Operation of the Perkin Elmer TGA-GC/MS

Summary of the TGA-GC/MS:

The TGA-GC/MS allows a user to decompose a sample by heating, measure its loss of mass, and simultaneously analyze the chemical composition of the sample. The instrument consists of three primary components: a thermogravimetric analyzer (TGA), a gas chromatograph (GC), and a mass spectrometer (MS). It can run in three operating modes: TGA, TGA-MS, and TGA-GC/MS. The TGA can operate at temperatures ranging from room temperature to 1100 ˚C under flowing air, nitrogen, or helium. The mass spectrometer (MS) is used to measure the composition of the evolved gases, while the gas chromatograph (GC) can be used to separate different gas components with the column for easier identification of individual molecular compounds.

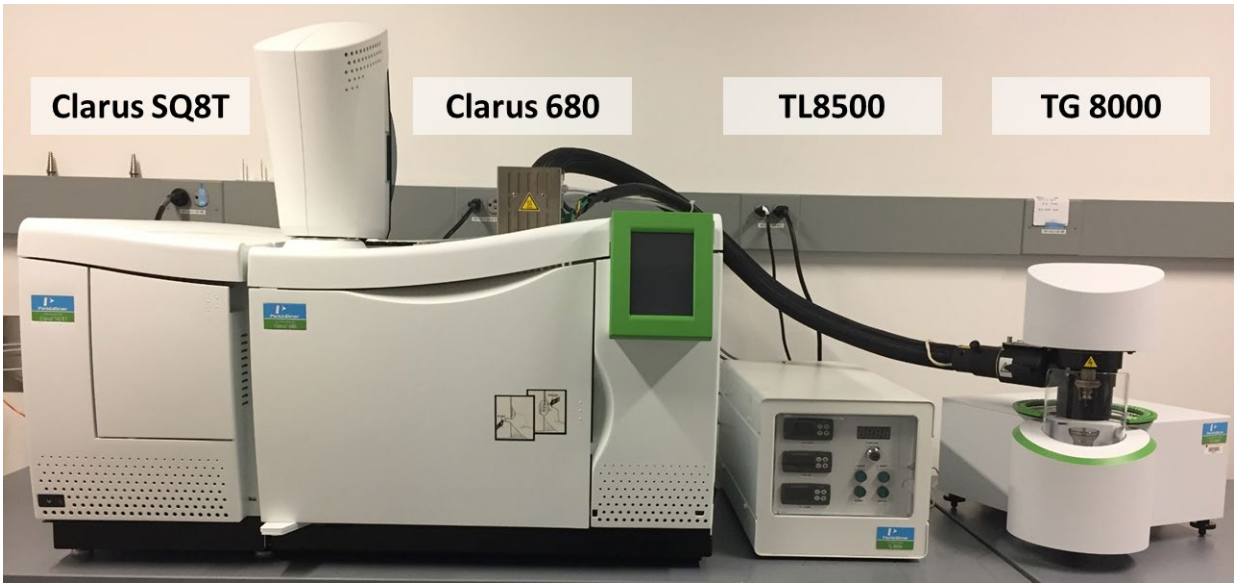


Figure 1. Perkin Elmer TGA-GC/MS Assembly.

Modes of Operation:

I. TGA
II. TGA-MS
III. TGA-GC/MS

Note: For TGA-GC/MS operation you will need to run a TGA-MS experiment first, find the decomposition temperature of your sample, configure the method in Pyris to trigger at the decomposition point of your sample, and then run a TGA-GC/MS experiment.

Safety Concerns and Precautions:

- Check the temperature of the TGA before lowering the furnace.
- **Warning –** When using the MS, helium must be used as the sample purge gas.
- **Warning –** Ensure that your sample pan is stable at the experimental temperatures that you will utilize. Aluminum pans cannot be heated above ~550 ˚C.
- **Warning –** The flow rate of the balance purge must be higher than the sample purge, otherwise corrosive gases may enter the balance and cause damage. Typical settings are: Sample Purge: N_2, Air, or He = 30 mL/min; Balance Purge: N_2 = 50 mL/min.

Preliminary Instrument Inspection:

Make sure the instrument is configured properly before starting your experiment. Contact the IAC staff if you have any questions. Helium must be used as the sample purging gas in experiments utilizing the MS (air cannot be used).

- Check the purge gas valve located behind the TGA. For GC/MS use, the valve must be opened for helium flow pointing left and the sample purge in **Pyris** software should be set to "air". For TGA-only use, you may choose between nitrogen and air.
- If helium is to be used, check that the He cylinder is not empty, i.e. there is some gas pressure shown by the gauges on top of the cylinder.

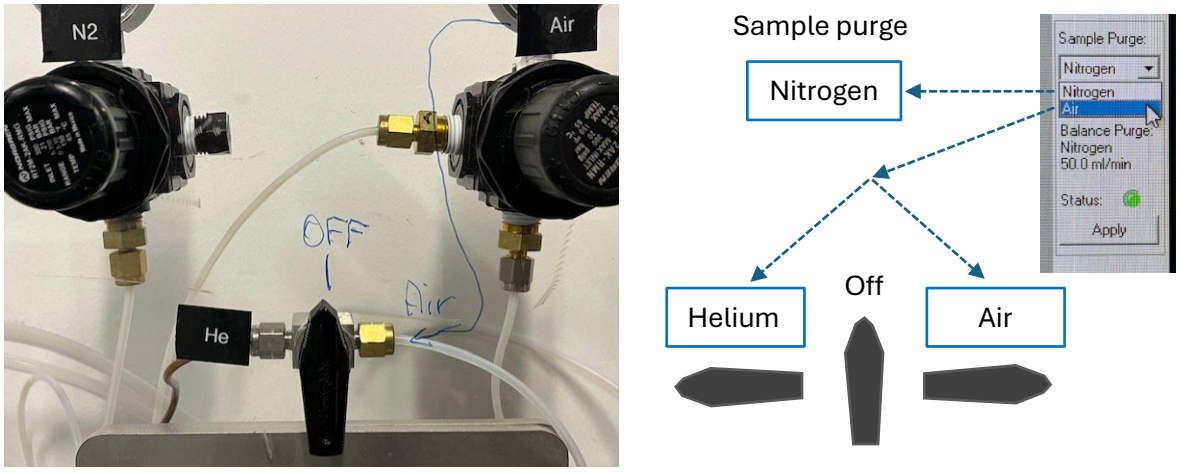
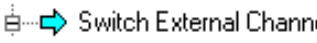
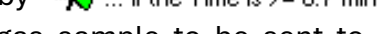


Figure 2. Sample purge gas selection. For N_2 select "Nitrogen" in Pyris window and leave the valve in Off position. For He (as for TGA-GC/MS or TGA-MS operation) or air, select "Air" in Pyris and turn the purge gas valve left or right for helium of air respectively.

TGA Operation:

1. **Turn on the TGA –** Flip the "Toggle Switch" on the back of the instrument.
2. **Open Pyris –** Click the Pyris Software icon on the PC, click on "TGA 8000" button.
3. **Create a TGA Method –** Select "Method Editor" window. Press "File" then "Open Method". A series of preset methods have been created in Pyris; select:
   - "General TGA Method" for TGA alone experiment
   - "General_Helium_TG-MS" for TGA-MS experiment
   - "General_Helium_TG-GC-MS" for TGA-GC/MS experiment.

   Select "Save Method as" to create a unique method for your measurements.
4. **Enter sample information**, date, directory and file name for saving data, etc. in "Sample Info" tab.
5. **Load the Autosampler –** Place your empty pan in the TGA's autosampler.
6. **Tare your Empty Pans –** Press the autosampler button and enter the slot number where you placed the pan. Tare the empty pan by using the following buttons: "Load Sample", "Raise Furnace", "Zero Weight", "Lower Furnace", and "Unload Sample".
7. **Load Each Pan –** Weigh 5 - 10 mg of sample and load the sample pan. Press "Sample Weight" button on the right.
8. **Select Sample Purge Gas –** In the pulldown menu under "Sample Purge" select Nitrogen or Air and if you selected Air, flip the gas valve to Air of He. For TGA-MS and TGA-GC/MS you need to use He (See Figure 2). Press "Apply".
9. **Enter Initial State Information –** Select "Initial State" tab. You can modify the initial state if needed.
10. **Enter the Program Info –** Set the temperature profile for the measurement in "Program" tab. It consists of dwell and temperature ramp steps.
    - Use the buttons on the right and properties section below to modify the profile. The total run time can be viewed by selecting "Temperature Program"
    - In cases of TG-MS and TG-GC-MS methods there will be plus icons next to some steps that you can expand by clicking. 

      This is a trigger signal to start GCMS. It is placed at the beginning in TG-MS method but in TG-GC-MS method it is conditioned by 

      Change this time to the time when you want the gas sample to be sent to chromatography column determined form preliminary TG-MS run.
    - Make sure the end condition is: Go to 50 °C, this will prevent the furnace from dropping immediately at the end of the run and exposing the MS to air.
11. **For TG-MS and TG-GC-MS** measurements, setup and **start** the experiment in Turbomass software (see below).
12. **Start the TGA Run –** In Pyris hit the "Start/Stop" button: 

13. **Remove Samples from Furnace –** After the run has completed, allow the furnace to cool. Use the autosampler to remove your sample pan. Clean the pan. For the carbonaceous residue "Burnout Method" in combination with air sample purge can be used.
14. **Turn off the Instrument –** Set the temperature of the furnace to 30 °C. Change the Sample Purge gas to Nitrogen if you used Air, press Apply. Flip the "Toggle Switch" on the back on the TGA. Turn the gas valve to Off position (Fig. 2). Close Pyris window.

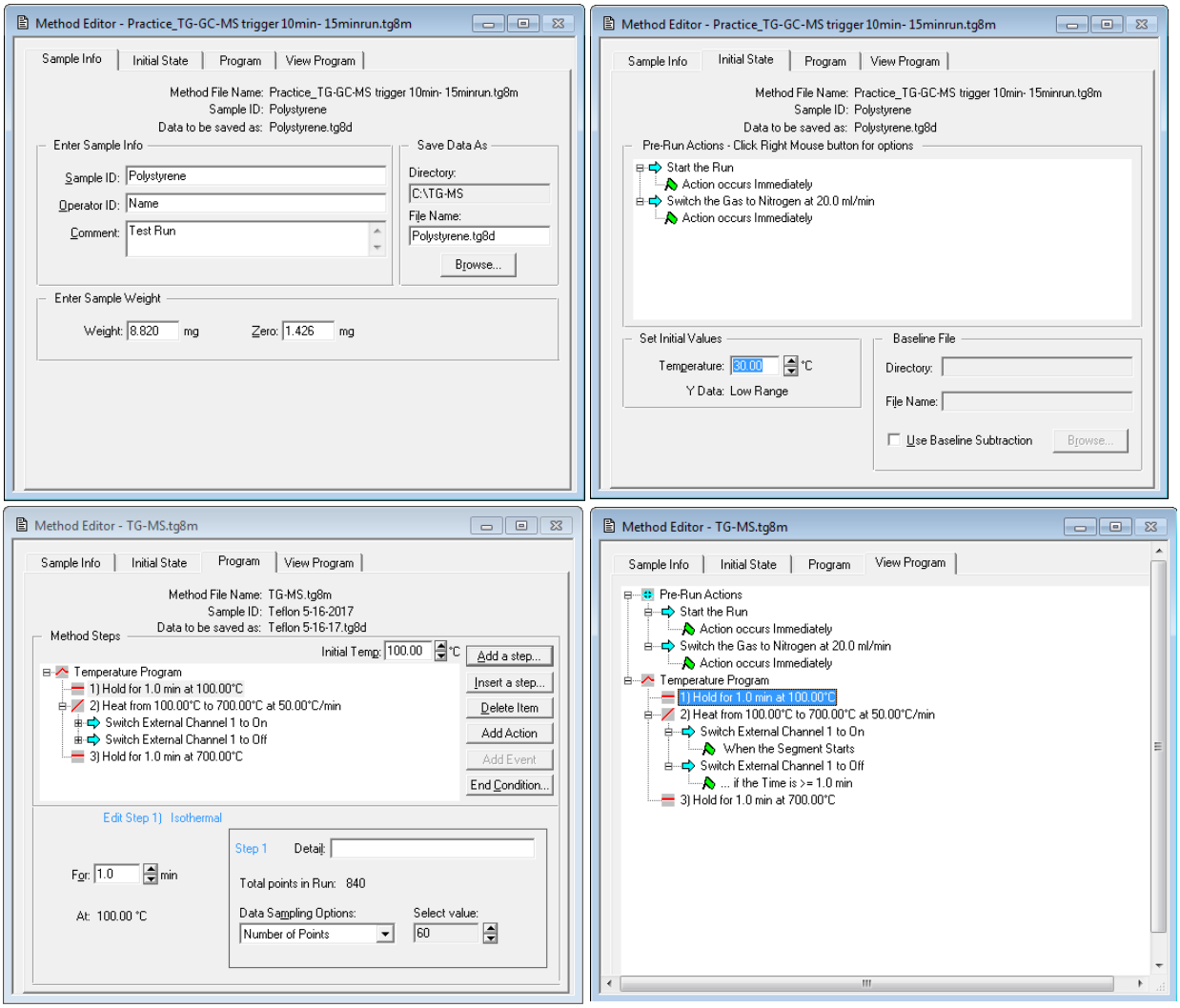
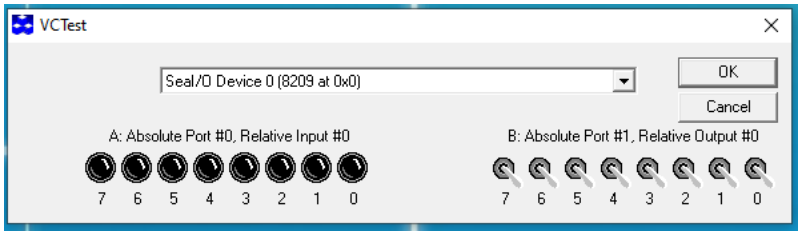


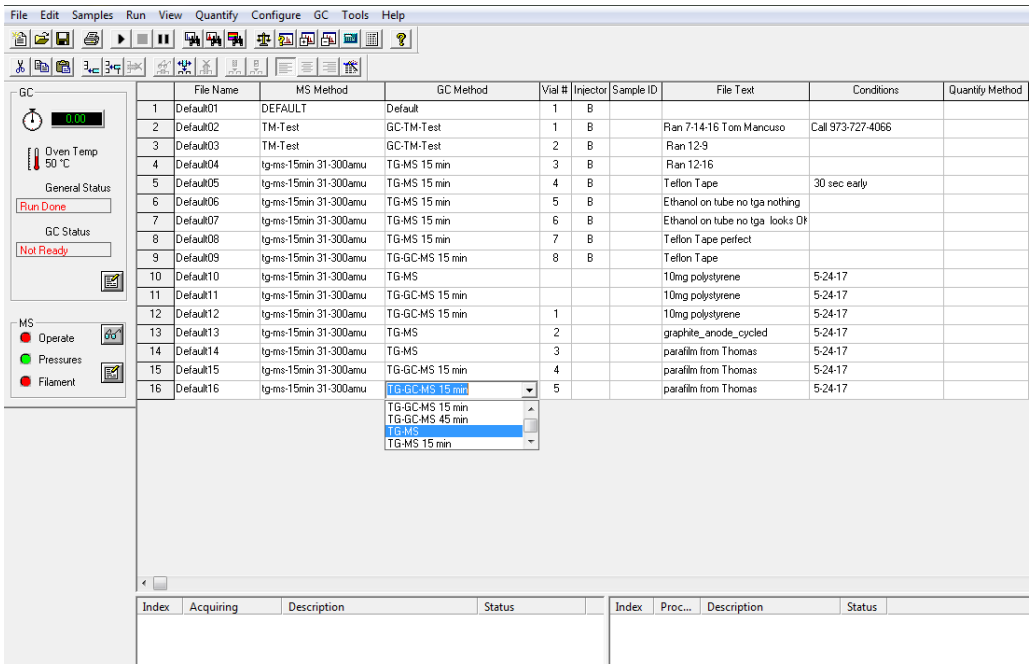
Figure 3. Examples of Method Editor window tabs.

GCMS Operation:

1. **The GCMS unit** will be normally running. If it is off, ask IAC staff to turn it on or follow separate printed instructions (not simple, it takes more than an hour).
2. **Turn on the Controller Box –** Flip the "Toggle Switch" on the back of the TL8500. The controller will begin heating the transfer line. Depending on the sample being analyzed, the temperatures should be set ~ 260 - 330 ˚C.
3. **Open VC Test Software** – It delivers the start signal from Pyris to Turbomass. It runs in the background and has no user accessible controls. Don't press "OK"



4. **Open Turbomass Software** – It controls the GC/MS (no password, just press OK).

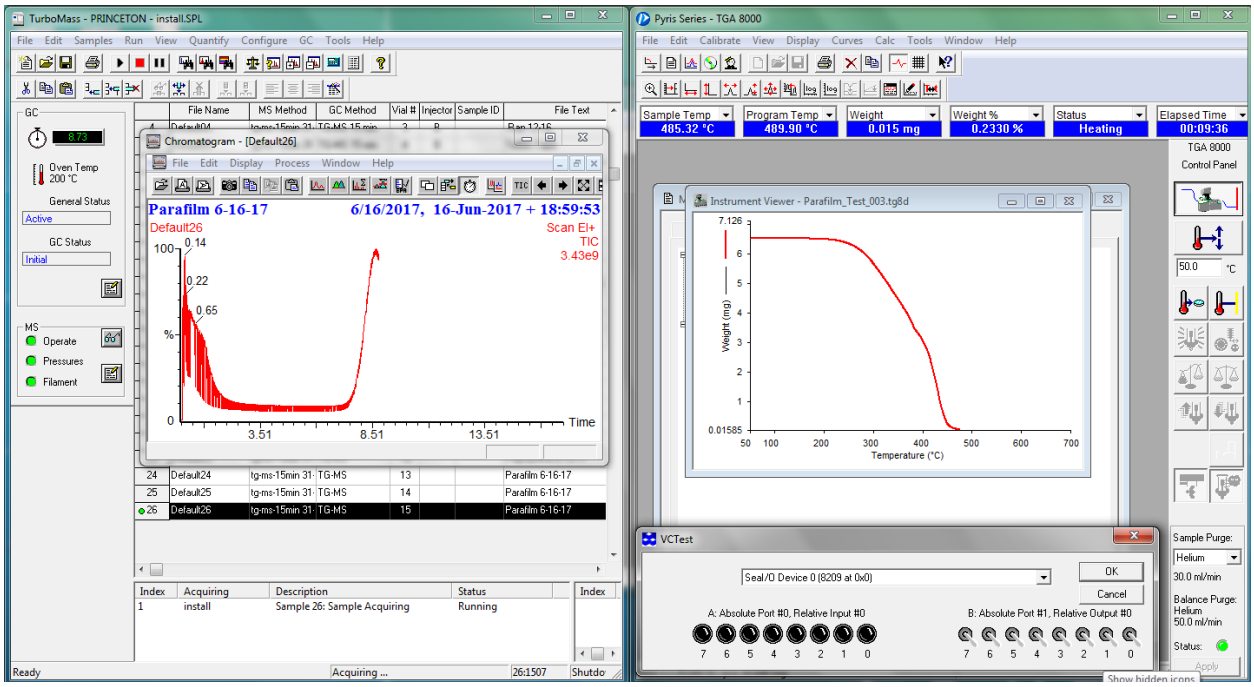


5. **Check the Tuning of the MS** – Open the "Tune Page" by clicking on the glasses icon in the MS box on the left of the Turbomass window
   - Click the "Press to Operate" button in the bottom right corner of the Tune Page.
   - Click the "Gas" option on the top tool bar and select "Reference Gas On"
   - Check the ions: The peaks should appear at ion masses 69 and 502.
   - Turn off the Reference Gas by clicking the reference gas icon.
   - Turn off the Filament by pressing the "Press for Standby" button on Tune Page.

6. **Create an Experiment** in Turbomass – Select the last line in the table and click "Add Sample"  and enter sample details. Enter an appropriate sample ID, conditions, notes, etc. For convenience widen "MS Method" and "GC Method" columns.
7. **Select a MS Method** – General experimental methods have been created for sample analysis. However, the instrument can also be tailored to meet the needs of your sample. To select an existing method, double-click on the "MS Method" cell of the table. Select a method from the pulldown menu. A typical method name is as follows: "tg-ms-15min 31-300amu continuum"
    - "tg-ms" refers to TGA-MS experiment. For TGA-GC/MS use "tg-gc-ms" methods
    - "15min" refers to the total length of data collection. It should match the duration of the TGA run in Pyrus (see above).
    - "31-300amu" refers to the ion mass range.
    - "continuum" refers to MS spectrum with several datapoints per single ion mass. In the absence of this ending, the data is collected in one value for one ion mass (centroid) format.
8. **You can edit a MS method** by right clicking on it and selecting "Open". In the new window, double-click the blue line. You can choose any ion mass range, duration time, etc. Save the method with an appropriate name. Then you can find it in the pulldown menu.
9. **Select a GC Method**. To select an existing method, double-click on the "GC Method" cell of the table. Select a method from the pulldown menu. A typical method name is as follows: "TG-MS-15min".
    - "TG-MS" refers to TGA-MS experiment. For TGA-GC/MS use "TG-GC-MS" methods
    - "15min" refers to the duration of data collection.
10. **You can edit a GC method** by right clicking on it and selecting "Open". In the new window, Click on "Instrument Control" icon. You can choose oven temperature, experiment duration, valve to operate (use V4 for TG-MS and V3 for TG-GC/MS experiments). Save the method with an appropriate name. Then you can find it in the pulldown menu.
11. **Start the Run** in Turbomass – Highlight the row that you would like to run and press the "Start Run" button  . In the series of popup windows, you must press "OK", "Save", "OK". A green dot should appear next to the row. **Wait** until both GC Status boxes on the left read "**Ready**" in green. It may take up to 10 min. At this point the GC/MS is waiting to be triggered by the TGA.

Running the Experiment

Start the experiment in Pyrus. At selected time Pyrus sends a trigger signal to GCMS. Below is the example of Turbomass and Pyrus simultaneously running. Note that status boxes in GC on the left should read "**Active**" and "**Initial**" in blue. Use binoculus buttons to view the MS signal and press stopwatch button to refresh the data in real time.



Saving the data.

1. **In Pyris**. The raw data is saved automatically after the experiment is done. To save data in externally usable format, go to File, Export Data. There you have a choice of saving the data in ASCII or CSV format.
2. **In Turbomass**. The raw data is permanently saved in the experimental table. It will be accessible on the instrument's computer for future analysis. Unfortunately, there is no known way to save the entire dataset in an externally usable format. But you can save individual mass spectra or chromatograms (signal as a function of time) for individual times or ion masses. For that, open an empty Excel spreadsheet. Then open the data by selecting an appropriate line in the table and clicking "View map" button . There, by moving horizontal and vertical cursor lines you can view chromatograms and mass spectra respectively. Double clicking on the chromatogram or mass spectrum opens them in separate windows. There you can copy data for data points or make a snapshot for a graph. Then paste them into the spreadsheet and save the Excel file.

Safety requirements

The laboratory in room ACEE 027B is a wet chemistry lab, in which some researchers may handle hazardous substances. The lab safety requires that users wear **eye protection** (safety googles or glasses with side shields), **lab coats**, and **gloves** while using equipment in this room.

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000
Room / facility emergencies: Contact Public Safety (609) 258-1000
Issues related to the instrument:

1. Contact IAC Staff.
2. If unsure, leave the system as is.
3. Try to power down the system.

Audible/Siren Emergency Alerts:

Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

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