

Operating Procedures: TA Instruments TGA

Purpose and Scope:

This document describes the procedures and policies for using the MSE TA Instruments TGA. The scope of this document is to establish user procedures. Instrument maintenance and repair are outside the scope of this document.

Responsibilities:

This document is maintained by the department Lab manager. The Lab Manager is responsible for general maintenance and for arranging repair when necessary. If you feel that the instrument is in need of repair or is not operating correctly please notify the Lab Manager immediately. The Lab Manager will operate the instruments according to the procedures set down in this document and will provide instruction and training to users within the department. Users are responsible for using the instrument described according to these procedures. These procedures assume that the user has had at least one training session.

Background:

TGA stands for 'Thermogravimetric Analysis'. Using a very accurate, sensitive balance, the instrument measures the change in weight as a function of temperature. From TGA we can determine a number of dynamic properties.

- Thermal stability of materials
- Oxidative stability of materials
- Composition of multi-component systems (no elemental data)
- Estimated lifetime of a product
- Decomposition kinetics of materials
- The effect of reactive or corrosive atmospheres on materials
- Moisture and volatiles content of materials.

-Customer training course TA Instruments

These properties can be determined from either weight gain or weight loss. It is also important to remember that this is a kinetic measurement so rates and masses should be carefully chosen and they should be included in any reporting of data.

Features and Specifications of the UW_MSE TA Instruments TGA Q50:

- Low mass furnace
- EGA Furnace
- RT – 1000 C
- TGA-FTIR
- N₂ and/or dry air purge
- Mass accuracy: +/- 0.5%
- Mass precision: +/- 0.01%
- Sensitivity: 0.1 ug
- Dynamic Drift : <50 ug from 50 – 1000 C at 20C/min with empty Pt pan
- Temperature precision: +/- 0.1 C

The instrument is calibrated for weight and temperature whenever work is done on it. If you feel that a calibration is necessary please let the Lab Manager know. Do not attempt a calibration yourself.

It is important to determine approximate mass and the rate most appropriate for you material. Powders are ideal because you can maximize surface area which can improve resolution. For most applications 10 – 20 mg is sufficient mass. 10 C/min – 20 C/min are typical rates. Keep in mind that a scan of 5 mg of material at 20 C/min. will not be the same as 15 mg of material at 10 C/min. and would not be comparable. (see figures)

Below are a few application examples. These are taken from the TA Instruments training seminar and there is not documentation as sample prep, sample origin, etc. All the information available is presented.

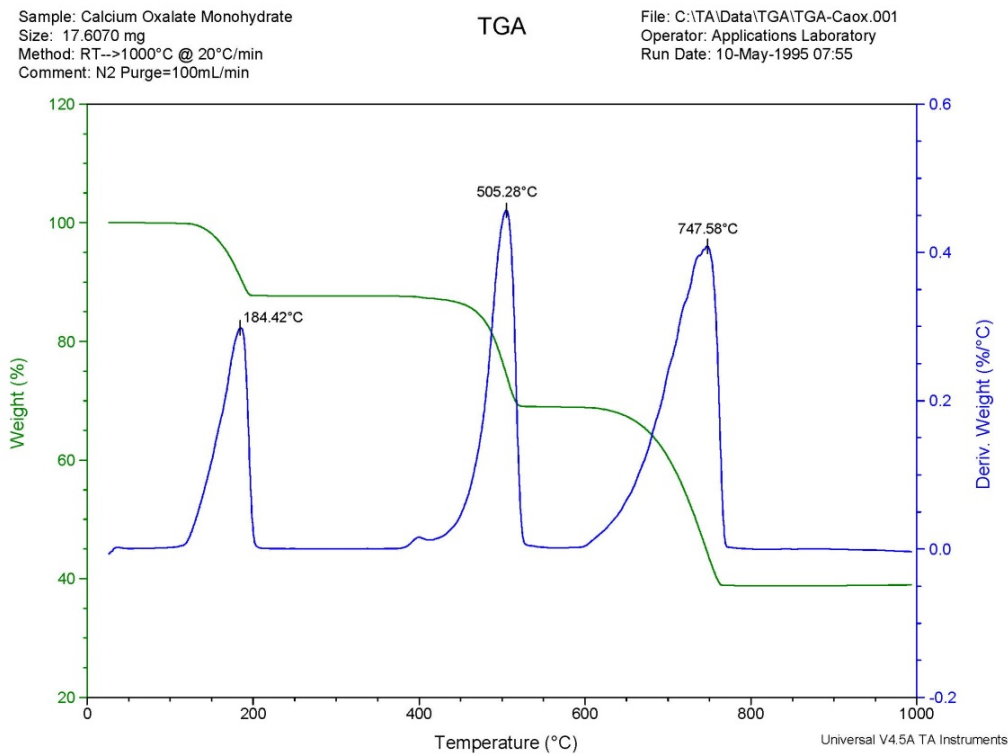


Fig. 1 Sample TGA result for CaOx (not UW-MSE Instrument)

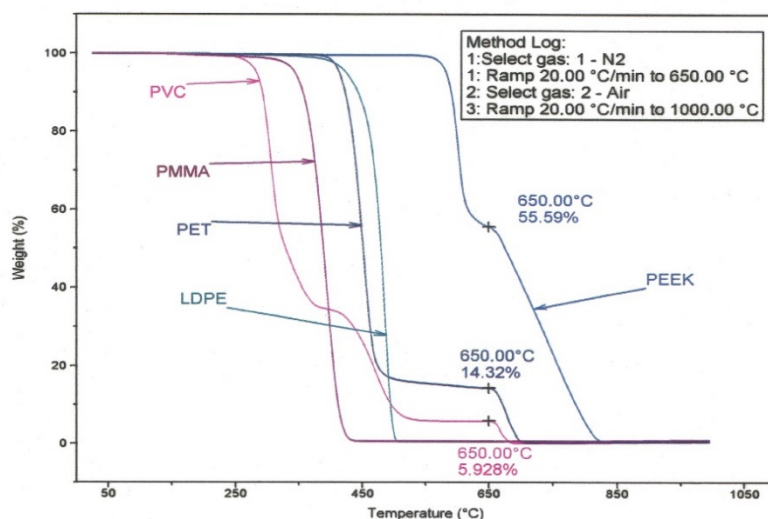
Calcium Oxalate Decomposition

- 1st Step $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O} (\text{s}) \longrightarrow \text{CaC}_2\text{O}_4 (\text{s}) + \text{H}_2\text{O} (\text{g})$
Calcium Oxalate Monohydrate *Calcium Oxalate*
- 2nd Step $\text{CaC}_2\text{O}_4 (\text{s}) \longrightarrow \text{CaCO}_3 (\text{s}) + \text{CO} (\text{g})$
Calcium Oxalate *Calcium Carbonate*
- 3rd Step $\text{CaCO}_3 (\text{s}) \longrightarrow \text{CaO} (\text{s}) + \text{CO}_2 (\text{g})$
Calcium Carbonate *Calcium Oxide*

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Fig. 2 Decomposition products for CaOx: TA Instruments training 02/2014

Thermal Stability of Polymers

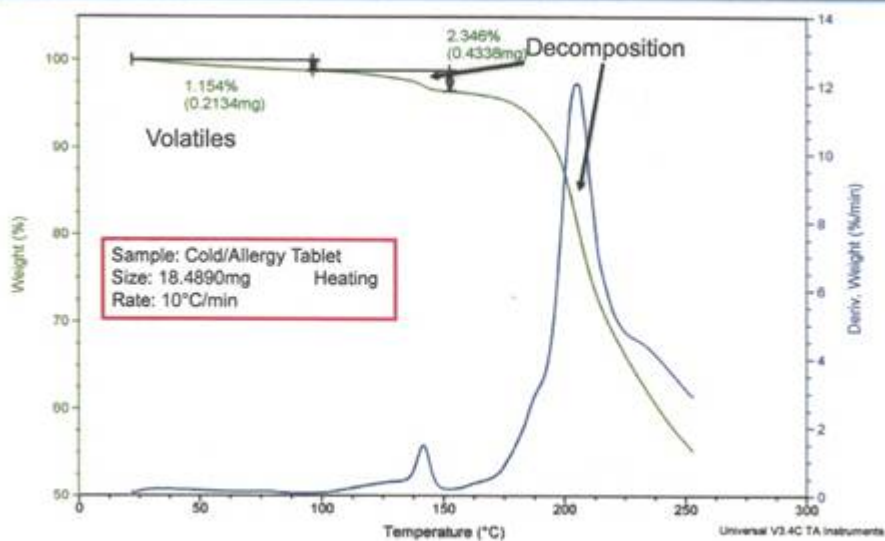


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Fig 3. Example 1

TGA Analysis of Cold/Allergy Tablet



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Fig 4. Example 2

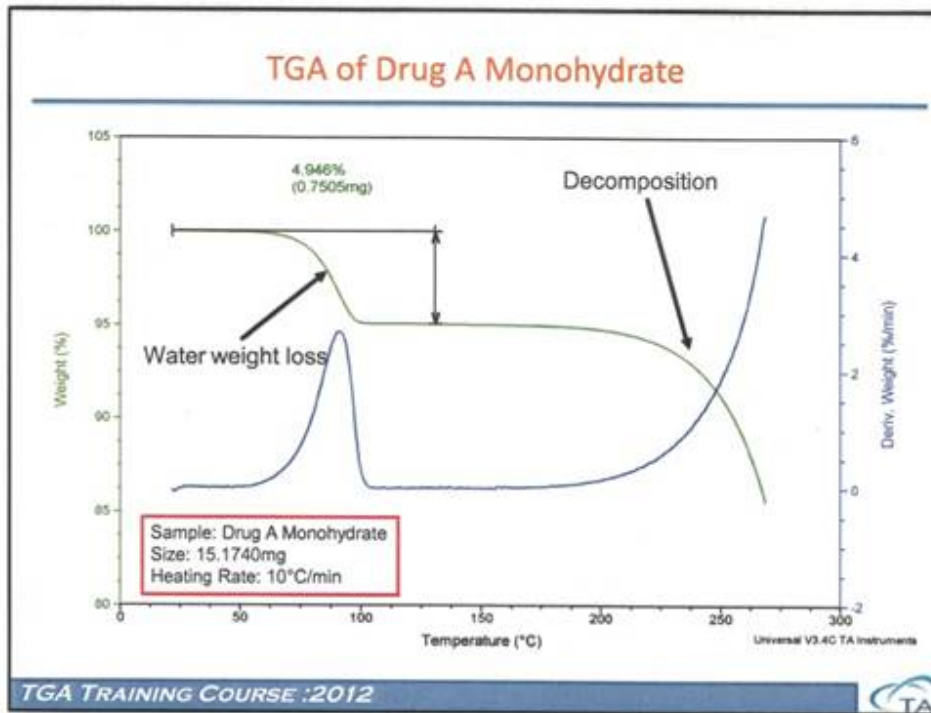


Fig 5. Example 3

Procedures and Sample Prep Considerations:

There are a number of parameters that must be determined before beginning, how much sample, heating rate, purge gas, etc. TGA is a kinetic measurement and each of these parameters will have an effect on the results. As shown below the same material at the same mass can have a decomposition temperature that varies significantly. It is important to determine this rate before doing TGA for the purpose of determining maximum temperature in the DSC.

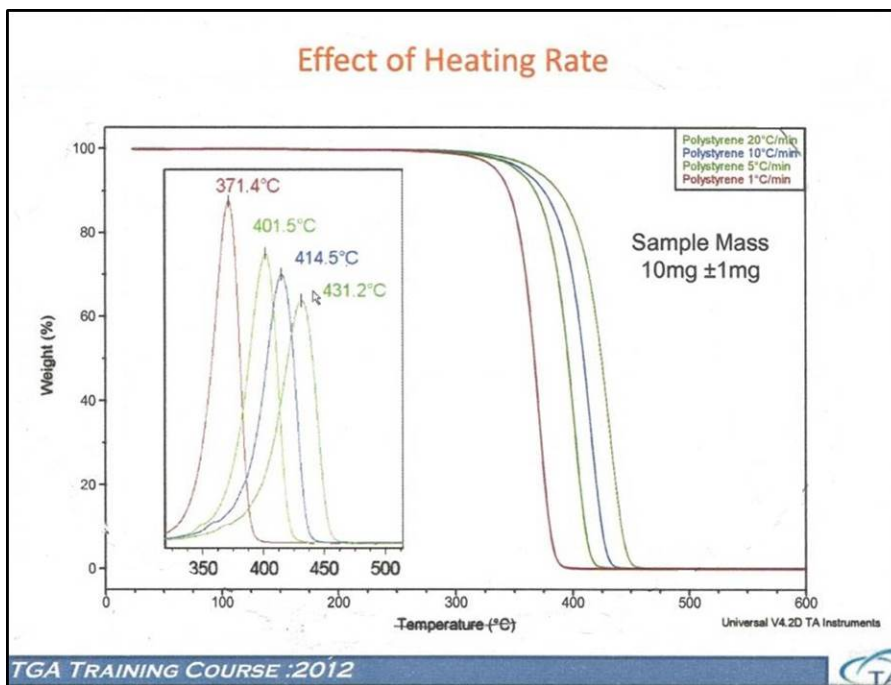


Fig. 6 Effect of heating rate on decomposition temperature results.

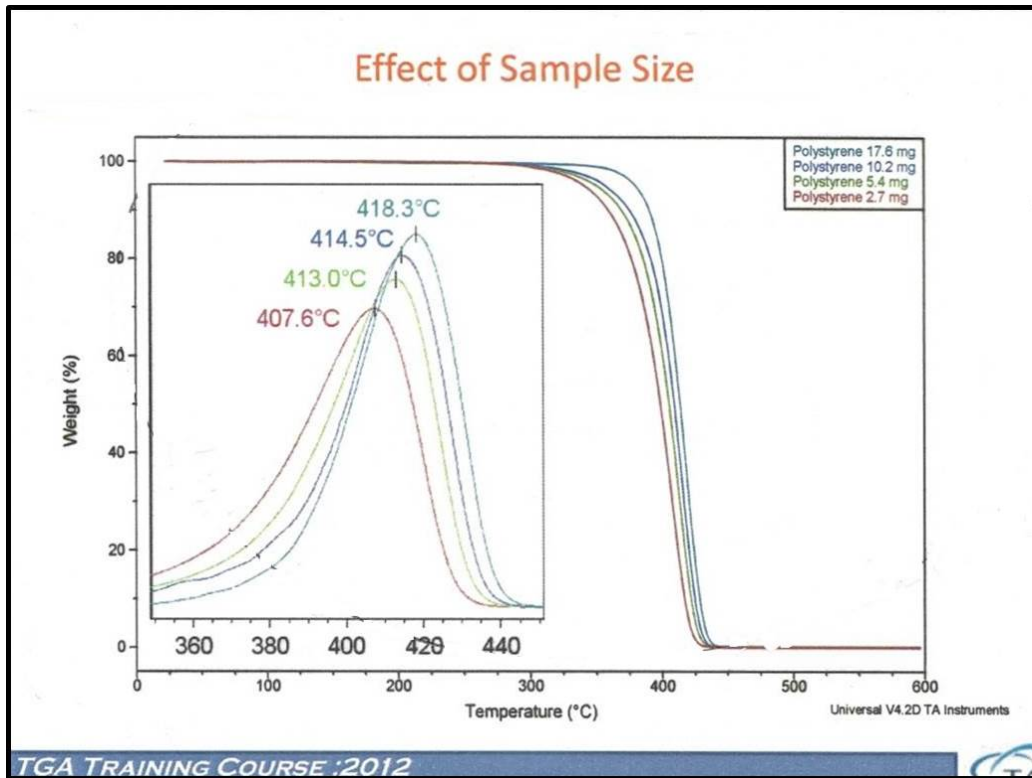


Fig. 7 Effect of sample size on decomposition temperature results

For making comparison measurements be sure to keep the mass of each sample within 10% of each other.

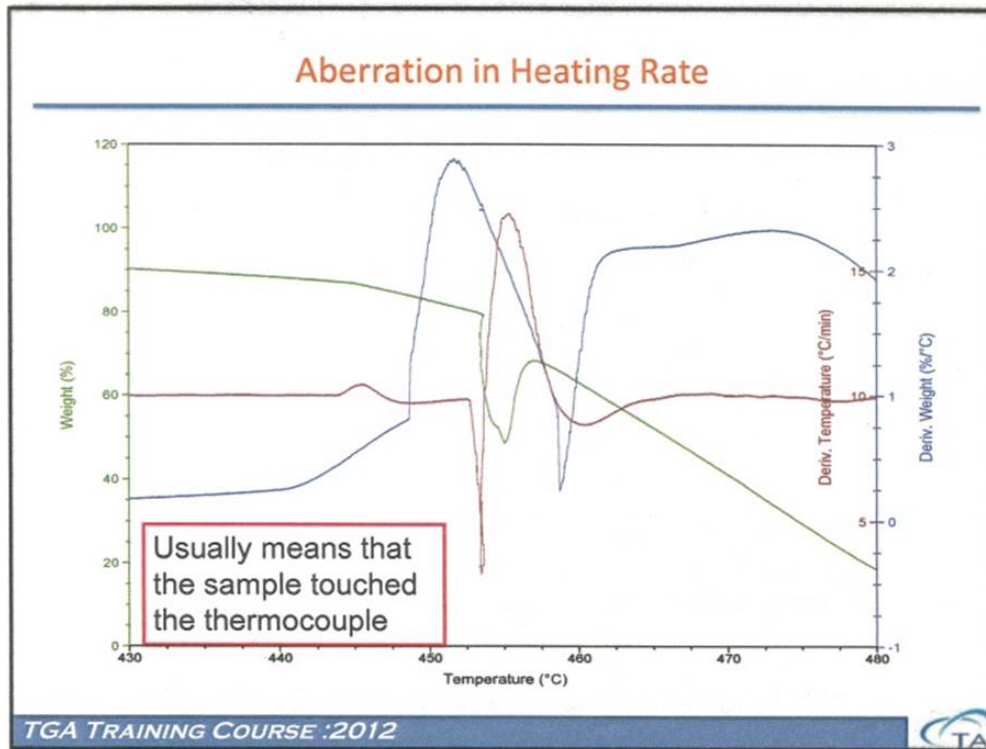


Fig. 8 Anomalous results

It is also useful to know how to interpret unexpected or anomalous results. The most common problems occur if there is too much sample and the thermal couple touches the sample or the crucible. Also, if the sample falls off or interacts/erupts during the measurement, this can cause odd results as well. If your results are unexpected or appear to be erratic please see the Lab Manager before continuing to reduce or prevent instrument damage.

Currently the TA Instruments EGA furnace is installed. The cross section shows how the sample and thermocouple are placed as well as indicates the gas flow. It is very important that the recommended gas flow rates be followed.

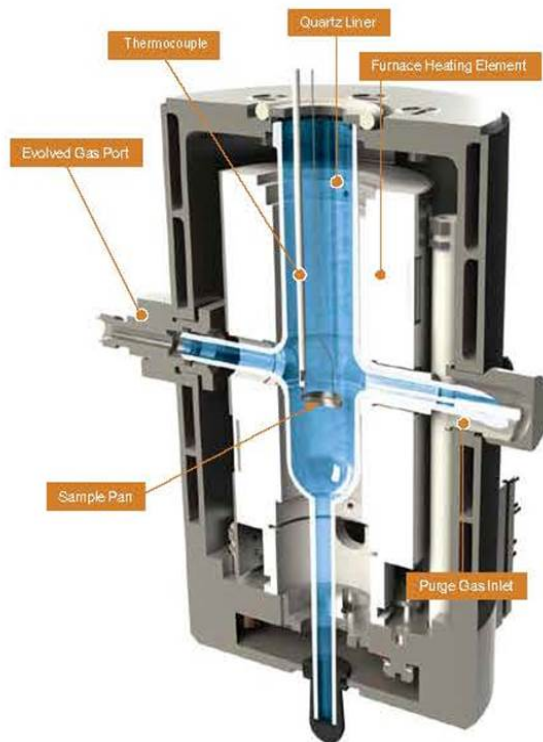


Fig 9. TA Instruments Cross Section with EGA

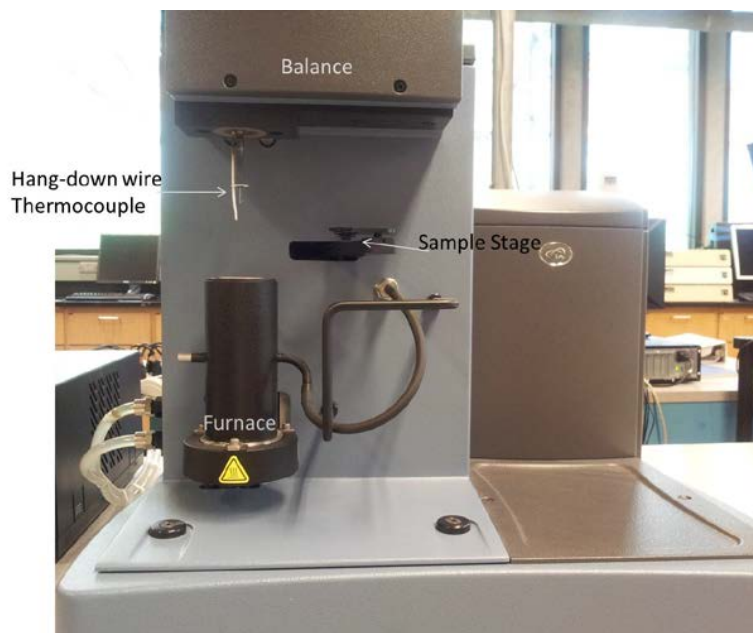


Fig 10.: MSE TGA

Warnings and precautions:

- Please do not eat or drink while operating this instrument.
- **NEVER** touch the hang-down wire or thermocouple.
- Never attempt to operate the instrument without nitrogen flow.
- Do not change any of the user preferences or instrument preferences.
- Notify the Lab Manger of any issues; do not attempt to 'fix' a problem.
- Once a measurement is running it is very important not to bump or disturb the instrument or the table it's sitting on, it is very sensitive to vibrations and can cause erroneous or anomalous behavior.
- The analysis software for the DSC and TGA are available on the TA Instruments web page at:

[Installation of Universal Analysis from the Advantage Software package](#)

- You will only need to download the "Universal Analysis" portion. There are also some training power-point videos available to further instruction on analysis. The computer that operates the instruments is not available for long term analysis.
- You are responsible for providing and caring for your own consumables for this instrument. Platinum crucibles are available from TA Instruments or from MSE.
- Before starting please verify that there is plenty of N₂ in the cylinder and that the flow rate is at 10/90 mL/min for the EGA furnace. (40/60 mL/min. for the standard furnace, purge and balance respectively)

Procedures:

- Place an empty crucible in the groove on the sample platform and tare. The instrument will load and unload the crucible.
- If the crucible fails to load, it may be out of alignment. The instrument will attempt to load it a second time. If that still doesn't work please contact the Lab Manager.
- Please leave the instrument undisturbed during this process and take care not to bump the table.
- After the tare is complete the pan will be returned to the sample platform.
- Remove the pan from the platform and fill with your sample. Please make sure that the sample fits comfortably in the pan. Do not allow sample to fall or flow out of the pan into the furnace.
- **IMPORTANT:** Some plastics expand as they heat up. If this happens and the material is close enough to the thermocouple it will "glue" the thermocouple to the pan. This will damage the thermocouple which is very expensive.
- At this time, if your program is already open and parameters have been defined simply press the run button and the instrument will load and weigh the sample and commence the measurement.

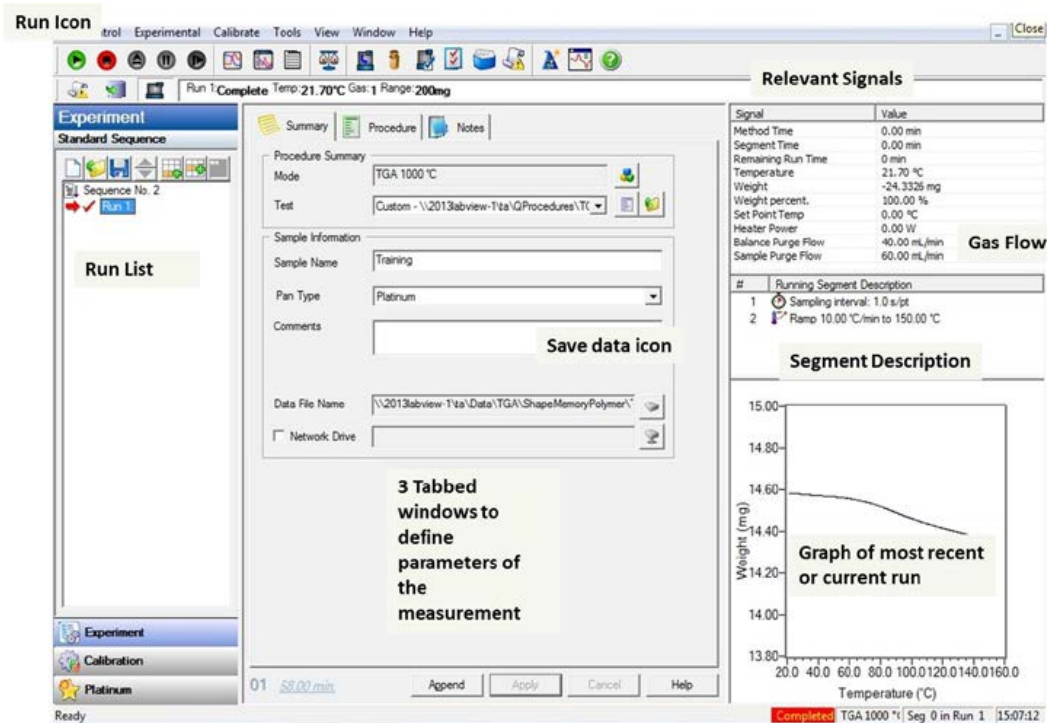


Fig.11 Screen shot of software

Software instructions:

- If the software isn't already open, open instrument explorer and select the Q50 TGA. The last user's information will be displayed.

If you already have completed procedures file:

- In the summary tab select <TGA 1000C > <Custom Test> and you can open it here are well by clicking on the file icon.
- Type in the sample name and select the pan type. In this case Platinum
- Select the icon to the right of "Data file name" and name the unique file.
- In 'Notes' enter your name.
 - Note: If you are running more than one sample you can name the first one and TA will add .00X to each subsequent run. Or you can provide a unique file name for each one.
- If necessary, select <Apply>

If you have not created a procedures file,

- select the second tab, <Procedure>
- Under method, select <Editor> to the right of the screen will be a list of steps you can select. The most common is simply the rate and temperature. Standard would be 10 C/min to a temperature not to exceed 1000C.
- Under end conditions please select air cool for no more than 30 minutes.
- Check the <Notes> tab and enter your name in the appropriate field. Make sure that the flow rate is appropriate for the furnace in use. If it is, do not change it, if it is not, please contact the lab manager before proceeding.
- Select <Apply> before beginning the run
- Select the run icon.
- Please remember to remove your crucible when your measurement has finished. MSE Management is not responsible for keeping track of your crucible and if it is lost, will not replace it.

When all of your measurements have been completed you can copy your data to an usb drive from the Workgroup share computer. No other action is necessary. Leave the computer on.

Date: _____

Email address _____