

GC-MS on the GCQ for Chemistry 355

It takes very little sample for GC-MS analysis. Generally a 1mg/mL sample in a volatile solvent is more than adequate. It is preferable to use light solvents such as ether, dichloromethane, or methanol as these come out of the GC very early, before the MS turns on. Do not use heavy solvents that come out later as these will overload the spectrometer and may damage or burn out the filament.

If you have a solid, weigh approximately 10 mg on a balance. Dilute the solid in 10 mL of solvent. Place one mL of the solution into a microvial with a septum cap (A solid cap is OK if you are doing manual injection at the GCMS. If you are unsure, ask your instructor).

If you have a liquid, weigh one drop accurately into a test tube on a balance. Dilute the drop to a concentration of 10 mg/mL with solvent. You may have to do a dilution of this solution if you do not have enough room in the test tube for the solvent. Alternately, since most liquids have a density of around 1 g/mL, dissolve 10 microliters of the liquid in 10 mL of solvent. Place one mL of the final solution into a microvial with a septum cap (A solid cap is OK if you are doing manual injection at the GCMS. If you are unsure, ask your instructor).

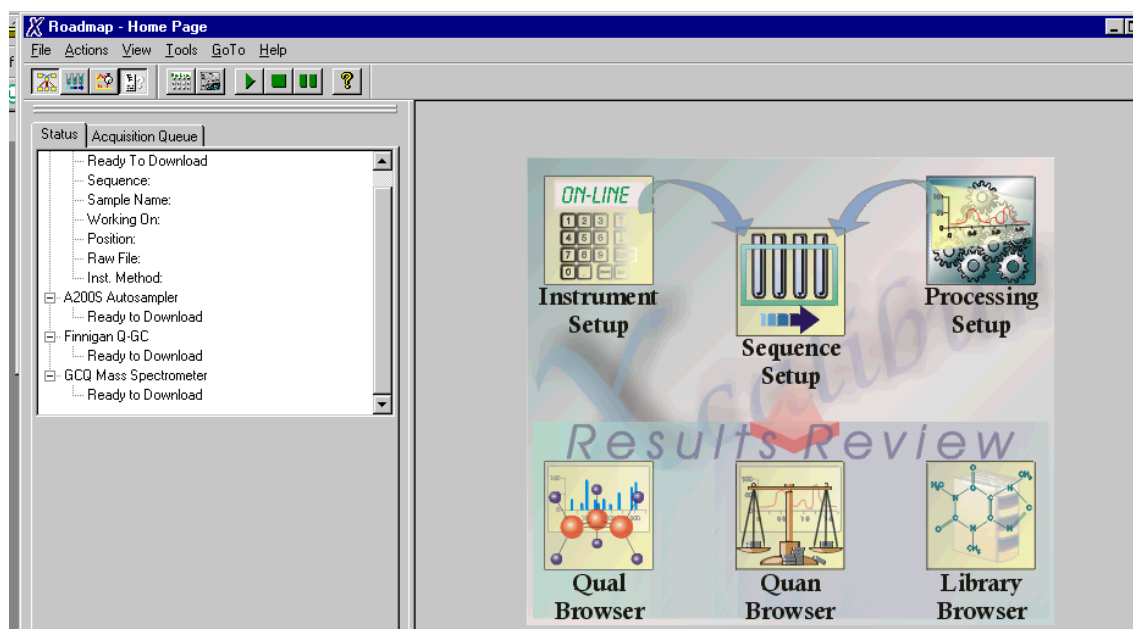
Using the Finnigan GCQ gas chromatograph-mass spectrometer

Bring your sample to the instrument. (If the autosampler is enabled, place your sample in the first open slot on the front row. Note the position number from the tray. Add methanol to the WASH solvent vial at the left end of the autosampler. *If you are unsure how to do this or which vial to use, ask your instructor.*)

Go to the computer. If it is not already open, start Xcalibur by double clicking on the icon on the desktop (right). The program will open in the roadmap view as shown below. Click on the icon labeled Sequence Setup.

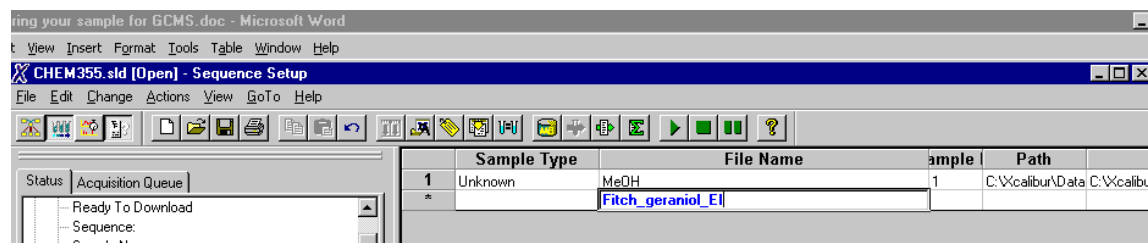


Click



From the File menu, select Open, then select CHEM355.sld to open the proper sequence file. A menu will appear with a spreadsheet (below) to enter your sample information. Click on the first open cell

under FILENAME and type the name of your sample. Do not use illegal characters (space, ., / \ ! ?, etc.) Your preferred filename should be your last name followed by the sample name followed by the type of spectrum desired (electron impact or chemical ionization, ex. Fitch_geraniol_EI or Fitch_epoxidation_CI). Avoid very long filenames as the older computers cannot handle over about 20 characters. (If you are using the autosampler, cursor to the right and enter the position number from the tray.) Press the Enter key and the computer will fill in the remaining information regarding the data path and instrument method.

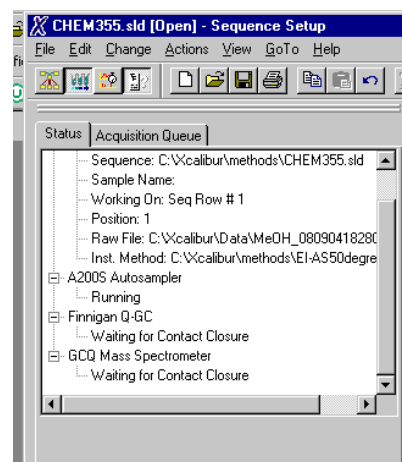


Click on the entry you just made and then on the icon at the top of the page that looks like a test tube with an arrow through it. This will submit your sample for analysis. A dialog box will appear but no information needs to be entered. Simply click on OK or press the Enter key to start the analysis.

The instruments will begin getting ready and you can observe the progress in the window on the left side of the screen. When each instrument is ready, it will say Waiting for Contact Closure. The last one to finish will be the gas chromatograph. Don't inject before the instruments are all ready.

If you are using the autosampler, it will start the run automatically (right).

If you are performing manual injection, take the 10 μ L syringe from the bench and rinse it several times with methanol into the waste vial (DO NOT TOUCH THE NEEDLE TO THE WASTE VIAL – This will contaminate the needle and your sample or the solvent). Then draw up five microliters of your sample and squirt it into the waste vial, again not touching the vial. Finally draw up one microliter of your sample and inject it into the GC port. Press the START button on the GC and the analysis will begin.

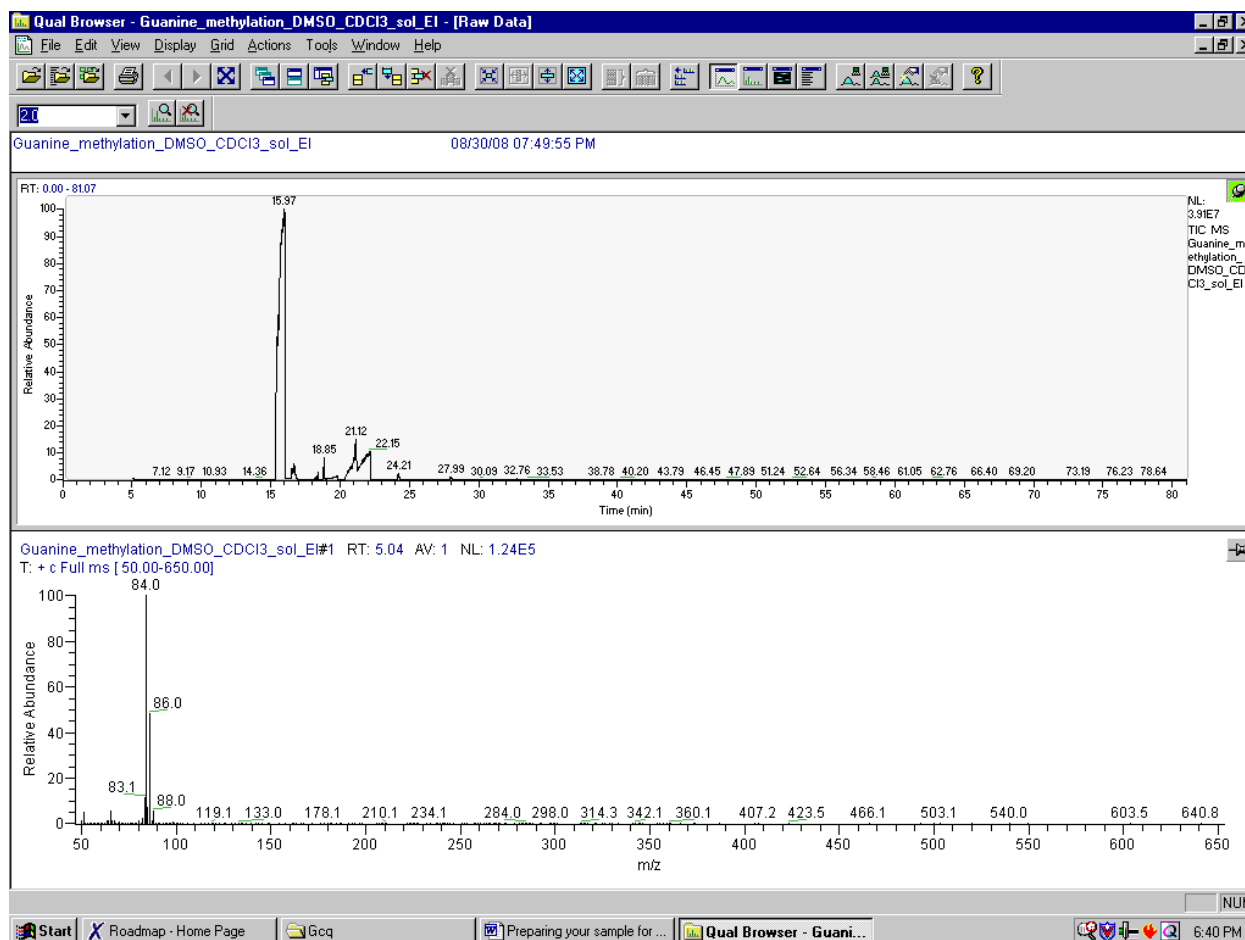


When you have finished with the instrument, close your file and retrieve your sample. Be sure to rinse out the syringe well with methanol if you did a manual injection. The rule is rinse five times with solvent to remove 99.999% of contamination.

When your run is finished, you can analyze your data by going into the Qual Browser. This can be accessed from the menu under GoTo – Qual Browser.

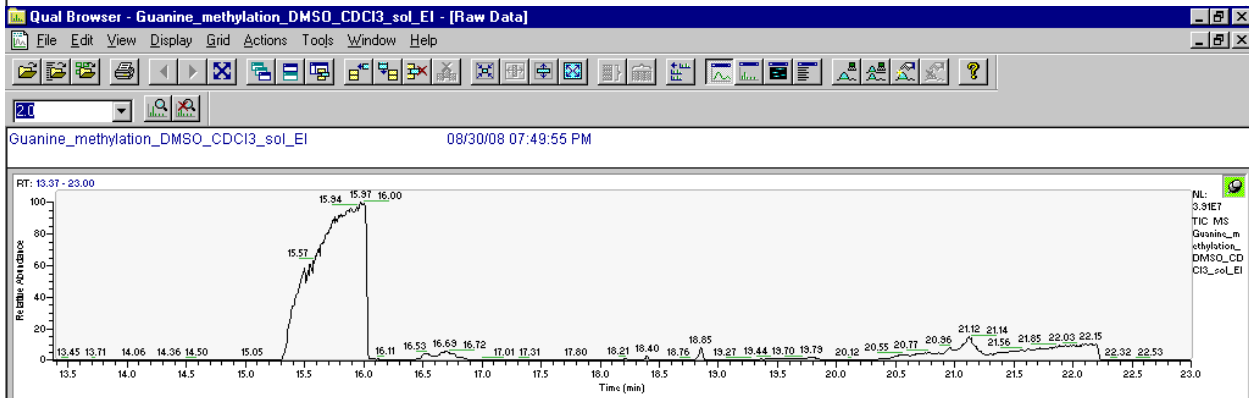
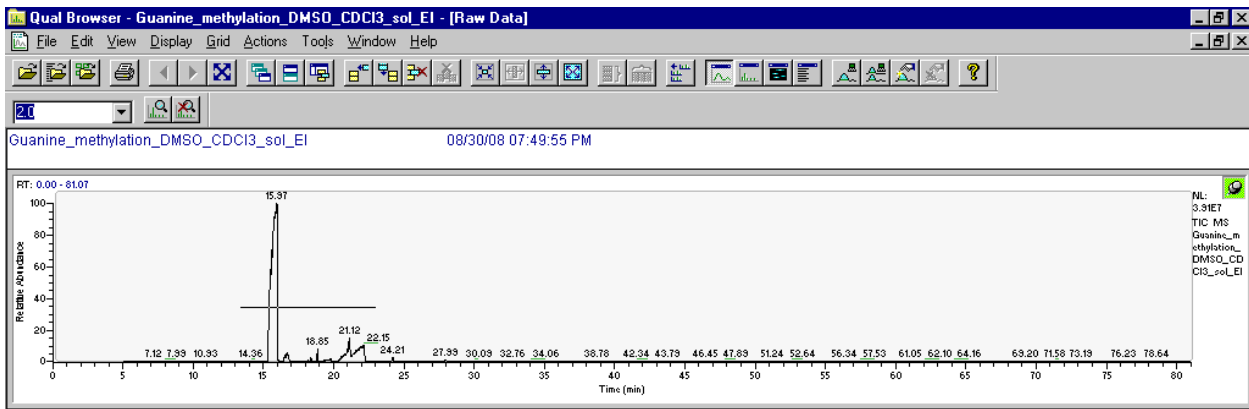
Open your file by going to File – Open and select your dataset.

Your dataset should open with one to three windows, most often three open with a pushpin icon in the upper right corner of each one (right). If the pushpin is highlighted and pushed in the window is active and can be manipulated. Select the layout by selecting File – Layout – Apply, and select CHEM355.lyt. This will give you two windows as shown below. The upper window will show the chromatogram and the lower will show the mass spectrum at a given point (it starts with the first scan acquired).

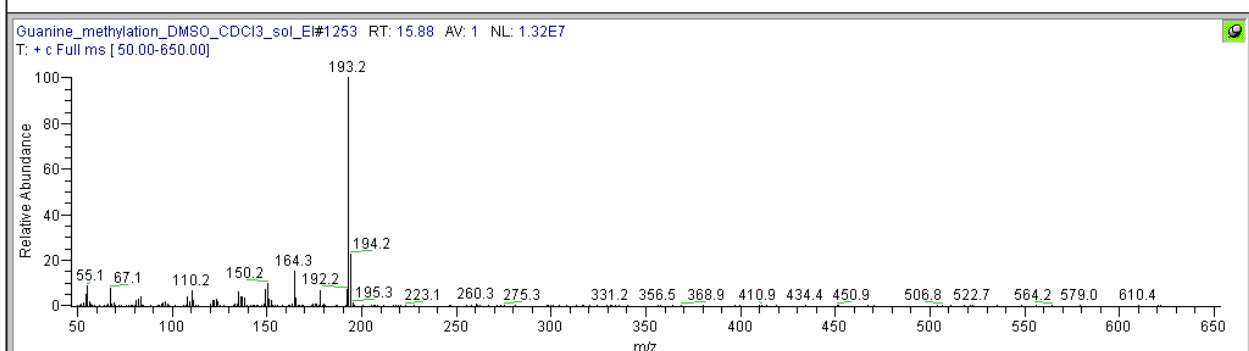
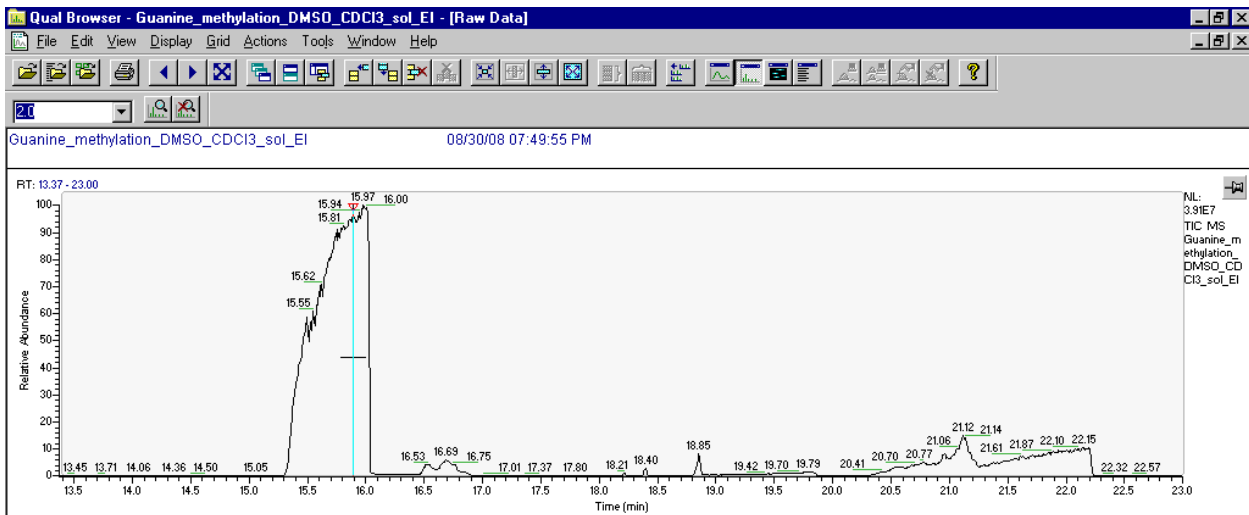


Press the pushpin for the top window showing the chromatogram for your sample. The chromatogram can be expanded in three ways:

- by clicking and dragging a window over the desired area which expands the area in the box
- by clicking and dragging a horizontal line over the desired time range, which autoscales the chromatogram to the tallest peak in the range (below)
- by selecting Display – Ranges from the menu and specifying a time range to plot



Clicking on the pushpin in the spectrum window will activate it. Then clicking on the chromatogram where your peak is will display the mass spectrum at that point.

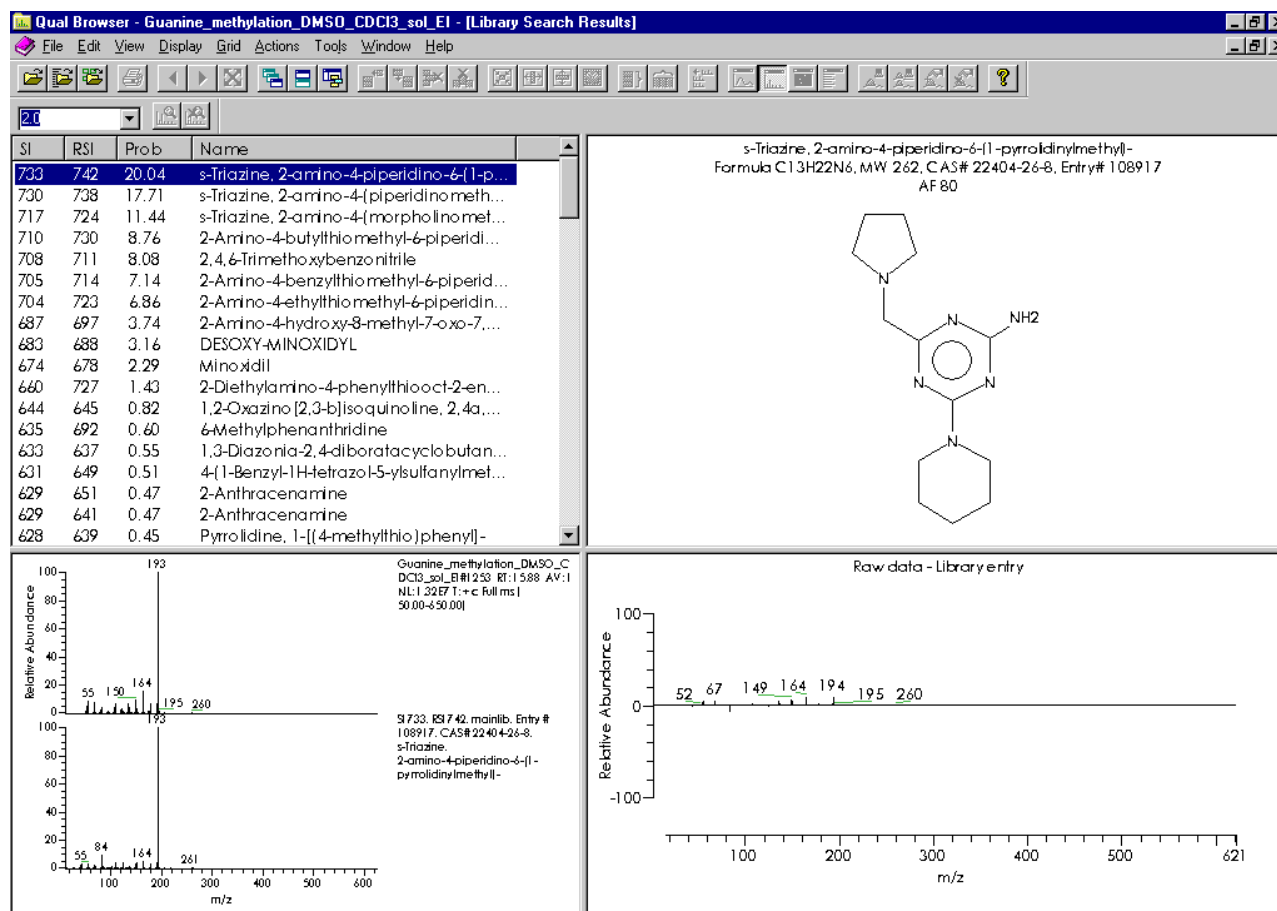


If you click and drag across a range, you will get the averaged mass spectrum for that range. For smaller peaks, this is a good idea. Also, if you have a small peak, you may wish to subtract the baseline noise so as to get a cleaner spectrum of the compound. You can do this by selecting Actions – Subtract Spectra – Range. Here you can also select a single point to subtract or a time range. If you wish to subtract two ranges, you can do so as well. (If you then obtain a new spectrum by clicking again on the chromatogram, you will need to clear the range(s) by selecting Actions – Subtract Spectra – Clear. This is because the background may vary significantly with time, so it is good practice to subtract areas close to but not including your peak or other peaks. If you are unsure about where you should subtract, ask your instructor.)

When you are satisfied with your chromatogram and spectrum, click on the printer icon to print the window (right).



If you wish to search the library for your spectrum, select Actions – Library – Search. This will search the spectral library for compounds whose spectra are similar to yours. The window that comes up will display the hit list, the structure of the current hit, and two windows comparing your spectra (below). You can browse the hits by clicking on the appropriate entry.



Be careful here as different compounds can have very similar spectra and your compound may resemble but not be the first compound that shows up or may not even be in the library. Use good judgment when evaluating library matches. If you see a compound that doesn't make chemical sense, it probably is just something with a similar pattern. If you find a close hit that makes sense, you can print out the result by selecting File – Print. The default is all cells on a single page and this is best to save paper.