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 the gases, while the gas chromatograph (GC) can be used to separate different gas components with the column for easier identification of individual molecular compounds.

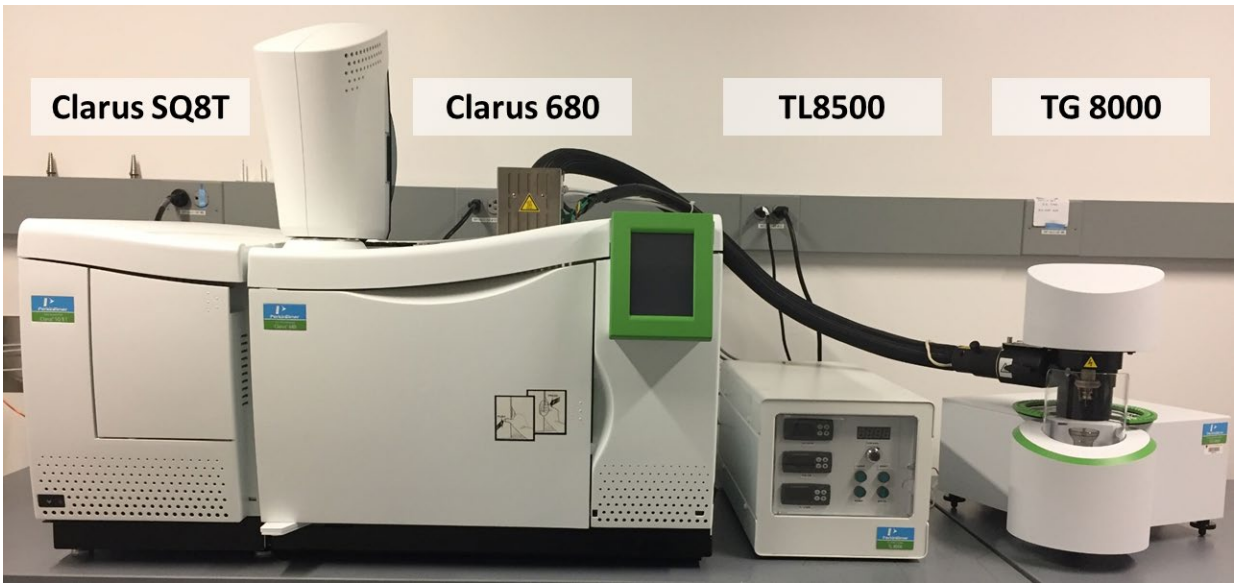

Figure 1. Perkin Elmer TGA-GC/MS Assembly.

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 gas and 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.

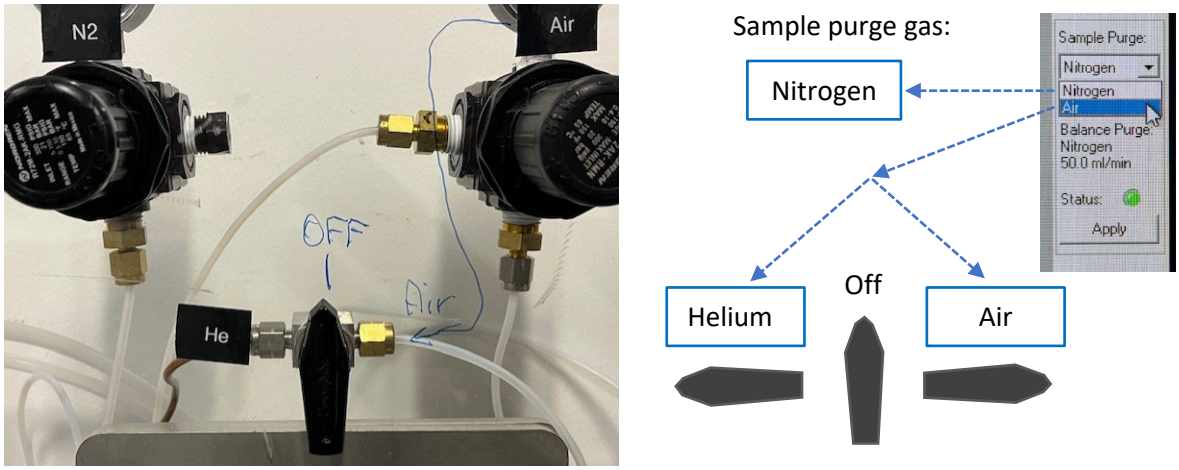

Figure 2. Sample purge gas selection. For N_2 select "Nitrogen" in Pyris window and leave the valve in Off position. For He (as for TGA-GC/MS or TGA-MS operation) or air, select "Air" in Pyris and turn the purge gas valve left or right for helium of air respectively.

I. TGA Operation:

1. **Log onto the Instrument –** Log onto the PC in the IAC.
2. **Turn on the TGA –** Flip the "Toggle Switch" on the back of the instrument.
3. **Open Pyris –** Click the Pyris Software icon on the PC, click on "TGA 8000" button.
4. **Create a TGA Method –** Press the "Method Editor" button. Press "File" then "Open Method". A series of preset methods have been created in Pyris, select "General TGA". Select "Save Method as" to create a unique method for your samples.
    - Enter sample information, date, directory and file name for saving data, etc.
5. **Load the Autosampler –** Place your empty pans in the TGA's autosampler.
6. **Tare your Empty Pans –** Press the autosampler button  and tare each empty pan by using the following buttons: "Load Sample", "Raise Furnace", "Zero Weight", "Lower Furnace", and "Unload Sample".
7. **Load Each Pan –** Weigh ~1-10 mg of sample and load each sample pan.
8. **Check Purge Gas Settings –** Go to Tools, Preferences, Purge Gas Tab; Sample Purge and Balance Purge should be set to nitrogen or air (only for TGA analysis).
9. **Enter Initial State Information –** Select the "Initial State" tab and enter the proper settings for your sample. Nitrogen or air can be used only when the GC/MS is not in use.
10. **Enter the Program Info –** Set the instrument to ramp and dwell at a desired rate.
11. **Start the TGA Run –** In Pyris hit the "Start/Stop" button: 
12. **Monitor the Experiment –** Ensure that the experiment heats to the desired temperature.
13. **Remove Samples from Furnace –** After the run has completed, allow the furnace to cool. Use the autosampler to remove your sample pans from the furnace.
14. **Turn off the Instrument –** Set the temperature of the furnace to 30 °C. Flip the "Toggle Switch" on the back on the TGA. Close Pyris and log off of the instrument.

II. TGA-MS Operation: (* - skip these steps if GC/MS is already on)

1. **Log onto the Instrument –** Log onto the PC in the IAC.
2. *** Open the Helium Tank –** Open the main valve on the cylinder and the smaller valve connected to the copper tubing. Leave the regulator in its standard position at 90 psi.
3. *** Turn on the MS –** Flip the "Toggle Switch" on the side of the instrument.
4. *** Turn on the GC –** Flip the "Toggle Switch" on the side of the instrument.
    - Wait a few minutes for the green light on the front of the MS to turn on.
5. **Turn on the TGA –** Flip the "Toggle Switch" on the back of the instrument.
6. **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 final temperatures should be set between ~270-330 ˚C.

7. **Turn on the Pump –** Press the green "Pump Button" on the TL8500 controller box after it reaches 50 ˚C to initiate the pump. Note: it could be already on.
8. **Open Pyris Software** – Pyris is used to control the TGA.
9. **Open Turbomass Software** – Turbomass controls the GC/MS.
10. **Open VC Test Software** – It delivers the start signal from Pyris to Turbomass.

Configuring the GC/MS in Turbomass:

11. *** Pump out the GC/MS** – Open the "Tune Page" in Turbomass by clicking on the glasses icon. Click "Options" then "Pump/Vacuum System On". Wait 15 min. for the instrument to equilibrate. The MS should reach 200 ˚C and the pressure should be in the green.
12. **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
    a) Click the "Press to Operate" button in the bottom right corner of the Tune Page.
    b) Click the "Gas" option on the top tool bar and select "Reference Gas On"
    c) Check the ions: The peaks should appear at ion masses 69 and 502.
    d) Turn off the Reference Gas by clicking the reference gas icon.
    e) Turn off the Filament by pressing the "Press for Standby" button on the Tune Page.
13. **Create an Experiment in Turbomass –** Click "Add Sample" and enter sample details.
    - Enter an appropriate file name, sample ID, conditions, notes, etc.
14. **Select a MS and GC Method –** General experimental methods have been created for sample analysis. However, the instrument can also be tailored to meet the needs of your sample. For a general TGA-MS experiment select:
    - **MS Method –** TG-MS 15min 31-300 amu
    - **GC Method –** TG-MS
15. **Start the Run in Turbomass –** Highlight the row that you would like to run and press the "Start Run" button. 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.

Configuring the TGA in Pyris:

16. **Check Purge Gas Settings –** Helium must be used for TGA-MS analysis (see Fig. 2).
17. **Create a TGA-MS Method –** Press the "Method Editor" button. Press "File" then "Open Method". A series of preset methods have been created in Pyris, select "General Helium TG-MS". Select "Save Method as" to create a unique method for your samples.
    - Enter sample information, date, directory and file name for saving data, etc.
18. **Load and weigh the samples** following steps 5 – 7 from "**I. TGA Operation"** above.
19. **Enter Initial State Information –** Select the Initial State info tab and enter the proper settings for your sample. Helium must be used when utilizing the MS.
20. **Enter the Program Info –** Below is an example program used for TGA-MS.

- 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.
- Make sure that the total time of TGA method is longer than the MS/GC methods.
- Ensure that "Helium Gas" is selected during MS in the bottom right corner of Pyris.

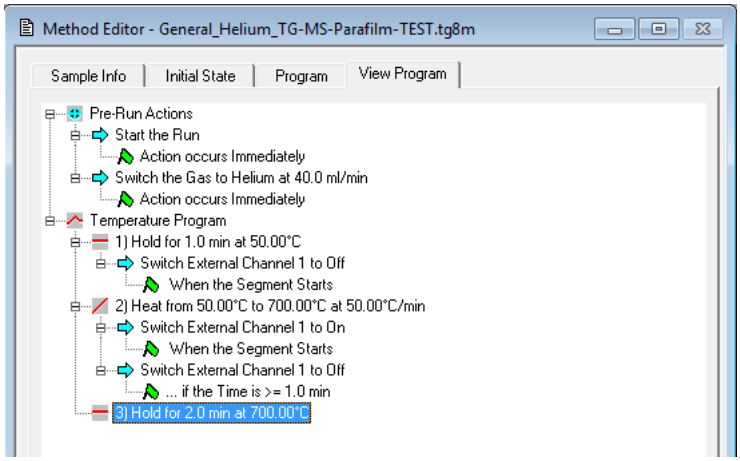

Figure 3. General TG-MS method in Pyris.

21. **Start the TGA-MS Run –** In Pyris hit the "Start/Stop" button: 
22. **Monitor the Experiment –** Ensure that the TGA triggers the GC/MS.
23. **Remove Samples from Furnace –** After the run has completed, use the autosampler to remove your sample pans from the furnace.

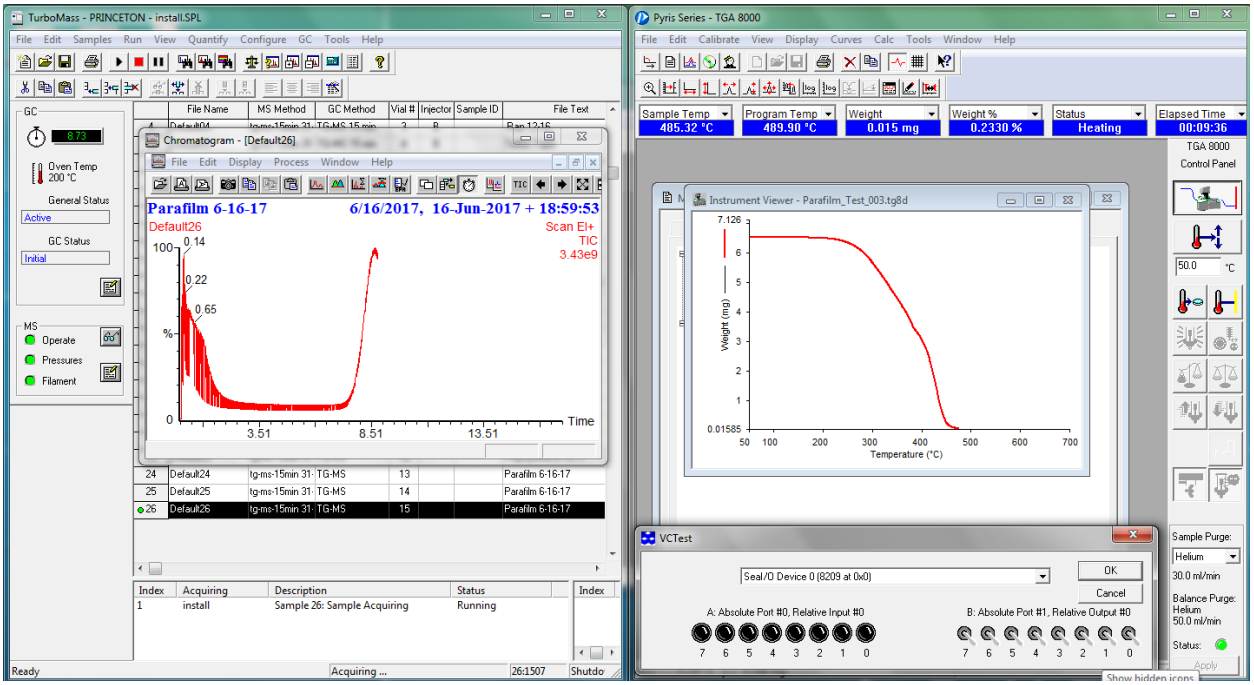

Figure 4. Turbomass, Pyris, and VC Test during TG-MS operation.

III. TGA-GC/MS Operation:

For TGA-GC/MS operation you will utilize the same heating method utilized for TGA-MS operation; however, you will set the Pyris TGA-GC/MS method to trigger the GC/MS at the time in which a peak was detected in the preliminary TGA-MS experiment. Therefore, all the steps of TGA-GC/MS procedure are **identical to that of "II. TGA-MS Operation"** procedure above **except**:

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14. **Select a MS and GC Method –** General experimental methods have been created for sample analysis. For a general TGA-GC/MS experiment select:
    - **MS Method –** TG-MS 15min 31-300 amu
    - **GC Method –** TG-**GC**-MS 15 min

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17. **Create a TGA-GC/MS Method –** Press the "Method Editor" button. Press "File" then "Open Method". A series of preset methods have been created in Pyris, select "General Helium TG-**GC**-MS". Select "Save Method as" to create a unique method for your samples.
    - Enter sample information, date, directory and file name for saving data, etc.

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20. **Enter the Program Info –** Below is an example program used for TGA-GC/MS.
    - Make sure the end condition is: Go to 50 ˚C.
    - Make sure that the total time of TGA method is longer than the MS/GC methods.
    - Ensure that "Helium Gas" is selected during MS in the bottom right corner of Pyris.
    - **Program GC/MS Trigger Time –** In the "Program" tab on the method editor page, **edit External Channel 1 to turn on and turn off at a desired point in time** during your experiment. This trigger should occur at the point in time where a broad peak was observed during the TGA-MS experiment

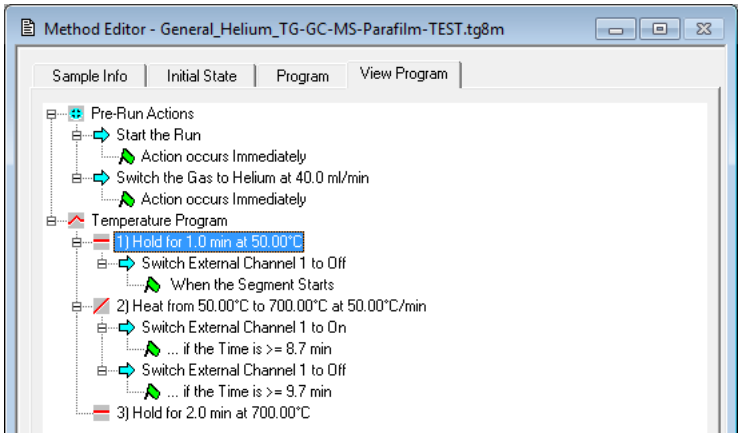

Figure 5. General TGA-GC/MS method in Pyris.

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Appendix.

Pyris Software

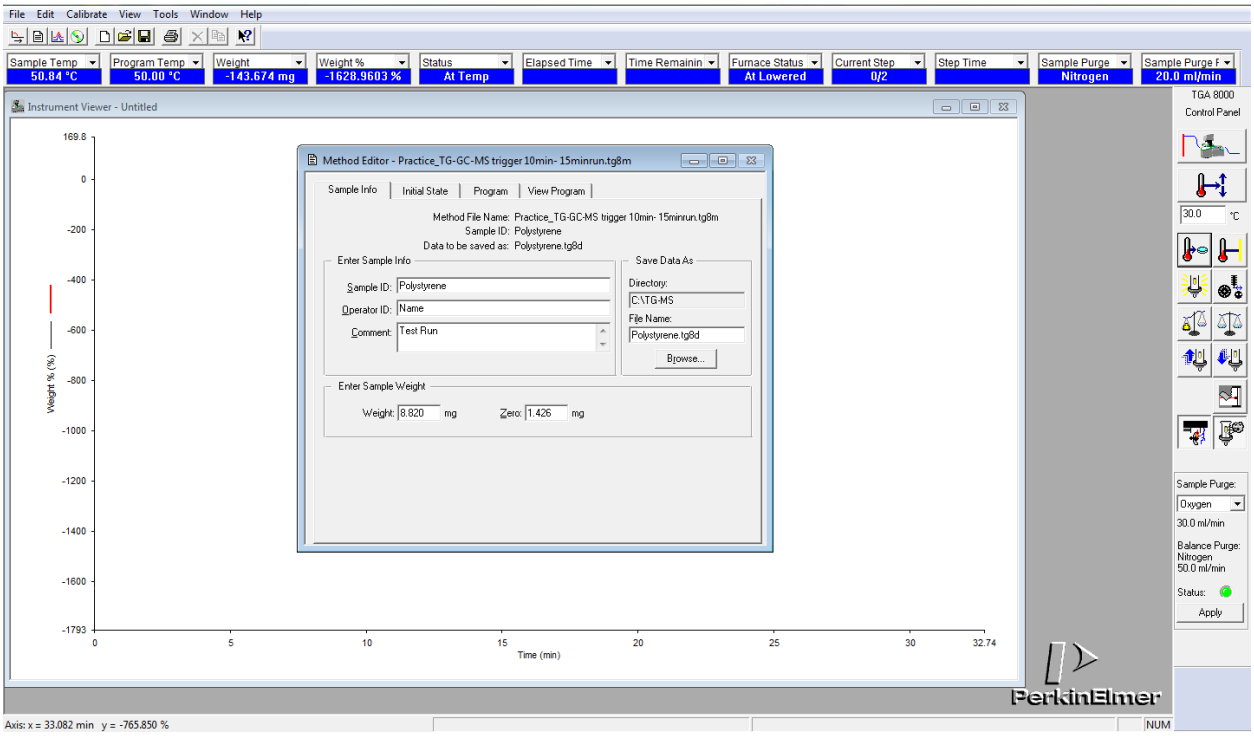


Figure 7. Pyris software window layout.

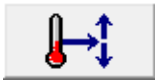
 Instrument View

 Method Editor

 Pyris Player

 Data Analysis

 Start/Stop

 Go to Temp

 Go to Load

 Go to Hold

 Clean Furnace

 Autosampler Control

 Sample Weight

 Zero Weight

 Raise Furnace

 Lower Furnace

 Reset Monitor

 Antistatic

 Fan

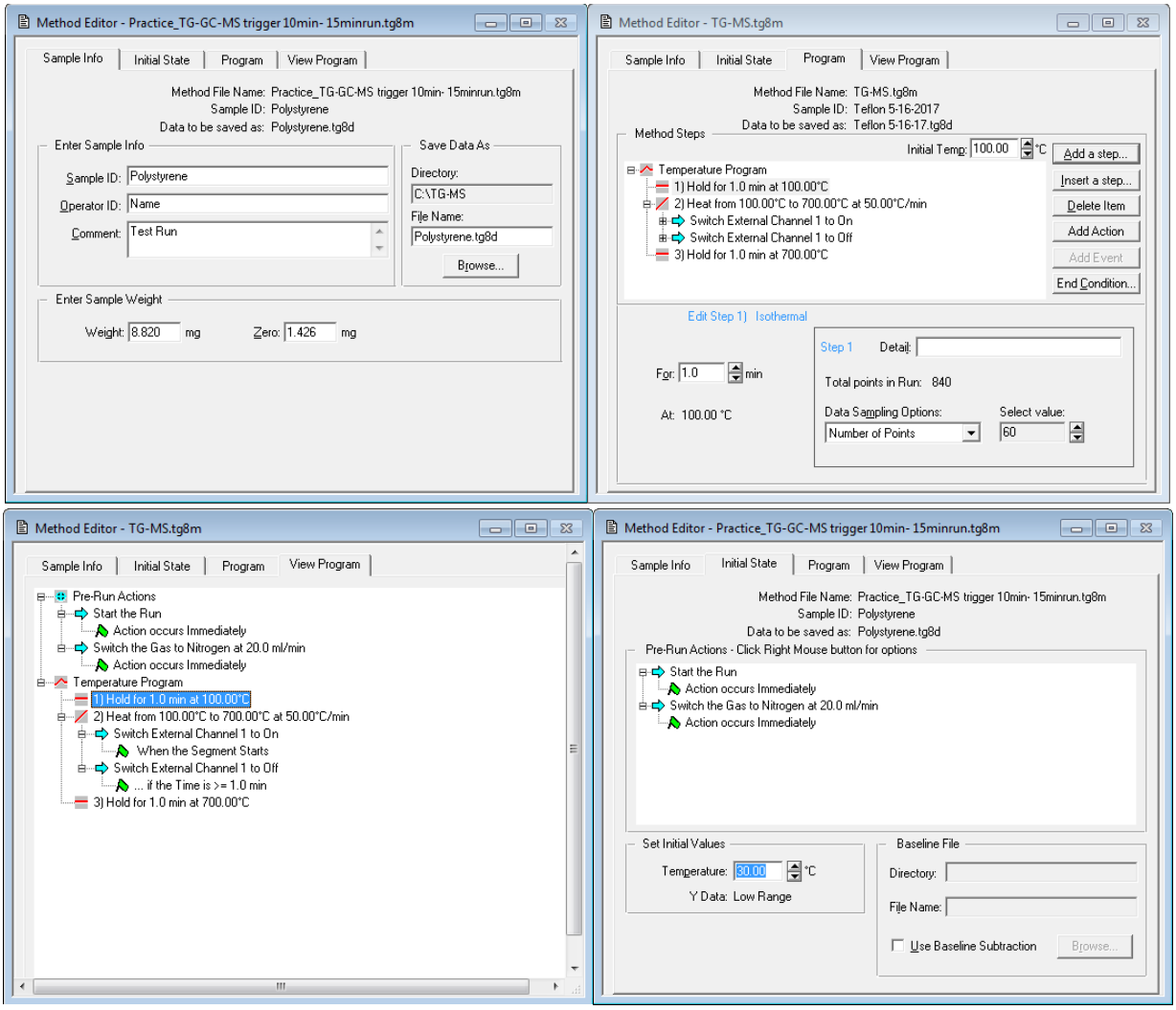


Figure 8. Method Editor window tabs.

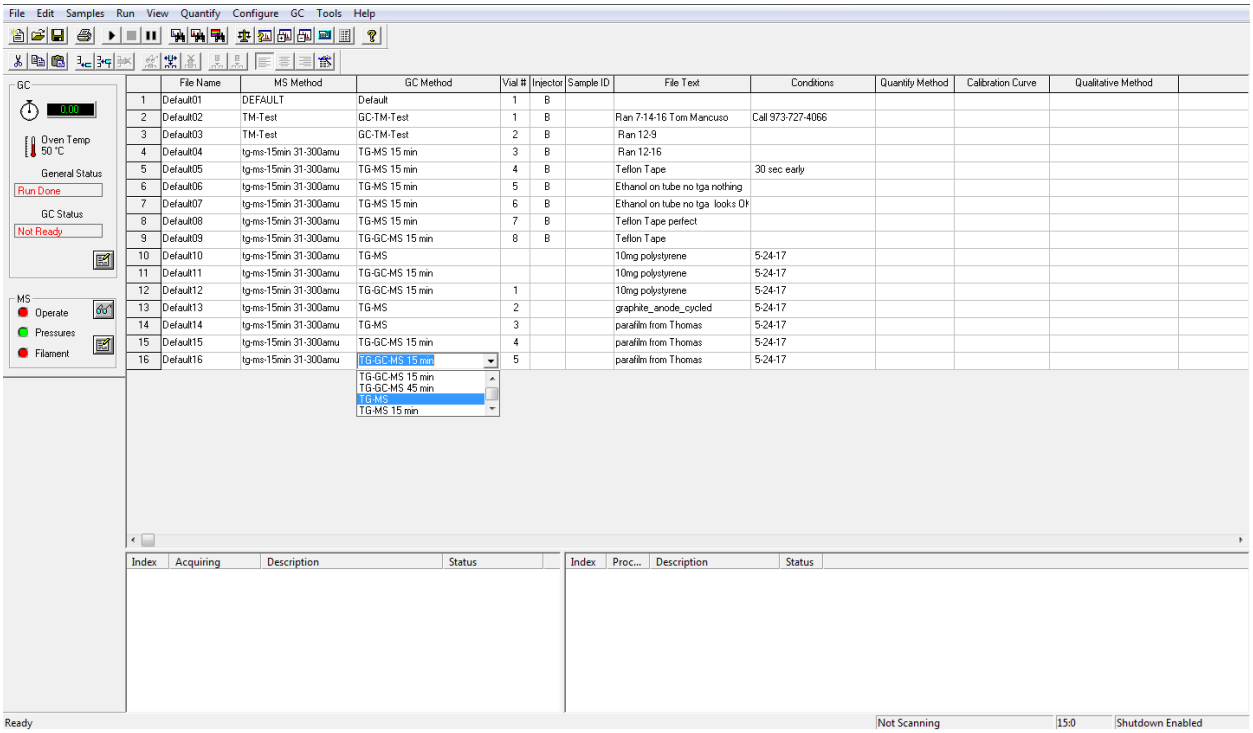


Figure 9. Turbomass software

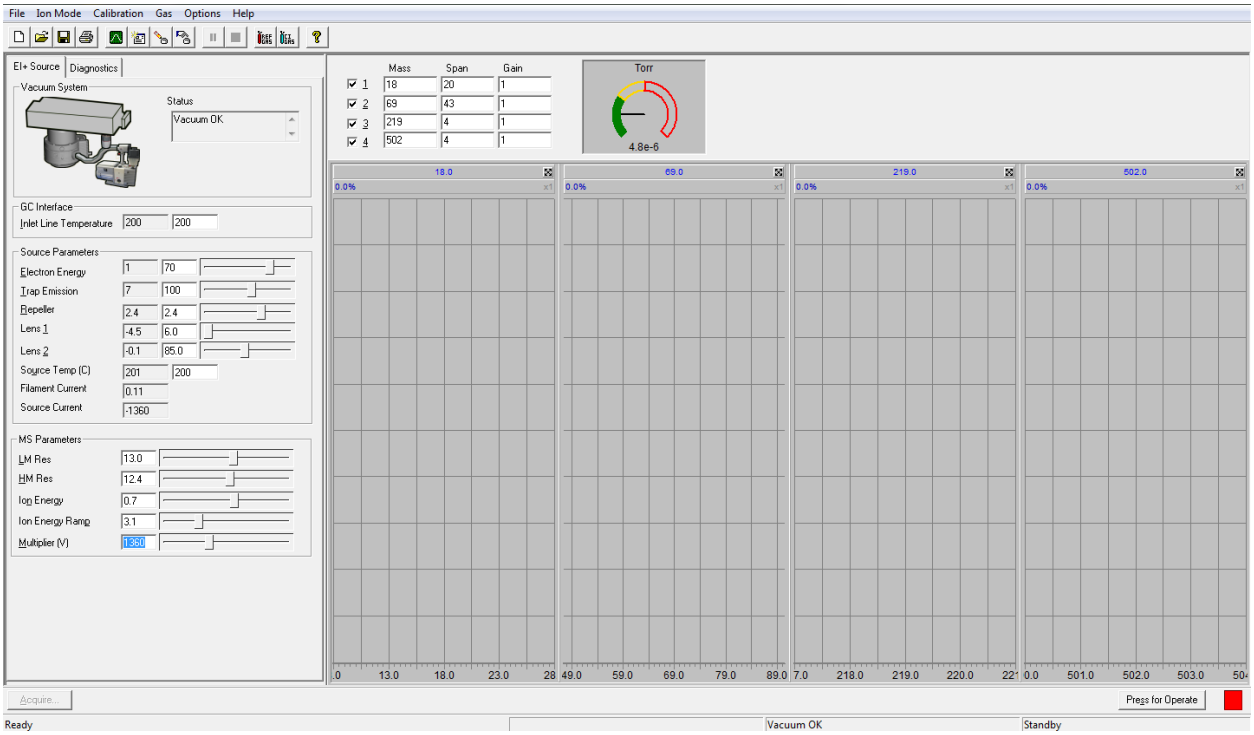


Figure 10. Turbomass tune page

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 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:

Nan Yao: Office (609) 258-6394; Cell (908) 922-2236 Email: nyao@princeton.edu
Denis Potapenko: Office (609) 258-7956; Cell (718) 551-6810 Email: denisp@princeton.edu
John Schreiber: Office (609) 258-0034; Cell (215) 431-4670 Email: js51@princeton.edu
Paul Shao: Office (609) 258-3851; Cell (847) 721-0861 Email: pshao@princeton.edu