

GC-MS Barebones Guide


revised 7-20-06

GC Sample Prep:

1. All samples must be organic (NO AQUEOUS OR WATER!). Typical solvents are chloroform, ethyl acetate or hexane.
2. NMR samples may be used. Proton samples need not be diluted. Ideal concentration for GC-MS is 1-2 mg/mL.
3. Filter your sample.
4. Know the approximate boiling point of your sample. If it's over 350°C, GC may not be the best chromatographic method for your experiment.

Software:

1. Sign in the logbook.
2. Log into the computer – same login and password as for NMR.

3. Open up the GC-MS Instrument #1 by double-clicking the  icon.

The software should look like this when you open it:



- Open the DEFAULT.M method by clicking Method -> Load, selecting DEFAULT.M and clicking “okay”. The default temperature parameters are:
 - Inlet temp: 250
 - Initial column temp: 100
 - Temperature ramp: 20°/min
 - Final column temp: 280
 - Detector temp: 280
- These parameters can be changed as needed to accommodate the boiling point and



separation needs of your sample. Click on the Oven and chose “Oven Temperature” to change temperature parameters.

The screenshot shows the "Temperature Instrument 1" software interface. It is divided into several sections:

- Zone Temperatures:** A table with columns for "On", "Setpoint", and "Actual".

On	Setpoint	Actual
<input type="checkbox"/> Inl. A:	C	0 C
<input checked="" type="checkbox"/> Inl. B:	250 C	250 C
<input type="checkbox"/> Det. A:	C	0 C
<input checked="" type="checkbox"/> Det. B:	280 C	278 C
<input type="checkbox"/> Aux.:	C	0 C
- Oven Parameters:**
 - Oven Equip. Time: 0.50 min
 - Oven Max: 325 C
 - Oven On
 - Cryo On Ambient: 25 C
 - Cryo Blast On
- Oven Program:**
 - Init. Temp: 100 C
 - Init. Time: 4.00 min
 - Final Rate Temp. Time (C/min) (C) (min) table:

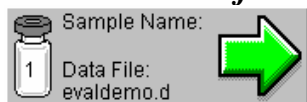
	Final Rate (C/min)	Final Temp (C)	Final Time (min)
Level 1	20.0	250	2.00
Level 2(A)	0.0		
Level 3(B)			
 - Next Run Time: 13.50 min
- Graph:** A line graph titled "Oven Temp vs. Time" showing a temperature ramp from 100 C to 250 C over time. The x-axis is labeled "Time" and the y-axis is labeled "Temp".

To change the temperatures you use the Oven Program on the right half of the screen. Final temp should be set at least 5° higher than the highest boiling point component of your sample. If your boiling point is above the column maximum temperature, set the final temp as 280°. The run time for your method is shown below the box.

If you wish to use two or more gradients, use the Level 2 and 3 program boxes. Otherwise leave blank.

- When you are done programming any changes, save the new method by clicking Methods -> Save and save the method with your initials.

Running your sample – Manual Injection:



1. Click the arrow when you are ready to start acquisition.
2. After the software downloads your method, the Start Run box will appear. Enter your file name in the Data File Name box, be sure the box next to Data Acquisition is checked, and click 'Start Run'.
3. To Inject: Wash the needle with sample, then fill the needle (be careful to avoid air bubbles!) to .5-1 μL . Use .5 μL for concentrated samples. **DO NOT USE MORE THAN 1 μL OF SAMPLE EVER!!!** This will overload the column, resulting in poor chromatography and a broken MS!
4. Insert the needle into the injection port on top the GC. Simultaneously inject and hit START on the panel on the GC itself. Quickly remove the syringe from the injection port.
5. A box will open, asking "Override Solvent Delay?" Click **NO**! If you click yes, then the MS will begin taking data of your solvent, which will break the MS!
6. As your experiment is running you can observe the chromatogram in the Total Ion box. To extend the run time, click on the Oven icon, select "Oven Temperature..." and change the Final Time for your last program step (see Step 5 of Setup section above).

Data Analysis:



1. Open the offline data by double-clicking the Data Analysis icon on the desktop.
2. If your sample is still running, you can click File->Take Snapshot to observe the chromatogram and MS spectra so far. If the acquisition is completed, open your file by clicking File->Load and select your file.
3. To Zoom: Press and hold LMB and drag a box around the peaks of interest. Double-click the LMB to zoom out.
4. To view mass spectra, double click the RMB at the apex of the peak. The mass spectrum will open below the chromatogram.
5. Click File->Print->TIC and Spectrum to print out the chromatogram. You can also print Selected Window; enter "2" for the chromatogram and "1" for the mass spectrum.