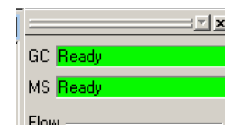
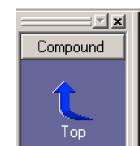


GCMS Operation

1. Double-click the “GCMS Real Time Analysis” icon to start the spectrometer-control software. A dialog box will appear prompting you for a password. Do **not** enter a password, just click “OK” or enter Return.
2. If the instrument has not been used recently, it may need to be tuned with the Autotune feature. (In most cases, the instrument will have been tuned recently, so check with Brian before doing a tune.)
 - Tune by clicking on the icons on the left: first you’ll need to find the Tuning icon. On the vertical list of icons on the left, you need to go up one level by clicking the arrow up key.
 - Next click on the “Tuning” icon and then the “Start Autotune” icon.
 - Save the tune file, print it, and place it in the Tuning Binder near the mass spec.
3. If you did not do a tune, then you can proceed to the next step. If you did run an autotune, then you will need to click on the “Data Acquisition” icon on the list of icons to the left to begin a sample run.
4. Prepare the sample. Put a tiny drop (just one!) or a very, very small spatula tip full of your compound in a MS vial and dissolve it to about half to $\frac{3}{4}$ full with methanol. Place the vial in sample slot 1, which is the slot closest to the wash and waste vials in the rack. You can use other slots, but then you need to specify the appropriate slot in step 6.
5. Load the appropriate method file by choosing “File”, “Open Method”. [For the Unknown lab in CHEM 234 or CHEM 306 the appropriate method file is Chem234unk. The full pathname is: GCMSsolutions/data/Chem234/Chem234unk. This is a pretty robust method and will work for many if not most compounds]
6. Click the “Sample Login” icon and enter your information in the dialog box. The only really important entry is the filename and the vial number (see below). I think the filename for the previously run sample is the default, so if you don’t want to overwrite someone else’s data, be sure to enter a new filename. Also, make sure that you are saving to the appropriate directory (GCMSsolutions/data/Chem234/). The default vial number is 1, so don’t change this unless you placed your sample vial in one of the other slots, in step 4.
7. Click on the “Standby” icon to get the GCMS ready for your run.

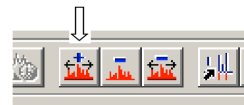
It may take a while for the machine to equilibrate to the method’s starting parameters, especially on the first run. When the machine is ready, it will indicate that both the GC and MS are ready on the right side of the window. When ready, the “Start” icon will become active.
8. Begin the run by clicking on the “Start” icon.
9. You can wait until the run is complete, or if you are sure your sample has eluted, you can stop the run with the “Stop” icon. If you stop the run early, you should also push the STOP button on the mass spectrometer itself. If you don’t, the GC oven continues to heat and finishes the temperature routine. Not pushing the STOP button doesn’t hurt anything, but you won’t be able to begin a new injection until the temperature program has finished and the machine resets itself, which may be several minutes.
10. To analyze the data, you need to open a new program. On the desktop, click on the “GCMS Postrun Analysis” icon.



GCMS Operation

11. Load your data file: “File”, “Open Data File”. The GC trace appears with a mass spectrum window below.
12. You can select the peaks on the GC trace you want to analyze, one at a time. On the GC trace, you can zoom around peaks by dragging a box around the peak of interest, with the mouse (left mouse button). To undo a zoom, click the right mouse button and select “Undo Zoom”; doing so will undo the last zoom you did. To go back to the full GC trace (if you have zoomed in more than once), click the right mouse button and select “Initialize Zoom”.

13. To get the mass spectrum of a GC peak, use the “Average Spectrum” icon on the toolbar that runs across the top of the window:
Click and drag the cursor across the peak of interest.



14. To subtract any background junk from the spectrum, which may be dribbling off the GC column throughout a run, click on the “Average and Subtract” icon and click and drag across a section of the GC trace near the peak, but which has no signal – i.e. just baseline. This will subtract the mass spectrum of material eluting in this baseline region (mostly noise, or column junk) from the averaged spectrum of the peak of interest. Your spectrum should appear.



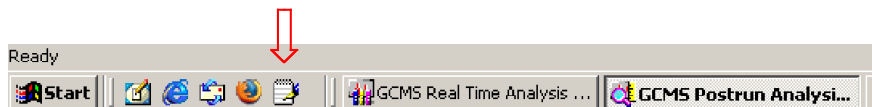
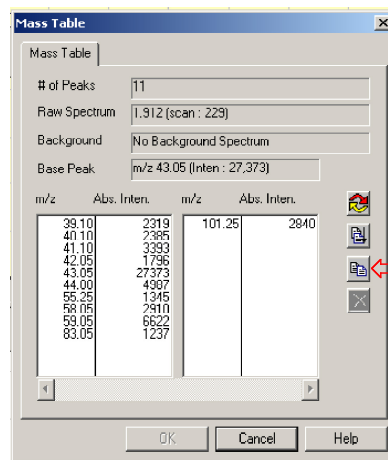
15. Print the spectrum: “File”, “Print Image – Print”; or you can just click on the “print” icon.
16. If you have some very small peaks that you would like to get a better look at, you can change the vertical scale on the mass spectrum by clicking on the + and – buttons found off to the lower right side of the mass spectrum.



17. If you would like a Mass Table—a table listing all the peaks and their relative and absolute intensities—right click anywhere on the mass spectrum and select “Mass Table” from the options that appear in the dialog box. You can’t directly print this table, but you can copy the data and paste it into the PC’s Notepad program, and then print.

To copy the data, click on the copy to clipboard icon (denoted by the red arrow on the figure) on the right side of the Mass Table window.

Then paste into a text document using the Notepad program. The Notepad program can be accessed from its icon on the toolbar that runs across the bottom of the screen, as indicated by the red arrow in the figure below.



18. When you are completely done for the day, go back to the “GCMS Real Time Analysis” and shut the instrument down using the “Daily Shutdown” routine -- under the “Tools” menu.