

# GC-MS of Organic Unknowns

## Method Selection

In order to analyze a sample using the GC-MS, we must first choose the correct method in Chemstation. The method contains all the GC-MS parameters needed for the analysis.

1. If you are not in the “Instrument Control” window, open it by choosing “Instrument Control” from the View menu.  
Click on the Method menu, and select Load...
2. Select the method “organic1.m”.
3. After the method has been loaded, click on the button with the large green arrow to start the analysis.
4. Enter your name in the “Operator Name” box that appears
5. Enter “organic.d” in the data file name box. Note: normally we do not wish to save these chromatograms, so we will continuously overwrite to this file!
6. Enter the name of your sample in the “Sample Name” box.
7. If you desire, enter any miscellaneous information in the “Misc. Info” box.
8. Click on the “Start Run...” button at the bottom of the screen.
9. Click on the “Yes” button that appears on the overwrite file dialog box prompt.
10. At this point, the computer will send all the information needed to the GC and the MS to perform the analysis.
11. You are now ready to inject your sample and perform the analysis.

## Sample Preparation and Injection

12. Using a glass Pasteur pipet, transfer about 4 mL of  $\text{CH}_2\text{Cl}_2$  into a sample vial. Note: a Pasteur pipet contains about 2 mL of fluid when filled.
13. Using another glass Pasteur pipet, add 1–2 drops of your unknown sample to the  $\text{CH}_2\text{Cl}_2$ , and mix.
14. Take a 10  $\mu\text{L}$  GC-MS syringe (make sure it has a blunt, cone shaped tip) and pull the plunger back to the 1  $\mu\text{L}$  mark. Place the tip of the needle in your sample, and withdraw the plunger to the 2  $\mu\text{L}$  mark. Remove the tip of the needle from your sample, and pull back the plunger to the 3  $\mu\text{L}$  mark.
15. Your unknown sample is now held between two air gaps.
16. Hold the plunger in place while you align the syringe over the injection port. We are using injection port B, which is the port the furthest from the front of the GC. Inject the sample by inserting the syringe needle into the injector until the barrel of the syringe rests on the injector. Press the plunger all the way into the syringe. Remove the syringe from the injector, and then **immediately** press **START** on the GC keypad. (Or click **START** on the computer screen.)
17. **Do not choose** “override solvent delay” from the window that pops up. If you ignore this window, it will eventually disappear. Overriding the solvent delay will greatly reduce the life expectancy of the mass spectrometer.

## Data Analysis

During a run, from the “MS Top/Enhanced” window, click on the “View” menu, and select “Data Analysis (offline).”

18. If you would like to analyze your chromatogram as it is being collected, click on the “File” menu, and select “Take Snapshot”. Otherwise, wait for the data to be collected, and choose “Load Data File” from the “File” menu. Your data file name should be “Organic.d”.
19. Make sure the Organic1.m method has been loaded. It should say “ORGANIC1.M” in the title bar at the top of the screen. If it does not, then click on the “Method” menu, and select “Load Method...”. Choose “Organic1.m” from the list that follows.
20. You can zoom-in on any peaks by holding down the left mouse button and drawing a rectangle over the area you would like to zoom in on.
21. You can zoom-out by double clicking the left mouse button anywhere on the chromatogram.
22. To display the mass spectrum at any point in the chromatogram, double **right** click on a point in the chromatogram. The mass spectrum will be displayed below. You may also average several data points together by drawing a rectangle over the chromatogram peaks with the **right** mouse button depressed.
23. You may print a copy of the mass spectrum and the chromatogram by selecting “Print” from the “File” menu. If you select “TIC and Spectrum”, you will receive a print-out of the TIC (total-ion-chromatogram) and the mass spectrum you selected.
24. If you need to integrate your chromatogram, select “Autointegrate” from the “Chromatogram” menu. Do not save the autointegration parameters to the Method. You can view and print the integration report by selecting “Integration Results...” or “Percent Report” from the same menu.
25. When you are finished analyzing your chromatogram, you can quit the **Data Analysis** program by selecting “Exit” from the “File” menu.

## Finishing Up

26. If you are finished with your unknown sample, throw the solvent away in the Halogenated Organic Solvent waste bottle located in the Northern most fume hood in the Organic Chemistry lab.
27. It is very important to clean the syringe out after every injection. You can do this by putting a small amount of acetone in a beaker, and drawing up 5–10  $\mu\text{L}$  of acetone into the syringe, followed by squirting it out into a kim-wipe (where it will quickly evaporate). You should do this 5–6 times to ensure the syringe is clean. Failure to do this will result in carry over of your sample into the next sample introduced into the syringe. Also, the plunger may freeze-up if the sample is not washed off of the inside surfaces of the syringe.