Instructions for HP mass spectrometer

Unless otherwise noted, all clicks use the left hand button on mouse. The mass spectrum is run with windows software.

1. Turn on computer and screen. When the cow in space screen appears, double click on **HP Chem Station** Icon

2. In **Utilities Menu**, choose **Start**, and click on only choice, **5899**, then click **OK**.

3. The Window should say 5988 Top. In the **Method Menu**, choose **Load and Run Method**. Scroll through methods, and choose **Supscan.m.** Click **OK**.

4. In the **Start Run window**, type in your name, same name and any misc. information you want. Click **OK**.

For a typical organic chemistry analysis, you will be using the gc inlet, scan mode. There are two ways that you can modify the conditions for any mass spec run: In the **Method Menu** under **Edit Entire Method,** where one can go through a long list of menus to edit the two of importance, **Normal Scan Acquisition** and **Temp Information**. Changes made here will change the Supscan program for everyone who follows you. It is better to modify the conditions to **Supscan.m** using the methods described below.

5. In the **Acquire Data Menu**, choose **Main Panel** (only option)**.** Then choose **Edit Parameters.** In this menu there are two windows you should call up:

1. SIM/Scan: [The window is similar to the Normal Scan Acquisition in the Edit Entire Menu mention above.] In this window you may want to adjust three parameters:

a. Solvent delay. This tells the mass spec when to start detecting. One wants the solvent to have come off the gc before any efluent is sent to the ms source. A one minute solvent delay is good of diethyl ether; a two minutes solvent delay for solvents with bp's below 100°C and a higher solvent delay for solvents like DMSO.

b. Mass ranges. You should define the mass ranges you will search. Mass of 35 is a good low range. The upper range can be adjusted depending on the masses of the compounds you are analyzing or expecting. For the upper level choose a mass about 50 or 100 amu higher than the MM of your heaviest compound. Set Threshhold to about 50 or 100. (this sets the lower limit for the number of ions that the ms will detect.)

c. Types of plots that appear during analysis. Set Plot #1 to Total- This give a total ion chromatograph, which looks like a gc trace. Set Plot #2 to Extracted, which can be

adjusted to look at a different mass range from the Total plot.

2. GC Temperatures: [This window is similar to the Temp Information in the Edit Entire Menu mentioned above.] Here you set the temperatures of the injector, detector and oven. We use injector and detector B. For most organic work they should be set between 200-250°C. Generally the best separation of a mixture of compounds results with a programmed temperature gradient for the oven. A typical gradient might be **Initial Temperature** 40°C and **Initial Time**, 1 - 2 minutes (how long the oven stays at initial temp.), with a **Rate** (how fast the oven heats up) of 20 - 30°/minutes) to a **Final Temperature** of 200 - 250°C, and **Final Time** (how long it stays at the final temp) of 5 minutes. The **Run Time** should then automatically calculate what your run time is based on your gradient.

6. When the Method is modified, go to the **Acquire Data Menu** and choose **Acquire Data.** Fill in the Data file name (8 digits) of your choice, the operators name etc and click OK. [For the file name I like to use my initials and the page number from lab book containing the experiment that led to the solution being analyzed.]

7. Now you are ready to make your injection. When the temperatures are ready the **red** "not ready" light on the gc will go off. When that happens, make your injection and press the start button on the gc button pad. [A window on the computer will remind you of these instructions if you wait a minute.] When it asks if you want to override the solvent delay click **NO**.

8. When the run is complete, go to the **Top Menu Window**, and choose the **Data Analysis Menu**, in which you choose **Main Panel**.

10. In the **Chromatograph Menu**, choose **Draw Chromatograph**. Click **OK**. The TIC (Total Ion Chromatograph- which looks like a gc trace)will appear. With the arrow you can double click on any peak with the **right** button and the mass spectrum of that peak will appear below.

11. To Zoom in on peaks, Click with the left button and holding it down, frame the peaks of interest. Double click on the screen to return to the TIC.

12. To integrate the peaks, in the **Chromatograph Menu**, choose **Autointegrate**. To view the results, in the **Chromatograph Menu**, choose **List Results.**

13. To tabulate the results of mass and abundance choose the **Spectrum Menu** and choose **Tabulate.**

Generally any screen can by printed by choosing **Print** under the **File Menu.**