and use an online browser to access [www.srigc.com](http://www.srigc.com).
- From the menu on the left hand side of the screen select **Download PeakSimple** and then download the latest version. Windows XP and some Vista computers need to download the 32-bit version, other Vista computers and Windows 7 need to download the 64-bit version. If you are unsure, right-click on **My Computer** then select **Properties** in order to determine what bit operating system you are using.
- Save the file to a temporary folder and double-click on the setup file when it is finished downloading, or, just click **Run** to install PeakSimple without saving the setup file.
- Follow the onscreen instructions provided by the installation wizard.
- For instructions on loading the driver, please refer to the Quick Start Document located in the PeakSimple folder or on the [www.srigc.com](http://www.srigc.com) website.



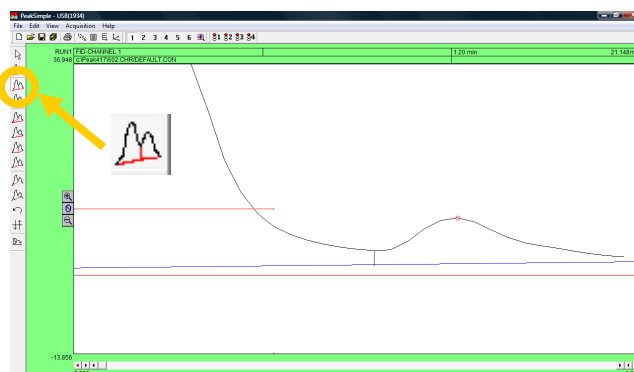
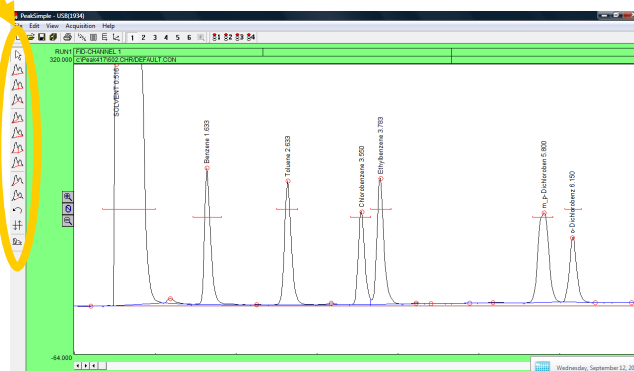


# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

### Manual Integration

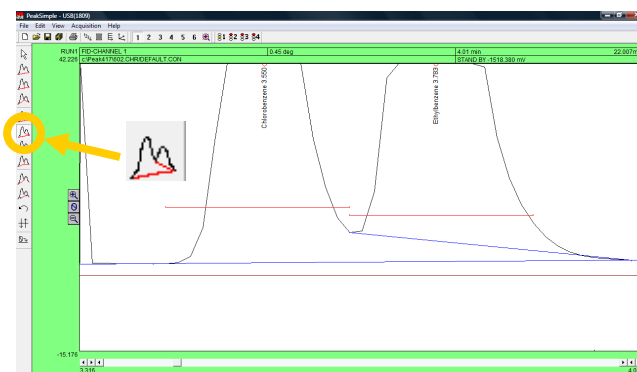
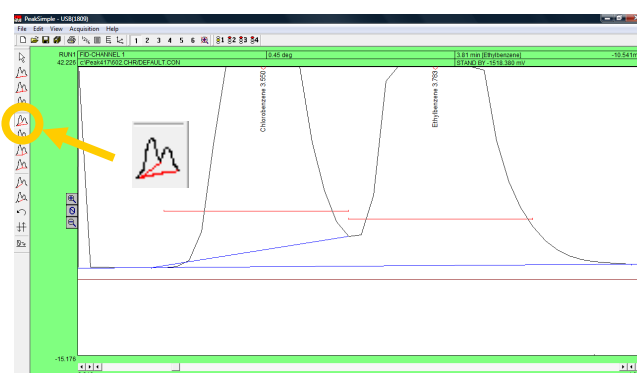
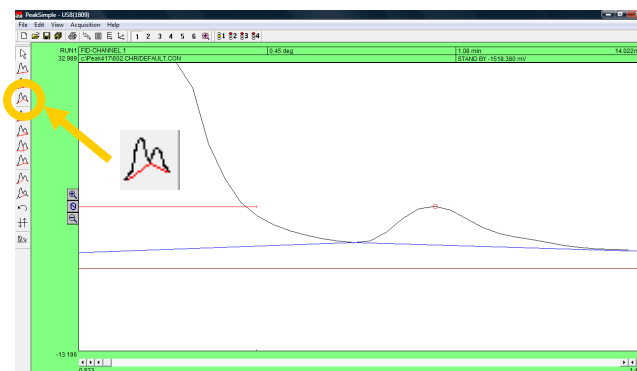
1. To manually integrate the PeakSimple baseline in a chromatogram use the manual integration tools found in the manual integration toolbar. To open the manual integration toolbar first have chromatogram 602.CHR and component file 602.CPT loaded and then select **Edit** from the PeakSimple menu bar. From the drop down menu select **Manual integration** with the mouse cursor. The manual integration toolbar will now be displayed on the left-side of the PeakSimple screen.
2. Use the None integration tool to add the area of the smaller peak to the area of the Solvent peak. First, zoom in on the solvent peak, the smaller peak to its right, and their baselines. Once the chromatogram is zoomed in select the **None** integration tool from the manual integration toolbar. With the None integration tool selected click once, using the left mouse button, on the valley between the solvent peak and the smaller peak.
3. Use the Drop integration tool to drop the baseline from the valley of the two peaks to an existing baseline. To drop the baseline select the **Drop** integration tool from the manual integration toolbar. Using the mouse cursor, click on the valley between the solvent peak and the smaller peak to drop the baseline.



# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

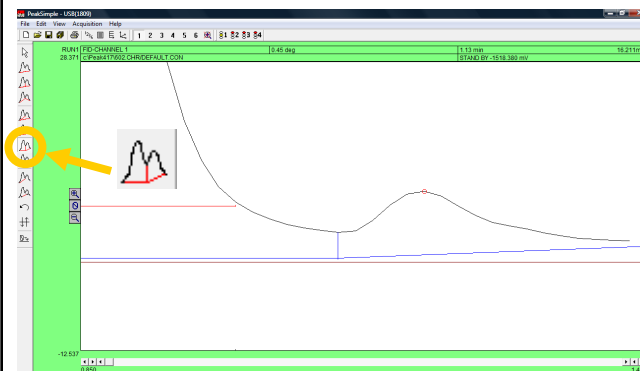
- The Based integration tool raises the baseline to the valley between two specified peaks. With the baseline dropped, click on the **Based** integration tool button and then click on the valley between the solvent peak and the smaller peak to its right to raise the baseline to the valley.
- The Lead skim integration tool allows a peak's area to be skimmed off of the leading edge of another peak. To use the Lead skim tool first unzoom off of the solvent peak and the other smaller peak and then zoom in on the Chlorobenzene peak, the Ethylbenzene peak, and the baseline. After the chromatogram is zoomed click on the **Lead skim** integration tool button and then click on the valley between the two peaks with the mouse cursor.
- The Trail skim integration tool is similar to the Lead skim tool except a peak's area is now skimmed off of the trailing edge of another peak. Select the **Trail skim** tool button from the manual integration toolbar and then click on the valley between the Chlorobenzene and Ethylbenzene peaks with the mouse cursor to see the Ethylbenzene peak skimmed off of the Chlorobenzene peak.



# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

7. The Lead horizontal tool constructs the baseline horizontally for the leading peak while the trailing peak's baseline stretches from the horizontal line to the next valley. Unzoom off of the Chlorobenzene and Ethylbenzene peaks and instead zoom in on the Solvent peak, the smaller peak to its right, and the baseline. Click on the **Lead horizontal** integration tool in the manual integration toolbar and then click, using the left mouse button, on the valley between the solvent peak and the other smaller peak.



8. The Trail horizontal integration tool drops the baseline horizontally for the trailing peak while the lead peak's baseline stretches from the horizontal line to the previous valley in the chromatogram. After selecting the **Trail horizontal** tool in the manual integration toolbar click with the mouse cursor on the valley between the two zoomed in peaks.



9. The Inhibit tool ends the baseline after a valley effectively inhibiting a peak's area from being counted with the rest of the chromatogram. To use the Inhibit integration tool select the **Inhibit** tool button from the manual integration toolbar and click on the valley of the Solvent peak and the smaller peak to its right.

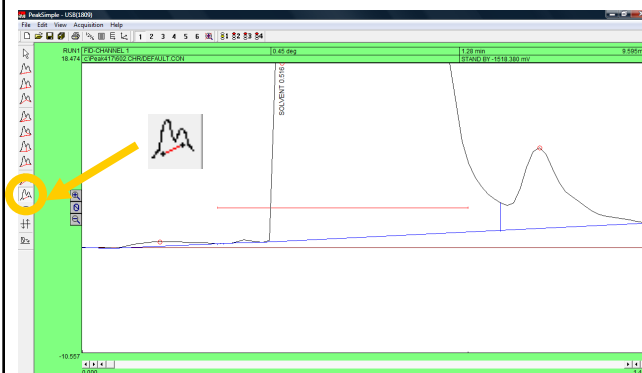




# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

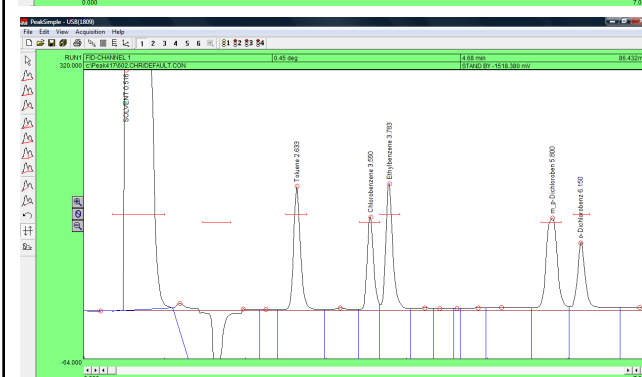
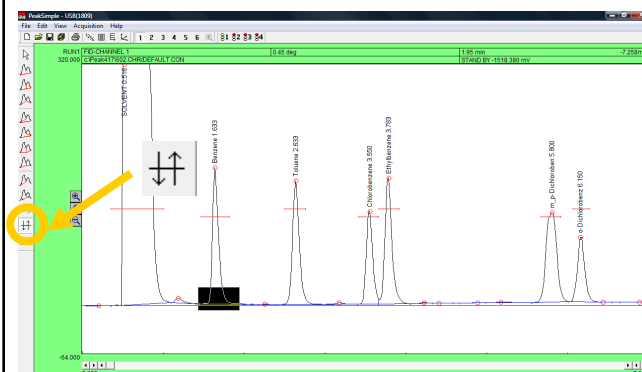
10. The Rubber Band tool is used to manually draw the baseline in a chromatogram. To use the Rubber Band tool first scroll the X-axis scrollbar all the way to the left to **0.000**. Select the **Rubber Band** tool from the manual integration toolbar and draw a line from the valley between the Solvent peak and the small peak to its left to the valley between the smaller peak to the right of the Solvent peak and the peak to its right.



11. To undo a change made to the baseline of a chromatogram with the manual integration tools use the Undo button found in the manual integration toolbar. To undo the changes made to the baseline using the Rubber band tool click on the **Undo** button with your mouse cursor. All changes made to the baseline will now be undone.



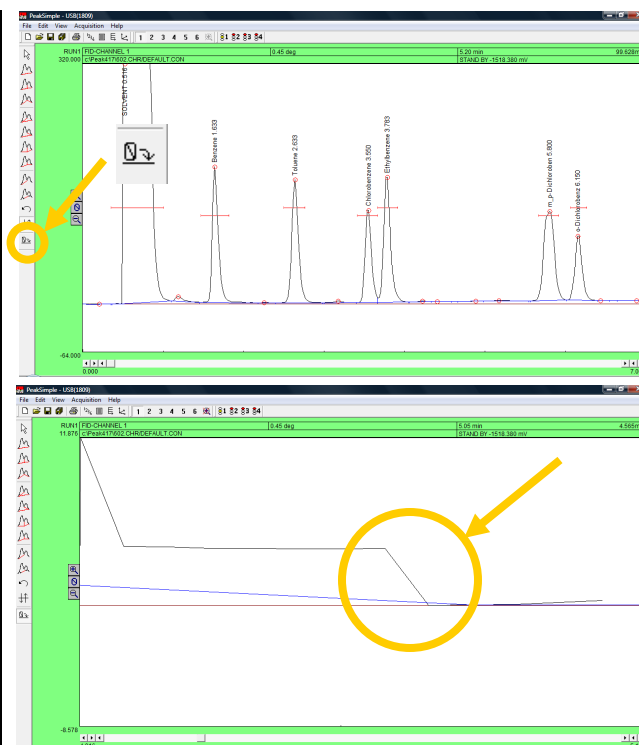
12. The Reverse tool allows the inverting of a peak in a chromatogram. First unzoom off of the Solvent peak and the smaller peak to its right and then select the **Reverse** tool from the manual integration toolbar and click and hold the left mouse button while the area of the chromatogram you want to reverse is dragged over with a black box. Let go of the mouse button when the desired area is selected to reverse the orientation.



# PeakSimple Advanced Tutorial

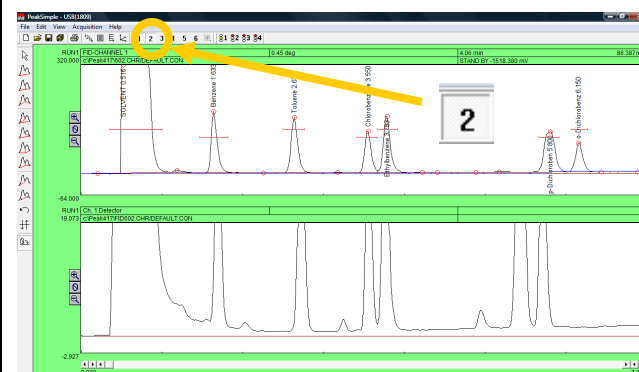
## Version 4.17, September 2012

13. The Zero tool is used to set the value of the data line at a selected point and following in the chromatogram to zero. First undo the changes done to the chromatogram by the Reverse tool by reopening 602.CHR in the PeakSimple menu bar. **Note:** Changes made to a chromatogram by the Reverse tool and the Zero tool cannot be undone with the Undo tool. Once the file is re-opened click on the **Zero** tool and click anywhere on the baseline between the Ethylbenzene peak and the two peaks to its right with the mouse cursor to set the data line at zero.



### Creating Component Tables

1. To create a component table from scratch open up a second channel in the PeakSimple window by clicking on the Display Channel 2 button in the PeakSimple toolbar. Once the second channel is open click on **File** and then **Open** to get to the Load chromatogram file window. Select the Channel 2 radio button and then file **FID602.CHR** from the list of files to open the file in channel 2. Click **OK** with the mouse cursor to load the file.



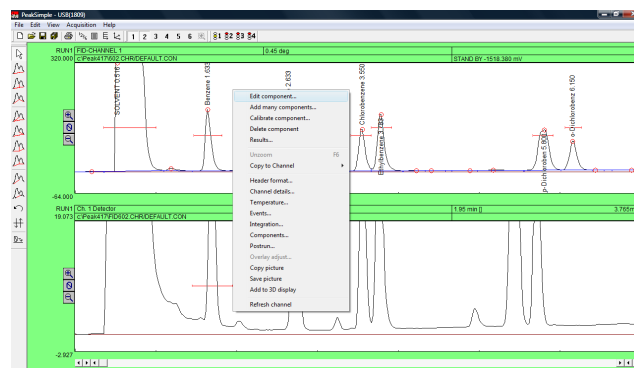
# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

2. In channel 2 locate the second tall peak from the left and right click on it with the mouse cursor. From the resulting menu select **Add component** to add a retention window bar to the peak. Once again right click on the peak and select **Edit component** from the menu to open up the Component details window.

3. Once the Component details window is open locate the Peak number dialogue box and add the number **1**. Immediately underneath the Peak number box is the Peak name dialogue box. In the Peak name dialogue box input **benzene** to name it. Locate the Units box and put **ppm** to make the units parts per million. Locate the In case of multiple peaks options box and select the radio button for **Show largest peak only**. Click on **OK** with the mouse cursor to close the window.

4. Go to **Edit** in the PeakSimple menu bar and then **Channels** from the resulting menu. The Channel controls window is now open. Locate the Channel 2 options box and the Integrate checkbox. Check the **Integrate** checkbox and then click on **OK** with the mouse cursor to close the window. The peak in the second channel should now identify itself as benzene.



Component details

Peak number: 1

Peak name: benzene

Start: 1.47 End: 1.97 Expected: 0.00

Internal standard: 0.000 Units: ppm

Internal standard peak: 0 Ref peak: 0

In case of multiple peaks:

- ☐ Show each peak separately
- ☐ Show first peak only
- ☐ Show last peak only
- ☒ Show largest peak only
- ☐ Show total of all peaks

Measure peak:

- ☒ Area
- ☐ Height

Alarms...

User calculations...

Multiplication factor: 0.00000000

☐ Calculate area as time-slice

OK Cancel

Integrate ☒

Channels

Channel	Active	Display	Integrate	Details	Temperature	Events	Integration	Components	Postrun
Channel 1: FID-CHANNEL 1	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>						
Channel 2: Ch. 1 Detector	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>						
Channel 3: Channel 3	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>						
Channel 4: Channel 4	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>						
Channel 5: Channel 5	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>						
Channel 6: FID-CHANNEL 1	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>						

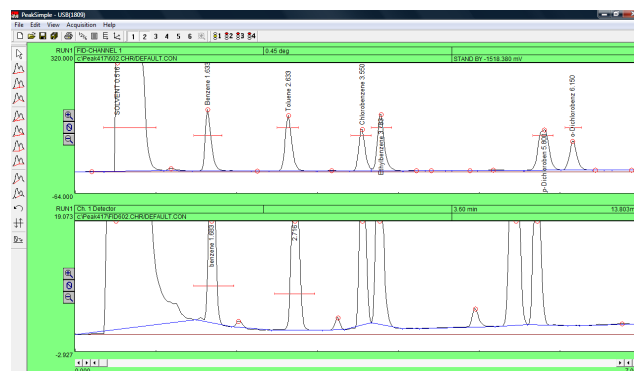
OK Cancel



# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

5. Locate the large peak to the right of the benzene peak in the second channel. Right click and then select **Add component** to add a retention window bar to the peak. Right click again and go to **Edit component** to open up the Component details window. Change the Peak number to **2**, the Peak name to **toluene**, the Units to **ppm**, and the In case of multiple peaks options box to **Show largest peak only**. Click on **OK** with the mouse cursor to exit the window.



Component details

Peak number: 2

Peak name: toluene

Start: 2.43 End: 2.93 Expected: 0.00

Internal standard: 0.000 Units: ppm

Internal standard peak: 0 Ref peak: 0

In case of multiple peaks:

- ☐ Show each peak separately
- ☐ Show first peak only
- ☐ Show last peak only
- ☒ Show largest peak only
- ☐ Show total of all peaks

Measure peak:

- ☒ Area
- ☐ Height

Alarms...

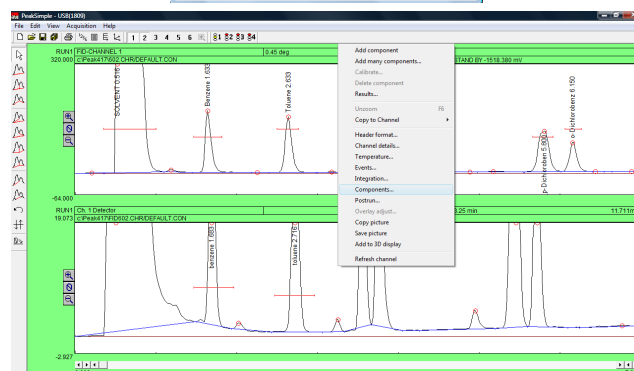
User calculations...

Multiplication factor: 0.00000000

Calculate area as time-slice

OK Cancel

6. Right click anywhere on the second channel and select **Components** from the list of options. Once the Channel 2 components window is open make sure all the data is correct and then click on **Save** to save the Component data to disk. Name the file **Ctable** and then click on **OK** to close the window. An unlimited number of component windows may be added to the component table.



Channel 2 components

Ctable.cpt

Peak	Name	Start	End	Calibration
1	benzene	1.470	1.970	
2	toluene	2.480	2.980	

Add... Change... Remove Calibrate...

Load... Save... Clear Print

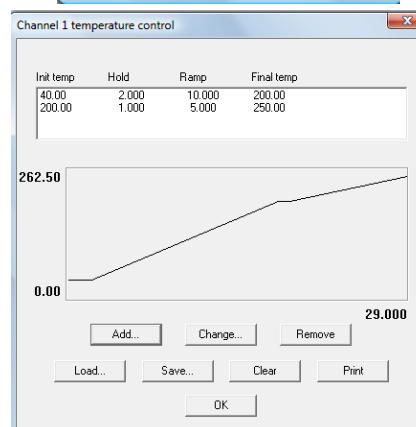
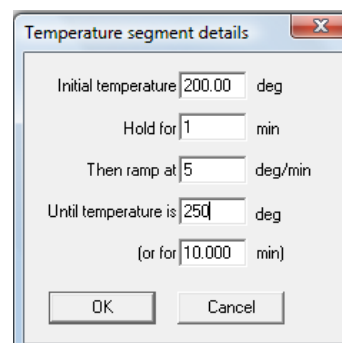
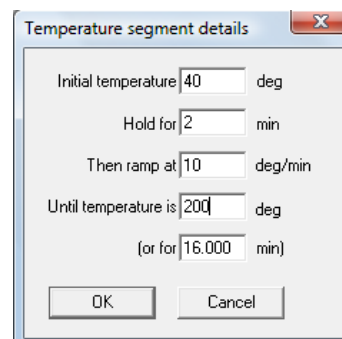
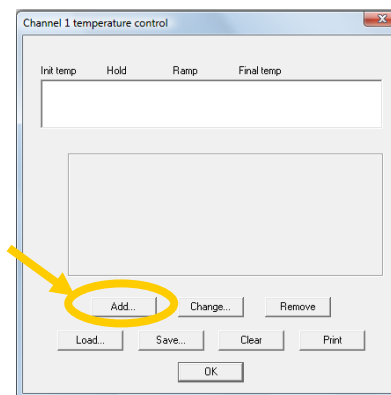
OK

# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

### Temperature Programming

1. To modify the temperature programming in PeakSimple right click anywhere on the chromatogram and choose **Temperature** from the drop down menu. This will open up the Temperature control window.
2. In the Temperature control window select **Add** from the group of buttons. The Temperature segment details window will open allowing the addition or modification of the temperature programming. Enter the numbers shown in the picture to the right in the appropriate fields. Click on **OK** to close the window and go back into the Temperature control window.
3. Select the **Add** button from the Temperature control window to open up the Temperature segment details window once again. Leave the Initial temperature at 200 and insert a **1** in the Hold for dialogue box. Change the Then ramp at dialogue box to **5** and the Until temperature is box to **250**. Click on **OK** to close the window and to see the new temperature data added to the temperature box. Click on **OK** to close the window.

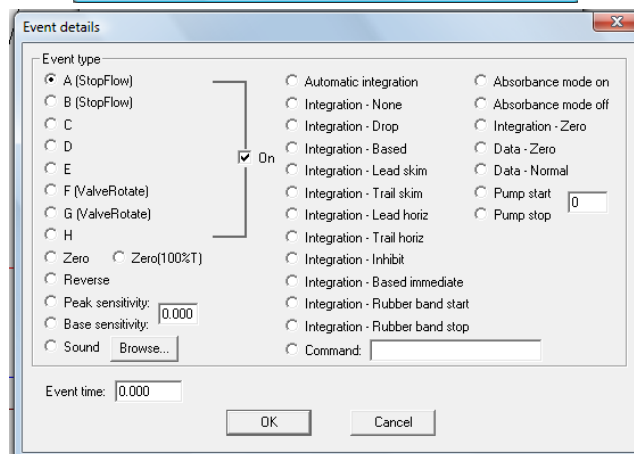
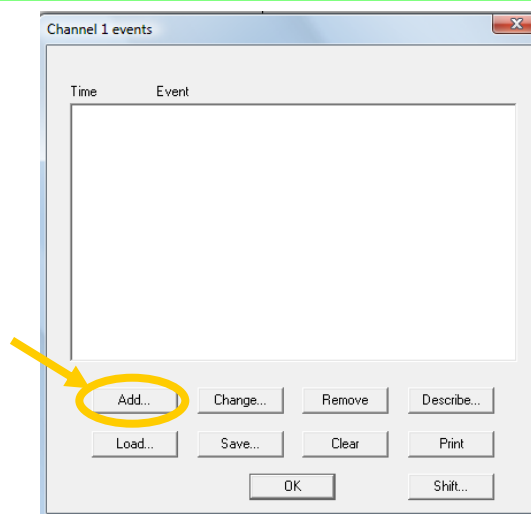
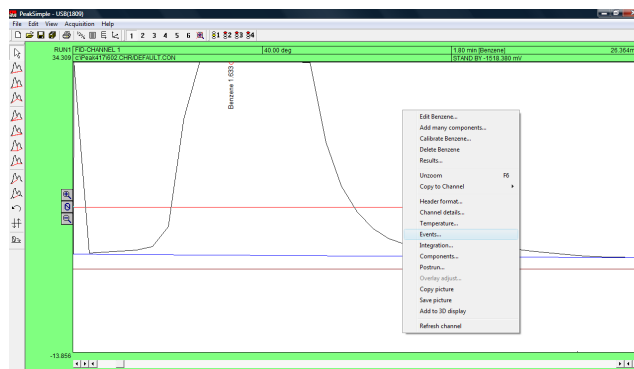


# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

### Events Table

1. To modify the Events table in PeakSimple open up chromatogram 602.CHR and zoom in on the benzene peak, the smaller peak to its right, and the baseline. Right click anywhere on the chromatogram and select **Events** from the drop down menu. Doing this will open up the Events window where specific events can be added to the chromatogram.
2. Click using the mouse cursor on the **Add...** button to view the Event details window. A list of event types are available with their radio buttons to either select or deselect the event. **Note:** *The event types to the left of the window are real-time and thus will only affect the chromatogram when A/D hardware is connected. The event types to the right are concerned only with integration and their changes will be immediately evident after returning to the main screen and selecting **Re-integrate** from the **Edit** menu bar.*



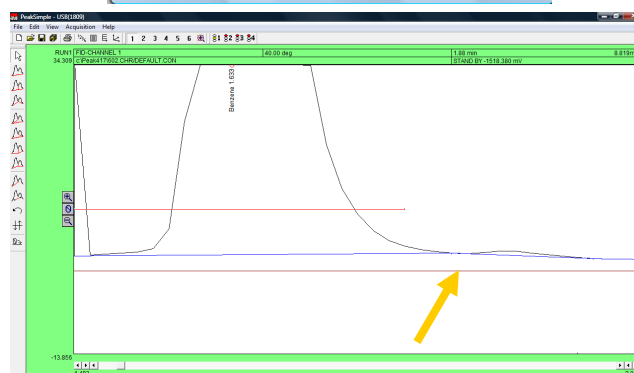
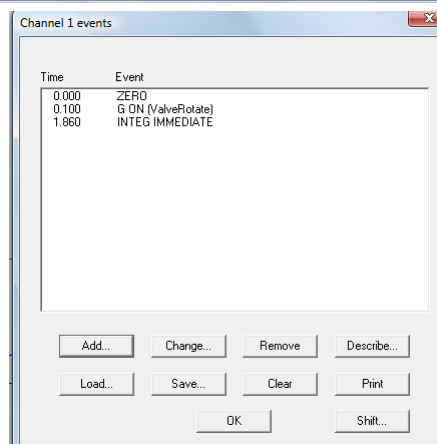
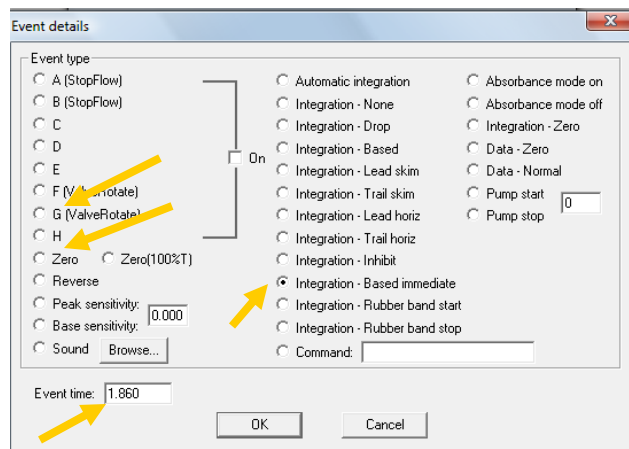


# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

3. In the Event details window locate and select the relay **G** radio button with the mouse cursor and then locate the Event time dialogue box and enter **.1** in the box. Click on **OK** to exit the window. **Note: The relay might be used to actuate a valve when hardware is connected.** The event type will now be added to the Events table. Select the **Add** button and now locate and select the **Zero** event type radio button. Leave the Event time box at 0.000 and once again click on **OK** to exit the window and add the event to the Events table. **Note: The Zero event auto-zeros the detector signal at the beginning of the run.** Click on the **Add** button again and select the **Integration-Based immediate** radio button in the Event details window and input **1.86** in the Event time dialogue box. Select **OK** to exit the window.

4. There are now three events in the Events table. Click on **OK** to exit the Events window and then hit the **Enter** button on the keyboard to reintegrate the baseline according to the events in the Events table. Notice that the baseline is connected to the data line at 1.86 minutes.

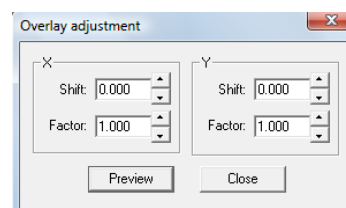
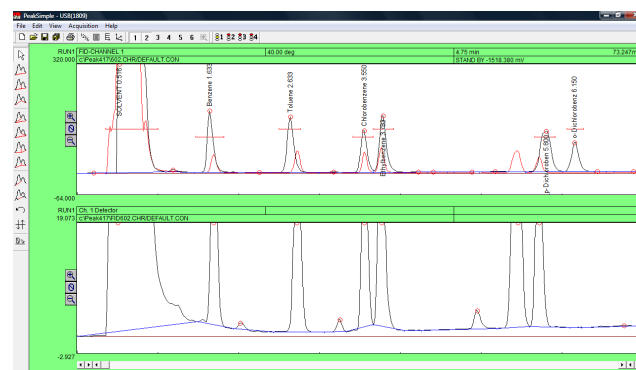
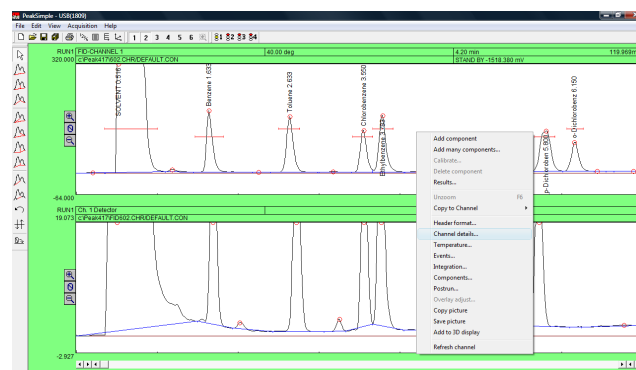


# PeakSimple Advanced Tutorial

## Version 4.17, September 2012

### Overlay and Subtract

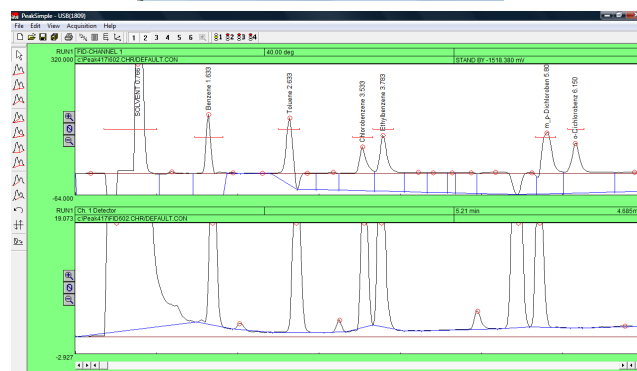
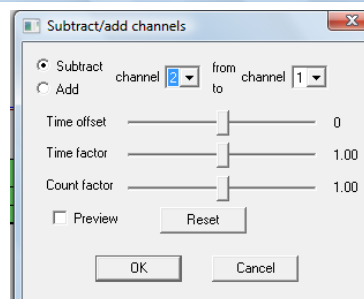
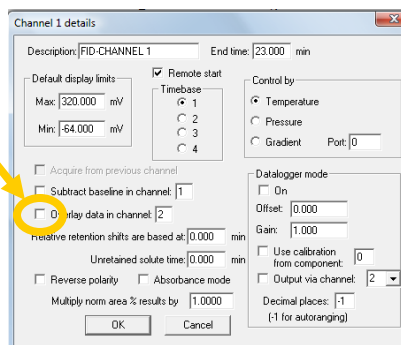
1. To overlay one PeakSimple chromatogram on top of another chromatogram open up a second channel in the main screen and load chromatogram 602.CHR in the first channel and chromatogram FID602.CHR in the second channel. Right click anywhere in the first channel and select **Channel details** from the drop down menu.
2. In the Channel 1 details window locate the Overlay data in channel checkbox and check it and then input a **2** in the dialogue box to the right. The chromatogram in channel 2 is now overlaid on top of the chromatogram in channel 1. The overlay appears in a different color.
3. Right click anywhere on the first channel and select **Overlay adjustment** from the drop down menu. In the Overlay adjustment window locate the Factor scroll box in the X box. Experiment scrolling the X factor up or down to shift the overlaid chromatogram to its right or left. Locate the Factor scroll box in the Y box and experiment scrolling the Y factor up or down to move the overlaid chromatogram up or down. Click on the **Close** button to close the window.



# PeakSimple Advanced Tutorial

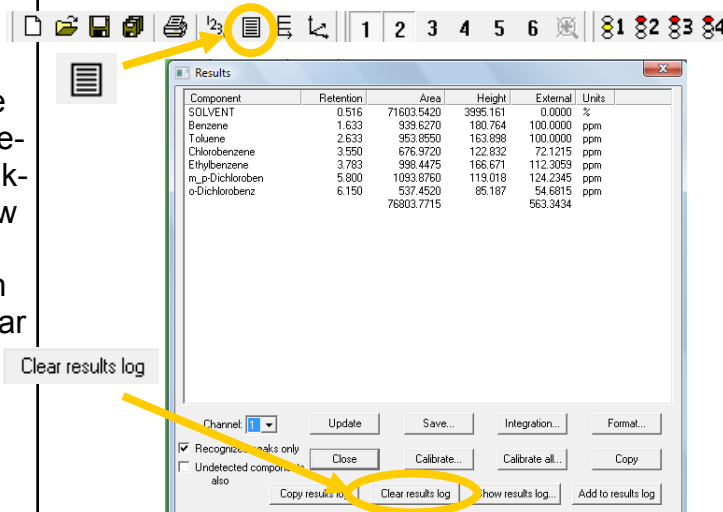
## Version 4.17, September 2012

- To subtract a chromatogram in one channel from another channel, right click using the mouse cursor on channel 1 and select **Channel details**. From the Channel 1 details window deselect the Overlay data in channel checkbox and then click on the **OK** button to exit the window.
- Go to the **Edit** menu bar and select **Subtract/Add channels** from the drop down menu. In the Subtract/add channels window make sure the Subtract radio button is selected and that channel 2 is being taken from channel 1. Click on the **OK** button to make the changes take effect and have channel 2 subtracted from channel 1. The normal way to use this feature is to subtract a drifting baseline from a chromatogram.



### Results Log

- Open chromatogram 602.CHR in the PeakSimple main screen and then select the **Results** button from the PeakSimple toolbar. In the Results window click on the **Clear results log** button at the bottom of the window. Click on **Yes** from the resulting window to clear the results.

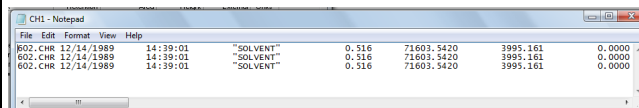




# PeakSimple Advanced Tutorial

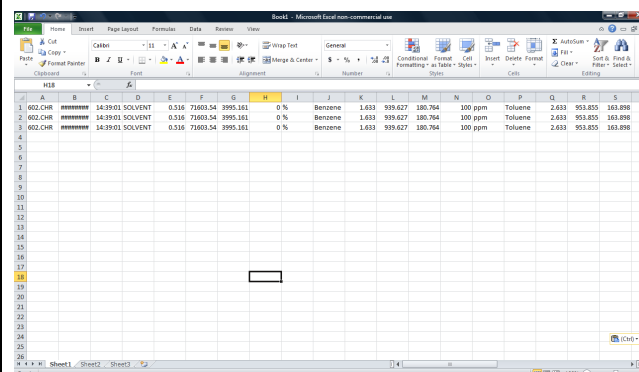
## Version 4.17, September 2012

2. Locate the **Add to results log** button and click on it three times to add the results on the screen to the Results log three times. Click on the **Show results log** button to view the results log in the Windows Notepad. Exit the Windows Notepad program by selecting **File** from the menu bar and then **Exit**.
3. In the Results window locate the **Copy results log** button at the bottom of the window and click on it with the mouse cursor (don't confuse the Copy button with the Copy results log button). Open up Microsoft Excel (or if Excel is not loaded Microsoft Word or PowerPoint) and select **Edit** from the menu bar and then **Paste** to copy the results log to Excel.
4. Go back into PeakSimple and close the Results window by selecting the **Close** button. Right click using the mouse cursor on the chromatogram and select **Postrun** from the drop down menu to open the Post-run actions window. From the window locate the Add to results log checkbox and add a check to the box. By selecting the Add to results log checkbox all results from data analysis will automatically be added to the results log after the run is done. Click on **OK** to exit the window. In this way a summary of many analyses can be automatically created and then exported from PeakSimple.

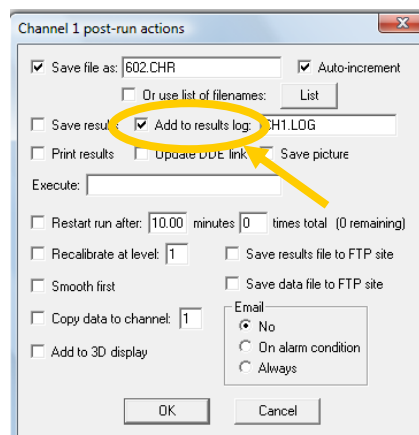


File	Edit	Format	View	Help
602.CHR	12/14/1989	14:39:01	"SOLVENT"	0.516 71603.5420 3995.161 0.0000
602.CHR	12/14/1989	14:39:01	"SOLVENT"	0.516 71603.5420 3995.161 0.0000
602.CHR	12/14/1989	14:39:01	"SOLVENT"	0.516 71603.5420 3995.161 0.0000

Copy results log



	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S
1	602.CHR	12/14/1989	14:39:01	SOLVENT	0.516	71603.54	3995.161	0.0000											
2	602.CHR	12/14/1989	14:39:01	SOLVENT	0.516	71603.54	3995.161	0.0000											
3	602.CHR	12/14/1989	14:39:01	SOLVENT	0.516	71603.54	3995.161	0.0000											



Channel 1 post-run actions

☒ Save file as: 602.CHR ☒ Auto-increment

☐ Or use list of filenames: List

☐ Save results ☒ Add to results log: CH1.LOG

☐ Print results ☐ Update DDE link ☐ Save picture

Execute: \_\_\_\_\_

☐ Restart run after: 10.00 minutes 0 times total (0 remaining)

☐ Recalibrate at level: 1 ☐ Save results file to FTP site

☐ Smooth first ☐ Save data file to FTP site

☐ Copy data to channel: 1

☐ Add to 3D display

Email: ☒ No ☐ On alarm condition ☐ Always

OK Cancel

This concludes the PeakSimple Advanced Tutorial

Further documentation can be obtained by going to:  
[www.srigc.com](http://www.srigc.com)

If you have questions or would like to place an order call:  
(310) 214-5092



# SRI MG#5 HINTS

NO OEM **EVER** tells customers THE limitations of their "designs"  
s'ware writers invariably NEVER test them in "live" situations only "ivory tower" situations  
> hence MS Windows "traumas" etc > so **Buyer B'ware !**

WIN 10 Unique! / can be unreliable Do NOT Use PeakSimple v4.88

Particularly conflicts Anti virus s'ware etc > IT Network(s) **Use 4.54 > more reliable !**

**Never use Cloud Office/XLS etc definite "latency" effects responsiveness of PS "on-line" calculations**

**similarly > be wary of "lack memory" resources effects and responsiveness of PS "Tools" PC RAM / offer-heads etc**

**Troubleshooting :**

**IF PC>GC Does NOT Connect !**

Uninstall Peak Simple completely ( as a last resort !)

- 1 Plug in PC to the SRI GC via USB Cable & After switching the GC ON
  - 2 **then** open v4.54 64-Bit PS Data System or as appropriate
  - 3 In **PeakSimple [Overall]**
- 2 Set USD Port No (eg 5216 ) but as per detail on RH Side Panel of the GC  
Set Model #333 for 1-Channel; Model 302 for 6-Ch  
SEQUENCE IS IMPORTANT  
Initially save ALL Files to THE PeakSimple Folder > becomes very cluttered > quickly . . . at least until you become familiar with the file structure of PS  
  
.CON supposedly saves all the GC parameters  
> SAVE regularly & after changes  
  
To be "doubly safe" > [SAVEALL]
- 3 Later you can transfer to a specific Method Folder
- 4 Set up [Control File .CON ] for each GC APP and save regularly ( and after any changes ) **[SAVE ALL] regularly just in case !**  
**After each PS Closedown re-load the .CON file > to reset GC parameters**

## Calibration

For (*ultimate*) accuracy > Ideally you need Custom Gas mixtures ( usually imported ) covering components of interest at several ppm levels spanning your samples to be measured .

TCD and FID are fundamentally Linear response Detectors over a reasonable range ( >x4>5 orders of Mag) but this needs to INITIALLY be confirmed experimentally . . . under the strictly defined set GC parameters required for your separation > THEN

Simple Calibration - Using CAC(1) High Concentration O<sub>2</sub>/N<sub>2</sub>,CH<sub>4</sub>/CO<sub>2</sub> at % levels Note on FID CH<sub>4</sub> is overloaded at 2.5% ( Ignore that result! ). Do a 50:50 dilution as a regular [CALIB] Check ADJUST each Component calibration table if/as needed For subsequent sample runs the ppm levels is THEN Auto-calculated

- To verify linearity > Do successive dilutions ( of CAC1 with preferably a Glass GasTight Syringe with ambient Air Minimum of 3 to 5 dilutions eg 50:50 ; dilutions >> 1000ppm > but may cause non linearity. particularly for CO@ due to "natural CO<sub>2</sub> "offset" needed. Both TCD and FID-Meth should be linear down to 10000ppm or less !

## 6 CO<sub>2</sub> in ambient Air is quite variable !

Dilution in ambient air > U must allow for the ppm actually in the Air depending on room air stuffiness ( temperature and humidity versus outside Fresh Air ~400ppm up to 1000ppm CO<sub>2</sub> . . . Human Breath is ~ 2% v/v 20,000ppm despite the "experts" saying Average World CO<sub>2</sub> is 419ppm +/- ? > **Catastrophic Climate "heat"**

## 7 The PS DDE Link to NotePAD is automatic/manual or When set ! **BUT It can be a "re-design" MESS !**

So! > **Minimise** noise peaks ( Area reject ) - format issues can be a complete mess and XLS needs re-formatting > into a more "decent" report etc.



# Op Hints > SetUp SRI GC MG#5 for Gas Analysis > Ambient Air : CO2, Methane O2/N2 eventually C1-C6 capability : H2-GEN operation, Mini Air FID-Methaniser UNIQUE GC System !

8 **MY (emphasis) suggestion** > write down ALL relevant data on paper FIRST ! Before trying to “unscramble” the XLS data  
**Keep TCD and FID LOG Files separate** 9 **Uhave many ALT Menu ways of doings things in PS . . . > so Explore them !**

10 THE MAIN CAUSE OF UNCERTAINTY IN RESULTS > errors

- Get a decent Custom made Gas Standard ( preferably CERTIFIED )
- check baseline integration Manual Integration on EACH relevant peak can be awkward > a bit “flakey”

**Simple Calibration** with much care ! **Syringe Dilution** use appropriate type PP but All Glass Gas-tight preferred

~ “?” maybe to +/-5-10%

**IF ambient CO2 =500ppM : CORRECTION**

	<b>Dilution X</b>			<b>“Actual ppM”</b>	
50:50	1	9000	250	9250	
50:50	2	4500	250	4750	Good linearity expected +/-5% or better but CHECK Via PS
50:50	3	2250	250	2500	
50:50	4	1125	250	1375	
50:50	5	612.5	250	862.5	Errors DO accumulate !
50:50	6	306.2	250	556	> non linearity ?

Preferably > ideally > Use custom STD(s)

eg for ambient+ CO2 > 0.5% std then dilute

**BUT Be wary of any Industrial Grade Gas eg AIR, N2, He re O2, Moisture content For ANY dilution can be 99% or even worse !**  
**some are misleading ie purpose created > “specific” simple gas detecors**  
**( matrix is NOT important > very crude devices ONLY!)**

- guesstimate CO2 content from initial CALIB and correct above ppM Table
- THEN Enter the NEW Data into PS calibration table for EACH Component
- Water Content of Ambient air can also effect linearity ie 0-2.0%
- NOTE : U cannot EASILY dry small ambient air samples with a MS-5A/SilicalGel Trap at small flows, low pressure or spasmodic flow rates  
despite ANY theory

**Gas Syringes** for . . .

- Quick Field transfer PP Syringe with Double Check Valve is Good ! > little loss or adsorption of “air” components
- USE a **GasTight Glass / Teflon Tipped** if in doubt or for longer term Storage
- Use Laminated Foil Bags
- BUT Suggested analysis by GC within 3 to 5 Days MAX > recommended BUT U must Check This first !
- **Useful Fittings**  
Needle-Free Valve - SmartSite > ASK !  
Our Gas Sample Bags have a 6mm thread Top Teflon Stopcock Valve
- Use a 1/4-26 Female Union (J R-060-5 )( > SmartSite ( 2000-E ) for your Syringe Connection > Don’t bother with Needles  
/on **LuerLok** Syringes





## 11 Sampling

Use the SRI GC **Vacuum Pump Interface** for Sampling I through the **Gas Sample Valves** within the GC Valve Oven

- far more precise ( Don't use a Manual Syringe Injection Port EVER ! for CALIBRATION

- automated via PS / [ Post Run ]

Optional Accessory

A **10-Port Auto Gas Sampling Manifold** is available from SRI for automating Multiple Sample Runs > ASK ! *A simple CO2 > water GC Sample Run will take about 12-15minutes run time*

*This sytem will allow overnight Sample Runs ( but Only when your overall system has proven reliable ! )*

CO2 STD 18000 ppm Diluted in ambient Air		nominal ppM	If ambient CO2 =500ppM CORRECTION	"Actual" ppM	
50:50	1	9000	250	9250	
50:50	2	4500	250	4750	Good linearity expected
50:50	3	2250	250	2500	
50:50	4	1125	250	1375	
50:50	5	612.5	250	862.5	Errors accumulate > non linearity ?
50:50	6	306.2	250	556	

BUT "ideally" Use custom STD(s) **once YOU know what U R doing !**

0.5% std then dilute BUT Be wary on any Industrial Grade Gas eg AIR, N2, He re O2, Moisture content For ANY dilution

- guesstimate CO2 content from initial CALIB and correct above ppM Table
- THEN Enter the NEW Data into PS calibration table for EACH Component
- Water Content of Ambient air can also effect linearity ie 0-2.0%
- NOTE : U cannot EASILY dry small ambient air samples with a SilicaGel / MolSieveTrap at small intermittent flows, low pressure or spasmodic flow rates despite ANY of your "theory"

THEY ONLY WORK at continuous flow after stabilisation !

**NAFION Drier** - ONLY useful where large Qtys of wet air/gases which may slowly deactivate GC Columns ( 100'samples ??? )

THEY ARE NOT QUANTITATIVE but can reduce water significantly from ~2% down to ~100-200ppM

other gases are NOT lost significantly

eg CO2 ? . . . maybe loses a few "%" from "ambient air-type concentrations"



## Gas Analysis Options - Accessories

HandPump > Bag

Gas-Tight Syringes

"proto" Martix Sample Drying

MG#5 Valve/Column Oven/Detector Layout

CAC Gas Std Cylinder 100liter

Adaptor Gas Standard Cylinder - LowDead Volumne

some Useful GC Tools

MG#5 H<sub>2</sub>-Gen LapTop SetUp > LAYOUT

Sample Fltation > for "Dirty" Samples

## Gas Sampling Bag - Multi-layer "Foil" via Syringe



**Laminated Foil**  
**500cc**  
**Teflon Stopcock**  
**6mm Thread**  
**Septim Cap**

**Double Check**  
**Valve DCB-115**

**Fem Luer-Luer**  
**Connector**

**Improvised**  
**Connector here !**  
**but ASK !**

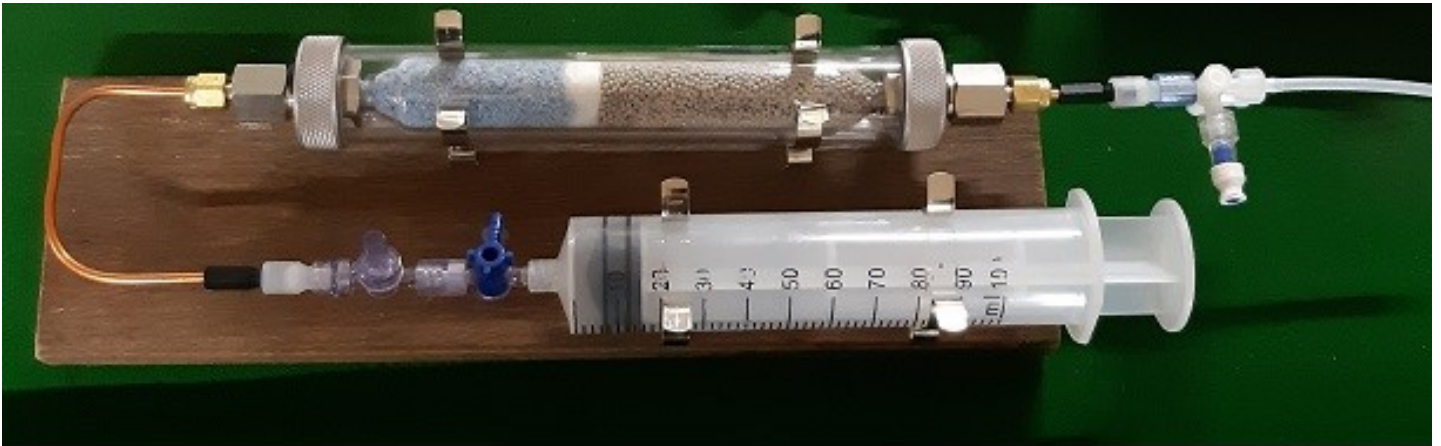
## HandPump > Bag





## "proto" Martix Sample Drying

but DON'T Bother > absolutely Useless ! concept . . . don't "re-invent" this ! )



**"prototype" Gas Sample > Purifier Air Device**  
a bit speculative >>>> ASK !  
an indicative idea ? ONLY !

**PSA Nitrogen Gen are a completely different "dynamic" prospect !**  
**& THEY DO WORK !**

**High(er) Pressure AND continuous "(high) flow rates**  
**> require stabilisation time re "pore" diffusion effects etc**



*Sample Preparation > for Dirty Samples*  
Op Hints > SetUp SRI GC MG#5 for Gas Analysis > Ambient Air : CO2, Methane O2/N2  
eventually C1-C6 capability : H2-GEN operation, Mini Air FID-Methaniser UNIQUE GC System !

## MG#5 Valve/Column Oven/Detector layout



**SRI 8610C**

**MG#5**

**Multi-Gas Analyser**

**H2 Generator**

**H2-100**

**for Bottleless**

**TCD / FID**

**Methaniser**

**Operation**

**FID has a built-in**

**mini Air**

**Compressor**

**ALT Dual Liquid  
Injection Ports**

**ASK !**

**LapTop inc**

**PeakSimple**

**Operating**

**System**

**VacuumPump Interface**  
**( inc internal H'ware PCB**

**[www.chromtech.net.au/pdf2/  
MG5-Jan2018\\_23Update2019-A.pdf](http://www.chromtech.net.au/pdf2/MG5-Jan2018_23Update2019-A.pdf)**



# CAC Gas Std Cylinder 100liter

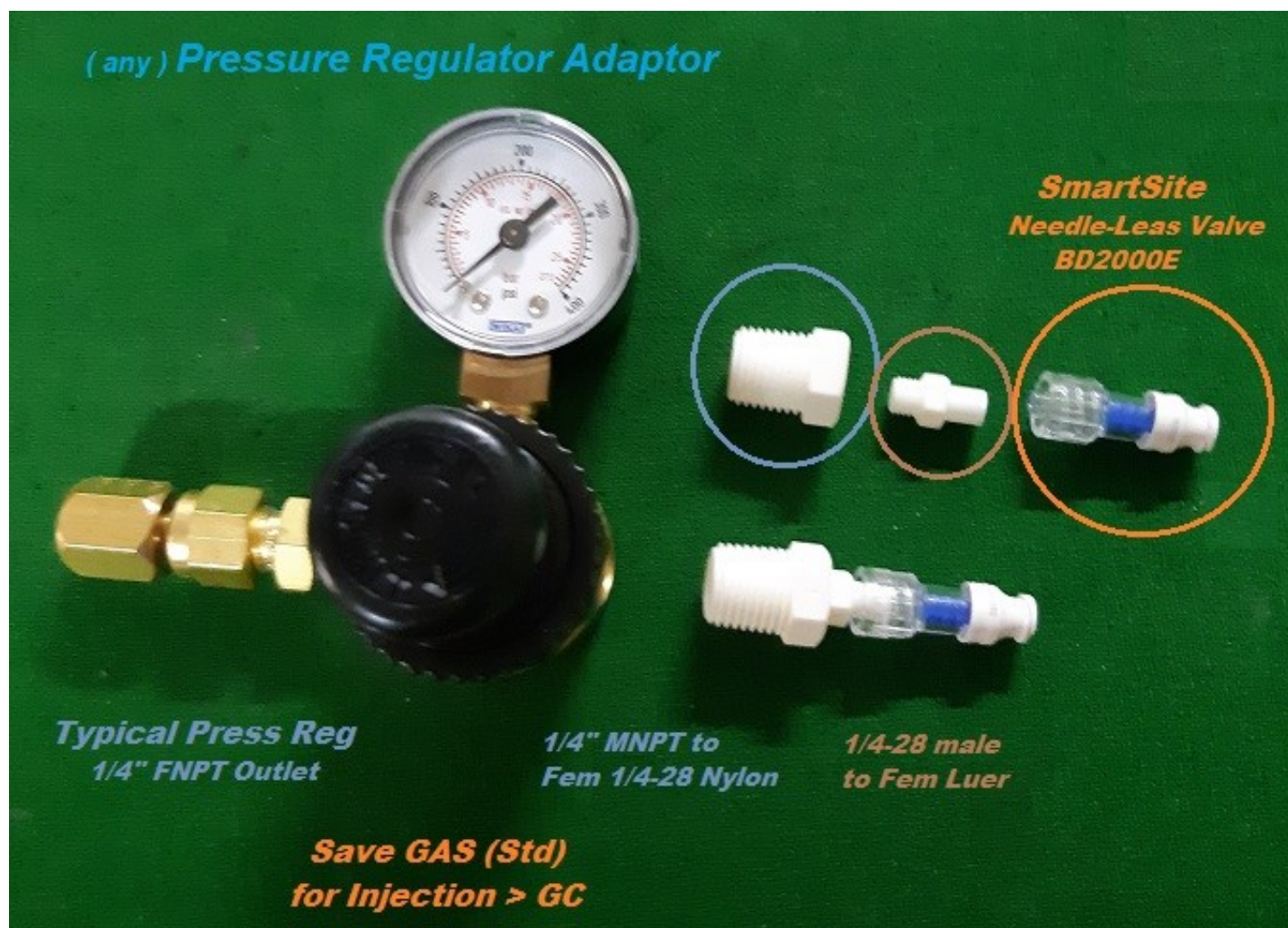
## Gas-Tight Syringes

### Sample Bags





## Adaptor Gas Standard Cylinder - LowDead Volume



## some Useful GC Tools



**some TOOLS  
extras**

**for GC**

**ASK !  
as too many  
"options"**

**Gas Analysis  
CO<sub>2</sub> / Moisture**

**GC Calibration  
"may" depend  
on ambient  
Temp /  
Humidity**

**CO<sub>2</sub> "average "  
"Worldwide CO<sub>2</sub> levels**

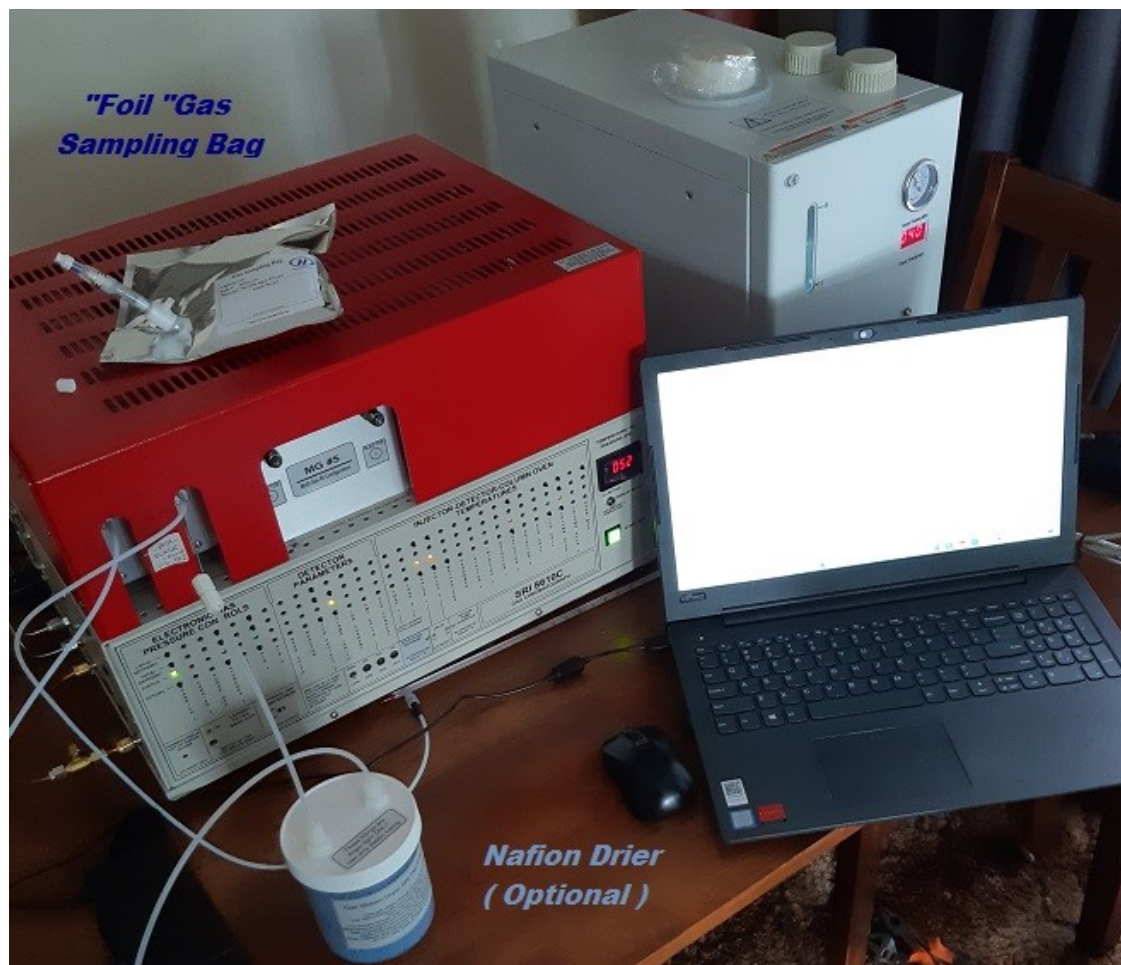
**UN "Consensus "  
is 420.0 ppm  
& rising fast !**

**a misnomer !...there is NO such thing at "ground" level 400 to 1500ppm  
CO<sub>2</sub> depending on indoor / fresh Air temp and humidity**

**city vs suburban vs forest vs agricultural ! North vs South Hemisphere !**



## MG#5 H2-Gen LapTop SetUp > LAYOUT



**H2 Generator**

**LapTop for GC  
/ PeakSimple  
Operating System**

**WIN 7 or WIN10  
IT Networked  
maybe NOT !**

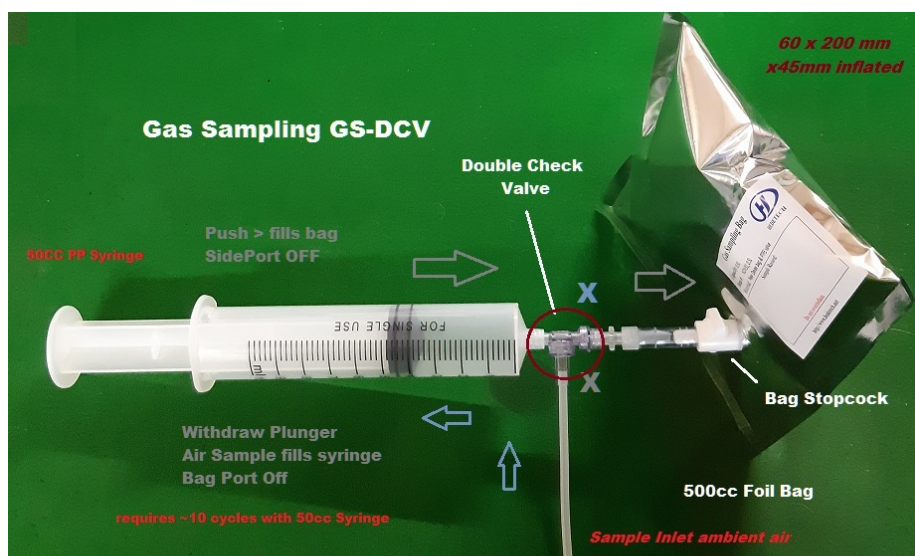
**MG#5  
SRI Multi-Gas  
Analyser  
"Bottle-less"  
GC System**

**Nafion Drier  
( Optional )**

**Vacuum Pump Interface ( Accessory )**

**Mobile Hardly ! "luggable" maybe ? Total about 50Kg**

*Fields Sampling  
from PP Syringe > Bag  
Double Check valve*

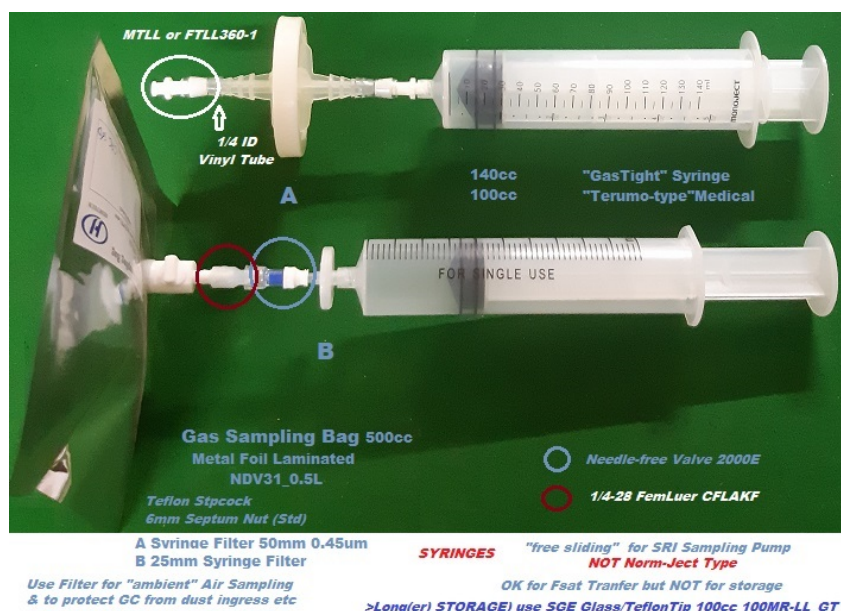


Double Check Valve DCV-01  
50cc PP Medical Syringe ( 140ml also available ! )  
OK for Inert Gases  
Use a Glass Gas-Tight ( Teflon -Tip plunger ) for reactive solvent vapors ) and Tedlar-type bag

PP, Polycarbonate/Silicone; Teflon components  
LuerLok fittings

Sampling > use Syringe Filters > to protect GC from contamination & damage long-term > to GC Gas Sampling Valves > Columns

## Sample Filtration > for "Dirty" Samples



### Gas Sampling Bags - Re-Usable ?

- for higher concentration "inert" gases Air / CO<sub>2</sub>
  - simple evacuate a few times with a Vacuum or Hand Pump or simply "press flat" manually
  - purge a few times with "clean air" if available Or with new Sample )
- Trace levels ( <100ppM? ) or more reactive gases > test !



# Peak Simple Detail I - Operating Hints

Peak Simple IS the Operating System for ALL SRI GCs and PROVEN Over 30+years

**IT IS A MOST POWERFUL TOOL . . . USED PROPERLY !**

but NO s'ware is infallible ! . . . not without their own "quirks" largely due to practical ignorance of Applications s'ware writers "themselves"

Inevitably end-users are left to their own devices / "intuition" . . . to work around issues

> with Many "tricks" of the the trade . . . for the unwary ! so **ATTENTION To "THE Detail" IS Required !**

[Events Table]

[Post Run] setup > File "Run Labelling", repeat runs AND DDE Link to XLS .log etc

[CALIB] see pages here ( coming soon ! )

for ultimate accuracy and proof of method certification

**Ideally Gas Standards are needed for each Gas component the APP requires** ( multi-component mixes are generally OK

- but these can be very expensive particularly if certification is required)
- in the concentration range required ( "stradle" the expected sample peak concentration ! )
- DO NOT EXTRAPOLATE over wide range ( eg x100 for TCD/FID ) UNTIL PROVEN

. . . **CHROMTECH Are Satisfied with Reason !**

preferably use the same sample matrix

**SIMPLIFIED** eg CO2 water in ambient AIR gives potentially SIMPLE

Use a High Concentration ( certified standard high purity matrix )

but DON't use /rely on "compromised" stds ( eg wet air /impure matrix gases) . . . maybe appropriate for "specific-type" gas detectors )

- "dilute in a Syringe" ( proven accurate over x6 50:50 dilutions ) . . . > for a simple **Detector Linearity check !**

or in a Gas Sampling Bag ( Metal Foil Laminated or Tedlar and as appropriate )

- PP Medical Syringes are gastight but only reliable for short term high concentrations and quick transfers )
- SGE All Glass Gas Tight Syringes are preferred / recommended

IF ambient air is used as diluent gas THEN

allow for "matrix" background levels eg CO2 and Water in air

but Note **CO2 ambient !**

> there is No such thing as an average CO2 level at ground level ( room vs external etc ) that makes ANY sense

. . . desert/tropical, land sea; urban vs country-side; altitude above sea level; Nth /Sth Hemisphere or the surface of the MOON ! for that matter !

O2 17%

N2 78%

Methane 2.5%

CO2 1.8%

plus supposedly CO 40ppM ( not present from Chromtech experience )

plus Water estimated at ~420ppM

despite what the "global consensus" might interpret 1

. . . levels can be from the generally accepted World "average" of 400ppM to 1000ppM or more in confined rooms / vehicles or enviro situations



for a diverse range of associated products  
and SRI Options / Accessories  
Syringes, Gas Sampling bags, tubing etc

Getting [RESULTS] **IT CAN BE DONE** and can give good 'ball park' Calib data > And SIMPLY ! . . . but after YOUR "Learning Curve"

**TCD** measures ALL likely components

base on GC Peak Area [RESULTS]

linearity to reasonable accuracy is known ( reaffirmed here by Chromtech "experimentally" for the "CO2 in Air" project )

1 providing "Mlar Response Factors are used

a **Single Point Calibration**

**Chromtech** has used ( temporarily ) a Gas Standard from **CAC**

**CAC Gas Reference Standard**

O2 17%

N2 78%

Methane 2.5%

CO2 1.8%

plus supposedly CO 40ppM ( not present from Chromtech experience )

	CEC Assay %	#27 AMB AIR	a%	CEC 100% #17	AREA %	fx	
		GC Measured					
o2/Ar	17.9	1258	21.88	21.37871	952.90	17.0789	1.048
n2	77.38	4569	78.09	77.64651	4436.00	79.50676	0.973
ch4-1	2.5	0			99.60	1.785138	1.400452
air/co		7000.3			4593.00		
ch4-2					85.79		
co2	1.8	3.26		0.055401	67.40	1.208015	1.490047
water 65%H21C	0.42	54.1		0.919386	23.50	0.421192	
16260 ppM	100	5884.36			5579.40		

In **SRI PeakSimple** >

**TCD** Simply go to for example CO2 CALIB from [RESULTS] enter 18000 (ppM) and Area 67.4

A SinglePoint line is indicated

This has been substantially verified for both CO2 and CH4 as being linear and valid over the range 2% - approx 0.1% ppM

But can be assumed to extend low to 400ppM

Errors may occur due to overlapping peaks and the Manual Integration Tool being used

- For simplicity perhaps use the TCD CO2 Peak for range above 1000ppM

NOTE : the CO@ peak does NOT elute from the MolSieve 4A Column ( first half of the chromatogram )

**FID-Meth** > A similar linearity extends from 2% down to an estimated 20ppM as a Limit of Detection

Similar results can be obtained using either [CH4-1] or the [CH4-2] Peak if present in Samples with the corresponding CALIB Data

- For lower levels of CO2 the FID-Methaniser is obviously more sensitive

- Either peak can be used for higher levels on TCD And FID or for lower levels FID for CH4-2

the following pages attest to CHROMTECH experimental Validity if the SRI MG#5 results with some emphasis on some of the finer points using PeakSimple  
many hours of work > consult as needed may save end-user a lot of time . . . but experiment with care ! NO Data is lost in PeakSimple

**Once a Sample RUN is initiated U can work elsewhere on Your PC and PS runs in the background !**



for a diverse range of associated products  
and SRI Options / Accessories Syringes, Gas  
Sampling bags, tubing etc



# My Advice **VIP**

1 H<sub>2</sub> is being used as Carrier Gas + FID-Meth Fuel Gas

If there is any need to disconnect column (DON'T) to regenerate MolSieve (at 300degC)

- **Check for leaks THOROUGHLY** (DO NOT USE SOAP SOLUTION ("SNOOP") UNDER ANY CIRCUMSTANCES

partial leaks can "back-seep" into fittings columns etc and can cause "bleed" havoc

Buy a H<sub>2</sub> Leak Detector ! SRI 8690-5600.pdf (~A\$750) or Restek Leak Detective 28500 (~A\$2300) IF YOU *have any doubts whatsoever* !

- maybe use Methanol/Water mix from a syringe OR rely on "pressure drop" techniques

40 ml/min (the abs Max for MG#5) of H<sub>2</sub> leaking into the GC Oven (500cc) just maybe builds up to a "potentially explosive hazard (4% LEL in Air) (miniscule as it may be ?, because of the high speed GC oven fan ! etc)

2 If GC is on StandBy TCD Oven is at 125C but filaments are redhot) TURN FILAMENTS OFF to conserve their lifetime !

3 IF ? WHEN the GC "Red Lid" is up ALL Power to heaters is tuned OFF ) > need to restabilise !

## **SRI GC Start-Up sequence**

1 Turn H<sub>2</sub>-GEN ON > Pressure set at 50psi Tank may require 10mins to reach and stabilise flow (normally at 40ml/min (for the MG#5)

2 After 5 mins turn GC Power On

If H<sub>2</sub> Flow OK thru columns the Green LED On Carrier EPC is Green

If RED then no flow thru' the TCD (check for leaks ! ) > Filament Protect circuit for filament safety )

3 Check TCD Oven stabilises 125C takes 10mins, FID-MET maube 20mins

4 Turn Filament GAIN Switch (normally for HighSensitivity for Air analysis/CO<sub>2</sub>) > "Hi" 5

Ensure H<sub>2</sub> Flame is alite (goes "pop" or check at FID Chimney with "fog" on a cold spanner" 6

Plug-in GC USB Cable to PS DataSys

Then load **PeakSimple DS... experiment ! any data collection is live in realtime and PS is "multifunctions" ) U wil not loose ANY live data !**

IF Correct PS Model is set to #302 for 6-Ch, AND the USB Port No (eg 1512) on RHS GC panel) PS {EDIT}>{OVERALL}

THEN PS should "log" in OK and be "active" ( "standby" / flickering mV signal on Top Menu Bar > confirms this !

Details PeakSimple instructions see **PEAK SIMPLE BASIC (p78) ADVANCED Section (p98)**

**Superficial maybe ? APP specific detail Screen shots on following pages**

7 Never BOTHER with Syringe Injection and Manual Injection port )

Always use the VacPUMP Interface for Injection of Samples

Automated via [Events TABLE],

Label Sample Run File [POST RUN]

SET Column Temperature program [TEMPERATURE]

Inject a "room" air sample

**CALIB MUST** Be checked regularly AND / OR (if in ANY Doubt) or if ANY changes in ANY GC parameters have been made

8 Inject CAC Gas Standard

Identify PEAKS of INTEREST in chromatogram SET Peak "Window" for peaks that ARE TO BE INTEGRATED

Optimise Base-line and Integration Parameters

ENTER ONLY SENSIBLE Peak Area Data in Component Integration Channel within PS [CALIB]

SUBSEQUENT Results are THEN AUTO-CALCULATE relative to that CALIB

**"Guessimated" Accuracy non-linearity od Syringe Diltion +/-2-5% of measured value for CO<sub>2</sub> / CH<sub>4</sub>**

9 Gas Samples (actual)

For real samples from "the field" use a **Syringe Filter** to remove dust/dirt (can cause long term adsorption problems within the GC and GSV damage ! ) (see p122)

- PolyProp Medical Syringes are Gas-Tight and OK for fast transfer < 60mins storage ? ) for "Air" but not long term due to adsorption permeability at trace levels ( use **SGE Gastight Teflon-Tipped Plunger** type Glass Syringes ( expensive ! ) ( free sliding type are needed for the **SRI Vacuum Pump Interface Auto-Injection** device (p121)

- Use **Gas Sample Bags** for transfer from "field site"

- Air samples > permanent gases suggested storage time MAX of 5 to 7 days ? ( Laminated "Foil" bags )

: Tedlar Type gas are recommended for more reactive VOCs

: highly "Inert" "SF<sub>6</sub> U can use either type ? ( "Foil" bags preferred ? ) to maximise storage time at trace levels ( *ppT or less lifetime ???* )

: ultimately for trace levels VOCs use **Canisters** ( TO-14 or Silcosteel > expensive ! ) ( **Restek** )

**10 Results Export to MS Excel via Notepad - SET Area Reject to eliminate noise and non-interest peaks to simplify Excel < Label and Set Log in [PostRUN]**

**Separate Detector [LOG]Files foto avoid "clutter" of [XLS] fields**



> for a diverse range of associated products and  
SRI Options / Accessories  
Syringes, Gas Sampling bags, tubing etc

[BaseLine Tracking] > automatic BUT Check visually AND Adjust as necessary  
as [ Manual INTEGRATION ] may still be necessary to avoid Area "errors" see these pages 122-125

from MS5A Column

FID - Methaniser

SRI 8610C MG#5

from HS-D column

PS Manual Integration TOOL # 9

NOTE > some PS Tools are "flakey"

PS

Autotracked ( depends on Integ Settings )

Manual Correction > Re-integration / update required

BEFORE

AFTER

3125ppM

CH4-1

4591

4937

CH4-2

5717

5688

& perfect !

~2600ppM

CO 2 (as methane)

5077

4220

X-X

X-----X  
Z                  Z

Z

Z

" visually check baseline & manual correct as needed Else errors of 10-15% as above "

same GC Run (dual columns)

CH4-1 and CH4-1 should in theory be same A but ! different GSV Loop size,  
Column Flow Rates / elution temp but ???

sample GC #21Mar20 #21

**Peak Integration** see Peak Sensitivity Peak %, Baseline pre GC run > **no manual override post run !**

% Area reject override OK post run ( reduces noise peaks and minimises Data Log XLS "mess"

**Event Table** > X---X Valve switch cause noise peaks so PS turned Off to avoid base-line/integration "huge BL Errors"

[Z] occasional auto-baseline correct > Zero pre-set to offset "any" BL drift

**GC Detector Sensitivity** : TCD / FID Both have over-ride GAIN Setting > DO separate Calibration on each AND Do NOT MIX / so  
optimise FID A/D overloads at ~ 20000ppM with normal 1cc loop on HIGH > can even get "flat-top" (>Errors) or "double" peaks ( may still

RAW DATA SRI GC MG#5 accessed from WEB [www.chromtech.net.au/pdf2/SRI-DATA](http://www.chromtech.net.au/pdf2/SRI-DATA)

Peak Identified and Integrated  
ONLY if straddled by pres-set (red) "Window"

FID-Meth CO2 (as methane)

from MS5A

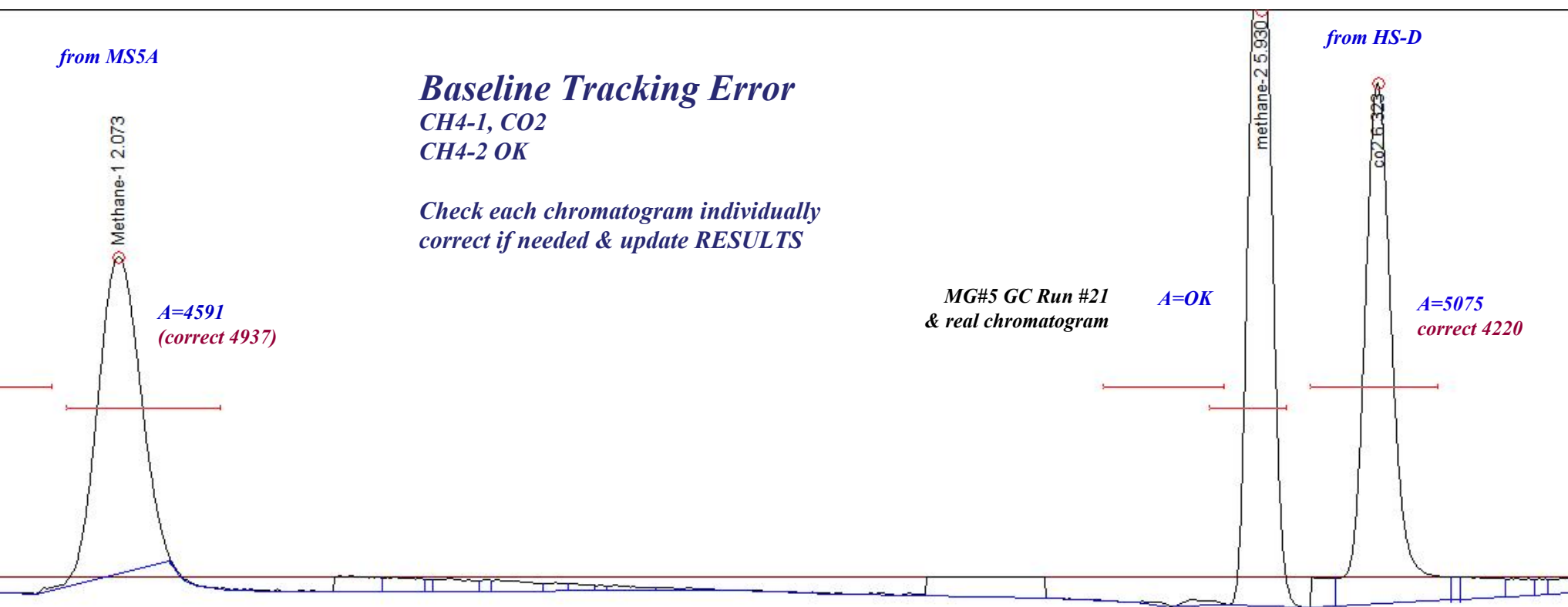
## Baseline Tracking Error

CH4-1, CO2

CH4-2 OK

Check each chromatogram individually  
correct if needed & update RESULTS

from HS-D



Integration Parameters  
- supposedly

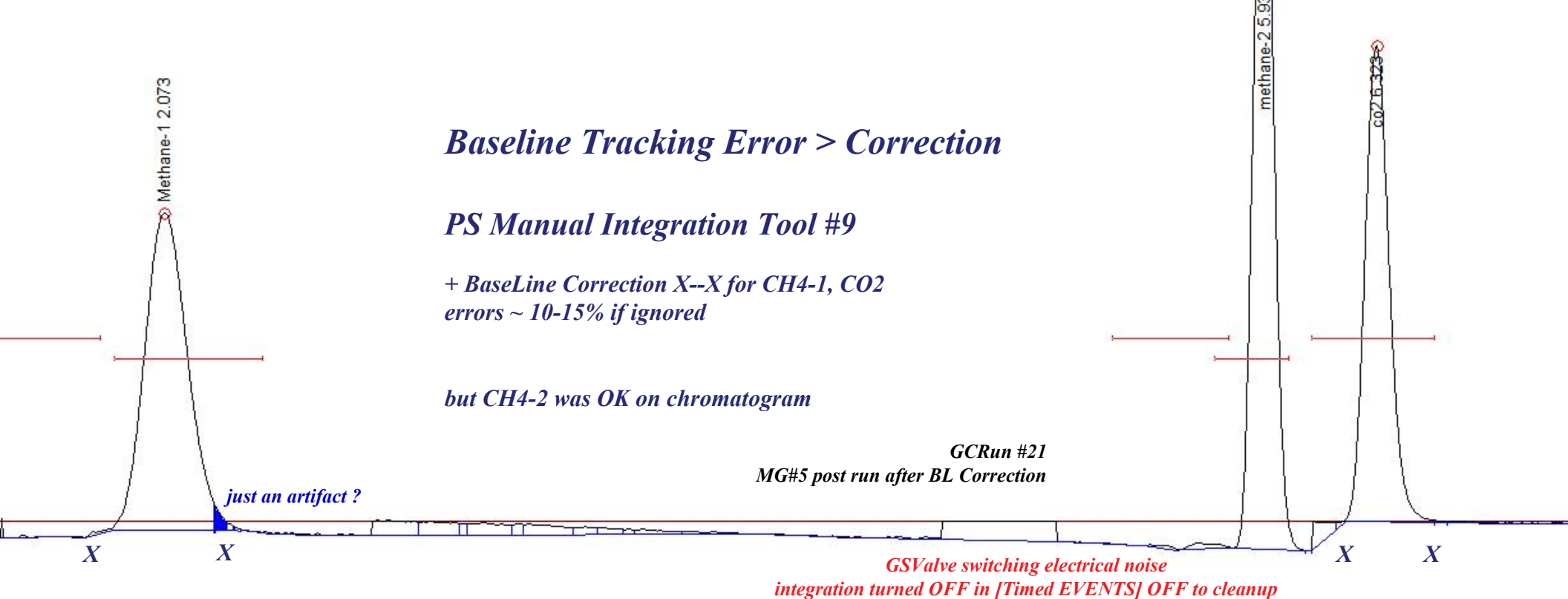


## Baseline Tracking Error > Correction

### PS Manual Integration Tool #9

+ BaseLine Correction X--X for CH4-1, CO2  
errors ~ 10-15% if ignored

but CH4-2 was OK on chromatogram



### MORE Hints

- to “remove” a component from [RESULTS] simply turn off [[Peak Window] then > Zero AREA eg CO/Air and 2 x Methane Peaks is a “double up” peak as artifact of requiring dual column MG#5 set up
  - Optimise the Manual Integration Tool for Best Results
- THis tool can be a bit flake ( nonfunctional depending on PC internal resources ( RAM Network Speed . . . > whatever . . . be patient !
- Set [Area Rejcct [ in [INTEGRATION} to a sensible value to remove integration “clutter in display BUT MORE-SO in the “clutzy” “Data LOG” and import into Excel **OTHERWISE IT IS A MESS !**

Peak Integration  
Tool  
(a bit "Flakey")

Component	Retention	Area
o2	0.000	0.0000
n2	0.000	0.0000
Methane	2.073	4591.2872
CO Trace ( IF present )	0.000	0.0000
Methane-2	5.930	5688.3018
co2 ( Methanised )	6.323	5077.4920
water?	0.000	0.0000
		15357.0810

1.	2.0000	0.0000	0.0000	1	0.0000	0.0000
2.	1.2500	50.8000	0.0000	0.0000	1	50.8000
3.	0.6250	22.2000	0.0000	0.0000	1	22.2000
4.	0.3125	10.45	0.0000	0.0000	1	10.4500
5.	0.0000	0.0000	0.0000	0.0000	1	0.0000
6.	0.0000	0.0000	0.0000	0.0000	1	0.0000
7.	0.0000	0.0000	0.0000	0.0000	1	0.0000
8.	0.0914	3.6290	0.0000	0.0000	1	3.6290

Statistics...

Use

☒ Current area/height only

☐ 1 previous area also

☐ 2 previous areas also

Accept new

Method...

Load...

Save...

Clear

Print

Copy to all other components

Copy

Curve fitting options

☐ Single line through origin (Ax) [average CF]

☒ Single line through origin (Ax) [least squares]

☐ Single line (Ax+B)

☐ Multiple line segments

☐ Parabolic (Ax<sup>2</sup>+B)

☐ Quadratic through origin (Ax<sup>2</sup>+Bx)

☐ Quadratic (Ax<sup>2</sup>+Bx+C)

☐ Power (Ax<sup>B</sup>)

OK Cancel

(blue) auto base-line tracker

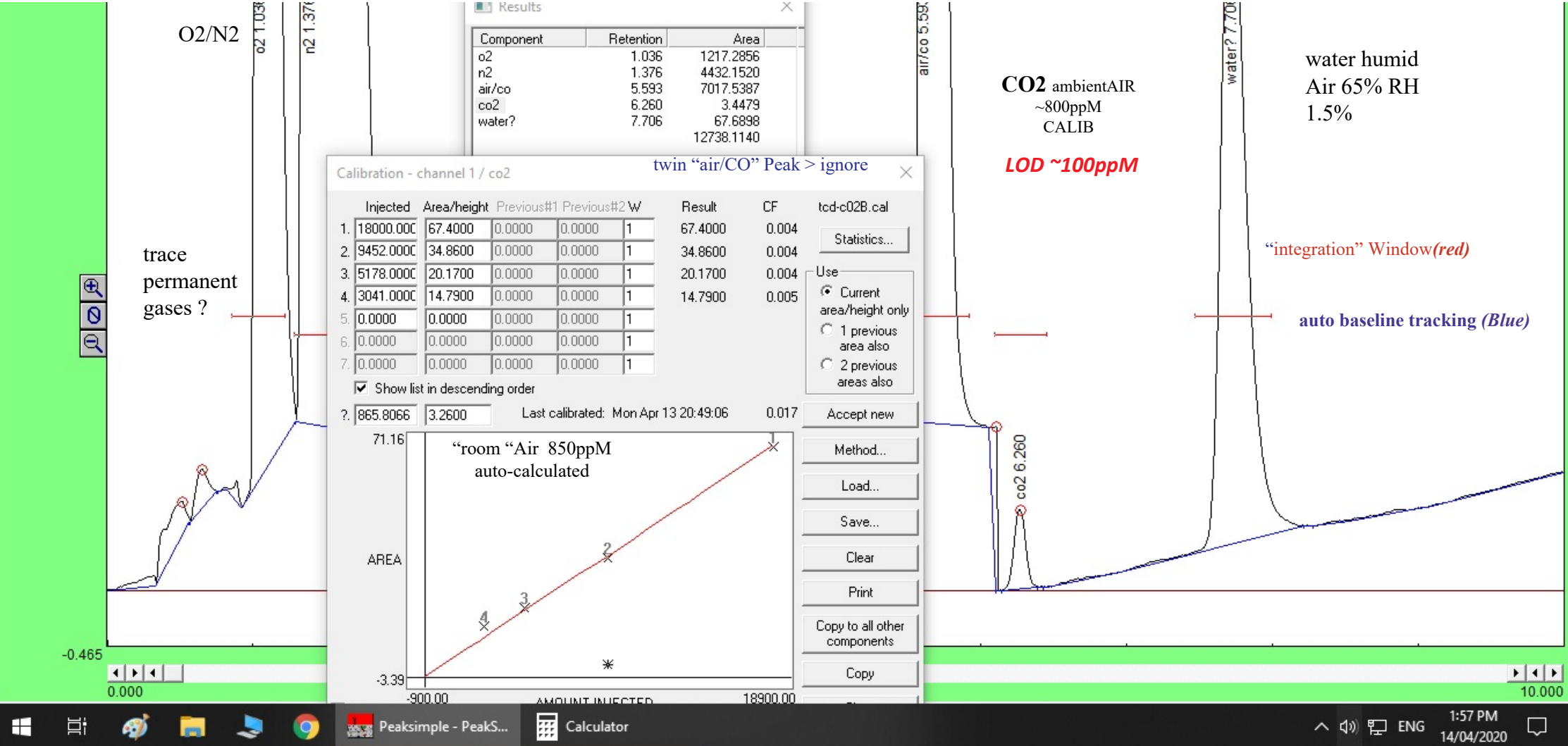
(red) Circle for Integrated Peaks "Window:"

auto area reject set at "500"

SRI 8610C MG#5 GC HS-D + MS5A  
+ backflush column; Dual GSV's  
Dual Channel PeakSimple DS  
TCD + FID-Methaniser

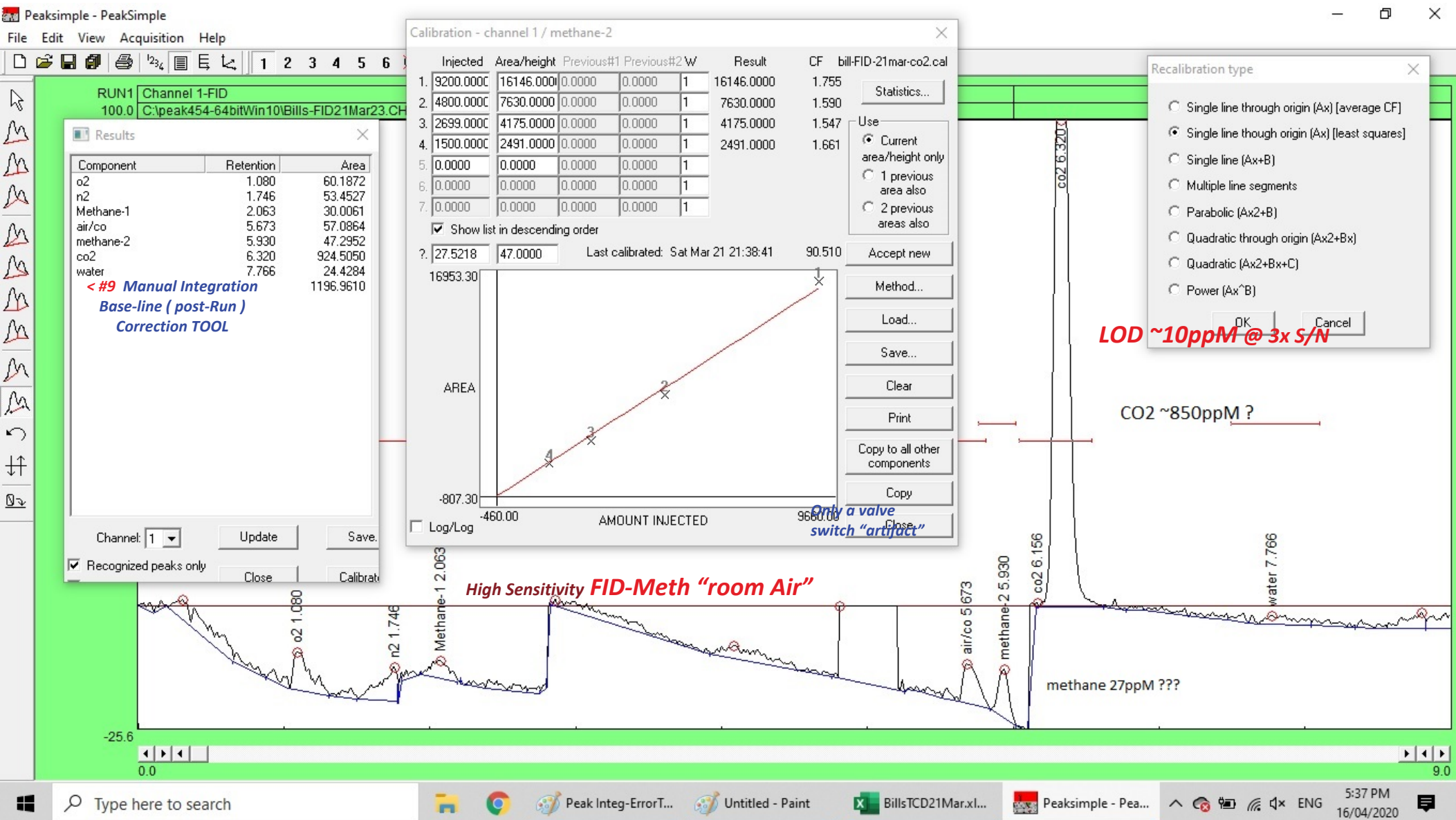
Methane from  
HS-D Column  
~ 90ppM

CO2 ( as CH4 from  
FID-Methaniser  
~100ppM  
\_100ppM)



RAW DATA SRI GC MG#5 accessed from WEB [www.chromtech.net.au/pdf2/SRI-DATA](http://www.chromtech.net.au/pdf2/SRI-DATA)





RAW DATA SRI GC MG#5 accessed from WEB [www.chromtech.net.au/pdf2/SRI-DATA](http://www.chromtech.net.au/pdf2/SRI-DATA)

**Red Files Are VIP for All GC APPS save these Ap specific into the “specific” APP Folder ALL**

**Win10 Use PS v4.54 v4.88 has “bugs” (2019)**

## **PEAKSIMPLE SOFTWARE** **File types used by PeakSimple**

- .CHR** = chromatogram or data file. The chromatogram is the graphic depiction of raw analytical data in binary format.
- .ASC** = ASCII file. You can save your raw data in binary format (.CHR), ASCII format (.ASC), or both.
- .THU** = thumbnail file. PeakSimple keeps thumbnail snapshots of your .CHR files to facilitate browsing. In PeakSimple version 3.21 and newer, you can elect to not save thumbnails to save disk space.
- .3D** = three dimensional file. Load multiple chromatograms (.CHR files) for viewing in the 3D display window. Save the 3D display in a .3D file for later viewing.
- .TEM** = temperature file. TEM files contain the column oven temperature program information: initial and final temperatures, hold times and ramping rates. .TEM, .GRA, and .FLO files perform the same function for different applications; the only real difference between them is their extension. The user selects which application and file extension to use in the Channel details window: on the right side under “Control by,” are three radio buttons with the choices Temperature, Pressure, or Gradient.
- .GRA** = gradient file for HPLC. Gradient files contain the solvent and sample mixing percentages for one or two pumps. PeakSimple uses this extension when the “Gradient” radio button under “Control by” in the Channel details window is selected.
- .FLO** = pressure flow file. PeakSimple uses this extension when you select the “Pressure” button under “Control by” in the Channel details window. This is useful when varying the carrier gas pressure, making use of the Electronic Pressure Controllers (EPCs) in your SRI GC. The user must move a wire to switch from temperature to pressure control.
- event files. Events are controlled by turning relays ON and OFF. Event tables allow you to automatically turn relays ON/OFF at specified times during an analysis. Integration events can also be automatically performed using an event table.
- .EVT** = components file. Each channel has its own Components table that displays the list of expected components, their retention times, and calibration files. All component information is input by the user.
- .CPT** = component calibration file. The calibration curve is calculated from user-generated results obtained at several different concentrations that span the expected range to be encountered in actual samples. Calibration is required for each component you expect to be present in your sample, and for each detector to be used in the analysis. When a component calibration file is saved, it will appear in the Components window, next to the appropriate component.
- .CAL** =
- .LOG** = results log file. Use the Post-run actions window to have PeakSimple add the results to the results log after the analysis.
- .RES** = results file. The results file displays analytical results in ASCII format. The results window is accessed through the View menu. The results log (.LOG) may be viewed by clicking a button in the results window.
- .CON** = Control files contain user-defined analytical parameters such as column oven temperature program (.TEM files), components (.CPT files), and relay event tables (.EVT files), as well as print information and many other parameters.
- .QUE** = autosampler queue files. The autosampler queue lets you load multiple control files for use with an autosampler. In batch reprocessing mode, the autosampler queue is used to re-run data under the parameters of a particular control file.

**IMPORTANT** > SAVE [.CON] Regularly but [SAVE ALL] . . . > just to be sure !



# Q & A's SRI MG#5

## **H2Gen-100** ( Purity > 99.995% ) U generally Don't need High Purity Traps for low ppM GS analysis

- Don't use if Silica Gel has turned blue>brown) saturated ( MS5A may also have become saturated

Symptom wet carrier gas O2/N2 ( on MS-5A Column, Ch-2 ) peak retentions tend to drift lower over time and > loss of peak resolution.

- refill with demin of distilled water when 10% empty( H2Gen itself is <ppM H2O and desiccant only cleans up residual water.
- empty both desiccant tubes in the H2-Gen and reactivate at 250C+ overnight ( in a separate lab oven )
- the few 100g desiccant should last 100s of CubFt ( months at 40cc/min )
- SRI H2-100 has SS Isolation valve if GC Disconnected turn off to minimise ingress of ambient moisture > desiccant traps.

### **START UP**

- Turn on H2 Gen> takes 10mins for pressure to generate/stabilise from ambient
  - Then turn ON the GC Main Power ensure the Column Inlet Pressure is set (2-3psi)
- AND the TCD Filament PROTECT Circuit LED IS GREEN NOT RED ! BEFORE Turning TCD Filament ON**

- Reverse sequence to turn GC "System" OFF

"later" peaks get backflushed anyway from the HSD

**Purge GC for 10minutes before turning on GC HOT zones AND BEFORE TURNING ON THE TCD filaments**

Water from the **H2Gen** Not generally a problem the PEM membrane normally diffuses thru' virtually NIL Water Only H2 .  
at low ppM levels the (>>100g) of desiccant is just to clean up traces for High Sensitivity work  
> it might be relevant at extreme sensitivity

**Desiccant capacity should last at least >100(0)s of hours at normal 40cc/min**

1 Tank of distilled Water ( ~ 7days operation time ) Measure the megOHM ( 1-2mohm is OK !

## **MG#5 GC**

### **Calib - Ambient Air**

Linearity checked down to ~500ppM TCD, 50ppM CH4 FID Methaniser estimated ~+/-5%  
single point daily calibration (in duplicate) eg 50:50 CAC Std (1.8%CO2, 2.5%CH4  
actually measure water and correct end ppM result Note 2.5% OffScale on FID(HI)  
Offset Ambient CO2 in retrospect if diluting Gas Std with ambient Air (65% RH - ~1.6%water),  
( ambient CH4=Only 1.8ppM, but 10-20ppM not unusual indoor - offset IF need be ! )

### **Column Conditioning**

U may have to cycle Oven TEMP up to 200C occasionally to MINIMISE baseline drift.

Backflushing ( pre-set in Events table ( Valve=D? should cope for simple ambient level of other peaks.

- DO NOT HEAT MG#5 Column Set above 200C ( or if baseline drifts too high ?)

The MS-5A may need reactivating occasionally ( due to build up of CO2 AND Water over extended time  
(100s of samples ?) at 300C overnight.

- REMOVE the HaySEP Columns first from GC Oven **TURN OFF TCD Detector filaments / HEAT FIRST !**

CAP the detector ports if heated and NO carrier gas flowing.

### **CAUTION**

On Disassembling columns / detector ports etc

**CHECK CAREFULLY FOR ANY H2 leaks INSIDE the GC Oven**

H2 at 40ml/min potentially can build up an explosive environment side the GC oven ( the High Speed SRI Oven does help ! )

- methanol /water via a Syringe needle maybe ? > ideally use a proper electronic Leak Detectors ( SRI or Restek ) > seepage can occur thru a minute leak even against +ve flow/pressure.

### **Abort > Quick column(s) bake-Out**

Set Up a Simple PS [Events Table] !

[ Temperature Program ] the oven with carrier gas flowing : eg 50degC > 200C @ 5C per min

An "Abort" run does NOT reset ALL Valve setting peaks remain in columns > GC becomes confused DO A MANUAL "RESET" /

"Reinitialise" PS or even reload the correct [Ctrl File] > As necessary !

**Peak Simple ? > like ANY S'ware > & NOT "FOOLPROOF" many options !**

**BE PREPARED TO THINK and to Experiment ! ... & It IS far Simpler than that "dog" Windows 10 !**





## for GC Beginners > Novices

NO LIABILITY Whatsoever is accepted by Chromalytic / SRI GCs for the use OR misuse of this equipment !  
through incompetence, ignorance, stupidity lack of common sense > ASK ! WE WILL ADVISE > SUGGEST WITH ALL DUE DILIGENCE  
Use advisedly and Only after ADEQUATE Training or Supervision - suitably licensed > accredited by YOUR Organisation

### MG#5 GC System for . . .

#### Perm Gases; O<sub>2</sub>, N<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>, C<sub>2</sub>-C<sub>6</sub> HCs

H<sub>2</sub> is installed as Carrier Gas by Chromalytic for “mobile” / portability APPS lab situations

> a Gas Bottle-less installation ( the FID uses air from a unique built-in “mini” Air Compressor.

- \* potentially SF<sub>6</sub> traces (low ppB) ( with SRI ECD ( Electron Capture ) requires ARPANSA Radioactive license a \* requires installation by SRI-USA prior to GC Ordering
- \* H<sub>2</sub> Analysis as a separate Channel with Argon Carrier Gas is possible TCD > low ppm analysis

**Carrier Gas H<sub>2</sub>** > no response for H<sub>2</sub> ( of course ! ) : Helium CG > very poor response for H<sub>2</sub> and impossible to quantitate “sensibly”

USE WITH CARE re H<sub>2</sub> leaks in Column Oven > potentially a remote explosive environment

The MG#5 is a complex Instrument FAR BEYOND NORMAL SIMPLISTIC GC Systems

- MolSieve-5A for O<sub>2</sub>, N<sub>2</sub>, CO, Methane
  - HayeSep-D for Air, Methane, CO<sub>2</sub>, Water and higher MW C<sub>2</sub>-C<sub>6</sub>
  - a 2nd HS-D columns enabling Backflush of components not required ( via Valving and automated via SRI PeakSimple [Events Table]
- a STOP-FLOW Isolation Valve ( for specialise Apps NOT covered in this Note

dual Detectors

TCD for most gases - generally LOD 200 ppm to mid %

FID - HCs / VOCS - generally from 1 ppm to low % range

FID-Methaniser (300degC) ( accessory) - allows CO, CO<sub>2</sub> detection on the FID down to low ppm (> %)

with standard 1cc Sample Loops automated - via a Vacuum Pump Interface accessory ( & recommended ! )

2 heated automated Gas Sample Valves ( in separate oven ) - Injection controlled by PeakSimple - simultaneous injection into separate columns

2 Liquid Injection ports are included for “extended Apps” requiring “spot” VOC samples > limited by the columns installed ( eg “some” C<sub>2</sub>-C<sub>6</sub> VOCs

## NOT for GC Novices OR inexperienced Operators

*The default App is critically setup by SRI/Chromalytic !*

### Timing IS CRITICAL

> via [Events Table] > see Bill01.ev

sample injection [PostRun] to automate; AutoVac Interface & time duration determines flush volume

Valve Switching ( for back-flushing of any unwanted components )

Flow Rates are Critical DO NOT TAMPER UN-Necessarily with these  
Column Lengths, flow rates are BALANCE to do this “particular job

Take Care in changing ANY columns ( eg for baking the MolSieve )  
& ONLY WHEN NECESSARY !

( remove the HS-D Column where column oven is > 200degC  
& CHECK FOR LEAKS !

YOU MUST APPRECIATE and UNDERSTAND the GC FLOW SCHEMATICS !

YOU MUST REINSTALL AND ADJUST THINGS ACCORDINGLY TO  
EXACTLY AS IT WAS ORIGINALLY !

In fact I suggest U Physically write down EXACTLY how the “Whole” system is SetUp  
SO > you have a chance of re-establishing the same IDENTICAL “column Setup”  
DON’T Treat this MG#5 GC as a “Univeral” Do-ALL general purpose instrument  
> dedicate it for the Gas Analysis project it is designed for !

**Analogy** Just because you may have a license to drive a car  
IT DOES NOT MEAN . . . that you are a racing car driver !  
You might be able to put fuel in the tank ( providing you know it’s  
petrol, gas, diesel, hybrid >> electric or H<sub>2</sub> perhaps

But these days I’d advise you DON’T even contemplate opening the bonnet  
“( unless U have a PhD AND Know what you are actually doing ! )  
Let alone doing any internal repairs ( apart from emptying the ash trays  
or changing a light bulb “perhaps” ! )  
Of Course ! you’d NEVER attempt ANY engine repairs in ANY context  
> take it to a car “mechanic” > service engineer  
( even then you are at risk being inadvertently “diverted” to an “  
apprentice” and getting things stuffed up anyway !

GC / chromatographers ( tech “savvy” ? )

require a combination of skills

> chromatography, separation chemistry awareness, electronics, gas fluidics, compressed gases, some physics, computer s’ware literacy . . .

> certainly a degree of hands on “how to use tools” safely and with commonsense

All NOT necessary taught in schools > Universities ? . . . but IF U DO Have ANY COMMON SENSE U May Learn . . . but the Hard Way !

IF IN “ANY” DOUBT

DON’T DO IT ! OR . . . ASK Someone who might ! . . . BUT IT IS NOT A PERFECT WORLD !

YOU CAN Easily stuff up that “car” in that case !  
or Irreversibly destroy the functionality of the GC itself

To REPEAT > Far more so for the “complex” SRI MG#5 GC than for a stock standard simplistic GC



# GC

## TROUBLESHOOTING GUIDE



 **phenomenex**<sup>®</sup>  
...breaking with tradition<sup>SM</sup>



[www.phenomenex.com/GC](http://www.phenomenex.com/GC)



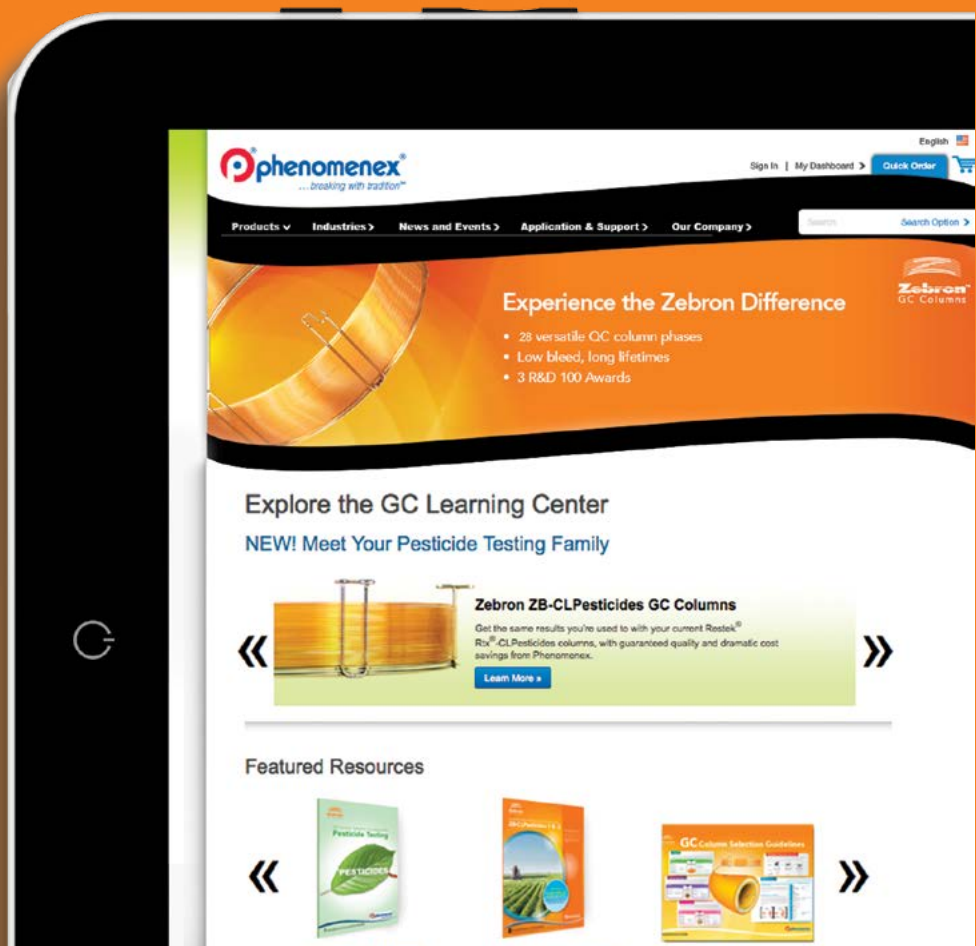


## Explore Online Tips & Tools

**You asked for a better online experience. One filled with technical resources, easy-to-find products, and useful tools to make your GC work easier. Good news... it's here!**

- Troubleshooting resources and on-demand webinars
- Get thousands of applications – save and access them anywhere!
- Watch technical demonstrations and “how-to” videos
- Create a virtual stockroom for your lab
- Try tools that make selecting the right product easier than ever
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# GC Troubleshooting Guide

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# BEFORE YOU START

## Goals & Approaches

An important first step in troubleshooting is understanding the problem. This is best approached systematically; once you have a good understanding of what is causing the problem, it will be easier to implement a logical solution. Understanding the problem can also allow you to alter analysis or maintenance habits to avoid the problem in the future. Prevention is usually the most cost effective solution!

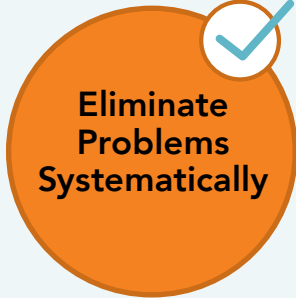
### Common Troubleshooting Approaches



Changing everything relating to the problem may allow a remedy, but won't allow you to fully understand the solution. In the long run, this may be more costly than other approaches.



Narrowing down or eliminating parts of the process may give a good starting point to determine the cause of the problem, but does not necessarily allow you to solve or prevent the problem in the future.



A systematic approach includes checking the chromatographic system from one end to the other, isolating the problem, learning what went wrong, solving the problem, and then preventing it. This is the recommended approach!

### Problem Prevention

Many GC problems can be prevented if the column and system are maintained routinely. *Problem Prevention* (see p. 32) outlines maintenance practices that will reduce the frequency of common issues. These suggestions should be modified to fit your GC column and instrument, and then made a regular part of your laboratory routine.

## Troubleshooting Tools

### What To Have On Hand

Have your instrument manual and these diagnostic tools at hand:

- Flow meter with a range of 10 to 500 mL/minute
- New syringes
- Non-retained, detectable compound such as methane or propane
- Septa, ferrules, inlet liners, and other consumables
- Electronic leak detector
- Reference sample
- Reference column with known performance

# BEFORE YOU START

## Tips For Effective Troubleshooting

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- When troubleshooting, remember to look at recent changes in the system, especially if the system was working previously. Was there something that changed and may be causing the problems now? Can you undo the changes and go back to the original performance?
- Try to isolate the problem to one specific cause to minimize the changes that have to be made to the system that may result in other malfunctions. This will also make it easier to prevent the problem in the future and shorten troubleshooting times if a similar problem does occur in the future.
- Remember to check every part of the process. Don't overlook the obvious. If you are not getting peaks for example, the makeup flow to the detector may be off or the syringe may be clogged. Has the sample preparation method been altered? Verify your samples on another instrument if possible.
- Keep good records of the troubleshooting process – closely observe and note operating parameters (temperatures, flow rates, columns used, etc.). Reliable system maintenance records (inlet liner changes, detector cleanings, etc.) are also important.

## For Additional Help

- The operator's and service manuals for the instrument should be consulted. These contain exploded diagrams, troubleshooting procedures for specific models, and part numbers to help you order replacement parts.
- Other people in the lab may have had experience solving a problem that is giving you trouble; they can be a helpful resource.
- The manufacturer of your instrument can help you. Most GC manufacturers offer free technical support to their customers. Phenomenex has experienced technical consultants who can assist you with almost any problem. We welcome your phone calls or emails!





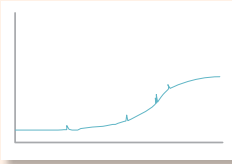
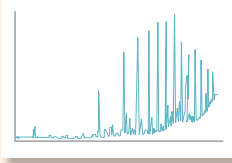
# BASELINE PROBLEMS

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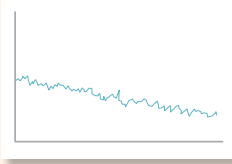
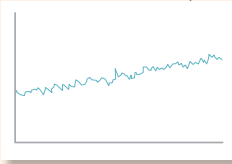
# BASELINE PROBLEMS

## Bleed

Symptom	Possible Cause	Suggested Remedy
<p>Column Bleed</p> 	<p>Improper column conditioning.</p> <p>Contaminated column.</p> <p>Contaminated injector.</p> <p>Leak in system causing column oxidation.</p>	<p>Properly condition the column. <a href="#">See column installation.</a></p> <p>There are several options:</p> <ul style="list-style-type: none"><li>– Trim the column</li><li>– Bake out the column</li><li>– Solvent rinse the column</li><li>– Replace the column</li></ul> <p>Perform inlet maintenance – clean the injector, replace the inlet liner, replace glass wool. <a href="#">See inlet maintenance.</a></p> <p>Check for leaks in the system. Tighten or replace connections; replace seals and filters. If column is severely damaged, replace. <a href="#">See column installation.</a></p>
<p>Septum Bleed</p> 	<p>Septum is not conditioned.</p> <p>Septum core is present in the flow path.</p>	<p>Condition septum prior to analysis or use pre-conditioned septum. Check septa temperature rating – should be sufficient to run at method temperatures.</p> <p>Remove septum core from the inlet. Check septum nut and make sure it is not over tightened. Inspect injector syringe for bent or blunt tip and replace as necessary.</p>

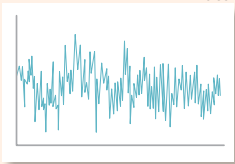
## Drift

Slow movement of the baseline in one direction (either up or down).

Symptom	Possible Cause	Suggested Remedy
<p>Downward</p> 	<p>Downward drift for a few minutes is normal after installing a new column.</p> <p>Unequilibrated detector or oven.</p> <p>Downward drift is frequently due to the "bakeout" of contaminants from the detector or other parts of the GC.</p>	<p>Increase the oven temperature to the maximum continuous operating temperature for the column. Maintain that temperature until a flat baseline is observed. If the detector signal continues to raise or does not drop in 10 minutes, immediately cool the column and check for leaks. <a href="#">See column installation.</a></p> <p>Allow sufficient time for (temperature) equilibration of the detector or oven.</p> <p>Clean out contamination. <a href="#">See detector maintenance.</a></p>
<p>Upward</p> 	<p>Excessive damage to the stationary phase of the GC column.</p> <p>Drift in gas flow rates.</p>	<p>Determine the cause of the damage. It may be due to impurities in the carrier gas or to excessive temperatures. Replace column. <a href="#">See column installation.</a></p> <p>Clean or replace flow or pressure regulator(s). Adjust pressure. <a href="#">See column installation.</a></p>

# BASELINE PROBLEMS

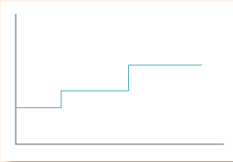
**Noise** Rapid, random movement of the signal amplitude.

Symptom	Possible Cause	Suggested Remedy
	<p>The column may be inserted too far into the flame of an FID, NPD or FPD detector.</p> <p>An air leak can result in noise in ECD and TCD detectors.</p> <p>Incorrect combustion gases or flow rates can generate noise in FID, NPD, or FPD detectors.</p> <p>Contaminated injector.</p> <p>Contaminated column.</p> <p>Drift in gas flow rates.</p> <p>Defective detector board.</p>	<p>Reinstall the column. Be sure to insert the column into the detector exactly the correct distance specified in the instrument manual. <a href="#">See column installation.</a></p> <p>Eliminate the leak.</p> <p>Be sure your gases are the proper grade, as well as clean and dry. Reset the flow rates of the gases to their proper values.</p> <p>Clean injector. Replace inlet liner. Replace glass wool. <a href="#">See injector maintenance.</a></p> <p>Bake out the column. Cut off first 4 inches of column. Solvent rinse or replace column. <a href="#">See column installation.</a></p> <p>Clean and/or replace parts as necessary. <a href="#">See detector maintenance.</a></p> <p>Consult GC instrument manufacturer.</p>

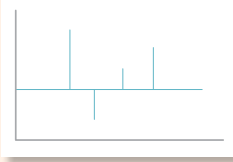


# BASELINE PROBLEMS

**Offset** Sudden unexplained changes in baseline position.

Symptom	Possible Cause	Suggested Remedy
	<p>Line voltage changes.</p> <p>Poor electrical connections.</p> <p>Contaminated injector.</p> <p>Contaminated column.</p> <p>Column inserted too far into the flame of FID, NPD, or FPD detectors.</p> <p>Contaminated detector.</p> <p>Gas generator cycle.</p>	<p>Monitor line voltage for correlation with offset. If correlation is found, install voltage regulator.</p> <p>Check electrical connections. Tighten any loose connections. Clean any dirty or corroded connections.</p> <p>Clean injector. Replace inlet liner. Replace glass wool. <a href="#">See injector maintenance.</a></p> <p>Bake out the column. Cut off the first 4 inches of column. Solvent rinse or replace the column. <a href="#">See column installation.</a></p> <p>Reinstall the column. Be sure to insert the column into the detector exactly the correct distance specified in the instrument manual. <a href="#">See column installation.</a></p> <p>Clean the detector. <a href="#">See detector maintenance.</a></p> <p>Baseline fluctuations can occur as the generator turns on and off. Add a tank with the appropriate volume after the generator to buffer any pressure changes.</p>

**Spiking** Peaks with no width, either positive or negative.

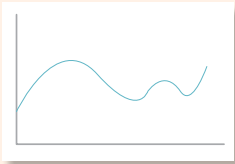
Symptom	Possible Cause	Suggested Remedy
	<p>Electrical disturbances entering the chromatogram through power cables, even shielded cables.</p> <p>Particulate matter passing through the detector.</p> <p>Pressure may build up and gas may escape through a seal and thus reduce the pressure below the point where the escape occurs. If this is the cause, the frequency of spikes will be pressure-dependent.</p> <p>Loose, dirty, or corroded electrical connections in the detector or at connections along the signal path can cause spiking.</p>	<p>Try to correlate spikes with events in equipment near the chromatogram. Periodicity is often a clue. Turn off equipment or move it. If necessary, install a voltage regulator.</p> <p>Clean the detector and eliminate the source of particles. A clean hydrogen flame is invisible. Most organic matter generates a yellow flame. <a href="#">See detector maintenance.</a></p> <p>Fix leaking seal.</p> <p>Check electrical connections. Tighten any loose connections. Clean any dirty or corroded connections. Replace badly corroded FID parts.</p>

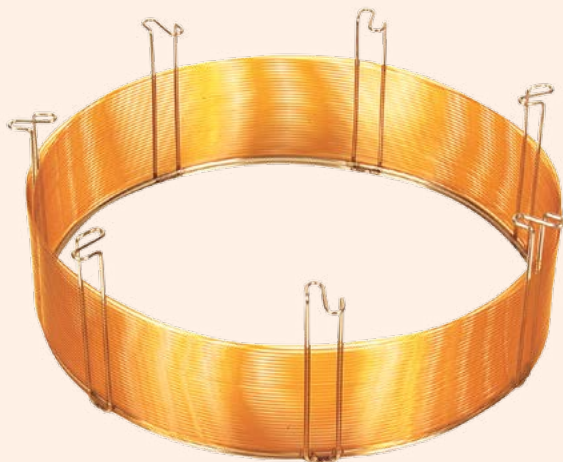




# BASELINE PROBLEMS

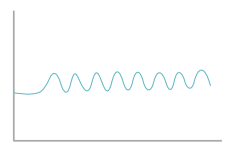
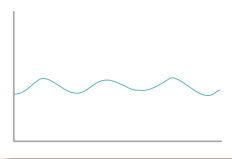
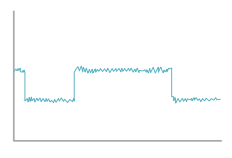
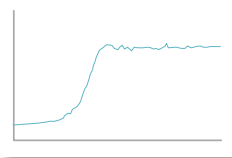
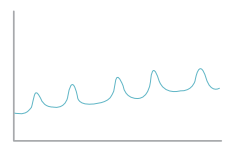
## Wander Low frequency noise.

Symptom	Possible Cause	Suggested Remedy
	<p>Baseline wandering may be caused by changes in environmental conditions such as temperature or line voltage.</p> <p>Inadequate temperature control. Check if variations can be correlated with changes in the baseline position.</p> <p>Wandering while using isothermal conditions may be due to contaminated carrier gas.</p> <p>Contaminated injector.</p> <p>Contaminated column.</p> <p>Poor control of gas flow rates.</p>	<p>Try to correlate the wandering with environmental parameters. If a correlation is observed, you will know what to do. Good luck.</p> <p>Measure detector temperature. Check detector, if TCD is used.</p> <p>Change the carrier gas or the gas purification traps.</p> <p>Clean injector. Replace inlet liner. Replace glass wool. <a href="#">See injector maintenance.</a></p> <p>Bake out the column. Cut off first 4 inches of column. Solvent rinse or replace column. <a href="#">See column installation.</a></p> <p>Clean or change flow controller(s).</p>



# BASELINE PROBLEMS

**Waves** Baseline oscillations different from typical noise.

Symptom	Possible Cause	Suggested Remedy
<div>Fast</div> 	Detector related problem.	Baking the detector at the maximum temperature (450 °C for FID) for 30 min to 1 hour may provide temporary relief. For a lasting solution, physically clean the detector.
<div>Slow</div> 	Gas pressure fluctuations.	Storage tank pressure varies, causing dips in flow. Adding a dual stage regulator can minimize pressure fluctuations and help to alleviate the problem.
<div>Square</div> 	Unbalanced column-switching or gas-sampling valves (for TCD detectors).	Measure, check, and set flows accurately.
<div>S-Shaped</div> 	AC power fluctuations; interference from other equipment lines.	Use a dedicated AC source with sufficient power.
<div>Wander</div> 	Excessive column bleed.	Check max column temperature, re-condition column. If column is damaged beyond repair, replace column.
	Oxygen contamination is degrading the column phase.	Install or check oxygen traps. Check system for oxygen leaks.
	Contamination during isothermal parts of a run.	The column is separating the contaminants (commonly siloxanes or hydrocarbons) as peaks. Changing samples, wash solvents, liner, gold seal, and sometimes the syringe may be required to eliminate the contamination.



# PEAK SHAPE PROBLEMS

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# PEAK SHAPE PROBLEMS

## Reduced Size Some or all peaks are reduced in size.

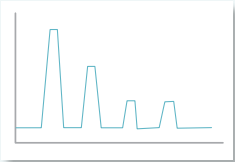
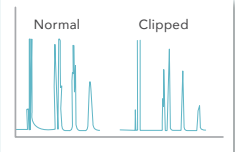
Symptom	Possible Cause	Suggested Remedy
<p>All Peaks Reduced</p> 	<p>Defective or plugged syringe.</p> <p>“Blown” septum or other massive leaks at the inlet or with carrier gas flow. Poor peak shapes usually result from bad leaks.</p> <p>Purge flow or split ratio too high.</p> <p>Injector and/or column temperature too low for high molecular weight or low volatility samples.</p> <p>NPD detector may be coated with silicon dioxide due to column bleed or residual derivatization reagents.</p> <p>NPD damage by loss of rubidium salt as a result of exposure to overheating, heating in the absence of clean gas flow, or humidity.</p> <p>For splitless injection, if the split vent is closed for too short a period of time or if the initial column temperature is too high, this may hinder refocusing of the sample.</p> <p>Detector-sample mismatch.</p> <p>Inadequate signal amplification.</p> <p>Sample invalidity.</p>	<p>Try a new or proven syringe.</p> <p>Find and fix leaks and adjust gas flow. <i>See column installation.</i></p> <p>Adjust gas flow rates.</p> <p>Increase injector and/or column temperature(s).</p> <p>Replace the active element. Avoid exposure to silicon containing compounds.</p> <p>Replace the active element. Turn off detector whenever the gas flow is interrupted. Avoid overheating. Keep element warm (150 °C) when not in use. Use a desiccator for extended storage.</p> <p>Increase the time the split vent is closed. Decrease the initial column temperature or use a less volatile solvent so that the initial temperature is below the boiling point of the solvent.</p> <p>Ensure that the detector will respond to the analytes.</p> <p>Check output signal levels.</p> <p>Check sample concentration and stability.</p>
<p>Some Peaks Reduced</p> 	<p>Activity in the inlet liner or column if the reduced peak is an active compound.</p> <p>Leak in the injector if the reduced peak is a more volatile compound.</p> <p>Initial temperature too high for splitless or on-column injections.</p> <p>Analytes are decomposing or breaking down for active or thermally labile compounds.</p>	<p>Clean or replace the inlet liner. Ensure an inert column is used. If necessary, replace the column. <i>See column installation.</i></p> <p>Find or repair the leaks and adjust gas flow.</p> <p>Lower the initial column temperature.</p> <p>Use a higher boiling solvent.</p> <p>Check the integrity of the sample.</p> <p>For thermal lability, lower the temperature and use on-column injection, a column with thinner stationary phase, a shorter column lengths, or a higher carrier gas flow rate. For active compounds, ensure an inert column is used. If necessary, replace the column. <i>See column installation.</i></p>



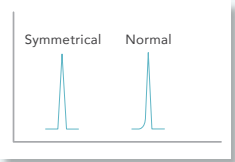
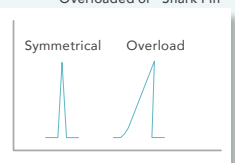


# PEAK SHAPE PROBLEMS

**Clipped/Flat** Peaks are clipped and flat at either the top or the bottom.

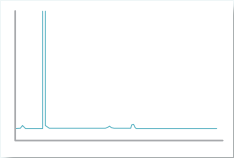
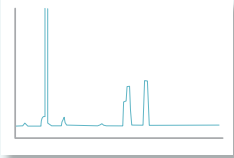
Symptom	Possible Cause	Suggested Remedy
<p>Flat Top Peaks</p> 	<p>Detector overload. The broad peaks may have a rounded top or even valleys in the top.</p> <p>Overload of the signal processing electronics. The peaks are clipped with flat tops.</p>	<p>Reduce sample volume, dilute with solvent, or increase or add a split flow.</p> <p>Attenuate detector output reduce sample volume, or add a split flow</p>
<p>Flat Bottom Peaks</p> 	<p>Detector, recorder, or integrator set too low; detector drifted below zero.</p>	<p>Correctly set zero. Reconnect leads from recorder and set zero of recorder baseline to ~5 % of full scale. Check the integrator threshold and adjust accordingly.</p> <p>Use an auto-zero function.</p>

**Fronting** Moderate to severe asymmetry towards the front or left side of the peak.

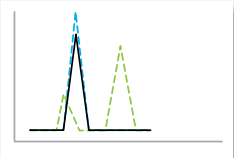
Symptom	Possible Cause	Suggested Remedy
<p>Slight Fronting</p> 	<p>Improper column installation.</p> <p>Sample is condensing in the injector or column.</p>	<p>Reinstall the column. <i>See column installation.</i></p> <p>Check injector and oven temperature with an accurate thermometer. If accurate, increase temperature as necessary, but do not exceed the maximum temperature limit of the column.</p>
<p>Overloaded or "Shark Fin"</p> 	<p>Column is overloaded as a result of injection volume and split ratio.</p> <p>Polarity mismatch.</p>	<p>Reduce the injection volume; add or increase split flow.</p> <p>Use a column with greater capacity. Columns with larger diameter or thicker stationary phase coatings generally have larger sample capacities; however, resolution may be reduced.</p> <p>Polar compounds will have lower concentration capacity on a non-polar phase and vice-versa. Choose a phase with the appropriate polarity and selectivity for your sample.</p>

# PEAK SHAPE PROBLEMS

**Ghost Peaks** Peaks observed when no sample has been introduced into the system.

Symptom	Possible Cause	Suggested Remedy
<p>Normal</p> 	<p>Remnants of previous samples in the inlet or column are most likely to occur when increasing inlet or column temperature(s).</p>	<p>Increase the final temperature and lengthen the run time to allow for the complete elution of previous samples. If ghost peaks continue to occur, clean the inlet.</p> <p><i>See injector maintenance.</i></p>
<p>Ghost Peaks</p> 	<p>Sample expanded to exceed the volume of the injector liner. These vapors may come in contact with colder spots, such as the septum and gas inlets to the injector. Less volatile components may condense. These condensates may vaporize later and interfere with subsequent analyses, sometimes producing "ghost peaks".</p> <p>Bleed from the septum or fragments of the septum lodged in the inlet or liner</p> <p>Syringe contamination.</p>	<p>Condition the column at a higher temperature that is still lower than the maximum isothermal limit for the column. Cut 4 inches off the inlet end of the column and/or reverse it (end-for-end) before reconditioning. Solvent rinse or replace the column.</p> <p><i>See column installation.</i></p> <p>Minimize backflash by using:</p> <ul style="list-style-type: none"><li>• a septum purge</li><li>• small injection volumes</li><li>• large inlet liners</li><li>• optimal injector temperatures</li><li>• pulsed pressure programming</li><li>• increased split flow</li></ul> <p>Clean the inlet. Replace the inlet liner or glass wool, and septum.</p> <p><i>See injector maintenance.</i></p> <p>Replace the syringe.</p>

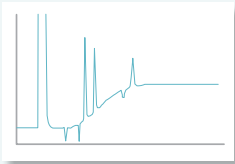
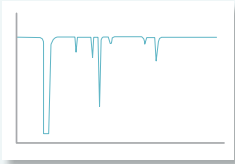
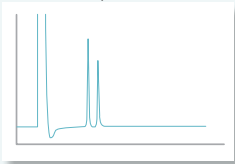
**Irreproducibility** Peak heights, areas, or retention times are inconsistent from injection to injection

Symptom	Possible Cause	Suggested Remedy
<p>Irreproducibility</p> 	<p>Inconsistent injection.</p> <p>Distorted peak shapes can adversely affect quantitative determinations.</p> <p>Baseline disturbances.</p> <p>Variations in GC operating parameters.</p>	<p>Develop a reproducible injection technique. Use autosampler or replace injection needle.</p> <p>Correct any problems that result in the distortion of peak shape.</p> <p>Disturbances in baseline are affecting peaks.</p> <p><i>See baseline problems.</i></p> <p>Standardize parameters.</p>



# PEAK SHAPE PROBLEMS

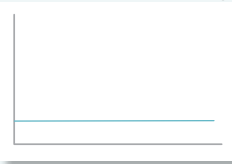
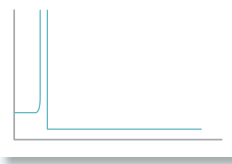
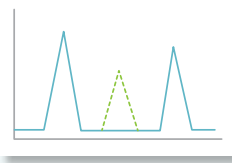
## Negative Peaks Some or all peaks dip below the baseline.

Symptom	Possible Cause	Suggested Remedy
<p>Some Peaks</p> 	<p>Detector overload in element-specific detectors such as ECD, NPD, FPD, etc. can produce both positive and negative peaks.</p> <p>Dirty ECD detector can give a negative peak after a positive one.</p> <p>Sample contaminants (hydrocarbons or other non-responders) are present when using ECD, PID, or NPD (thermoionic specific) detectors.</p>	<p>Have the compounds of interest arrive at the detector at a different time from the solvent or other compounds in high concentration. H<sub>2</sub> produces negative peaks with a TCD and helium carrier gas.</p> <p>Clean or replace the ECD detector. <i>See <a href="#">detector maintenance</a>.</i></p> <p>Improve sample preparation and cleanup methods prior to injection.</p>
<p>All Peaks</p> 	<p>Incorrect polarity of the recorder connections results in nearly all peaks being negative.</p> <p>Recorder-integrator wires reversed.</p> <p>Sample injected onto the wrong column for dual-column setups.</p>	<p>Reverse polarity of recorder connections.</p> <p>Correct connections.</p> <p>Reinject the sample onto the correct column.</p>
<p>Dip After Solvent Peak</p> 	<p>Detector contamination.</p> <p>Sample contamination.</p> <p>Often normal for NPD (thermoionic specific) detectors.</p>	<p>Clean or bake out the detector. <i>See <a href="#">detector maintenance</a>.</i></p> <p>For PID detectors, check that the sample has not been contaminated with methanol or water. If necessary, prepare a fresh sample.</p> <p>No correction necessary.</p>

# PEAK SHAPE PROBLEMS

## No Peaks

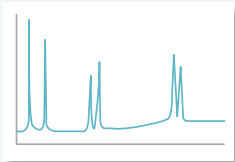
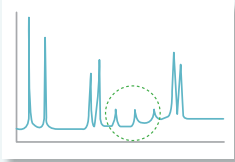
Some or all peaks are missing from the run.

Symptom	Possible Cause	Suggested Remedy
<p>All Peaks Missing</p> 	<p>Defective or clogged syringe.</p> <p>“Blown” septum or massive leaks at the inlet.</p> <p>Problems with carrier gas flow.</p> <p>Column may be broken or installed in the wrong detector or inlet.</p> <p>The detector is not functioning or is not connected to the recorder or integrator.</p> <p>Incorrect injector temperature:</p> <ul style="list-style-type: none"><li>• Injector too cold: sample is not vaporized.</li><li>• Injector too hot: thermally labile sample is decomposing.</li></ul>	<p>Try a new or proven syringe.</p> <p>Find and fix leaks.</p> <p>Adjust gas flow.</p> <p>If breakage is close to the beginning or end, cut off the short piece. Breakage in the middle can be repaired with a press-fit connector. For multiple breakages, replace or reinstall the column. <i>See column installation.</i></p> <p>Ensure detector is working properly. (e.g. is the flame in a FID lit?) Check connection to the output device.</p> <p>Cold injector: check injector and oven temperature with an accurate thermometer. If accurate, increase temperature as necessary, but do not exceed the maximum temperature limit of the column. Inject the sample directly onto the column.</p> <p>Hot injector: check injector and oven temperature with an accurate thermometer. If accurate, reduce temperature as necessary, ensuring compatibility with sample and column minimum limit.</p>
<p>No Peaks After Solvent Peak</p> 	<p>Sample volume is too high.</p> <p>For FID detectors, the flame is blown out by the solvent peak.</p> <p>Carrier gas flow is too high.</p> <p>Incorrect column temperature; column is too hot and sample is eluting in solvent peak.</p> <p>Column cannot separate components from solvent.</p>	<p>Inject less sample or use a higher split ratio.</p> <p>Check the detector temperature.</p> <p>Reduce the flow rate.</p> <p>Check oven temperature with an accurate thermometer. If accurate, reduce temperature as necessary, ensuring compatibility with sample and column minimum limit.</p> <p>Change solvent or column.</p>
<p>Some Peaks Missing</p> 	<p>Activity in the inlet liner or column if the missing peak is an active compound.</p>	<p>Clean or replace the inlet liner. Ensure an inert column is used. If necessary, replace the column. <i>See column installation.</i></p>



# PEAK SHAPE PROBLEMS

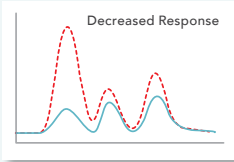
## Peaks Added There are more peaks than normal in the run.

Symptom	Possible Cause	Suggested Remedy
<p>Normal</p> 	<p>Septum bleed (especially for runs with oven ramp).</p>	<p>Turn off the injector heater. If extra peaks disappear, choose a higher temperature rated septum or use a lower injection temperature.</p>
<p>Extra Peaks</p> 	<p>Carryover of sample or contaminants from previous runs.</p> <p>Contaminants in current sample or solvent.</p> <p>Impurities in carrier gas are eluting.</p> <p>Analytes are decomposing or breaking down for active or thermally labile compounds.</p>	<p>Increase the analysis time prior to the next run or bake out the column between runs.</p> <p>Inject solvent by itself using a clean syringe. Switch to a higher quality solvent if extra peaks appear. If only solvent appears, run the solvent through any sample preparation methods, analyzing the solvent at each step of the process to identify the source of extra peaks. If only the solvent peak appears, the extra peaks are part of the sample.</p> <p>Install or check gas purifiers. Replace if necessary. Ensure only high-quality gases are used.</p> <p>If compounds are thermally labile, lower the temperature and use on-column injection, a column with thinner stationary phase, a shorter column lengths, or a higher carrier gas flow rate.</p> <p>If compounds are active, ensure an inert column is used. If necessary, replace the column. <i>See column installation.</i></p>

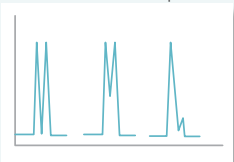


# PEAK SHAPE PROBLEMS

## Sensitivity Loss Some or all peaks are displaying decreased response.

Symptom	Possible Cause	Suggested Remedy
	<p>Contamination of column and/or liner can lead to loss of sensitivity for active compounds.</p> <p>Injector leaks reduce the peak height of the most volatile components of a sample.</p> <p>Initial column temperature is too high for splitless injection prevents refocusing of sample. This affects the more volatile components most.</p> <p>Inlet discrimination: injector temperature is too low. Later eluting and less volatile compounds have low response.</p> <p>Issues with the sample.</p>	<p>Clean liner. <i>See injector maintenance.</i></p> <p>Bake out the column. Solvent rinse or replace the column. <i>See column installation.</i></p> <p>Find and fix any leaks.</p> <p>Lower temperature below the boiling point of the solvent. Decrease the initial column temperature, or use a less volatile solvent.</p> <p>Increase the injection temperature or use on-column injection with direct connect liner.</p> <p>Check the sample concentration, any sample preparation procedures, and shelf life. Prepare a fresh sample ensuring the proper concentration.</p>

## Split Peaks Peaks are duplicated or separated.

Symptom	Possible Cause	Suggested Remedy
	<p>Poor (jerky or erratic) injection for manual injection.</p> <p>Bad column installation.</p> <p>Solvent mismatch: polarity of the stationary phase does not match the polarity of the solvent.</p> <p>Wrong inlet liner is not vaporizing samples in one location.</p> <p>Fluctuations in column temperature.</p> <p>Mixed sample solvent for splitless or on-column injections.</p> <p>Improper use of "solvent effect" refocusing techniques result in broad, distorted peaks because solutes are not refocused into a narrow band near the beginning of the column. The solvent must form a compact, continuous flooded zone in the column. If the solvent does not wet the stationary phase sufficiently (as might be the case for methanol used with a nonpolar phase), the flood zone may be several meters long and not of uniform thickness.</p>	<p>Use smooth, steady plunger depression. Use autosampler.</p> <p>Reinstall column. <i>See column installation.</i></p> <p>Change solvents, use a very large split ratio, install a retention gap, or change the stationary phase.</p> <p>Use a liner with glass wool in the middle of the liner if possible.</p> <p>Repair temperature control system.</p> <p>Use a single solvent.</p> <p>Install a retention gap (5 meters of uncoated but deactivated column) ahead of the column to reduce or eliminate the problem.</p> <p>Change solvent or GC column phase.</p> <p>Use a very high split ratio.</p>

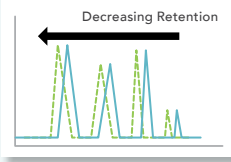
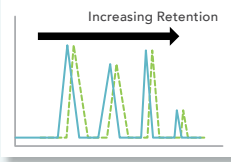
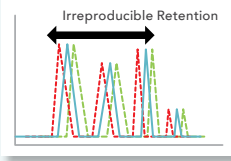
# PEAK SHAPE PROBLEMS

**Tailing** Moderate to severe asymmetry towards the back or right side of the peak.

Symptom	Possible Cause	Suggested Remedy
	<p>Contaminated inlet liner or column.</p> <p>Activity in the inlet liner or column if the missing peak is an active compound.</p> <p>Dead volume due to poorly installed liner or column.</p> <p>Ragged column end.</p> <p>Solvent-phase mismatch.</p> <p>A cold region in the sample flow path.</p> <p>Column or inlet liner temperature is too low for tailing hydrocarbons.</p> <p>Debris in the liner or column.</p> <p>Injection takes too long.</p> <p>Split ratio is too low.</p> <p>Overloading the inlet.</p> <p>Some types of compounds such as alcoholic amines, primary and secondary amines, and carboxylic acids tend to tail.</p>	<p>Clean or replace inlet liner. Bake out or replace the column. <i>See column installation.</i></p> <p>Clean or replace the inlet liner. Ensure an inert column is used. If necessary, replace the column. <i>See column installation.</i></p> <p>Confirm by injecting inert peak methane; if it tails, column is not properly installed. Reinstall liner and column as necessary. <i>See column installation.</i></p> <p>Score the tubing lightly with a ceramic scoring wafer before breaking it. Examine the end (a 20-power magnifying glass is recommended). If the break is not clean and the end square, cut the column again. Point the end down while breaking it, and while installing a nut and ferrule, to prevent fragments from entering the column. Reinstall the column. <i>See column installation.</i></p> <p>Change the stationary phase. Usually polar analytes tail on non-polar columns, or dirty columns.</p> <p>Remove any cold zones in the flow path or check the MS transfer line trap.</p> <p>Check injector and oven temperature with an accurate thermometer. If accurate, increase temperature as necessary, but do not exceed the maximum temperature limit of the column.</p> <p>Clean or replace the liner. Cut 4 inches off the end of the column and reinstall it. <i>See column installation.</i></p> <p>Improve injection technique.</p> <p>Increase split ratio to at least 20:1.</p> <p>Decrease the sample volume or dilute the sample.</p> <p>Try a more polar column. Derivatize the sample.</p>

# PEAK SHAPE PROBLEMS

## Retention Time Shifts Peak retention times drift or move.

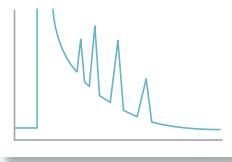
Symptom	Possible Cause	Suggested Remedy
	<p>Increase in column temperature.</p> <p>Increase in gas flow rate (linear velocity).</p> <p>Change of solvent.</p> <p>Significant loss of stationary phase due to column bleed.</p>	<p>Check GC oven temperature and adjust as necessary. Ensure run conditions do not exceed the minimum temperature limits of the column.</p> <p>Inject a detectable unretained sample such as methane to determine the linear gas velocity. Adjust gas pressure to obtain proper values for your analytical method.</p> <p>Use the same solvent for standards and samples.</p> <p>Reduce oven temperature. Ensure run conditions do not exceed the maximum temperature limit of the column.</p> <p>Replace the column if necessary. <i>See column installation.</i></p>
	<p>Leak in the injector.</p> <p>Decrease in column temperature.</p> <p>Decrease in gas flow rate (linear velocity).</p>	<p>Find the leak and fix it. Check the septum first. Change if necessary.</p> <p>Check GC oven temperature and adjust as necessary. Ensure run conditions do not exceed the maximum temperature limits of the column.</p> <p>Inject a detectable unretained sample such as methane to determine the linear gas velocity. Adjust gas pressure to obtain proper values for your analytical method.</p>
	<p>Poor (jerky or erratic) injection for manual injection.</p> <p>Contaminated column.</p> <p>Leak in the injector.</p> <p>Near-empty carrier gas tank.</p>	<p>Use smooth, steady plunger depression. Use autosampler.</p> <p>Bake out the column. Cut 4 inches off the end of the column. Solvent rinse or replace the column. <i>See column installation.</i></p> <p>Find the leak and fix it. Check the septum first. Change if necessary.</p> <p>Check and replace the tank if necessary.</p>



# PEAK SHAPE PROBLEMS

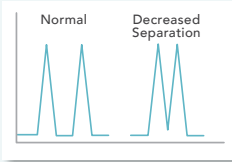

## Solvent Peak Broad

The solvent peak is wide and coeluting with analyte peaks.

Symptom	Possible Cause	Suggested Remedy
	Bad column installation.	Reinstall column. <a href="#">See column installation.</a>
	Injector leak.	Find and fix leak.
	Injection volume too large.	Decrease sample injection volume or dilute to 1:10.
	Injection temperature too low.	Increase injection temperature so the entire sample is vaporized "instantly." An injection temperature higher than the temperature limit of the column will not damage the column.
	Split ratio is too low.	Increase split ratio.
	Column temperature too low.	Increase column temperature. Use a lower boiling solvent.
	Initial column temperature too high for splitless injection.	Decrease the initial column temperature.
	Purge time (splitless hold time) too long for splitless injection.	Use a shorter purge valve closed time.

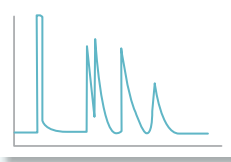


## Resolution Loss

Peaks begin to coelute or overlap.

Symptom	Possible Cause	Suggested Remedy
	Change in column dimensions or stationary phase; excessive column trimming.	Differences in retention time or peak shape of other compounds will be apparent. Check the column phase and dimensions switch the column if necessary.
	Damage to column stationary phase.	This is usually indicated by excessive column bleed. Replace the column. <a href="#">See column installation.</a>
	Damage to column stationary phase.	This is usually indicated by excessive column bleed. Replace the column. <a href="#">See column installation.</a>
	Injector problems.	Check for: <ul style="list-style-type: none"><li>• leaks</li><li>• inappropriate temperature</li><li>• split ratio</li><li>• purge time</li><li>• dirty liner</li><li>• glass wool in liner</li></ul>

# PEAK SHAPE PROBLEMS

## Performance Loss (Column) The column deteriorates too quickly after installation.

Symptom	Possible Cause	Suggested Remedy
<p>Poor Resolution or Performance</p> 	<p>Column too hot for too long.</p> <p>Exposure to oxygen, particularly at elevated temperatures.</p> <p>Chemical damage due to inorganic acids or bases.</p> <p>Contamination of the column with nonvolatile materials.</p>	<p>Stay below limits specified for the column. Replace column. <i>See column installation.</i></p> <p>Find and fix any leaks. Be sure carrier gas is sufficiently pure.</p> <p>Keep inorganic acids or bases out of column. Neutralize samples.</p> <p>Find and fix any leaks. Be sure carrier gas is sufficiently pure.</p> <p>Keep inorganic acids or bases out of column. Neutralize samples.</p> <p>Prevent nonvolatile materials from getting into column. For example, use a guard column or a Zebtron™ column with Guardian™ integrated guard.</p>
<p>Pitting</p>  <p>Shattered</p> 	<p>Broken column.</p>	<p>Replace column. <i>See column installation.</i></p> <p>Avoid damaging the polyimide coating on the column. Except when using Zebtron Inferno™ GC columns, avoid temperatures above 370 °C; abrasion of columns (for example, do not install a column so that it touches the side of the oven, because vibration may then damage the polyimide coating); or excessive bending or twisting, which will damage this protective coating. Remember, even if the column does not break immediately, when the protective coating is damaged the column may break spontaneously later.</p>

### A Note On Solvent Rinsing

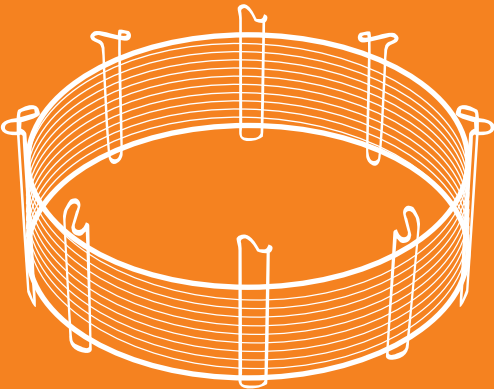
Solvent rinsing (where a pressurized vial of solvent is forced through the column with 10 to 15 psi pressure) may remove most soluble contaminants and restore column performance as a last resort. In most cases, it is better to replace the column.

Use a series of solvents, starting with the most polar and finishing with the least polar. Include the injection solvent if practical. Successive solvents must be miscible with their predecessors. Begin with water followed by methanol for water-based samples (or aqueous extracts). Avoid halogenated solvents as a final rinse if you are using an ECD. Avoid acetonitrile as a final rinse if you are using an NPD. Methanol, followed by methylene chloride and then hexane, is a useful combination.

Each solvent should remain in the column for at least 10 minutes. There is no need to remove the previous solvent before introducing the next. After the last solvent has been removed, the column should be purged with pure carrier gas for 10 minutes before reinstallation. Program the oven temperature at 2 °C/min until the normal conditioning temperature is reached, then condition the column as usual.

# COLUMN SELECTION PROBLEMS

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# COLUMN SELECTION PROBLEMS

## The Impact of Selectivity

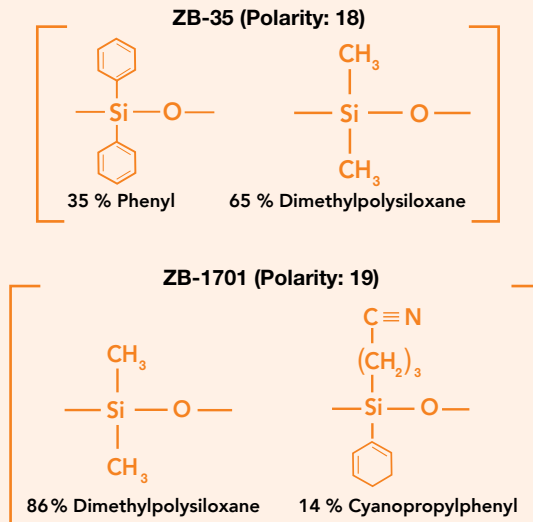
Resolution between two analytes is mainly determined by the selectivity of the stationary phase. By increasing the resolution between two compounds, the total analysis time can often be reduced significantly!

### The Master Resolution Equation

$$R_s = \underbrace{\left[ \frac{\sqrt{N}}{4} \right]}_{\text{Efficiency Term}} \times \underbrace{\left[ \frac{\alpha - 1}{\alpha} \right]}_{\text{Selectivity Term}} \times \underbrace{\left[ \frac{k}{k + 1} \right]}_{\text{Retention Term}}$$

## Selectivity vs. Polarity

Column polarity and selectivity are often confused – polarity gives a general guideline for sample capacity and separation, which can affect peak shape and resolution. However, two columns may have similar polarity but show very different separation profiles because of differences in phase chemistry. Example: The ZB-1701 cyanopropyl group makes it very different from ZB-35 in terms of selectivity though polarity is similar.



## The 3 Most Prevalent GC Interactions

### Dispersive Forces (Van der Waals Interactions)

- Weakest of all intermolecular forces and occurs between non-polar compounds
- Separation is based on boiling point (classic example – hydrocarbon separation in simulated distillation analysis)

### Dipole-Dipole Interactions

- Either permanently present or induced by analyte-stationary phase interactions
- Higher dipole-dipole interaction can help separate compounds with similar boiling points, but different chemical structures

### Hydrogen Bonding (Acid-Base Interactions)

- Can cause poor peak shape or irreversible binding to the inlet liner or to the column itself



# COLUMN SELECTION PROBLEMS

## Dimension Selection

### Short

(15 m or less)

#### Applications

- High boilers
- GC/MS applications

#### Advantages

- Faster run times
- Higher temp. limits
- Lower bleed
- Higher efficiency

#### Disadvantages

- Less inert
- Limited retention

LENGTH

30m



### Narrow

(0.10, 0.18, or 0.20 mm)

#### Applications

- Complex samples

#### Advantages

- Faster run times
- Better resolution

#### Disadvantages

- Lower sample capacity
- Easily overloaded

INTERNAL DIAMETER

0.25mm



### Thin

(0.10 or 0.18  $\mu$ m)

#### Applications

- High boilers
- GC/MS applications

#### Advantages

- Faster run times
- Higher temp. limits
- Lower bleed
- Higher efficiency

#### Disadvantages

- Less inert
- Limited retention

FILM THICKNESS

0.25 $\mu$ m



# COLUMN SELECTION PROBLEMS

## Dimension Selection

---

### Long (60 m or more)

#### Applications

- Complex samples with closely eluting peaks
- Low boilers
- Less active samples
- Complex temperature ramps

#### Advantages

- Better resolution

#### Disadvantages

- Slow run times

### Wide (0.32 or 0.53 mm)

#### Applications

- Dirty samples
- Highly concentrated samples

#### Advantages

- Increased sample capacity
- Increased sample

#### Disadvantages

- Decreased efficiency
- May need higher flow rates unsuitable for GC/MS

### Thick (0.50 $\mu$ m or more)

#### Applications

- Low boilers
- Gases, solvents, purgeables, volatiles
- Purity testing

#### Advantages

- Better inertness
- Higher capacity

#### Disadvantages

- Slower run times
- Lower temp. limits
- Higher bleed

# COLUMN SELECTION PROBLEMS

## Phase Selection Chart

Polarity	Phase	Composition	Temperature Limits (Isothermal/TPGC)	GC/MS Certified
5	ZB-1	100 % Dimethylpolysiloxane	-60 to 360/370 °C*	✓
5	ZB-1ms	100 % Dimethylpolysiloxane	-60 to 360/370 °C	✓
5	ZB-1HT Inferno™	100 % Dimethylpolysiloxane	-60 to 400/430 °C**	✓
5	ZB-1XT SimDist	100 % Dimethylpolysiloxane	-60 to 450 °C*	✓
8	ZB-5	95 % Dimethylpolysiloxane 5 % Phenyl	-60 to 360/370 °C*	✓
8	ZB-5ms	95 % Dimethylpolysiloxane 5 % Phenyl-Arylene	-60 to 325/350 °C	✓
8	ZB-5MSi	95 % Dimethylpolysiloxane 5 % Phenyl	-60 to 360/370 °C	✓
8	ZB-5HT Inferno	95 % Dimethylpolysiloxane 5 % Phenyl	-60 to 400/430 °C**	✓
8	ZB-SemiVolatiles	95 % Dimethylpolysiloxane 5 % Phenyl-Arylene	-60 to 325/350 °C	✓
9	ZB-XLB	Proprietary	30 to 340/360 °C*	✓
9	ZB-XLB-HT Inferno	Proprietary	30 to 400 °C	✓
11	ZB-MultiResidue™-1	Proprietary	-60 to 320/340 °C	✓
13	ZB-624	94 % Dimethylpolysiloxane 6 % Cyanopropylphenyl	-20 to 260 °C	
15	ZB-MultiResidue-2	Proprietary	-60 to 320/340 °C	✓
18	ZB-35	65 % Dimethylpolysiloxane 35 % Phenyl	40 to 340/360 °C	✓
18	ZB-35-HT Inferno	65 % Dimethylpolysiloxane 35 % Phenyl	40 to 400 °C	✓
19	ZB-1701	86 % Dimethylpolysiloxane 14 % Cyanopropylphenyl	-20 to 280/300 °C*	
19	ZB-1701P	86 % Dimethylpolysiloxane 14 % Cyanopropylphenyl	-20 to 280/300 °C*	
24	ZB-50	50 % Dimethylpolysiloxane 50 % Cyanopropylphenyl	40 to 320/340 °C	✓
52	ZB-WAX <sub>PLUS</sub> ™	100 % Polyethylene Glycol	20 to 250/260 °C*	
57	ZB-WAX	100 % Polyethylene Glycol	40 to 250/260 °C	✓
58	ZB-FFAP	Nitroterephthalic Acid Modified Polyethylene Glycol	40 to 250/260 °C	
Proprietary	ZB-CLPesticides-1 & -2	Proprietary	40 to 320/340 °C	
	ZB-BAC-1 & -2	Proprietary	40 to 320/340 °C	✓
	ZB-Drug-1	Proprietary	-20 to 260/280 °C	✓
	ZB-Bioethanol	Proprietary	-60 to 340/360 °C	✓



## Recommended Applications

Amines, Drugs, Essential Oils, Ethanol, Gases (Refinery), Hydrocarbons, Mercaptans, MTBE, Natural Gas Odorants, Oxygenates and GROs, PCBs, Pesticides, Semi-volatiles, Simulated Distillation, Solvent Impurities, Sulfur Compounds (Light)

Acids, Amines, Diesel Fuel, Drugs, Flavors & Fragrances, PCBs (EPA Method 1668), Pesticides

Diesel Fuel, High Boiling Petroleum Products, High Molecular Weight Waxes, Long-chained Hydrocarbons, Motor Oils, Polymers/Plastics, Simulated Distillation

ASTM Methods (D2887, D2887X, D3710, D6352, D7169), Crude Oil, Gasoline Fractions, Petroleum Distillates, Petroleum Fractions, Simulated Distillation, Vacuum Distillates

Alkaloids, Dioxins, Drugs, Essential Oils/Flavors, FAMES, Halo-hydrocarbons, PCBs/Aroclors, Pesticides/Herbicides, Phenols, Residual Solvents, Semi-volatiles

Acids, Alkaloids, Amines, Dioxins, Drugs, EPA Methods (525, 610, 625, 8100), Essential Oils/Flavors, FAMES, Halo-hydrocarbons, PCBs/Aroclors, Pesticides/Herbicides, Phenols, Residual Solvents, Semi-volatiles, Solvent Impurities

Drugs, EPA Methods, FAMES, Nitrosamines, Pesticides, Phenols

Diesel Fuels, High Boiling Petroleum Products, High Molecular Weight Waxes, Long-chained Hydrocarbons, Motor Oils, Polymers/Plastics, Simulated Distillation, Surfactants, Triglycerides

Semi-volatiles (SVOCs), PAHs, EPA Methods (525, 610, 625, 8100, 8270D)

PCBs, Pesticides/Herbicides

EPA Methods, PCBs, Pesticides/Herbicides

Aroclors/PCBs, Haloacetic Acids, Herbicides, Insecticides, Multi-Pesticide Screening, Nitrogen Containing Pesticides, Organochlorine Pesticides, Organophosphorous Pesticides

EPA Methods (501.3, 502.2, 503.1, 524.2, 601, 602, 624, 8010, 8015, 8020, 8021, 8240, 8260), Pharmaceuticals, Residual Solvents, Volatile Organic Compounds (VOCs)

Aroclors/PCBs, Haloacetic Acids, Herbicides, Insecticides, Multi-Pesticide Residue/Screening, Nitrogen Containing Pesticides, Organochlorine Pesticides, Organophosphorous Pesticides

Amines, Aroclors, Drugs, EPA Methods (508, 608, 8081, 8141, 8151), Pesticides, Pharmaceuticals, Semi-volatiles, Steroids

Amines, Aroclors, Chemicals, Drugs, EPA Methods (508, 608, 8081, 8141, 8151), Pesticides, Pharmaceuticals, Semi-volatiles, Steroids

Alcohols, Amines, Aromatic Hydrocarbons, Drugs, Esters, PAHs, PCBs, Pharmaceutical Intermediates, Phenols, Solvents, Steroids, TMS Sugars, Tranquilizers

Aroclors, Nitrogen Containing Pesticides, Organochlorine Pesticides, Organophosphorous Pesticides

Antidepressants, Aroclors, Cholesterols, Drugs of Abuse, EPA Methods (508, 608, 8081, 8141, 8151), Glycols, Pesticides/Herbicides, Steroids, Triglycerides

Alcohols, Aldehydes, Aromatics, Essential Oils, Flavors & Fragrances, Free Fatty Acids, Glycols, OVIs, Pharmaceuticals, Solvents / Residual Solvents, Styrene, Xylene Isomers

Alcohols, Aldehydes, Aromatics, Basic Compounds, Essential Oils, Flavors & Fragrances, Glycols, Pharmaceuticals, Solvents, Styrene, Xylene Isomers

Acrylates, Alcohols, Aldehydes, Free Fatty Acids, Ketones, Organic Acids, Phenols, Volatile Free Acids

Dual-Column Chlorinated Pesticides by GC/ECD (EPA 8081, 8082, 8151, 504, 505, 508, and 552)

Abused Inhalant Anesthetics, Blood Alcohol Analysis

Drug Screening (6-MAM, Amphetamines, Barbiturates, Benzodiazepines, Opiates, PCP, THC)

Alcohol, Bioethanol, Fusel Alcohols



# COLUMN SELECTION PROBLEMS

## GC Column Cross-Reference Chart

Zebron™ Phase	Restek	Agilent® Technologies
ZB-1	Rtx®-1, Rtx-1PONA, Rtx-1 F&F	DB®-1, DB-2887, DB-1 EVDX, HP-1, HP-101, HP-PONA, Ultra 1, CP-Sil 5 CB
ZB-1ms	Rtx-1ms	DB-1ms, HP-1ms, CP-Sil 5 CB MS, VF-1ms
ZB-1HT Inferno™	Rxi®-1HT	DB-1ht, CP-SimDist
ZB-1XT SimDist	MXT®-1HT SimDist	CP-SimDist UltiMetal DB-HT SimDis
ZB-5	Rtx-5	DB-5, HP-5, Ultra 2, HP-PAS-5, CP-Sil 8 CB
ZB-5MSi	Rtx-5ms, Rtx-5Amine, Rxi-5ms	DB-5, HP-5ms, HP-5msi
ZB-5HT Inferno	Stx®-5HT, XTI®-5HT	DB-5ht, VF-5ht
ZB-5ms	Rtx-5Sil MS, Rxi-5Sil MS	DB-5ms, DB-5.625, DB-5ms EVDX, VF-5ms, CP-Sil 8 CB MS
ZB-SemiVolatiles	Rxi-5Sil MS Rxi-5ms	DB-5ms Ultra Inert HP-5ms Ultra Inert
ZB-35	Rtx-35, Rtx-35ms	DB-35, DB-35ms, HP-35, HP-35ms
ZB-35HT Inferno		
ZB-50	Rtx-50	DB-17, DB-17HT, DB-17ms, DB-17 EVDX, HP-50+, CP-Sil 24 CB
ZB-624	Rtx-1301, Rtx-624	DB-1301, DB-624, DB-VRX, HP-VOC, CP-1301, CP-Select 624 CB
ZB-1701	Rtx-1701	DB-1701 , CP-Sil 19 CB
ZB-1701P		DB-1701P
ZB-WAX	Rtx-WAX, Famewax, Stabilwax-DB	DB-WAXetr, HP-INNOWax, CP-Wax 57 CB
ZB-WAX <sub>PLUS</sub> ™	Stabilwax®	DB-WAX, CAM, HP-20M, Carbowax 20M, CP-Wax 52 CB
ZB-FFAP	Stabilwax-DA	DB-FFAP, HP-FFAP, CP-Wax 58 (FFAP) CB, CP-FFAP CB
ZB-MultiResidue™-1	Rtx-CLPesticides, Stx-CLPesticides	
ZB-MultiResidue-2	Rtx-CLPesticides2, Stx-CLPesticides2	
ZB-CLPesticides-1	Rtx-CLPesticides, Stx-CLPesticides	
ZB-CLPesticides-2	Rtx-CLPesticides2, Stx-CLPesticides2	
ZB-XLB	Rtx-XLB	DB-XLB, VF-XMS
ZB-XLB-HT Inferno		
ZB-Drug-1		
ZB-BAC1	Rtx-BAC-1	DB-ALC1
ZB-BAC2	Rtx-BAC-2	DB-ALC2
ZB-Bioethanol		

This section is, neither in terms of manufacturers nor in terms of their products, a complete list, and the accuracy of the data is not guaranteed. Small differences in dimensions or performance might be possible and slight adjustments to your application may be necessary.



Supelco	Alltech	SGE	OV
SPB-1, SPB-1 TG, SE-30, MET-1, SPB-1 Sulfur, SPB-HAP	AT-1, AT-Sulfur, EC-1	BP1, BP1-PONA, BPX1-SimD	OV-1
MDN-1, Equity-1	AT-1ms	SolGEL-1ms	
Petrocol 2887			
MDN-5, SPB-5, PTE-5, SE-54, PTA-5, Equity-5, Sac-5	AT-5, EC-5	BP5, BPX5	OV-5
MDN-5S			
HT-5			
MDN-35, SPB-35, SPB-608	AT-35	BPX35, BPX608	OV-11
Phenomenex Exclusive			
SP-2250, SPB-17, SPB-50	AT-50	BPX50	
SPB-1301, SPB-624	AT-624, AT-1301	BP624	OV-624
SPB-1701, Equity-1701	AT-1701	BP10	OV-1701
Met-Wax, Omegawax	EC-Wax	SolGEL-WAX™	
SUPELCOWAX 10	AT-Wax, AT-AquaWax	BP20	Carbowax 20M
Nukol, SPB-1000	AT-1000, EC-1000	BP21	OV-351

guarantee

MDN-12

Phenomenex Exclusive

Phenomenex Exclusive

Phenomenex Exclusive

If Zebron columns do not provide you with equivalent or better separations as compared to any other GC column of the same phase and comparable dimensions, return the column with comparative data within 45 days for a FULL REFUND.

# PROBLEM PREVENTION

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# PROBLEM PREVENTION

## The Inlet: Sample Injection Techniques

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One size does not fit all – there is no single injection mode that accommodates all samples and all columns. Instead, an appropriate injection mode introduces the sample so that it:

- Retains its original composition (i.e. there should be no sample degradation or selective losses during injection)
- Occupies the shortest possible length of column (the shorter the initial sample band, the sharper the peaks, the better the sensitivity, and the better the resolution)

### Injection Modes: Split

In split injection, the sample is rapidly vaporized and mixed with carrier gas. Most of the sample is vented through the split vent, while a small amount enters the column. The flow through the split vent divided by the flow through the column is called the “split ratio”. This rapid sample introduction provides the basis for sharp peaks and good resolution; it may however be inappropriate if sample components vary widely in their boiling points.

**Inlet Discrimination** | The less volatile components of a sample will not vaporize as rapidly, so immediately after injection the vaporized sample has a greater proportion of the more volatile compounds than the original sample. This effect is called “discrimination”. The longer the sample spends in the heated inlet, the less the discrimination – but the broader the peaks.

**Backflash** | Backflash occurs when the vaporized sample expands and exceeds inlet liner volume. Vapors may come in contact with cold spots (e.g. the septum or inlets to the injector) and less volatile components may condense. These condensates may vaporize later and interfere with subsequent analyses, sometimes producing “ghost peaks.” Expansion outside the liner may also expose the sample to active metal surfaces and reactive components of the sample may be lost. Minimized backflash by using a septum purge, small injection volumes, large volume inlet liners, and optimal inlet temperatures.

**Inlet Temperature** | The temperature should be hot enough to ensure rapid vaporization of the entire sample, but not too hot to degrade any analytes. Experimentation may be required to mitigate inlet discrimination and backflash. A good starting point is 250 °C.

**Septum Purge** | Gas sweeping the bottom face of the septum and through a purge vent carries contaminants out. Higher than optimum purge flows may result in the loss more volatile sample components. Septum purge flow rates are usually between 0.5 and 5 mL/min.

**Sample Size & Concentration** | Split injection is used for highly concentrated samples. Typical concentrations are from 0.1-10 µg/µL. Injection volumes of 1 to 2 µL are common, and up to 5 µL can be used without great problems, depending on the solvent used. If the sample volume is too large, backflash may occur.

### Injection Modes: Splitless

In splitless injection, the entire flow through the injector passes into the column for the first 15 to 90 seconds, and is then refocused.

**The Solvent Effect** | To avoid the broad peaks that would otherwise result from slow split injections, samples are refocused before starting the chromatographic process following splitless injection. Refocusing can be accomplished by adjusting the initial column temperature to 10 °C or more below the boiling point of the sample solvent. When the vapor leaves the injector and enters the cooler column, the solvent condenses at the front of the column as a liquid band; vapors will condense in this band and be trapped and refocused. This process is called the “solvent effect.” Improper use of solvent effect techniques result in broad, distorted peaks because solutes are not refocused into a narrow band near the beginning of the column. The solvent must form a compact, continuous flooded zone in the column. If the solvent does not wet the stationary phase sufficiently (as may be the case for methanol with a nonpolar phase), the flood zone may be several meters long and not of uniform thickness.



# PROBLEM PREVENTION

## The Inlet: Sample Injection Techniques

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**Cold Trapping** | Solutes that boil at 150 °C or more above the initial column temperature do not require the solvent effect in order to refocus. These high boiling compounds will condense at the beginning of the column in a short band without the aid of the solvent. This process is called “cold trapping.” Both the solvent effect and cold trapping can be achieved by operating in a temperature programmed mode.

**Sample Volume** | Samples are usually limited to 2 µL or less to avoid overloading the inlet liner and the column. Sample injection volumes must be reproducible in order to obtain reproducible retention times or quantitative data.

### Injection Modes: On-Column

On-column injection can eliminate syringe and inlet-related discrimination. If polar solvents are used with non-polar column linings, a retention gap is recommended. If the solvent is below boiling point at injection, the sample is distributed over a flooded zone at the front of the column and less volatile compounds are distributed in the phase. As the carrier gas evaporates the solvent at the front end, volatile components are concentrated and refocused.

**Sample Focusing** | The distribution of solutes in the area of the flooded zone is not homogeneous and this leads to peak broadening; this can be neglected for many applications and good quantitative results can still be obtained. If the compound boiling points are vastly different from the solvent, ballistic heating to high temperature can be employed. If compound boiling points are close to the solvent, temperature programming can be used. Wide-bore columns make on-column injection easier; alternatively a deactivated but uncoated wide-bore retention gap may be connected to a narrow-bore column.

**Sample Size** | Samples between 1 and 2 µL can be injected rapidly into a column below the boiling point of the solvent. To keep the flooded zone short, sample size should be limited to 1 µL.

### Injection Modes: Direct Injection

Direct injection should not be confused with on-column injection. It is a flash vaporizing method in which the inlet system is heated independently from the column oven. Sample evaporation occurs in the inlet.

### Injection Modes: Programmed Temperature Vaporizing (PTV)

In PTV injection, the liquid sample is injected into a cold glass liner. After withdrawal of the syringe needle, the vaporizing tube is heated in a controlled manner (usually rapidly) to vaporize the sample. This injection method permits special handling of the sample to vent the solvent, or to avoid thermal decomposition of thermally labile compounds, etc.




# PROBLEM PREVENTION

## The Inlet: Setting A Maintenance Schedule

Many GC troubleshooting issues arise because system parts need to be replaced. It is not always obvious which part needs replacing. Therefore, a fair amount of time can be spent locating the problem part before it can be fixed.

Instead of waiting for a problem to occur, it is best to be proactive about your GC maintenance. For instance, the majority of GC issues are inlet related. By replacing inlet parts such as liners and septa on a regular basis, problems will occur far less frequently. Since problems will be occurring less often, there will be fewer instrument downtimes, resulting in greater productivity.

Below is a list of inlet parts which should be replaced regularly to prevent instrument downtime. Depending on how dirty the samples are, some parts will need to be replaced more or less often. In such instances, adjust the length of time or number of injections that is appropriate for the samples.

	Item	Replacement Frequency
	Septa	100 Injections (depends on needle style)
	Inlet Liner	Sample & Matrix Dependent <i>Common Replacement Frequencies</i> <ul style="list-style-type: none"><li>• Dirty/Soil Samples: &lt; 2 weeks</li><li>• Water Extracts: ~ 4 weeks</li><li>• Headspace Extracts: ~ 6 months</li></ul>
	O-Ring	6 months (or with each liner change)
	Inlet Seal	Sample Dependent (no more than 6 months)



For a much more in-depth discussion of setting your GC inlet maintenance schedule, please contact your GC Specialist at [GCSpecialist@Phenomenex.com](mailto:GCSpecialist@Phenomenex.com).



# PROBLEM PREVENTION

## The Inlet: Maintenance & Cleaning

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**Warning!** This procedure involves the use of compressed gas and therefore eye protection should be worn.

**Note:** It best to have clean replacement liners or inserts available for quick exchange.

Full maintenance cleaning procedure:

1. Turn off inlet heat and allow inlet to cool.
2. Remove septum.
3. Remove liner or insert.
4. Remove base seal if applicable.
5. Use dry air or nitrogen to blow out any loose particles.
6. Use swab and solvent to clean interior walls if required.
7. Replace septum, liner or insert, and base seal.
8. Vent lines may also require replacement or cleaning.
9. Reassemble inlet and purge with clean, dry gas to remove solvent.

**Note:** Light maintenance may not require changing of septum or base seal. Avoid touching any parts that go inside the inlet with fingers as fingerprints will cause contamination.

# PROBLEM PREVENTION

## The Detector: Maintenance & Cleaning

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**Warning! Wear eye protection when working with fused silica tubing or compressed gas.**

### Electron Capture Detectors (ECD)

Because of the use of radioactive nickel in this type of detector, it should not be disassembled by those without specialized training and an appropriate license. Cleaning is limited to baking it out at 350 °C from 3 hours to overnight. Verify there are no leaks and the carrier gas is clean and dry before baking.

### Flame Ionization Detectors (FID)

The collector bore and the jet require occasional cleaning to remove deposits. The deposits, which usually consist of white silica from column bleed or black carbonaceous soot, cause noise and spikes.

#### Cleaning procedure:

1. Turn off detector and its heater.
2. Turn off gases to the detector.
3. Allow time for the detector to cool.
4. Open up the detector and use mechanical means (brush, wire, etc., and compressed gas) to remove contamination.
5. Wash out the collector with distilled water and organic solvents as required.
6. Dry in an oven at about 70 °C for more than half an hour.

### Flame Photometric Detectors (FPD)

#### Cleaning procedure:

1. Set instrument temperatures to cool to safe temperatures.
2. Turn off gasses to the detector.
3. Turn off power to the gas chromatograph and unplug main power cord.
4. Remove detector covers, disconnect, and remove the detector.
5. Remove and inspect jet assembly. Remove any deposits mechanically, for example, by using a wire.
6. Inspect and clean, if necessary, the glow plug and the quartz windows.
7. Blow loose particles away with compressed gas.
8. Replace the jet if it is damaged or difficult to clean with a wire.

### Nitrogen Phosphorous Detectors (NPD)

**Caution:** If the hydrogen gas used to fuel the NPD detector is left on after the detector is disconnected from the column, this gas can accumulate in the oven and create an explosion hazard.

The collector bore and the jet require occasional cleaning to remove deposits. The deposits, which usually consists of white silica from column bleed or black carbonaceous soot, cause noise and spikes.



# COLUMN CARE & USE GUIDELINES



# COLUMN CARE & USE GUIDELINES

## Column Installation

### Pre-Installation Checklist

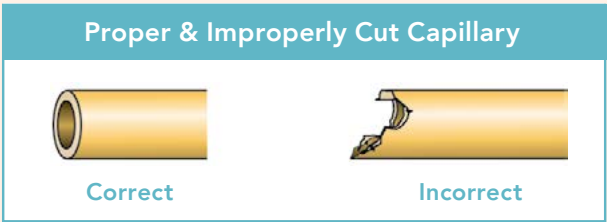
- Replace oxygen, moisture and hydrocarbon traps as necessary.
- Ensure that the injection port is clean and free of sample residues, septum, or capillary debris.
- Check and replace as necessary critical injector components such as seals, liners, and septa.
- Check and replace detector seals as necessary.
- Carefully inspect your column for damage or breakage.
- Check gas cylinder pressures to ensure that an adequate supply of carrier, make-up and fuel gases are available. Carrier gases should be of the highest purity. **Note:** It is critical that oxygen and water be removed from the carrier gas by the appropriate use of filters and adsorbents.

### Designate A Flow Direction

**Note:** GC columns do not have a specific directional flow when received from the manufacturer. Upon initial use of your new Zebron™ column, Phenomenex recommends the practice of dedicating one specific end of the column for injector installation only. This is particularly important when dealing with active/caustic or contaminating compounds. If these compounds are routinely injected onto the column, degradation of the phase will occur—leading to higher bleed. A typical first step to remedying (removing) this bleed would be to trim 10 cm from the front (injector) end of the column and keep trimming this inlet end of the column as necessary. Trying to remedy any bleed issues by trimming the column may not work if both ends have been interchangeably installed into the inlet.

### Ensure A Proper Column Cut

- 1 Score capillary with smooth edge of wafer at a 45° angle
- 2 Apply force in a downward direction
- 3 Tubing should break cleanly
- 4 Inspect cut with a magnifying glass - the cut should be smooth, not jagged



# COLUMN CARE & USE GUIDELINES

## Column Installation

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### Installation Into the Injector

1. Place a capillary nut and ferrule on the injector end of the GC column, allowing a section of column to protrude. Trim one to two centimeters from the protruding end to remove ferrule contamination that may have entered the column. Inspect the cut with a magnifier to ensure that a smooth, clean, square-cut edge has been made - recut if necessary.
2. Carefully hang the column in the GC oven, being cautious not to scratch or damage the polyimide coating on the capillary tubing. Rotate the column to avoid sharp bends of the capillary column and any contact of the column with oven surfaces.
3. Insert the column into the injector exactly the correct distance specified in the instrument manual. Tighten the ferrule nut finger-tight then 1/2 turn with a wrench. If the column can still be moved, tighten another 1/4 turn until the column is secure.
4. Adjust the carrier gas to obtain the flow rate listed on the test chromatogram.

### Installation Into the Detector

**Note:** For users with sensitive detectors such as MS and ECD, column conditioning steps should be performed before installing the column to prevent contamination and frequent maintenance of the detector.

1. Place the column nut and ferrule past the end of the column and cut a centimeter or two off the end of the column. Be sure that the ferrule is the right size and pointing in the correct direction. Inspect the cut with a magnifier and ensure that the cut is square and smooth. Recut if needed.
2. Insert the outlet end of the column into the detector exactly the distance prescribed in the instrument manual. Distances will vary between detectors. Tighten the ferrule nut finger-tight then 1/2 turn with a wrench. If the column can still be moved, tighten another 1/4 turn until the column is secure.
3. Inspect the column connections for leaks using an electronic leak detector. Leaks at the inlet end may introduce oxygen to the column that will result in increased column bleed and damage to the column phase.

# COLUMN CARE & USE GUIDELINES

## Conditioning Basics: The Column

### Column Conditioning Steps

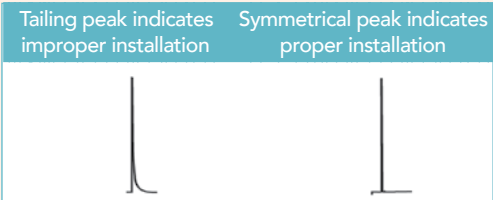
1. Allow sufficient time for the carrier gas to flow through the column to purge any oxygen that may be in the system.
2. Raise the temperature of the column to the maximum isothermal operating temperature that is listed on the individual Zebron™ GC Column Test Report. Maintain this temperature until a constant baseline is achieved. Conditioning times will depend on the phase identity and thickness, with thicker films taking longer to stabilize. In order to minimize the downtime of the instrument, columns can be conditioned overnight at the maximum isothermal temperature.

### Installation Testing

1. Inject a detectable unretained sample, such as methane for an FID, to determine dead volume time and linear gas velocity at the desired column temperature. Adjust gas pressure for optimal flow depending on carrier gas selection.
2. The non-retained peak must have ideal peak shape or installation is faulty and needs to be redone.

If the peak is broad and/or tailing, check the following:

- Improper column positioning/insertion into inlet or detector
- Gross contamination of the splitter sleeve
- Chipped or cracked splitter sleeve
- Improper sweeping of sample at column end by makeup gas
- Damaged or crushed column end



### Unretained Peak Times and Markers

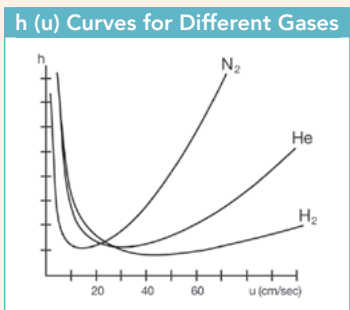
**Methane with FID/TCD:** Calculate linear velocity by injecting 25-100  $\mu\text{L}$  of 1% methane in  $\text{N}_2$  gas blend. Measure the retention time of the methane peak and calculate the following:

**Linear Velocity ( $u$ ) =  $L/t_0$**

Detector Type	Marker Compound
ECD	Methylene chloride <sup>2,3</sup> , Dichlorodifluoromethane
FID	Methane, Butane <sup>1</sup>
NPD	Acetonitrile <sup>2,4</sup>
PID ELCD	Vinyl chloride
TCD, MS	Methane, Butane <sup>1</sup> , air
<ol style="list-style-type: none"><li>1. From a disposable lighter</li><li>2. Place 1-2 drops in an autosampler vial and tightly cap. Shake and inject 1-2 <math>\mu\text{L}</math> from the headspace of the vial. Do not inject any liquid.</li><li>3. Use a column temperature above 55 <math>^{\circ}\text{C}</math>.</li><li>4. Use a column temperature above 95 <math>^{\circ}\text{C}</math>.</li></ol>	

### Recommended Non-Retained Retention Times

Length (m)	$\text{H}_2$ (sec)	He (sec)	$\text{N}_2$ (sec)
15	38	75	150
30	75	150	300
60	150	300	600





# COLUMN CARE & USE GUIDELINES

## Conditioning Basics: The System

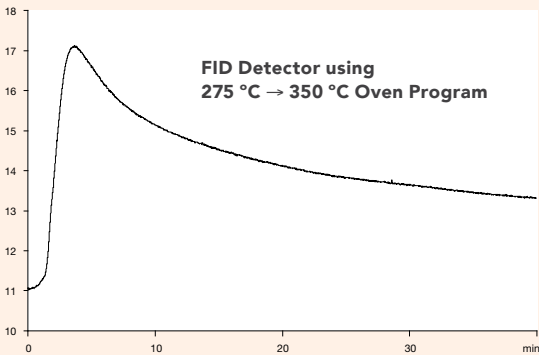
Increased signal is common and expected during the conditioning step when new components are installed. After a new column installation, detectors show increased signal response that decrease slowly over time with constant increased temperature. Misconception is that the baseline rise is solely due to column bleed. It may be a combination of many causes that collectively can be called system bleed. In fact, at temperatures below 200 °C, almost all background signal is the result of system noise.

### Detector Effects

During new column installation, detectors are sometimes allowed to cool for convenience. If the column is connected to the detector during conditioning, the detector can become contaminated. When the detector is heated following column installation an increased signal will be observed that can be interpreted as column bleed as shown below. An increase in detector temperature from a constant temperature of 275 °C to 350 °C caused a signal increase of 6 pA!

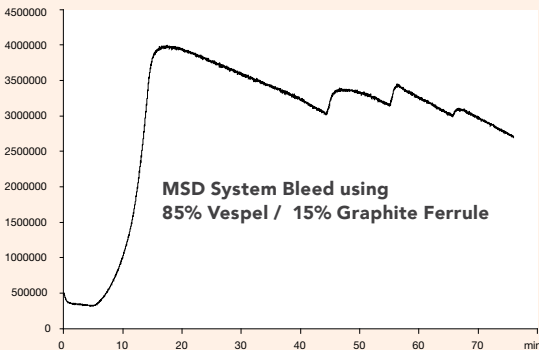
The effect would be greater if the detector had been constant at room temperature for an extended period of time. It is not uncommon for more sensitive detectors such as Electron Capture Detectors (ECD) to show very high signals that may persist for days after prolonged dormancy.

Repetitive heating and cooling of detectors can cause seal and ferrule distortion, allowing leaks to form. This might introduce oxygen into the system resulting in increased signal response.



### Accessory Effects

If the detector temperature remains constant, other causes for baseline increases are still possible. Ferrules absorb gases and other substances that offgas when heated. Figure shows a spectrum that was obtained when an uncoated capillary column, which does not contain stationary phase, was installed using new ferrules. The signal is due to system noise only and not column bleed. Notice that the intensity of the signal is very high and would be greater than most analyte peaks. This would decrease the signal to noise ratio making detection limits much worse than if the system was conditioned and the baseline minimized.

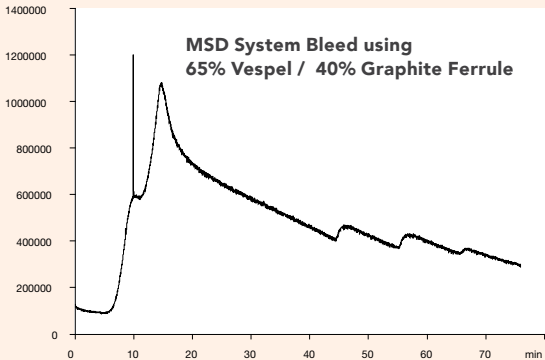


**Oven Program:**  
40 °C for 5 min to 320 °C at 30 °C/min for 30 min to 340 °C at 30 °C/min for 10 min to 360 °C at 30 °C/min for 10 min to 370 °C at 30 °C/min for 10 min.

# COLUMN CARE & USE GUIDELINES

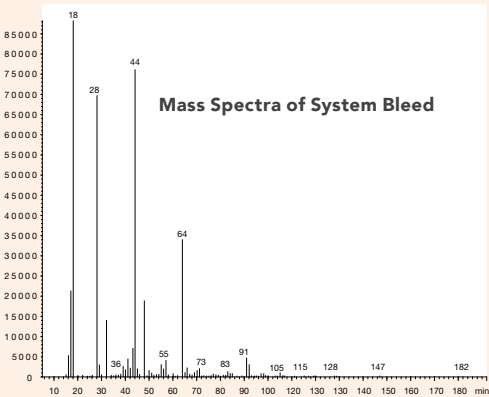
## Conditioning Basics: The System

Ferrule composition also effects system bleed. After the completion of the same oven program using a ferrule with higher graphite content, the signal is 90% lower.

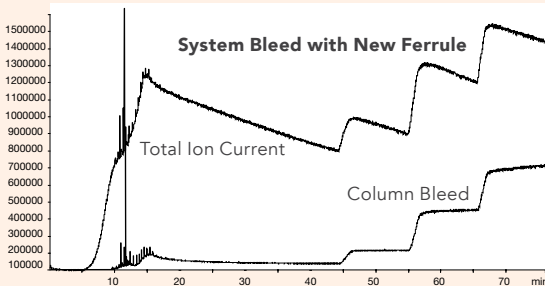


**Oven Program:**  
40 °C for 5 min to 320 °C at 30 °C/min for 30 min to 340 °C  
at 30 °C/min for 10 min to 360 °C at 30 °C/min for 10 min to  
370 °C at 30 °C/min for 10 min.

The major ions seen are 17, 18, 28, 32, and 44, and 64. Most masses can be easily explained by common gases adsorbing on the ferrule (such as water, carbon monoxide, nitrogen, oxygen, and carbon dioxide.) Since these same ions are indicators of a gas leak or contaminated vacuum chamber, an air and water check was run and passed before analysis. Subsequent runs after initial conditioning showed drastically reduced signals for these ions. An air leak would likely remain constant.



New 60/40% Vespel-Graphite ferrules were installed with a Zebron™ ZB-5 column. Subtracting the ions associated with system bleed shows bleed from column conditioning only. At normal operating conditions, over 90 % of the ion intensity is completely due to system bleed derived from ferrule offgassing.



Installation of any column should be followed by a heating cycle to condition the system. During this conditioning cycle, the entire system is being conditioned, not only the column. To prevent detector contamination, it is recommended that you do not connect the column to the detector. Ferrule composition can also determine the extent of conditioning needed, as it is the key component responsible for most offgassing. Injector contributions should also be considered when determining system bleed; they may appear as peaks.





## TROUBLESHOOTING GUIDE

Learn more and access free resources at  
**[www.phenomenex.com/TroubleGC](http://www.phenomenex.com/TroubleGC)**

### Australia

t: 02-9428-6444  
f: 02-9428-6445  
auninfo@phenomenex.com

### Austria

t: 01-319-1301  
f: 01-319-1300  
anfrage@phenomenex.com

### Belgium

t: 02 503 4015 (French)  
t: 02 511 8666 (Dutch)  
f: +31 (0)30-2383749  
beinfo@phenomenex.com

### Canada

t: (800) 543-3681  
f: (310) 328-7768  
info@phenomenex.com

### Denmark

t: 4824 8048  
f: +45 4810 6265  
nordicinfo@phenomenex.com

### Finland

t: 09 4789 0063  
f: +45 4810 6265  
nordicinfo@phenomenex.com

### France

t: 01 30 09 21 10  
f: 01 30 09 21 11  
franceinfo@phenomenex.com

### Germany

t: 06021-58830-0  
f: 06021-58830-11  
anfrage@phenomenex.com

### India

t: 040-3012 2400  
f: 040-3012 2411  
indiainfo@phenomenex.com

### Ireland

t: 01 247 5405  
f: +44 1625-501796  
eireinfo@phenomenex.com

### Italy

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f: 051 6327555  
italiainfo@phenomenex.com

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f: 001-310-328-7768  
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nzinfo@phenomenex.com

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f: +45 4810 6265  
nordicinfo@phenomenex.com

### Puerto Rico

t: (800) 541-HPLC  
f: (310) 328-7768  
info@phenomenex.com

### Sweden

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f: +45 4810 6265  
nordicinfo@phenomenex.com

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t: 01625-501367  
f: 01625-501796  
ukinfo@phenomenex.com

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f: (310) 328-7768  
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### All other countries: Corporate Office USA



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info@phenomenex.com



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# PeakSimple MG#5 [TABLES]

Channel 1 events

Time	Event
0.000	ZERO
0.000	SOUND
0.100	D ON
0.130	D OFF
0.300	G ON (ValveRotate)
0.850	G OFF (ValveRotate)
2.000	ZERO
2.010	INTEG IMMEDIATE
4.800	DATA ZERO
5.000	F ON (ValveRotate)
5.200	DATA NORMAL
5.200	ZERO
5.250	INTEG IMMEDIATE
5.900	ZERO
6.000	INTEG IMMEDIATE

Add... Change... Load... Save...

Event details C:\Peak488Win10\Bill01.evt

Event type

☐ A (StopFlow)

☐ B (StopFlow)

☐ C

☒ D **VacPump I'Face** ☒ On

☐ E

☐ F (ValveRotate) **BackFlush**

☐ G (ValveRotate)

☐ H

☐ Zero ☐ Zero(100%T)

☐ Reverse

☐ Peak sensitivity:

☐ Base sensitivity:

☐ Sound 

☐ Automatic integration

☐ Integration - None

☐ Integration - Drop

☐ Integration - Based

☐ Integration - Lead skim

☐ Integration - Trail skim

☐ Integration - Lead horiz

☐ Integration - Trail horiz

☐ Integration - Inhibit

☐ Integration - Based immediate

☐ Integration - Rubber band start

☐ Integration - Rubber band stop

☐ Command:

☐ Absorbance mode on

☐ Absorbance mode off

☐ Integration - Zero

☐ Data - Zero

☐ Data - Normal

☐ Pump start

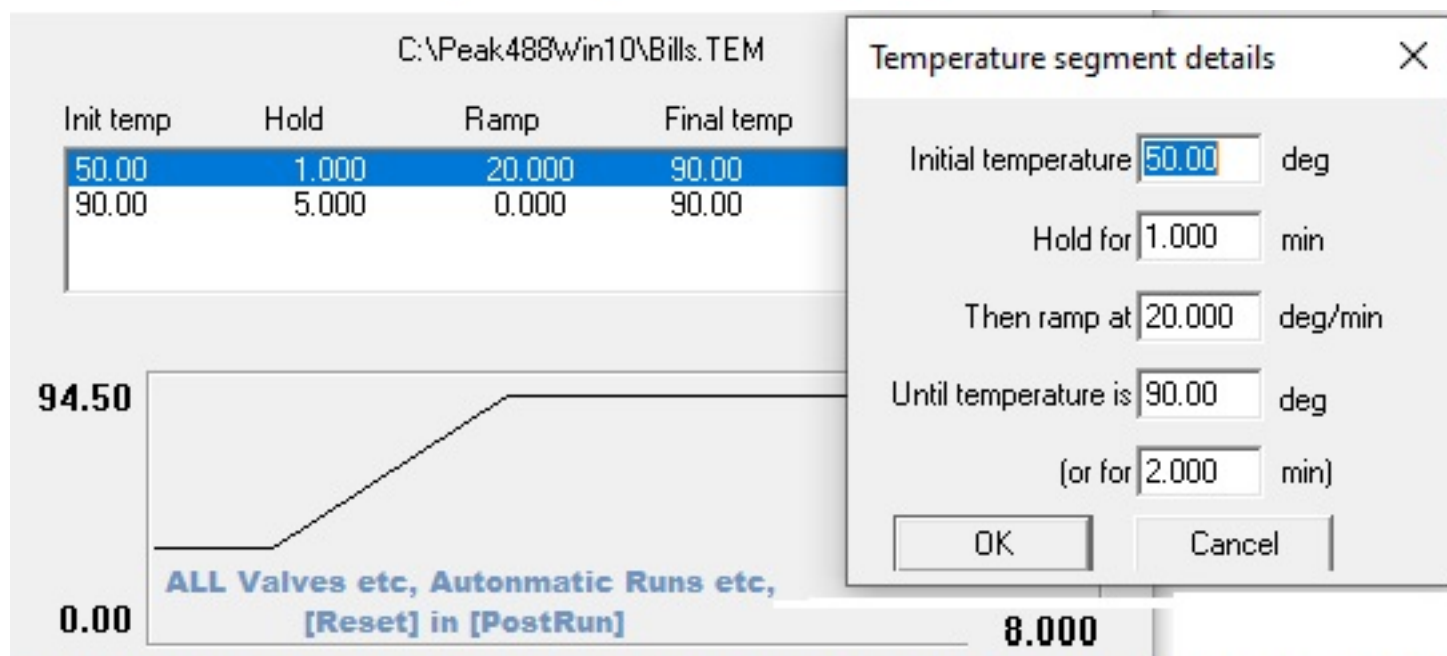
☐ Pump stop

☐ Autosampler control script:

Event time:

OK Cancel

[Event Table] MG#5 Bill01.evt Air; O2, N2, CO2-Methane-water



a NEW [.TEM] for column BakeOut] with **ALL Columns (200degC MAX)** / monitor Detectors **MS5A Column ONLY** to 300degC WITH DETECTORS DISCONNECTED & TURN-OFF !  
MAX # Hours recommended & ONLY WHEN NEEDED !



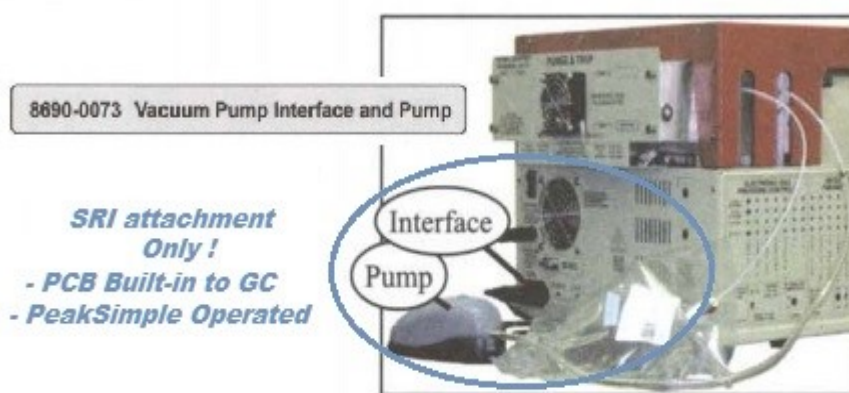
# Air Sampling Accessories – 2019

Air Sampling Labs cover a diverse range of sampling requirements and accessories—more a matter of detailed methodology

SRI GCs are more generally aimed at analytical laboratories rather than dedicated GC process control applications offer many as low cost easily installed integrated components based on the range of Multi-Gas Analysers.

- **Some built-in to the GC**— generally requires SRI factory installation
  - 1 **Vacuum Pump Interface** (8690-0073); ( SRI GC Cat 200 p89 ) including external vacuum pump and gas sampling bag use ( for accurate GC calibration )
  - 2 Non-standard Solenoid valves requires extra built-in circuit boards

The SRI GC is relatively “user-friendly” in terms of internal accessibility
- **Recommended but for proficient and suitably “trained” operators )**



- **Add-on — DIY**
- 2 **Gas Purifiers**, [PDF](#) High Purity Gases and gas regulators; particular for trace gas analysis < low ppm levels.  
- > many options



**OXYTraps** are recommended for any GC for all GC type columns to minimise background signals often attributed to bleed even where GC columns are to be operated at high temperatures ( even capillary columns at <100degC)

- care required ( monitor color , a fast change-over is mandatory ! )
- use isolation valves if /when GC is NOT being used > best to leave carrier gas running 24/7 if need be !

**H2 generator** (2014-HO20-2 new addition [PDF](#)) for FID H2 fuel gas and/or where H2 can be used as carrier gas for safety/ or mobile applications ( 100ml/min is adequate for a single

- NOT necessarily recommended for spasmodic GC operation but long terms stability ( >days) for system stability.  
Lab applications > where possible Helium or H2 gas bottle supply ***might be*** preferred.
- A Gas Purifier (OXYTrap many options see [PDF](#)) is also recommended installed as close to the GC as possible with a minimum of fittings.



**H2-100 Hydrogen Generator #2014-H100-2**  
**Hydrogen Generator**  
 Make GC quality hydrogen from distilled water  
 Hydrogen generators are perfect for labs that would prefer, or can't have hydrogen cylinders in them.

- 100mL/min flow, 50 psi outlet pressure
- 22" x 14" x 20", shipping weight 52 lbs
- 115 or 230VAC, 100 watts
- Quiet operation
- One year warranty
- Ideal for FID H2 Fuel with built-in mini Air Compressor
- As H2 Carrier Gas requires OxyTrap for Capillary columns and trace gas analysis



- All SRI **FID GCs** have a built-in **mini Air Compressor as standard** (8690-2270 ([SRI Cat200 p89](#))) enabling a gas bottle-less GC system for that extra portability as carrier gas; > Change over of a few fittings may be re-quired.

### Built-in "Whisper Quiet" Air Compressor

- Built into the GC Chassis
  - Powerful enough to supply FID air (300mL/minute)
  - Convenient—Recommended for Field Work
- NO Air Cylinder Required !**

8690-2270 Built-in "Whisper Quiet" Air Compressor 220 VAC



- **Sample stream in-line accessories**

- 4 **Sample Stream "Nafion" Drier** (8670-5870, [SRI Cat200 p88](#)) for "wet" humid samples (% water) tends to deactivate MolSieve and adsorbent type columns over-time requiring periodic high temperature re-activation. Similarly with many high concentration samples of CO2(%) will also deactivate MolSieve columns .



### "Nafion" Sample Stream Drier

- Uses rechargeable Molecular Sieve dessicant beads and Nafion tubing
- Water is absorbed while gases pass through unaffected
- For use with water sensitive columns
- A simple, economic way to dry gas samples for GC

8670-5850 Sample Stream Drier





- 5 **10 vial Gas Autosampler** ( 8690-0047 [PDF](#) ) for external field collection of gas samples in VOA glass vials ( requires Any pre-installed auto GSV GC system )

### 10 Vial Gas Autosampler 8690-0048

- 20/40ml Screw VOA Vials x10
- fit to any SRIGC with a built-in auto GSV - SRI factory fitted



- 6 **7ml custom vials - 500ea Bandalero Belt 7ml Gas Tube Sampler** [PDF](#) - typically custom sample tubes
- > field loaded ( onto an "ammunition-type-belt" ) for automated injection into a suitably modified SRI GC back in the lab - requires some expertise level for operation, setup and use ]
  - > extra unique skill in getting "vacuum safe vial seal !

### GC Multi-Tube Autosampler - as fitted to a standard GC



### "Unique" Crimp Top Evacuation Chamber

- A GC Attachment
- for Any GC with GSV Data SYS Control  
eg SRI 8610 Greenhouse Gas GC
- To 500 tubes per belt
- With many options !



- & Tends to be a one shot analysis per vial ! ( any duplicates of same sample can be problematic !  
( 2nd is under a reduced pressure )

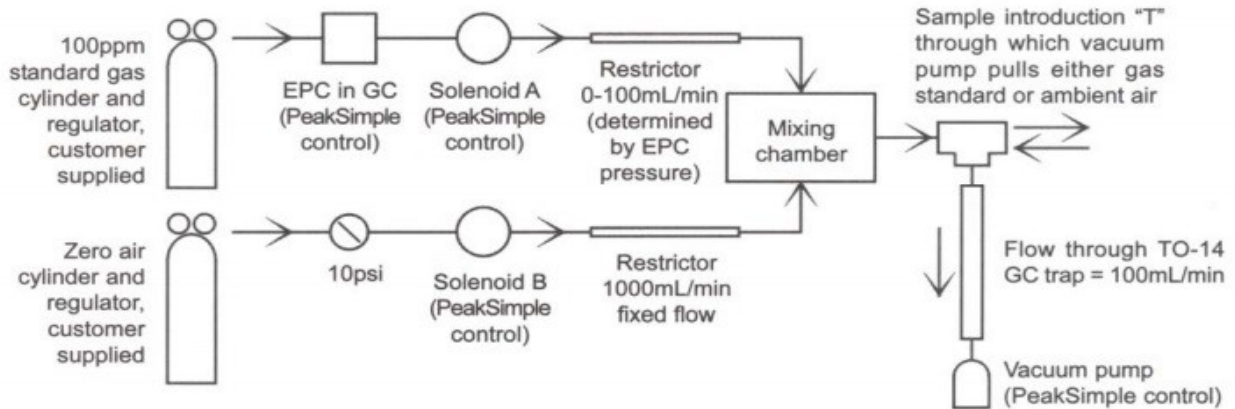
**GC run time per sample** - can be 2-6mins up to 15mins if temperature programming is required for typical simple MG#5

- as a brief test sample you can use ambient air or as a dilute Natural Gas.

- **strictly for experts process control Apps requiring high accuracy & regular calibration at low ppm>ppB levels (~AUD6000) eg for auto-generation of non-linear calibration curves**

## Automated Calibration System (ACS)

For the SRI TO-14 Air Monitoring GC



### Other Consumables

- **Columns** ( generally packed columns for gases > also PLOT .  
> long term life with care >> 12months )
- **Gas Syringes** - for simple gas apps plastic **Norm-Ject** type ( [PDF](#) )are adequate 1ml manual injection larger syringes for "spot" sample dilution. Via plastic fitting Connectors

## Norm-Ject Syringes

### re-Usable

**NOT necessarily throw-a-way**  
**NO "black tip" to clog or dry-out**  
**NO Built-in obsolescence**

**Gas-Tight ALLPolyProp**

**also similar up to 140cc**  
**and Jumbo 1-2 liter**



- **Carrier Gas Lines** 1/8"OD High Purity pre-cleaned Copper is adequate except for ECD Detectors
- ◇ **Sample transfer Lines**—simple applications 1/8"OD Teflon or SS is adequate
- ◇ For <ppm > ppB and for reactive compounds eg ( S-Cpds ) Silcosteel/Sulfinert is mandatory—also for faster response for moisture at <ppM levels compared to SS



- ◇ **Calibration Gas Standards**  
A multitude of possibilities ( All generally customised but some standardised ) and in different size cylinders — depending on your anticipated use-age rate ?

**Specify ; matrix; components to be analysed, concentration (ppM v/v) of each component to be quantitated**

for SRI MG#5# & TO14 GC

**see Restek Gas Standards>**

**Natural Gas Standard 34438** x10 components, 13litre cylinder (AUD1100)

**Refinery Gas #1 34441**(x28components)AUD1600) Restek PDF (p431) but we also get from Linde Gas, Air Liquide . . . depending . . . ! see p11 for Typical Refinery-type

**2nd Source TO-14A/TO-15 Gas Calibration Standards**

- Standards from TWO manufacturers provide second source on one order.
- 12 month stability in transportable cylinders.
- Drop shipped for fast delivery and maximum shelf life.

A. Spectra (Linde) 104L Cylinders  
B. Scotty (Air Liquide) 110L Cylinders (Pi-marked Cylinders for EU Regulations)

For regulators, see page 433.

For more available gas standards, visit [www.restek.com/air](http://www.restek.com/air)

- **Some “unique” SRI GC Features**

**SRI GCs** have a built-in Operating System **Peak Simple Data System ( 1-channel per Detector, 1or6 CH-system built-in )** via a s'ware “timed events” Table Manual GC Injection Port / gas sampling timing, column switching on/off switching of external or built-in solenoid switches or valves

- also for control of conventional GC Autosamplers ( *Liquid & Headspace* )

- ◇ Each MGA GC configuration is a completely function internally automated as required with all valves, solenoids, multiple columns if/as required and installed for the designed application

- **New features MGA#5**

**Methaniser - High Capacity** Methaniser FID jet now standard to automate many aspects of the overall GC system

- ◇ Additional Gas sample injection by syringe via the septum injection port or by sample loop Gas Sampling Valve (GSV) automated injection normally 1ml max **Dual GSVs** now standard

Detectors : **TCD, FID-Methaniser**

**TCD** 200ppM-% Concentrations—all permanent gases (plus C1-C6 HCs); except H<sub>2</sub> in simple MG#5 system ( requires Argon carrier gas ); % to low ppM

**FID** low ppM to mid % **for HCs ONLY !**

**FID/Methaniser** low ppm CO and CO<sub>2</sub>

cont from p5

- ◇ Standard SRI GC MGA GCs use SS packed columns 1/8"OD.
- ◇ 3 Columns Mol Sieve, HaySepD and a 3rd Haysep G for back-flushing requirement.
- ◇ Optional 4th Column capability - MXT-1 capillary column for higher MW components and/or S-Compounds ( requiring an extra optional **Dual FID/FPD** Detector ).
- ◇ Marked improvement can be achieved with high resolution capillary GC columns reLimits of detection and component resolution .

**— BUT WITH THE ADDED NEED FOR DETAIL ATTENTION**

***Limit of Detection — largely determined by baseline noise drift etc,  
a function of carrier gas purity.***

***This PDF is but a brief summary of gas analysis as implemented by SRI-GC & some of our Other Suppliers***

- NOT exhaustive !
- VERY Generalised !
- NOT ALL Options are necessarily still available — ASK!
- Please select options judiciously before placing you Order —be prepared to discuss full details of your APP  
***Our DISCLAIMER APPLIES !***
- ANY Prices quoted here are in AUD Ex-GST and INDICATIVE ONLY
- Some options are difficult to install in Australia / retrofit after “initial” delivery has been made
- internal GC components may be required  
***Generally the GC needs to be Return to the SRI Factory in CA-USA— IN THE ORIGINAL PACKING CASE > ALL freight costs are customer responsibility***

***Come back later ! we add to this PDF from time - to - time !***

# Gas Sampling Devices

Gas Sampling Bags are a Cost-Effective Alternative to Cans and Tubes for Many Applications

TO-14 Can  
➤ **SilcoCanisters**



	Canister	Gas Sampling Bag	Solvent Desorption Tube
Media Type	whole air	whole air	adsorption
Sensitivity	ppb	ppb	ppm
Technique	passive (no pump)	active	active
Sample Type	grab or integrated	integrated	integrated
Analyte	wide range of VOCs	permanent gases	sorbent specific
Applications	ambient, IAQ, emergency response, IH	ambient, IAQ emission	IAQ, IH
Durability	reusable	one time use	one time use
Inertness	excellent	excellent	fair
Stability	30 day	30 day	varies by analyte
Sample Volume	0.4-6 L	0.4-6 L	varies by analyte
Sampling Time	minutes to days	minutes to hours	minutes to hours

- Quickly confirm vacuum or pressure inside canister.
- Monitor pressure changes.
- Fully protected by canister frame.
- Can be heated to 90°C during cleaning.

## Gas Sampling - Devices

**SS Canister** : 0.5, 1.0, 6.0, 15liter

- TO-14 : for permanent gases and VOCs > generally >100ppB to %

- **Silco Canisters** : [PDF](#) ultimate inertness - Sulfinert > H<sub>2</sub>S <10ppB, VOCs < 1ppM

requires extensive cleaning between sampling.

( multiple Vacuum heat cycling purging to EPA "protocols ")

**Gas Sampling Bags** : [PDF](#)

0.5, 3, 5, 10, 25liter in **Altef** (Tedlar) LowppM VOCs ( NOT ketones, acetates, H<sub>2</sub>S )

1.3.5, 10liter in **Multi-Layer Foil** > low MW VOCs ( but NOT <lowppM ), permanent ga



**ALTEF Gas Sampling Bags**

**Multi-Layer Foil Gas Sampling Bags**

**Get the convenience of having both a hose connection and a syringe port in a single valve!**

**Polypropylene Combo Valve**

- Inert polypropylene
- 1/8" diameter valve stem
- Replaceable Teflon®-faced septum



**Vacuum Bag Sampler**

- Fast bag sampling without contamination from sample passing through pump.
- Bag capacity up to 10 L.

## Solvent Desorption Tubes - high MW VOCs in air

> similar in principle to :simple : DRAEGAR Tubes

( Color change tubes > concentration dependant, crude OH&S industrial use )

(> see elsewhere NOT Chromalytic )

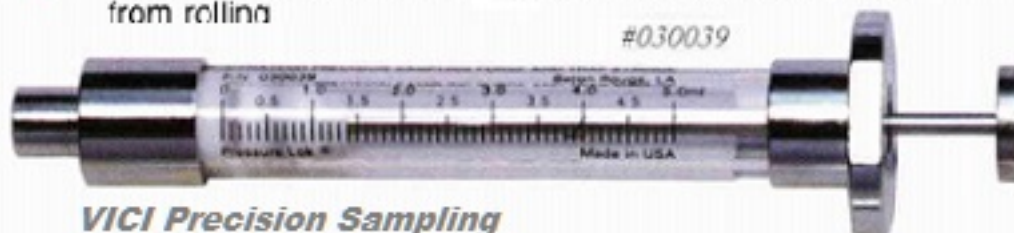


# Gas-Tight Syringes

## Gas Syringe for VOCs

### Purge and Trap Syringe Accept standard Luer hub needles

- ▶ **Luer lock** – for use with purge and trap analysis
- ▶ **Frosted glass on syringe barrel** – allows easy volume reading and accepts writing
- ▶ **Plunger tip of stress-formed virgin PTFE** – self-lubricating and durable.....
- ▶ **Heavy duty rear flange** – flat edges keep the syringe from rolling



**VICI Precision Sampling**

**5 ml    10 ml**

030039    030040

Luer needles must be ordered separately.

**Luer needles**

**Size:** .028" x .016" x 2"

**Type:** Bevel, open end

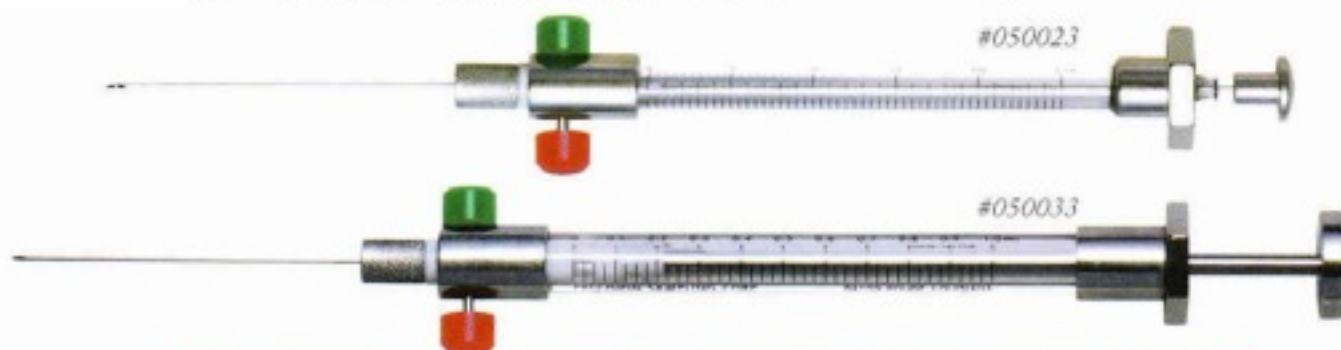
**Pkg. of 3:** 943061

## Precision Sampling Gas-Tight Syringes

### Series A-2

Removable needles: .028" x .005" x 2", bevel, open end on 25, 50, and 100 µl  
.029" x .012" x 2", bevel, open end on all others

- ▶ **Push-button valve** – allows instantaneous injection
- ▶ **Smaller volumes** – great for small volatile samples



	25 µl	50 µl	100 µl	250 µl	500 µl
<b>Standard:</b>	050023	050024	050025	050031	050032
<b>Luer:</b>	050043	050044	050045	050051	050052
	1 ml	2 ml	5 ml	10 ml	
<b>Standard:</b>		050033	050034	050035	050036
<b>Luer:</b>		050053	050054	050055	050056

#### Replacement needles for standard syringes (Luer needles on page 11)

<b>Size:</b>	.028" x .005" x 2"	.029" x .012" x 2"	.029" x .012" x 2"
<b>Type:</b>	Bevel, open end	Bevel, open end	Side port, taper
<b>Pkg. of 3:</b>	943050	943051	943052



# more Syringes - devices

## Jumbo Syringes

*Polycarbonate Body  
Silicone O-ring*

Jumbo Syringes (500mL to 2L)

- Designed for holding and dispensing large volumes of gas
- Heavy duty acrylic barrels
- Easy access to sample for the addition of standards or removal of subsample via secondary port
- Plunger stem can be unscrewed for ease of transportation and storage

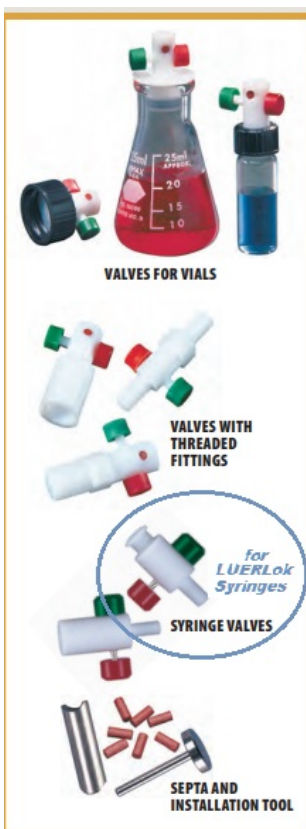


Part no. ▴ ▾	Syringe Volume ▴ ▾	Syringe Code ▴ ▾	Replacement O-Ring
009910	500mL	500MAR-LL-GT	032527
009920	1L	1000MAR-LL-GT	032532
009930	2L	2000MAR-LL-GT	032537



### MININERT™ VALVES

Mininert™ push-button valves are highly dependable, leak-tight closures for screw-cap vials and other laboratory containers. When used with a glass vial, only PTFE and glass are in contact with the contents. Their unique features make Mininert valves the ideal closure



**Stopcock Valves 3-Way**  
(also 2-Way)

*inert gases ONLY  
NOT necessarily for  
Trace Gases  
< ~ 100ppM*

### Plastic Syringes "Terumo" -type

*limited Use?*



Various  
Sizes  
1-140cc

*depends on  
siliconised  
"O-ring) seal*

**LuerLok preferred**

*"slides" more easily !*

*both OK for "inert" gases - pressure to > 50psi / Vacuum Use  
short term Only re inertness / leak-tight !*

### Norm-Ject® All Plastic Syringes



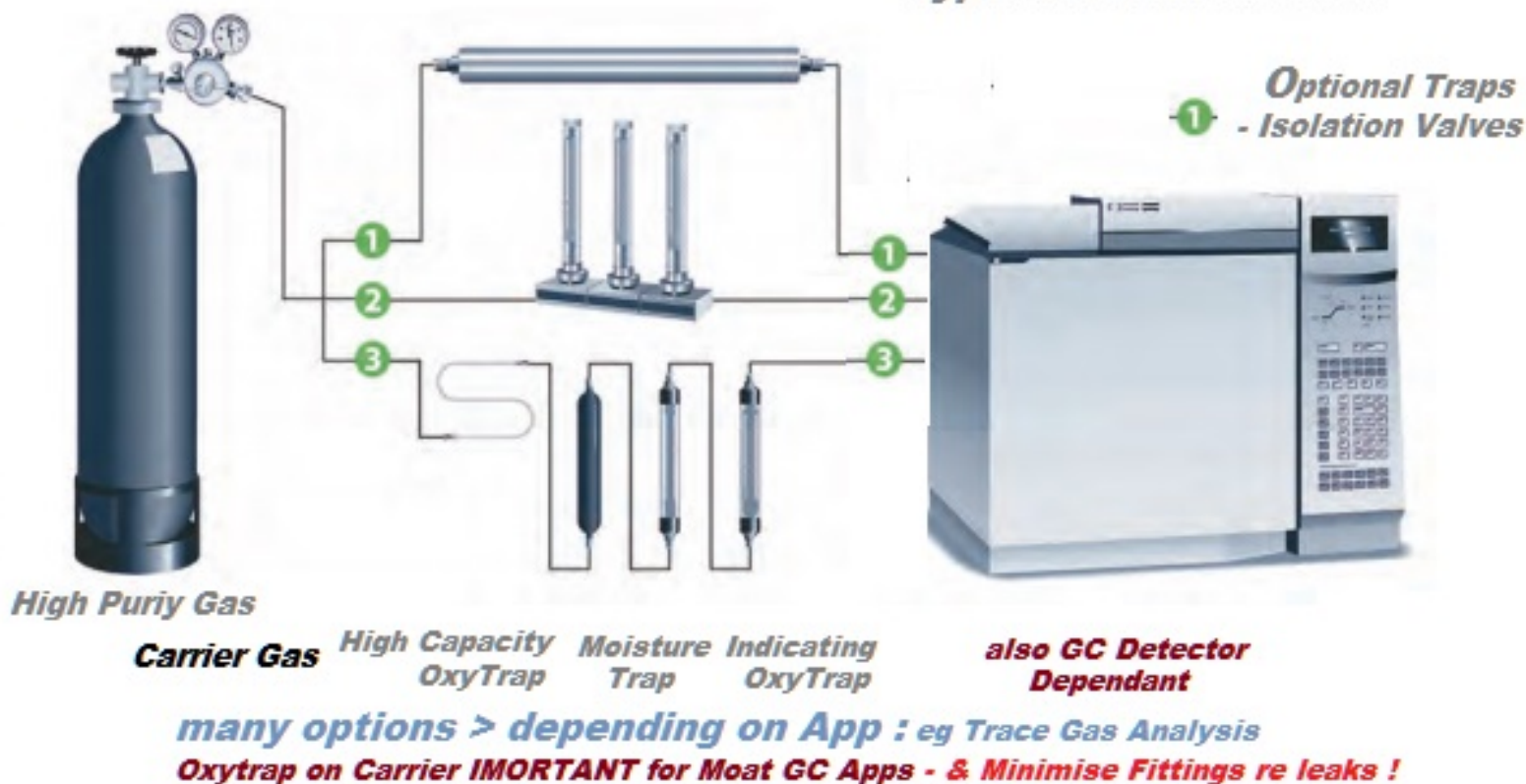
*more Robust Multi-use*

*Norm-Ject see PDF*

# Typical GC Installation [>PDF](#)

High Purity Gas Regulator

Typical GC Installation



## some Purity Specs

[see PDF](#)

## Gas Purification Hints

[see PDF](#)

**Gas Purity** for ANY instrumentation purpose IS Important !

> many compromises

- depends on the Gas type

**Analytical requirements**

GC Transfer Lines Ultra-clean Copper is required )

NOT for ECD ( use Silcosteel)

**Type of Detector** and sensitivity required

Trace Gas Analysis are ultra-sensitive vs macro gases

GC : TCD < FID < ECD & GC/MS

## Gas Samples

Reactivity of components and sampling device compatability

Syringes need to be Gas-Tight > time dependant

Inert for VOCs ( Glass / Teflon vs Polyprop for simple Air Analysis ?

**Process Control APPS**

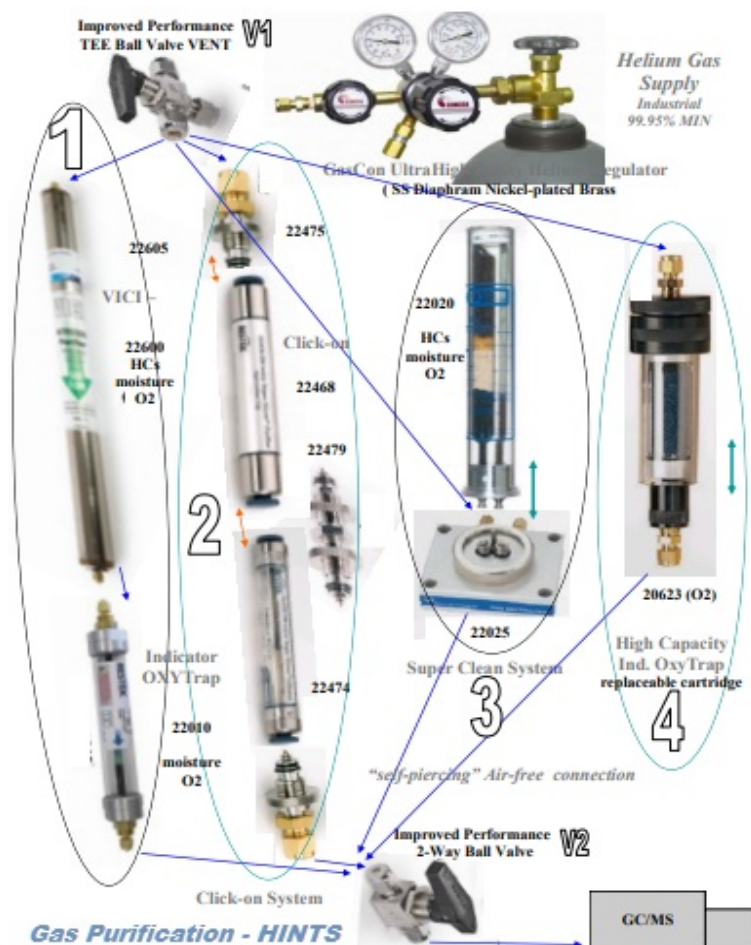
- Transfer Line dependant > even moisture in SS > Silcosteel required

> re response to stabilise !

**Reactive gases** can be especially problematic !

eg H<sub>2</sub>S i Natural Gas or Air (at <1ppm)

even CO<sub>2</sub> in "moist" air at trace levels !

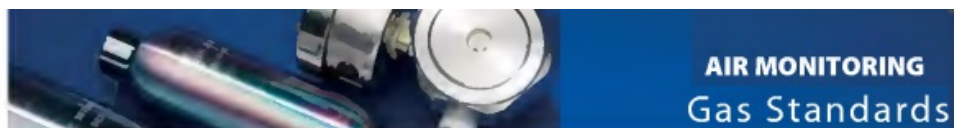




# Typical Gas Standards see Restek Catalog PDF

## Smoke Stack Plume Gas

- Crude standards can be made in a Syringe Transfer or by Gas Bag to ~10% accuracy?
- 18Liter - but can be conserved . . . with care !
- Use MinCyl Gas Regulator and Luer Adaptor



### Typical Refinery Gas - Type

Description	Shelf Life	Scotty 48 (48 Liter) cat.#
<b>Multi-Component Mixtures</b>		
Carbon monoxide, carbon dioxide, hydrogen and oxygen in nitrogen (0.5% each)	2 yrs.	34505
Carbon monoxide, carbon dioxide, hydrogen and oxygen in nitrogen (1% each)	2 yrs.	34508
Carbon monoxide, carbon dioxide, methane, ethane, ethylene and acetylene in nitrogen (1% each)	1 yr.	34511
Carbon monoxide, carbon dioxide, nitrogen, and oxygen, (5% each) and methane and hydrogen (4% each) in helium	2 yrs.	—
Carbon monoxide (7%), carbon dioxide (15%) and oxygen (5%) in nitrogen	2 yrs.	—
Carbon monoxide (7%), oxygen (4%), carbon dioxide (15%) and methane (4.5%) in nitrogen	2 yrs.	34516
C1-C6 n-Paraffins: methane, ethane, propane, butane, pentane, hexane in nitrogen (15ppm each)	2 yrs.	34529
C1-C6 n-Paraffins: methane, ethane, propane, butane, pentane, hexane in helium (100ppm each)	2 yrs.	34522
C1-C6 n-Paraffins: methane, ethane, propane, butane, pentane, hexane in helium (1000ppm each)	2 yrs.	34525
C1-C6 n-Paraffins: methane, ethane, propane, butane, pentane, hexane in nitrogen (100ppm each)	2 yrs.	34528
C2-C6 Olefins: ethylene, propylene, 1-butene, 1-pentene, 1-hexene in helium (100ppm each)	2 yrs.	34530
C2-C6 Olefins: ethylene, propylene, 1-butene, 1-pentene, 1-hexene in nitrogen (100ppm each)	2 yrs.	34532
Branched Paraffins: 2,2-dimethylbutane, 2,2-dimethylpropane, isobutane, 2-methylbutane, 2-methylpentane, 3-methylpentane in nitrogen (15ppm each)	2 yrs.	—
Methane, ethane, ethylene, acetylene, propane, propylene, n-butane, propyne in nitrogen (15ppm each)	1 yr.	34537
n-butane, isobutane, cis-2-butene, trans-2-butene, 1-butene, iso-butylene, 1,3-butadiene, ethyl acetylene in nitrogen (15ppm each)	1 yr.	34539

## Scotty® 48

Contents: 48 liters  
Pressure: 300psig (21 bar)  
Outlet Fitting: CGA 165  
Weight: 1.75 lbs/0.8 kg  
Dimensions:  
4" diameter x 16 1/4" height (10.2 x 41cm)  
DOT Specifications:  
39 NRC



### Regulators for use with 14-liter and 48-liter Scott Transportable Gases



### Syringe Adapter Kit for Single-Stage VOC Regulator



## VOC TO-14/15 Standards are far more diverse and multi-components for ambient Air Analysis

for use with FID, HID, ECD etc ultra sensitive GC Detectors,  
FID/Methaniser ( for ~10pM Limit of Detection ( 1cc sample injection)  
often at <100ppM - low ppB

Syringe and Gas Sampling bags are limited by adsorption and permeability problems

- glass OR
- Silcosteel-type Canisters are preferred
- cost depends on . . .

cylinder size, number of components & type, concentration  
accuracy required (within limits)

**Dangerous Goods Freight Charges apply for Import & Local Freight**

- **Custom Standards - Quotation required**

GreenHouse Gases

EPA TO-14

Chlorinated Hydrocarbons

S-Compounds

# some Bookmarks ( see Table of Contents )

