CHEM450 Experiment: Gas Chromatography/Mass Spectrometry (GC/MS)
Analysis of Citrus Fruit Extracts
(Modified from Chem. Educator, 2001, 6, 28-31)

PRELAB:
Know the answer the following questions before performing the lab.
1. What are the stationary phase and mobile phase in this gas chromatography experiment?
2. Which would you expect to elute from a GC column first, propane or hexane? Why?

PROCEDURE:
Extraction:
This experiment has been simplified by skipping the extraction process and using “generic” citrus fruit extracts in hexane.

GC/MS Analysis 1:
1. Obtain a set of unknown citrus extracts (dissolved in hexane) and a set of known citrus components (camphene, myrcene, limonene and linalool, also dissolved in hexane) from your lab instructor.
2. Using the parameters recommended by your instructor, analyze each known and unknown on the GC/MS. Make sure that you print the chromatogram from each run, as well as the mass spectrum of each component per unknown. Label each of the printouts. This will be used later for identification.
3. Using the retention time and mass spectra, identify the components of each of your unknowns by comparison with retention time and mass spectra of the known components. Which peak corresponds to the solvent hexane? Why?

GC/MS Analysis 2:
In this part of the experiment you will come up with a set GC parameters that will result to at least a baseline resolution of each of the components of one of your unknowns while minimizing analysis time. You may do this by looking at the GC parameters above and devising and executing a plan to accomplish the goals of this part of the experiment. However, you may not do more than 3 variations of the GC parameters.

DATA and RESULTS:
You must have the following information in the Data and Analysis section of your lab report.

<table>
<thead>
<tr>
<th>Instrument Model:</th>
<th>Column Type:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carrier gas pressure (from regulator):</td>
<td>Column length:</td>
</tr>
<tr>
<td>Oven temperature program:</td>
<td></td>
</tr>
<tr>
<td>Injector temperature:</td>
<td>Detector temperature:</td>
</tr>
<tr>
<td>Detector type:</td>
<td></td>
</tr>
<tr>
<td>Unknown #s:</td>
<td></td>
</tr>
</tbody>
</table>

Tabulate your data by including (1) the retention time of each component of the unknowns as well as that of the known components and (2) major peaks in their mass spectrum which helped you identify the components of your unknown citrus extracts. (3) Also include in your table the structure of each component, which can be found in the literature.

Questions: Incorporate the answers to the following questions in your DISCUSSION.
1. Look up the boiling point of hexane, camphene, myrcene, limonene and linalool. Compare these boiling points to the retention times presented in your table. Does the order of elution (value of the retention times) make sense relative to the boiling points? Explain.

2. How does the mass spectrum tell you what compound a given GC peak corresponds to?