Operating Procedures for the TGA

Purpose and Scope:
This document describes the procedures and policies for using the MSE-TGA (Thermogravimetric analysis) The scope of the 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 or Scientific Instructional Technician (SIT). The SIT 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 SIT immediately. The SIT 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.

Definitions:
N/A

Prerequisites:
All users must read this document and obtain approval and training from the SIT.

Precautions:

1) Check that your sample is compatible with the platinum crucible at temperatures, atmospheres and times you plan to use. Metals will most likely react with the Pt if you get close to their melting temperature. Glassy materials (SiO2 containing) will stick and possibly react at temperatures greater than \( \sim 0.8T_g \). For example many polymers use SiO2 as a filler- if you go to a high temperature, the fillers may react with the crucible.

2) Maximum operating temperature is 1200 C, max heat up rate 20 C,

3) Nonreactive balance gas is required ( N2, Ar or He recommended). See #2 of procedure
4) Do not let anything drop in to the furnace tube. If anything does notify the SIT immediately and do not start a run. Place a note on the TGA as well.

**Procedure:**

**Part 1:**
The TGA is part of the MSE cost center. The log MUST be filled out. It is recommended that this be done before beginning a measurement so as not to forget.

**Part 2:**
Please remember to check that the nitrogen gas is on and that it is connected to the instrument in the “balance” port. If the tank regulator reads below 400psi, please contact the SIT immediately. Do not attempt to change the tank unless approved to do so.

**Part 3:**
Open up “Pyris” manager. Select the TGA button at the top of the screen. Click only once. The software is slow and if you double click it will attempt to open twice. When the software is open there will be two dialogs within the application (see figure) one is the **instrument viewer** and the other is the **method editor**. At the right is a column of icons that are used to send direct commands to the instrument. There are diagrams at the end of this document that show the commands. Terms in bold can be found in the diagrams. Select the `<cool furnace>` icon if the furnace is not already in that position.

**Part 4:**
Using the tweezers supplied with the TGA, load a clean empty crucible with the wire basket (stirrup) onto the **hang-down wire**. This must be done VERY carefully. Do not attempt until after a few practice runs with the SIT. The hang-down wire is connected to the **microbalance** at the top of the instrument and is very expensive to fix or replace. Do not pull on the hang-down wire as it may be broken off from the arm on the balance mechanism.

After the crucible has been installed, select the `<raise furnace>` icon. The furnace will move into position and raise up over the crucible. When the furnace is in position select the `<zero balance>` icon. You will need to wait a few minutes to allow the crucible to stabilize and you may zero the balance repeatedly until it stabilizes.

When the balance has been satisfactorily zeroed, select the `<cool furnace>` icon to move the furnace out of the way. Put the **sample stage** in position under the crucible and hang down wire and carefully remove the crucible by lifting the wire.
Part 5:
In Method Editor, input information about your sample. Select browse to name and save your data file. Select the program tab and program as follows:
- Starting temperature
- First temperature scan
- First isothermal
- Second temperature scan
- Repeat as necessary.

Maximum operating temperature is 1200 °C, max heat up rate 20 °C. The furnace does tend to overheat so this should be taken into consideration. A slower heating rate may be better.

When you are satisfied with your method, if you have not done so already, save your method. Please be careful not to overwrite someone else's method. At this time weigh your sample using the <weigh sample> icon. Select the start icon.

Part 6:
When your measurement is complete you may put the furnace in the cool position and remove your crucible. If you need to run another sample the furnace will cool down faster in the raised position. Please keep your crucibles with you and do not leave crucibles or samples on or around the TGA workspace.

**Implementation and Training:**

This SOP will be available to all users and must be adhered to. The SIT will train users in the implementation of this document.

**References:**
N/A
Fig. 2

- Micro Balance
- Hang down wire
- Thermocouple
- Sample Stage
- Furnace