**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 slow down the flow of each component through the column and better distinguish a sample’s chemical composition.

![Figure 1. Perkin Elmer TGA-GC/MS Assembly.](image)

**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, find the decomposition point of your sample, configure the GC to trigger at the decomposition point of your sample, and then run a TGA-GC/MS experiment.
Safety Concerns and Precautions

- Inspect the instrument to ensure that it is configured properly for your experiment.
- 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, alumina pans cannot be heated above 700 °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: He = 30 mL/min; Balance Purge: He = 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 gas must be used for 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 (pointed towards the copper tubing). For TGA use, the valve should be opened for nitrogen flow (pointed towards the plastic tubing).

Figure 2. Purge gas valve (a) when set to helium for TGA-GC/MS or TGA-MS operation; and (b) the purge gas valve when set to nitrogen for TGA operation.

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.
4. **Load the Autosampler** – Place your empty pans in the TGA’s autosampler.
5. **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”.

6. **Load Each Pan** – Weigh ~1-10 mg of sample and load each sample pan.

7. **Check Purge Gas Settings** – Go to Tools, Preferences, Purge Gas Tab; Sample Purge A and Balance Purge should be set to nitrogen or air (only for TGA analysis).

8. **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, weight, file name, etc.

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:**

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.

8. **Open Pyris Software** – Pyris is used to control the TGA.

9. **Open Turbomass Software** – Turbomass controls the GC/MS.
Configuring the GC/MS in Turbomass:

10. Pump out the GC/MS – Open the “Tune Page” in Turbomass by clicking on the glasses icon. Click “Options” then “Pump/Vacuum System On”.

11. Allow the System to Equilibrate – Wait 15 minutes for the instrument to equilibrate.
   • The MS should reach 200 °C and the pressure on the tune page should be in the green.

12. Check the Tuning of the MS – Check to make sure the instrument is properly tuned.
   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.
   c) Press “Reference Gas On” allow a few minutes for the instrument to equilibrate.
   d) Check the ions: The nitrogen ion (ion 28) should be less than the reference gas ion (ion 69) and there should be a small quantity of ion 502; otherwise there may be a leak in the system.

13. Turn off the Reference Gas – Turn off the reference gas by clicking the reference gas icon.

14. Turn off the Filament – Press the “Press for Standby” button on the tune page.

15. Create an Experiment in Turbomass – Click “Add Sample” and enter sample details.
   • Enter an appropriate file name, sample ID, conditions, notes, etc.

16. 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

17. 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. At this point the GC/MS is waiting to be triggered by the TGA.

Configuring the TGA in Pyris:

18. Load the Autosampler – Place your empty pans in the TGA’s autosampler.

19. Tare the 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”.

20. Load Each Pan – Weigh ~1-10 mg of sample and load it into each pan.

21. Check Purge Gas Settings – Helium must be used for TGA-MS and TGA-GC/MS analysis. Go to Tools, Preferences, Purge Gas Tab. Sample Purge A and Balance Purge should be set to helium.

22. 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 proper sample information, weight etc.
23. **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.

24. **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.
   - Check to make sure the total TGA method is longer than the MS and 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.](image)

25. **Start the TGA-MS Run** – In Pyris hit the **“Start/Stop”** button:

26. **Monitor the Experiment** – Ensure that the TGA triggers the GC/MS.

27. **Remove Samples from Furnace** – After the run has completed, use the autosampler to remove your sample pans from the furnace.
Figure 4. Turbomass and Pyris during the initial stages of TG-MS operation.

III. TGA-GC/MS Operation:

Note: 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 TGA-MS chromatogram.

1. Starting from Step #15 in the above TGA-MS Procedure:
2. Create an Experiment in Turbomass – Click “Add Sample” and enter sample details.
3. 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
4. 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. At this point the GC/MS is waiting to be triggered by the TGA.
5. Load the Autosampler – Place your empty pans in the TGA’s autosampler.
6. Tare the 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 it into each pan.
8. Check Purge Gas Settings – Helium must be used for TGA-MS and TGA-GC/MS analysis. Go to Tools, Preferences, Purge Gas Tab. Sample Purge A and Balance Purge should be set to helium.
9. **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, weight etc.

10. **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.

11. **Enter the Program Info** – Below is an example program used for TGA-GC/MS.
   - Make sure the **End condition is: Go to 50 °C**, this will prevent the furnace from dropping and exposing the MS to air.
   - Check to make sure the total TGA method is longer than the MS and GC methods.
   - Ensure that “Helium Gas” is selected in the bottom right corner.

![Method Editor - General_Helium_TG-GC-MS-Parafilm-TEST.jpg](image)

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

12. **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.

13. **Start the TGA-GC/MS Run** – In Pyris hit the “Start/Stop” button:

14. **Monitor the Experiment** – Ensure that the GC/MS is triggered at the proper time.

15. **Remove Samples from Furnace** – After the run has completed, use the autosampler to remove your sample pans from the furnace.

16. **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.
Figure 6. Turbomass and Pyris during TGA-GC/MS operation.

GC/MS Shutdown Procedure:

*Note: The GC/MS should be turned off and the helium cylinder should be closed if you are the last user of the week.

1. **Open Turbomass**
2. **Open the Tune Page** – Click the “Glasses Icon” to open the Tune Page.
3. **Change Temperature Set Points** – Change both the “Inlet GC Line Temperature” and the “Source Temperature” (in the source parameters area) from 200 °C to 50 °C.
   *Note: Allow the temperatures to drop below 100 °C before continuing.
4. **Turn Off the Vent/Vacuum System** – Once the temperature has dropped below 100 °C, go to “Options” at the top of the menu bar and select “Vent/Vacuum System Off”.
5. **Turn off the Turbo Pump** – Open the “Diagnostics” tab and change “Turbo Speed” from 100% to 0%. Allow several minutes for the turbo pump to power down to zero.
6. **Exit Turbomass** – Close the Tune Page and Turbomass windows. Allow the software to fully close.
7. **Turn Power Off on the GC & MS** – Flip the “Toggle Switches” on the sides of both the GC and MS.
8. **Close the Helium Tank** – Close the main valve on the helium tank.

*Note: Check the helium cylinder to make sure there is adequate helium for future operation. Contact IAC staff if the pressure on the helium cylinder is low.
Appendix
Pyris Software

Figure 7. Pyris software window layout.
Figure 8. Method Editor window tabs.
Turbomass Software

Figure 9. Turbomass software

Figure 10. Turbomass tune page
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
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