

## Organic TA's Guide to the Agilent GC/MS Autoinjector

As a TA, you will set samples up for analysis on the Agilent GC/MS and transfer the resulting data-folders to the NMR drive, so that students may process their GC/MS data as they do their NMR data. Like the NMR, the GC/MS has an automated mode, but the injector can only hold sixteen samples at a time, making it necessary to empty and reload the carousel if there are two lab sections in a given afternoon.

The first time the GC/MS is used, you should take entire lab section to the GC/MS (in CSC-403), to point out the injector, the oven and column, and the MS, and to watch the first sample run. Also open one of the data files and point out how the MS relates to the GC trace.

**Before the lab period**, tune the mass spectrometer:

1. Switch to the GC7890B software, which will most likely already be running.  
(If not, click the green icon at the top of the Desktop.)
2. Click the tuning-fork icon and select "Quick Tune". Press OK.

The autotune will run for about five minutes, then print a report when it is finished.

**Before leaving the lab with samples**, verify that:

1. Samples are in septum-capped GC/MS vials with students' names written in Sharpie.
2. All vials contain a reasonable depth of solution (above the 0.5 mL line).
3. All samples are clear and well mixed — uniform color and no droplets or suspended particles.

If any samples are not properly made, the student should fix the problem or make a new sample.

**To set up the Automation Sequence**

1. Sign into the log-book by the instrument.
2. Check the three large vials on the carousel: Vial A (rinse solvent) should be at least half full, and Vial W (waste) should be reasonably empty. (This method does not use Vial B.)
3. Switch to the GC7890B window.
4. Recall the Injection Sequence through the menus: SEQUENCE → LOAD SEQUENCE. The sequence will be named something like *chm234\_FriedelCrafts.sequence.xml*, and be located in the directory D:\MassHunter\GCMS\1\sequence\ .
5. Fill in the Sequence Table:
  - i. From the menus, select SEQUENCE → EDIT SEQUENCE.
  - ii. Use the **New Samples** button to create rows in the table.
  - iii. Fill in the first row of the table:
 

Vial:	the slot number in the carousel
Method Path:	D:\MassHunter\GCMS\1\methods
Method File:	eg, <i>chm234_FriedelCrafts_with_Autoinj.M</i>
Data Path:	D:\MassHunter\GCMS\1\Data\Organic_Lab
Data File:	the student's name. (Use the same naming convention as for NMR data files.)
Vol:	0.1 $\mu$ L (This field may be irrelevant)

- iv. Copy the first line of the table and paste it into each of the subsequent lines, changing the vial number and the student's name as appropriate.
  - v. Press OK.
6. From the menus, choose SEQUENCE → RUN SEQUENCE. Verify that the information looks correct, and click the Run Sequence button at the bottom left.
- Respond "NO" at the prompt to override the solvent delay — overriding it will shorten the lifespan of the detector.
- If a sample is bad, you may abort it with the stop-sign button.
7. Each run should take about six minutes, so you will know when to return from lab to collect the samples and transfer the data.
- If you have more samples to run, press the edit button in the yellow bar, add the names to the list, and save it. (You may do this while the sequence is running.)

### Shutting Down

Bring a flash-drive with you to the instrument.

When the sequence has finished, the yellow stripe will disappear.

- (8) If your section is the last of the day, clean the column:
- i. Load the cleaning method from the menus: METHOD → LOAD METHOD → *Bakeout.M*. The method will be in D:\MassHunter\GCMS\1\methods.
  - ii. Click the green arrow at the top of the Acquisition Window.
    - Select D:\MassHunter\GCMS\1\data\ as the path, and *Bakeout.D* as the data file. (ok to overwrite it.)
    - Click OK and Run Method.
  - iii. Press the Start button on the front right of the GC, and verify that the oven temperature begins to increase. The cleaning cycle will stop after three hours.
9. Minimize the Data Acquisition window.
10. Browse to the organic data-folder, D:\MassHunter\GCMS\1\data\Organic\_Lab\. There is a helpful *Data* shortcut on the Desktop.
11. Copy the data folders created during the run (which will end in ".D") onto the Flash Drive, and move the originals from the *Organic\_Lab* directory into the appropriate *Transferred* subdirectory.
12. On a networked computer (eg, the Chem Club room), access the NMR drive folder for your lab section. (\\wu-labshare\labshares\Chemistry\...).
- Create a new "MassSpectra" folder within the folder for your lab section (as if it were a student's name). Transfer the data folders from your flash-drive into the new folder on the NMR drive. (eg, Chemistry\Organic Lab\Mon\data\mon\nmr\MassSpectra\DebbieF.D)

### Troubleshooting

*Front Inlet Pressure error:*

This error usually means that the septum needs changing. Ask Dr. Mencer or Dr. Trujillo for help.

*Broad, Stray GC Peaks*

This suggests that the column needs cleaning. When you are done your section, run the *Bakeout.M* method, as described in step 8. (It will require three hours, so don't do this during the lab).