

## **Instructions for using Haverford College's Agilent 1100 LC/MSD**

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Instructions\LCMS\LCMSinstructions.doc*

*Please send proposed revisions to Rob Scarrow via email (rscarrow@haverford.edu).*

### **SAMPLE PREPARATION:**

1. It is important to remove any possible particulates from your sample. This may be conveniently done using disposable 2 mL plastic centrifuge tubes (with snap-on lids) and a tabletop centrifuge (the kind commonly used in Biology labs). Be sure to balance the centrifuge by placing another tube with an (eyeballed) equal amount of water in the opposite position in the centrifuge. Run the centrifuge for about 30 seconds.
2. Remove the centrifuge tube from the centrifuge and using a pipette, remove the liquid leaving any solid behind. The pipette should be drained into a LC/MS sampling vial with a screw-top puncturable lid. Your sample is now ready to run on the LC/MS.
3. Once the method has been changed for your specific purpose load your vial/sample into one of the numbered slots (1 to 100) on the LC/MS injection unit. When the door is opened and the tray is removed the MS will notice this and will put a yellow warning on the screen. You may ignore this – the warning will go away when you close the door again.

### **TURNING THE INSTRUMENT ON:**

1. Turn on the computer, log in as Administrator and use the password provided on the disk drive.
2. Double click on the **hplc msd Online** Icon.
3. The Chem Station window will pop up and will be ready to use. It will take approximately 1-2 minutes for the program to load.

### **RUNNING A SINGLE SAMPLE:**

1. If you have a method to load, go to File, Load, Method. If you do not have a method, follow along below.
2. In looking at the main setup screen, you will see a line of icons. In the top left corner of the screen, be sure to have the single bottle highlighted.
3. Below, click on the sample bottle, and then click Sample Info.
  - In Data File, type in an operator name. Click Prefix/Counter. Make sure the subdirectory is your lab.

- In Sample Parameters, type in the location of the vial, then the rest of the information is optional if you wish.
  - Click OK.
4. Click on the syringe icon, and then click Setup Injector.
    - In injection, set injection volume. (Suggested volume is 25 micro liters)
    - Click injection with needle wash, which will clean the needle.
    - In Needle Wash, make sure location is set to vial 100 (you should empty and refill this vial before you run your sample. Make sure that the content of the needle wash vial is the same as the solvent to be used.
    - Click OK.
  5. Click on the pump icon, and then click Setup Pump.
    - In Control, put in flow rate, and set stop time. (Suggested values to start with are a flow rate of 0.4mL/min and a stop time of 2 minutes.) Select the appropriate solvents, and make sure that the solvents listed on the window correspond to the actual bottles to the left of the apparatus.
    - **To do a solvent gradient:** In Timetable, click insert, then input time, percent of solvent B, flow rate, and the maximum pressure. To add another gradient, click append, and continue until completed.
    - Click OK.
  6. Click the column icon, and then click Setup Column Thermostat.
    - For direct inject, make sure the column switching valve is set to Column 2.
    - Click OK.
  7. Click the MSD icon, and then click Setup MSD Signals.
    - This option allows you to scan for positive and/or negative masses within a specified range, or you can also scan for a specific positive/negative peak.
    - Click OK.
  8. Click the MSD icon a second time, and then click Spray Chamber.
    - In this window, you can set the drying gas temperature.
    - Click OK.
  9. Click the DAD icon, and then click Setup DAD signals.
    - In this window, you can search at the excitation wavelength and see the UV-Vis spectrum in analysis when completed. This exists with one caveat the wavelength for the spectrum should be known and checked in the DAD setup.
    - Click OK.

10. Next, click on each small box icon at the bottom right of each icon, turning the appropriate apparatus. Wait for all the icons to turn green. Then click the start button on the left side of the screen.

**RUNNING A SERIES OF SAMPLES:**

1. Click on the multiple bottle icon in the top left corner.
2. Click on the icon that resembles the sample tray, and then click Sequence Table.
  - In this window, input the location of each vial, the sample name, and the appropriate method.
  - Click OK.
3. Then follow the same steps as above (Steps 3-10).

**CHANGING SOLVENTS:**

1. In the pump icon, click on the desired new solvent.
2. Set the sample vial to the wash vial, and be sure that the content of the needle wash vial is the same as the solvent used. Run the solvent for 1-2 minutes. Your sample is ready to run.

**INSTALLING NEW SOLVENTS:**

1. Obtain the new solvent, undo the old solvent and replace it with the new one.
2. On the far left of the machine on the third panel there is a black knob - this is the purging valve. Turn the valve counter clockwise (PUMP SHOULD BE OFF).
3. Now that the valve is open, in the pump icon, set the pump rate to 5 ml/min and 100% of the new solvent. Also, make sure the correct solvent is chosen. Run the pump (turn it on) for 2.5 minutes. Stop the pump, close the valve and reset the pump parameters to the correct amounts. You are ready to run the sample.

**FOR ADDITIONAL HELP:**

Go to the link [www.chem.agilent.com](http://www.chem.agilent.com) for additional help available online. Also useful are the instructional videos for the various procedures and maintenance of the LC/MS at the following address:

[www.chem.agilent.com/cag/cabu/lcvideoindex.asp#LC-MSD](http://www.chem.agilent.com/cag/cabu/lcvideoindex.asp#LC-MSD)