Purpose

You will use gas chromatography to separate, identify, and determine the concentrations of the components of a mixture of up to three hydrocarbons.

Equipment

- Perkin-Elmer Autosystem GC, with integrator and printer
- Hamilton syringes, 10 μL

GC operating conditions

- Packed column
- Carrier gas: Helium, 30 mL/min flow rate
- Oven: 200 °C, isothermal
- Detector: Thermal conductivity detector (TCD); operating temperature 170 °C
- Injection: 2 μL injection size, injection port temperature 200 °C
- Run time: 4 minutes

Chemicals required

- Pentane (Density @ room temperature: 0.6262 g/mL; Boiling point: 36.1 °C)
- Heptane (Density @ room temperature: 0.6795 g/mL; Boiling point: 98.42 °C)
- Octane (Density @ room temperature: 0.7050 g/mL; Boiling point: 125.52 °C)

Theoretical

See the two GC handouts available on the course web site, http://scienceattech.com, for all background information on this experiment.

Procedure

Setting up the instrument

If necessary, assist your instructor with setup of the Perkin-Elmer Autosystem GC:

Initial setup

1) Turn on the equipment
   - Turn on the helium gas at the cylinder. Outlet pressure at the helium cylinder should be approximately equal to 90 psig.
   - Turn on the instrument, printer, and integrator (computer).
2) Verify gas flow
   - Attach the tubing of the soap-bubble flow meter to the reference TCD outlet. (The
reference outlet is the upper port on the TCD.)
• Access the internal stopwatch by pressing [System][System][Enter].
• Enter a volume of 10 mL.
• Squeeze the bulb of the flowmeter to release a bubble.
• Time the progress of the bubble through two divisions of the bubble flowmeter (each division represents 5 mL). Start the stopwatch by pressing [Enter], and stop it by pressing [Enter] again.
• When you stop the stopwatch, the display will indicate the flow rate. If the rate is not 30 mL/min, adjust the flow rate valve and measure the flow rate again. The timer may be reset by pressing [Enter].
• Repeat the previous steps for the analytical outlet. (The analytical outlet is the lower port on the TCD.)
• If the flow rates are more than 20% different, then adjust the gas flow through the reference channel by turning the [TCD Ref Flow] knob on the top of the instrument until the flow is within +/- 20% of the flow in the analytical channel. For example, if the analytical channel has a flow rate of 30 mL/min, the reference channel may have a flow rate ranging from 24 mL/min to 36 mL/min.

**Oven setup**

1) Set oven temperature
   • Press [Oven Temp]. “Oven TEMP 1” appears on the display.
   • Enter 90 (90 °C is our desired temperature) using the number keys.
2) Set the run time
   • Press [Time]. “Oven TIME 1” appears on the display.
   • Enter 4 (the run time in minutes).
   • On the integrator (computer) make sure the run time is displayed as 4 minutes.
3) Make sure the rate is set to zero. (This tells the instrument to keep the oven at the same temperature throughout the experiment)
   • Press [Rate]. “Oven RATE 1” appears on the display. Set a rate of “0” with the number keys. (After you hit [Enter], the display will say “End” instead of “0”. That's normal.)

**Thermal conductivity detector (TCD) setup**

1) Set the detector temperature.
   • Press [Det Temp] twice. The display should read “TCD 2 Temperature”.
   • Enter 170 (170 °C is the desired temperature for this experiment) with the number keys, then press [Enter].
   • Wait for “NOT RDY” to disappear from the display. This will happen when the temperature inside the detector has stabilized.
2) Activate the detector.
   • Press [Range] twice. “TCD 2 Range” should appear in the display. Enter “1” to apply current to the filaments in the detector. (You can enter “0” to turn off the
current to the detector. DO NOT ENTER ANY NUMBERS OTHER THAN “0” OR “1” WITHOUT PRIOR AUTHORIZATION FROM MR. TAYLOR. ENTERING A LARGE NUMBER HERE CAN DESTROY THE TCD!

3) Zero the detector.
   • Press [Autozero] twice. The display should read “TCD 2 A/Z”. Wait about 20 minutes.
   • Verify that the voltage reading on the display is between 0.0 mV and 20.0 mV. If it isn't, then adjust the TCD Balancing Potentiometer knob (near the TCD, labeled “Channel 2”) until the display reads between 0.0 mV and 20.0 mV.

Injector setup

1) Set the injector temperature (200 °C for this experiment)
   • Press [Inj Temp][Inj Temp]200[Enter]. “Pkd 2” should appear on the display.
2) Allow the injection port to come to operating temperature.
   • Press [Status Escape] and wait for "READY" to appear.

Analysis of samples

You will need to obtain chromatograms for five different samples:
   • Pure pentane
   • Pure heptane
   • Pure octane
   • A 1:1:1 mixture of pentane, heptane, and octane
   • An unknown mixture of up to three of the above hydrocarbons

1) Verify that the instrument is ready to acquire data
   • Press [Status Escape].
   • Wait until the GC display reads “READY”.
2) Load the 10 μL Hamilton syringe
   • Rinse the syringe several times with sample by completely filling the syringe with sample, then squirting this sample into a waste container.
   • Load 5 μL of sample into your syringe, making sure there are no visible air bubbles inside the syringe.
3) Begin your run
   • Inject the sample into the packed column injector port and press [Run] immediately. The integrator (computer) will automatically begin collecting data.
   • After the run, the integrator will automatically print out the chromatogram. Tear it off and immediately write (on the printout itself) which sample was run.

Repeat the three steps above until all five samples have been analyzed.
Analysis of data

Data collection

Your first three samples were pure hydrocarbons. Each of these should have only one prominent peak. On the data sheets, write down the retention times of the peaks corresponding to pentane, heptane, and octane.

The 1:1:1 mixture contains all three hydrocarbons. Write the retention times and peak areas of the major peaks of the 1:1:1 mixture. Identify the peaks as pentane, heptane, or octane. Mark the identity of each peak on both the data sheets and the chromatogram itself. Repeat the above for the unknown mixture.

Calculations for the 1:1:1 mixture

Calculate the theoretical mass percentage of each hydrocarbon in the 1:1:1 mixture using the density information given at the beginning of this handout. Assume that the 1:1:1 solution was made by mixing 1.00 mL of each hydrocarbon together. (Hint: Convert 1 mL of each substance to mass, then add all three masses together to get the weight of the mixture.)

Instrument response should be similar for all three hydrocarbons tested, and response depends on the amount of material passing through the detector. Calculate the experimental mass percentage of each hydrocarbon from your chromatogram by dividing the area of one major peak by the total area of all major peaks. For example:

\[
\%\text{pentane} = \frac{\text{pentane peak area}}{\text{pentane peak area} + \text{heptane peak area} + \text{octane peak area}} \times 100\%
\]

Calculate the percent error for each hydrocarbon.

\[
\%\text{error} = \left| \frac{\text{expected value} - \text{experimental value}}{\text{expected value}} \right| \times 100\%
\]

Calculations for the unknown mixture

Calculate the experimental mass percentage of each hydrocarbon from your chromatogram by dividing the area of one major peak by the total area of all major peaks, as you did for the 1:1:1 mixture.

Fill in the data sheets on the following pages, answer the questions, and turn pages 5 through 8 - along with the chromatograms from the instrument - in to your instructor.
CHM 111: Gas Chromatography: Separation of a three-component mixture (r10)

**Group information::**

<table>
<thead>
<tr>
<th>Member #1</th>
<th>Member #2</th>
<th>Member #3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Write the date the experiment was performed in the box below.

**Data**

**Chromatograms of pure hydrocarbons**

<table>
<thead>
<tr>
<th>Hydrocarbon</th>
<th>Retention time (min)</th>
<th>Peak area (arbitrary units)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PENTANE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HEPTANE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>OCTANE</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Chromatogram of 1:1:1 mixture**

<table>
<thead>
<tr>
<th>Hydrocarbon</th>
<th>Retention time (min)</th>
<th>Peak area (arbitrary units)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PENTANE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HEPTANE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>OCTANE</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Chromatogram of unknown**

<table>
<thead>
<tr>
<th>Hydrocarbon</th>
<th>Retention time (min)</th>
<th>Peak area (arbitrary units)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PENTANE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HEPTANE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>OCTANE</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Calculations for 1:1:1 mixture

<table>
<thead>
<tr>
<th>Hydrocarbon</th>
<th>Theoretical mass %*</th>
<th>Experimental mass %*</th>
<th>Percent error</th>
</tr>
</thead>
<tbody>
<tr>
<td>PENTANE</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HEPTANE</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>OCTANE</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Report these mass percentages to the nearest hundredth of a percent. Ex: 21.42%

**Sample calculation:** In the space below, show how you calculated the theoretical mass percent, experimental mass percent, and percent error for PENTANE.
Calculations for unknown mixture

<table>
<thead>
<tr>
<th>Hydrocarbon*</th>
<th>Experimental mass %**</th>
</tr>
</thead>
<tbody>
<tr>
<td>PENTANE</td>
<td></td>
</tr>
<tr>
<td>HEPTANE</td>
<td></td>
</tr>
<tr>
<td>OCTANE</td>
<td></td>
</tr>
</tbody>
</table>

*If no peak was observed for a particular hydrocarbon, leave the row blank.
**Report mass percentages to the nearest hundredth of a percent. Ex: 21.42%

Sample calculation: In the space below, show how you calculated the experimental mass percent, for PENTANE***

***If no peak was observed for PENTANE, show a sample calculation for HEPTANE or OCTANE instead.
Questions

1) How do the retention times of the hydrocarbons in today's lab relate to their boiling points?

2) $n$-Hexane is a hydrocarbon that is similar in structure to the hydrocarbons tested in today's lab. It has a boiling point of 69 °C. Estimate its retention time under the conditions used in today's experiment.

3) If you mistakenly injected less sample than you anticipated (due to your syringe containing an air bubble), what would happen to the observed peak areas for each peak? What would happen to the retention times?

4) Today's experiment was run at an oven temperature of 90 °C. If the oven temperature was raised to 110 °C, what would happen to the observed retention times for each hydrocarbon peak?

5) Today's experiment was run with a carrier gas flow rate of approximately 30 mL/min. If the flow rate was changed to 20 mL/min, what would happen to the observed retention times for each hydrocarbon peak?