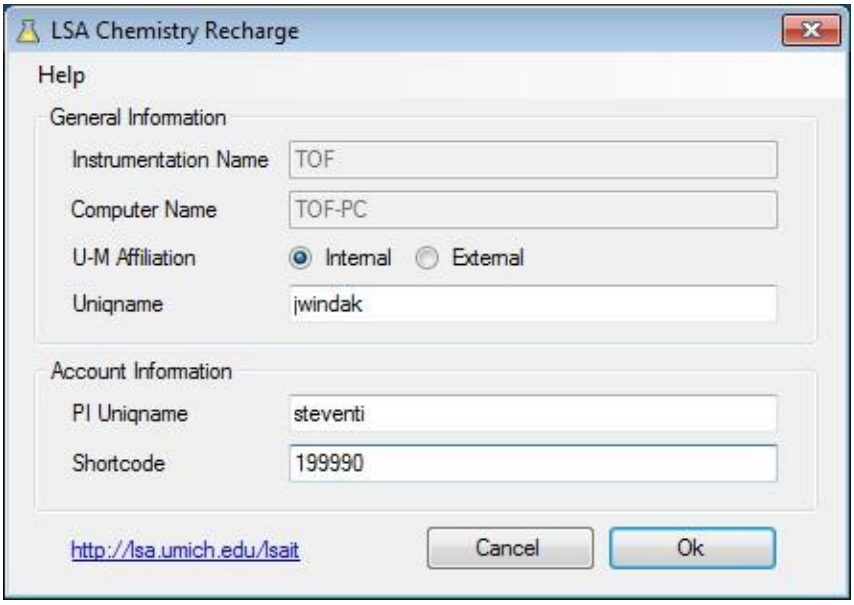
Agilent GCMS User's Booklet

1) Double-click the LSA Chemistry Recharge icon:

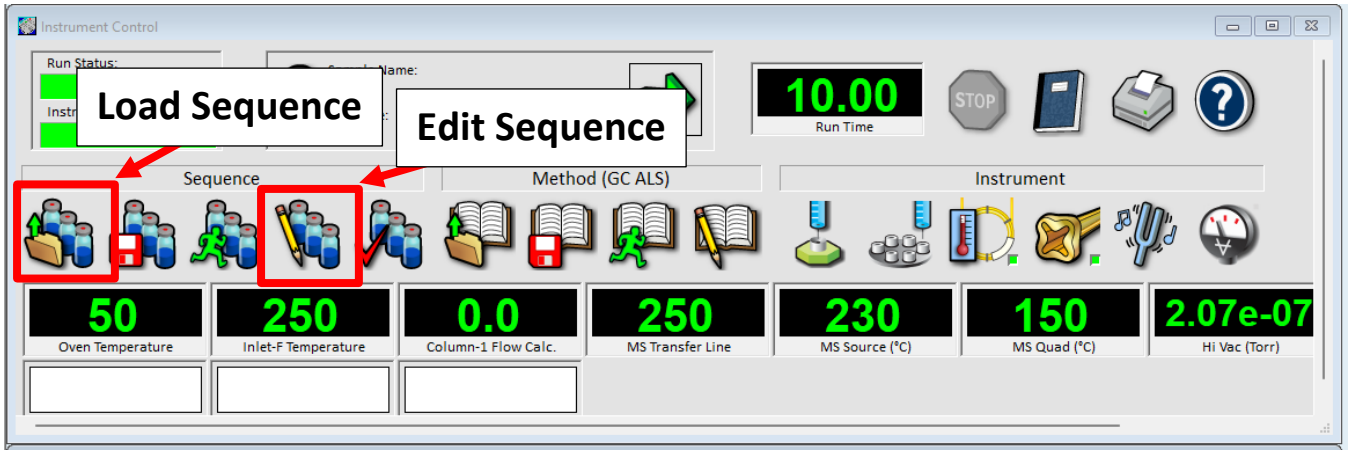


Type in your uniquename, your PI's uniquename, and your short-code account number and click Ok:

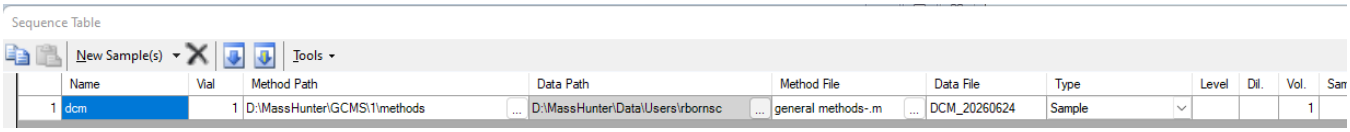


After you login, the data acquisition software will automatically come up.

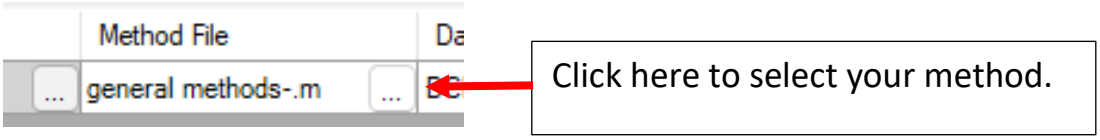
2) After the data acquisition software comes up, you need to set up the *Sequence Table* for running your batch of samples. First click the load sequence icon to load an existing sequence, then click the Edit Sequence icon.



In the *Sequence Table*, you need to fill in one row for each sample that you want to run. In each row, you there are 5 important items you need to fill in.
The Vial is the location where you put the vial in the autosampler tray.
The Data Path is your data folder where you will save your data file.
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 Vol. is the amount in microliters that you wish to inject.

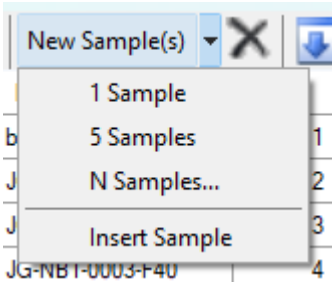


If you need to change the Method File, click the ... icon, you can then Browse to where your method is located and select it.

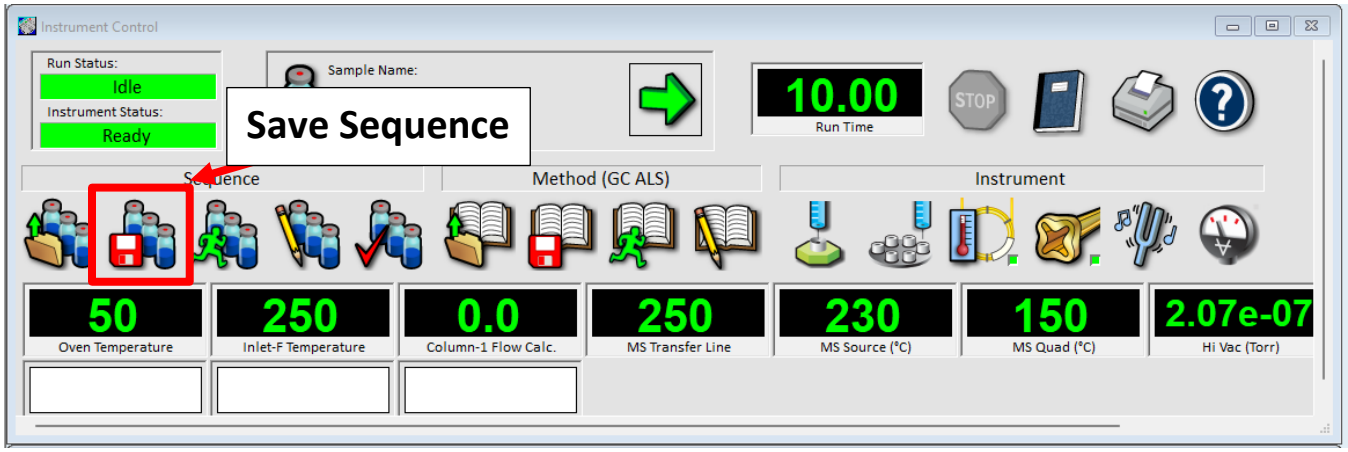


To select where your Data will go, click the ... icon for the Data Path and you can navigate to where you want to put the data, and what you want to name it.

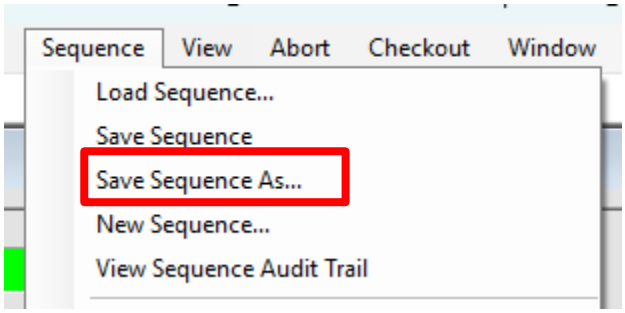
If you need more rows click on New Sample(s) at the top to add a single row. Clicking the icon lets you add multiple samples to the end of the sequence or insert a row into the sequence. If you need to delete rows, highlight the row(s) and click the icon.



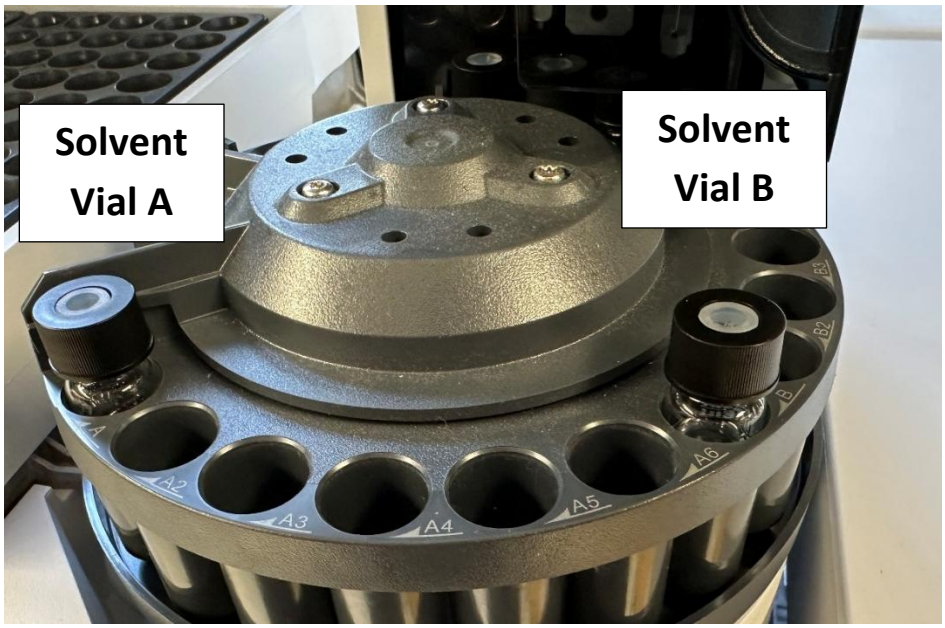
After the table is filled out, select ok and over write the current sequence file by clicking the Save Sequence icon.



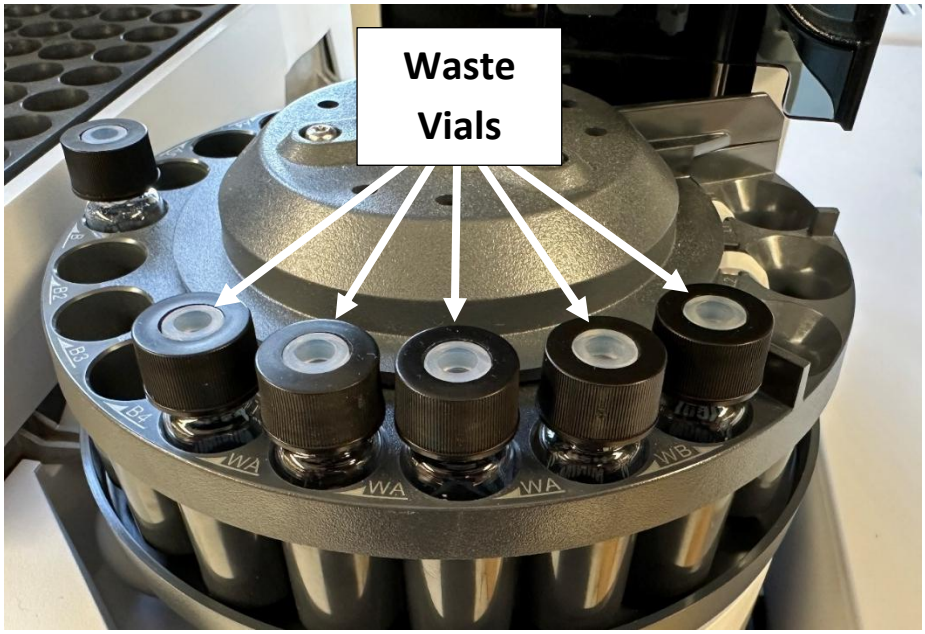
Alternatively, you may “Save Sequence as…” under Sequence in the menu bar.



3) Rotate the solvent tray from the Auto-injector and make sure that there is solvent in the 2 rinse vials. **<u>If the vials are less than ½ full, the syringe may not get cleaned between sample runs. If the 2 vials need to be refilled, please fill them in the hood using the methylene chloride rinse bottle. Make sure to keep open vials in the hood until fully capped.</u>**



Rotate the solvent tray again to verify the 5 waste vials are possitioned appropriately.

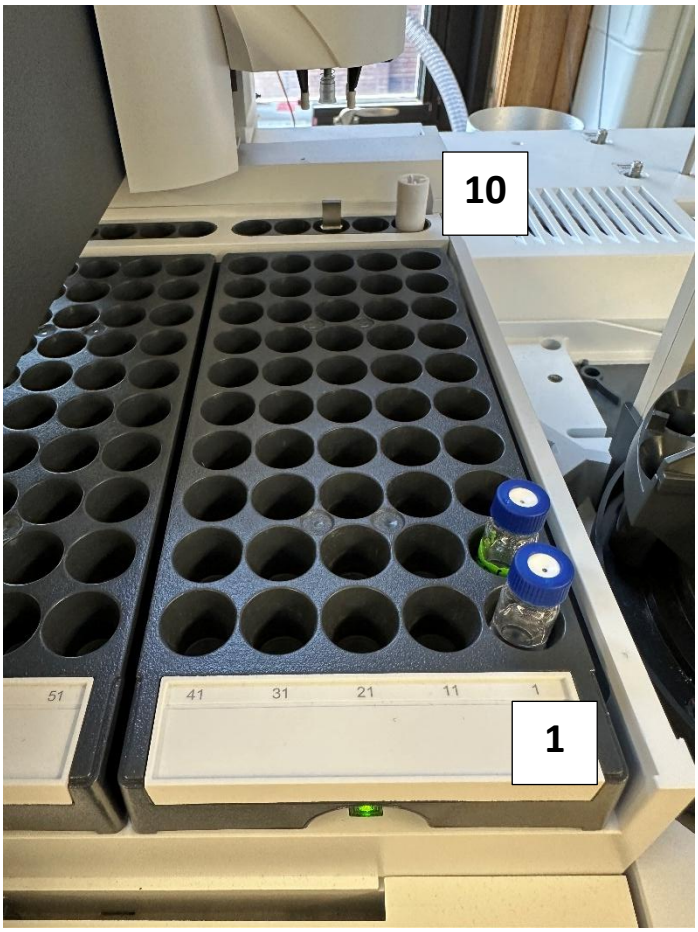


The tray will move to the correct position when the sequence is started.

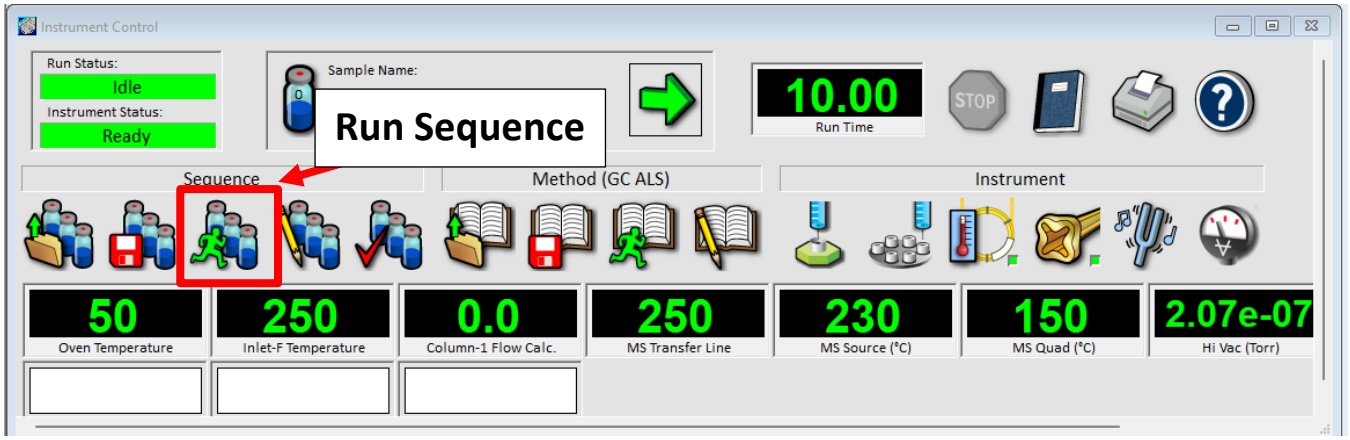
4) Put your samples into sample vials. You need to use a 2 ml autosampler vial with a Teflon-lined cap, such as Fisher Scientific part # 03-391-15.



5) Put your samples into the autosampler locations that you specified in the Sequence Table.



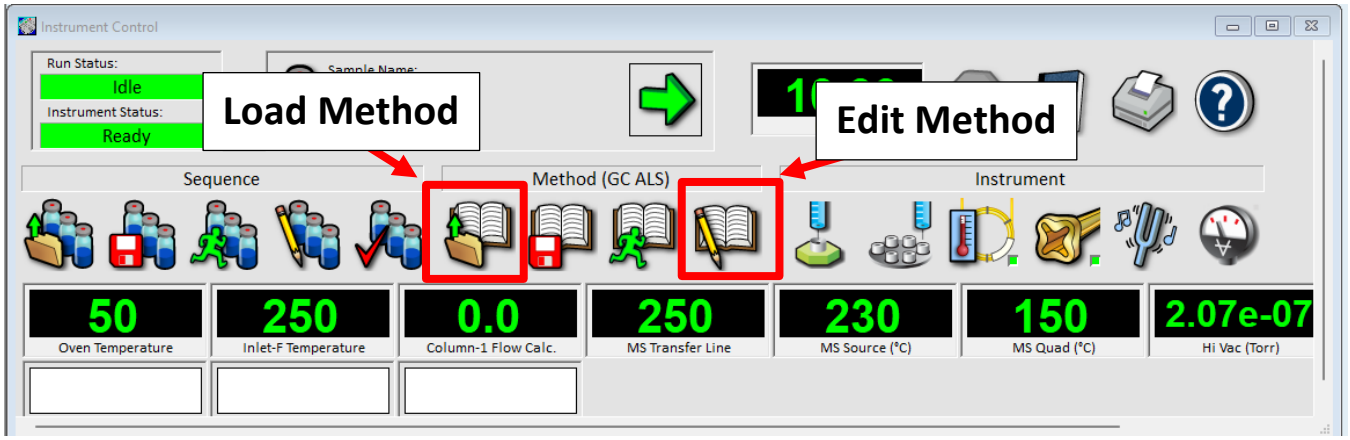
6) Click the Run Sequence icon button to start the run:



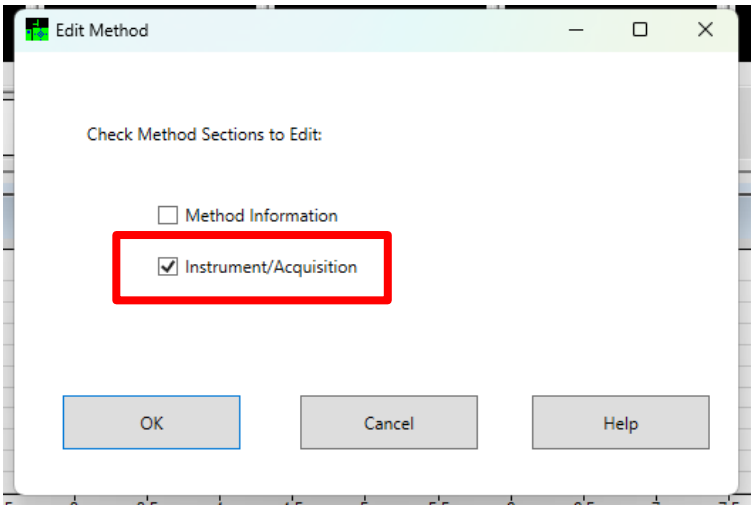
How to Modify a Method

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

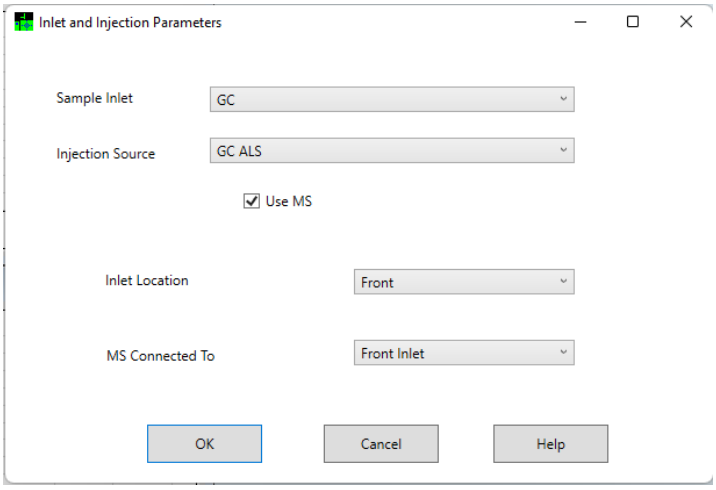
To modify a method, first click the Load Method icon and load an existing method, then click the Edit Method icon.



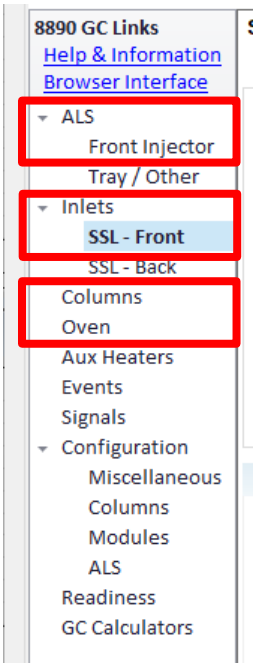
In the pop up window, only select Instrument/Acquisition to edit and click OK:



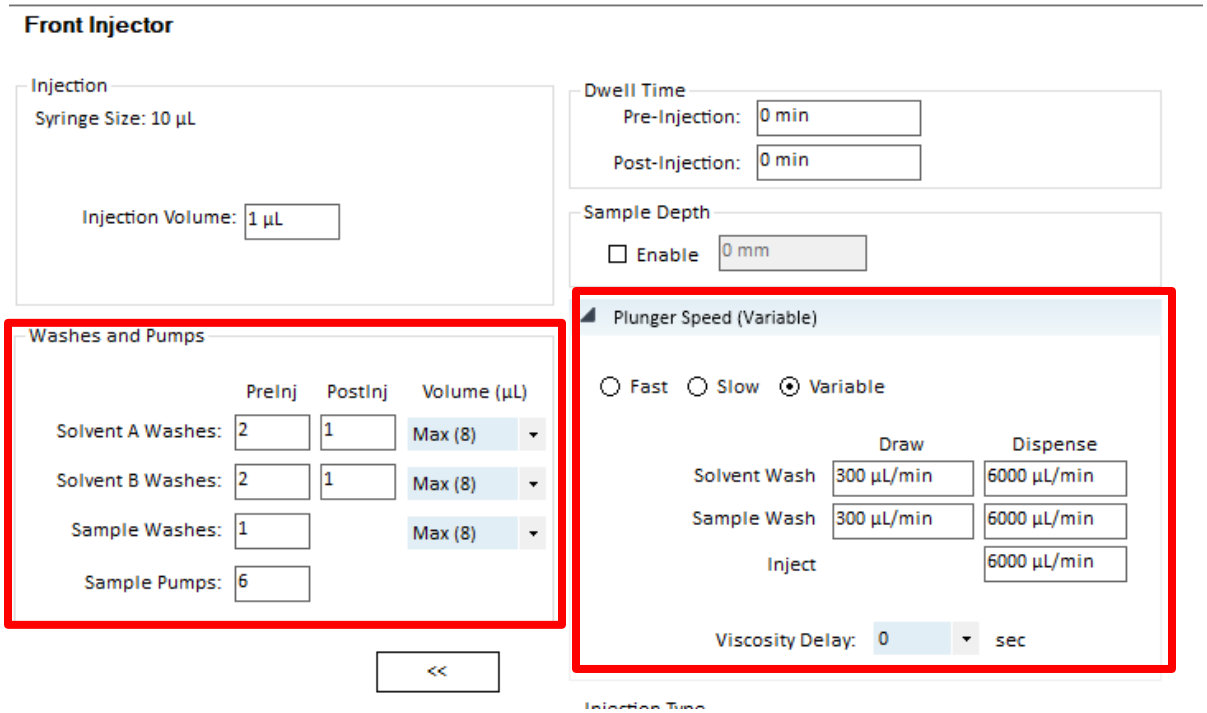
Do not change Inlet and Injection Parameters, just click OK:



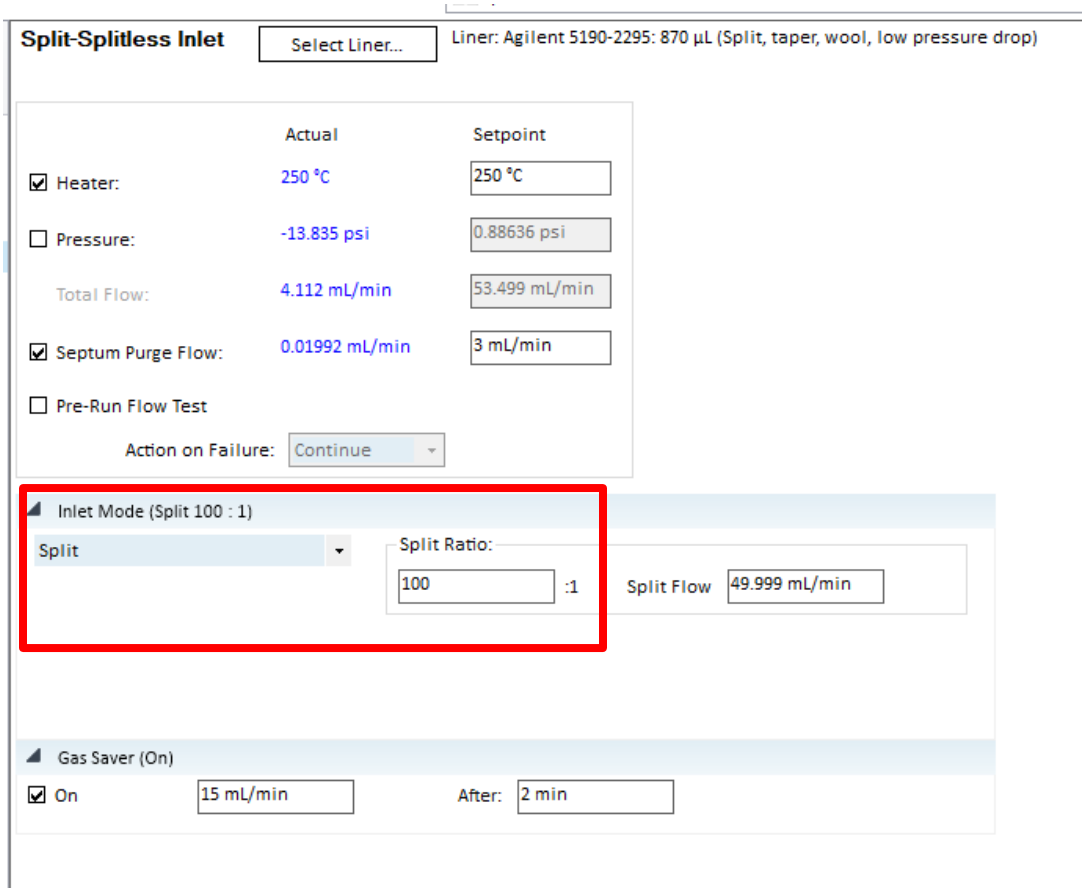
The GC Edit Parameters window will open. Along the left side, GC Links can be selected for different parts of the method. The main options for the GC are ALS: Front Injector, Inlets: SSL – Front, Columns, and Oven.



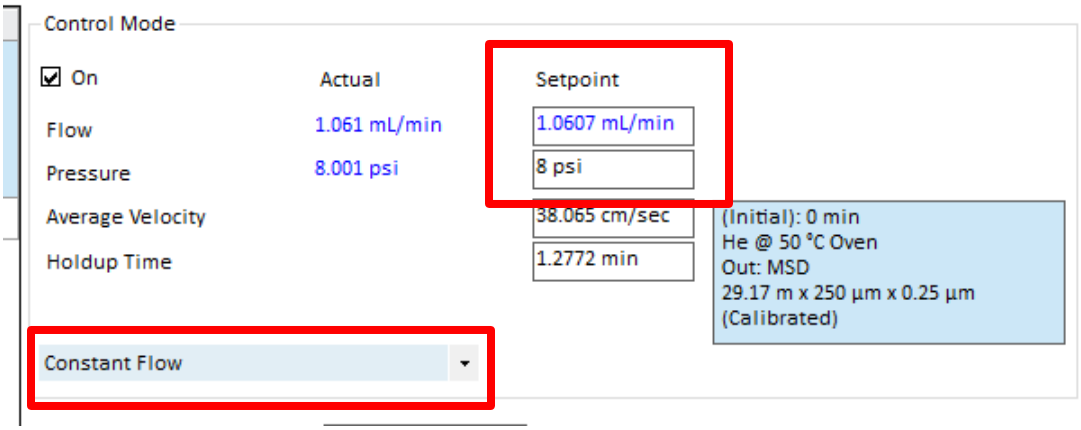
On the Front Injector page, the needle wash conditions and plunger speed can be controlled. Under Washes and Pumps, the number of washes from each Solvent Vial can be changed for pre- and post-injection. Under Plunger Speed, conditions can be adjusted to compensate for viscosity.



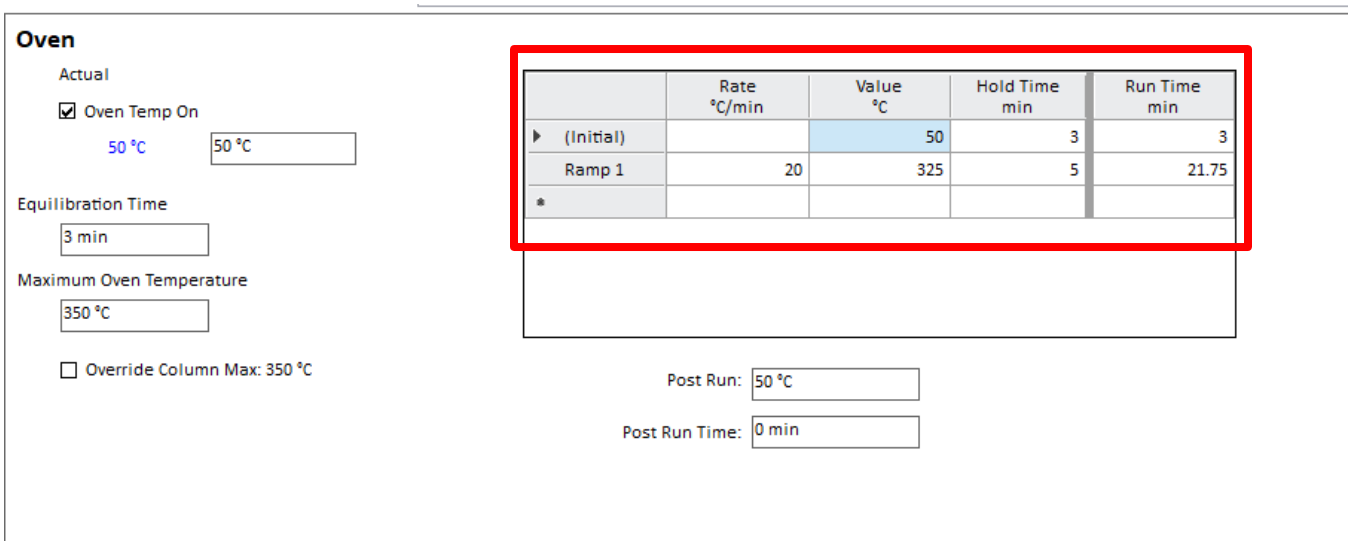
On the SSL – Front page, choose the Inlet Mode, as either Splitless or Split. For very dilute samples (low nanogram per microliter concentrations), you should use Splitless**. For more concentrated samples, you should use Split.** For example, if the sample concentration is 1 mg/ml, use a split injection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



On the Columns page, the gas flow can be controlled. If constant flow is being used, the gas flow rate can be adjusted. If constant pressure is being used, the gas pressure setpoint to be adjusted.

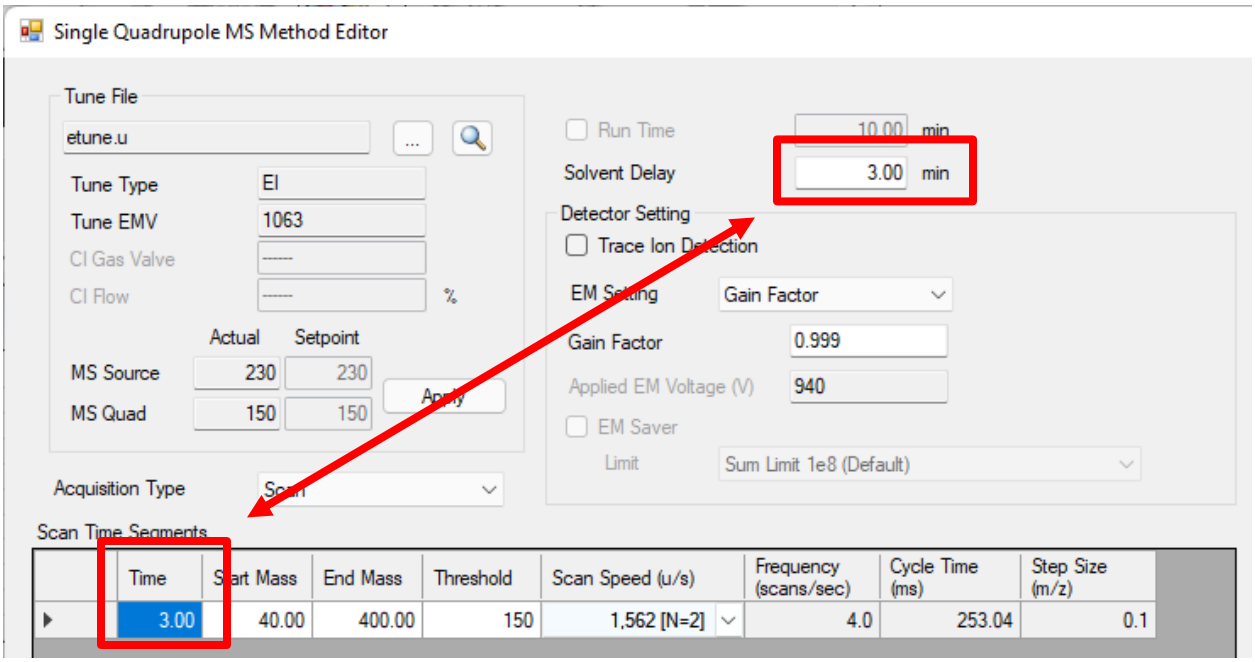


On the Oven page, the GC temperature program can be set with the table. For example, this temperature program holds the GC oven at 50°C for 3.00 minutes. Then it ramps the temperature up to 325°C at 20°C/minute. Then it holds it at 325°C for 5 minutes. The Run Time for this is method is 21.75 min.

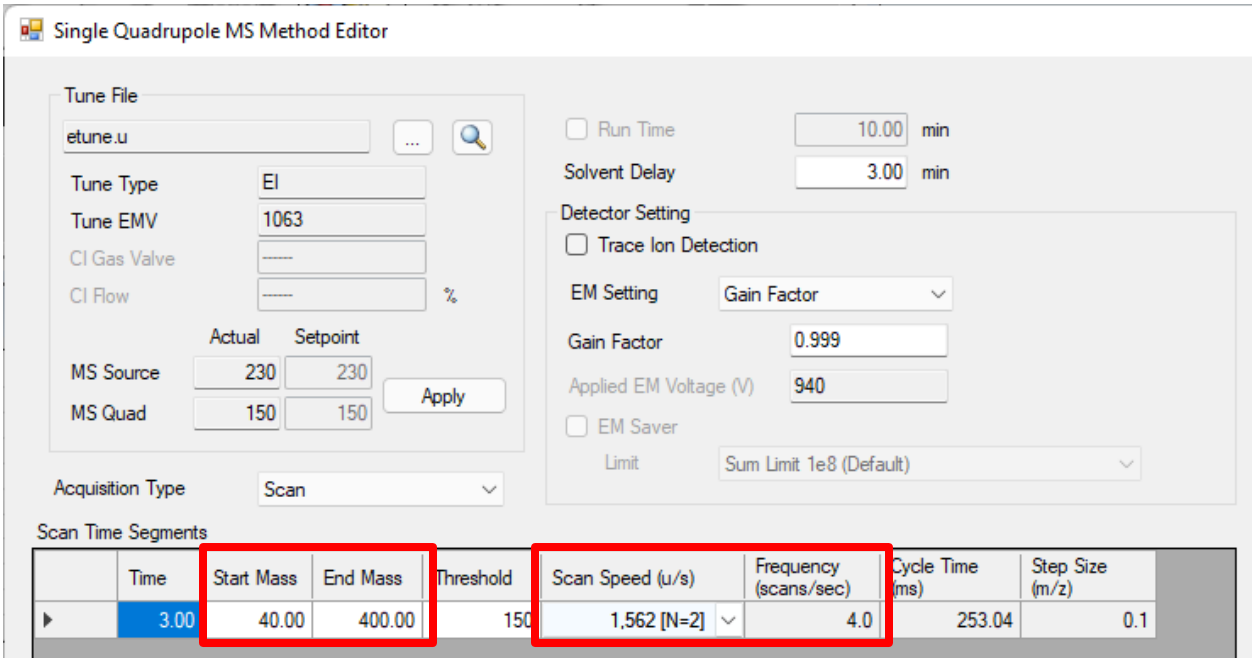


After GC parameters are set, click the Apply button to send conditions to the instrument. Click OK to move to the Real Time Plots page. Select OK again without making changes.

The MS Method Editor window is where the mass spec parameters can be defined. The Solvent Delay should be set to 3-4 minutes; this will automatically update the time in the Scan Time Segments. Do not set the solvent delay to values <3 minutes as it can damage the instrument. An end time does not need to be set.

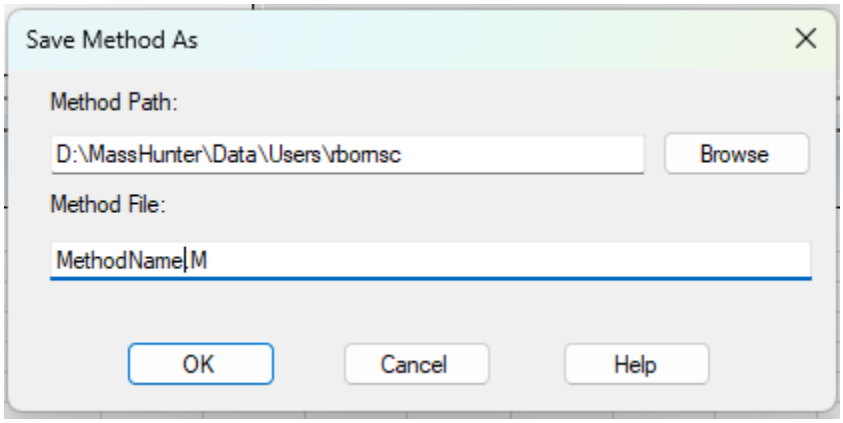


The Start Mass and End Mass define the range of the mass spectrum. Do not set the Start Mass below 35. The End Mass should be minimized to conserved space on the hard drive, but it cannot go above 1091. Once the ranges is set select a scan speed such that the frequency is 2-4 scans/sec.



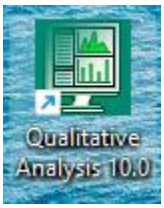
Select OK once parameters are set. Select OK on the Select Monitors page and say No to the “Arrange Monitors?” prompt.

Finally save your method to your user folder.

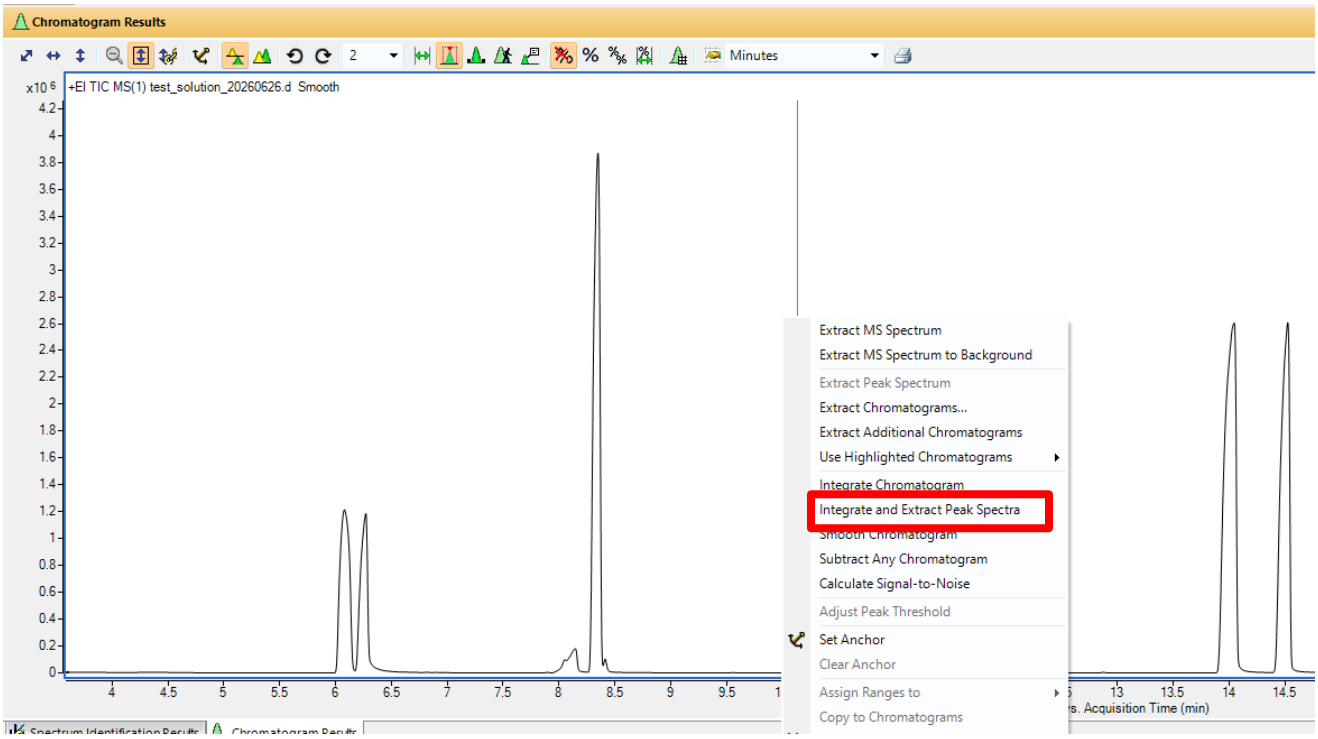


How To Batch Process Data

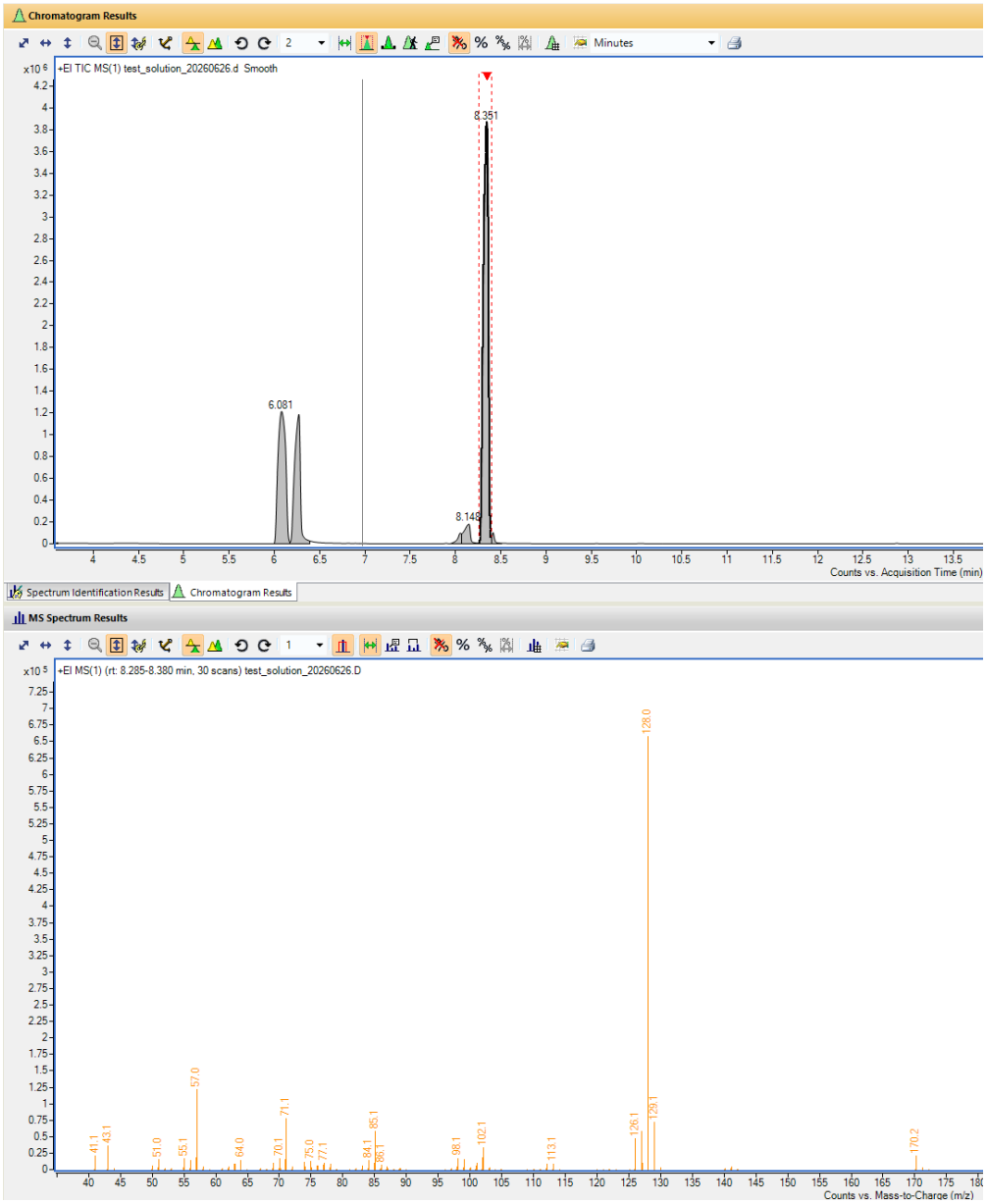
Lauch the Qualitative Analysis application and open your data. If the application is already open, click the File Menu and select Open Data File… Alternatively you may click the  icon to bring up the Open Data File Window.



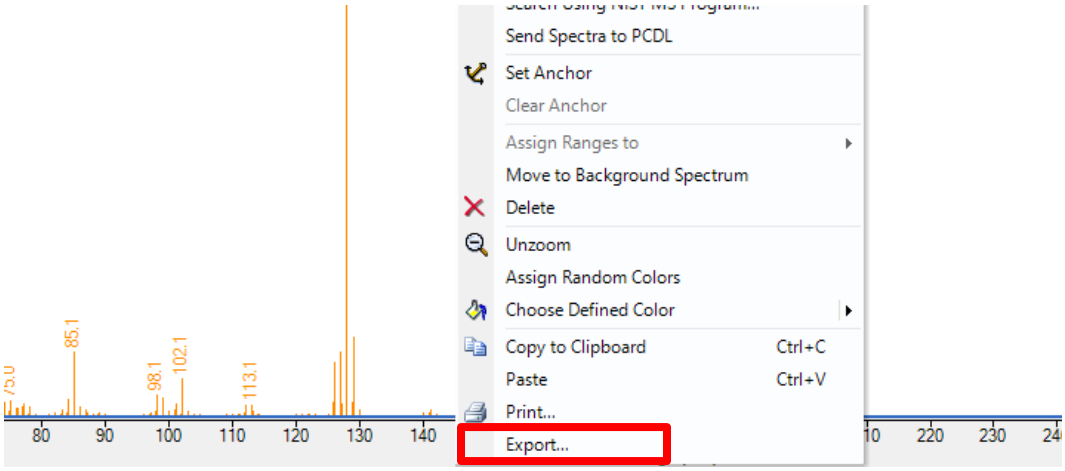
The software will integrate peaks and extract mass spectra automatically. Right-click on the chromatogram and select Integrate and Extract Peak Spectra.



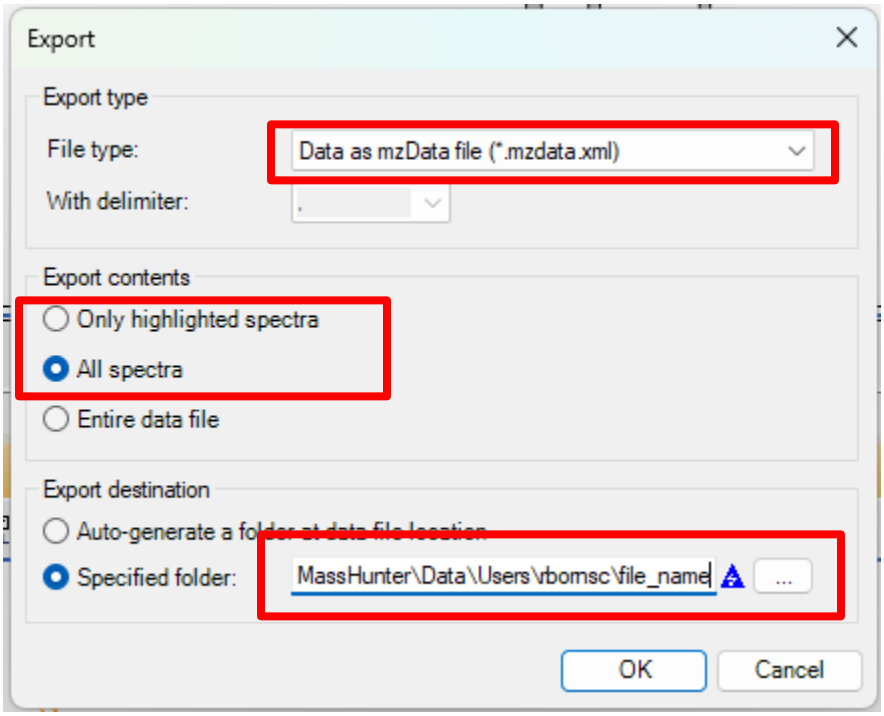
After integration and extraction the selected peaks on the chromatogram will be shaded. Make sure the  icon is selected so that when you select a peak, the mass spectrum will appear in the Spectrum Results Window.



Mass spectra need to be exported as .mzdata files. To export the spectra, right-click on the mass spectrum and select Export.



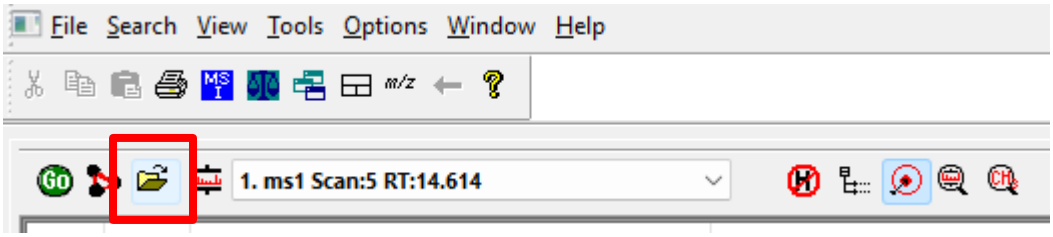
Set the file type to Data as mzData file. You can choose to export all spectra or just the spectra selected on the Data Navigator prior to export. Select Specified folder as the Export destination and click the ... icon to navigate to your data folder. After you have selected your data folder, add the desired name for the exported file to the path. **Do not select the raw data file as your destination. The software will delete the contents of your data file.** Once the export parameters are set, select OK.



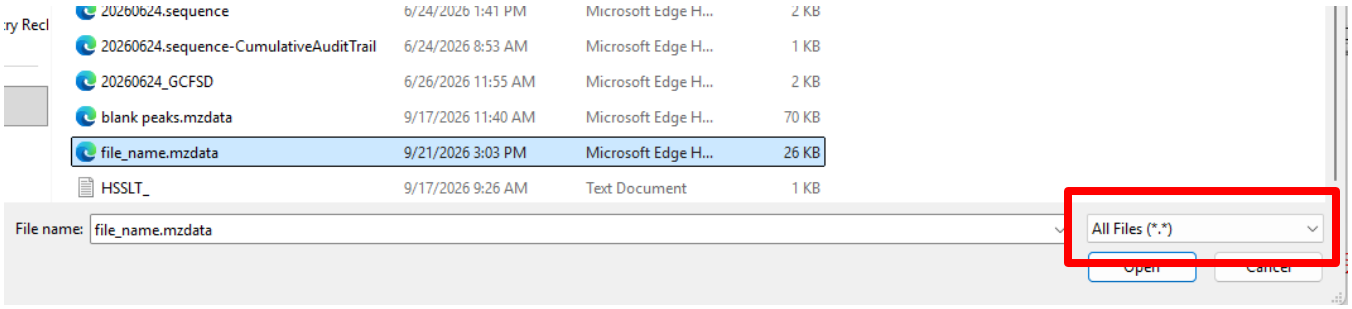
Open the MS Search application from the taskbar.



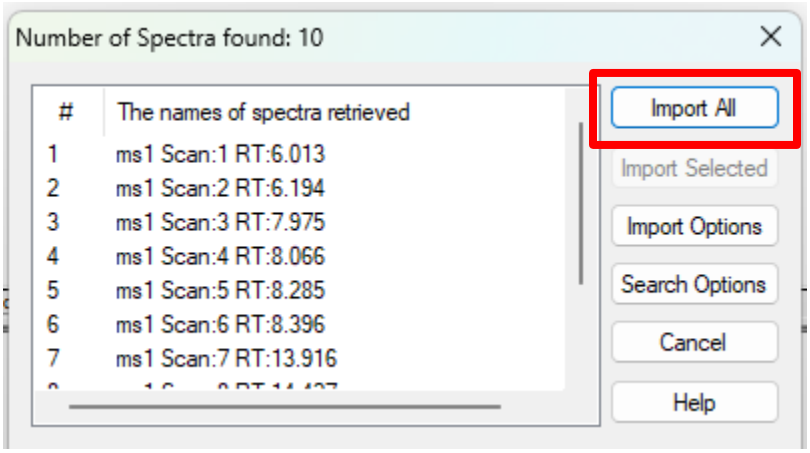
Select the Import icon.



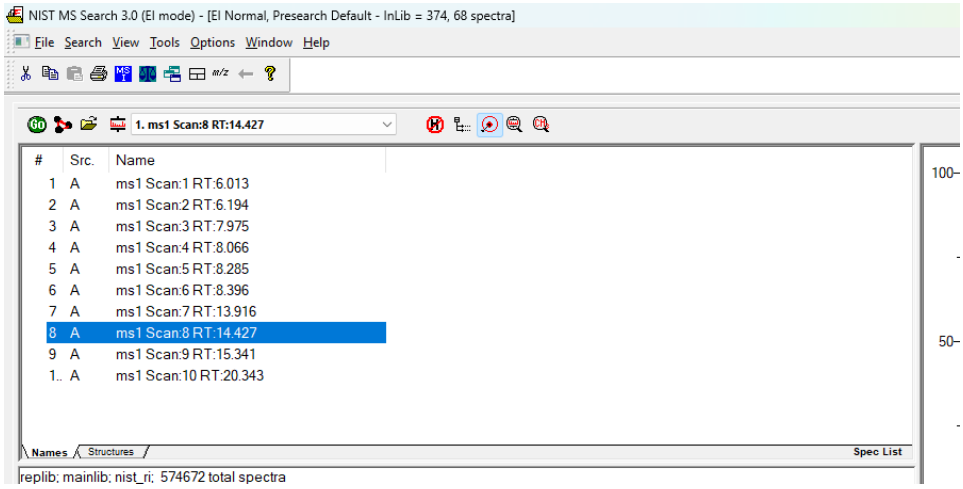
Navigate to your data folder. In order to find your data file, you will need to change the view to All Files. Select the desired file and click Open.



The software will report the number of spectra found in the file that can be imported. You may select specific spectra based on retention time or just click Import All.



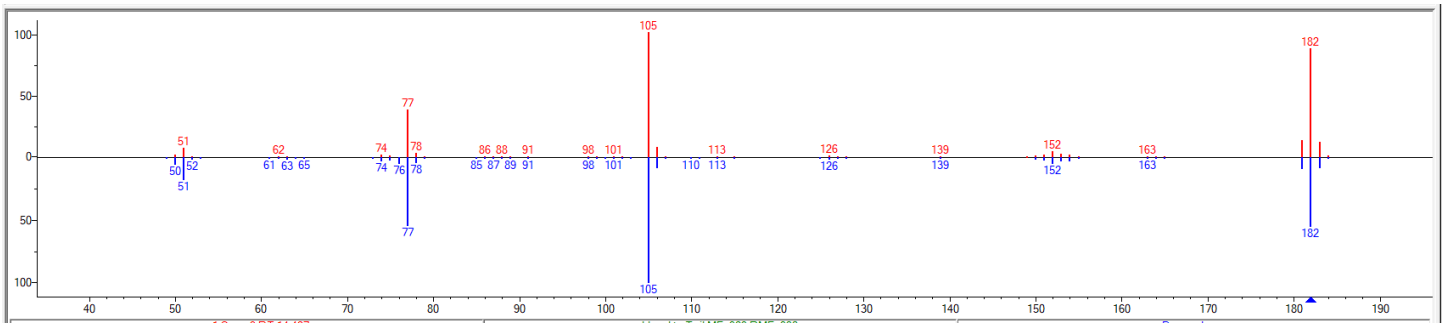
The various spectra and search results can be viewed by double clicking the name from the spectra list on the upper left side of the window.



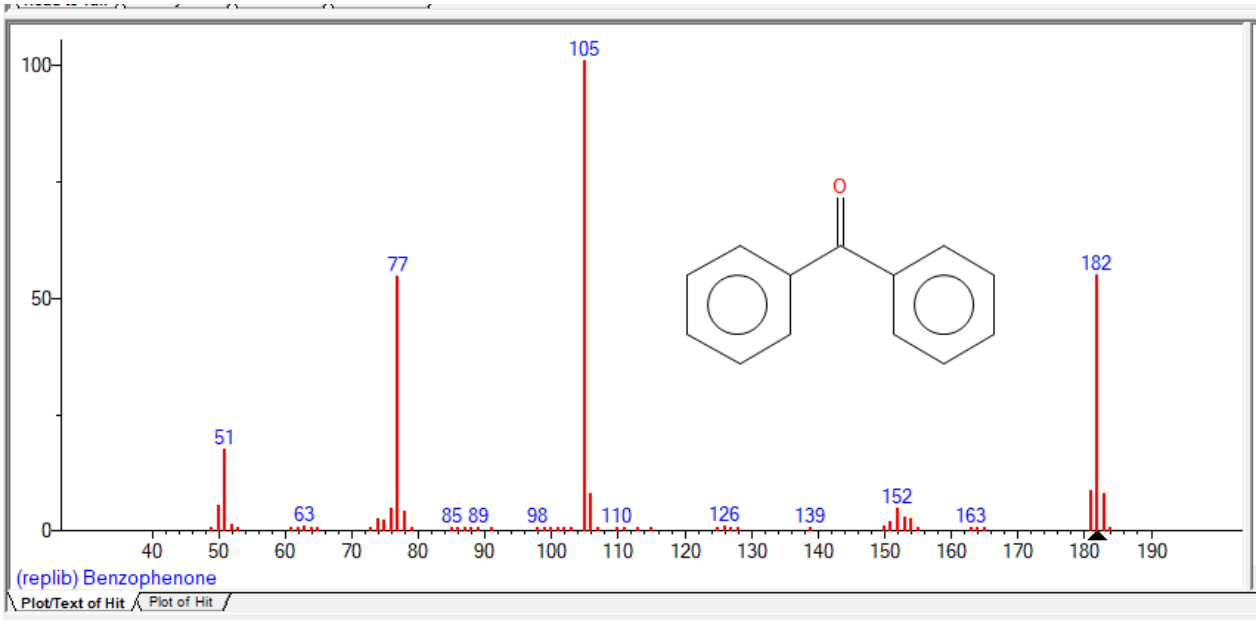
The results table will provide the names and probabilities of various possible matches. Each match can be selected

#	Lib.	Match	R.Match	Prob. (%)	RI	PSS.Match	DBs	Name
⊞ 1	R	929	930	86.8	1635	936	40 ...	Benzophenone
2	M	809	819	4.19	2337*	855		1,2,4-Trioxolane, 3,3,5-triphenyl-
3	M	807	844	3.96	2941*	809		1,2,4,5-Tetroxane, 3,3,6,6-tetraphenyl-
4	M	790	807	2.36	2064*	900		2,5-Dimethyl-7,7-diphenyl-3-aza-4,6-diox...
⊞ 5	R	745	750	0.62	1658*	838	5 E	Benzophenone dimethyl ketal
6	M	737	756	0.49	2458*	867		4-Acetyl-7,7-diphenyl-6-oxa-2,4-diazabic...
⊞ 7	R	732	769	0.42	1607	738	23 ...	Azobenzene
⊞ 8	M	686	689	0.10	2676*	841		1-Propanone, 1,3-diphenyl-3-(phenylami...
9	M	681	682	0.09	2060*	716		3-Oxetanecarbonitrile, 3-amino-2,2-diph...
1..	M	665	666	0.05	2582*	854		Benzamide, N-(2'-benzylphenyl)-
⊞ 1..	R	663	663	0.05	2052*	758	14 ...	Benzoic acid, 2-benzoyl-
⊞ 1..	R	658	690	0.04	1678*	665	8 E...	Phenyl 3-pyridyl ketone
1..	M	656	656	0.04	2032*	745		Benzenepropanoic acid, β-hydroxy-β-ph...
⊞ 1..	R	654	655	0.04	2182*	762	1 G	Tritylamine
⊞ 1..	M	654	654	0.04	1960	674	1 E	Hydracrylonitrile, 2-methyl-3,3-diphenyl-

The experimental and library spectra for a selected match can be compared.

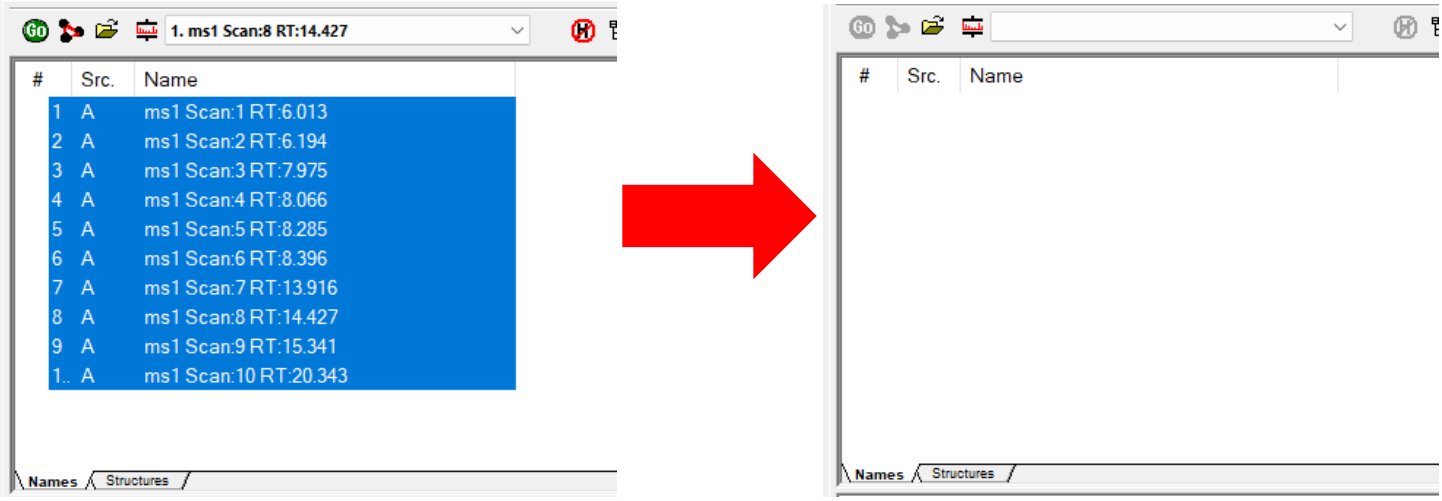


Chemical Structures of the matched compounds can be viewed.



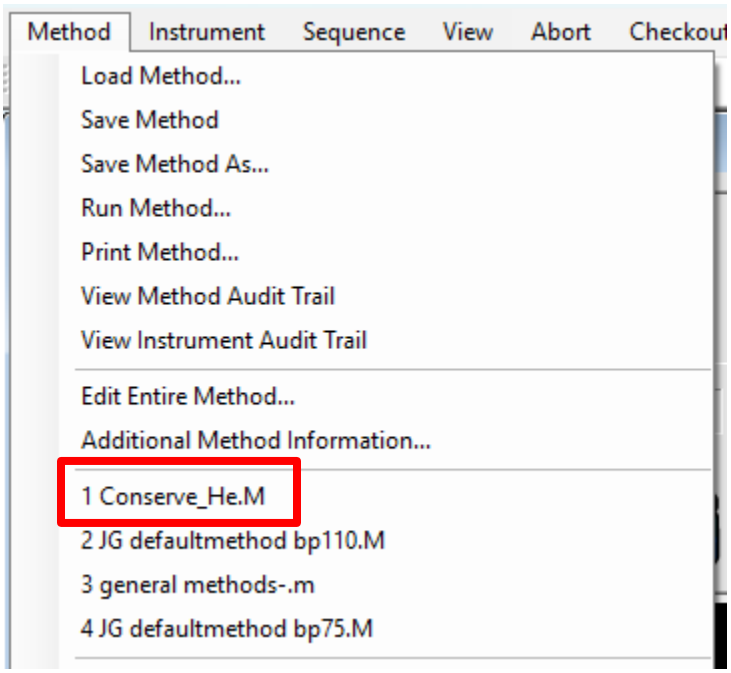
(Optional)

Once you are done processing the data, it can be cleared from the software by selecting all (ctrl+A) the spectra from the spectra list and hitting the “delete” on the keyboard. The search history can also be cleared by clicking the icon.



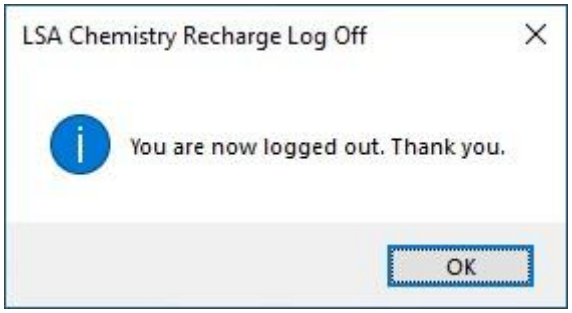
How to Shut the Instrument Down When Done

1) Go to the Method menu in the menu bar and select the “Conserve_He.M” method. This will automatically export low-flow conditions to the instrument.



If the method is not on the list of recent methods, select Load Method… and find the method in D:\MassHunter\GCMS\1\methods.

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



3) Remove your sample vials from the Autosampler.