

# Using a mini-Gas Chromatograph (GC): Identifying Unknown Compounds

There are many different types of chromatography: paper, thin layer, liquid, high-pressure liquid (HPLC) and gas (GC). Chromatography is applied in many fields. Biochemists use liquid chromatography to separate proteins; chemists use GC and HPLC to identify organic compounds. Technicians use GC for drug tests, toxin screens and environmental analysis. Many forensic tests involve chromatography.

All chromatography approaches operate under the same principles. There is a stationary phase and a mobile phase. The mobile phase travels along the stationary phase (in the column or on a plate) from a start point to an end point. Compounds can travel from the start to the end at different rates, depending on whether they tend to “stick” to the stationary phase or “float” in the mobile phase. Compounds stick to the stationary phase through dipole interactions, dispersion forces or ionic interactions.

The Vernier Mini GC uses a metal column with the inside of the column coated with the stationary phase. A sample, consisting of one or more compounds, is injected onto the column and is pushed through by air, which acts as the mobile phase. Organic compounds flowing out of the chromatography column are seen as a *peak* on a chromatograph, as seen in Figure 1. The amount of time it takes for a compound to exit the column after it is injected is called the *retention time*. With a GC, a compound can be identified from a mixture of chemicals by its retention time.

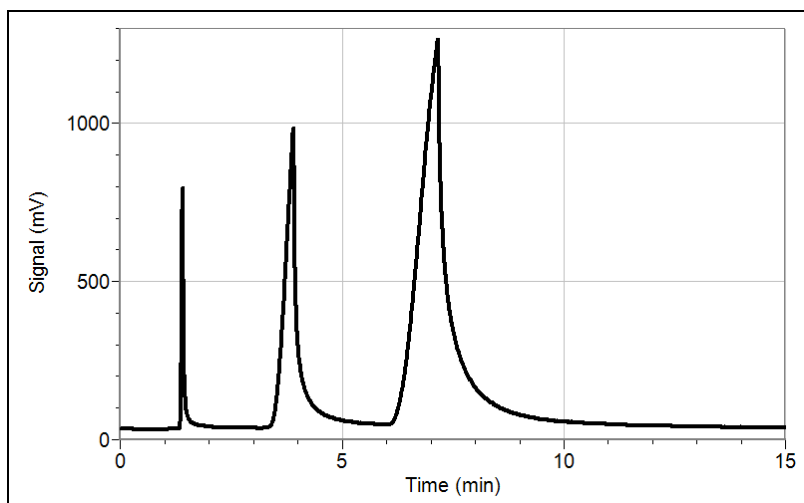


Figure 1: Sample gas chromatogram

Several factors can affect the interaction of a compound with the GC. More volatile compounds (i.e., compounds with a lower boiling point) tend to move through the column faster because they are flowing in the mobile phase and interacting very little with the stationary phase. The functional groups present on the compound are also a factor. For example, alcohols may interact with a polar stationary phase more than esters because alcohols can form stronger hydrogen bonds. The molecular weight of a compound can also play a role, although it is not a simple matter of saying that the heavier the molecule, the slower it will travel through a GC column.

## OBJECTIVES

In this experiment, you will

- Measure and analyze the retention time of five ketones and a known mixture of the ketones as they pass through a Vernier Mini GC.
- Measure and analyze the retention time of an unknown mixture of ketones.
- Identify the ketones present in an unknown mixture based on retention times.

## PRE-LAB EXERCISE

Complete the table below. Look up the boiling points for the ketones listed and predict the order in which these compounds will exit the GC column (known as *elution order*). Use the Merck Index reference book or chemspider.com. Rate the compound you think will exit the column first with the number 1; the last compound to exit the column is given the number 5.

Compound	Boiling point (°C)	Predicted elution order (1–5)
2-butanone		
2-hexanone		
4-methyl-2-pentanone		
acetone		
2-pentanone		

## PROCEDURE

**VERY IMPORTANT** – The glass syringe is designed so that the plunger is **NOT** meant to be pulled out of the syringe barrel!!! \*\*\*\*\*If the plunger is accidentally pulled out of the glass barrel, the syringe is no longer useable. \*\*\*\*\* Also, the glass syringe is fragile and can be easily damaged. Be careful not to bend the needle or bend the plunger.

### 1. How to prepare the Vernier Mini GC for data collection:

- a. Click Collect in Logger *Pro*, or tap ► in LabQuest, to bring up the Temperature-Pressure profile.
- b. Set the Temperature-Pressure values to:

Start temperature	35°C
Hold time	2 min
Ramp rate	5°C/min
Final temperature	55°C
Hold time	9 min
Total length	15.0 min
Pressure	5.0 kPa

- c. Select Done to initiate the Mini GC warm up. Note: A new message will appear, “Do not inject until GC is ready”, and the LED on the Mini GC is red. The Mini GC will take a few minutes to warm up and stabilize. When the Mini GC is ready for injection, the message will read, “Inject and select Collect simultaneously”, and the LED will turn to green. Continue with the next step during warm up.

## 2. How to clean the syringe with acetone:

**AGAIN:** The glass syringe is designed so that the plunger is NOT meant to be pulled out of the syringe barrel!!! \*\*\*\*\*If the plunger is accidentally pulled out of the glass barrel, the syringe is no longer useable. \*\*\*\*\*

**Important:** The glass syringe is fragile. Be careful not to bend the needle or bend the plunger. Never pull the plunger back more than 50% of its total volume. Be careful not to bend the plunger as you press it down.

- Depress the plunger fully.
- Submerge the tip of the syringe needle into the vial of acetone.
- Pull back the plunger to fill the barrel about 1/3 full of acetone. Examine the barrel of the syringe and estimate the amount of acetone in the barrel.
- Expel the liquid onto a Kimwipe or a paper towel.
- Repeat Steps a–d at least two times, until you are comfortable pulling up a liquid into the syringe and measuring the volume in the syringe barrel. Use a Kimwipe or a paper towel to carefully pat around the tip of the syringe needle.

## 3. How to collect a volume of 2-butanone for injection:

- Submerge the needle into the vial of 2-butanone.
- Draw up approximately 0.2  $\mu\text{L}$  of liquid. It is not critical that the volume be exactly 0.2  $\mu\text{L}$ ; a tiny bit more or less volume is all right.
- After collecting your sample, use a Kimwipe to gently wipe the needle from barrel to tip.

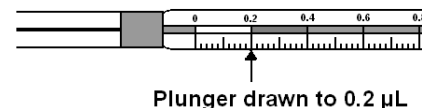


Figure 2

## Experiment 1

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### 4. How to prepare for injection and the start of data collection:

It is important for you and your lab partner to divide the tasks in this step. One person will operate the syringe and the other person will operate the computer controls.

- When the Mini GC has reached the correct start temperature and pressure, the message reads, "Inject and select Collect simultaneously," and the LED on the Mini GC is green.
- To insert the needle of the syringe into the injection port of the Mini GC, hold the syringe with one hand and steady the needle with your other hand. Insert the needle into the injection port until the needle stop is fully seated, as shown in Figure 3. If the needle sticks, rotate it slightly while inserting. Do not move the plunger yet.
- Simultaneously, depress the syringe plunger and select Collect to begin data collection. Pull the needle out of the injection port immediately.

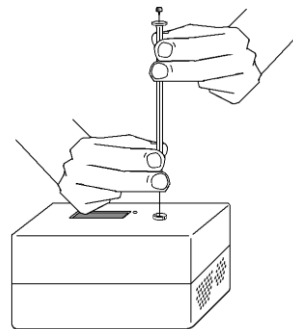


Figure 3

While the data collection proceeds, clean the syringe and needle with acetone. It may take more than three flushes to feel the syringe plunger move smoothly again, which is your indicator that the syringe and needle are both suitably clean.

Data collection will end after fifteen minutes. Make sure you go back to it and do the analysis.

### 5. How to analyze your results:

- Choose Peak Integration from the Analyze menu.
- Select and integrate the left-most peak. To do this, drag from a little before the peak to a point far enough to the right that includes all of the peak. Then choose Add.
- Record the retention time in your data table.
- Enter the name of the compound, if known.
- To analyze another peak on the same graph, repeat Steps b and c.
- When you are finished with all of the peaks, select OK.

(optional) You can choose to save this chromatogram and peak analysis for later use, with a unique file name, by choosing Save from the File menu.

**Repeat all the steps above (#1-5) to inject more ketones, the ketone mixture, and the unknown.**

**When you have completed your final data collection run, turn off the Mini GC.**

**DATA TABLE**

Compound	Boiling Point (°C) (from prelab)	Retention Time (min)
2-butanone		
2-hexanone		
4-methyl-2-pentanone		
acetone		
2-pentanone		

Unknown Mixture	Retention Time (min)
Peak 1	

**\*\*Sketch the chromatograms of the ketone mixture and your unknown mixture in your notebook.**

**POST-LAB QUESTIONS**

1. In the prelab, you predicted the elution order of the ketones. Did the chromatogram of the mixture support your prediction? Explain.
2. From the chromatograph of the known ketone mixture, measure the retention time for each peak in the chromatogram. Identify the ketone that produced each peak.
3. 2-hexanone and 4-methyl-2-pentanone are isomers, thus their molecular masses are equal. Suggest reasons for their differing boiling points and GC retention times.
4. Identify the ketones that are present in your unknown mixture. Support your identification.
5. Based on the results of this experiment, predict the retention time of diethyl ketone. Explain your prediction.