Gas Chromatography

Gas Chromatography (GC) is a technique used to separate components of a mixture. GC can determine how many components a mixture has, the relative proportions of each component, and the identity of the components (especially when combined with mass spectroscopy). In CHM245 you will use both a Vernier Mini GC Plus or GoDirect Mini GC to separate compounds.

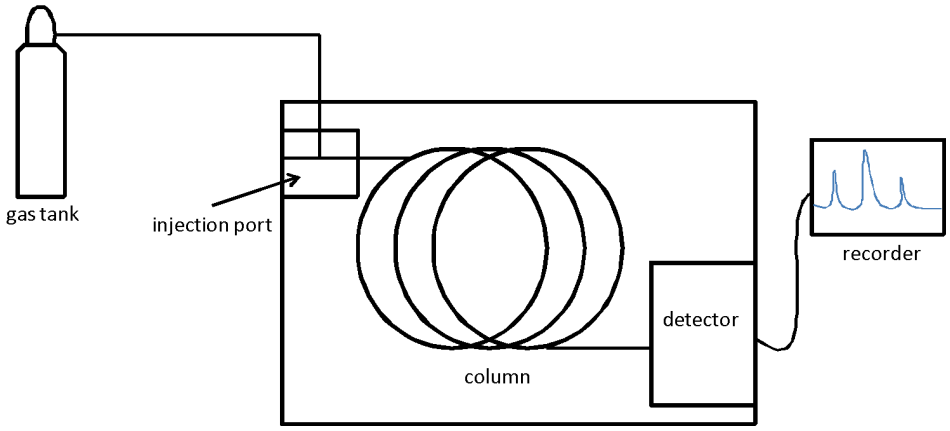




images: vernier.com

The Gas Chromatograph

The basic parts of a GC are the injection port, the column and the detector. The separation depends on the type of stationary phase inside the column, the rate of flow of the mobile phase, the temperature of the column and the length of the column. GC can only be used with compounds that can be vaporized and do not decompose in the gas phase.



<u>The Column</u>

GCs have a long metal column that contains the stationary phase. The stationary phase is a wax coating or other packing material. Different column compositions may be used depending on the polarity of the compounds.



GC columns can be over 15 meters and are coiled so they will fit inside the machine. The longer the column, the better the separation. The column temperature can be kept steady or gradually increased during an experiment. The temperature and gradient are adjusted based on the boiling point of the compounds to be separated. (image: https://sites.pitt.edu/~ceder/lab6/exp6text.html)

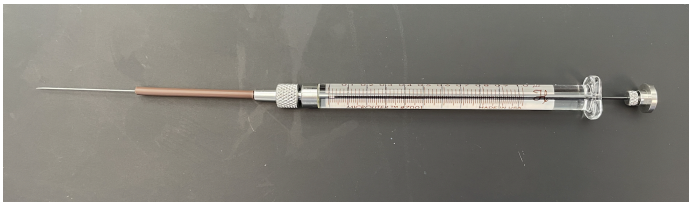
<u>The Carrier Gas</u>

The mobile phase in GC is an inert gas, usually helium or nitrogen. The mini GC uses air. The gas carries the sample through the column. The flow rate of the gas must be optimized for different types of samples. If the flow rate is too fast it may lead to poor separation.

Sample Injection

Typically 0.1 to 3 microliters of sample are injected into the GC. The sample is measured in a microsyringe. Once the sample is injected, it is vaporized at that injection port then gets carried by the inert gas to the column to be separated.

Microsyringes are expensive and delicate. Bending the needle or plunger will make it unusable.

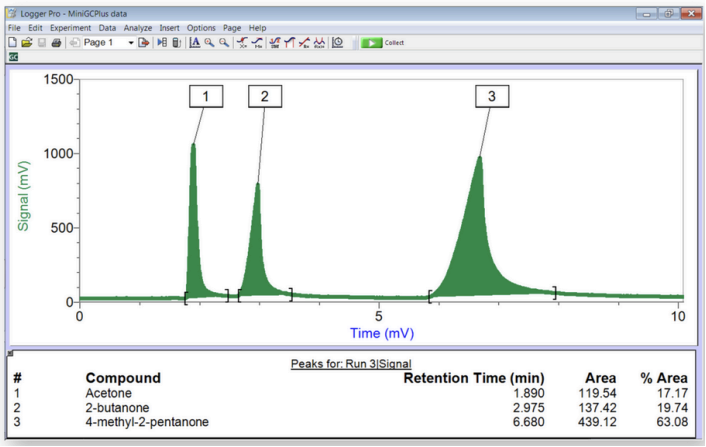


Retention Time

The time it takes for a compound to go from the injection port to the detector is known as the retention time. The retention time in GC is analogous to the R_f in TLC. Generally, higher boiling points will result in longer retention times. A compound will have a characteristic retention time under a set specific set of GC conditions (flow rate, column length, temperature, etc.) therefore compounds can be identified by their retention time.

The Detector and the Recorder

The detector senses different thermal conductivity between the carrier gas and the vaporized gas of the sample. When a component of the mixture passes the detector, this information is converted into an electric signal that is sent to the recorder. Peaks are plotted versus time and appear on the recorder as each component of the sample passes by the detector. The area under each peak is related to the amount of that chemical present in the mixture



Procedure for the Mini GC

**The syringe is fragile! Do not bend the needle or plunger*
**Do not pull the plunger back more than halfway (0.5 µL)*

Cleaning the syringe with acetone

- Submerge the needle tip in a vial of acetone. Pull back the plunger about ⅓ of the way.
- Remove the needle from the vial. Press the plunger to expel acetone on a kimwipe. You should see a tiny drop of the end of the needle if the syringe is working properly.
- Repeat two more times.

Injecting a sample. (image vernier.com)

- Follow the procedure above to flush the microsyringe with your sample.
- Submerge the needle in the vial of your sample again and pull up **0.2 µL.**
- Wipe the end of the needle with a kimwipe.
- The GC parameters will be set for you. Check that the GC has a green light and says "ready to inject"
- Hold the syringe vertically to inject. Guide the needle with one hand and the barrel with the other. Push the needle down into the port all the way to the brown bumper guard. If there is a lot of resistance, remove and try another area.
- Depress the plunger and press the collect button at the same time. (The button is on the Vernier screen for Mini GC plus and on the GC for Go Direct Mini GC)
- Gently pull up and remove the needle immediately

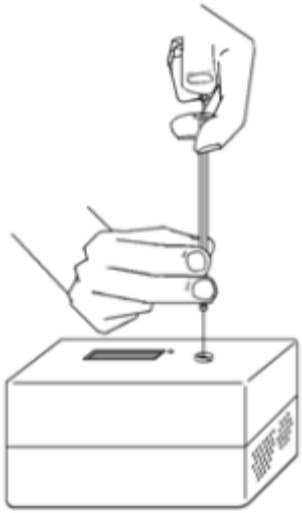


Figure 3

Analyze your sample

- When data collection has stopped, tap the menu for Analyze > Advanced > Peak Integration.
- Tap and drag from the left to the right side of each peak to integrate.
- Write the Retention time and area percent of each peak in your notebook.