BRIEF INSTRUCTIONS FOR GCMS

1. Your sample should be clean, passed over silica, column chromatographed, or recrystallized. Solids that are not volatile below 300 deg C should be injected by the DI probe, not the GC. Concentrations should be about 0.5micro gram per ml. Double click GCMS Real Time Analysis icon in upper left corner. A user name and password will be required. Wait for initialization.

2. Check settings at right side panel: GC system:ON, Heater:ON, Flow1:ON, GC:Ready, MS:standby Col temp:100 (idle setting) INJ temp:220 (idle setting) DET temp:280 Carrier gas mode:Split vacuum: 1.3 Pa or smaller. Check green light on turbo pump(lower right of instrument): steady, Autosampler: (red)000. If something is wrong, advanced users can run a system check to try to find out what's wrong. If the last icon in the upper left row is a question mark click on FILE in the far upper left corner and pull down to open. Pick test.qgm and click open then okay.

3. Sample login, far upper middle right of screen. Name the sample and give a unique data file name, __________.QGD. Click little folder icon to see previous names. Make yours different, other-wise you could overwrite an already existing data file. It's handy to incorporate the data file name into the sample ID field. Set vial #:1, Inj Vol: 1, Multi inj:1, unless other settings are needed. The most recent tuning file is automatically selected unless you specify one.

4. Method Development (icon in left column) Click little icon of a syringe in folder tab settings near center left under GC temp profile chart. Deafult settings: # of rinses with solvent(Pre-run):1, # of rinses with solvent(Post-run):3, #of rinses with sample:1 unless otherwise required. Other parameters can be left at their default settings.

5. Click GC folder tab under temp profile chart. Setup GC values: Typical injection temp is 200-250 deg, Keep interface at 280 deg, Control mode is usually split, typical column flow is 1 ml/min. Inlet pressure between 70 to 130 Kpa. Split ratio between 10-100. It's best to match a pressure program to
the temperature program to maintain constant flow. Temperature profile as desired. Preferably a GC profile has been worked out before you come to the machine. MAXIMUM column temperature is 325 deg C. If something has been left in the column, do a dummy run at 300 deg C. See step 26.

6. Click MS tab under temp profile. Acquisition mode usually: scan, Solvent cut time usually: 3 min, but not lower than 2 min. Leave detector voltage alone unless you know from a previous run that there will be too much sample (detector overload) or sensitivity problems. Start time: Solvent cut time plus 0.5 min typical (2.5 min minimum). Typical run takes 11 minutes but depends on GC temp profile. Longer runs will produce more spectra and a bigger data file. Estimate maximum mass and add ~50 amu to get even division. 900 amu is maximum. CO_2 appears at 44 so 45 is a good minimum. Max scan speed is 8000, but 2000 is a reasonable choice for good signal-to-noise Asking for a higher mass range will automatically recalculate a new scan rate.

7. IMPORTANT, if you changed anything in the method, pull down the FILE icon in the upper left corner and select: save method file as.... Otherwise you will overwrite a previous method file.

8. Place rinse solvent, empty vial and sample vial in tray. Make sure tray is installed properly, with

little tabs in slots and gear wheel engaged. Press reset on autosampler. Don't force the autosampler.

9. Click data acquisition icon in left column. Once you go beyond here you cannot return except by pressing the stop acquisition button in far upper middle right. Click standby icon in column. A box may appear asking if you want to overwrite the method file. Saying NO defaults to the previous method. Saying: YES is harmless if step 7 was done properly. It takes a couple minutes for the machine to set the parameters, and the GC has a built-in equilibration period.

10. Click start icon in left column. If the autosampler is jammed a warning box will appear. Clear the jam, close the door, press reset. The tray should go back and forth a few centimeters. If the scan is terminated early by clicking the stop button and another run is to be started immediately, press stop on the GC panel to abort the temperature program in the GC, and wait for the column to cool. To see the MS for a TIC peak that has gone by click Data Analysis in lower left corner.

11. When the data are finished accumulating, note the data file name from step 3 above. Do a cleaning run at 300 deg C. See step 26. Minimize the GCMS real-time acquisition window (far upper right "_ " button). Save the current data file if it prompts you to do so. Take data off for remote analysis (skip to step 18) or double click the GCMS post run analysis icon in the upper left corner.

12. Pull down the FILE button (far upper left corner) to open data file and click on the data file name you just generated (step 3 above or step 21 below).

13. Double click on the TIC (total ion current) retention peaks to see the mass spectrum of each peak of interest as a first scan of the data. (Is it reasonable? If not repeat step 12). It is necessary and the following will define how to number each TIC peak. The procedure may pick too many peaks or it might skip a peak of interest. If by choice or by error the display becomes excessively expanded, place the mouse arrow in the display, click AND RELEASE the right mouse button, and drag the mouse down to undo zoom again clicking the right button.

14. Pull down from the qualitative parameters at the very top center of the menu to peak integrate and across to highlight TIC. A table appears. Change the slope to 10,000/min, and the width to 3 sec. Click OK. The integrated peaks are defined with red arrows. If it did not define enough peaks repeat this step with lower slope and/or smaller width. If it defined too many peaks increase slope and width, and repeat this step. It is possible to define the peaks manually as follows. Start(step 12) with a clean TIC graph. Set the parameters with the icon at the top right center just to the left of the little yellow hand, and then click the icon with the yellow hand. Place the mouse at the lower left of a peak and drag the mouse across the peak defining the peak with a blue rectangle. Click OK to the default baseline. Do this for each peak of interest left to right. Once manual peaks are defined the peak table will be scrambled if you then attempt to let the software define peaks as above. Once you've done this three or four times it becomes obvious.

15. Once the peaks are defined pull down FILE from the far upper left to report. (Don't use the Report Generator) If a box appears on the page which is not wanted click the mouse in it and hit delete on keyboard. Click on the second (blue)icon in the third row(sample information if you leave the

mouse on it but don't click). Place the mouse on the page where the sample info is to be plotted and simultaneously click and drag to define the sample info box. A gray table appears. Select sample info in the tabs at top and highlight in blue any text you don' t want, then hit delete key. Click on the blue(5th from left in 3rd row) icon, place the mouse on the page and click and drag where the chromatogram should be plotted. A gray table appears. Select graph in the tab at the top and select peak# and area. Click OK

16. Click on red spectrum (6th from left in 3 rd row) and place the mouse on the page. Simultaneously click and drag to define the spectrum box for peak #1. A gray table appears. Select spectrum at the top where the page tabs appear. Pull down Spectrum process table and select peak#. Define the peak to be displayed in the two boxes to the right. Repeat this step for each TIC peak of interest. The report could be two or three pages in length depending on how many peaks are displayed. Page controls are found in black-and-white icons to the upper right.

17. Once the report looks right, pull down the FILE button from the far upper left corner to print preview. Check it. Make sure printer switch is set to GCMS. Pull down FILE button to print. Exit post run analysis saving the file if it so prompts. Put the GCMS in standby conditions, step 26.

18. To take the data off on a 3.5" floppy or a ZIP drive, close all applications. A typical data file occupies 650KB of disk space so two data files will fit on one 3.5" floppy. Open the Microsoft Explorer on the middle right. The data files are inside

    C:\GCMSsolution\Data\Project1\ (name of data file)

    Provide and insert an IBM-formatted ZIP or 3.5" disk cartridge. Pick up the data file with the mouse and drag it to disk A:\ (3.5" floppy) or to removable disk Q:\ (the ZIP drive).

19. Do a dummy run with the column set to 300 deg C and a hold time of 5 min to purge out any residual from the column. The procedure is saved in Quickclean.qgm. If necessary hit stop on the GC to abort the last run and start again. Put an empty vial in the sample tray, and login a new sample name to save the last run. In the menu at the far top find tools and pull down to daily shutdown (even if someone else is going to use the instrument an hour from now). Set the column to 100 deg C, the injector to 220 deg C, and the interface to 280 deg C. Set the carrier gas pressure to 10 Kpa and the total flow to 1 ml/min. Click shutdown. Leave the autosampler tray empty. Sign the log book.