Directions for using Agilent GC (6890) with MSD (5973)

If the computer isn't on, log-in with username "Administrator" and password "administrator". Check to make sure the gas line is open and set to at least 20 psi. You may need to adjust the pressure later to satisfy the pressure needed by your method.

From the desktop, double-click "1 GCMS" icon. Three windows will open – leave all three open. We will assume that the MSD is already pumped down and ready. If it is not, please see your instructor or Dr. Kramer for help.

Tuning the instrument

If it has been more than 24 hours since the instrument has been tuned, you need to perform a quick tune to confirm the settings.

1. In the "Instrument Control" window, choose "View – Manual Tune."
2. In the "Manual Tune" window, choose "Tune – Quick Tune."
3. The MSD will click on and perform a scan of three ions from a standard stored in the instrument. It will adjust to make the peaks as narrow as possible and as accurate as possible.
4. If the scan completes, you are ready to move on. If it does not, please see your instructor.
5. Go to "File – Save Tune Values" to save the tune. Choose "atune.u" for the file name.

Setting up a method

In order to run the instrument, you will set up a method in Chemstation.

1. If you are not in the "Instrument Control" window, open it by choosing "View – Instrument Control."
2. If you plan to use a stored method, choose "Method – Load" and choose your method. If you plan to start a new method, choose "Method – Edit Entire Method."
3. In the "Edit Method" window, make sure that only "Method Information" and "Instrument/Acquisition" are selected. Select "OK."
4. In the "Method Information" window, enter any comments about the method and make sure that only "Data Acquisition" is selected. Select "OK."
5. In the "Inlet and Injection Parameters" window, make sure that "GC" is chosen as the inlet, "Manual" is chosen as the injection source (unless an auto-injector is to be used), "Front" is chosen as the injection location and an "x" is in the box next to "Use MS." Select "OK."
6. Now you will edit each step of the analysis. These parameters can also be edited individually later by choosing the icons on the "Instrument Control" screen. The parameters you will most likely want to edit are:
    a. Inlets: Two options are available, split or splitless. For a "Split" run, choose "Split" and set the ratio below. For a "Splitless" run, choose "Splitless." On this screen you should set the heater temperature, type of carrier gas, pressure and total flow for the inlet.

b. Column: For Mode, choose "Constant Flow." For Inlet, choose "Front." For Detector, choose "MSD."
c. Oven: On this screen, you can set the starting temperature and any temperature gradient using the table of times and temperatures. Any changes you make will be expressed in the graph of temperatures at the top of the screen. Make sure that your temperatures will not go over the "Oven Max" chosen on this screen.
d. Aux: This refers to the MSD. On this screen you should set the temperature for the transfer line to the MSD.

7. After editing all of the instrument parameters, select "OK."
8. In the "GC Real time plot" window, make sure that no signals are selected. These would only apply if you were using a different GC detector. Select "OK."
9. In the next window, make sure to select "atune.u" or the name of the tune file you have saved previously.
10. In the "MS SIM/Scan Parameters" window, you can set up the parameters for the mass spectrometer. The instrument can be run in "Scan" mode for a total ion chromatogram (TIC) or in "SIM" mode for selected ion monitoring.
    a. Set the solvent delay. Three minutes is usually sufficient. This delay keeps the MSD from turning on until after the solvent in a liquid sample has passed. If you are injecting a gaseous sample, it is unnecessary.
    b. Scan: If you would like to adjust the range of m/z ratios that the instrument will monitor or the amount of time it will spend on each m/z, do so in the table at the bottom of the screen. Most likely, you will not need to adjust this table.
    c. SIM: If you know the compounds you will be detecting and have their mass spectra, you may use this screen to choose specific ions to be detected at different times during the run.
11. After selecting "OK," you will be given a place to save your method. Type in a method name. All names used in this program must be 8 characters or less.

Running a Method

Make sure your method is loaded.

1. Choose "Method – Run" in the "Instrument Control" window.
2. Choose a name for your file. To find a list of files and folders, type "?" in the "File name" box.
3. Fill in the rest of the boxes with information about your run.
4. Choose "Run Method" (not "OK") to start the method.
5. When "GC Ready" window appears, prepare your sample to be injected. See your instructor for information about appropriate volumes used. Make sure to get rid of any bubbles in the syringe.
6. On the GC itself, press the "Prep run" button. When the "pre-run" light turns on, quickly inject your sample by piercing the septum in the front inlet and pressing the plunger of the syringe. **Immediately** press the "Start" button on the GC.
7. **Do not choose** "override solvent delay" unless you are injecting a gas into the GC. Overriding the solvent delay when injecting a liquid sample will greatly reduce the life expectancy of the detector. Ignore the window.

8. Your chromatogram will appear in the "Total Ion" window. Double click on it to expand the window.

Data Analysis

During a run, from the "MS Top/Enhanced" window, choose "Open New Data Analysis." Otherwise, open a data analysis window by choosing "View – Data Analysis (offline)."

1. If you would like to analyze your chromatogram as it is being collected, choose "Take Snapshot" under the "File" menu. Otherwise, choose "Load Data File" under the "File" menu.
2. To display the mass spectrum at any point in a chromatogram, double **right** click on a point in the chromatogram. The spectrum will be displayed below. You may also average several points together by drawing a rectangle with the **right** mouse button clicked.
3. To search the library for a given spectrum:
    a. Choose "Spectrum – Select Library."
    b. Type "?" in one of the boxes. Scroll down to choose "Wiley275.L" as the library to search.
    c. Double **right** click on a spectrum and a list of potential library matches will be displayed along with the probability of a match ("Qual" column – nearer to 100 is better). To find out more information about a certain choice, highlight the compound and then click the "Text" button.
4. To determine the area of the peaks in the chromatogram:
    a. Select "Chromatogram – Integrate." The retention time of the peaks will be listed next to each peak.
    b. If too many or too few peaks are chosen, go to "Chromatogram – Select Integrator." Choose "RTE Integrator." Choose "Chromatogram – MS Signal Integrator Parameters" and change the minimum peak area detected (increase if too many peaks are chosen, decrease if too few were chosen). Integrate again by selecting "Chromatogram – Integrate."
    c. To display the areas of each peak, choose "Chromatogram – Integration Results."