GAS CHROMATOGRAPHY / MASS SPECTROMETRY

This experiment will serve as an introduction to the "hyphenated" technique of gas chromatography/mass spectrometry (GC-MS). The resolution that is available using an open-tubular (capillary) GC column coupled with the structural information that is offered by a mass spectrometric detector makes GC-MS a powerful technique for the analysis of complex mixtures. GC-MS is used extensively in food, drug, and pesticide analysis.

In Part A of the experiment, familiarity with the basic operation of the instrument will be gained and the precision of a GC-MS analysis will be studied. In Part B, the quantitative analysis of acetic acid in a food sample will be performed by GC-MS using the standard addition method. Finally, a student-chosen and prepared mixture of compounds will be analyzed.

General Information and Procedures:

GC column:	Hewlett-Packard FFAP open-tubular column, 0.2 mm inner column diameter (FFAP is a polyethylene glycol stationary phase.)
MS lower mass limit:	mass/charge = 50
Injection Port Temp:	275°C
Solutions Provided:	1000 ppm acetic acid in water 10,000 ppm acetic acid in water

Some Basic Operating Procedures:

START-UP PROCEDURE

1. Turn on the computer, monitor, data transfer module and the printer (if they are not already on)
2. Remove the MS detector from STAND-BY and engage the pumping unit and heater.
3. Turn on the gauge controller - depress the power and degas buttons simultaneously. Do the same for the filament button. Both the filament and degas buttons should be lit. The gauge should show a pressure of less than 10×10^{-5} torr.
4. You are now ready to prepare to make a run.

It is impractical to give an exhaustive description of the software commands used in this experiment. Instead, some commands and their descriptions used in a typical run are listed below. Commands that are soft key choices are in ALL CAPS.

1. DATA ACQUISITION - gets you into data acquisition mode
2. EDIT - pulls up choices for changing experiment parameters including: TEMP. PROFILES and RUN PARAMETERS.
3. Important: after changing parameters and using EDIT, these new parameters must be stored (use STORE PARAMETERS) and loaded (use LOAD PARAMETERS).
4. PREP TO INJECT - prepare to inject a sample. You must choose a data file in which to store data from the run.
5. After temperature equilibrium is reached, inject and press GO.
6. After run is complete, QUIT and select DATA EDITOR.
7. View the TOTAL ION CHROMATOGRAM (TIC), and view the SPECTRUM at any point in the chromatogram.
8. INTEGRATE can be assessed through the CHROMATIGRAPHIC KEYS command.

9. "Print Screen" can be used to obtain a hard copy of data, temperature profiles, and integration results.

Part A - Precision

Operating Conditions:

Column Temp. Profile:	Initial temp. = 110°C, 4 min. hold
	Ramp at 15°C/min to 140°C
	Hold at 140°C for 2 min
Solvent Delay:	4 min.
Sample:	1000 ppm acetic acid in water

1. Inject 1.0 μL of the 1000 ppm acetic acid solution and observe the chromatogram. Locate the acetic acid peak and print the total ion chromatograph with the mass spectrum of acetic acid. Integrate the acetic acid peak and print the result.

2. Repeat this procedure four more times. The same person should make all five injections while other members of the group take turns at the GC-MS keyboard.

3. In your report, tabulate the retention times and peak areas for the five injections. Perform the Q-test on your retention time and peak area data to check for discordant values that should be discarded. Use critical Q-values at the 90% probability level for comparison with your experimental Q-values. (See your Quant book or R. B. Dean and W. J. Dixon, Anal. Chem. 23, 636 (1951)). Show one of you Q-test calculations and explain your decision to keep or reject the data points. With the remaining data, calculate and report the mean, the range, the standard deviation, and the percent relative standard deviation for the retention times and peak areas. Comment on the performance of the method.

PART B - Quantitative Analysis of Acetic Acid in a Food Sample

Operating Conditions:

Column Temp. Profile:	Initial temp: 105°C, 4 min. hold.
	Ramp at 15°C/ min to 145°C
	Hold at 145°C for 2 minutes
Sample:	Supernatant from commercial dill pickles.
	Dilute by taking 1 mL and diluting to 10 mL.
Injection Volume:	1.0 μL throughout

1. Devise and carry out the quantitative determination of acetic acid by the standard addition method. You will want to start by obtaining a "ball park" figure for the acetic acid in the sample and devise your experiment on that basis. Plan carefully!
2. Explain the procedure that you used, including volume and concentration, to obtain your "first" standard addition point. Show the calculation for "added concentration".
3. Tabulate "added concentration" and peak area for each run. Graphically determine (show your graph) and report (in ppm) the concentration of acetic acid in the sample. Discuss sources of error.

PART C - Separation of A Mixture:

1. Choose, prepare and separate a mixture of compounds. Choose a sample designed to illustrate some basic principle about GC-MS, i.e. make and test a hypothesis! One sample per group will suffice.

   Things to keep in mind:
   - the concentration of sample components should be less that 1%, in a volatile solvent
   - the nature of the FFAP column
   - the finite nature of time: design you mixture to be elegant but simple
   - get instructor approval

2. Identify all peaks, and record and interpret mass spectrum.

3. Do your experimental data support your hypothesis? Discuss what your experiment has illustrated.