Quick Guide to Operating TA Instruments Discovery DSC 250

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Instrument Overview

TA Instrument Discovery Differential Scanning Calorimeter [DSC] 250. This DSC is equipped with the Refrigerated Cooling System 90 [RCS 90]. This configuration can be used for experiments requiring cooling within an operating range of -90°C to 550°C. The maximum rate of cooling depends on the temperature range of your experiment. This system is usually calibrated for measurements between –90°C to 400°C. Additionally the DSC/RCS90 system is equipped with a four-axis robotic autosampler to safely and accurately load the sample and reference pans. Unattended processing of up to 53 samples + 1 reference is possible with the use of the autosampler and TRIOS software's sequence design tools. This tool is capable of measuring melting point and latent heat of fusion with an accuracy of 1% and 3% respectively.[1]

Before Starting a Session

Log into your instrument session at https://fom.engr.pitt.edu/fom/.

Specimen Decomposition and Handling

Ideally samples should be able to withstand the entire experimental temperature range without decomposing. Sample decomposition increases the risk of DSC cell contamination. Oil, dust, and other material can contaminate the DSC cell. Gloves should be worn when handling sample pans, press dies, and related tools.

System Startup

1.1. Open the purge gas supply and verify there is sufficient gas to last the duration of your measurements.

✍The GDM light in the **Status Bar** will turn green and the base purge signal will indicate approximately 370 ml/min of flow.

1.2. Verify that the DSC 250 instrument is turned on and the RCS 90 cooler is both turned on and switched to "Event" mode.

1.3. Open TRIOS software and connect to the DSC 250 instrument.

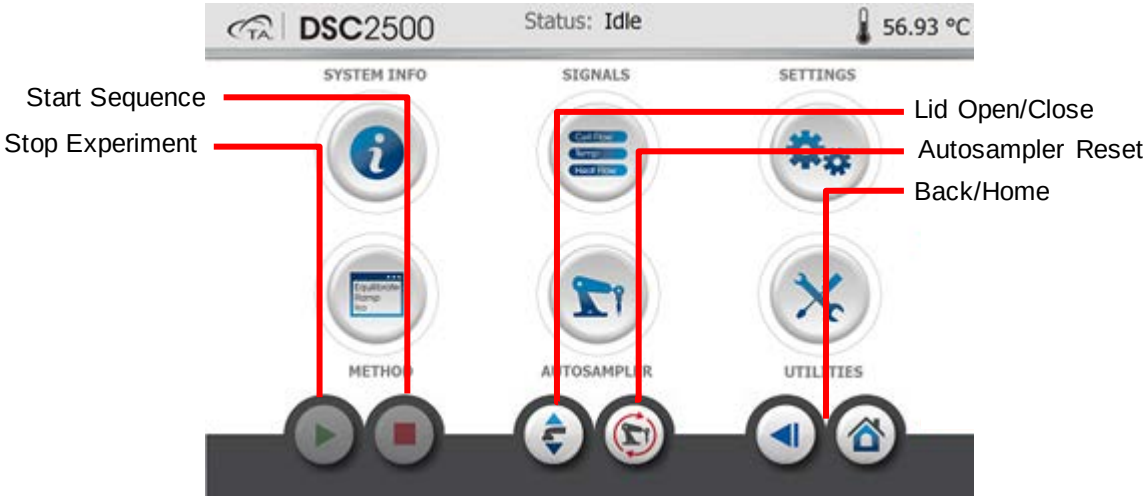
1.4. Turn on the RCS 90 cooler using the "Temperature" subsection of the **Control Panel** window.

✍The flange temperature will approach -90°C approximately 15 minutes after the RCS 90 is switched on.

[1] Trios Discovery DSC Help

DSC User Interface

TRIOS DSC software interface



DSC Touchscreen interface

✍ "Autosampler Reset" button is a good way to correct most autosampler errors.

Basic DSC Experiment Outline

The general outline for DSC experiments include:

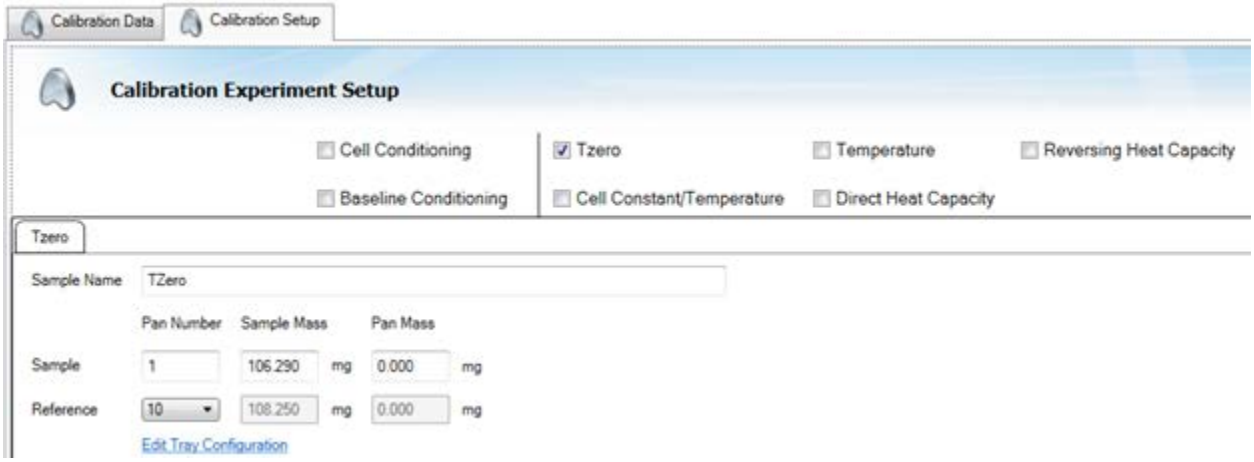
1. Calibration / verification of instrument performance. (Optional)
2. Selecting sample pan / lid system.
3. Preparing sample.
4. Configuring the autosampler or manually loading the DSC cell.
5. Designing the experimental run or sequence of runs using the TRIOS software.
6. Running the experiment or sequence.

Calibration

The DSC requires periodic recalibration. Tzero calibration is typically completed every 4-6 months or as required due to DSC cell contamination. Temperature and enthalpy calibrations should be completed with appropriate melting standards when highly accurate experimental results are required or after a Tzero calibration.

🌪 For more information please consult "Calibrating the Discovery DSC" help topic.

✍ The current calibration data set may viewed by opening the "Calibration" sub-panel of the **File Manager Panel**. Additionally new calibrations runs may be created using this sub-panel.



Tzero Calibration

✍The recommended heating rate for Tzero calibration is 20 °C/min. Rates lower than 10 °C/min are not recommended.

2.1. Access the Calibration sub-panel of the **File Manager Panel** and activate the Calibration Setup tab.

2.2. Select Tzero checkbox.

2.3. Enter the following information:

- a. Sample Name: Tzero
- b. Sample Pan Number: Autosampler tray location of the **clear** sapphire disk.
- c. Reference Pan Number: Autosampler tray location of the **dark** sapphire disk.
- d. Operator/Project/Notes: Completed with user supplied information.

2.4. Modify the method parameters as required. Default Method Parameters:

- a. Ramp Rate: 20 °C/min

b. Lower Temperature: -90 °C
c. Upper Temperature: 400 °C
d. Isothermal: 10 min
e. Perform Verification Run After Calibration: Checked.

2.5. Press the Run All button. A sequence of two calibration runs plus an additional baseline verification run will now begin.

Enthalpy and Temperature Calibration

✍ A Tzero calibration is required before the enthalpy and temperature calibrations can be completed.

🌪 See Preparing Sample and Setting up DSC Experiments section for additional information.

3.1. Measure and record the weights of empty sample and reference pans. Use Tzero pans and standard lids.
3.2. Prepare a 1-5 mg sample of indium. Load indium into sample pan, crimp close the pan and lid, and load into an autosampler tray position.
3.3. Crimp close an empty pan and lid and load into an autosampler tray position.
3.4. Access the Calibration sub-panel of the **File Manager Panel** and activate the Calibration Setup tab.
3.5. Select the Cell Constant/Temperature checkbox.
3.6. Enter the following information:
   a. Reference Pan Number: Autosampler tray location of the empty pan.
   b. Operator/Project/Notes: Completed with user supplied information.
   c. Premet: Checked
   d. Calibration Experiment parameters:
      i. Reference Material: indium
      ii. Melt Temp.: 156.598
      iii. Lower Limit: 106.60
      iv. Upper Limit: 186.60
      v. Pan Number: Autosampler tray location of indium sample.
      vi. Sample mass: Measured sample mass.
      vii. Pan Mass: Measured pan/lid mass.
   e. Calibration / Perform Verification after Calibration: Checked.
   f. Verification Experiment parameters are the same as the Calibration Experiment parameters.
3.7. Press Run All button to begin the calibration run.

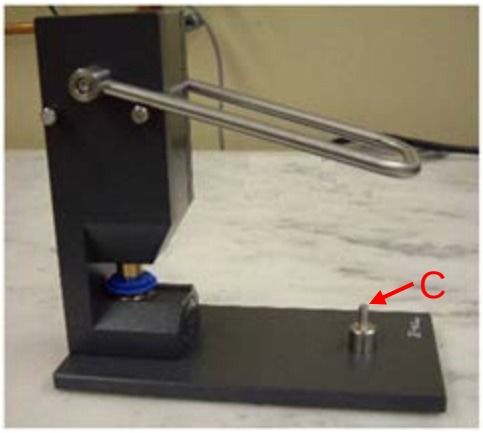
Preparing Samples and Setting up DSC Experiments

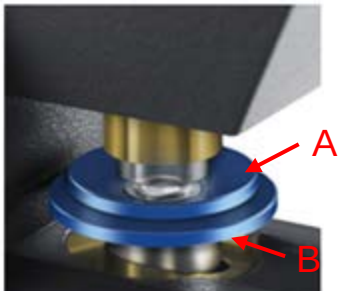
Supplied for use with this instrument are Tzero pans, Tzero lids, and Tzero hermetic lids. The sample pan is loaded with a sample of a known mass. The optimal sample weight is dependent on the type of measurement being conducted (see table). Pans can be sealed using the Tzero press and die sets which are compatible with a range of pans.

🌪*See "Selecting a Sample Pan" and "Preparing Samples" help topics for more information.*

Sample/Size & Heating Rate[1]			**Pan / Die Selection[1]**		
Type of Measurement	Sample Size (mg)	Heating Rate (°C/min)	Pan / Lid System	Die Set	Capacity
Glass transition	10 -- 20	10 -- 20	Tzero aluminum pan / Tzero lid	**BLACK**	20 µL
Melting point	2 -- 10	5 -- 10			
Kinetics (Borchardt & Daniels)	5 -- 10	5 -- 20	Tzero aluminum pan / Tzero hermetic lid	**BLUE**	40 µL
Kinetics (ASTM)	2 -- 5	.5 -- 20	Standard aluminum pans / lids	**Green**	NA
Heating Capacity	10 -- 20	20			
Purity	1 -- 3	.5 -- 1			
Crystallinity	5 -- 10	5 -- 10			
Oxidative stability	5 -- 10	5 -- 10			
MDSC	2 -- 10	1 -- 5			

4.1. Determine the appropriate sample mass for your DSC experiment (see Sample/Size & Heating Rate table).
4.2. Determine the appropriate pan, lid and press die set. (see Pan / Die Selection table)
4.3. Install appropriate die set into the Tzero press.
4.4. Using an analytical balance record the mass of the sample.
   ✍When not using Tzero pans, it may be necessary to record the mass of the sample and reference pans.
4.5. Spread the sample as evenly and as flat as possible into the sample pan.
   ✍Non-flat samples may need to be pressed using the cupped upper die available in the **BLACK** and **GREEN** die sets.
4.6. Using tweezers position the lid onto the pan, and place the assembled pan into the lower die.
4.7. Cycle the press handle to crimp the lid onto the pan.
   ✍No significant resistance should be encountered during pres operation.
4.8. If using hermetic sample pans use the pan ejection pin to remove the sealed pan from the lower die.
4.9. Place the closed sample pan into any available position in the autoloader tray.
   ✍Some experiments, such as isothermal oxidation, and specimens require an open pan. When using open pans, the risk of DSC cell contamination may increase.





Hermetic
Upper Die

The Tzero sample press with handle in the closed position and the hermetic die set installed.

Setting up Discovery DSC Experiments

See "Setting Up Discovery DSC Experiments" help topics for more information.

The DSC/RCS90 is equipped with autosampler and design software which facilitates the serial execution of a sequence of experiments or "runs". In each run the sample parameters, procedure parameters, and other experimental information are defined. Runs and/or a sequence of runs can be saved and reused from session to session.

5.1. Select the "Experiment" tab in the **File Manager Panel**. The "Design Run" experimental window is displayed in the **Display Panel Window**.

5.2. Enter the sample information.

a. Enter the sample name.
b. Enter the sample pan number, sample mass, and if not using Tzero pans the pan mass.

5.3. Enter the reference information.

a. Press the "Edit Tray Configuration" link. The tray configuration graphic will appear. Modify the tray configuration to accurately represent the types and pan numbers of the reference pans loaded into the autosampler tray.
b. Select the reference pan number from the drop down list.

Setting the sample pan or reference pan number to 0 implies manual loading of the pan, the autosampler will not be used.

5.4. Define the Operator, Project and Note fields.

5.5. Define the DSC procedure.

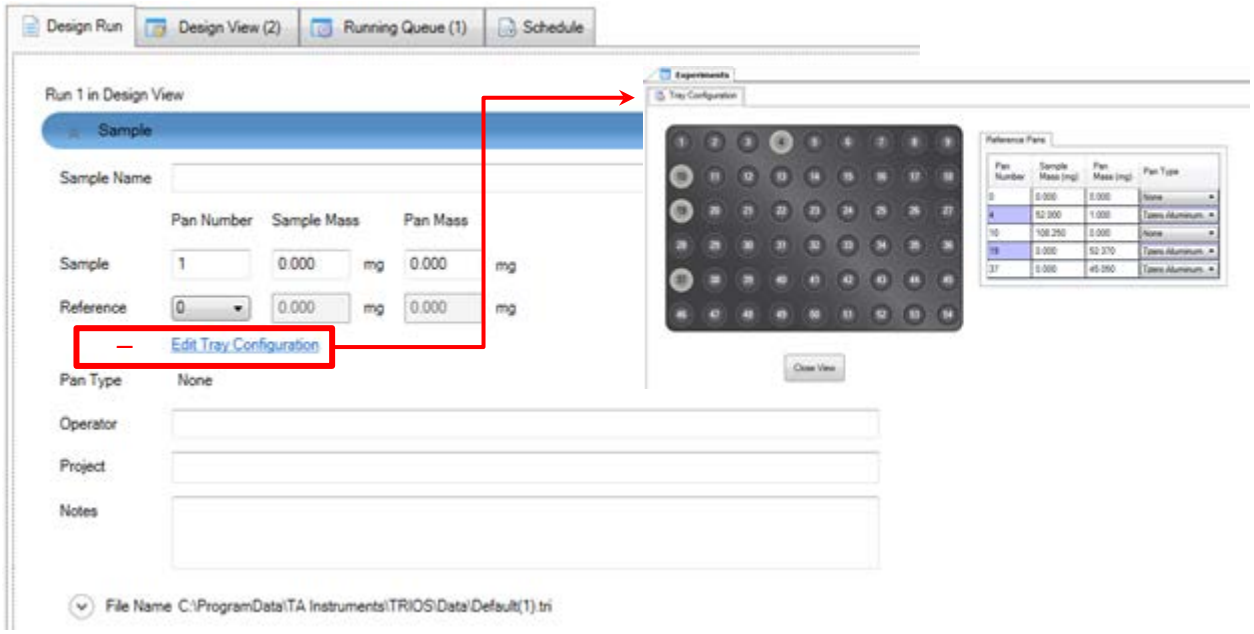
a. Using the test drop-down list you can select a pre-programmed test template or, by selecting "Custom", create your own method.
b. Optionally name the test
c. Edit the segments to define the specific experimental conditions of your DSC experiment.

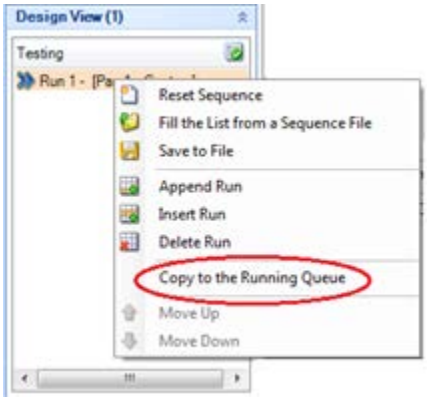
5.6. In the "Design View" sub-panel of the **File Manager Panel**, right-click on the currently designed run, and select "Copy to the Running Queue".

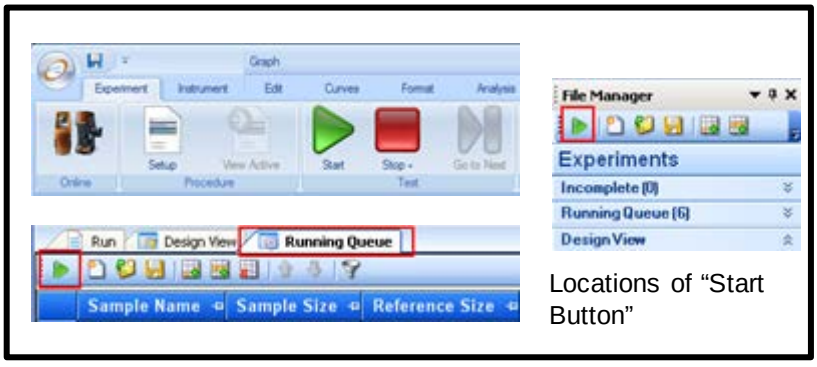
5.7. To begin executing runs located in the running queue, press a green start button located in several positions of the user interface (see below).

✍Runs may be inserted or appended into the running queue from the “Design View” window, previously completed runs, or saved custom templates.

✍It is possible to modify the currently running method by interacting with the “Running Method List” sub-panel of the “Control Panel”







Locations of “Start Button”

Instrument Shutdown

⛔ ✍ It takes approximately 20 minutes for the flange temperature to approach the standard standby temperature. If purge gas is turned off prematurely, condensate may form inside the DSC and deteriorate performance.

4.1. Turn off the RCS 90 cooler using the "Temperature" subsection of the **Control Panel** window.
4.2. If changed during the course of your experiment, set the "Standby Temperature" to 40°C.
4.3. When the "Flange Temperature" reaches 40°C, stop the purge gas by closing regulator.
4.4. Exit the TRIOS software, Leave DSC and RCS 90 powered on
4.5. Log out of FOM.

Other Information

This section contains procedures directly from the Trios Discovery DSC Help document. They are included here for reference purposes. If you are unsure how to correctly implement these procedures please seek advice from NFCF staff.

Cleaning a Contaminated Discovery DSC Standard Cell

1. Determine that the anomalies seen in the baseline are actually caused by contamination, and not a faulty Tzero calibration. Contamination is normally manifested in very sharp, distinct, and sometimes noisy peaks in an empty baseline (no pans) run.
2. If you have determined that the anomalies in the baseline are in fact caused by contamination, select a solvent that is most appropriate for dissolving the contaminant. Note that in most cases an organic solvent such as acetone or methanol may be necessary, but an aqueous solvent might also work for some contaminates.
3. Dampen the end of a small cotton swab with the chosen solvent. Avoid saturating the swab, simply moisten it.
4. Gently dissolve the contaminants with the swab, starting at the outside of the silver housing within the sample chamber and working inwards. Repeat the process until the contaminants have been removed. Do not rub hard with the swab.
5. Dry the interior of the cell with a clean dry swab.
6. Heat the cell to 200˚C in nitrogen purge at approximately 50 mL/min, and hold for 5 minutes to remove all traces of solvent.
7. Verify by running an empty baseline test. If the anomalies are gone, but the baseline shape or slope is not acceptable, then Tzero calibration may be necessary. If the anomalies are still there, but are less evident, repeat the above steps and re-verify the baseline.

✍Cleaning of the cell is performed with the lid open to prevent the gas exit tube from becoming plugged by condensing off-gases. If your instrument is equipped with an Autolid mechanism, open the cell manually at the start of the method. You can open the cell by selecting Lid on the DSC user interface or by clicking on the Lids icon within the Control panel.

Drying the DSC-RCS or DSC-LN Pump System

Under certain conditions, it may be necessary to dry out the DSC-RCS or DSC-LN Pump system. To remove moisture in the DSC cell and cooling head, access the Calibration Setup page and run a Cell Conditioning experiment.

The default Conditioning Temperature (75°C) and Hold Time (120 min) values should be used in most cases.

⛔ Never set the Conditioning Temperature over 100°C.