

## DSC Manual

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1. **POWER** should be always **ON**.
2. Turn **ON- Cooling system and the Heater**. Push **STOP** on the instrument to disconnect the DSC from the computer.
3. Check the purge gas (nitrogen). The recommended flow rate is **120** and **40**.
4. Open **TA Thermal Advantage**. Be sure that you have DSC page. You can use DSC and DMA at the same time.
5. After turning **ON** the DSC please run "**TEST**" to keep the DSC at room temperature. Run 1 cycle, temperature 24-25 °C, save as test in your file (see **RUN EXPERIMENT**).

### RUN EXPERIMENT

1. **Prepare** and load a pan with 5-20 mg of the polymer (see **Table 1**). Never open DSC if the cell is cold because you will condense moisture. Don't touch pans with your fingers.
2. **Select the instrument mode**: click the mode button on the tool bar. You can select the mode you want to use (Standard or Modulated).
3. **Summary Page**: Enter **Sample Name**, **Sample Size** (mg). Enter **Comments**, **Data Filename** by clicking on the browse button to the right of the **Data File**, then select the desired storage location and enter in the filename. Click **OK** when finished. Choose one of the common test templates on the Summary Page (Cyclic, Custom, Heat/cool/Heat etc.). If Cyclic test was selected a preprogrammed test template will display notes specific to the test chosen. Click the **Apply** button to apply the information on this page to the current run.
4. **Procedure Page**: enter **Test**, **Notes**, **Start Temp.**, **Heating and Cooling Rate** (10 °C/min typical rate- see Table 1). Enter **Final Temperature**. Select a final temperature that does not cause decomposition of the sample in a DSC cell. Degradation products can condense in the cell and cause corrosion of the cell and baseline problems. **For unknown polymer TGA MUST BE** used to determine stability of the polymer. Insert number of cycles (3-5). Check the **Post Test**. If desired, you can change the conditions that will apply at the end of the run. Click **Apply** button when finished to save these changes.
5. **Notes Page**: Enter the name of **Operator**. Specify the pan type used for this experiment from the list of available pans. Add "extended text" to include additional information about sample or experiment, sample treatment, data sampling parameters. **Gas #1**: Nitrogen, Flow Rate 40 mL/min. **Gas #2** NO. Click the Apply button to apply the information on this page to current run.
6. **START** from the tool bar. Experiment will be saved automatically.
7. Write your name/advisor name/ VLID account # / time (from- to) in the **LOGBOOK!** If error occurs, make a note of the test number in which the error occurred.
8. When you finish **TURN OFF** cooling system. After 10-15 min **TURN OFF HEATER** and push **STOP** on the DSC.

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### ANALYSIS

1. Open **TA UNIVERSAL ANALYSIS** from the desktop. File- Open. Data/ DSC / Users / your File. Make any corrections needed to the information displayed on the screen. You can change size or exotherm up or down. Click **OK**.
2. Use the **Analyze menu** to choose the type of analysis that you want to perform on the current data file ( $T_m$  or  $T_g$ ). Use the Tools menu to select from the list of different types of functions that can be performed on the graph. You can smooth, shift, and/or rotate the curve. TA Instrument **Manual** is available on the Desktop.

**Table 1.**

Type of Measurements	Sample Size (mg)	Heating Rate (°C/min)
Glass transition	10-20	10-20
Melting point	2-10	5-10
Heat capacity	10-70	20*
Purity	1-3	0.5-1
Crystallinity Or oxidative stability	5-10	5-10

**MORE**

1. Begin a DSC analysis about 40 °C below the first event of interest. A heating rate of 5 - 10 °C/min gives four to eight minutes for the instrument to equilibrate before reaching the temperature of interest. Select a final temperature that does not cause decomposition of the sample in a DSC cell. Degradation products can condense in the cell and cause corrosion of the cell and baseline problems. **TGA must be used** to determine stability of the unknown polymer.
2. Sample pans should have high thermal conductivity, and low heat capacity. Pans may be made from aluminum, copper, gold, platinum, glass, carbon, stainless steel, or mild steel, but typically aluminum pans are used. The selection of the sample pan depends on specimen size and shape, the maximum temperature of the experiment, the possibility of chemical reaction between pan and sample, and whether or not the pan needs to retain vapors. It is better to cut out a representative sample rather than crush it, which may impart stress to the sample. Cutting a sample from the bulk with a razor blade or clipper imposes a minimum stress to the sample.
3. When analyzing **thermoplastics** by DSC, the experimental program should include three cycles at 10 °C/min: heating/cooling/heating. The results of the first heating depend on the unknown thermal history of the material and unreliable for analysis. The cooling cycle gives the sample a known thermal history, and the scan can be used to compare polymer crystallization properties. Finally, the results of the heating are a function of the material and the known thermal history imparted during cooling cycle, and useful for comparison of polymers.
4. When analyzing **thermoset polymers** by DSC, the experimental protocol should begin with an annealing period, should progress to the maximum analysis temperature, and then progress to a very rapid cooling (quenching). Finally, the analytical curve is obtained by heating at a rate 10 to 20 °C/min. Annealing at approximately 25 °C above the onset of the  $T_g$  eliminates effect of enthalpic relaxation.