TA Instruments TGA Instructions

This handout explains how to use the TA Instruments Thermogravimetric Analyzer (TGA) found in CP-107. This is a research instrument, so please be exceedingly careful in its use. Follow these instructions EXACTLY. You will use this instrument to analyze your sample of YBa$_2$Cu$_3$O$_{7-x}$.

Step by Step Instructions

Sign in on the logbook.

On the computer the Instrument Control screen will be displayed. There are a number of function key descriptors at the bottom of the screen. **Press F4** (Experimental parameters).

On the Experimental Parameters screen you will see a variety of information. **Press F1** (Sample Info).

On the Sample Info screen, type in information for operator and comments (be descriptive). Say "yes" to saving the datafile. The filename should look like "C:CHE450.002" -- do not touch the filename (but write it down, we will need it later)!!

**Press F2** (Method Editor). On the next screen, press F5 (Select Method). Enter the number corresponding to the CHE450 method and then hit F8 (Accept this Form).

**Press the ESC** key on the keyboard twice go back to the Instrument Control screen (or hit F12 on the keyboard to go there any time....)

Now turn on the gas flow: We will be using a mixture of 4% hydrogen and 96% nitrogen. This should be in a blue cylinder (double check the label,
don't rely on the tank color!). Simply turn on the tank -- the rest of the valves etc. will be preset.

**Now we need to load the sample pan:**

The pan should have been left clean (clean is a relative term). NEVER TOUCH THE PAN WITH YOUR FINGERS -- TWEEZERS ONLY!!!! The oils in your fingers are enough to throw off your readings!

The pan should be positioned on the sample loading stage (A) of the TGA. It should be centered on the small black circle with the sample pan handle oriented perpendicular to the benchtop.

Press the **TARE** button on the TGA control panel (B). The sample loading platform will swing over and (if you positioned it correctly) will deposit the pan on a thin wire hook. The furnace (C) will then raise itself, come to equilibrium, tare itself and then redeposit your pan. The process takes about 5 minutes.

UNDER NO CIRCUMSTANCES ATTEMPT TO PLACE THE PAN ON THE HOOK (OR REMOVE IT) BY HAND. YOU WILL DAMAGE THE INSTRUMENT AND YOU WILL BE SEVERELY PENALIZED.

Using tweezers, place the pan on a clean KimWipe on the benchtop. Place your powdered sample into the pan with a spatula, filling the bottom half or so of the pan (a small mound is OK). Make sure no sample is stuck to the arm or sides of the pan and that no material is in danger of falling out of the pan.

Replace the pan on sample loading platform as you did when you tared it.

Press the **LOAD** button on the TGA control panel. Once the pan is hanging from the wire, press the **FURNACE** button on the TGA control panel once. The furnace will move up and you are ready to run!

**Press the F1 (Start) key on the computer.** Your run will begin with an equilibration to 30 degrees C and then ramp at 20 degrees per minute to 930 degrees C. The run will continue isothermally for up to 60 minutes.

**We can monitor the progress of your TGA by pressing F5 (Realtime Plot).**

Press F1 (Plot 1 Parameters) followed by F3 (Parameters). Set the x-axis value to "time", curve 1 to "signal A", curve 2 to "Temperature" and then
**Press F8** to accept your choices. **Pressing F1** will start the real time plot of your data.

When your run is complete, the machine will take care of itself. However, you will want to plot out your data using a separate data analysis program that will calculate weight changes for us. To access this program **press F11 (Data Analysis)**.

**Press F1** (Get New Program) and select the program called "TGA 5.1".

**Press F1** (Start Program). You will be asked for your filename -- type it in. Your sample information will be displayed and we are ready to set up your plot. **Press F1 (No Limits)** and your plot will be displayed.

**Press F5** (Analyze Curve). We want to measure the weight change between the beginning and end of your run so we can use **F2 (Weight Change)**.

A cursor will appear on the left. Move it with the arrow keys and hit return. Now move the right cursor and then press return. The weight change will be calculated and displayed...follow the computer prompts.

Once you have your plot displayed the way you like it, make sure that the plotter is loaded with a sheet of paper and then press **F4 (Go to Hard Copy)**. **Press F1 (Plotter)** to get your plot.

If you wish you can try plotting your data in other ways (T on the x-axis or maybe derivative curve etc.).

**To finish up do the following:**

Press **F12 (Instrument Control)** to leave the computer on the Instrument Control screen.

Clean out the sample pan using tweezers, a spatula (do not stab, scrape or dent the pan) and/or a brush. **DO NOT TOUCH THE PAN WITH YOUR FINGERS!!!!!**. Ask your instructor if the pan is clean enough.

Turn off the gas cylinder.

Sign out on the logbook.

As always, immediately report any problems to an instructor.