Organic Chemistry 211 Laboratory
Gas Chromatography

MATERIALS

Computer
Logger *Pro*
Vernier Mini GC
Temperature Probe
1 μL glass syringe

vials of:
ethyl acetate
butyl acetate
collected fractions from Exp. 5
Kimwipes® or paper towels

PROCEDURE

1. Obtain a glass syringe and three vials containing acetone, ethyl acetate, and butyl acetate. The acetone will be used only to clean the syringe. **CAUTION**: *Ethyl acetate and butyl acetate are both hazardous in case of ingestion or inhalation.*

2. Prepare the Vernier Mini GC for data collection.

   a. Turn on the Mini GC.
   b. Connect the Mini GC to the USB port on the laptop.
   c. Start the data-collection program, and then choose New from the File menu.
   d. To bring up the Temperature-Pressure profile, click Collect in Logger *Pro*.
   e. Set the Temperature-Pressure values to:

Start temperature	40°C
Hold time	1 min
Ramp rate	10°C/min
Final temperature	65°C
Hold time	5 min
Total length	8.5 min
Pressure	5.0 kPa

   f. Select Done to initiate the Mini GC warm up. When the Mini GC is ready for injection in Step 6, the message will read, "Inject and select Collect simultaneously", and the LED will turn to green. Continue with Step 3 during warm up.

3. Follow the steps below to clean and flush the syringe with acetone. **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.**

a. Depress the plunger fully.
b. Submerge the tip of the syringe needle into the vial of acetone.
c. 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.
d. Expel the liquid onto a Kimwipe or a paper towel.
e. 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.

4. Follow the process in Step 3 to clean and flush the syringe with ethyl acetate, the first sample to be injected into the Mini GC.

5. Collect a volume of ethyl acetate for injection.
   a. Submerge the needle into the vial of ethyl acetate one last time.
   b. Draw up 0.20 μL of liquid.
   c. After collecting your sample, gently wipe the needle, from barrel to tip, with a Kimwipe.

6. Prepare for injection and the start of data collection.
   a. The Mini GC should now have reached the correct start temperature and pressure, and the LED should be green.
   b. 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, as shown in Figure 3. Insert the needle into the injection port until the needle stop is fully seated. If the needle sticks, rotate it slightly while inserting. Do not move the plunger yet.
   c. Simultaneously, depress the syringe plunger and select Collect to begin data collection. Pull the needle out of the injection port immediately.

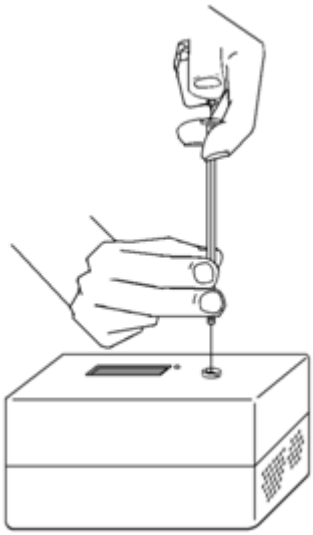

Figure 3

7. While the data collection proceeds, repeat Step 3 to thoroughly clean the syringe and needle. 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.

8. Data collection will end after 8.5 minutes. Observe the graphed data that characterize an ethyl acetate chromatogram.

9. Analyze your chromatogram.
   a. Choose Peak Integration from the Analyze menu.
   b. Select the left-most peak. To do this, drag from a little before the peak to a point far enough to the right so that the entire peak is included. Choose Add.
   c. Record the retention time and the peak area in your data table.
   d. Click Cancel to return to the graph.

10. Label and store your run of data:
    - In Logger *Pro*, double-click Latest, which is the title of your run of data in the table, to the left of the graph. In the Data Set Options dialog box, type in the name of your compound. Click OK. Choose Store Latest Run from the Experiment menu to store your chromatogram.

11. Prepare the butyl acetate sample.
    a. Click Collect in Logger *Pro* to bring up the Temperature-Pressure profile. This profile will be the same as for your previous run. If you are satisfied with these values, click Done.
    b. While the Mini GC adjusts to its Temperature-Pressure profile, repeat Steps 4 and 5 with the butyl acetate sample.
    c. After the Mini GC is ready, repeat Steps 6–10.

12. Repeat Step 11 for the ethyl acetate/butyl acetate mixture and the fractions you collected from the distillation in Part I.

13. After you have completed the test of the mixture, save your file for use at a later time. Save the file as directed by your instructor.

14. Flush and clean the syringe with acetone.

15. Turn off the Mini GC. Keep your test results open in Logger *Pro*; you will need to refer to the various chromatograms to answer the Data Analysis questions.

DATA TABLE

Compound	Retention time (min)	Peak area
Ethyl acetate (EtOAc)		
Butyl acetate (BuOAc)		
Original EtOAc/BuOAc mixture		

Recorded from last experiment

Fraction	Temperature range (°C)	Volume collected (drops or mL)

Simple 1st		
Simple 2nd		
Simple 3rd		
Fractional 1st		
Fractional 2nd		
Fractional 3rd		

Analysis of the Chromatograms

	Peak area EtOAc	Peak area BuOAc	% Peak Area EtOAc	% Peak Area BuOAc
Original EtOAc/BuOAc mixture				
Simple 1st fraction				
Simple 2nd fraction				
Simple 3rd fraction				
Fractional 1st fraction				
Fractional 2nd fraction				
Fractional 3rd fraction				

FOR YOUR REPORT

- Discuss the theory of the GC technique in details.
- Make a Simple Distillation graph of % Peak Area (y) vs. Temperature (x) for both EtOAc and BuOAc on the same graph.
- Make a similar graph for Fractional Distillation.
- Explain how and why the two graphs are different.

- How well did your fractionating column separate the chemicals? What could you change to achieve better separation?
- Other discussions.