

Gas Chromatograph (GC) Operation in Hollister 152

Egidio Tentori (eft35), Richardson Lab – Oct 2018



A gas chromatograph (GC) is an instrument that is used to separate and measure gaseous compounds of interest by using a carrier gas to draw the sample gases past a stationary phase of composition that differentially binds compounds present in the injected sample. It can be used to detect compounds that volatilize at temperature of 250-300°C. Several factors affect the ability to separate, or "resolve" compounds, including: carrier gas flow rate, temperature, column type and length and the different in partition coefficients of compounds present in the sample.

Once the compounds are separated by the GC, they are detected as they elute from the column at their corresponding retention time. To correctly identify and measure a compound you need to know its retention time for the set GC conditions. The available GCs are equipped with a Flame Ionization Detector (FID) or a Thermal Conductivity Detector (TCD). GCs and corresponding computers are labeled 'FID' or 'TCD', make sure you know the differences in setting them up and that you are using the correct detector to measure your compound(s) of interest. Refer to 'Method Summary' section of this protocol for the operating conditions for each GC detector.

To turn on GC and set up for sample injection:

1. Check that the gas tanks needed for GC operation are not empty or running low, the gas tanks are to the left of the room as you enter. Open both the tank and regulator valves. If you are unsure which gases you need to operate the GC, refer to 'Method Summary' section of this protocol for the operating conditions for each GC detector.
2. Check that tank valve pressures are above 500 psi. If they are lower, keep in mind that the tank might need to be replaced soon and lower pressures (<250 psi) could affect your results.
3. Turn on the GC power switch located on the bottom right hand side near the back. After display screen on GC instrument shows 'EMULATION MODE OK', click on the 'TurboChrom' software on the computer to operate GC. Hit 'OK' to any prompts that appear. *(Gas Chromatography)*
4. Once the software is open, gently open the required gas switches for the gas lines that feed into the GC. They are located on the wall above each GC and are labeled by gas type.
5. Check that the individual pressure gauges which are located behind a panel on the left of the GC are rising, the column pressure on the front of the GC should also rise to ~30 psi.
6. On the 'TurboChrom' software click on 'Set Method', this is where the temperatures and other parameters of your method are set up. If your method is different than the set method, change the values. Now click 'OK'.
7. A screen will appear that shows the heaters and temperatures of key elements of the GC, the injector, column and detector. Here you will toggle the heaters for each these elements. Toggle the heaters for the injector, column and detector to 'ON' by clicking on each lever which turns them green, signaling they are now on.
8. Next the detector needs to be turned on, make sure GASES ARE ON, operating the GCs without gases flowing through them can cause damage to the detector. Click the 'Detector Status' button to 'ON', a prompt warning shows up and click 'OK gases are on'.
9. The GC elements now need to reach the set method temperatures. The 'OK' button will show up on this window once all the elements have reached the set temperatures. Alternatively, you can 'Click' cancel and check the temperatures on the GC instrument display by pressing the button for each element.
10. After temperatures are reached, now the GC detector baseline signal needs to stabilize before you begin sampling. Click 'Measure Sample' without injecting anything to bring up a dialog box and click 'OK'. The detector signal will show on the screen, if you click 'Stop Run' the measurement will end.

(Detector B)
for CH₄, N₂ & O₂

0-100

Stabilize
Signal

Wait if
not stabilized
✓ Baseline

11. If you are using the GC-TCD the baseline should be in the range of 5-40 which can take 15-60 minutes. If you are using the GC-FID you first need to ignite the detector, this is done by starting a run and then pressing and holding the 'FID Ignitor' button, located on the front top left of the GC-FID instrument, for about 3 seconds. This can take a few tries, once lit you should hear a small pop sound and the baseline should now be in the 5-20 range (the baseline for an unlit FID is in the 0-1 range). Once lit, the GC-FID baseline signal should take ~5 minutes to stabilize.
12. You can leave and come back periodically, clicking 'Measure Sample' again to check on the baseline signal.
13. Once you are satisfied with your baseline signal, you can begin your sampling by injecting your gas samples in the injection point at the top of the GC.

Repeat
fore each
sample

Record
or Results
"Copy
to Excel"

14. To inject your samples, first click 'Measure Sample' on the TurboChrome software, a prompt will appear to start your run. Now inject your sample in the injector port on the top of the GC. Now return to the computer and click 'OK' on the prompt to start measuring your sample.
15. After all your peaks have eluted for your sample, click 'STOP this RUN' and write down sample data (for example: retention times, peak areas, peak heights and baseline data) for your peaks of interest.

To shut down the GCs:

Log
Injections

Heaters
Turn
off everything

Quit

Turn
off GC power

off Toggle Switch

Tanks: Rightly tighty.
(Heater)

1. Log your number of injections on the GC Sign-in sheet which should be near each corresponding GC. Injection port septa need to be changed approximately every 100 injections, make note on the GC Sign-in sheet if the septa should be replaced before the next run. GCs have to be off in order to change septa as in injection port will be very hot during operation.
2. Click on 'Set Method' on the TurboChrome software. Click 'OK' to go to the window showing the heater status of the GC elements. Set all heaters and detectors to 'OFF' and click 'Cancel' to leave this window.
3. On the main software window click 'Quit', a prompt reminding you to turn off gas flows will appear, click 'OK' and program will close.
4. Turn off the GC power switch located on the bottom right hand side near the back.
5. Next, gently switch off the gas lines on the wall that feed into the GC.
6. Head to the gas tanks and close both the tank and regulator valves.

Method Summary:

GC TCD

Column type: Supelco 60/80 Mol Sieve 5A, 6ft x 1/8 in x 2.1 mm ss
Carrier gas: Helium
Method:
Temperatures
 Injector: 200°C
 Column: 30°C
 Detector: 250°C
Pressures
 Column pressure: 30 psi

GC FID

Column type: Supelco Custom Column 052300, 60/80 Carbopack B
Carrier gas: Nitrogen
Method:
Temperatures
 Injector: 200°C
 Column: 90°C
 Detector: 250°C
Pressures
 Column pressure: 30 psi

 Nitrogen: 50 psi
 Air: 40 psi
 Hydrogen: 20 psi

Troubleshooting:

Issue	Potential Solution
Something is wrong	Gases are off, check that your gases are on!
Poor column pressure	Gas leak through injector septa. Shut off GC and change septa.
Extraneous peaks	Contaminants or moisture present in column. Bake out column by turning on GC and setting column temperature to 300°C and leaving GC on for 1-2 hours. Proceed with regular method afterwards.
Peak shifts	
Odd peak shapes	
GC software unresponsive or error	Software turned on before GC instrument established communication with computer. Shut off GC and exit software and try again.
No peaks observed after injection (FID)	FID not ignited,