

GC Instructions

1. Turn on GC (external switch) and plug in the laptop computer with PeakSimple installed
2. Check that “carrier setpoint” is ~ 40 psi; if not, adjust the setscrew accordingly.
3. Helium carrier gas should be regulated to 15-20 psi higher than the setpoint, ~55 psi.
4. Detector temperature should be 125-130°C; if not, consult local expert.
5. BEFORE turning on the TCD detector (“under the hood”), check carrier gas flow at delivery tube outlet (inside the “oven”) by bubbles in water.
6. Switch TCD amplifier to LOW. (Lift red cover, look to the right of the oven box.)
7. Must close lid to operate, which is a safety feature.
8. To use “remote start” you must push and hold for ~1 sec. The green light of the channel will indicate when a sample is still running. The yellow light indicates “hold” status for the active channel(s).
9. When your lab is finished, turn off the TCD amplifier. Allow the oven to cool down to near room temp before turning it off.
10. Turn off GC and finally the carrier gas, both the regulator valve and the top tank valve.

Peak Simple

1. Run and change settings under Channel 1 via Edit/Channels menu. Deactivate any other channels. You may integrate directly in Channel 1 by checking the Integrate box.
 - a. Note: Channel 1 is labeled “FID” for flame ion detector even though our system is using a TCD or thermal conductivity detector.
 - b. Default components may be labeled and saved in a *.cpt file for later use:
Edit/Channels/Components/Load/ (e.g.) “dist.cpt.” You may also add your own component ranges or alter the current settings for start and end times for each peak. These times allow the software to identify what GC peak should be assigned to which species.
2. Select remote start box and use the start button on the GC simultaneous with aliquot injection (see Sample Injection below).
3. Set temperature for 185°C (T initial and final, Hold = 0, Range = 0) for basic operation; current run times should be ~10 minutes to reach the baseline for butanol (the last peak) with all above operating specifications.

Note: Lower start temperatures help separate the methanol and water peaks, but will increase total run time, followed by a temperature ramp strategically placed between peaks. {This is rather tedious to optimize during the lab time, as peak elution times shift with composition changes. I recommend this option only if you have spare time.}

4. Do not cut your runs short or the area normalizations under each peak will be wrong!

5. Click and drag the center or ends of the line intervals on the plot to include correct peaks for expected species; the default values are determined by the ranges loaded from the components file (above) or by adding your own component information (see #2).
6. Save chromatogram and immediately copy or analyze your results (see below).

Sample Injection

1. Run and watch baseline before injecting a sample to check for signal stability with carrier gas only.
2. Use ~1 μL with the 10- μL manual-injection syringe and accompanying needle.
3. Flush the syringe with your sample; eliminate air bubbles.
4. Auto-zero the baseline Channel 1 output; DO NOT FORGET to do this, or your integrations will not be accurate and you must repeat the run.
5. BE CAREFUL not to bend or break the needle while punching through septum!
6. Inject sample and quickly push GC "run" button or space bar on the computer to start the run.

Analyzing Results

1. Channel 1 can be used to integrate the peaks: activate, display, and integrate.
2. Set Integration reject to ~1; you may adjust this to exclude/include peaks by count size, which corresponds to area under the curve.
3. Load your components file for the alcohols into the Channel; while this is not necessary, it can be helpful by showing default ranges for each species peak.
4. Load your chromatogram data if it is not already shown, adjusting the windows for integration if necessary so the peaks are correctly labeled.
5. If the area peaks are not automatically labeled by the software when they appear, it means the component details have not been loaded or input correctly. You must do so, and then span the peak area by clicking and dragging the line intervals. (Read above.)
6. Peaks will shift in their elution time ranges depending on relative volume fractions and species in the sample. We expect water first followed by methanol, isopropanol and butanol, on a mass basis assumption only, which may not be true for water.
7. Area counts must be multiplied by the GC weights factors (in the Excel file on the desktop or under My Documents) and normalized to converted to mass fractions.
8. Manual integrations should be selected after the sample run is completed. Because there is almost always baseline drift. The button menu appearing at the left of the screen will allow you to compensate somewhat for the loss of peak area due to overlap or baseline drift. To alter the way the integration line (red) is measuring the area, you may have to "join" to peaks, for example, before dropping the red line to the horizontal and then splitting the peaks again. The instructor(s) will demonstrate this if/when needed.