

# TGA SOP

Purge gas A OR B: Purge with Nitrogen (and Oxygen gas if needed) around the cooling mechanism of the sample to avoid condensation

System Purge: Purge with Nitrogen the balance to eliminate any fume coming out of the sample to become residue on the balance

Avoid vibration, laying on the bench, putting any weight or hitting the bench while the test is running as the machine & scale are too sensitive to movement.

## Start-Up

- Turn ON the Nitrogen gas tank. Delivery pressure should be at 2 bar
- Turn ON the TGA machine by pressing the switch in the back
- Turn ON software PYRIS. Click on the TGA 4000 tab on top left side of screen to open the software
- Turn ON the chiller by pressing the switch on the back of chiller and the ON button on the front panel. Chiller should be at 15.0 C before we start the testing  
N.B: Distilled water in chiller should be changed every 4 months. Quantity is approximately 4 liters or enough to cover the coils

## Creating a new Method

- Click on File – New Method
- Under Sample Info tab
  - Name the method in the “File Name” window. Press “Save”
  - Add sample ID if needed
- Under Initial state tab
  - Leave as is. No need to change anything
- Under program tab
  - Set initial temperature
  - Click Add Step. Choose Temperature Scan OR Exothermic
    - ✓ Temperature Scan to add temperature ramp & rate
    - ✓ Isothermal to hold step at certain temperature and time
  - In the “To:” window add the next temperature to be reached
  - In the “Rate:” window add the rate in C/min at which you want the next temperature to be reached
  - In the “Data Sampling Options” window, select number of Points” and choose the number of points to be shown on the curve that ranges between 2 and 500 points

- Click “End Condition”, leave the default setting “End to Load”. Change value if you need to hold temperature at the end at other than the default temp of 30 C.
- Go to File – Save Method as – Click the folder named “Methods” – Choose the proper name – Click Save

## Testing Procedure

- Place the empty sample pan inside the machine, close the lid and wait for weight reading in the weight tab (top menu bar) to stabilize
- Click the Tare icon by clicking on the Even balance icon in the right panel (right icon) to zero out the sample pan
- Remove sample pan and place a small amount of sample (2-10 mg) and return it carefully with the tweezers inside the machine. Close the lid and wait for the weight reading to stabilize
- Click on the Uneven balance icon in the right panel (left icon) to record the current weight as the start-up weight
- Make sure gas flow is set at 20.0
- Press Start icon on top of the right panel to start the test
- Go to the Instrument view by clicking on the icon under File to view the graph as real time monitoring of weight vs temperature graph

## Data Analysis

- Once the run is over, it will be automatically saved and will open automatically in the data analysis window (3<sup>rd</sup> icon on the top panel)
- To remove any noises in the graph, click Math tab in the top panel, smooth then Calculate
- Click on Math tab again, click derivative, the highest peak will show up which is the highest point of change
- Click Calc. (Calculate) in the top panel, Click on Peak Area and check Peak height in the pop-up window. Mark the peak area on the graph with the mouse. Click Calculate. Peak height value will show up. Record the value shown
- Right click on the curve and remove curve
- Click on Calc. again then Onset. Insert the **value of peak height** as the start value. Insert the **value of the end temperature** as the end value is the. Press calculate
- Draw a tangent to each of the 2 points on the graph. Where the 2 tangents meet is the onset point