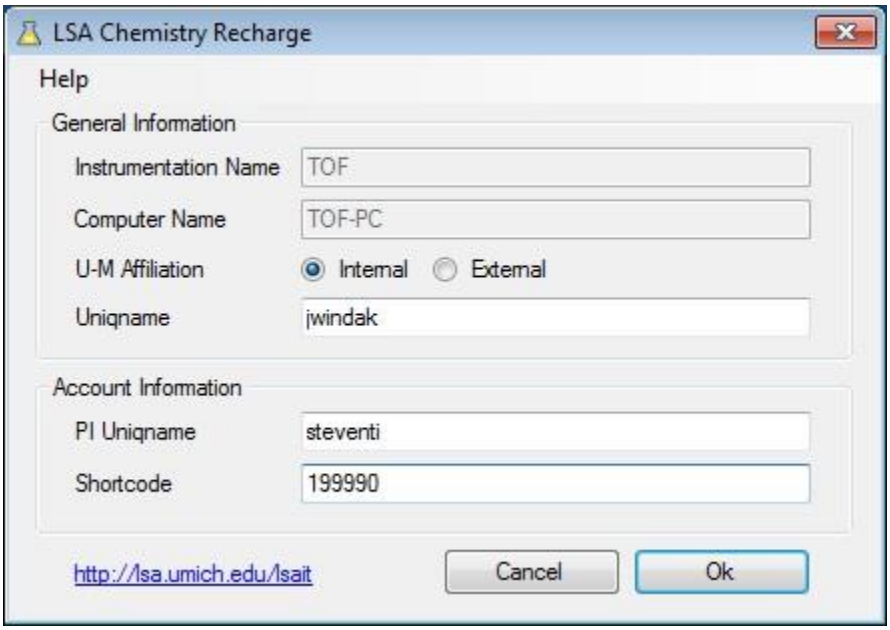
# Shimadzu GCMS User's Booklet

1) Double-click the LSA Chemistry Recharge icon:


Type in your uniquename, your PI's uniquename, and your short-code account number and click Ok:



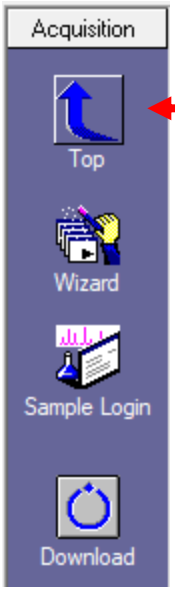

After you login, the data acquisition software will automatically come up. When you see this box, just click OK:

2) After the data acquisition software comes up, you will set up the Batch table for running your batch of samples. If the batch table is not visible, click on the 

icon on the left side of the screen. If the 

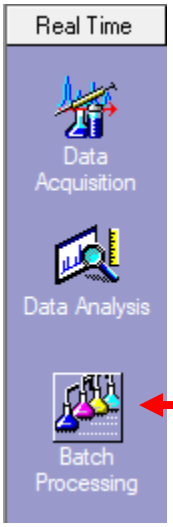
icon is not visible on the left side of the screen, then you may need to move up a level by clicking on the 

icon on the left side:




Click here to move up one level if the Batch Processing icon is not visible.

It should then say “Real Time” and the Batch Processing icon will be visible:



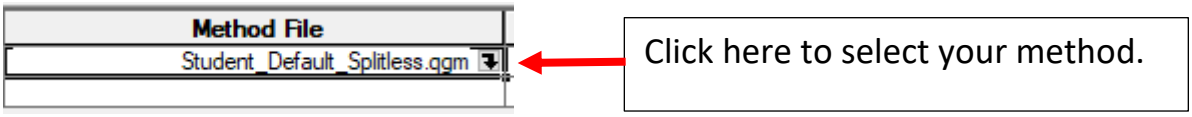

You can now click on the Batch Processing icon to bring up the Batch Table.

In the Batch table, you need to fill in one row for each sample that you want to run. In each row, you there are 4 important items you need to fill in.
The Vial# is the location where you put the vial in the autosampler tray.
The Method File is the method that you want to use to run this sample.
The Data File is the filename for the data.
The Inj. Volume is the amount in microliters that you wish to inject.

Folder: C:\GCMSsolution\Data\Project1

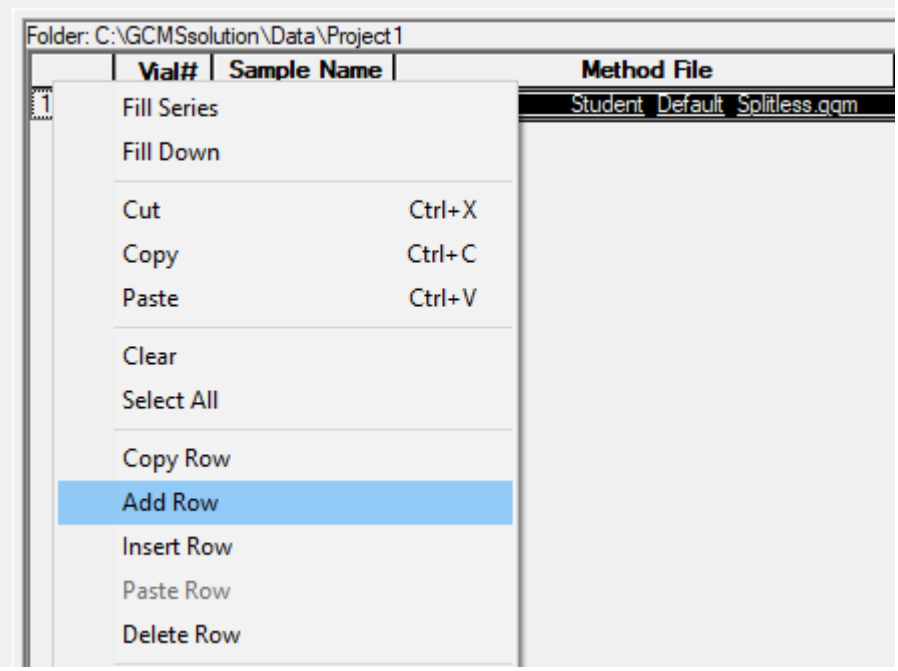
| | Vial# | Sample Name | Method File | Data File | Inj. Volume |
|---|---|---|---|---|---|
| 1 | 1 | GCMS test | Student_Default_Splitless.qgm | GCMS Test Solution 4-6-2020.gcd | 1 |
| 2 | 1 | | | | 1 |

If you need to change the Method File, click on the Method File cell in a row. You will see an icon. If you click on the icon, you can then Browse to where your method is located and select it.



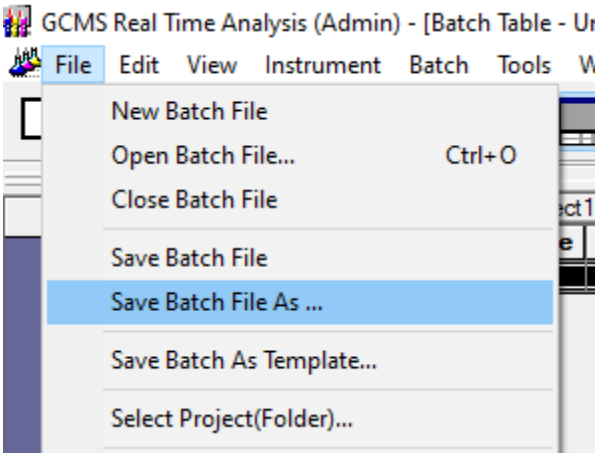

To select where your Data will go, click on a Data File cell. You will see the icon and you can click on it to navigate where you want to put the data, and what you want to name it.

If you need more rows, right-click to get a menu, and then click on Add Row:

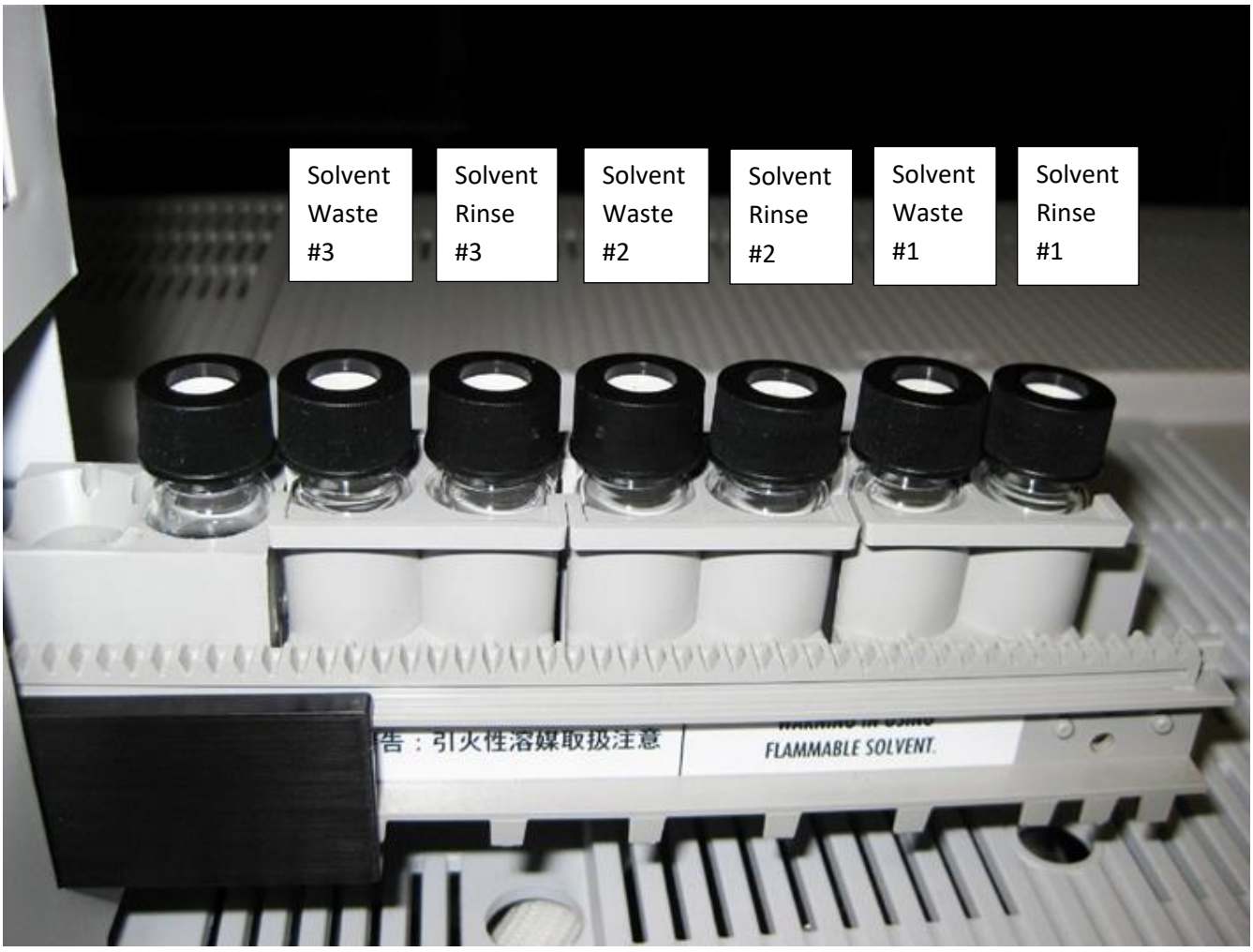



If you need to delete rows, highlight the rows, and right-click to get a menu, and then click on Delete Row.

After the table is filled out, save the table by clicking on File, and Save Batch File As…




**3)** Remove the solvent tray from the Auto-injector and make sure that there is solvent in the 3 rinse vials. **<u>If the vials are less than ½ full, the syringe may not get cleaned between sample runs. If the 3 vials need to be refilled, please use the methylene chloride rinse bottle in the hood to refill them.</u>**

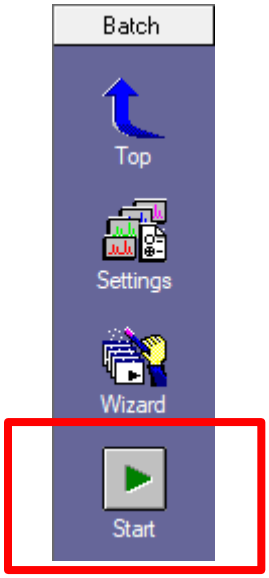


Push the tray back in, and press the “Reset” button on the front of the Injector. The tray will then move to the correct position.

3) Put your samples into sample vials. You need to use a 2 ml autosampler vial with a Teflon-lined cap, such as Fisher Scientific part # 03-391-15.

4) Put your samples into the autosampler locations that you specified in the Batch Table.

5) Press the Start button to start the run:

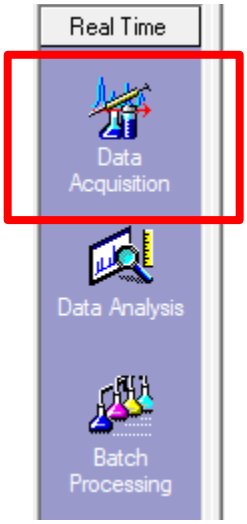



# How to Modify a Method

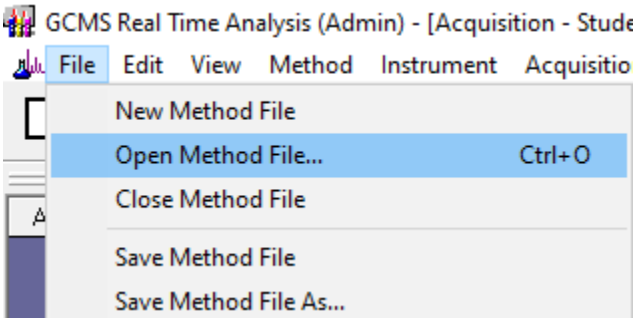
A method contains all of the instrument parameters used to control the autosampler, the GC, and the mass spectrometer.

To modify a method, first go the Data Acquisition page. If you are in the Batch Processing page, you will need to click on the 

icon to go up one level, and then click on the Data Acquisiton icon:

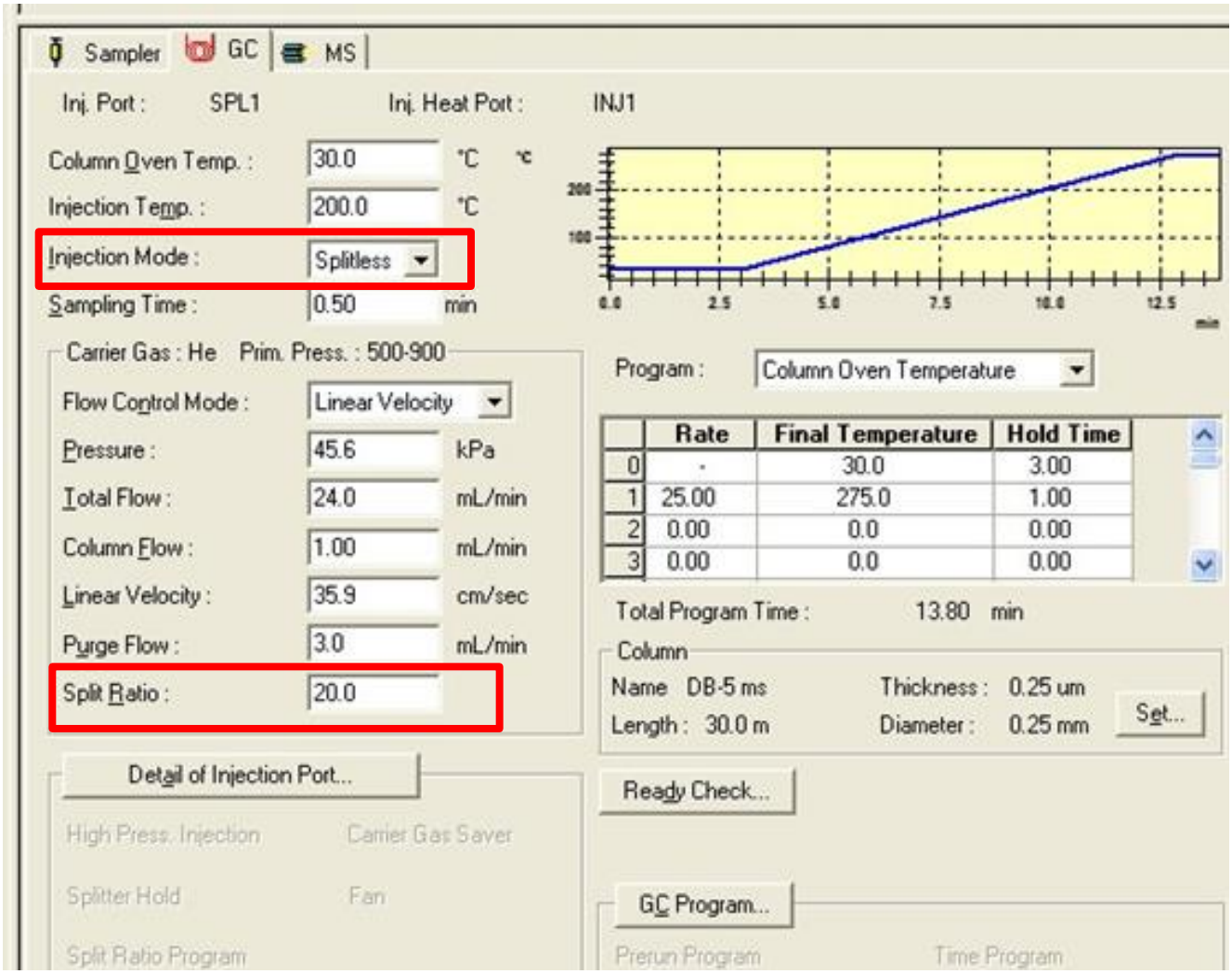


In the Data Acquisiton page, click on File, and Open Method File…

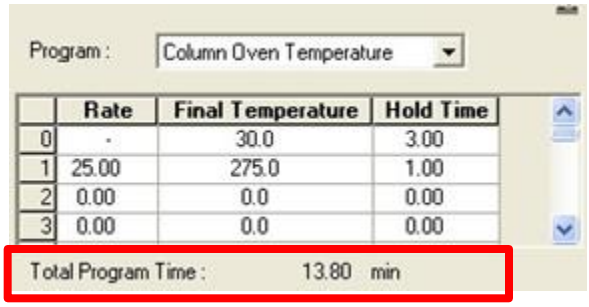



There are 3 tabs in the method editor. One is for the Autosampler settings, one is for the GC settings, and one is for the MS settings.

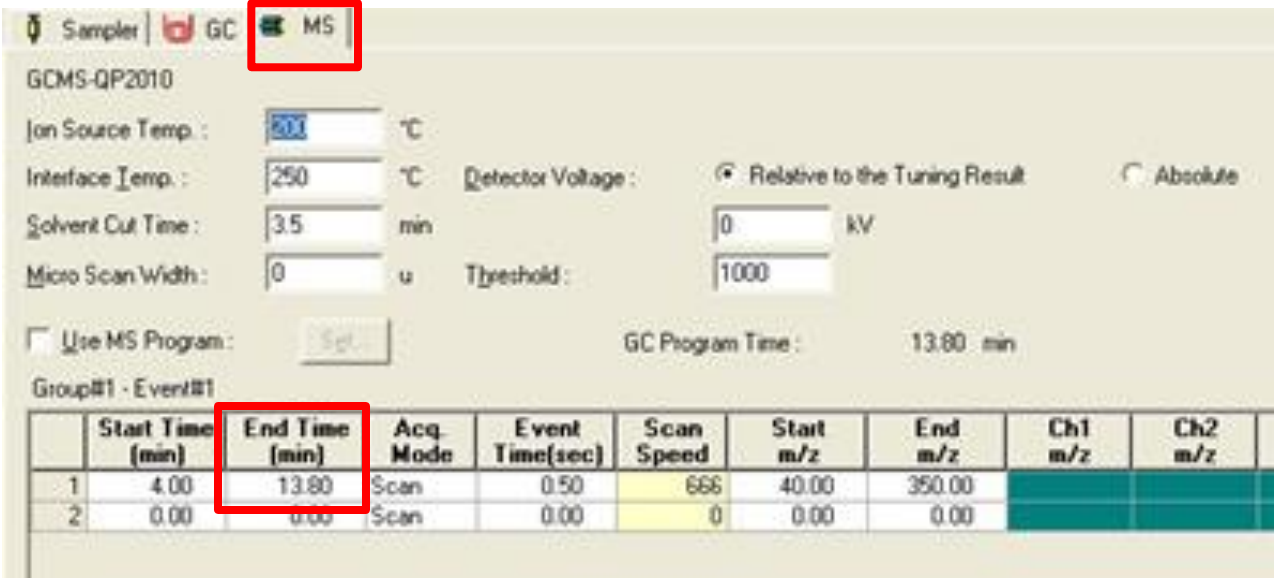
**On the GC page, choose the Injection Mode, as either Splitless or Split. For very dilute samples (low nanogram per microliter concentrations), you should use Splitless. For more concentrated samples, you should use Split. For example, if the sample concentration is 1 mg/ml, use a split injection and set the Split Ratio to 100 for a 1:100 split.**



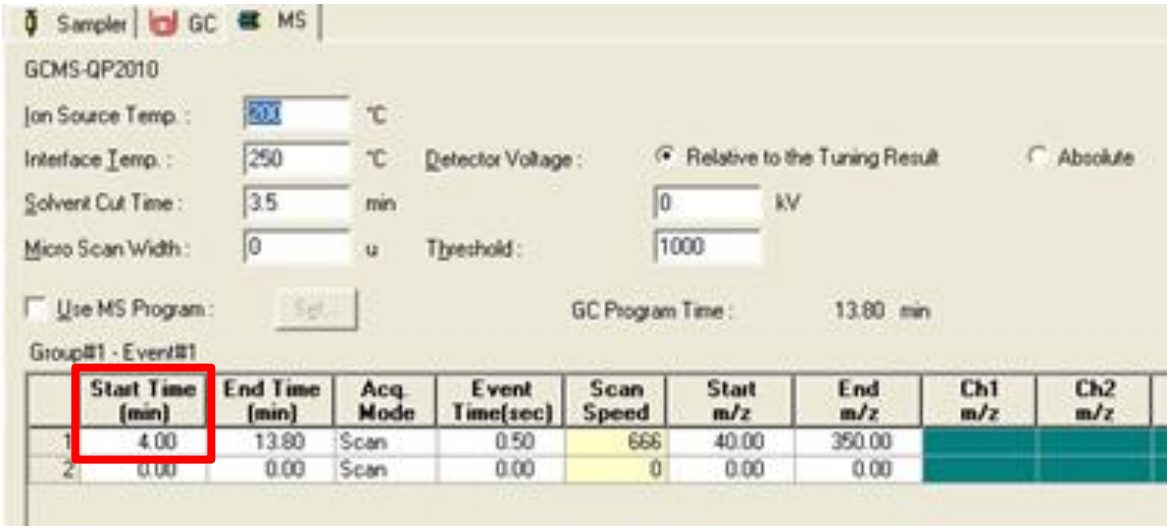
The GC temperature program can be set with the table on the right. For example, this temperature program holds the GC oven at 30 C for 3.00 minutes. Then it ramps the temperature up to 275 C at 25 degrees per minute. Then it holds it at 275 C for one minute. The Total Program Time for this is listed as 13.80 min.



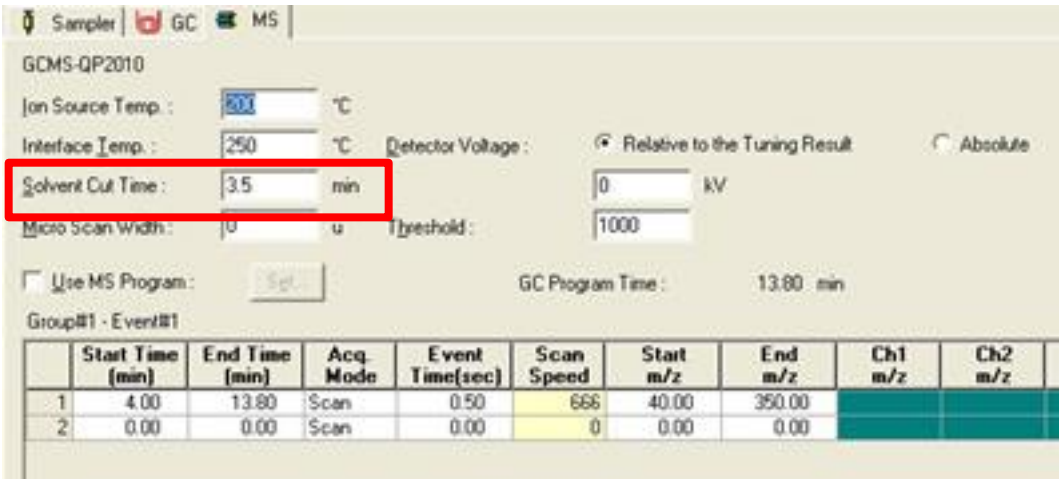

The MS data acquisition time should be set to the same amount of time as your GC program. For example, if your GC program takes 13.80 min, then the MS should acquire data until 13.80 min as well, so you should set the End Time (in the MS tab) to 13.80 min in this example:



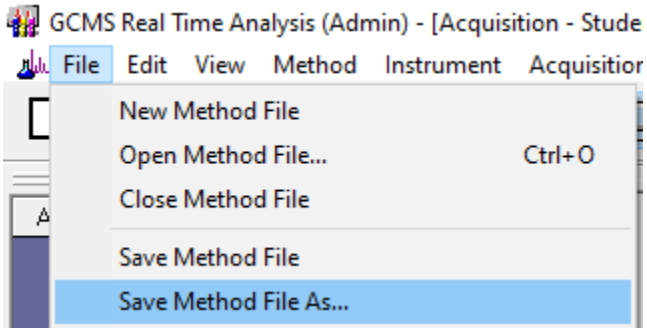

The Start Time for the MS should be set to 3 or 4 minutes, so that the solvent peak has sufficient time to elute before starting to collect MS data:



The Solvent Cut Time is the time when the MS turns on. This should be set to 0.5 minute less than the Start Time. In this example, the Start Time was set to 4.00 minutes, so the Solvent Cut Time should be set to 3.5 min.




After you have modified a method, click on File, and Save Method File As… to save your method:



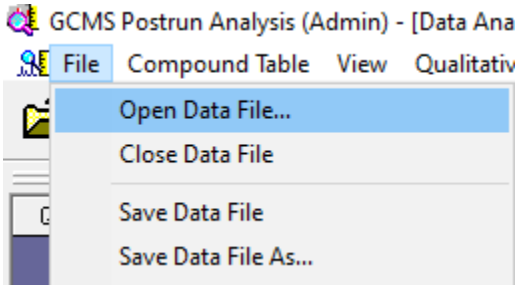
# How To Process Data

If the Data Acquisition program is open, you can click on the Data Analysis button to open the data processing software:


Alternatively, you can double-click on the 

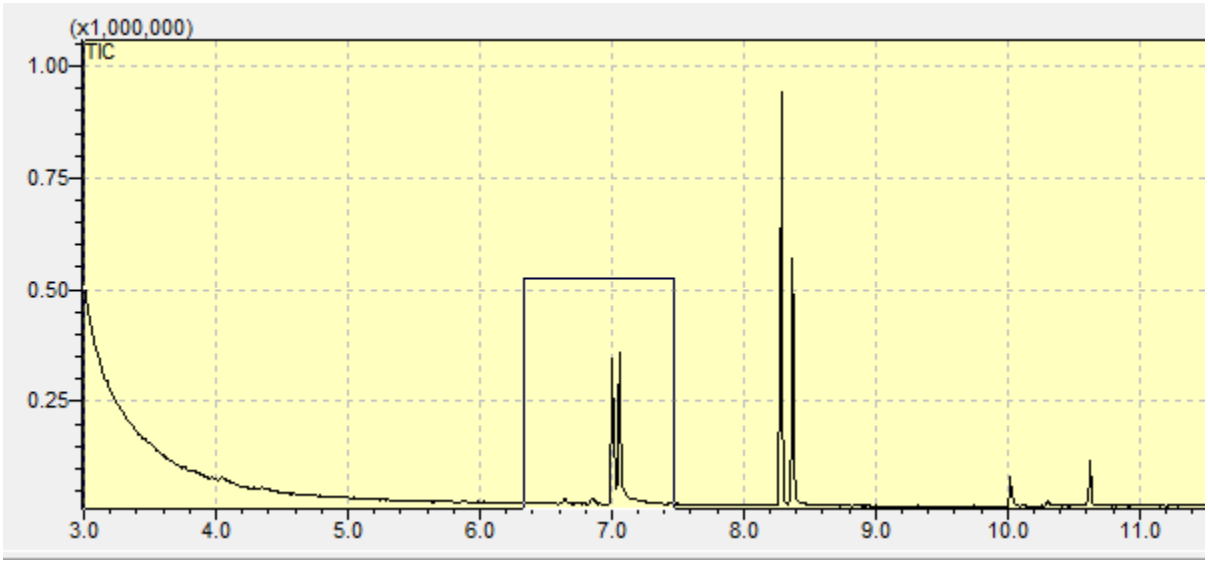
icon on the desktop to open the data processing software as well. There is no fee for data processing. We charge only for the time that the Data Acquisition software is open.

To open your data file, click on File, and Open Data File…

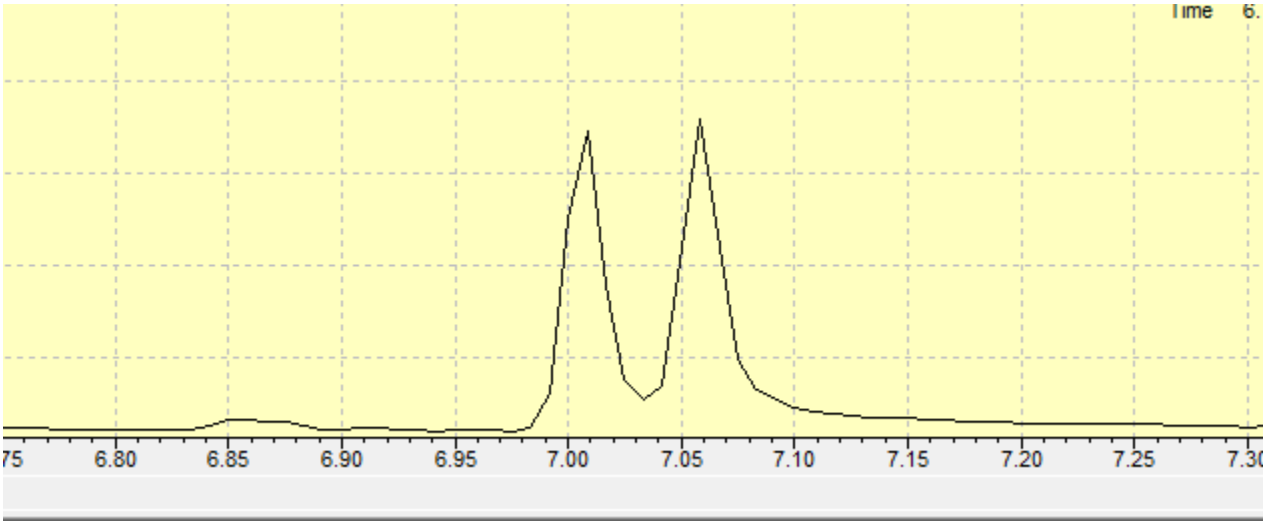


After your data file is open, you should see a TIC Chromatogram (TIC is Total Ion Chromatogram.  It is calculated from stored MS data).

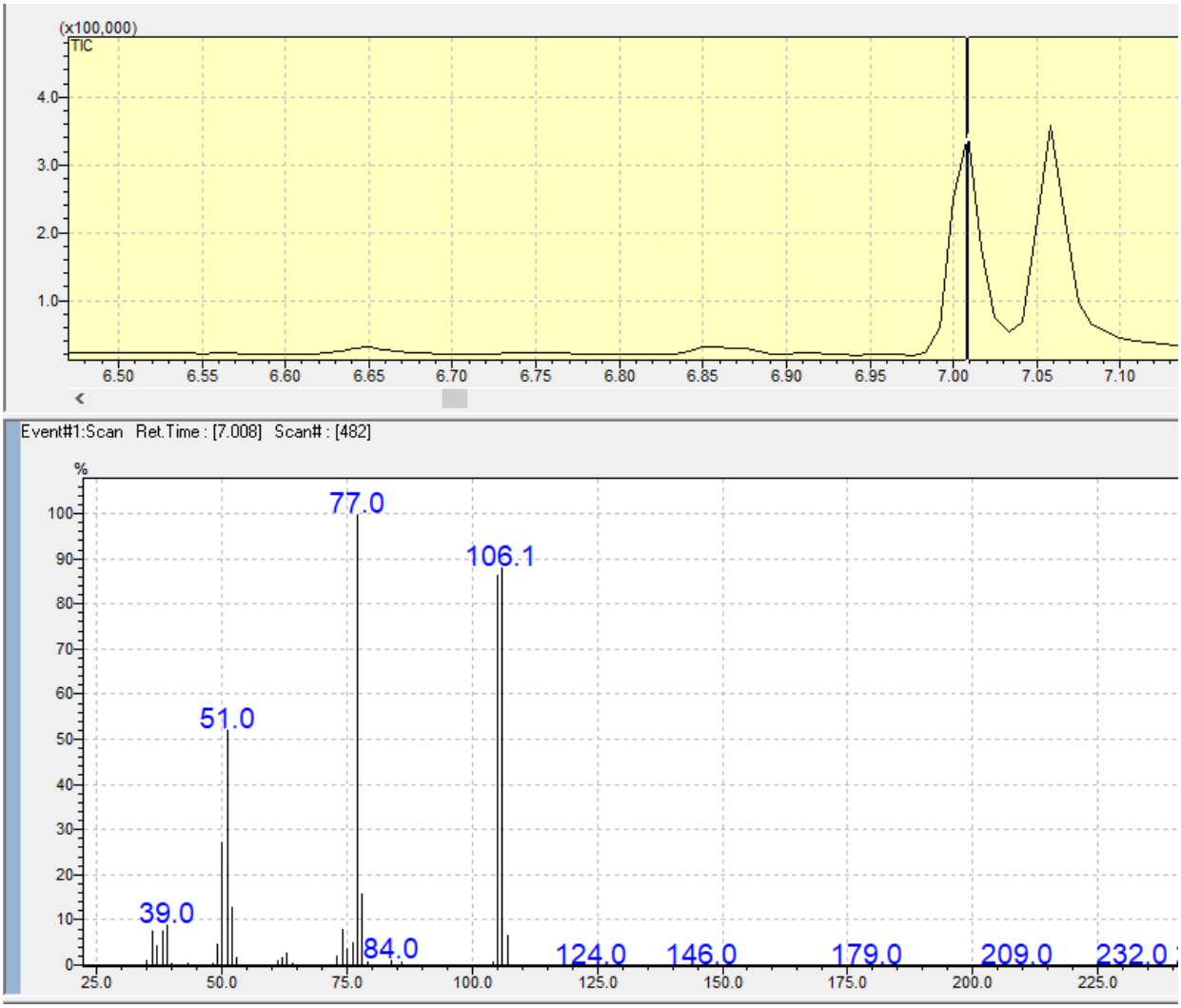
If you want to zoom into a region of the chromatogram, you can left-click and drag a box around the area you want to zoom into:



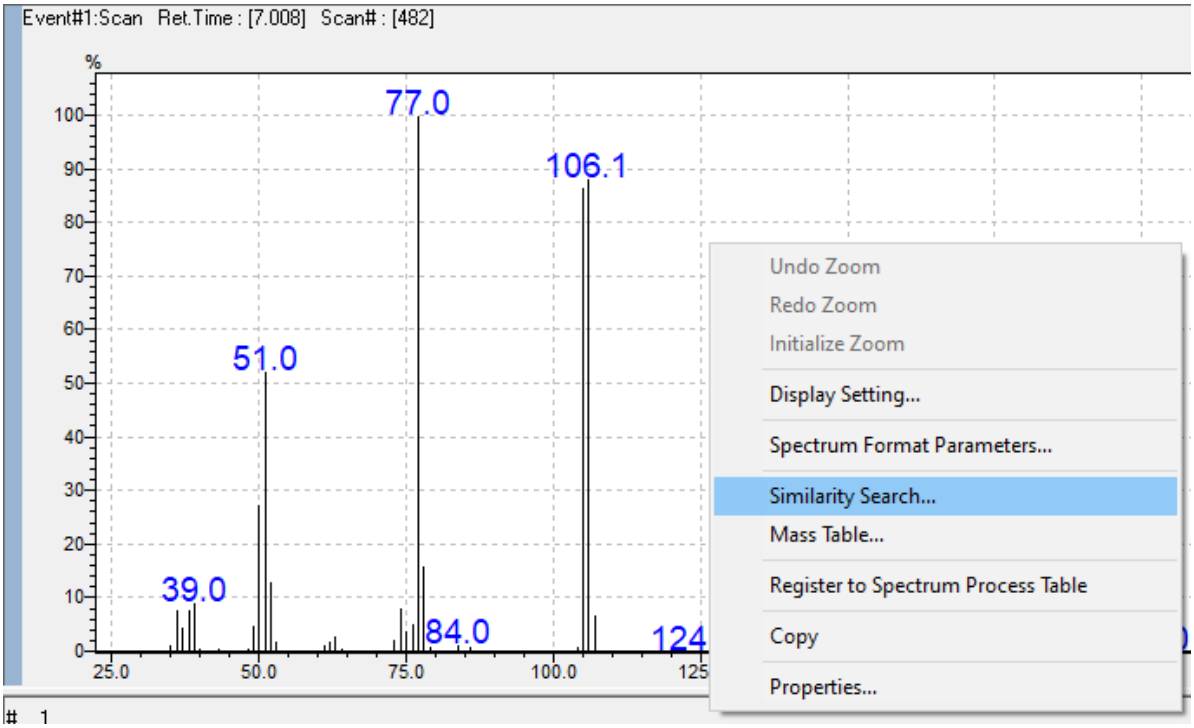

The chromatogram will then show the zoomed-in area:



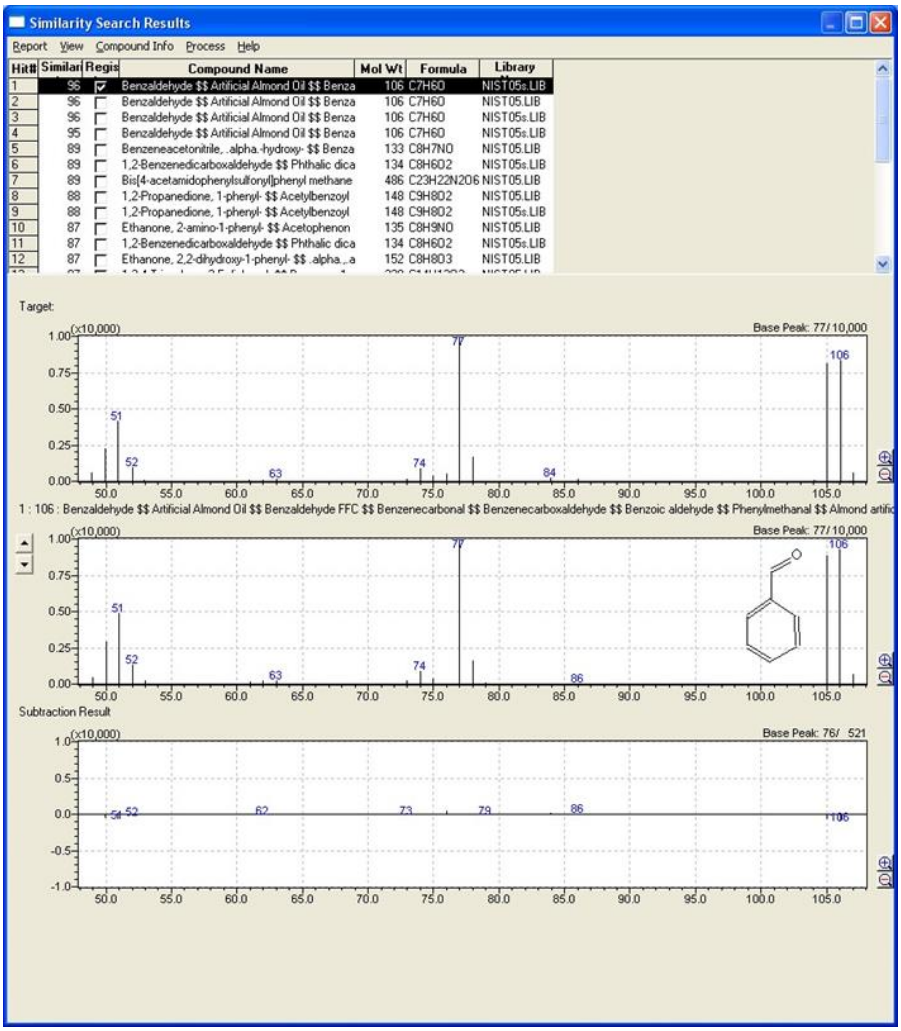
Double-clicking on a GC peak in the chromatogram will show the corresponding mass spectrum:



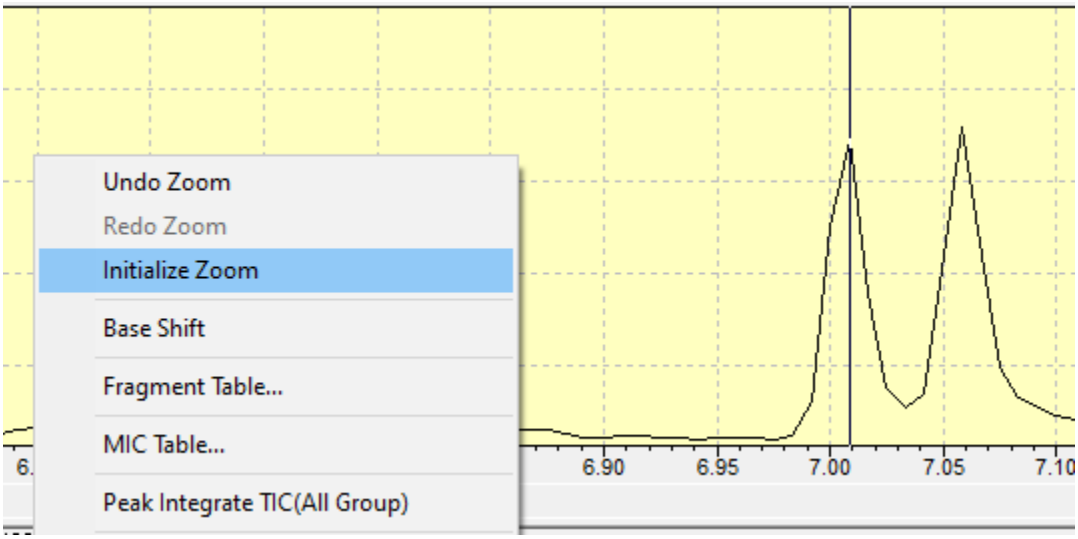
If you right-click on a spectrum, you will see a menu. If you click on Similarity Search…, it will search the NIST database to find the best match to your spectrum:




Here are the results for the Similarity Search:



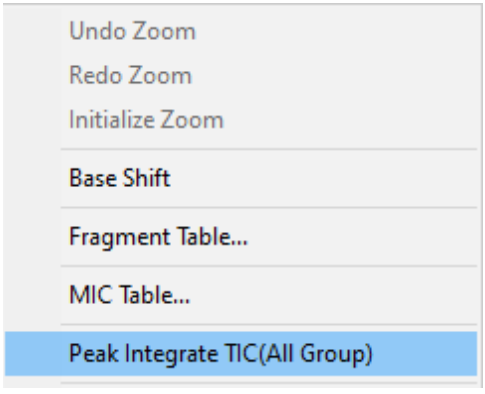
If you want to un-zoom a chromatogram display, right-click the chromatogram to get a menu, and then left-click Initialize Zoom:



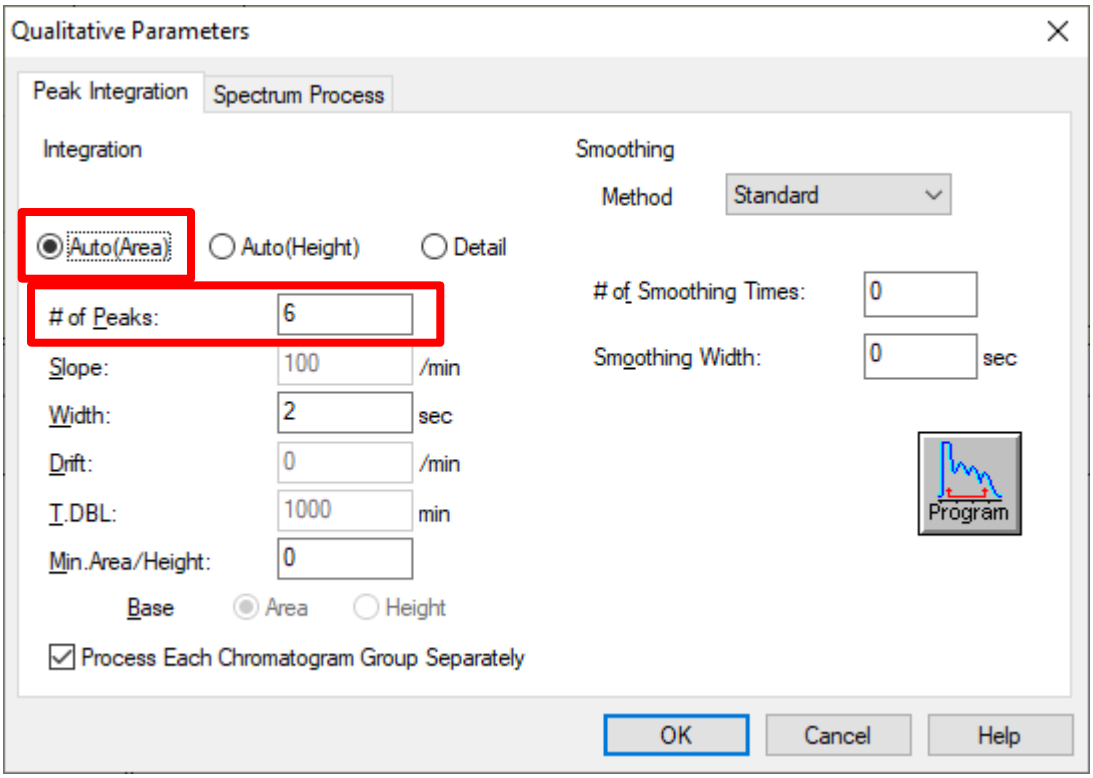

# How to Automatically Process the Data and Generate a Report

You can automatically process all of the major peaks in the chromatogram. For each peak, you can integrate the area under the peak. You can then generate a report that shows the sample method and conditions, the chromatogram, the mass spectrum of each peak, a NIST database search of each peak, and a summary table of all of the peaks.

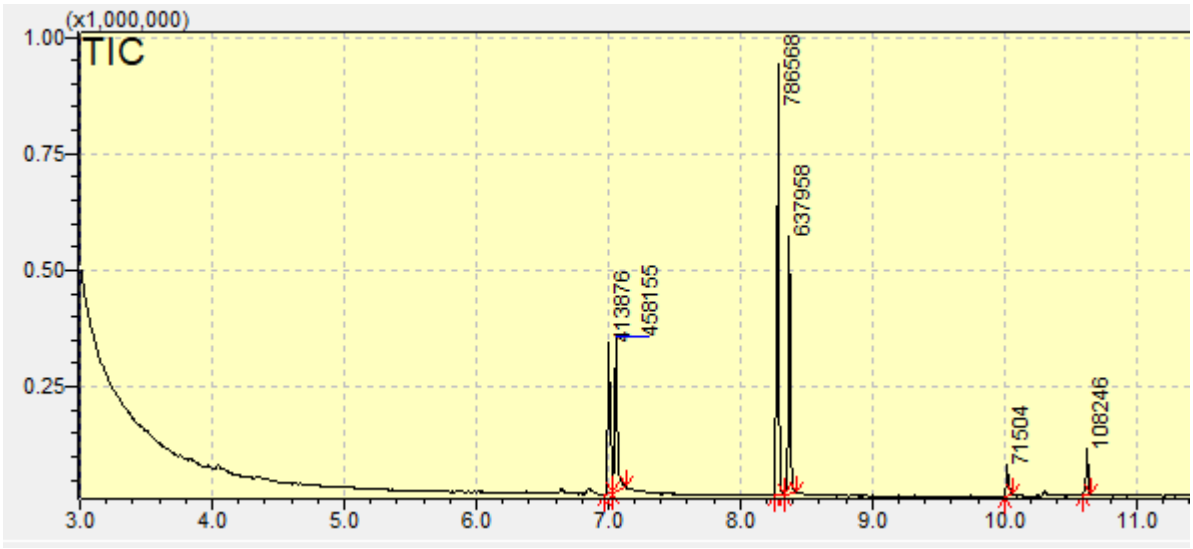
To do this, right-click the Chromatogram to get a menu. Then left-click on "Peak Integrate TIC (All Group):



You will then see a dialog box. Click on Auto(Area), and enter the number of peaks that you want it to process. For example, if there are six major peaks in the chromatogram, you could set this number to six. Then click on OK.

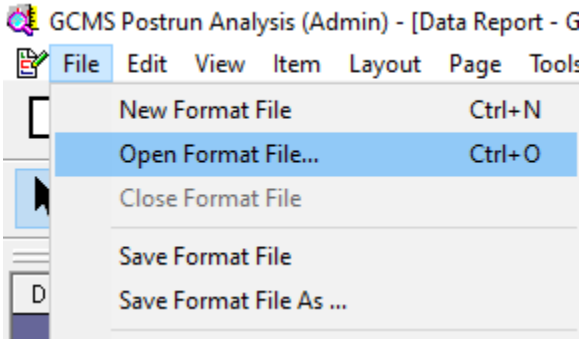



In this case it will automatically choose the six largest peaks, and process each one. The peaks that are processed will show the area under the peak in the chromatogram:



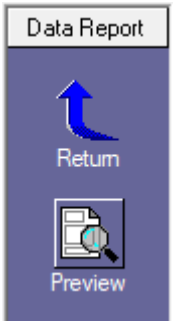
To generate a report, click on the Report icon:


When the Report page opens, click on File, and Open Format File…



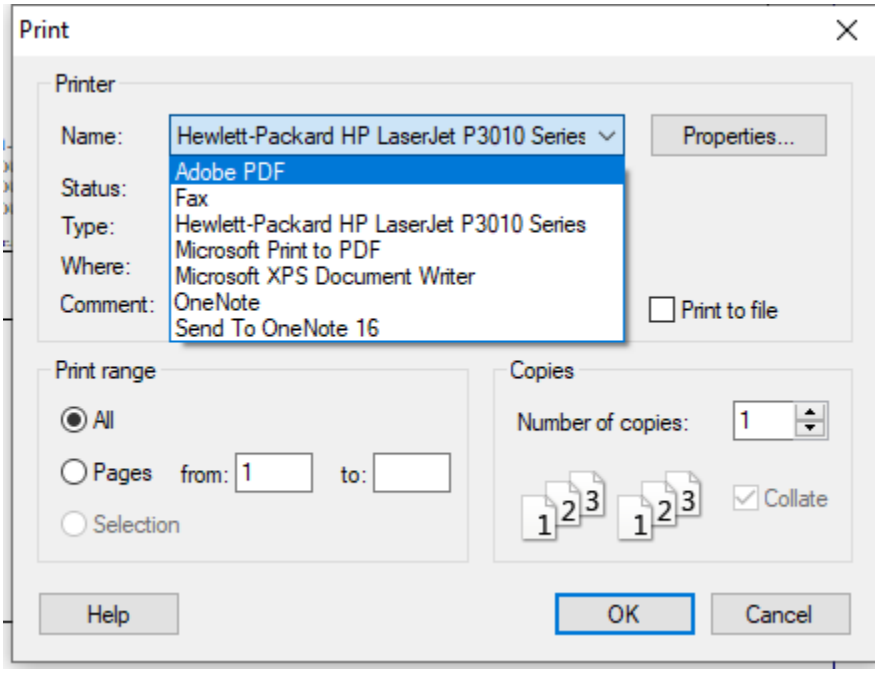

You should then choose the format file called “Student_Default_Report”.
The report will then be generated.

If you want to Preview exactly how the report will appear, click on the Preview button:



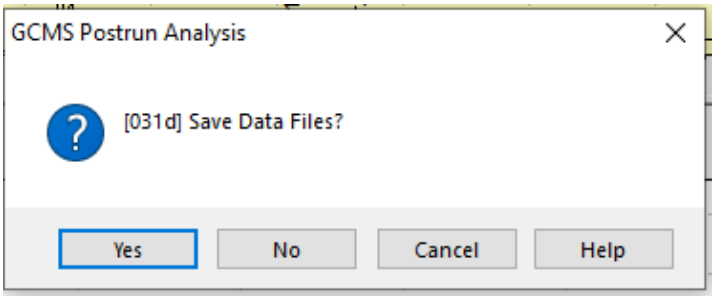

You can scroll through the pages of the report using the Next Page and Prev Page buttons.

If you want to save the report, click on Print. In the drop-down menu for choosing a printer, select “Adobe PDF”:



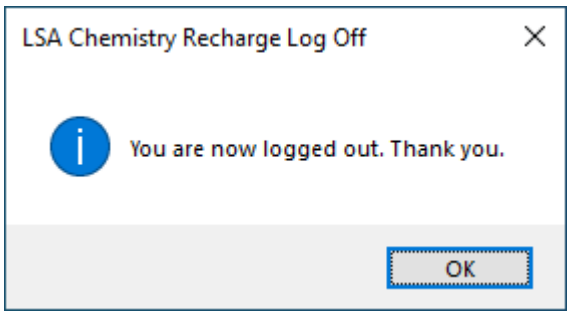

It will then let you navigate to where you want to save the report and what you want to call it.

When you close data files in the Post-Run data processing software, it will ask if you want to save the data files. What it is asking is whether or not you want to save the processing that you have done to the data files.



# How to Shut the Instrument Down When Done

1) Close the Data Acquisition software.  After the program closes, you should see this message:




2) Remove your sample vials from the Autosampler.