IMSERC User Manual for TA TGA5500

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INTRODUCTION

Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at http://imserc.northwestern.edu/pcm-instruments.html. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and also available under the corresponding instrument section at http://imserc.northwestern.edu/pcm-instruments.html by pressing on the 'User manual' button. If while using the system, something happens that you do not understand, please ***stop***, and ***get help***. In any event, be completely prepared to justify your actions. The cost of even minor repairs could be considerable.

SAFETY

All users of IMSERC must review the general safety policies at http://imserc.northwestern.edu/about-policies.html. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

- Hazardous Chemical Waste Management
- Laboratory Safety
- Personal Protective Equipment

You need the above certificates to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at https://learn.northwestern.edu. Upon completion of the certificate, it will take an overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found in outside of room B172 at the north side. Protective eyewear is required in this room, and gloves should be removed when using the computer.

DATA MANAGEMENT

Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder, otherwise you might not be able to access your data remotely. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied on your group folder on 'imsercdata.northwestern.edu' under 'others/TGA' every few seconds. Please follow instructions at http://imserc.northwestern.edu/about-general-faq.html#data for details about data access.

SOFTWARE

Data reduction and analysis of thermal data can be performed with the 'Trios' software. GC-MS data processing can be performed with the 'ChemStation' software. Software is installed on the instrument computer. For offline analysis after your instrument reservation is complete, please use the following resources:

- For registered IMSERC users, the 'Trios' and licensed to IMSERC 'ChemStation' software can be downloaded from 'imsercdata.northwestern.edu' under the folders 'public/TA' and 'public/STA', respectively. Software is available for Windows only. Please follow instructions under 'Data Access' at http://imserc.northwestern.edu/about-general-faq.html#data on how to connect to the 'public' folder
- Remotely via NUWorkspace which is operating system independent. After logging in with your netID credentials, please launch the 'IMSERC' workspace which contains the thermal analysis (no GC-MS) software
- You have the option to use the instrument computer for analyses, but you must reserve instrument time through NUcore

DEFAULT INSTRUMENT STATUS

The default measurement mode of TA TGA5500 is ***TGA/DTA under inert gas (Helium or Nitrogen or Argon) without GC-MS and without FT-IR***. Please notify the appropriate staff member well in advance if you would like to run an experiment in a different mode than TGA/DTA or using a different gas environment. For the full list of modes and capabilities, please check at http://imserc.northwestern.edu/pcm-instruments.html#tga. Additionally, put a note on your NUcore reservation indicating the preferred mode of your measurement.

The default working condition of TA TGA5500 is as follows:

1. Computer screen is by default deactivated. You must start your reservation through NUcore to be able to turn on the computer screen. If screen is already on, start your reservation through NUcore
2. The default 'TGA' user account should be logged in. In case the computer was restarted, the password for the 'TGA' account is (see hardcopy by the instrument)
3. Acquisition software (Trios) should be running. Leave the acquisition software open when you are done with the measurement
4. There should be no error messages on either the front panel of the instrument or the acquisition software. Please check the 'Troubleshooting' session for a potential solution before reporting the error
5. Crucibles, standards, and various attachments are in the yellow compartmentalized container (figure 5) located in the second drawer of the cabinet under the DSC high throughput instrument. When exchanging ceramic crucibles, please place uncontaminated ceramic crucibles back into the yellow container. Do not leave exchanged ceramic crucibles at the area near the instrument, as they might role, drop, and crack or break

If there is an error or problem with the instrument that is not covered under the 'Troubleshooting' section, please report the issue by following at least one of the steps below:

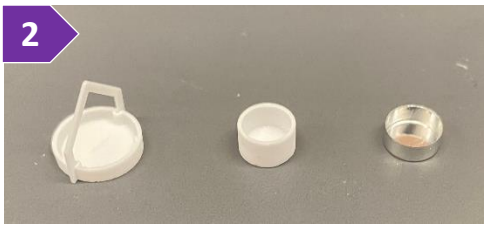
1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html and place the 'Stop' sign near the instrument computer
3. Contact a staff member for instructions

LOADING A SAMPLE INTO THE INSTRUMENT

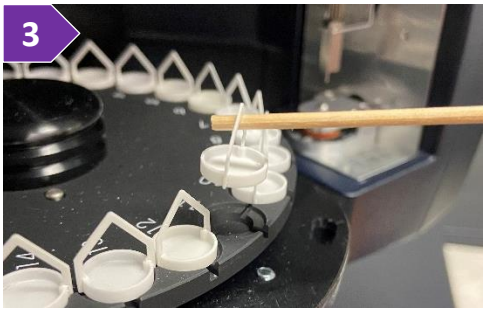
The following procedure should be followed by the users who want to use the default instrument configuration, which is 'TGA/DTA'. If you need to measure DSC, please ask for training on our dedicated DSC.

1. Verify that the instrument is idle, and the temperature of the furnace is near room temperature (20-40 °C) as indicated on the display panel in the front of the device (figure 1). Start your reservation in NUcore to have the screen of the instrument computer turned on. Also, please verify that there are no current collections running on the instrument by looking at the 'Running Queue' (lefthand side) under the 'Experiments' tab

2. Select which type of crucible you would like to use for your measurements. If only going up to 600°C the disposable Al crucibles can be used. If going higher than 600°C (up to 1200°C) then the ceramic crucibles must be used. Figure 2 shows (from left to right) a ceramic pan, a ceramic crucible, and an Al crucible. We use ceramic pans regardless of the type of crucible. All crucibles are in the yellow plastic case as listed in step #5 under 'Default Instrument Status'. Ceramic pans should be always left on the carrousel of the autosampler

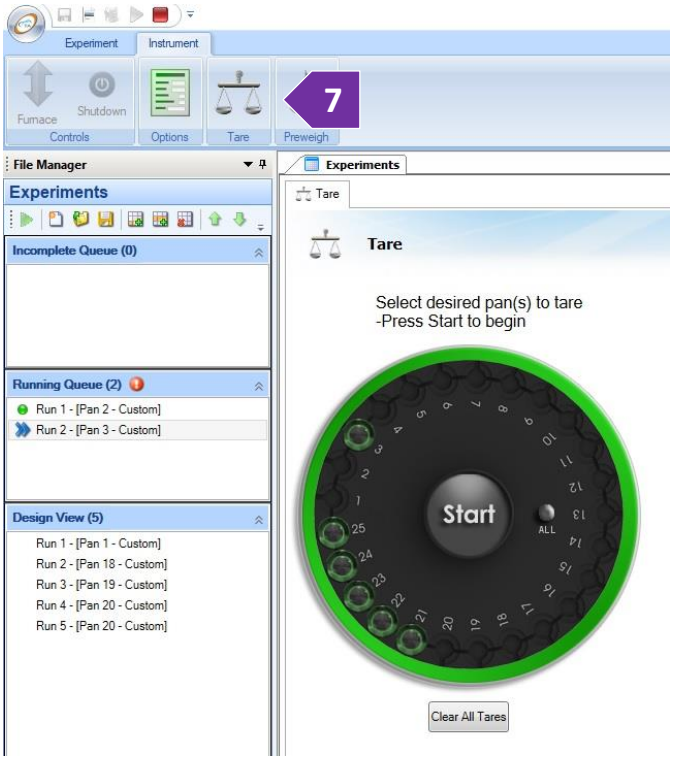


3. To load/unload your sample, rotate the autosampler by hand gently to a position that is convenient to remove the pans + crucibles. Take one of the pans off of the autosampler using a wooden stick (figure 3) and note the number on the carrousel. Pan may or may not have a used crucible onto it. Place the ceramic pan on the bench by the instrument. If an existing used crucible is found, please dispose of the sample using the waste vial located in the hood



4. Place the empty crucible of your choice (Al or ceramic) on the ceramic pan (figure 4)
5. Place the ceramic pan + crucible back in the same position that it was removed from on the autosampler, e.g., if you remove a pan in position #4 on the autosampler, once you place your crucible onto the crucible, place the pan back into position #4. Ensure that the crucible is centered into the ceramic pan

6. Repeat this process for each sample you would like to collect during your reservation
7. Tare the pans + empty crucibles by
   a. Selecting the 'Instrument' menu on the top of the window, and pressing on the 'Tare' icon (figure 7)
   b. Selecting the sample positions on the carrousel by pressing on each position (figure 7 has sample positions 3, 21-25 selected)
   c. Once all positions are selected, press on the large round 'Start' button in the middle of the carrousel graphics. Estimate 3-5 minutes for each tare
   d. Every tared position will be marked with a solid green color



8. Once all pans + empty crucibles are tared, remove them one by one to load your sample. Place each pan on the bench and remove the crucible. Load your sample in the crucible and place the crucible + sample onto the pan. Use a wooden stick to transfer the pan + crucible + sample into the carrousel of the autosampler

MEASUREMENT TYPES FOR THERMAL ANALYSIS EXPERIMENTS

Start the 'Trios' measurement program (icon on the desktop) if it is not running. Double press on the 'Trios' icon and select the instrument (TA TGA5500). It will take a few seconds for the software to connect to the instrument. Once synchronization between the software and instrument is successful, you will see the default 'Trios' window.

Depending on the measurement type, i.e., sample, baseline, or combination of both, you have different options with the software for measuring:

- a sample without pre-existing settings (new procedure)
- a sample with pre-existing settings. In this case, all you must do is open an old sample collection and provide a new file name
- a baseline for samples to be measured (optional)

Please read the detailed instructions below based on the measurement type of your choice.

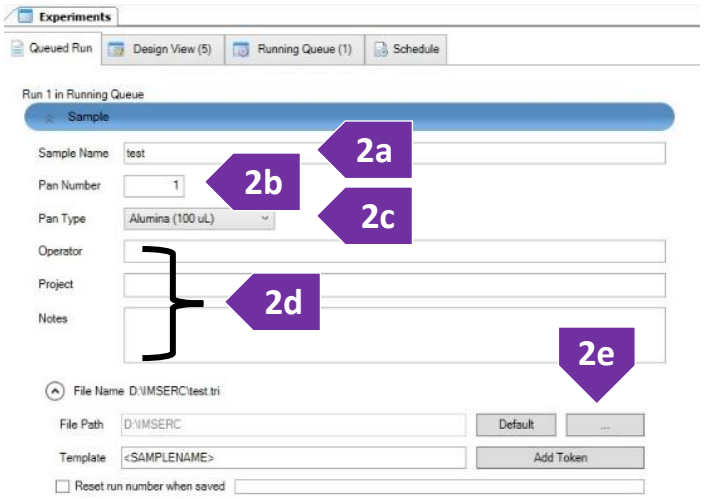
A. TGA/DTA SAMPLE MEASUREMENT WITHOUT AN EXISTING PROCEDURE (NEW PROCEDURE)

To create a completely new measurement without a pre-collected correction file (baseline) or existing procedure that is part of an old measurement:

1. Press on the 'Experiments' tab on the bottom left of the file manager portion of the 'Trios' software and select 'Create New Runs' under the 'Design View' (figure 1). Here we will be able to build a procedure, and ultimately add it to the 'Running Queue'
2. On the 'Design View' tab of the 'Experiments' window, you are going to set the main configuration parameters of your measurement (figure 2):
   a. 'Sample Name' is the name you would like to provide for this specific sample (figure 2a)
   b. 'Pan Number' is the position in the autosampler that this pan has been placed (figure 2b). Note that pan and empty crucible must be pre-tared using the software. See the 'Loading a sample into the instrument' section for more details

c. 'Pan Type' must be selected and accurately represent the type of pan/crucible combination that in on the autosampler. Currently we are only using Alumina pans (100 µl) + crucibles (figure 2c)

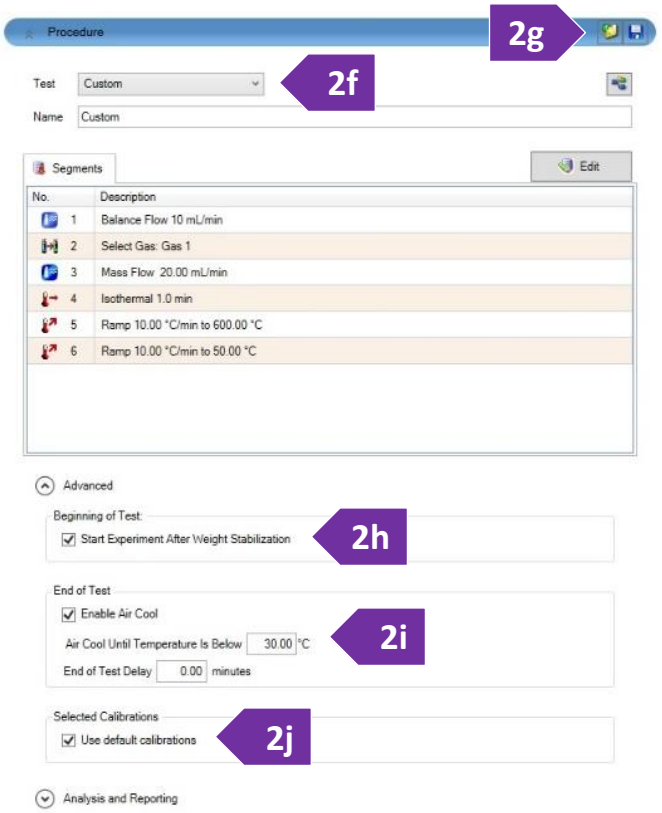
d. (optional) Provide your name (as the operator of this measurement) a project title, or notes. These will all be carried over in the final collection file but are not necessary to start a measurement (figure 2d)



e. 'File Path' is the folder that you would like to save your data. Press the three dots next to the file path field (figure 2e), find your PI's folder on the D drive, and then your own folder (example D:\PI'sLastName\YourName). Leave the template to '<SAMPLENAME>' this will make the filename the same as you have described at the sample name. Leave the 'Reset run number when saved' unchecked.

f. 'Test' field under the Procedure tab is where we can select from a set of pre-made procedures or create your own. The preset procedures include 'Heat and Hold', 'Ramp', and 'Stepwise Isothermal'. It is recommended to either make your own custom procedure or start with a template and then configure it to your needs. Check the 'Procedures' folder for examples of templates.

- To start with a pre-made procedure, simply select whichever pre-made procedure you would like (figure 2f), press on the segments tab, and then change the test type to 'custom'. This will allow a box that says 'Edit' to appear in the upper right-hand corner of the 'segments' field, and then add/take away segments that you do/do not want
- It is highly recommended to include segments that explicitly define the type of gas and the mass flow of the gas since the user before you might have used



completely different gas environments. Add an isothermal step after setting gases to allow for the system to stabilize. Ensure that the right type of gas is connected to the line used by your procedure by following the instructions at the 'Change type of gas' section

- A list of all the available 'Method Segments' is listed in 'Appendix A'. When done with edits, press on the 'Apply' button. Additionally, under the 'Procedures' folders, you'll find a list of available templates for various procedures

g. (optional) Once all the parameters have been selected, you have the option to save the procedure, by clicking on the 'Save Procedure' Icon (figure 2g). Procedure is also permanently saved in your file which you could reload for future measurements

h. Under the 'Advanced' header under the 'Procedure' tab, the box labeled 'Start Experiment After Weight Stabilization' should be checked. This will endure that the weight of the sample has stabilized before the measurement starts (figure 2h). In case your sample is sensitive and for example absorbs or releases moisture over time, you might want to uncheck the 'Start Experiment After Weight Stabilization' option

i. 'End of Test' header shows a box labeled 'Enable Air Cool' which should be checked with the values of 30° C for the field 'Air Cool Temperature Is Below' and 0 minutes of 'End of Test Delay' (figure 2i)

j. 'Selected Calibrations' shows a checkbox that states 'Use Default Calibrations'. This option should also be checked, and will use the calibrations that have been saved to the instrument (figure 2j)

k. Next is to add the run(s) to the running Queue. Right click the run(s) you would like to queue for collection. Do this by selecting all the run(s) of interest, right clicking, and selecting 'Copy to the Running Queue'. You will now see the list of run(s) that you created in the design view, in the 'Running Queue'

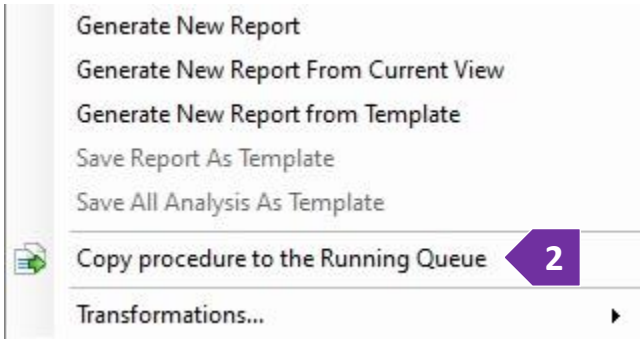
3. Repeat step #2 for each sample by pressing on the 'Append' icon or selecting the append option after right pressing on the 'Design View' or 'Running Queue' area

4. Once all the runs have been added to the running queue, press the green start/play button at the top of the experiment window of 'Trios'. All the runs in the 'Running Queue' tab will be collected in the order that they appear in the list. While the instrument/measurements are running, you could append new runs

B. REPEAT A TGA/DTA MEASUREMENT USING AN EXISTING PROCEDURE

To either repeat or load an existing procedure follow the steps below:

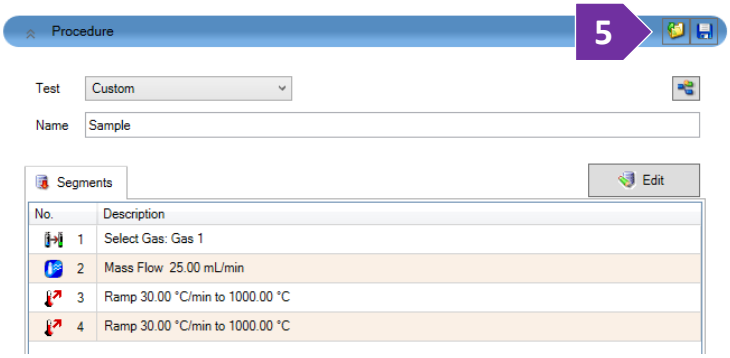
1. Double press on the TRI file that contains the procedure you'd like to reuse. File will be loaded on 'Trios' and data will be shown under the 'Results' tab
2. Right press on the sample name on the lefthanded side and select 'Copy procedure to the Running Queue' that is located towards the end of the menu (figure 2)



3. A new run is created using the existing procedure. Ensure that you have provided a new file name for the new run

Alternatively, you could:

4. Create a new blank run by adding/appending a new run in the queue
5. Load an exported procedure by pressing on the 'Load' button on the 'Procedure' ribbon (figure 5)



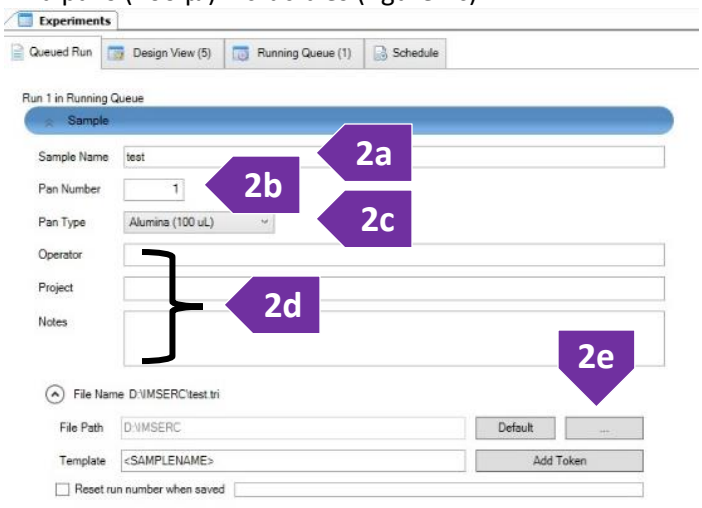
C. TGA-GC-MS MEASUREMENT

Overall, the process of running a thermal analysis measurement coupled with GC-MS, requires:

1. The creation of a thermal analysis procedure that contains a trigger ('Event1' segment) for starting the GC-MS collection
2. The creation of a GC-MS method using the GC-MS acquisition software
3. The connection of a transfer line between the TGA furnace and the GC-MS

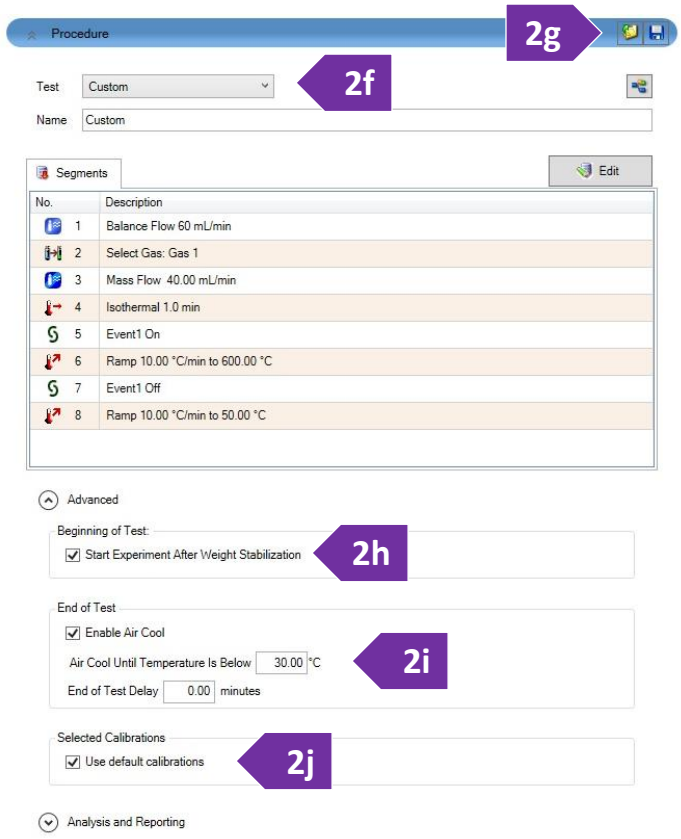
Please follow the steps below for setting up a TGA-GC-MS measurement for a single sample (without high throughput):

1. Press on the 'Experiments' tab on the bottom left of the file manager portion of the 'Trios' software and select 'Create New Runs' under the 'Design View' (figure 1). Here we will be able to build a procedure, and ultimately add it to the 'Running Queue'
2. On the 'Design View' tab of the 'Experiments' window, you are going to set the main configuration parameters of your measurement (figure 2):
   a. 'Sample Name' is the name you would like to provide for this specific sample (figure 2a)
   b. 'Pan Number' is the position in the autosampler that this pan has been placed (figure 2b). Note that pan and empty crucible must be pre-tared using the software. See the 'Loading a sample into the instrument' section for more details
   c. 'Pan Type' must be selected and accurately represent the type of pan/crucible combination that in on the autosampler. Currently we are only using Alumina pans (100 µl) + crucibles (figure 2c)
   d. (optional) Provide your name (as the operator of this measurement) a project title, or notes. These will all be carried over in the final collection file but are not necessary to start a measurement (figure 2d)
   e. 'File Path' is the folder that you would like to save your data. Press the three dots next to the file path field (figure 2e), find your PI's folder on the D drive, and then your own



folder (example D:\PI'sLastName\YourName). Leave the template to '<SAMPLENAME>' this will make the filename the same as you have described at the sample name. Leave the 'Reset run number when saved' unchecked.

f. 'Test' field under the Procedure tab is where we can select from a set of pre-made procedures or create your own. The preset procedures include 'Heat and Hold', 'Ramp', and 'Stepwise Isothermal'. It is recommended to either make your own custom procedure or start with a template and then configure it to your needs. Check the 'Procedures' folder for examples of templates.



- To start with a pre-made procedure, simply select whichever pre-made procedure you would like (figure 2f), press on the segments tab, and then change the test type to 'custom'. This will allow a box that says 'Edit' to appear in the upper right-hand corner of the 'segments' field, and then add/take away segments that you do/do not want
- It is highly recommended to include segments that explicitly define the type of gas and the mass flow of the gas since the user before you might have used completely different gas environments. Add an isothermal step after setting gases to allow for the system to stabilize. Ensure that the right type of gas is connected to the line used by your procedure by following the instructions at the 'Change type of gas' section. Set the balance flow of the TGA to 60 ml/min and the gas flow to 40 ml/min. To trigger the GC-MS valves, the 'Event1' segments must be utilized
- A list of all the available 'Method Segments' is listed in 'Appendix A'. When done with edits, press on the 'Apply' button. Additionally, under the 'Procedures' folders, you'll find a list of available templates for various procedures

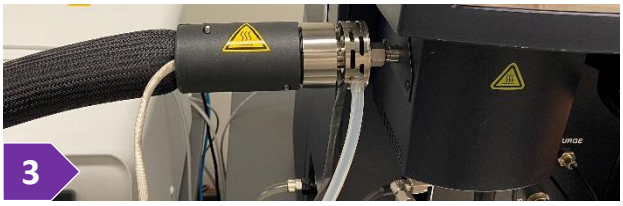
g. (optional) Once all the parameters have been selected, you have the option to save the procedure, by clicking on the 'Save Procedure' Icon (figure 2g). Procedure is also permanently saved in your file which you could reload for future measurements

h. Under the 'Advanced' header under the 'Procedure' tab, the box labeled 'Start Experiment After Weight Stabilization' should be checked. This will endure that the weight of the sample has stabilized before the measurement starts (figure 2h). In case your sample is sensitive and for example absorbs or releases moisture over time, you might want to uncheck the 'Start Experiment After Weight Stabilization' option

i. 'End of Test' header shows a box labeled 'Enable Air Cool' which should be checked with the values of 30° C for the field 'Air Cool Temperature Is Below' and 0 minutes of 'End of Test Delay' (figure 2i)

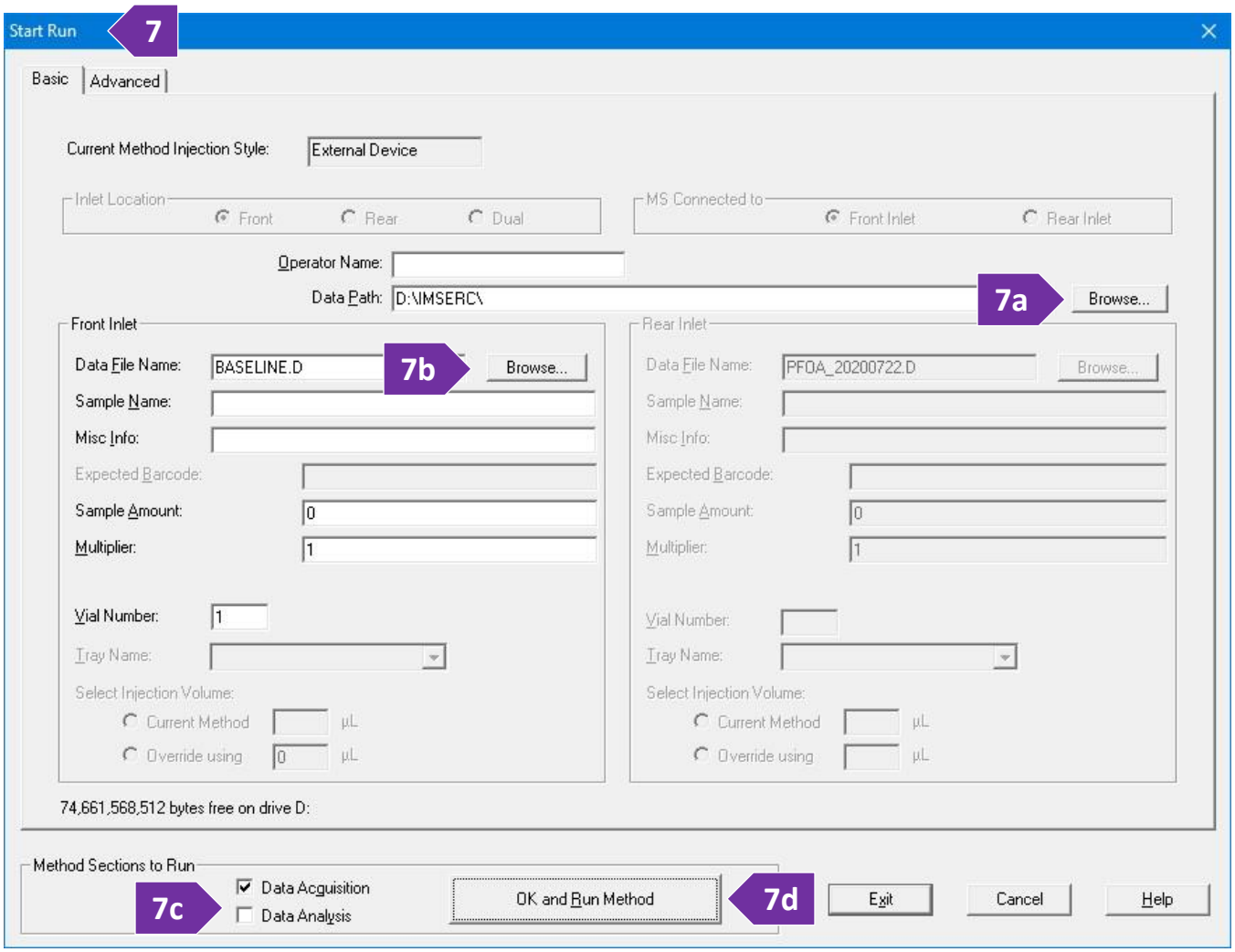
j. 'Selected Calibrations' shows a checkbox that states 'Use Default Calibrations'. This option should also be checked, and will use the calibrations that have been saved to the instrument (figure 2j)

k. Next is to add the run(s) to the running Queue. Right click the run(s) you would like to queue for collection. Do this by selecting all the run(s) of interest, right clicking, and selecting 'Copy to the Running Queue'. You will now see the list of run(s) that you created in the design view, in the 'Running Queue'



3. Ensure that the transfer line is connected on the left side of TGA furnace (figure 3)
4. On the controller between the GC-MS and FT-IR, ensure that:
   - The 'Heating' button is enabled, and the temperature of all components is at 280C (figure 4)
   - The 'Pump on' button is enabled, and the PV flow is at 80 µl/min (figure 4). If there is no flow, please contact a staff
5. On the STA (not TGA) computer, launch the GC-MS acquisition software (ChemStation) in case is not running already. Icon of the GC-MS software is on the desktop labeled as 'TGA-GC-MS'
6. Load the method that was created for you during your training session by pressing on the 'Method' menu and selecting 'Load Method'. Wait for a few seconds until the method is loaded and the cursor becomes responsive again

7. Run your method by pressing on the 'Method' menu and selecting 'Run Method'
   a. On the 'Start Run' window (figure 7), provide the path for where your GC-MS data will be saved under by pressing on the 'Browse' button next to the 'Data Path' field (figure 7a)
   b. Provide the folder name of where the GC-MS will be saved under by pressing on the 'Browse' button next to the 'Data File Name' field (figure 7b)

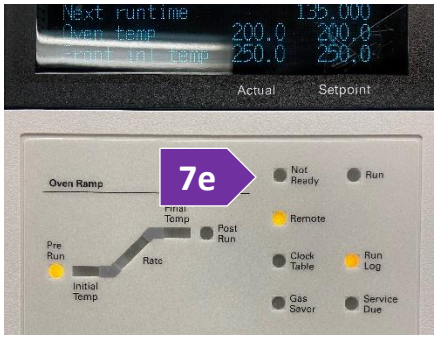


c. At the bottom-left corner of the window, tick the option 'Data Acquisition' and untick the option 'Data Analysis' (figure 7c)

d. Press on the 'OK and Run Method' to run the method (figure 7d). Within a few seconds, GC-MS instrument will go into standby mode and instrument will wait for the trigger signal from the thermal analysis software

e. The 'Not Ready' light indicator should be off (figure 7e) before going to the next step

8. On the thermal analysis program ('Trios'), press on the green play button on the toolbox to start the measurement. Once the measurement starts, the live TGA trace will be loaded and shown on the 'Results' tab



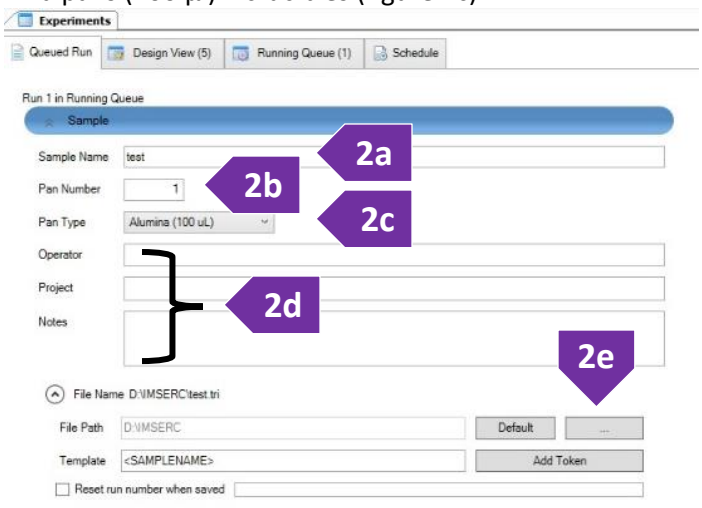
D. TGA-FT-IR MEASUREMENT

Overall, the process of running a thermal analysis measurement coupled with FT-IR, requires:

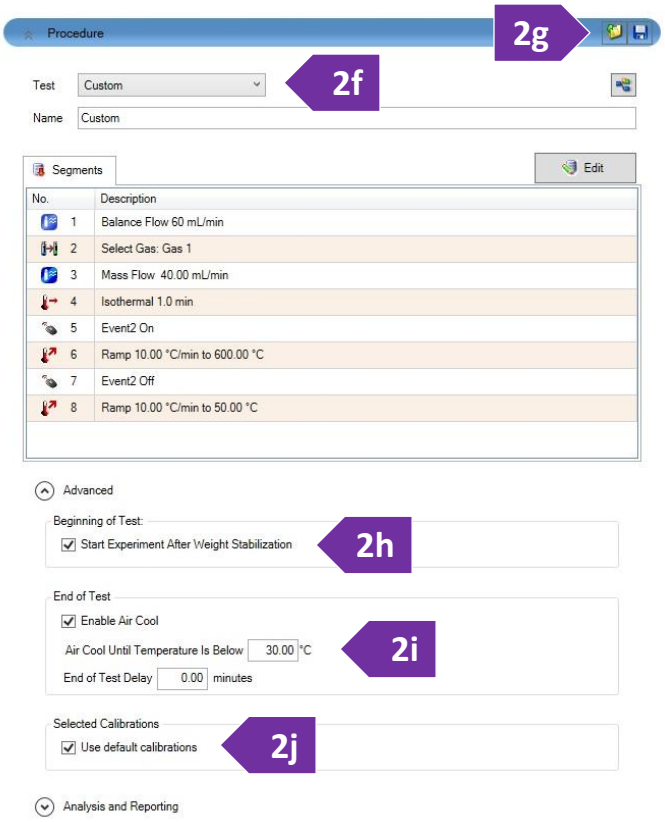
1. The creation of a thermal analysis procedure that contains a trigger ('Event2' segment) for starting the FT-IR collection
2. The creation of a FT-IR series using the FT-IR acquisition software
3. The connection of a transfer line between the TGA furnace and the FT-IR

Please follow the steps below for setting up a TGA-FT-IR measurement for a single sample (without high throughput):

1. Press on the 'Experiments' tab on the bottom left of the file manager portion of the 'Trios' software and select 'Create New Runs' under the 'Design View' (figure 1). Here we will be able to build a procedure, and ultimately add it to the 'Running Queue'
2. On the 'Design View' tab of the 'Experiments' window, you are going to set the main configuration parameters of your measurement (figure 2):
   a. 'Sample Name' is the name you would like to provide for this specific sample (figure 2a)
   b. 'Pan Number' is the position in the autosampler that this pan has been placed (figure 2b). Note that pan and empty crucible must be pre-tared using the software. See the 'Loading a sample into the instrument' section for more details
   c. 'Pan Type' must be selected and accurately represent the type of pan/crucible combination that in on the autosampler. Currently we are only using Alumina pans (100 µl) + crucibles (figure 2c)
   d. (optional) Provide your name (as the operator of this measurement) a project title, or notes. These will all be carried over in the final collection file but are not necessary to start a measurement (figure 2d)
   e. 'File Path' is the folder that you would like to save your data. Press the three dots next to the file path field (figure 2e), find your PI's folder on the D drive, and then your own



folder (example D:\PI'sLastName\YourName). Leave the template to '<SAMPLENAME>' this will make the filename the same as you have described at the sample name. Leave the 'Reset run number when saved' unchecked.



f. 'Test' field under the Procedure tab is where we can select from a set of pre-made procedures or create your own. The preset procedures include 'Heat and Hold', 'Ramp', and 'Stepwise Isothermal'. It is recommended to either make your own custom procedure or start with a template and then configure it to your needs. Check the 'Procedures' folder for examples of templates.

   - To start with a pre-made procedure, simply select whichever pre-made procedure you would like (figure 2f), press on the segments tab, and then change the test type to 'custom'. This will allow a box that says 'Edit' to appear in the upper right-hand corner of the 'segments' field, and then add/take away segments that you do/do not want
   - It is highly recommended to include segments that explicitly define the type of gas and the mass flow of the gas since the user before you might have used completely different gas environments. Add an isothermal step after setting gases to allow for the system to stabilize. Ensure that the right type of gas is connected to the line used by your procedure by following the instructions at the 'Change type of gas' section. Set the balance flow of the TGA to 60 ml/min and the gas flow to 40 ml/min. To trigger the FT-IR, the 'Event2' segments must be utilized
   - A list of all the available 'Method Segments' is listed in 'Appendix A'. When done with edits, press on the 'Apply' button. Additionally, under the 'Procedures' folders, you'll find a list of available templates for various procedures

g. (optional) Once all the parameters have been selected, you have the option to save the procedure, by clicking on the 'Save Procedure' Icon (figure 2g). Procedure is also permanently saved in your file which you could reload for future measurements

h. Under the 'Advanced' header under the 'Procedure' tab, the box labeled 'Start Experiment After Weight Stabilization' should be checked. This will endure that the weight of the sample has stabilized before the measurement starts (figure 2h). In case your sample is sensitive and for example absorbs or releases moisture over time, you might want to uncheck the 'Start Experiment After Weight Stabilization' option

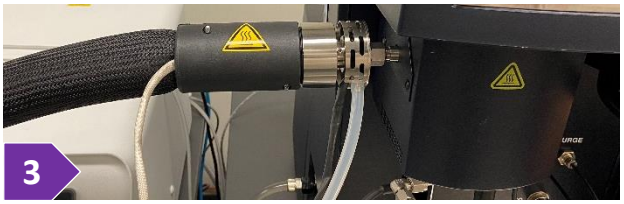
i. 'End of Test' header shows a box labeled 'Enable Air Cool' which should be checked with the values of 30° C for the field 'Air Cool Temperature Is Below' and 0 minutes of 'End of Test Delay' (figure 2i)

j. 'Selected Calibrations' shows a checkbox that states 'Use Default Calibrations'. This option should also be checked, and will use the calibrations that have been saved to the instrument (figure 2j)

k. Next is to add the run(s) to the running Queue. Right click the run(s) you would like to queue for collection. Do this by selecting all the run(s) of interest, right clicking, and selecting 'Copy to the Running Queue'. You will now see the list of run(s) that you created in the design view, in the 'Running Queue'

3. Ensure that the transfer line is connected on the left side of TGA furnace (figure 3)

4. On the controller between the GC-MS and FT-IR, ensure that:
   - The 'Heating' button is enabled, and the temperature of all components is at 280C (figure 4)
   - The 'Pump on' button is enabled, and the PV flow is at 80 µl/min (figure 4). If there is no flow, please contact a staff

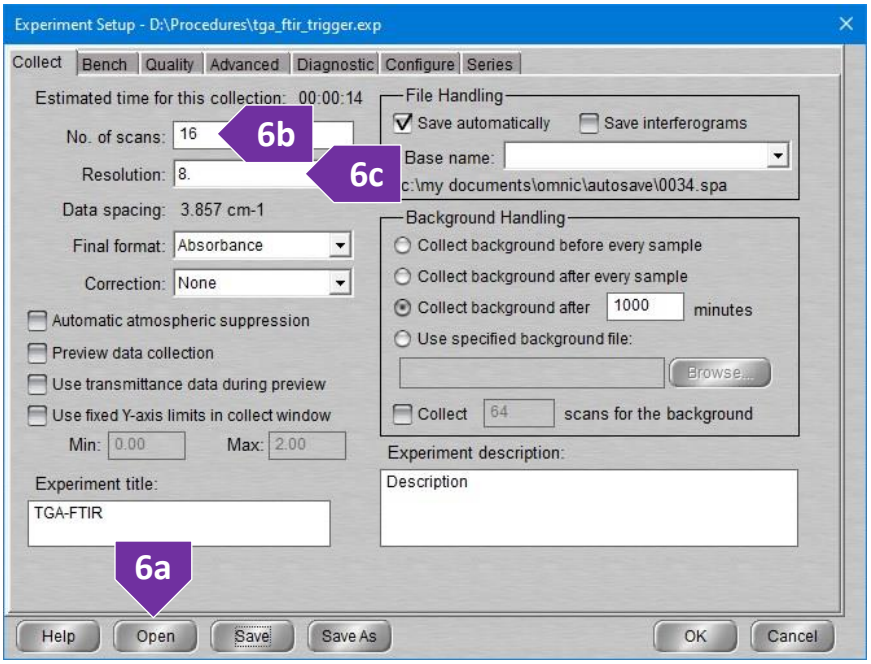


5. On the FT-IR (not TGA) computer, launch the FT-IR acquisition software (Omnic) in case is not running already. Icon of the FT-IR software is on the desktop labeled as 'Omnic'

6. Under the menu 'Collect', select the 'Experiment Setup' option. On the 'Collect' tab:
   a. Press on the 'Open' button (figure 6a) and load the 'tga_ftir_trigger.exp' method located under the 'Procedures' folder
   b. Select the number of scans needed for each spectrum (figure 6b)
   c. Select the desired resolution (figure 6c)

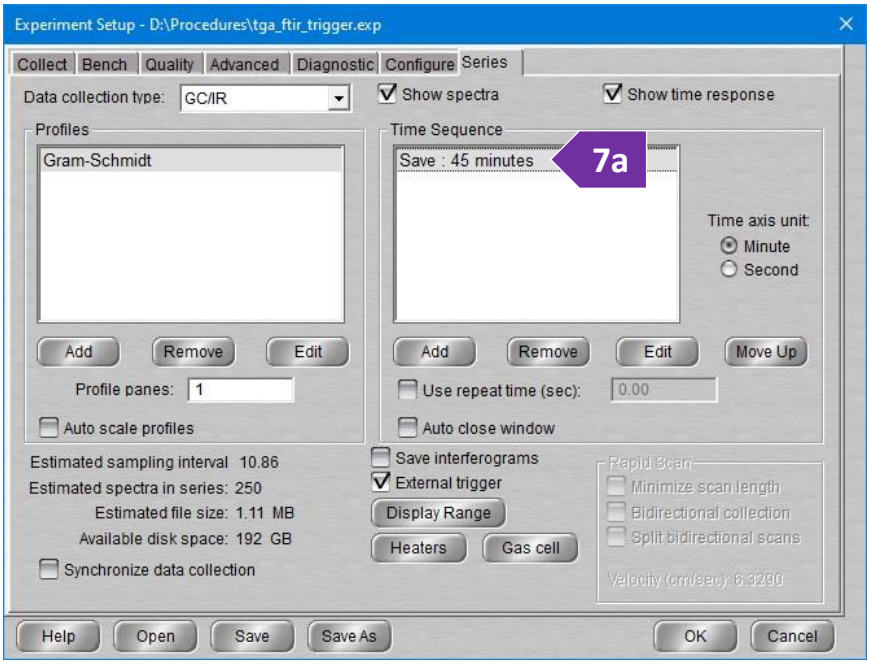
   d. Estimated collection time for the collection of one spectrum is indicated at the 'Estimated time for this collection' field. The higher the number of scans and/or the higher the resolution, the longer it will take for the collection. This information is important since the overall collection time will define the number of total spectra to be collected during the TGA measurement

7. On the 'Experiment Setup' window, select the 'Series' tab:
   a. In the 'Time Sequence' filed, double press the 'Save: 45 minutes' line and type in the duration of the overall FTIR experiment (figure 7a). Duration is typically a few minutes longer than the TGA heating segment
   b. Press on the 'OK' button to store the new time information



   c. Note the estimated number of spectra to be collected as indicated on the 'Estimated spectra in series' label. The total number of spectra will define the time resolution of the TGA-FT-IR collection
   d. Press on the 'OK' button to save and close the 'Experiment Setup' window

8. Start the FT-IR series method by pressing on the 'Collect' menu and selecting 'Collect Series'
9. Provide a file name for the FT-IR series file and save it under your personal folder in your group folder
10. On the thermal analysis program ('Trios'), press on the green play button on the toolbox to start the measurement. Once the measurement starts, the live TGA trace will be loaded and shown on the 'Results' tab



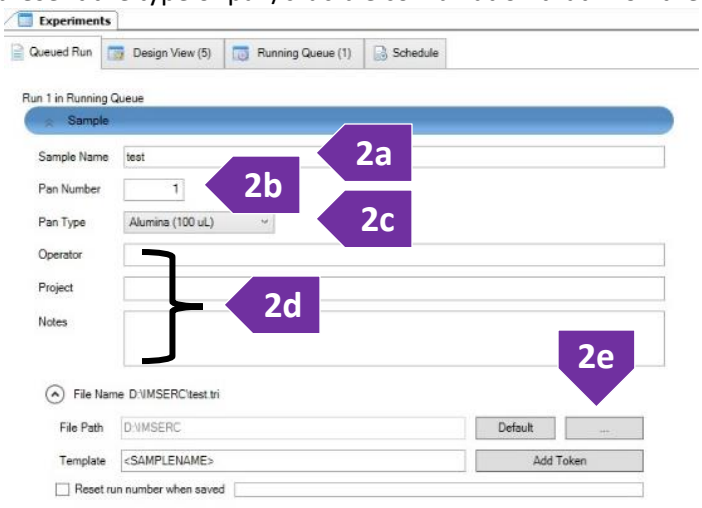
E. TGA-FT-IR-GC-MS MEASUREMENT

Overall, the process of running a thermal analysis measurement coupled with FT-IR-GC-MS, requires:

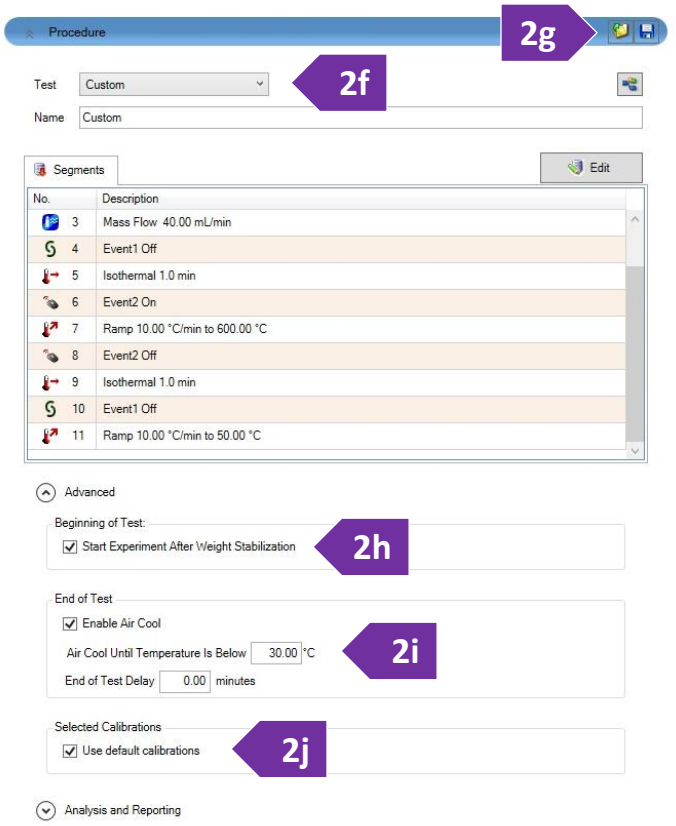
1. The creation of a thermal analysis procedure that contains a trigger ('Event1' segment) for starting the GC-MS collection and a second trigger ('Event2' segment) for starting the FT-IR collections
2. The creation of a GC-MS method using the GC-MS acquisition software
3. The creation of an FT-IR series method using the FT-IR acquisition software
4. The connection of a transfer line between the TGA furnace, the FT-IR heated gas cell, and the GC-MS

Please follow the steps below for setting up a TGA-FT-IR-GC-MS measurement for a single sample (without high throughput):

1. Press on the 'Experiments' tab on the bottom left of the file manager portion of the 'Trios' software and select 'Create New Runs' under the 'Design View' (figure 1). Here we will be able to build a procedure, and ultimately add it to the 'Running Queue'
2. On the 'Design View' tab of the 'Experiments' window, you are going to set the main configuration parameters of your measurement (figure 2):
   a. 'Sample Name' is the name you would like to provide for this specific sample (figure 2a)
   b. 'Pan Number' is the position in the autosampler that this pan has been placed (figure 2b). Note that pan and empty crucible must be pre-tared using the software. See the 'Loading a sample into the instrument' section for more details
   c. 'Pan Type' must be selected and accurately represent the type of pan/crucible combination that in on the autosampler. Currently we are only using Alumina pans (100 µl) + crucibles (figure 2c)
   d. (optional) Provide your name (as the operator of this measurement) a project title, or notes. These will all be carried over in the final collection file but are not necessary to start a measurement (figure 2d)
   e. 'File Path' is the folder that you would like to save your data. Press the three dots next to



the file path field (figure 2e), find your PI's folder on the D drive, and then your own folder (example D:\PI'sLastName\YourName). Leave the template to '<SAMPLENAME>' this will make the filename the same as you have described at the sample name. Leave the 'Reset run number when saved' unchecked.



f. 'Test' field under the Procedure tab is where we can select from a set of pre-made procedures or create your own. The preset procedures include 'Heat and Hold', 'Ramp', and 'Stepwise Isothermal'. It is recommended to either make your own custom procedure or start with a template and then configure it to your needs. Check the 'Procedures' folder for examples of templates.

- To start with a pre-made procedure, simply select whichever pre-made procedure you would like (figure 2f), press on the segments tab, and then change the test type to 'custom'. This will allow a box that says 'Edit' to appear in the upper right-hand corner of the 'segments' field, and then add/take away segments that you do/do not want
- It is highly recommended to include segments that explicitly define the type of gas and the mass flow of the gas since the user before you might have used completely different gas environments. Add an isothermal step after setting gases to allow for the system to stabilize. Ensure that the right type of gas is connected to the line used by your procedure by following the instructions at the 'Change type of gas' section. Set the balance flow of the TGA to 60 ml/min and the gas flow to 40 ml/min. To trigger the GC-MS valves, the 'Event1' segments must be utilized. To trigger the FT-IR, the 'Event2' segments must be utilized
- A list of all the available 'Method Segments' is listed in 'Appendix A'. When done with edits, press on the 'Apply' button. Additionally, under the 'Procedures' folders, you'll find a list of available templates for various procedures

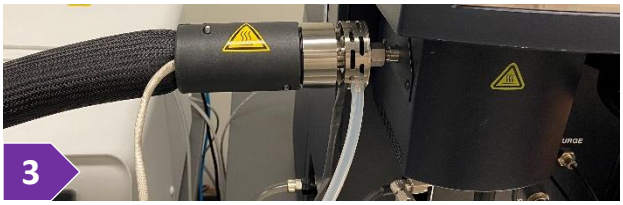
g. (optional) Once all the parameters have been selected, you have the option to save the procedure, by clicking on the 'Save Procedure' Icon (figure 2g). Procedure is also permanently saved in your file which you could reload for future measurements

h. Under the 'Advanced' header under the 'Procedure' tab, the box labeled 'Start Experiment After Weight Stabilization' should be checked. This will endure that the weight of the sample has stabilized before the measurement starts (figure 2h). In case your sample is sensitive and for example absorbs or releases moisture over time, you might want to uncheck the 'Start Experiment After Weight Stabilization' option

i. 'End of Test' header shows a box labeled 'Enable Air Cool' which should be checked with the values of 30° C for the field 'Air Cool Temperature Is Below' and 0 minutes of 'End of Test Delay' (figure 2i)

j. 'Selected Calibrations' shows a checkbox that states 'Use Default Calibrations'. This option should also be checked, and will use the calibrations that have been saved to the instrument (figure 2j)

k. Next is to add the run(s) to the running Queue. Right click the run(s) you would like to queue for collection. Do this by selecting all the run(s) of interest, right clicking, and selecting 'Copy to the Running Queue'. You will now see the list of run(s) that you created in the design view, in the 'Running Queue'



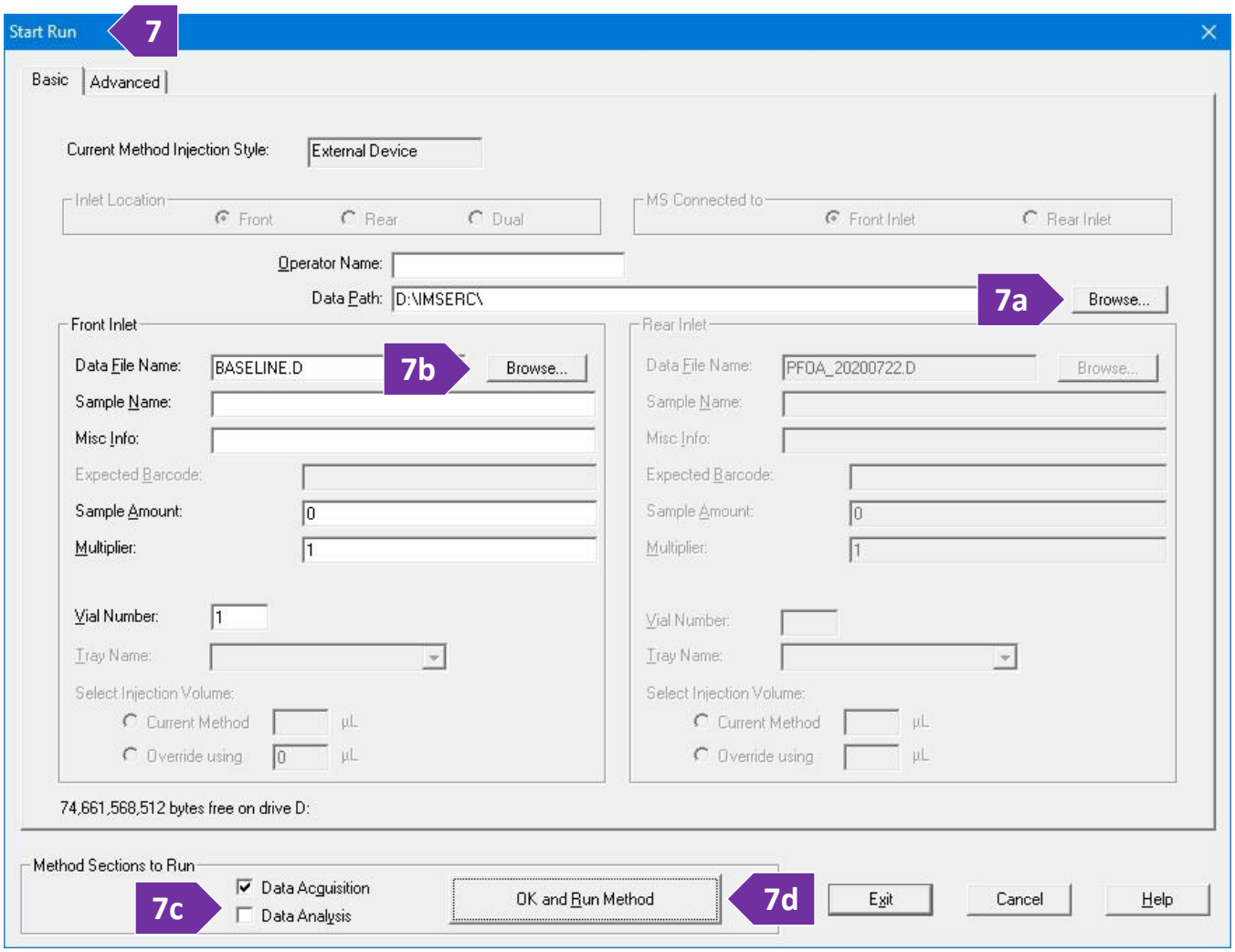
3. Ensure that the transfer line is connected on the left side of TGA furnace (figure 3)

4. On the controller between the GC-MS and FT-IR, ensure that:
   - The 'Heating' button is enabled, and the temperature of all components is at 280C (figure 4)
   - The 'Pump on' button is enabled, and the PV flow is at 80 µl/min (figure 4). If there is no flow, please contact a staff

5. On the STA (not TGA) computer, launch the GC-MS acquisition software (ChemStation) in case is not running already. Icon of the GC-MS software is on the desktop labeled as 'TGA-GC-MS'

6. Load the method that was created for you during your training session by pressing on the 'Method' menu and selecting 'Load Method'. Wait for a few seconds until the method is loaded and the cursor becomes responsive again

7. Run your method by pressing on the 'Method' menu and selecting 'Run Method'



a. On the 'Start Run' window (figure 7), provide the path for where your GC-MS data will be saved under by pressing on the 'Browse' button next to the 'Data Path' field (figure 7a)

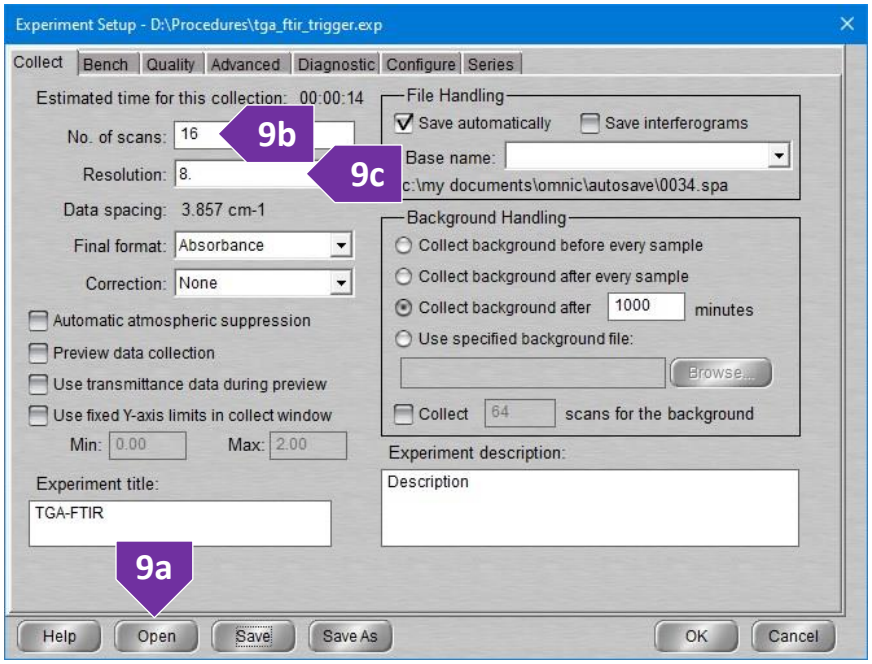
b. Provide the folder name of where the GC-MS will be saved under by pressing on the 'Browse' button next to the 'Data File Name' field (figure 7b)

c. At the bottom-left corner of the window, tick the option 'Data Acquisition' and untick the option 'Data Analysis' (figure 7c)

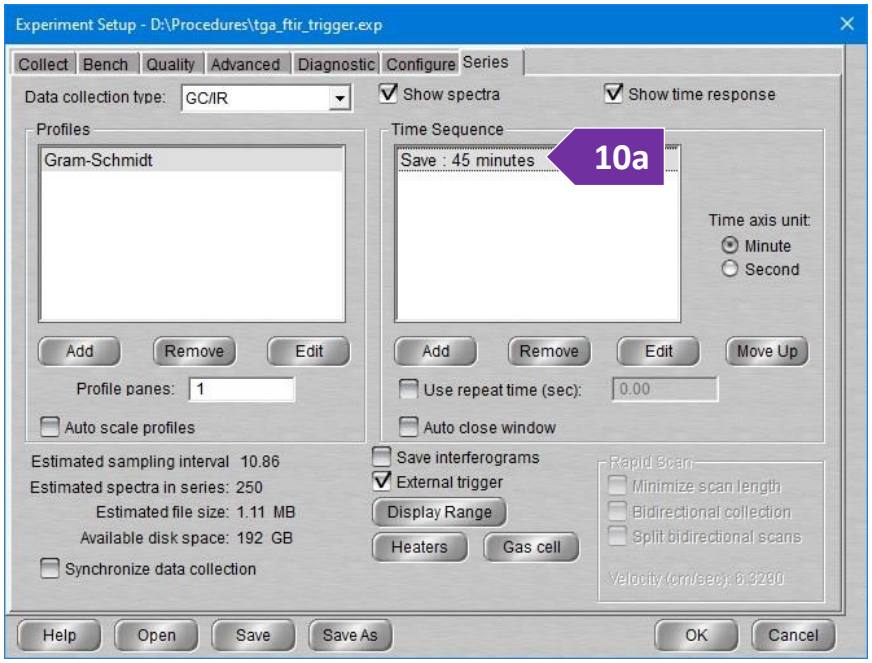
d. Press on the 'OK and Run Method' to run the method (figure 7d). Within a few seconds, GC-MS instrument will go into standby mode and instrument will wait for the trigger signal from the thermal analysis software

e. The 'Not Ready' light indicator should be off (figure 7e) before going to the next step

8. On the FT-IR (not TGA) computer, launch the FT-IR acquisition software (Omnic) in case is not running already. Icon of the FT-IR software is on the desktop labeled as 'Omnic'
9. Under the menu 'Collect', select the 'Experiment Setup' option. On the 'Collect' tab:
   a. Press on the 'Open' button (figure 9a) and load the 'tga_ftir_trigger.exp' method located under the 'Procedures' folder
   b. Select the number of scans needed for each spectrum (figure 9b)
   c. Select the desired resolution (figure 9c)
   d. Estimated collection time for the collection of one spectrum is indicated at the 'Estimated time for this collection' field. The higher the number of scans and/or the higher the resolution, the longer it will take for the collection. This information is important since the overall collection time will define the number of total spectra to be collected during the TGA measurement



10. On the 'Experiment Setup' window, select the 'Series' tab:
    a. In the 'Time Sequence' filed, double press the 'Save: 45 minutes' line and type in the duration of the overall FTIR experiment (figure 10a). Duration is typically a few minutes longer than the TGA heating segment



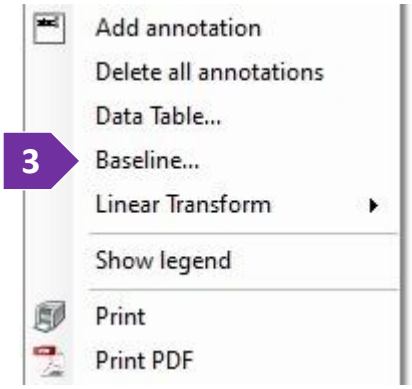
b. Press on the 'OK' button to store the new time information
c. Note the estimated number of spectra to be collected as indicated on the 'Estimated spectra in series' label. The total number of spectra will define the time resolution of the TGA-FT-IR collection
d. Press on the 'OK' button to save and close the 'Experiment Setup' window

11. Start the FT-IR series method by pressing on the 'Collect' menu and selecting 'Collect Series'
12. Provide a file name for the FT-IR series file and save it under your personal folder in your group folder
13. On the thermal analysis program ('Trios'), press on the green play button on the toolbox to start the measurement. Once the measurement starts, the live TGA trace will be loaded and shown on the 'Results' tab

F. COLLECTION FOR A BASELINE (OPTIONAL)

If you would like to collect a baseline and do a manual subtraction, you will follow the steps laid out in section 'A. TGA/DTA sample measurement without an existing procedure (New Procedure)' of this manual and collect data on just an empty crucible + pan. Be sure to collect under the same conditions for your baseline, as you did for your data collection (that includes using the same type of crucibles).

Once you have your data collection, and your baseline collection, we will need to go to the 'Results' tab of 'Trios' on the left-hand side.



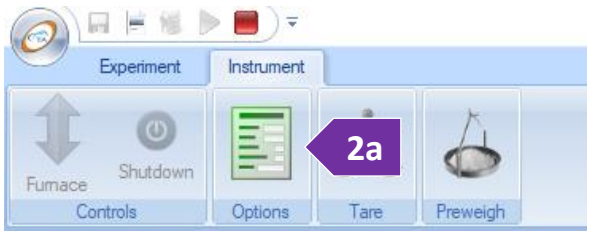
1. First, open the baseline file
2. Open the data collection file and select the signals that will be used to perform operations within the baseline file
3. Be sure that the data collection file is currently active, and then right-click the graph and select Baseline from the menu (figure 3)
4. Now the Baseline dialog box displays. Select the desired reference file to be used as the baseline from the available list (figure 4)
5. Check the desired signals from those shown in the variable box
6. Click on the desired base (Time or Temperature)
7. Click on the desired Operation (add or subtract the baseline file)
8. Click on the desired signal match (Name and units, or units only)
9. Select the 'Limit data to the range of the reference' if the baseline file has a smaller range than the data collection file
10. Finally press 'OK', and then save the corrected file. (It is recommended to use the same settings as shown in figure 4)

CHANGE TYPE OF GAS

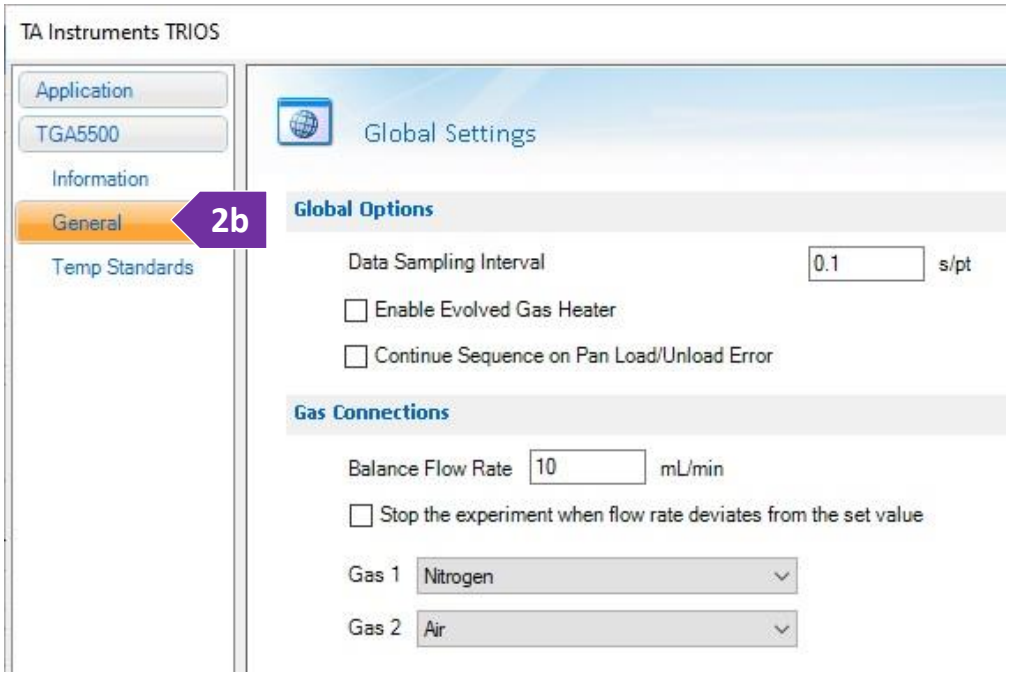
The TA TGA5500 is equipped with two mass flow controllers (MFCs) for an accurate control of the gases flowing through the furnace during a measurement. The two controllers are labeled as 'Gas 1' and 'Gas 2'. The 'Gas 1' controller is connected to an inert gas line. The 'Gas 2' controller is dedicated for any non-standard gas needed for your measurement. To change the type of gas flowing through either 'Gas 1' or 'Gas 2' controller, you need to follow two steps. First step is to select the appropriate gas using a valve control script, and the second step is to change the digital label of the gas defined in the acquisition software:

1. To change the type of gas to what is needed for your measurement, use the 'tga_gas1_port_control' or 'tga_gas2_port_control' script, depending on the gas controller used for your experiment:
   a. Look at the windows taskbar to see if the script is running (circled in figure 1a). If script is not running, double press on the 'tga_gasX_port_control' icon on the desktop
   b. Press anywhere on the large red button with the label of the preferred gas (figure 1b). Button of the selected gas should turn green within five seconds. If this is not the case, please press again on the button showing the gas of interest. Inert gases that are always available for 'Gas 1' include nitrogen, argon, and Helium. For any other special gas, please talk to a staff before scheduling your experiment. Special gases are used only on 'Gas 2'. Additionally, add a note in NUcore about the special gas request

   c. Minimize the 'tga_gasX_port_control' script. In case you accidentally closed the script, the selected gas valve will remain open



2. To change the digital label of the gas in the acquisition software:
   a. Select the 'Instrument' menu on the top of the window, and press on the 'Options' icon (figure 2a). It will take a few seconds for the 'Options' window to appear
   b. On the 'Options' window, select the 'General' tab on the lefthand side (figure 2b), and select the appropriate gas type for either 'Gas 1' and/or 'Gas 2' on the righthand side. Balance gas is of the same type as 'Gas 1'
   c. Press on the 'OK' button to save the changes


TA Instruments TRIOS
Application
TGA5500
Information
General
2b
Temp Standards
Global Settings
Global Options
Data Sampling Interval
0.1
s/pt
Enable Evolved Gas Heater
Continue Sequence on Pan Load/Unload Error
Gas Connections
Balance Flow Rate
10
mL/min
Stop the experiment when flow rate deviates from the set value
Gas 1
Nitrogen
Gas 2
Air

PUBLICATION

A. EXPERIMENTAL SECTION

Modify the text below according to the setup and conditions you used during the measurement:

"Thermogravimetric thermal analyses were performed in a TA TGA5500 instrument. ***XX mg*** of sample ***YY*** were placed in an ***Alumina | Aluminum XX ml*** crucible with weight of ***ZZ mg***. Sample was measured under ***ultra-high purity Helium gas (XX ml/min) | dry air (YY ml/min)***. Temperature was increased at a rate of ***XX C/min*** and gases were transferred to the **GC/MS and/or FT-IR** instrumentation via a heated (280 °C) transfer line. An Agilent Technologies 7890A GC system equipped with a non-polar capillary column (Agilent J&B HP-5 packed with (5%-Phenyl)-methylpolysiloxane) coupled with a 5975 MSD spectrometer was used for the analyses of the gases released from the samples. A gas injection was triggered every ***XX minutes*** from the beginning of the heating cycle and 0.25 ml of gas was sampled from the gases released by the compound and carrier gas (Helium). The detection limit is typically better than 100 fg but this value can be larger, and it highly depends on the ionization efficiency of the different molecules in the compound. Mass spectra were scanned in the range of ***XX-YY u***. A Nicolet 6700 FT-IR spectrometer equipped with a heated gas cell was used. The FT-IR and GC/MS were connected in series with a transfer line. Each FT-IR spectrum was averaged from **ZZ** number of scans and the collection time of each spectrum was approximately **X.X** mins. Multiple spectra were collected as a function of time. Performance of the thermobalance of the TA TGA5500 was verified by using a certified sample of calcium oxalate monohydrate (European Pharmacopoeia Reference Standard) up to 1000 °C."

B. ACKNOWLEDGEMENT

"This work made use of the IMSERC facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-2025633), and Northwestern University."

TROUBLESHOOTING

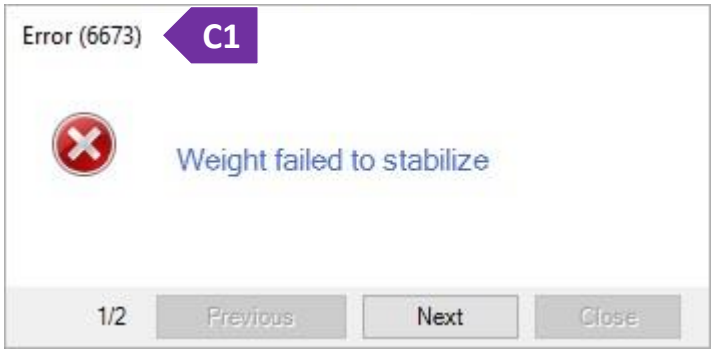
A. THE COMPUTER SCREEN WILL NOT TURN ON

Begin your reservation in NUcore to initiate access to the instrument

B. COMPUTER REQUIRES LOGIN AND A PASSWORD

The default 'TGA' user account should be logged in. In case the computer was restarted, the password for the 'TGA' account is (see hardcopy by the instrument). See 'Default instrument status' section for more details

C. SPECIFIC ERROR MESSAGES



1. ***Error (6673): Weight failed to stabilize*** (figure C1). Most likely the weight of the sample changes over time. Under the 'Advanced' header under the 'Procedure' tab, uncheck the box labeled 'Start Experiment After Weight Stabilization'. This will start the measurement without waiting for the default stabilization time (see figure 2h under section A)

D. FURNACE IS GLOWING

This is perfectly normal for the furnace to be glowing when above 600 °C (figure D)

D. THERE IS AN ERROR/PROBLEM WITH THE INSTRUMENT THAT IS NOT ADDRESSED UNDER THE TROUBLESHOOTING SECTION

If there is an error or problem with the instrument which is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue
2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html add place the 'Stop' sign near the instrument computer.

'Stop' signs are located on the shelf above the computers in BG51 and online at the link above. Email or talk to a staff member

3. Email or talk to a staff member

APPENDICIES

APPENDIX A: METHOD SEGMENTS EXPLANATIONS

Table below was taken directly from the user manual. Some of the segments might not be available based on the hardware configuration.

Segment	Description
Abort	The **Abort** segment skips over the next segment when specified limit conditions are met. • If the limit is reached at the beginning of a segment, then that segment is skipped and method execution continues with the next segment. • If the limit is reached during the execution of a segment, then the remaining portion of the segment is skipped. • NOTE: The **Abort** segment is generally followed by a **Ramp** or **Isothermal** segment. Example (DSC): 1. Equilibrate at 200°C 2. Abort next segment if mW>1 3. Isothermal for 100 min
Balance Flow Rate	This segment is used to alter the rate of flow of the selected gas to the balance. Example: Flow rate 50 mL/min
Data	The **Data** segment controls data collection during the experiment. If a **Data** segment is not used, data storage is automatically initiated by the first **Ramp**, **Isothermal**, or **Step** segment that appears in the method. Example: Data Storage: On
Electromagnet	The Discovery TGA has a magnetic coil surrounding the furnace. The **Electromagnet** segment allows you to apply a magnetic field during an experiment so that temperature calibration using Curie point standards may be performed. Example: Electromagnet: On at 100% Ramp 10°C/min to 250°C
Equilibrate	The **Equilibrate** segment heats or cools the furnace to the defined temperature, stabilizes the furnace at that temperature, then continues to the next segment. This segment does not automatically start data collection. Example: Equilibrate at 200°C

Event 1 / **Event 2**	The **Event** segment controls the external event relay through the event jack on the back of the instrument. This is used to synchronize control of additional hardware through the method. Example: Event 1: On
High Resolution Ramp/Sensitivity	*Available for TGA High Resolution Instruments only*:The **High ResolutionRamp** segment varies the heating/cooling rate of the furnace in response to changes in the rate of decomposition of the sample to improve weight change resolution. The resolution setting is a unitless number ranging from 0 (lowest resolution) to 8.0 (highest resolution). Increased resolution tends to increase the experiment time. The **High Resolution Sensitivity** segment sets an additional parameter that can be used to adjust the response of the High Resolution temperature control algorithm. This is sometimes necessary due to the wide variation in decomposition mechanisms of typical sample materials. The sensitivity setting is a unitless number ranging from 1.0 (lowest sensitivity) to 8.0 (highest sensitivity). Increasing sensitivity tends to increase experiment time. Example: High Resolution Ramp 50°C/min to 1000°C Res 5 Sensitivity 1
Heater PID	The **Heater PID** segment changes the performance of the instrument furnace during the execution of a thermal method. PID stands for Proportional, Integral, and Derivative, the three modes of traditional temperature control. The **Heater PID** segment specifies the control coefficients for each mode of temperature control. This segment is only maintained during the current method. At the end of the method, the Heater PID values are reset to the default values. Example: P= 35 I= 70 D=2
Increment	The **Increment** segment raises or lowers the temperature in a controlled step, lets the temperature equilibrate, then begins the next segment. Example: Increment by 5°C
Increment Gas	*Applicable to Blending GDM instruments only:* The **Increment Gas** Blend segment increases or decreases the specified gas blend percentage in a controlled step, then begins the next segment. A complementary change in the second gas being blended attends the execution of this segment. Example: Increment Gas 3 5%
Isothermal	The **Isothermal** segment holds the sample at the current temperature (as programmed by the previous segment) for a defined period of time. This segment automatically turns on data collection, except when preceded by a **Data OFF** segment. Example: Isothermal for 10 min
Jump	The **Jump** segment instantly changes the set point temperature, causing ballistic changes in the sample temperature. This segment then allows the immediate execution of the next segment (which is usually the **Isothermal** segment). Note that large temperature overshoots may result from the use of this segment. This segment does not automatically start data collection. Example: Jump to 200°C

Mark End	The **Mark End** segment places a marker in the data for use by the data analysis programs. In general, markers provide quick parsing of data to separate experimental segments (i.e., the heat-cool cycle). This segment is available but not necessary for TRIOS. Example: Mark end of cycle 0
Mass Flow	The **Mass Flow** segment alters the rate of flow of the selected gas when an instrument is equipped with a Gas Delivery Module (GDM). Example: Mass Flow 50 mL/min
Modulate Temperature	*Available for Modulated Instruments Only*: This segment allows you to enter the modulation temperature amplitude and period (frequency) parameters that will be used with subsequent ramp or isothermal segments. Data collection begins after two modulation cycles. Example: Modulate temperature amplitude 1°C period 60 seconds
Ramp	The **Ramp** segment heats or cools the sample at a fixed rate until it reaches the specified temperature, producing a linear plot of temperature versus time. This segment automatically turns on data collection, except when preceded by a **Data OFF** segment. Example: Ramp 10°C/min to 200°C
Ramp Gas	*Applicable to Blending GDM instruments only:* The **Ramp Gas** Blend segment is used to ramp the blend percentage of the sample purge gas stream, while the sample is held at a constant temperature. A complementary change in the second gas being blended attends the execution of this segment. Example: Ramp Gas 3 0.20%/min to 80.00%
Repeat	The **Repeat** segment does exactly what the name implies: it repeats a group of one or more segments within a method for the number of times specified. Example: 1. Ramp 5°C/min to 200°C 2. Ramp 5°C/min to 50°C 3. Repeat segment 1 for 2 times
Repeat Until	The **Repeat Until** segment repeats of group of one or more segments within a method until the specified final temperature is reached or passed. Example: 1. Equilibrate at 50°C 2. Isothermal for 5 min 3. Increment 10°C 4. Repeat segment 2 until 200°C

Sample Interval	The **Sample Interval** segment allows you to define or change the rate at which data is to be collected (in seconds per point). Example: Sample Interval 2 sec /pt
Select Gas	The **Select Gas** segment controls the switching of gas between Gas 1 and Gas 2 for an instrument with a GDM installed. This segment is used to synchronize gas switching at a specific time or temperature in an experiment. Example: Select Gas 1
Step	The **Step** segment causes the temperature to jump a specified number of degrees at a specified time interval until a final temperature is reached. This segment automatically turns on data collection, except when preceded by a **Data OFF** segment. Example: Step 5°C for 2 min to 200°C
Step Gas	*Applicable to Blending GDM instruments only:* The **Step Gas** Blend segment causes the relative gas to step a specified percentage, at a specified time interval, until the final blend is attained. A complementary change in the second gas being blended attends the execution of this segment. Example: Step Gas 3 10% for 10 min to 50.00%

REVISIONS

v1.21 2023/08/16	• Sections 'TGA-GC-MS', 'TGA-FT-IR', and 'TGA-FT-IR-GC-MS' were added
v1.12 2023/08/01	• Release of original version of the user manual for the acquisition software Trios 5.7.0.56