

Discovery TGA 5500 SOPs

Users must read the Instrument Hazards and Best Practices before using this instrument.

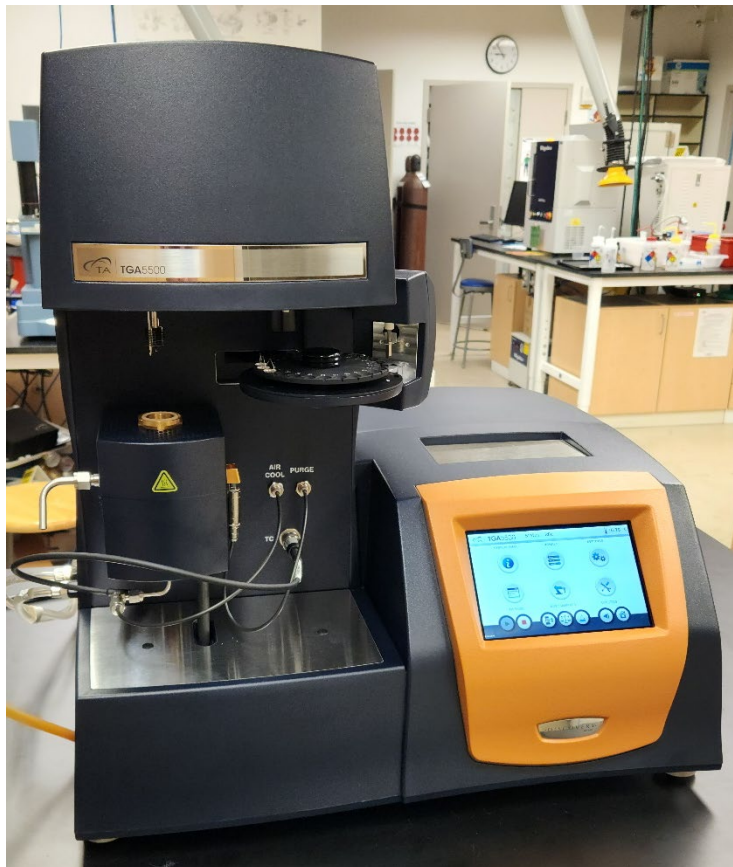


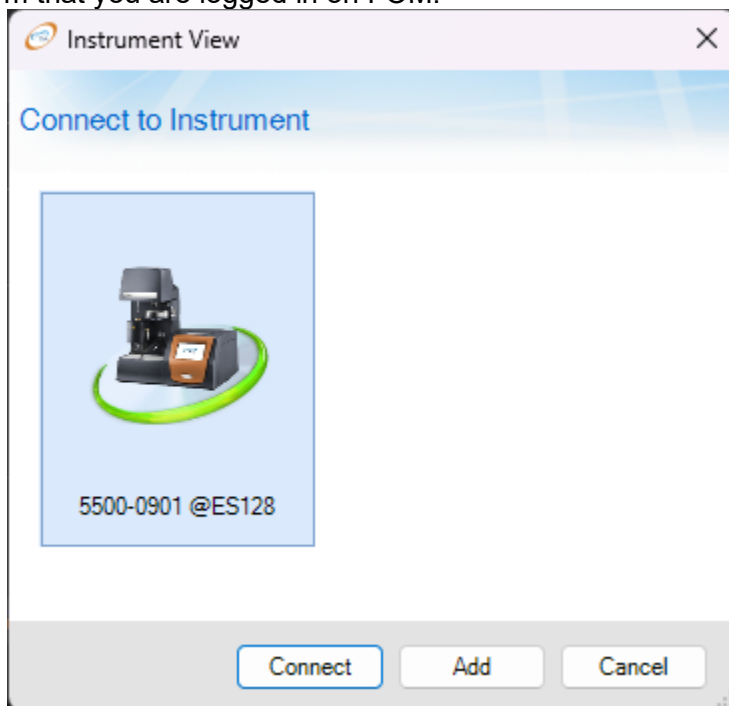
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1. Instrument connection

You must first login to FOM and login to the instrument in order to connect to the TGA. Ensure that you have reserved enough time for the experiments to complete. If not, you can reserve the time directly after your time slot if it is available. If you don't have enough time, and there is a user scheduled right after you, you will need to cut one or more runs to stay within your reserved time. The following user has the right to cancel your tests at the start of their timeslot.

Open **TRIOS** from the desktop once logged in to FOM. If the halo around the instrument is grey, you will need to restart **TRIOS** and confirm that you are logged in on FOM.



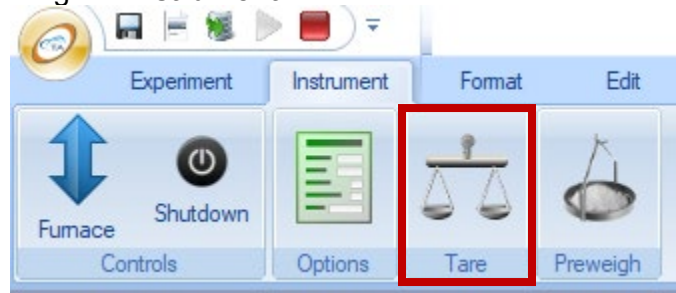
Double click the instrument or click Connect. The software will connect to the instrument and open.

2. Clean and tare pans

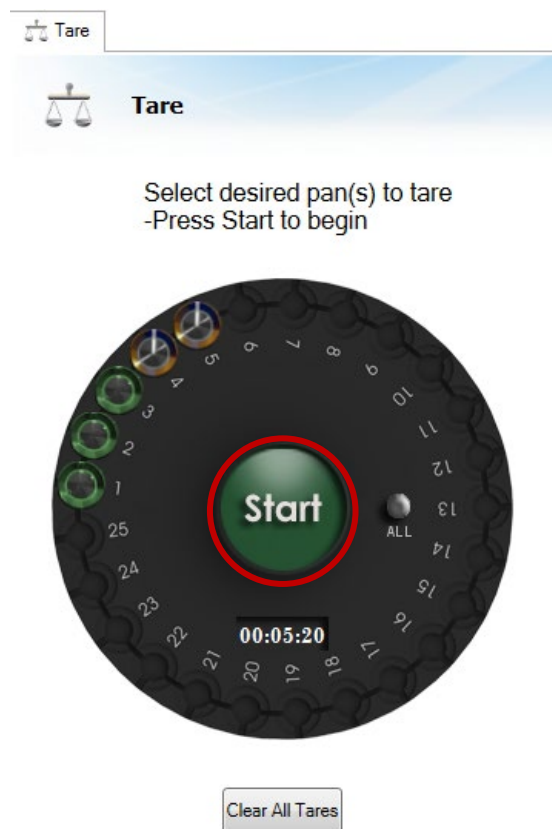
Clean pans with the normal procedure of torching. Please inspect all pans prior to use to ensure the instrument will load them correctly. No users shall manually load pans onto the hook. Failure to abide by this could result in damage to the instrument and loss of user privileges.

Clean as many pans as you need for samples to run. You cannot tare pans while the system is running. During taring pans must be empty and clean. Align the pans in the autosampler tray by aligning the wire with the slotted part on the tray.

Open the **Tare** window by clicking the **Instrument** tab from the ribbon and choose the **Tare** option.



The tare window will open. Select the pans you would like to tare. It is about 2 minutes 40 seconds per pan for taring and the system will give an estimate and a countdown timer once started. Click the **Start** button in the middle of the circle to begin.



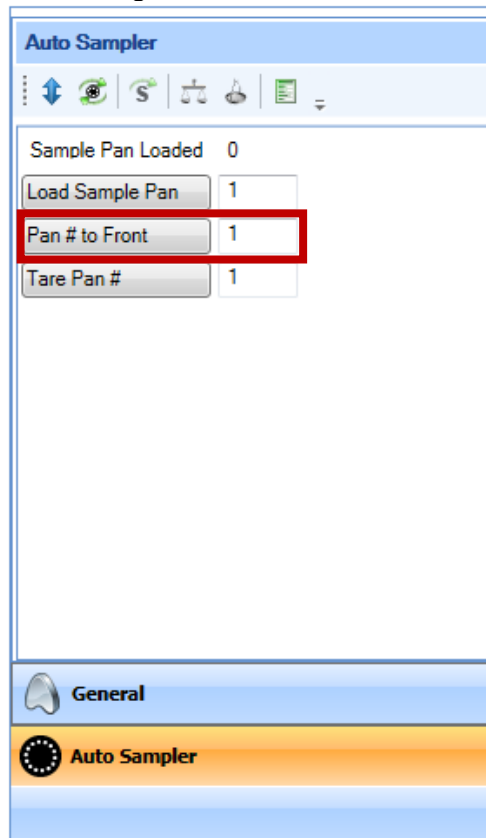
You can navigate around the software during the tare. You can return to the tare window via the same method used originally. There are instructions on loading pans in the section on [Loading pans](#).

3. Loading and choosing pans

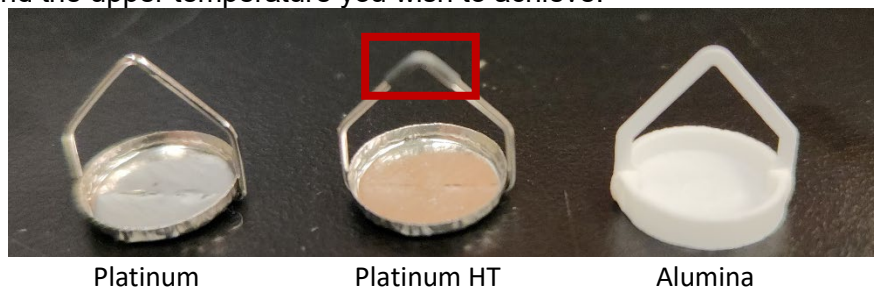
Platinum pans are ~\$400 each. Great care must be taken not to bend or break the pans. Users may lose privileges if multiple instances of pan destruction occur.

To load a pan in to the autosampler, the wire must be aligned with the slot in the autosampler tray. It will be useful to have the autosampler rotate so that the pan position needed is at the front for easy access. It is not recommended to reach over the top of other pans to load or unload them. You can knock samples out and potentially bend the pans.

To have a pan position rotate to the front, choose the **Auto Sampler** tab from the control panel and input the pan number desired and click **Pan # to Front**. The autosampler will rotate and the appropriate pan slot will be at the front of the instrument for ease of loading.



Choosing a pan is relatively easy. We don't have all the types of available pans on-site, but we currently have three different types: Platinum (100 μ L), Platinum HT, and Alumina (100 μ L). Which pan you choose depends on the sample type and the upper temperature you wish to achieve.



Platinum: Choose for most samples. Max temp 1000°C.

Platinum HT: Choose for samples up to 1200°C.

Alumina: Choose for samples that may alloy or react with platinum. All metals must use this pan or the alumina powder dusting technique. Failure to follow can ruin pans. Can be used up to 1200°C. Pan is porous and needs to be cleaned well between uses.

4. Sample prep

Pans must be cleaned and tared before loading samples. Check the section on [Cleaning and taring pans](#) for more info.

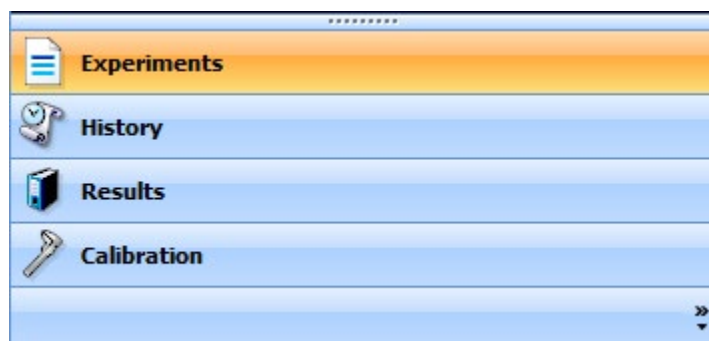
Recommended sample size is between 10-15 mg. You don't need to record the mass as the system itself is a very precise balance, but use the balance on the bench to get in the ballpark. Larger samples are not better as the sample will take longer to undergo each transition and the temperature of a transition displayed may not be accurate.

Ensure that once a pan is tared into a slot on the autosampler, that they are not mixed up. Each pan has a slightly different mass, so mixing them up would require them to be tared again so the system knows the base mass of your pan. If you mix them up, the data may seem valid, but is not.

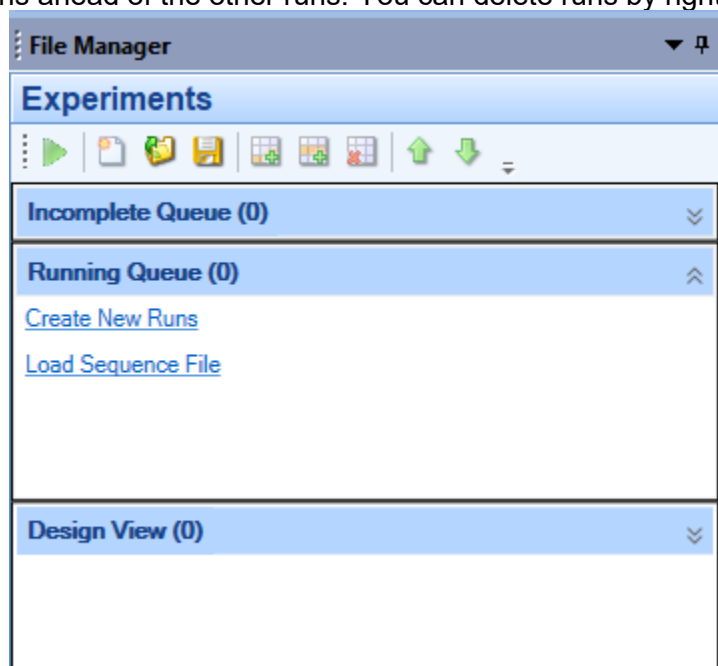
Place the samples in your pans after the pan has finished taring.

5. Setting up a run

In order to setup a run, you will need to navigate to the **Experiments** tab in the software. This is located in the bottom left of the software.



In the **File Manager** section, there shouldn't be any runs left. Runs that have completed successfully will disappear from the **Running Queue**. If there are runs when you arrive to your reserved time, you may remove them, or simply put your runs ahead of the other runs. You can delete runs by right-clicking on them.



Click on **Create New Runs** to add a run. You should see the following in the middle of the **Experiments** window. Enter the **Sample Name** and the corresponding **Pan Number** you wish to place the sample in. Choose the **Pan Type**. See the section on [Loading and choosing pans](#) for more info. You may optionally enter a **Project** and all the filenames generated will have the project name in them automatically as seen in the example.

The screenshot shows the 'Experiments' window with a 'Run 1 in Running Queue' section. The form contains the following fields:

- Sample Name: Test
- Pan Number: 1
- Pan Type: Platinum (100 uL) (dropdown menu is open showing options: Platinum (100 uL), Aluminum, Alumina (100 uL), Alumina (250 uL), Platinum (50 uL), Platinum HT, Sealed Aluminum, Other)
- Operator: AMSEC
- Project: SOP Writing
- Notes: (empty text area)

At the bottom, a red box highlights the 'File Name' field with the value 'C:\Data\Test_AMSEC_SOP Writing.tri'.

Expand the **File Name** caret to select the save location. In order to have your data backed up online automatically, you will need to choose a location within the C:\Data\ directory. Please keep this directory organized and cleaned up. Students must always save within their research advisor's folder. Click the button with the **ellipses** in order to choose a file save location.

The screenshot shows the configuration section for the file name:

- File Name: C:\Data\Test_AMSEC_SOP Writing.tri (with a caret icon)
- File Path: C:\Data
- Default button
- Ellipsis button (highlighted with a red box)
- Template: <SAMPLENAME>_<OPERATOR>_<PROJECT>
- Add Token button
- Reset run number when saved checkbox

Choose your test template or create a custom run from the dropdown. You can save and load procedures from the blue **Procedure** bar. Save these procedures in the same folder that you would save your data, or create a procedures folder within your research advisor's folder. Enter the appropriate run parameters into the fields. This will not be covered in the SOPs as there are many procedures used for many different reasons. You can ask the lab manager for assistance if you are not sure.

Procedure

Test: Custom

Name: Custom

Segments:

No.	Description
	Temperature Verification
	Heat and Hold
	Hi-Res Constant Reaction Rate
	Hi Resolution Dynamic
	Ramp
	Stepwise Isothermal
	Standard Modulation
	Modulated HiRes
	IR Bulb Check

Advanced

Beginning of Test:

Start Experiment After Weight Stabilization

End of Test:

Enable Air Cool

Air Cool Until Temperature Is Below: 30.00 °C

End of Test Delay: 0.00 minutes

Selected Calibrations:

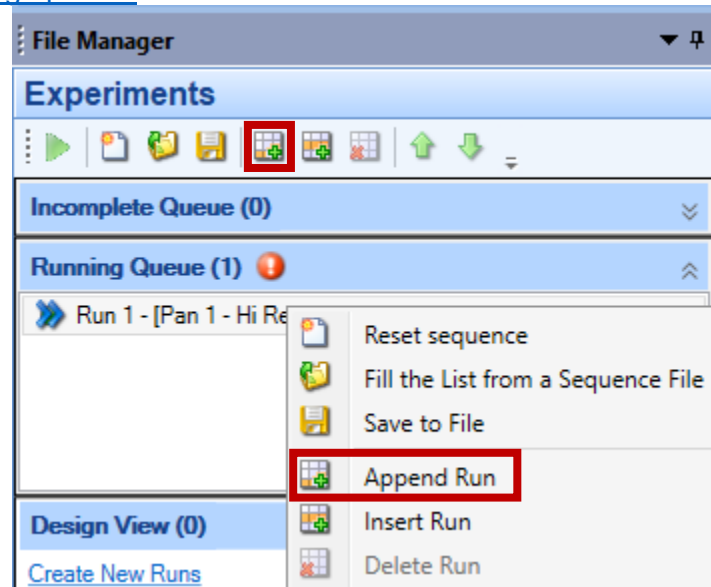
Use default calibrations

Under **Beginning of Test** decide if you want the system to wait for a stable weight reading. This is good to leave checked in most cases, however, if your sample has volatile components that may be evaporating while the sample is waiting, the mass won't stabilize and this should be unchecked. All the other options under **Advanced** should be left as is.

NOTE: On warmer days, it is a good idea to change the **Air Cool Until Temperature Is Below** to a value of 40°C so that it doesn't get stuck cooling. On warm days, the air that is cooling it is at or above 30°C so it won't ever reach the value and the system won't continue past the first run for a very long time.

6. Adding runs

To add runs, choose the append option from the **Running Queue** in the **Experiments** section. It is important to choose **Append Run** as opposed to insert run. Append will copy all the settings from the run that you selected when you chose it. This is helpful since users typically have multiple runs with the same parameters. Once the run is appended, you will need to fill in the sample info and double check all the run parameters. Check the section on [Setting up a run](#) for more info.



7. Starting and stopping runs

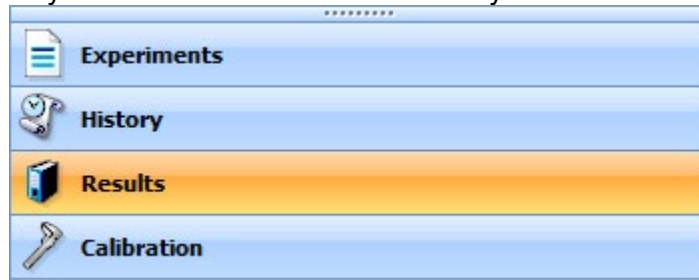
To start a run, choose the big green **Start** arrow from the **Experiment** tab on the main software ribbon at the top. This will start whatever run(s) that have been created in the **Running Queue** in the **Experiments** section of the software. The software will automatically take you to the **Results** section of the



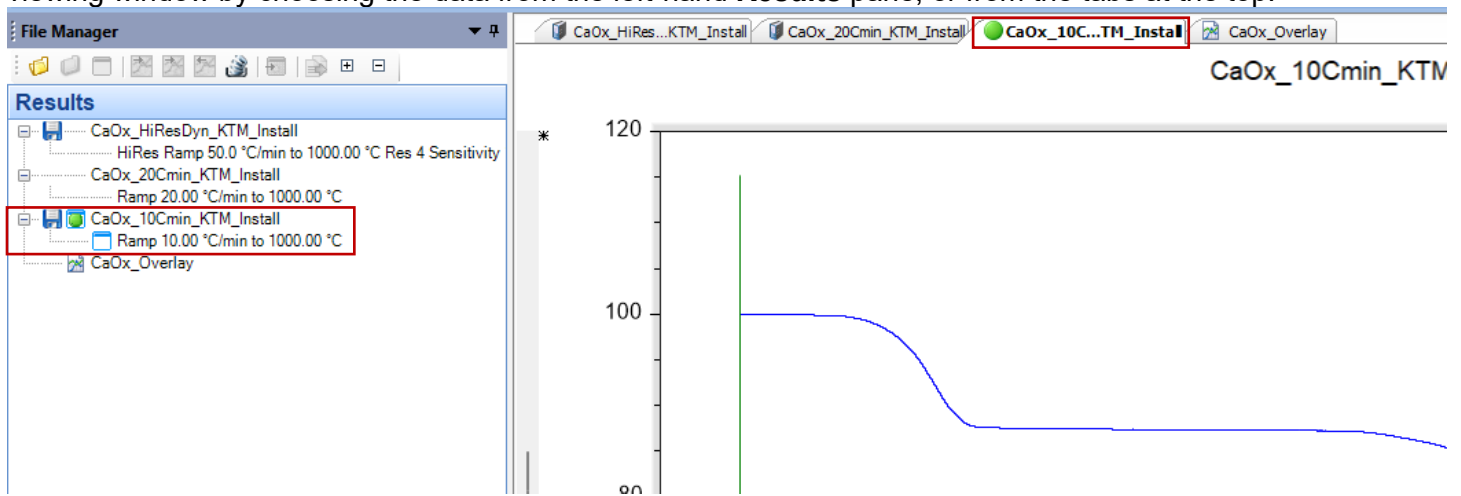
To stop a run, choose the big red **Stop** square from the **Experiment** tab on the main software ribbon at the top. You will then have to choose from the three options. Read them and choose accordingly. The system will then execute the selected option.

8. Viewing active runs and data

When a run is started, it will automatically change the view to the **Results** tab from the bottom left window selection. This is where you will view actively collecting data, successfully completed run data, and even open data collected on a previous day. It can all be done simultaneously.



The actively collecting data will be indicated by a green-filled circle in the **Results** pane as well as in the tabs above the plots. All data collected prior to that sample will show up higher up on the list and all data collected after the currently active sample will show up lower on the list. You can cycle which data is displayed on the viewing window by choosing the data from the left-hand **Results** pane, or from the tabs at the top.



In this SOP, we will not discuss all the possible ways to view and manipulate data in this software. This document is designed to help users operate the instrument. Data workup can be done with someone experienced on the system, or with the lab manager.

9. Instrument hazards and best practices: TGA

This document will cover the inherent hazards when utilizing this piece of equipment as well as the best practices and procedures to avoid danger. These hazards will not include basic things that may be included in the basic safety training document that each user has attested to have reviewed at fom.wvu.edu/documents

Lab coats are to be provided by the user unless special hazards exist in which case they are located at the PPE station.

Hazards:

- Chemical exposure
- Extreme Temperatures (Up to 2200 C)
- Hot Flames (propane torch)

1. Required PPE

Appropriate laboratory attire is required at all times in the AMSEC laboratories. Whenever chemicals are being used, an additional requirement of a lab coat is required. Lab coats are to be provided by the user.

Whenever a user is in the AMSEC labs, the minimum requirement for eye protection is wrap around impact glasses. Anytime liquid chemicals are present in the same room as the user without a direct barrier, all users in the lab must wear chemical splash goggles. Splash goggles must be approved by State of Washington Administrative Code (WAC 296-155-215).

If chemicals being used are considered toxic, caustic, corrosive, flammable solvents, carcinogenic, mutagenic, or teratogenic, a minimum of disposable nitrile gloves is required. Avoid chemical transfer by taking off gloves when using anything other than the chemical(s).

2. Extreme Temperatures

The TGA operates from room temperature up to 1200 C. When the furnace is in the operating position of up, it is very safe and not harmful to the touch. When the furnace is brought down after a run completes, it will be very hot. Avoid contact with the furnace during this period of cooling after a run. Avoid coming into contact with the cooling air stream exiting the top of the furnace as this may be hot as well. If no air cool executed after a run, it may remain hot for up to an hour. When an air cool is used after a run, it should be cool after 10 to 15 minutes. As a common practice, users should never touch the furnace. This is due to not only the temperature risks, but to keep the furnace from becoming contaminated.

3. Hot Flames

The procedure for cleaning the TGA pans includes the use of a propane torch to heat the samples pans up. The torch flame reaches temperatures over 2200 C and will rapidly heat or set aflame anything it comes in contact with.

Standard laboratory clothing is required. A minimum of impact glasses are required for this procedure unless liquid chemicals are present. It is recommended to wear flame resistant gloves and a flame resistant lab coat that are provided in the PPE station. Please put them back after use.

Torch operation is straightforward. Avoid using the torch anywhere except near the TGA as care has been taken to prevent anything flammable or combustible from being located in that vicinity. If you notice anything flammable or combustible near the TGA, please contact the lab manager immediately. To use the torch, turn the brass knob on the back counterclockwise until it stops. Then turn the red knob on the front to the on position. Then pull the red knob and hold it down. If the torch does not ignite at first click, try again until it does. Avoid pointing the torch in the direction of any persons or equipment or furnishings. Use the torch at least two feet from anything.

Standard practice should be to use the long tweezers provided near the instrument to hold the pans during torching. The tweezers should be over 8" and made from steel to prevent rapid heat transfer and temperature resistance. If these tweezers cannot be located, do not perform this procedure until they have been acquired. Contact the lab manager for assistance. To torch the pans, grab them with the long tweezers. Keep your hand being used to grip the tweezers as far from the flame source as possible. Grab the pan with the long tweezers by the corner of the wire. Avoid grabbing it by the top of the wire. Use the propane torch to heat the sample pan to red hot for at least 5 seconds or until no further change is observed. Let the pan cool down for 3 seconds before setting down. **Caution:** the tweezers will remain hot for several minutes after torching. Take care to avoid coming into contact with the tweezers or letting the tweezers encounter anything they may damage due to high temperatures.