

Q50 TGA Manual

School of Materials Engineering ARMS B-214

User Requirements

Training

- All users must receive training from Jameson Root (jmroot@purdue.edu) or a qualified super-user before using the TGA

Mailing Lists

- All users of the School of Materials Engineering TA TGA must sign up for the equipment mailing list available at <https://engineering.purdue.edu/ECN/mailman/listinfo/mse-tga-list>

Reporting Technical Issues

- Users are prohibited from performing any system maintenance not described in this manual
- Contact an administrator for help troubleshooting any equipment issues and report all unresolved lab and equipment issues to the mailing list (Mse-tga-list@ecn.purdue.edu)
- Failure to report equipment damage will result in a **permanent ban** from the *lab facility*

System Capabilities

- Standard Atmosphere – N₂, Compressed Air, etc. Novel atmospheres must be approved before use.
- Max Temperature – 1000°C
- Max Heating Rate – 100°C/minute
- Typical Heating Rate – 20°C/minute
- Typical Specimen Weight – 5 to 20 mg (depending on density)
- Thermocouple Accuracy – ± 5°C

IMPORTANT NOTES

- DO NOT PUT ANY MATERIAL CONTAINING THE FOLLOWING ELEMENTS IN A PLATINUM PAN: PHOSPHOROUS, BORON, SILICON, OR ARSENIC – EVEN IF OXIDES! USE ALUMINA PANS INSTEAD.
- ONLY RUN PHOSPHOROUS, BORON, SILICON, OR ARSENIC IN AIR (no other gases allowed).
- We have an autoloader for a reason – NEVER EVER MANUALLY LOAD OR UNLOAD THE PAN ON THE HOOK!
- NEVER RUN A DEFORMED PAN IN THE TGA – it could damage the instrument and you will be charged..
- The quality of your data depends on the quality of your sample. Samples with high specific surface area will result in tighter degradation transitions, so powders & thin films (solid samples) or a thin film of liquid will be the optimum test samples.
- The Universal Analysis software can be downloaded on your own machine here: <http://www.tainstruments.com/main.aspx?id=292&n=2&siteid=11>
- If the gas is empty, email Jameson or a super user and we will show you how to get a new tank from the loading dock if you do not know how.

SOFT MATERIALS CHARACTERIZATION CORE FACILITY OPERATING PROCEDURES

TGA

- 1) Operational limits: This instrument has a range of Room Temp to 1000 C. It only measures mass change and cannot measure heat flow or differential temperature (ie IT IS NOT A DTA/SDSC). Only use a reasonable mass of materials – 10-20mg.
- 2) Temperature: Know about what temperature your material loses mass - Only go about ~100 C above the lowest plateau (If there is no reason to go to 1000C then don't). If you don't know, then do a reasonable guess and run the material a bit high in temperature to see then adjust subsequent runs. Organic polymers typically are all done below 500 C or so.
- 3) Materials: Know your material – never go close to the boiling temperature. If your material evaporates, then stay WAY below the temperature that it does so. Know what your material reacts with, specifically platinum (There are a variety of platinum eutectics). If you ruin a pan or thermocouple, you will be charged for a new one.
- 4) Evolved gases: Know what your material off-gases – if it degrades to small molecule organics or CO₂ (run in air) then it is fine. However, if OFF-GASSES ARE CONDENSIBLE AT ROOM TEMPERATURE OR REACTIVE THEN IT IS NOT ALLOWED. This means that materials such as zinc, mercury, etc cannot be run.
- 5) Provide your own pan: You will be expected to provide your own pan for samples, although we will train you with ours. See below for proper pan size/material, but we can provide with the vendor. They cost ~\$400, but are reusable. They must be the proper type for this instrument and undeformed or you will damage the hook and you will pay for that as well.
- 6) Proper Pan: Your material should not be anywhere near the top of the pan – do not overflow. If it is close, use a different pan that holds more. Know if your material shows a eutectic with Pt (P, B, Si, As), in which case use alumina pans and do it in air.
- 7) Proper heating rate: Generally, 20C/minute is ok (for loose powder with space between grains). However, some materials need extra time to heat or evolve gasses – foams, packed powders, and solid materials with residual solvent/liquid may need slower ramp rates. DO NOT EXPAND, FOAM, OR EXPLODE SAMPLES IN THE INSTRUMENT.
- 8) Cleaning your pan: Only use a blow torch, low pressure air and possibly acid wash to clean the Pt Pan. Never scrape, wire brush, abrade or mechanically clean it. Also, treat the pan gingerly as it is mechanically weak. DEFORMING THE PAN CAN RUIN IT AND DAMAGE THE INSTRUMENT!

Quick Procedure Checklist (experienced users)

- Enable the TGA through Coral
- Weight calibrate the TGA for your pan type (if applicable)
- Place an empty sample pan on the autoloader arm, wire lined up in the groove
- Hit "tare" (~2 min to complete & unload, can set up testing procedure in computer now)
- Take the empty sample pan off the autoloader and load in your sample
- Place the sample pan on the autoloader, wire lined up in the groove
- Hit "sample" (the machine will load the pan and close the furnace)
- Set up testing procedure through the software
- Open gas valves
- Hit green run icon
- Double check green running man has appeared
- After your run is complete, remove the sample pan (can do even if it's hot)
- If doing multiple runs, enable air cooling to cool down the furnace quicker, clean the sample pan and repeat the procedure
- If you are done, turn off the gas, raise the furnace, clean the sample pan and place it on the autoloader arm

Detailed Procedure (all users)

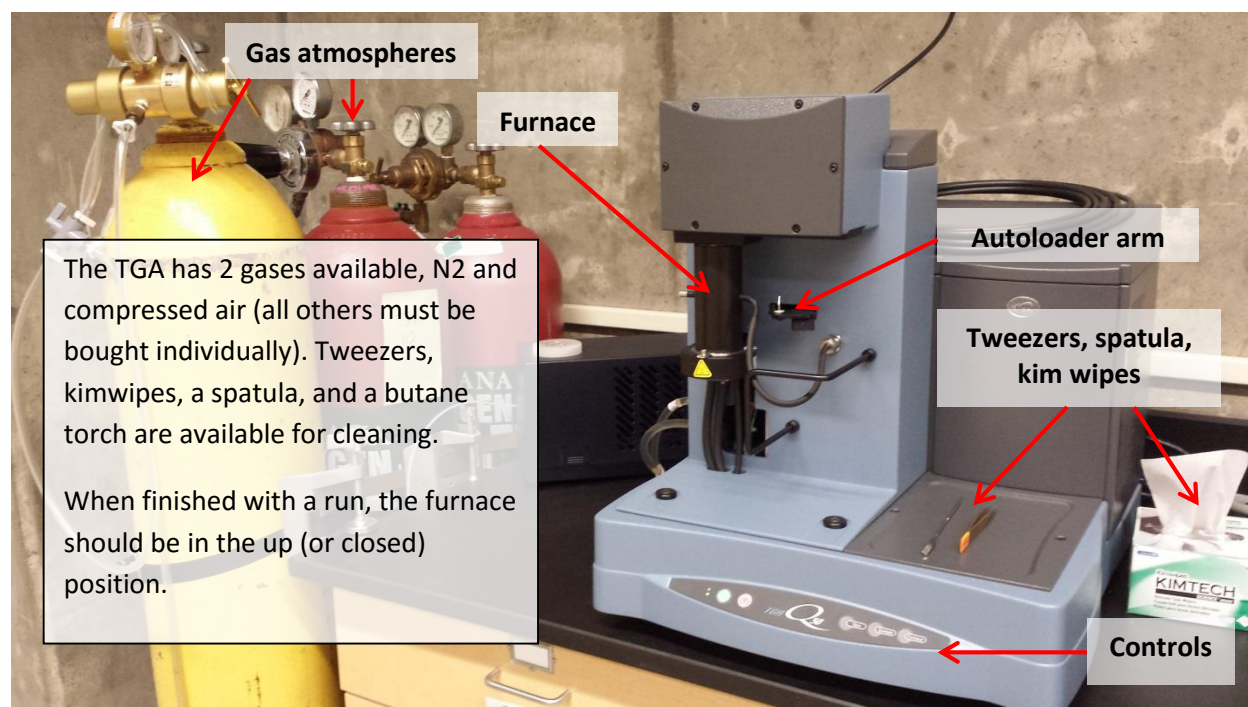


Fig. 1. How the TGA should look at the beginning and end of your use. Gas needs to be turned off, the furnace closed, and the tweezers and spatula present.

1. Enable the TGA through your Coral account (Coral -> MSE Electron Microscopy -> ARMS_B214 -> TGA). Billing policies are posted by the TGA.

2. Check to see that the TGA is in the same condition as in **Fig. 1**. Please let a superuser know if anything is out of place.

3. WARNING – the sample pan wires are extremely easy to deform. **DO NOT DEFORM THE PAN.** Only handle the sample pan with a pair of tweezers – never your fingers (**Fig. 2a**)! The pan should look similar to **Fig. 2b** (maybe a little less shiny).

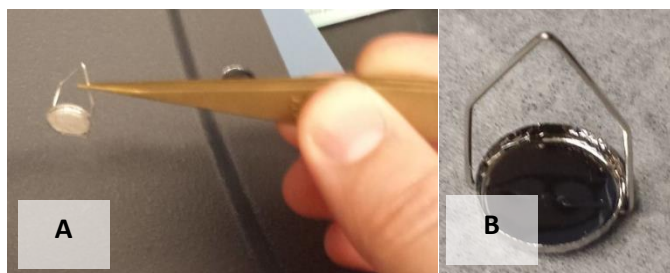


Fig. 2a, the proper way to handle the sample pan, **2b** a CLEAN, undeformed platinum sample pan. Notice that the hanging wire is very straight. Only handle the pan with tweezers to prevent damage!

4. If necessary, clean the sample pan using the butane torch, solvents, and/or prolonged periods in a hot furnace (1100C). . **BE VERY CAREFUL WITH THE PAN WHILE CLEANING – DO NOT DEFORM IT!** The butane torch is located in the locked cabinet under the TGA (lock combo is 8421). Ask a superuser if you need to use a furnace and have not used one before. The pan is made of platinum, but will accrue some residue over time

5. Weight calibration of your pan may be necessary if the previous user used a different pan type. If the pan type is different than yours, in the software go to calibrate -> weight. This will take you through the calibration wizard, follow those steps until complete. The calibration weights are located in the drawer under the TGA.

6. Now tare the empty sample pan by placing the clean, empty sample pan onto the autoloader with the wire lining up in the groove (**Fig. 3**). Hit the “Tare” button on the machine (**Fig. 4**).



Fig. 3, shows the groove in the autoloader arm. You need to line the sample pan wire up with the groove until it is in place. The autoloading function will not work if you do not do this.

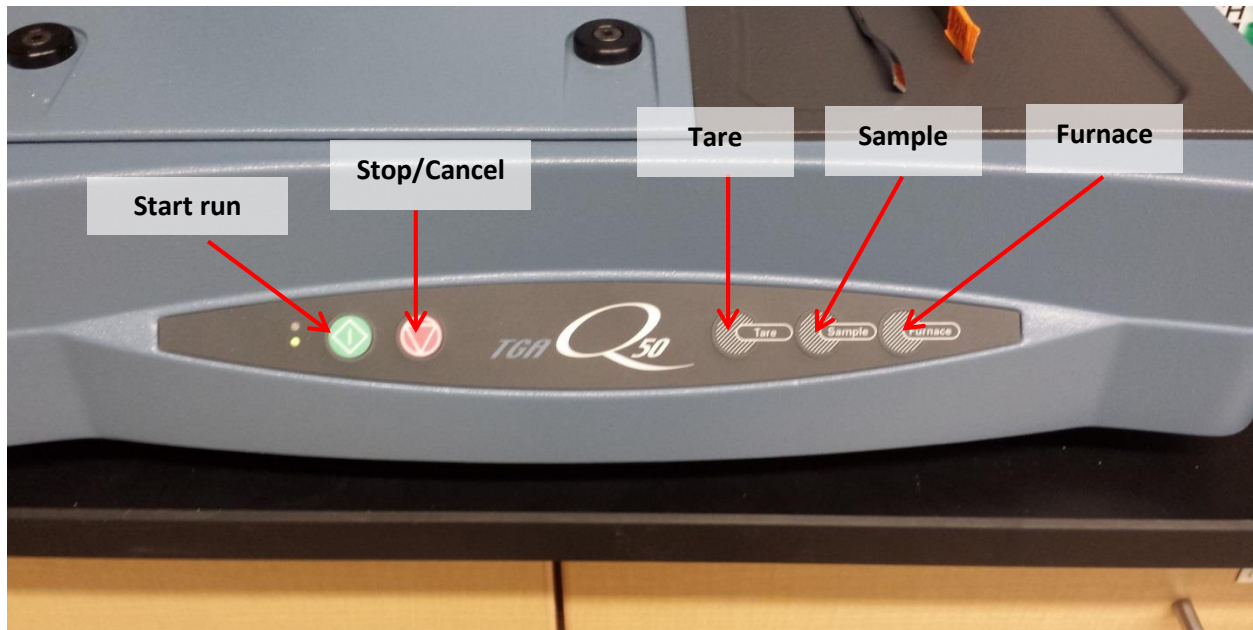


Fig. 4, shows the control buttons on the machine. Before hitting “tare” or “sample”, the pan should be on the autoloader. Furnace will raise or lower the furnace depending on its current position. “Stop” will stop whatever action the machine is doing.

7. After the machine is done taring, it will place the sample pan back on the autoloader. Use the tweezers to move the pan to the empty bench area in order to load your sample.

8. Load your sample. **Make sure that your sample is not overflowing or stuck to the bottom of the pan.** Your sample will increase in volume in the furnace – make sure that it will not overflow if that happens! Having as high of surface area to volume ratio is desirable in most samples in order for degradation to happen in the entire sample uniformly.

9. Place the loaded sample pan on the autoloader arm, with the wire lined up in the groove same as before.

10. Hit “sample” on the controls to load the pan. The furnace will close on its own after the sample pan is on the hook (**Fig. 5**).



Fig. 5, a close up of the furnace. The thermocouple should be very close to your sample but not in any danger of being touched by your sample when it undergoes thermal expansion.

11. Set up your testing procedure through the computer. Open the “TA Instruments Explorer” icon on the desktop, and double click on the left icon that is the TGA (Fig. 6). This will open the operating software.

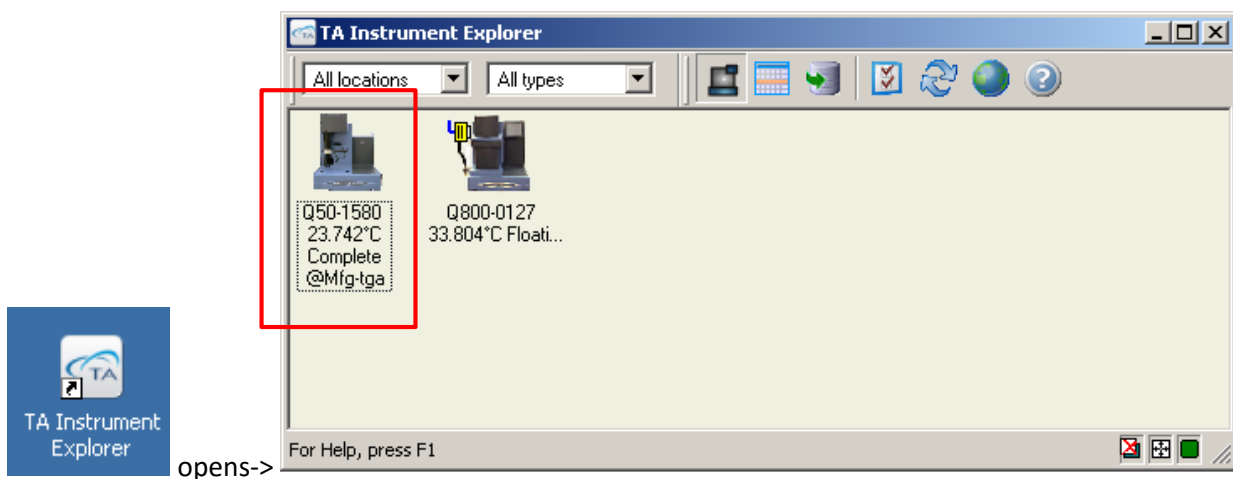


Fig. 6, The TA Instrument explorer icon is on the middle of the desktop. Double click the left icon in the explorer to open the TGA control software.

12. There are three main tabs in the middle section, Summary, Procedure, and Notes (**Fig. 7**). In the Summary tab enter your sample name and any comments. Specify where you would like to save by clicking the icon next to Data File Name.

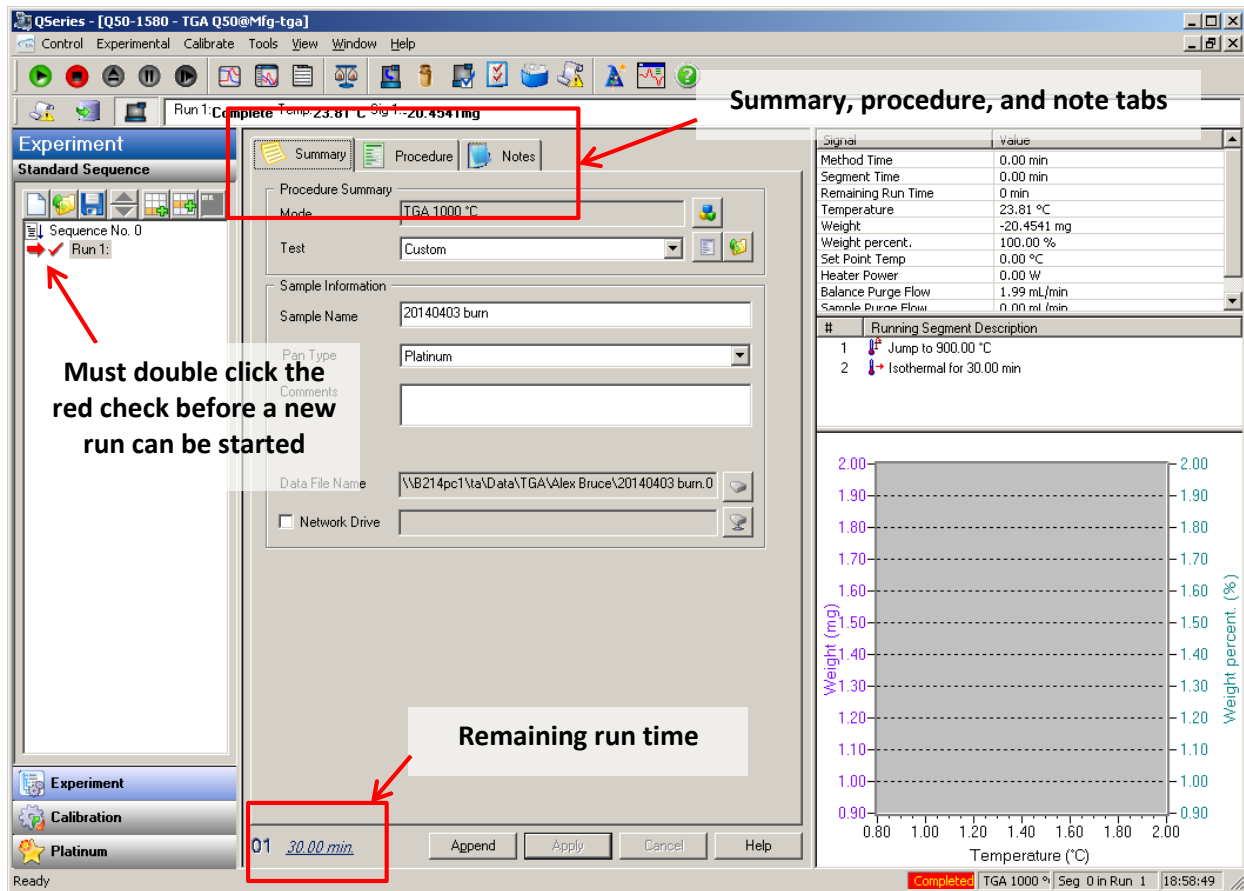


Fig. 7, the software screen as you open it.

13. In the Procedure tab, choose what test you would like, i.e. ramp, isothermal, custom, etc. In custom, you can create your own procedure by clicking the editor button, which will pull up the segment editor screen. Double click items in the Segment List to add them, and double click values in the Segment Description to edit them. You can drag and drop items into the correct series.

14. In the Notes tab, input your name as the operator. Do not change the gas flow values unless you are using a gas other than N2.

15 Make sure there is no red check mark next to the run (double click it if there is).

16. Open the gas valves (**Fig. 1**). It is important to close the gas when you're done or the gas will run out and be empty for the next user.

17. Hit “Apply” in the software (if necessary) and push the green icon in the top left to start the run. A new icon of blocks will appear where the red check mark was, and then a green running man will appear. The running man indicates that your test is running too... A warning will pop up if you forgot to open the gas valves or did something incorrectly.

18. Wait for your run to finish. The software will display your remaining run time at the bottom of the middle screen. You can leave the machine during this part.

19. If you will be running another sample when your first is completed, hit the “Sample” control button as soon as your run is done (even if it’s 900C). Once the pan is unloaded, enable air cooling through the software (Control -> air cool -> on) to cool the furnace down very quickly. Clean the sample pan & retare it as soon as the furnace has cooled down. Go through the procedure again.

20. If this is your last run, hit “sample” on the controls to unload the pan. Turn off the gas. Clean the sample pan and leave it sitting on the autoloader arm. Hit the “furnace” control button to close the furnace.

21. To get a useable data type (.txt), open the Universal Analysis software and open your raw files. Go to file -> export data -> file and plot signals. Choose your appropriate file type (usually spreadsheet, Unicode), and save the file in your folder or on a flash drive. The Universal Analysis program can be downloaded on your own machine for free here

<http://www.tainstruments.com/main.aspx?id=292&n=2&siteid=11>

21. Log out of Coral. You will be charged according to the hours it logs.

Common TGA issues

Error message saying “over/under weight”

The TGA needs to be weight calibrated for your pan type, start at #2 in the operation manual. If you don't think that's the problem, email a superuser.

Pan fell into the furnace

While this is not a big deal, please email a superuser to help you get the pan back out. It's very easy to damage the hang down wire or thermocouple while trying to retrieve the pan.

Hang down wire fell off/is in the furnace

Please email the superuser to replace the wire. It's tricky to do.

Taring or calibration is taking an extremely long time

Sometimes the thermocouple can be positioned too close to the pan which can either hit it or get stuck on it. This causes the machine to take a really long time to stabilize weight, and the data you get from a run like this will look crazy. Reposition the thermocouple to get it away from the pan.

The pan won't autoloading

This happens especially with old and bent pans. Let the superuser know, and they may be able to correct the shape of the pan to get the autoloading to work again.