

TA Instruments DSC 2500 – Quick Start Guide
Polymer Facilities - MRL @ UCSB
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Safety

Hot Surfaces. The DSC is capable of heating samples to 400°C. During testing, the furnace assembly becomes noticeably hot. After testing, the furnace assembly, sample, and lid may remain hot for some time. **Do not touch the furnace assembly** or sample stage area during heating or while the sample is cooling from an elevated temperature.

Cold Surfaces. The DSC is capable of cooling samples to -170°C through the use of liquid nitrogen. Because of its low temperature (-170°C), liquid nitrogen will cause tissue damage upon contact with your skin.

Users must wear gloves at all times when preparing samples and when the DSC is open in order to prevent contamination.

For new samples, a TGA must be run prior to DSC analysis

The maximum DSC upper temperature will be determined by 3% mass loss from the TGA.

The actual upper temperature should be no more than 3 minutes from TGA decomposition temperature based upon scan rate. TGA temp – scan rate*3 minutes = Upper DSC temperature

e.g. TGA temp 300 – (10C/min)*3 min = DSC temp of 270 C

There are two DSCs- each with a different cooling option. One has a refrigerated cooling system (RCS) and the other a LN₂ pump. The lowest temperature achieved with each pump depends on your scan rate. If you do not need to go below -70C, please use the DSC RCS. If you are not sure, please ask.

Controlled Cooling Rate	RCS 120 lowest temperature	LN2 Pump lowest temperature
50 C/min	120 C	0 C
20 C/min	-20 C	-100 C
10 C/min	-50 C	-150 C
5 C/min	-75 C	-165 C
1 C/min	-90 C	-180 C

Reservations

- Reservations are required for the DSC, as the run times are typically long. Reservations are made through FBS. There are two DSCs: RCS and LN₂. Each has a separate SKU, so you will need to make a reservation for either DSC RCS or DSC LN₂.
- If you are running more than 3 samples, please let Rachel know a day in advance (before 3pm) to make sure you have enough LN₂

Getting started

1. Turn on the computer by logging into FBS.
2. Open the respective valve from the nitrogen generator for Nitrogen flow to the instrument. The valve on the left is for the DSC RCS, and the valve on the right is for the DSC LN₂.
3. Note: The LN₂ tank is always open. **If you fill the little dewar before your run, you must wait 30 min before starting a run!**
4. From the touch screen, reset the autosampler.

Sample Preparation

- Approximately 5mg
- Aluminum TZero pans- best for solids/powders (Grey Press and Black Die Set)
- Aluminum TZero hermetic – uses TZero pan with TZero hermetic lid (Grey Press and Blue Die Set)

Setting up an experiment in Trios

- If the TA Instruments DSC software is not running, open it by clicking the "Trios" icon, then click the DSC icon (the only one in the folder) and click Connect.
- In the Single Run Form, you will enter these parameters in the Sample Box (top segment):
 - Sample Name
 - Sample Pan Number
 - Sample Mass (without the pan)
 - Pan Mass (mass of empty pan and lid)
- Reference Position (drop down box, positions 50-54). The pan type is linked to the reference pan, and the reference pan mass should be closely matched to the empty pan masses of your samples (± 0.3 mg).



Creating your Procedure (second segment). There are many preset 'Test' options in the Procedure segment. To create a custom procedure:

- 1) First, select a preloaded test from the 'Test' drop down menu (e.g. Heat Cool Heat)
- 2) Second, select custom from the same 'Test' drop down menu.
- 3) You now can edit the experiment by clicking the Edit button.
- 4) Drag segments from the right side of the screen into the segment list to create the desired procedure.
- 5) Once you are done, you can save your procedure into your folder with the save button on the upper right portion of the Procedure segment box.

Starting your Experiment

Once your sequence is complete, you can start the sequence by pressing the green start button at the top of the software. If the button is not illuminated, check the bottom of the screen for red circles indicating the not ready error.

Basic Data Analysis

If you have not already done so, please download TRIOS from TA Instruments (free-
<https://www.tainstruments.com/support/software-downloads-support/downloads/>)

1. Open the desired data file you wish to analyze by clicking on the Trios icon in the upper left hand corner and select open.
2. Display the Graph in the Step Overlay Tab (bottom of the page).
3. Configure the graph variables as desired.
4. Rescale the graph, if needed.
5. Click on the desired curve you wish to analyze (alternatively, select the desired curve from the graph legend).
6. Click the Analysis tab to display the toolbars.
7. Select the desired analysis/model from the drop-down menu.
8. Click **Analyze**.
9. Your analyzed data will now appear on the graph.

Exporting and Saving your Graph as a PDF

1. To Export, click the TRIOS button in the upper left corner of the main window.
2. Select Export → Excel to export as an Excel spreadsheet
3. A popup window will appear with options for your file.
 - a. Select include parameters. This will include your experimental parameters for this run (useful for future use).

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- b. Deselect separate steps into worksheets, unless you want each heating and cooling cycle as a separate tab.
 - c. If Open Excel after exporting is selected, your spreadsheet will open once exported. Deselect this if you do not want it to open.
- 4. To Print, click the TRIOS button in the upper left corner of the main window
 - 5. Select Print → Print PDF

Shutting down

- 1. Refill the small LNCS dewar if using the LN₂ pump
- 2. Turn off the valve to the Nitrogen flow.
- 3. Record number of samples in both FBS (update consumables) and on log sheet
- 4. Log out of FBS