

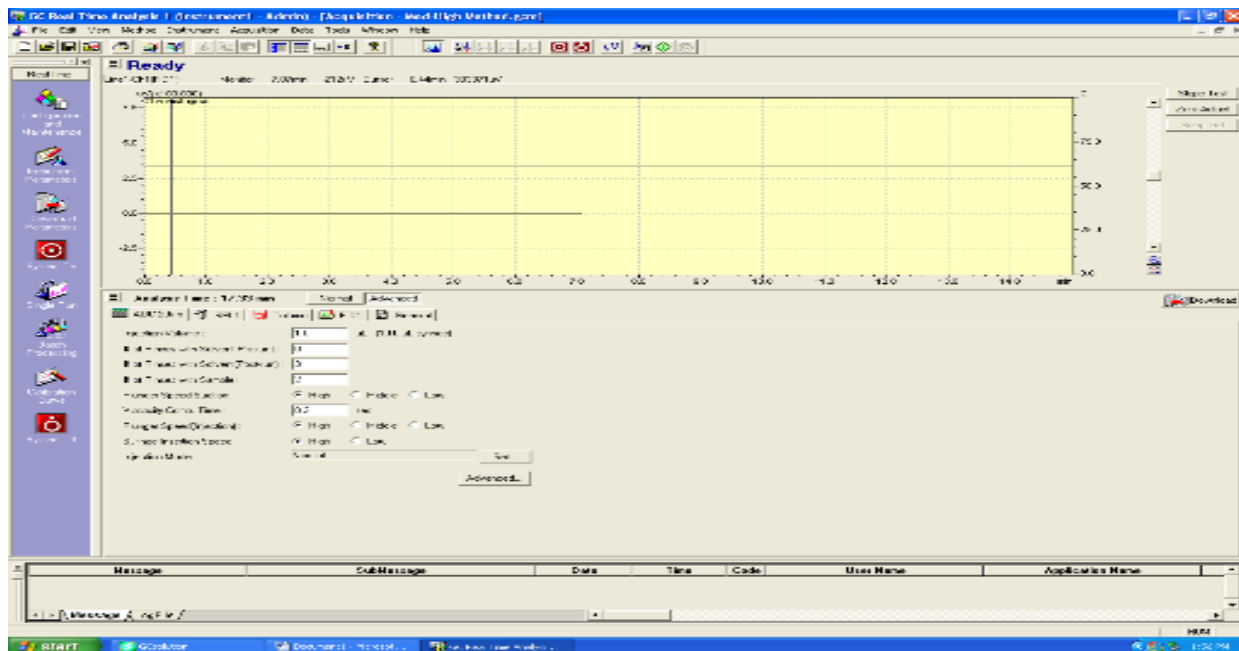
Shimadzu GC

Sample Preparation

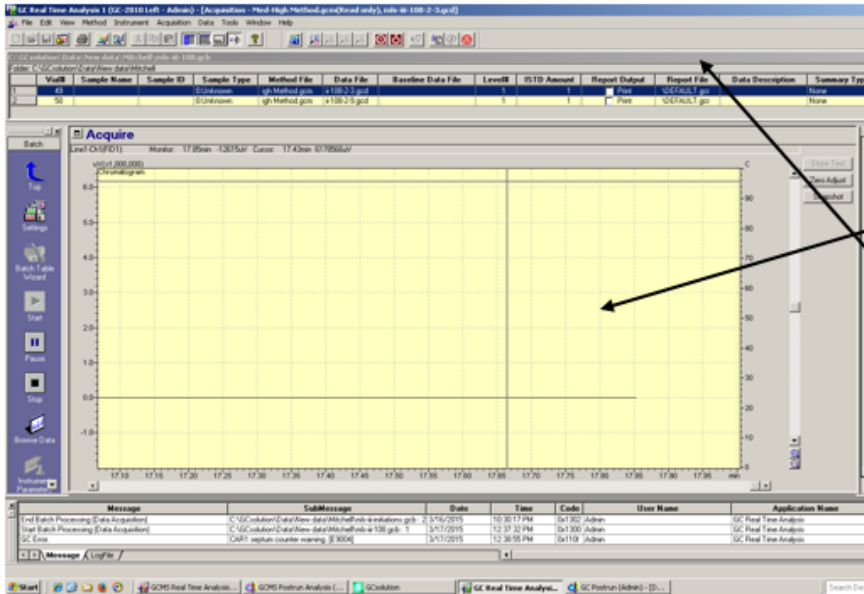
1. Your sample concentration should be no more than 5 mM (e.g., about <50 ul of a 0.2M reaction).
2. Never put metals, strong acids, bases, oxidants, water, or samples with particulates on the GC!
3. Sample with metals: filter through silica.
4. Samples with Pd/C: filter through celite.
5. Sample with strong acids or bases: aqueous work-up.
6. Samples with strong oxidants (e.g., Oxone, Br₂): quench with 10% aq NaSO₃ solution and work-up.
7. Samples with particulates: filter through celite.
8. Always check and refill the wash vials from the bottles next to the GC. Make sure to push the wash vial rack into its original position after refilling wash vials, or the sample will not run.

Running a Sample

1. Open the Shimadzu GCsolution operation window.
2. Click on the picture of a GC to open the login window. The username is Admin and there is no password, so click ok.
3. Once the program has loaded, click on the **Instrument Parameters** icon, in the bar on the far left side of the screen. If the **Instrument Parameters** icon is not visible click on the **Top** icon in the same bar until it appears.

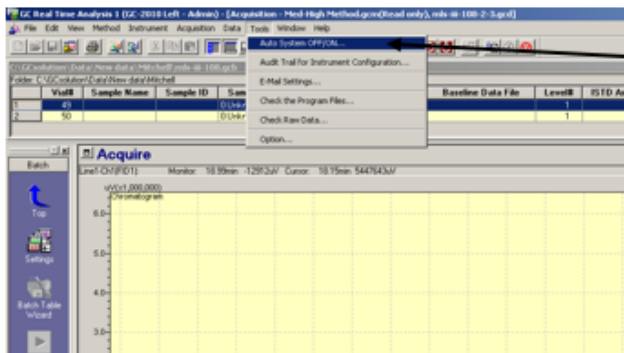


4. The screen should look like the picture above. If it does not, click the **Instrument Parameters** button again and the graph should appear.
5. Next, open the **Instrument Monitor** window, which is under **View > Instrument Monitor**. At the top of the **Instrument Monitor** window, click on **System ON**. The button will now read **System off**. The He gas should turn on and the oven injection port and FID will begin warming. The H₂ and air will also turn on and the flame will ignite when the appropriate temperature is reached.
6. You're now ready to make your sample batch. Click on **Batch Parameters**. Enter the vial position and sample name. Select a method and enter a data file name. When all your samples have been entered, hit the green **play** button. When prompted, enter a file name for your batch and hit **save**. **Note: when using the Wizard, the program automatically puts all your data files in the folder of the previous user. You can manually change the folder by clicking on each data file and selecting your folder, or before going through the Wizard, open an older batch file from your folder. This will ensure that your new data files are automatically saved to your folder.**
7. **Setting the Auto System Off.** If you are the last one to use the GC and need to leave lab before your run is finished, use the automatic "instrument off/on" tool. Make sure you have clicked inside the **instrument monitor** window.



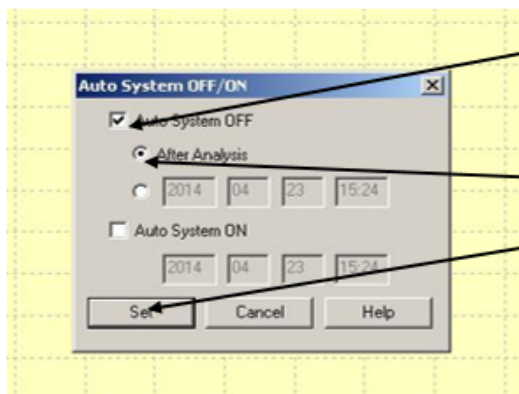
1. Click in this window

2. After clicking, sample queue should be greyed out



Click here

A new window will open; click the checkboxes for Auto System Off and After Analysis, then click Set. This window should remain on the screen afterwards.



1. Click Auto System Off

2. Click After Analysis

3. Then click Set. This dialog box will remain displayed on the screen.

If samples are already running:

1. Click the blue **pause** button. This will open up the batch parameters.
2. Enter the vial position and sample name. Select a method and enter a data file name.
3. When all your samples have been entered, unpause the batch by clicking **pause** again.

Processing the data

There are two ways to do this:

1. If you still have the batch file open: right click on the sample you would like to look at and select **browse data**. The data will open in **Postrun** in **Data Analysis** mode, where you can zoom in etc.
2. If the batch file is no longer open go back to the Shimadzu GCsolution Operation (which should be on open) window and select **Postrun** . This will open **Postrun** and then you can open the file you would like to analyze.

Printing data

In **Data Analysis** mode, select **Report In Data** . The go to **File>Print** to print data.

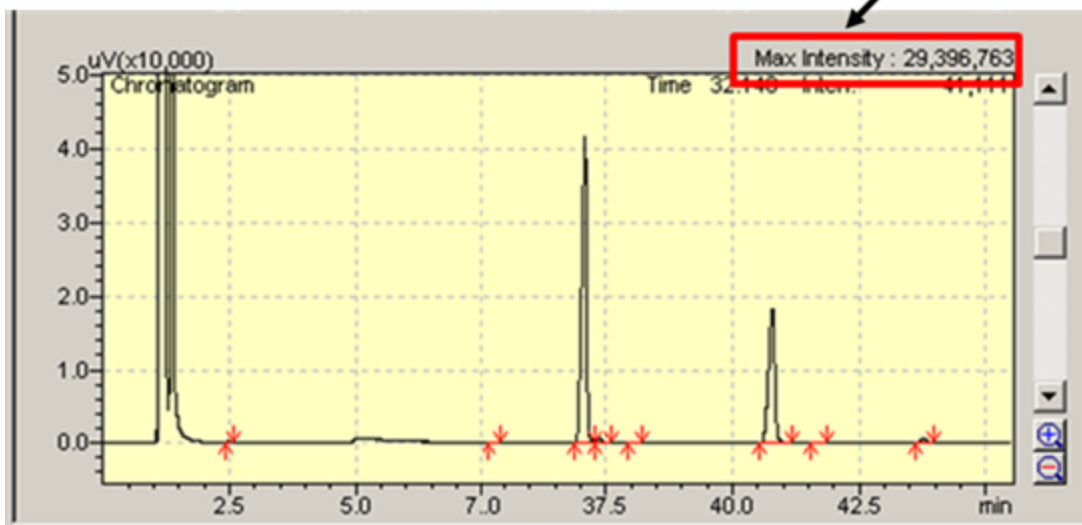
If you don't like the scale, right click on the chromatogram and select **Properties**. Select **Graph Range** and adjust the scaling.

Overlaying Multiple Chromatograms: In **Data Analysis** mode click on **Top**, then select **Data Comparison**. In this mode you can open more than one data set through **File>Open**.

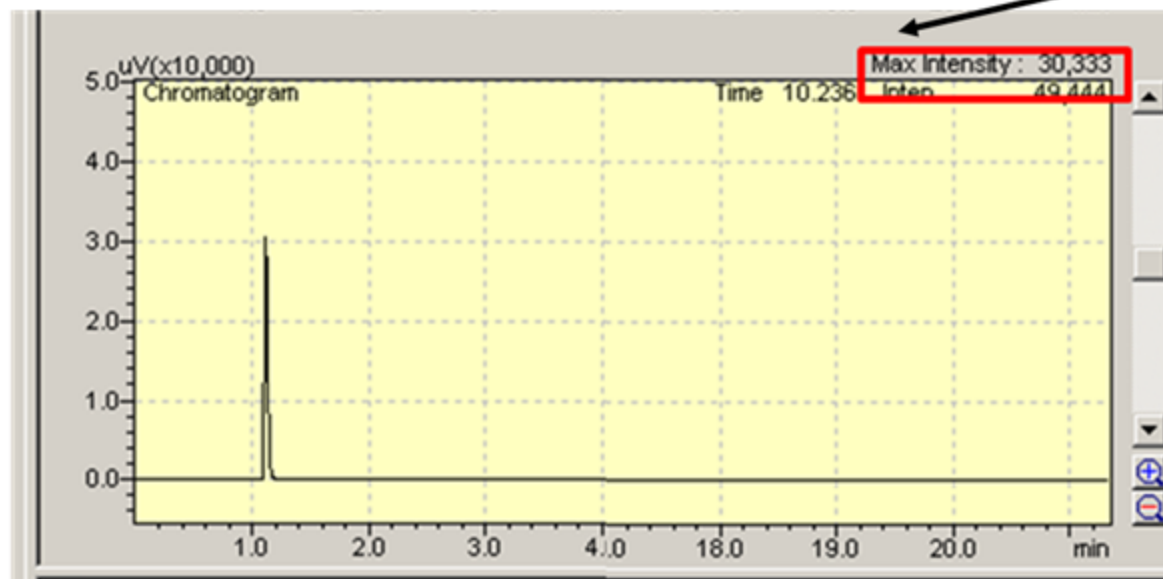
Determining if the Syringe is clogged:

Samples containing higher concentrations of polymer or other less soluble materials often crash out in the narrow injection needle. The sample will appear to run normally, however the only observable signal is solvent. When only solvent is observed, look at the intensity of the signal to determine if the needle is clogged or if the sample is too dilute.

In a normal run, the solvent signal has a “max intensity” of ~30,000,00



If the needle is clogged, the solvent signal “max intensity” will be ~30,000



When the solvent peak is orders of magnitude smaller than normal, the needle/syringe is clogged. **Stop** the run, contact Ariana, and make sure to **dilute** your samples before you rerun them.