Shimadzu GCMS QP2010 SE Standard Operating Procedure

Rochester Institute of Technology

Department of Chemistry and Material Science

SOP prepared by Andrew Layman on 6/23/15

SOP owned by Tom Allston

I. Purpose

To promote the effective use of the Shimadzu GCMS-2010 Gas Chromatography Mass Spectrometer, to establish an instrumental method, and to analyze the chromatogram obtained.

II. Scope

This SOP is intended for in-group use by trained and certified personnel in the Chemistry Department.

III. Prerequisites

This experimenter must be trained in proper instrument techniques before using this SOP

IV. Responsibilities

The responsibility for this instrument lies with Tom Allston

Room: 08-A174

Voice: 585-475-6034

Fax: 585-475-7272

E-mail: tdasch@rit.edu

School of Chemistry and Materials Science

85 Lomb Memorial Drive

Rochester Institute of Technology, Rochester, NY, 14623-5603

V. Procedures and Information

1.) Make sure that the GC-MS is switched on, then turn the computer on and log in.

   a.) Make sure the user name is…, and there is no password so just hit ok.

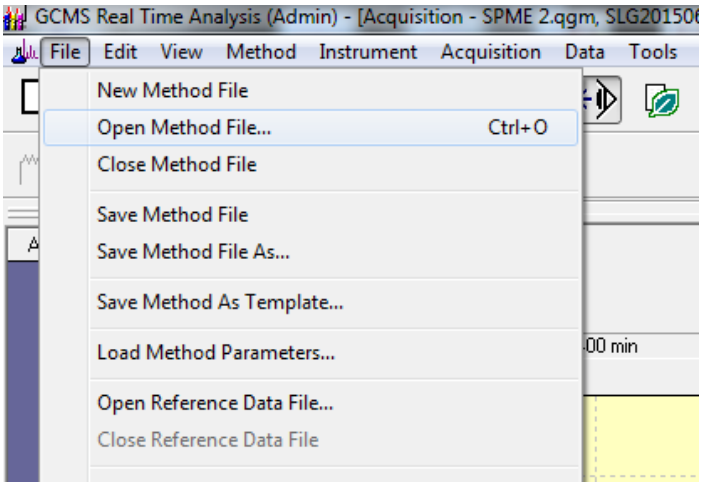
2.) Connect and Start the Instrument.

   a.) On the windows Desktop, double click on the "GCMS Realtime Analysis" tab. This will open the Realtime Analysis window.

   b.) Once this new window comes up click on the "Data Acquisition" tab.

3.) Loading a Preexisting Method

a.) Go to “File” \ “Open Method File” then select the previously saved method from the folder that it resides in.

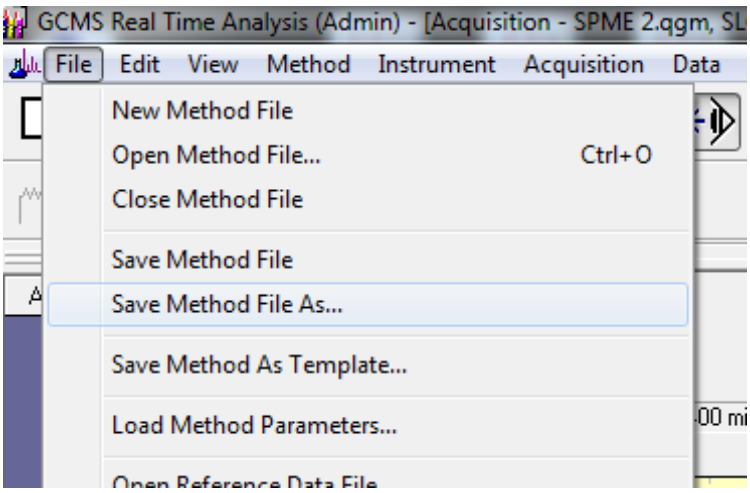


b.) This will open the previously saved method and its parameters can be edited if desired.

c.) To resave your method go to “File” \ “Save Method As”

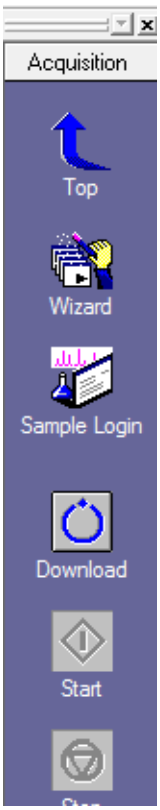
i.) Be sure to create YOUR OWN folder. This will ensure that all your data will be kept in one location.

ii.) Note: The chemistry department does NOT take responsibility for your data; you must back up any data file that you do not wish to be deleted.

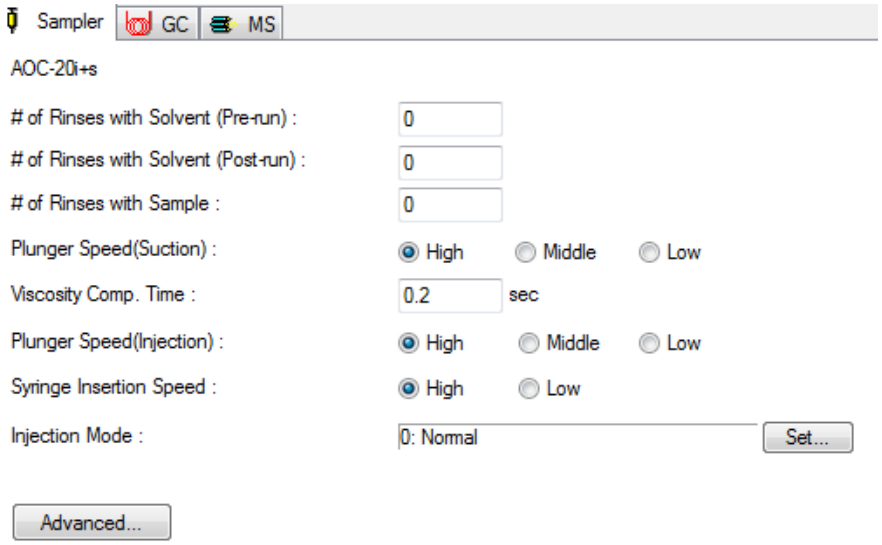


4.) Creating a New Method File

a.) Click on the "Data Inquisition" tab, this will allow you to start creating your method.

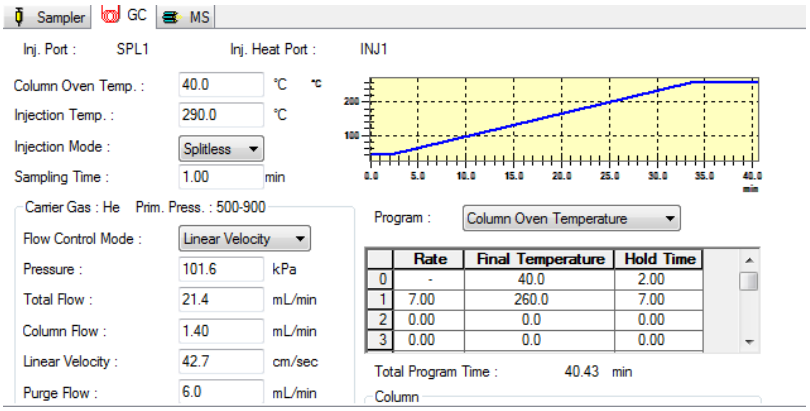


b.) First select the "Sampler" tab and make any changes necessary. If you wish to wash the injector before and after injections, make sure to place the proper solvents in the injector tray.

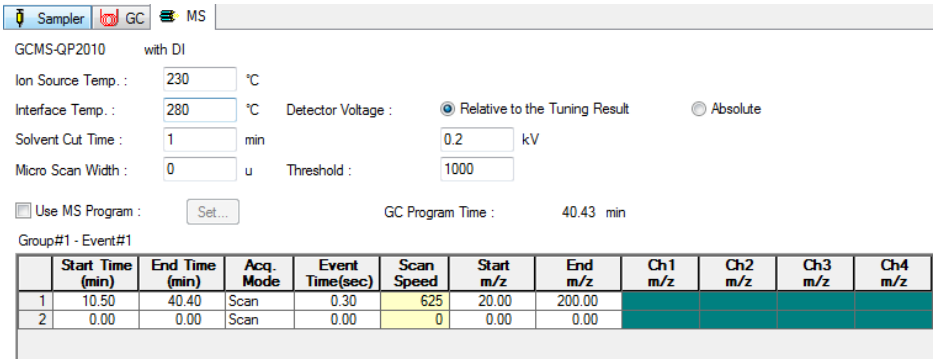


c.) Next click on the "GC" tab. This will allow you to change the injection temperature, the temperature of the column oven, as well as change the pressure going through the column.

i.) Make sure that the injection method matches the injector that is in the GC. If unsure ask Tom Allston.



d.) Finally select the "MS" tab. This will allow you to change the temperature of the Mass Spec detector as well as what ions to look for and at what times during the run you want the Mass Spec to scan.



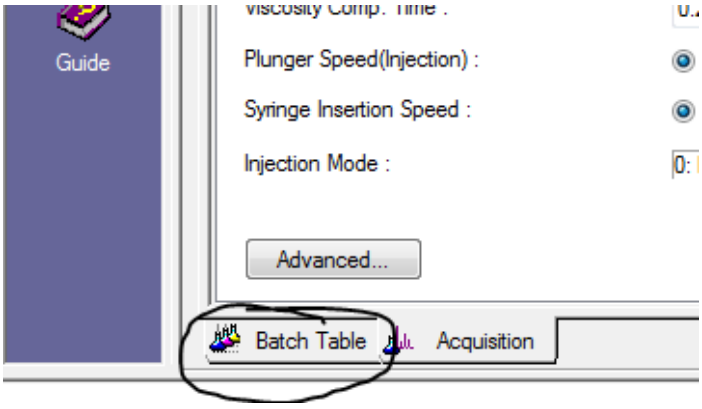
e.) When all of the parameters are set how you want them you want to save the method by going to "File" \ "Save Method As". Save your file in a place where it can easily be retrieved.

5.) Running Samples

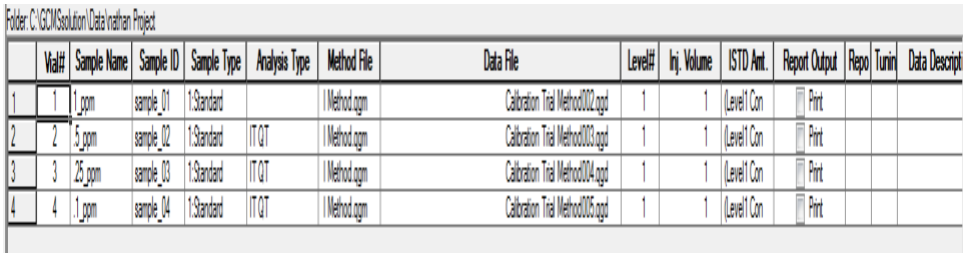
a.) Insert your samples into the autosampler rack taking careful note of the numbered slot that your samples are in.

b.) Click on the "Batch Processing Table" tab. This will open a table where you can input the name of your samples, whether or not the sample is

a standard, the method file used, and the data file that it will be stored in.



c.) Once the method and batch table are all set, hit the "Start" tab. This will download your method parameters and the processing of your samples will begin.


Folder: C:\GCMSsolution\Data\nathan Project

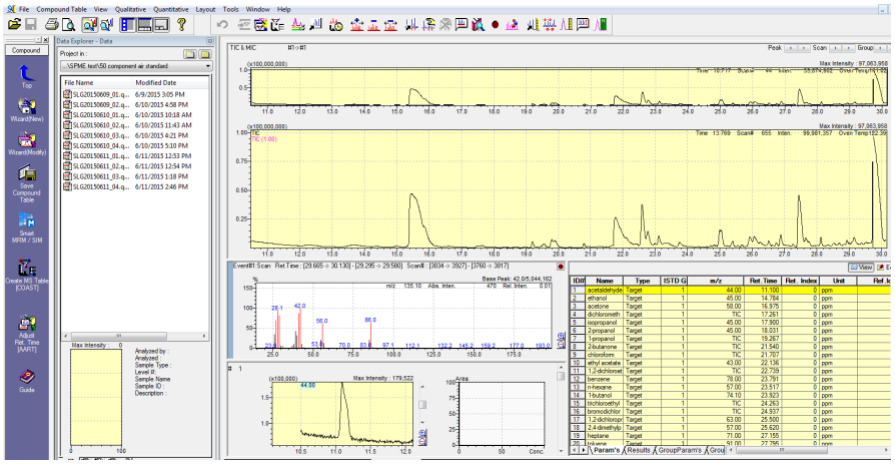
	Vial#	Sample Name	Sample ID	Sample Type	Analysis Type	Method File	Data File	Level#	Inj. Volume	ISTD Amt.	Report Output	Repo	Tunin	Data Descript
1	1	1_ppm	sample_01	1:Standard		I Method.qgm	Calibration Trial Method002.qgd	1	1	Level 1 Con	Print			
2	2	5_ppm	sample_02	1:Standard	IT QT	I Method.qgm	Calibration Trial Method003.qgd	1	1	Level 1 Con	Print			
3	3	.25_ppm	sample_03	1:Standard	IT QT	I Method.qgm	Calibration Trial Method004.qgd	1	1	Level 1 Con	Print			
4	4	.1_ppm	sample_04	1:Standard	IT QT	I Method.qgm	Calibration Trial Method005.qgd	1	1	Level 1 Con	Print			

6.) Data Analysis of Samples

a.) On the Desktop click on the "GCMS Postrun" tab. This will open the "Postrun Analysis" window.

b.) Next click on the "Quantitative" tab. This will open the Data analysis window. Here you can view the TIC and MIC, as well as the Mass Spec

chromatogram, and the compound table.

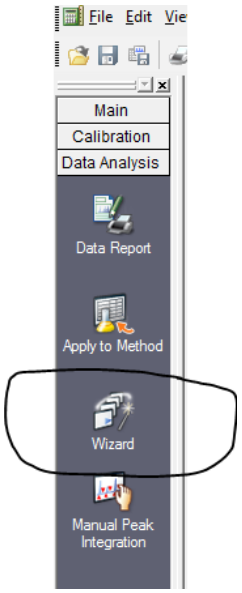


c.) To zoom in on a specific section of TIC or MIC click and drag with the left mouse button and the highlighted range will be zoomed in.

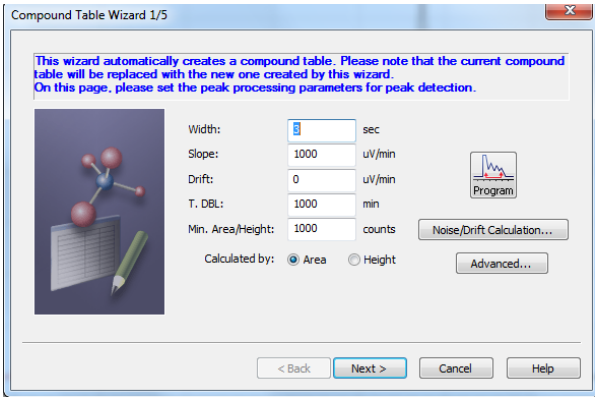
d.) Double clicking on a peak will show that peak's Mass Spec chromatogram. This can be found in the white box near the center of the window.

i.) Right clicking in this box will allow you to do a similarity search of the Mass Spec chromatogram. To get better results use the average spectrum and subtract baseline buttons found near the top of the window.

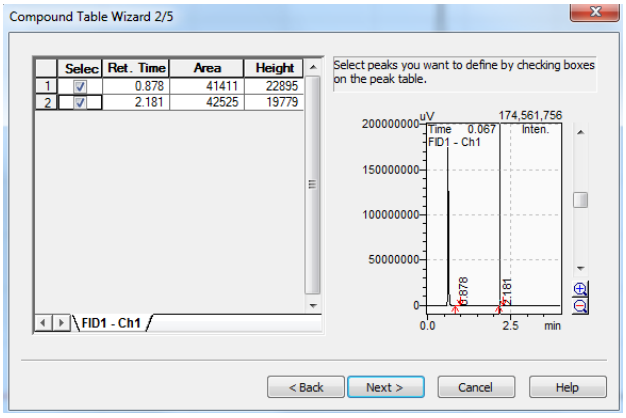
e.) Use the "Wizard" tab to create a compound table. You can choose which peaks to include in the compound table by selecting them. This will also integrate the peaks that you have selected. If the integration is not up to your standards, you can manually integrate the peaks that you wish.



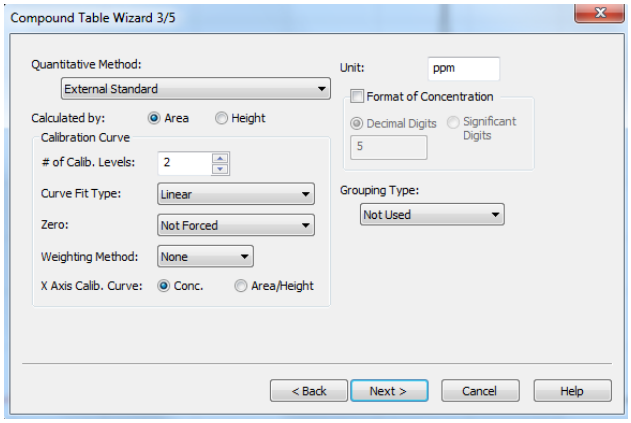
f.) Click on the “Wizard” tab and then choose the integration values that you wish and then click “Next >”.



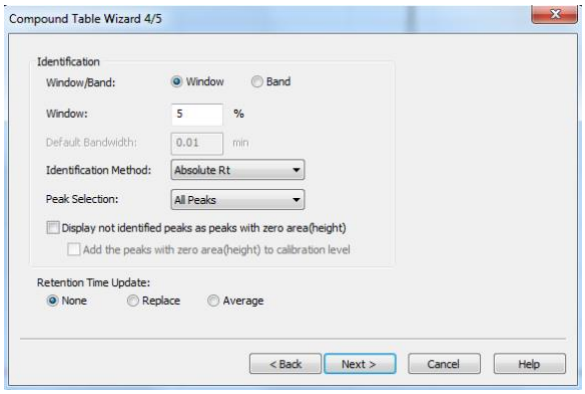
g.) Select the peaks that you wish to integrate and include in the compound table. When all of the desired peaks are selected click “Next >”.



h.) Now you can select the number of calibration levels, the units of concentration, and the type of calibration curve that can be created. When you have calibration curve settings as you wish then click “Next >”.

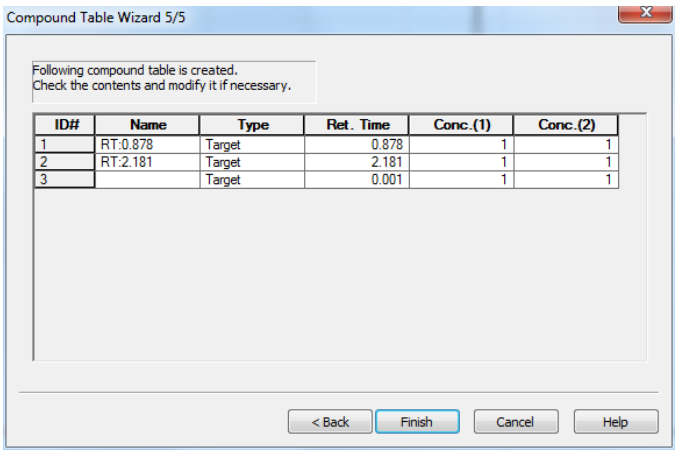


i.) Next choose how the peaks will be identified. Once this is done click “Next >”.

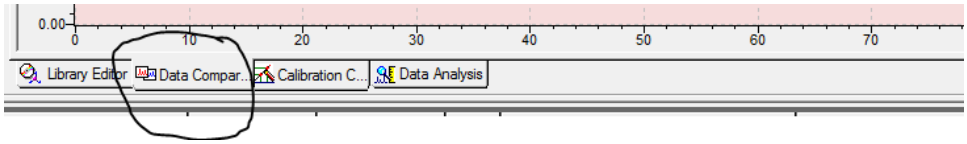


j.) Finally input the names of the compounds into the table next to the correct retention time. Hit “Finish” when you are done. This will create a peak

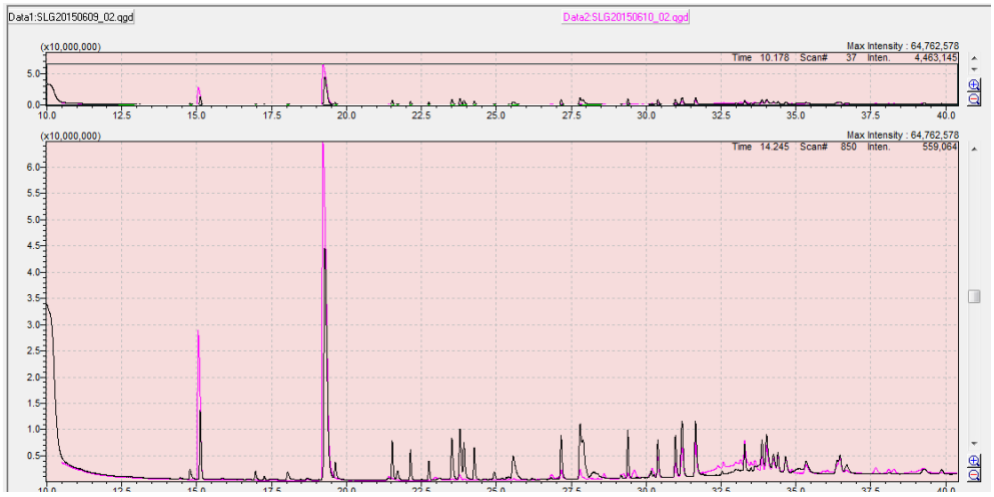
table, and a compound table.



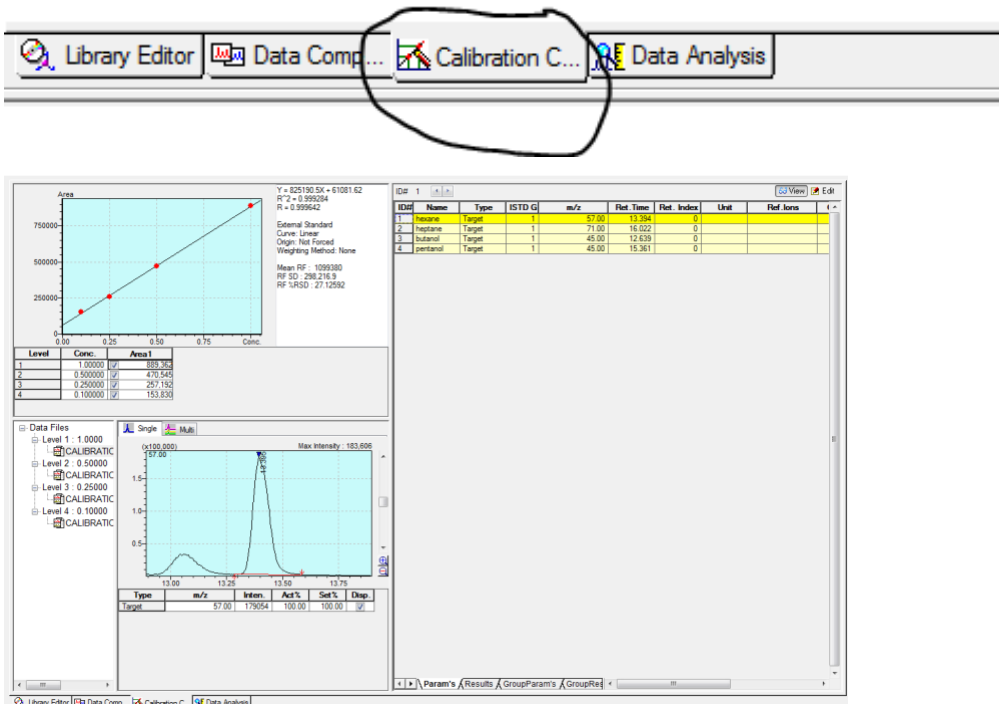
k.) Next click on the "Data Comparison" window. This will allow you to compare two data files, by overlapping their spectra.



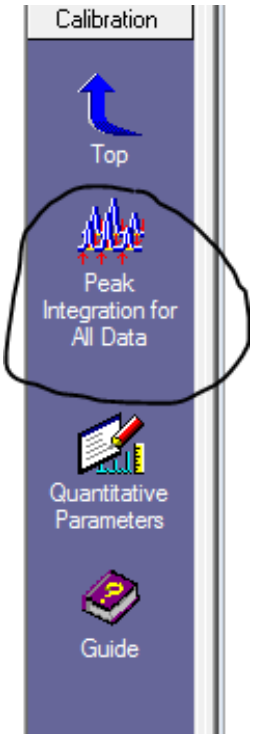
l.) Double click on one of the data files that you would like to look at, and then double click on the other file that you want to compare.



m.) Finally click on the "Calibration Curve" tab. This will open the Calibration curve window.



n.) Integrate the area of the peaks by either using the "Integrate All Peaks" tab or the "Wizard" tab.

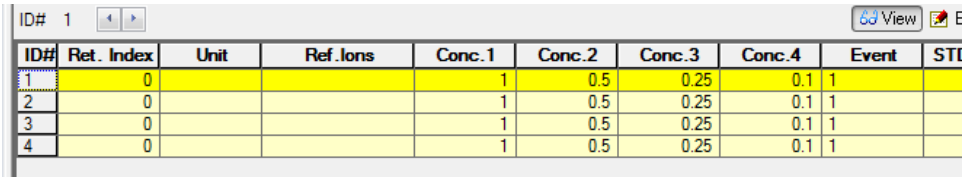


o.) Using the data explorer tab, drag the method file that you wish to use onto the tab

p.) Next switch the data explorer view to data files and then drag the data files you wish to use onto the different level tabs.

i.) In order to get different levels to show up use the “Wizard” tab and change the number of calibration levels to the amount that you desire.

q.) In the compound table change the concentration of each level and the calibration curve will be generated.


ID# 1

View

ID#	Ret. Index	Unit	Ref.Ions	Conc.1	Conc.2	Conc.3	Conc.4	Event	STI
1	0			1	0.5	0.25	0.1	1	
2	0			1	0.5	0.25	0.1	1	
3	0			1	0.5	0.25	0.1	1	
4	0			1	0.5	0.25	0.1	1	

7.) Logging Off

a.) Exit out of both the “GCMS Realtime Analysis” program and the “GCMS Postrun” program

b.) DO NOT SHUT DOWN THE GCMS make sure it is left on and running.

c.) Finally log off of the computer.