

Standard Operating Procedure: TA Instruments – Discovery DSC

APPROVAL REQUIREMENTS

TITLE	DEPT	NAME (PRINTED)	SIGNATURE	DATE
Author	RES	Chris Kuncho		20-FEB-15
Reviewer	RES	Patrick Casey		20-FEB-15

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PRIOR TO OPERATION, READ THE SAFETY PROVISIONS AND REVIEW THIS STANDARD OPERATING PROCEDURE IN ITS ENTIRETY.



This document should only serve as a reference. The TA Instruments Discovery DSC should not be operated by anyone without express permission and proper training by approved personnel.

SAFETY

The subsections below outline some of the obvious / major hazards that could exist during the operation of the TA Instruments Discovery DSC, and are divided to bring a level of clarity to such hazards.

Electrical Hazards: Electrical equipment should be installed, operated, serviced, and maintained only by qualified personnel only. Do not block the main switch as potential risk of personal injury or equipment damage if the instrument cannot be turned off in an emergency.

Inhalation Hazards: All materials being used should have Safety Data Sheets (SDS). Confirm that the material does not pose an inhalation hazard for the extrusion parameters being used.

Burn Hazards: The Furnace will be very hot. Be careful of hot surfaces with exposed skin and clothing.

Mechanical Hazards: Keep hands and long hair away from moving parts and feed sections. All long hair should be tied back.

Personal Protective Equipment (PPE): All personnel should utilize and implement proper PPE per OSHA requirements. Refer to OSHA requirements for proper use and implementation of PPE. The following items are suggested as a minimum to avoid injury based on the operation procedure outlined in this manual.

- Safety glasses
- Lab coat
- Long pants
- Close-toed shoes
- Proper gloves (when required)

INTRODUCTION

1.0 Purpose

- 1.1 The purpose of this Standard Operating Procedure is to guide the user in the basic use of the TA Instruments Discovery Differential Scanning Calorimetry (Discovery DSC).

2.0 Description

- 2.1 The TA Instruments Discovery DSC (**Figure 1**) is used in conjunction with a controller computer and associated software to make up a thermal analysis system.

The Discovery DSC measures temperatures and heat flows associated with thermal transitions in a material. Common usage includes investigation, selection, comparison and end-use performance evaluation of materials in research, quality control and production applications. Properties measured by DSC techniques include glass transitions, "cold" crystallization, phase changes, melting, crystallization, product stability, cure / cure kinetics, and oxidative stability.


 The Diffusion-Bonded Sensor is **VERY** sensitive and precise. Under no circumstances should the sensor ever be touched or manipulated by hand or with tools. Serious damage to the instrumentation could occur.



Figure 1 TA Instruments Discovery DSC

3.0 List of Related Internal Documents

3.1 UMass Lowell Chemical Hygiene Plan

3.2 RES-07-001 ETIC Operations Manual

4.0 List of Related External Documents

4.1 OSHA Laboratory Safety Guidance

4.2 Discovery DSC Getting Started Guide

4.3 Selecting a Sample Pan

4.4 Preparing Standard Aluminum Pans

4.5 Using the Tzero[®] Press

4.6 Calibrating Tzero[®]

4.7 Cleaning the Discovery DSC User Interface and Cell

OPERATION

5.0 Safety

5.1 Safety glasses

5.2 Lab Coat


5.3 Long Pants

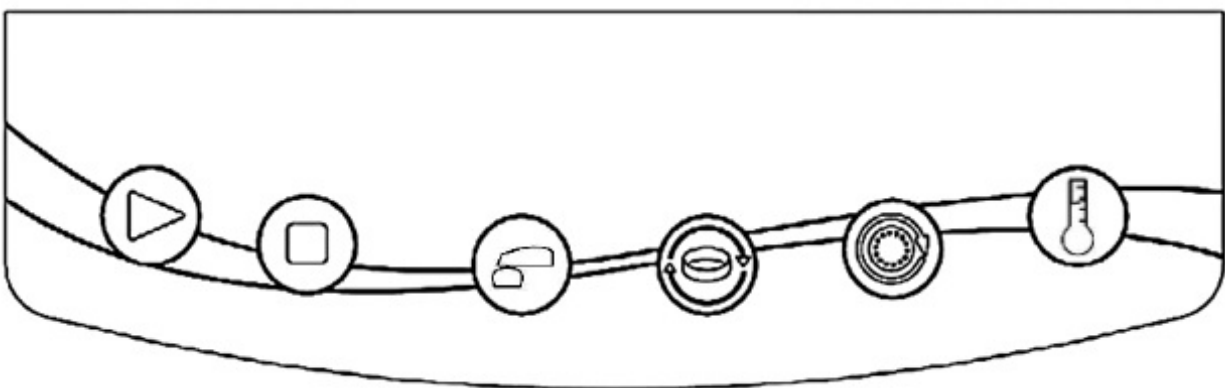
5.4 Close-toed shoes

5.5 Proper Gloves (when required)

5.6 UMass Lowell EEM CHP

5.7 Material Safety Data Sheet(s)

 Before continuing all personnel should familiarize themselves with the TA Instruments Discovery DSC.









Key Name	Description
Start 	<p>This key begins the experiment. This is the same function as Start on the instrument control software.</p> <p>This button is <i>white</i> when the system is in standby mode, <i>yellow</i> when a method is loaded (at least one run in the TRIOS Running Queue), and <i>green</i> when the system is running.</p>
Stop 	<p>If an experiment is running, this key ends the method normally, as though it had run to completion; i.e., the post test conditions go into effect and the data that has been generated is saved. This is the same function as Stop on the instrument control software. Press this key twice to stop temperature control.</p> <p>This button is always <i>red</i>.</p>
Open/Close 	<p>Toggles between the opening and closing of the Discovery DSC AutoLid.</p> <p>This button is always <i>white</i>.</p>
Load/Unload 	<p>Removes the pan from the sample cell position and returns it to its original location (if applicable), and loads the pan from the load position into the sample position. Does not load samples from the tray.</p> <p>This button is always <i>white</i>.</p>
Reset 	<p>Resets the Autosampler.</p> <p>Caution: This command erases all current statuses for the sample and/or reference pan positional information.</p> <p>This button is always <i>white</i>.</p>
Standby 	<p>Activates the pre-programmed standby temperature when system is in idle mode.</p> <p>This button is <i>green</i> when the system is in standby mode, <i>green</i> when a method is loaded (at least one run in the TRIOS Running Queue), and <i>yellow</i> when the system is running.</p>

Figure 2 TA Instruments Discovery DSC Keypad

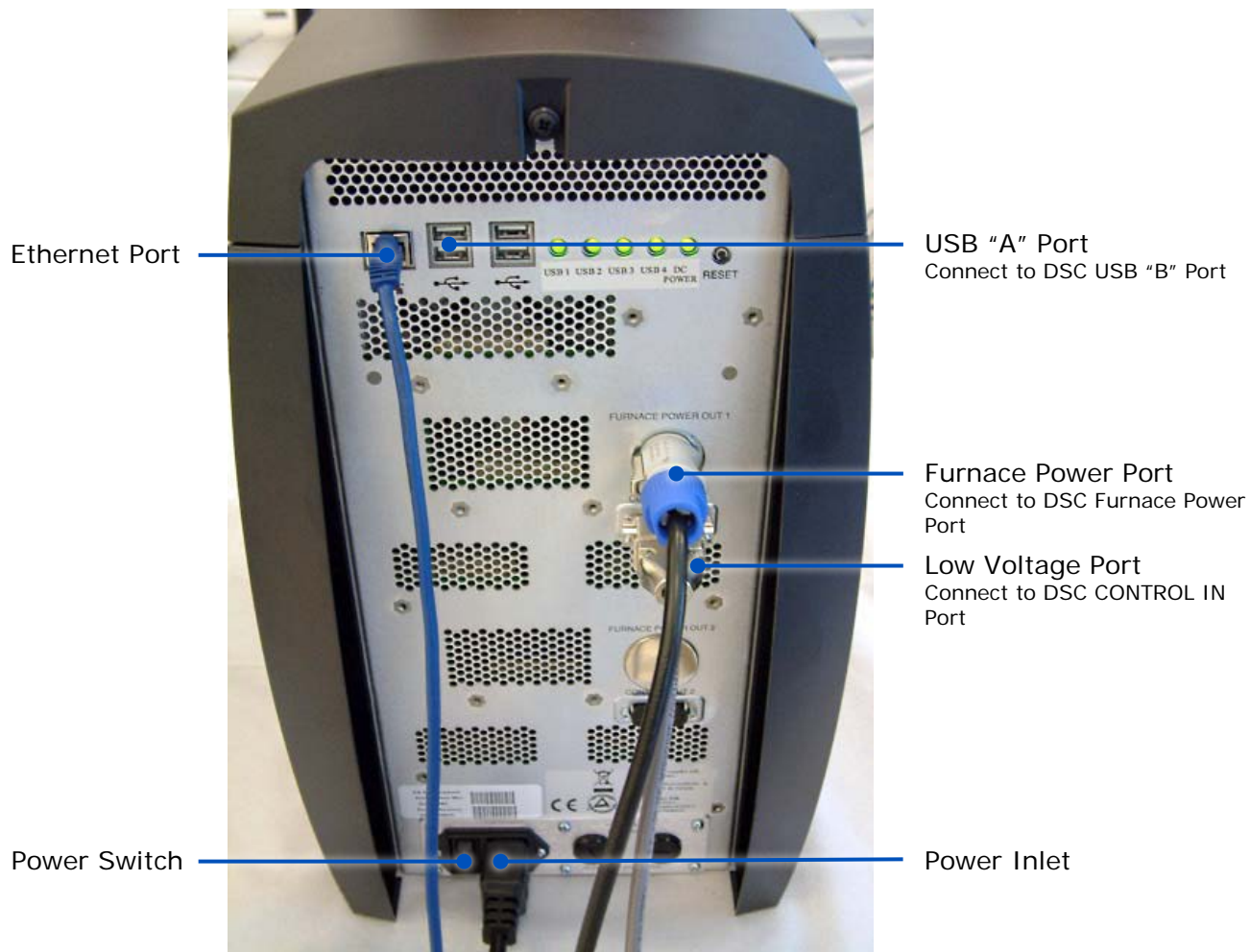


Figure 3 Common Cabinet Rear Panel Connections



Figure 4 Discovery DSC Rear Panel Connections

6.0 Process Description

6.1 Instrument startup

- 6.1.1 The DSC and Common Cabinet should always be on. If the DSC is ever found off, check power connections in the back of the instrument. The common cabinet has a power toggle on its lower rear panel near the power connector (see Power Switch in [Figure 3](#)).
- 6.1.2 If the PC is off, start up the PC by normal means.
 - 6.1.1.1 Power on the Tower.
 - 6.1.1.2 Power on the Monitor.
- 6.1.3 Lower the DSC AutoLid if it is raised by pressing **Open/Close** button on the DSC keypad (see [Figure 2](#) & [Figure 5](#)).

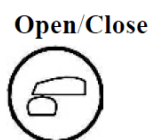


Figure 5 AutoLid Open/Close Icon

- 6.1.4 Start the software.

NOTE: The Discovery DSC can be run using the touch screen on the Discovery Common Cabinet or using the TRIOS Software on the PC.

- 6.1.1.3 Using the PC, click TRIOS Icon ([Figure 6](#))



Figure 6 TRIOS Icon

- 6.1.1.4 Select **Discovery DSC @Lab** and click **Connect** ([Figure 7](#)).

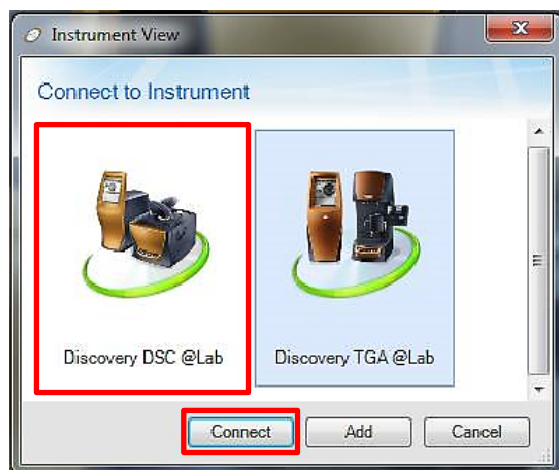


Figure 7 Discovery Instrument View

6.1.1.5 The experiment screen will appear by default.

6.2 Sample Preparation.

NOTE: For this section refer to the following external documents:

- Selecting a Sample Pan
- Preparing Standard Aluminum Pans
- Using the Tzero[®] Press

6.2.1 Select appropriate pan and lid.

NOTE: Samples that are liquid or have a significant quantity of volatiles will require a hermetic pan and lid.

6.2.2 Using an analytical balance, weigh and record the sample pan and lid weight.

6.2.3 Load sample into pan, reweigh pan and lid, and record this new value.

6.2.4 Based on pan type select appropriate crimping die set (reference external document: Selecting a Sample Pan).

6.2.5 Install top of die assembly into crimping vice (reference external document: Preparing Standard Aluminum Pans).

6.2.6 Load sample pan into lower die.

6.2.7 Place sample lid (concave down) onto sample pan.

6.2.8 Install lower die into crimping vice.

6.2.9 Pull vice lever handle forward smoothly. Then push vice lever handle completely back.

6.2.10 Remove lower die assembly.

6.2.11 Using the fixture on the vice carefully poke crimped sample pan out of the lower die.

6.2.12 Place sample pan into numbered position on auto-sampler carousel.

6.2.13 Repeat steps for all samples

6.2.14 When finished, add prepared samples into DSC auto-sampler.

IMPORTANT: take care to note where each sample is placed in the Autosampler.

NOTE: The DSC Autosampler can be removed for easier handling of multiple samples.

NOTE: Do not knock over pans, or damage Autosampler Carousel

NOTE: There is a pin on the Autosampler that lines up with a hole on the carousel. These must be lined up for proper working use of the Autosampler. Also, care must be taken to not damage the Autosampler, or jostle the pans.

NOTE: The DSC Autosampler can accommodate 50 samples for testing. Each individual sample must follow steps 6.3 through 6.4 to be properly tested.

6.3 Running an experiment

6.3.1 On the experiment screen click Empty in running queue, in the upper left section of the screen. This will populate the first run of the sequence (Figure 8).

Experiments		Experiments	
Incomplete (1)	∨	Incomplete (1)	∨
Running Queue (0)	∧	Running Queue (1)	∧
Empty		Run 1 - [Pan 1 - Custom]	

Figure 8 Starting the Experiment

6.3.2 Under the sample field input the following (Figure 9):

- Sample Name
- Sample Pan Number
- Sample Size
- Sample Pan Mass
- Reference
- Operator
- Project
- Notes (Optional)
- File Name and Specified Path (Optional)

Sample

Sample Name

Pan Number Size mg Pan Mass mg

Reference

Operator

Project

Notes

File Name:

Figure 9 Test Sample Experiment Information

6.3.3 If additional runs are needed (**Figure 10**):

6.3.3.1 At the left side of the screen

6.3.3.1.1 Mouse over **Run 1**

6.3.3.1.2 Right Click

6.3.3.1.3 Select Append Run

6.3.3.1.4 Repeat steps 6.3.3.1 as needed per sample and experiment.

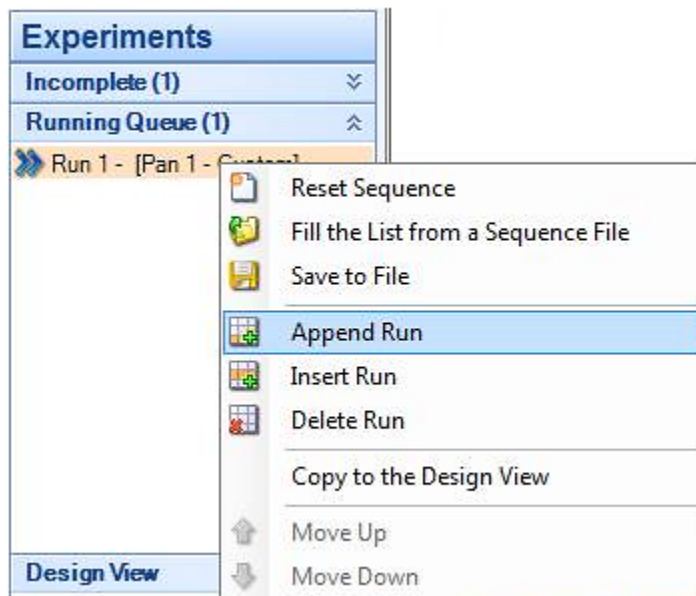


Figure 10 Creating Additional Runs

6.3.2 Click the **Start** icon to run the experiment(s) (**Figure 11**).



Figure 11 Start Icon

NOTE: The readout under the **Start** button gives an approximate time to completely tare all selected positions on the carousel.

6.3.3 Repeat step 6.3.2 for all test samples (if applicable).

6.3.4 Modulated DSC:

6.3.4.1 Sample prep is identical to standard DSC experiments.

6.3.4.2 To select this test, click **Experiments > Design View** in the **File Manager**. In the **Procedure** window, select **Modulated** from the Mode drop-down list (**Figure 12**).

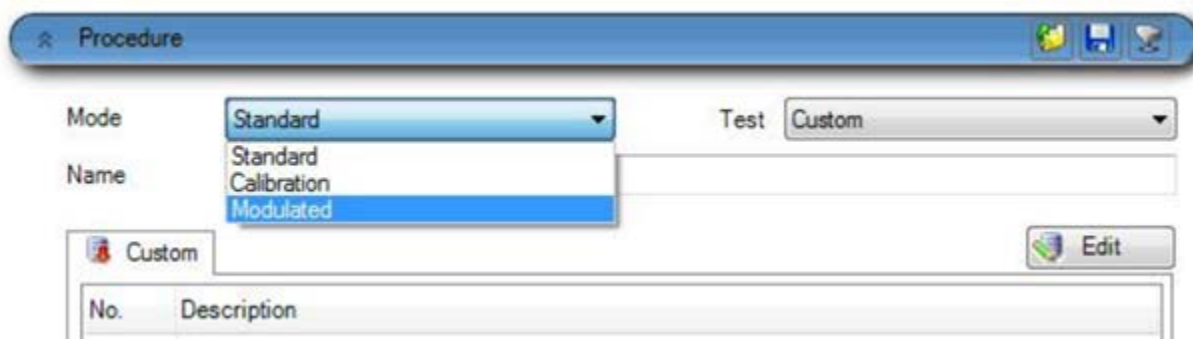


Figure 12 Starting Modulated DSC Experiment

6.3.4.3 Choose the following parameters after selecting the MDSC Conventional procedure:

6.3.4.3.1 **Modulate Temperature Amplitude:** Select a temperature amplitude of modulation between ± 0.2 and 2.0°C . Larger amplitudes (± 1.5 to 2.0°C) should be used when measuring weak transitions. Smaller amplitudes should be used for analysis of sharper transitions which are only a few $^{\circ}\text{C}$ wide.

NOTE: Amplitudes below $\pm 0.1^{\circ}\text{C}$ are not recommended.

6.3.4.3.2 **Modulation Period:** Select a modulation period of 20 to 200 seconds. For most materials in crimped pans, 40 to 60 seconds is recommended.

6.3.4.3.3 **Ramp Rate:** Select an underlying heating/cooling rate of 1 to $5^{\circ}\text{C}/\text{minute}$. Make sure that the rate chosen provides at least 4 complete temperature modulations (oscillations) over the temperature range of each transition studied.

6.3.4.3.4 **Start and Final Temperature:** The starting and final temperatures should be set to cover a wide enough temperature range to observe all the events of interest. As in conventional DSC, the maximum range is -180 to 600°C , but may be reduced depending on the type of sample pan and cooling accessory used. The higher temperature should be low enough to prevent the onset of decomposition.

6.3.4.3.5 (Optional) Left click run in running queue, and select append run (**Figure 10**).

6.3.4.3.6 Repeat steps for all sample pans prepared and placed in auto-sampler carousel for their applicable positions.

6.3.4.3.7 Press start at the top of the screen (**Figure 11**).

7.0 Maintenance

7.1 Preventative Maintenance Instruction: please reference the External Documents below for Discovery DSC Maintenance:

- Calibrating Tzero®
- Cleaning the Discovery DSC User Interface and Cell

7.2 Vendor Information:

TA Instruments

159 Lukens Drive

New Castle, DE 19720

TRAINING CERTIFICATION CHECK SHEET

	UCard ID	NAME (LAST, FIRST)	SIGNATURE	DATE
TRAINER				
TRAINEE				

OPERATION NUMBER	OPERATION DESCRIPTION	TRAINER INITIAL *
1	Can load software and add new experiment	
2	Can create sample pans and load test material	
3	Can define test parameters and start test	
4	Understands the potential hazards associated with equipment	
5	Knows the location of the UML Chemical Hygiene Plan (CHP)	
6		
7		
8		
9		
10		
11		
12		
13		
14		

* TRAINER'S INITIAL CONFIRMS THAT TRAINEE IS PROFICIENT *