## **IMSERC User Manual for TA DSC**

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### **INTRODUCTION**

Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at http://imserc.northwestern.edu/pcm-instruments.html#dsc. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and also available under the corresponding instrument section at http://imserc.northwestern.edu/pcm-instruments.html#dsc by pressing on the 'User manual' button. If while using the system, something happens that you do not understand, please stop, and get help. In any event, be completely prepared to justify your actions. The cost of even minor repairs could be considerable.

### SAFETY

All users of IMSERC must review the general safety policies at http://imserc.northwestern.edu/aboutpolicies.html. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

- Hazardous Chemical Waste Management
- Laboratory Safety
- **Personal Protective Equipment**

You need the above certificates to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at https://learn.northwestern.edu. Upon completion of the certificate, it will take an overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found in outside of room B172 at the north side. Protective eyewear is required in this room, and gloves should be removed when using the computer.



### DATA MANAGEMENT

Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder, otherwise you might not be able to access your data remotely. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied on your group folder on 'imsercdata.northwestern.edu' under 'others/DSC' every few seconds. Please follow instructions at http://imserc.northwestern.edu/about-general-fag.html#data for details about data access.

### SOFTWARE

Data reduction and analysis of thermal data can be performed with the 'TA Universal Analysis' software. Software is installed on the instrument computer. For offline analysis after your instrument reservation is complete, please use the following resources:

- The universal analysis software is free to download and install at https://www.tainstruments.com/installation-of-universal-analysis-from-the-advantage-software-package/
- You have the option to use the instrument computer for analyses, but you must reserve instrument time through NUcore

### **DEFAULT INSTRUMENT STATUS**

The default measurement mode of the TA DSC is Conventional Differential Scanning Calorimetry (DSC) under nitrogen gas from -80 to 500C. Please notify the appropriate staff member well in advance if you would like to run an experiment in a different mode or using a different gas environment. For the full list of modes and capabilities, please check at http://imserc.northwestern.edu/pcm-instruments.html#dsc. Additionally, put a note on your NUcore reservation indicating the preferred mode of your measurement.

The default working condition of the TA DSC is as follows:

1. Computer screen is by default deactivated. You must start your reservation through NUcore to be able to turn on the computer screen. If screen is already on, start your reservation through NUcore



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- 2. The default 'DSC' user account should be logged in. In case the computer was restarted, the password for the 'DSC' account is
- 3. Acquisition software (QSeries) should be running. Leave the acquisition software open when you are done with the measurement
- 4. There should be no error messages on either the front panel of the instrument or the acquisition software. Please check the 'Troubleshooting' section for a potential solution before reporting the error

If there is an error or problem with the instrument that is not covered under the 'Troubleshooting' section, please report the issue by following at least one of the steps below:

- 1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
- 2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html and place the 'Stop' sign near the instrument computer
- 3. Contact a staff member for instructions



### PREPARING AND LOADING A SAMPLE INTO THE INSTRUMENT

Your sample must be cold sealed in an Aluminum crucible and placed in the autosampler of the TA DSC. The following procedure is recommended:

1. Take a set of an Aluminum pan and lid labeled as 'DSC only' from the yellow container located in the middle drawer in the cabinet under the instrument computer (circled in figure 1). Always use tweezers to handle the pan and lid as contact with skin my affect the DSC measurement



- Weigh your sample by using the microbalance. Watch the 'microbalance' video for a visual demonstration on 2. how to use the microbalance:
  - a. Measure the mass of the empty pan
  - b. Remove the pan from the microbalance, load your sample into the pan, and place the pan with your sample on the microbalance
  - c. Record the weight of your sample in the pan
- Remove the pan with the sample from the microbalance, place them into the 3. blue stab of the press, and seal your sample. Watch the 'dsc-sealing-alcrucible' video for a visual demonstration on how to use the press. While handling the crucible with the tweezers, ensure that the rim of the sealed crucible is not deformed as this may create mounting problems with the



autosampler. Figure 3 shows an example of a deformed crucible (left) and a proper sealed crucible (right)



- 4. If you prefer to purge the sample + crucible with nitrogen during the measurement, create a small pin hole on the lid by using a needle (figure 3). A syringe needle is in the white tray next to the yellow case that contains the crucibles
- 5. Lift the protective lid of the autosampler, load your sealed crucible into the autosampler tray, and note the number of the sample position. Ensure that the lid of the autosampler tray is placed and aligned properly on the instrument. There is only one correct placement of the lid. The lid has a long groove that needs to be aligned with the corresponding notch (circled in figure 5)





### SETTING UP DSC EXPERIMENTS

DSC is a thermal analysis technique that measures heat flow into or out of a material as a function of temperature. Typical phenomena that can be measured include glass transitions, melting points, crystallinity fraction, heats of fusion and reactions, oxidative stability, rate and degree of cure, and heat capacity.

Most DSC experiments (conventional DSC) are performed while heating or cooling the material at a constant rate or while holding the material isothermally.

There are two main ways for creating a new measurement procedure:

- The DSC 'Experimental Wizard' will guide you through the setup of different types of DSC experiments, or
- The 'Experimental View' page will let you select from a list of available pre-programmed templates

If you do not have an existing procedure, it is recommended to start with the 'Experimental Wizard' (section 'A. Create a procedure using the experimental wizard') to build a procedure, and then customize the procedure (section 'C. Modify a procedure') with additional options not available through the wizard or template

Please read the detailed instructions below based on the measurement type of your choice.

#### A. CREATE A PROCEDURE USING THE EXPERIMENTAL WIZARD

This method is recommended in case you do not have an existing procedure, or you prefer to create a new method from scratch. Most options/settings on the software are self-explanatory but read the entire section for more details about each option.

- 1. Under the menu 'Experimental', select the 'Wizard...' option
- 2. Select the 'Conventional DSC' option
  - a. For 'Heat Flow Selection' select 'Heat Flow T1 (mW)'
  - b. For 'Cooler Type' select 'RCS (90)', i.e., Refrigerated Cooling System (usable from -90 to 550°C)
- 3. Press on the 'Next' button
- 4. Select the appropriate 'Measurement Type' that works for your measurement. There are four types (a-d):
  - a. 'DSC Ramp' experiments heat or cool the material at a constant rate (figure 4a). The results obtained are affected by the previous thermal history (often imparted on the material during processing, e.g., extrusion). Therefore, the results are designated "as received."





i. 'Start' and 'Final Temperatures': The actual start and final temperatures chosen for a specific experiment must be within the usable temperature of the DSC cell/cooling accessory combination that you are using. These temperatures should bracket the region where sample transitions are expected to occur, and

<b>4</b> a	This e It is us can b acces ramp
1	Enter

This experiment is designed to heat (or cool) the material at a constant rate.
It is used to evaluate materials "as received". The temperature range that
can be used for ramp experiments depends on the type of pan and cooling
accessory. Note: The QCA and FACS are not recommented for cooling
ramp experiments. Press the HELP button below for more details.

Start temperature	Use current	
	35.00	
Heating rate:	10.000	°C/min
Final temperature:	300.00	°C
	-	
	Start temperature Heating rate: Final temperature:	Start temperature     Image: Use current)       35.00       Heating rate:       10.000       Final temperature:

they should be far enough below or above the transitions to establish a stable baseline. The temperature range of analysis for the DSC cell is -80 to 555 C. You can choose to start your experiment from one of the following choices:

- 1st. Current Temperature—the temperature of the DSC cell at the current moment (usually ambient temperature, 30 to 35°C). In this case, the ramped heating begins immediately on starting the experiment
- 2nd. Another Temperature—you choose the specific temperature desired (default is 50°C). In this case, the system equilibrates at the chosen temperature before ramped heating begins
- ii. Ramp (Heating) Rate: The acceptable range of heating/cooling rates for conventional DSC is 0.01 to 100°C/minute. This range depends on a number of variables including the temperature range covered and the presence of a cooling accessory. Faster heating/cooling rates generally increase sensitivity, particularly for thermal events such as the glass transition. They also obviously shorten the time of analysis. Slower heating/cooling rates generally provide better resolution (separation of closely spaced thermal events). Heating and cooling rates of 5 to 10°C/minute are a good starting point for most materials
- iii. Additional Conditions: There are additional experimental conditions that can be changed to optimize specific situations. These are accessed by pressing on the 'Advanced Parameters' and 'Post-Test Conditions' buttons. For most experiments, the system default values shown are recommended
- b. 'DSC Heat/Cool/Heat' experiments are designed to erase previous thermal history by heating the material above a transition (e.g., glass transition or melting), where relaxation or molecular rearrangement can occur, then cooling at a known rate before heating again (figure 4b). The first heating curve provides the "as received" information. The cooling imparts a known thermal history. Therefore, any differences observed between similar materials in the second heating curve are related to real internal differences in the materials (e.g., molecular weight) rather than previous thermal history effects



i. Start Temperature: The actual starting, and upper temperatures chosen for a specific experiment must be within the usable temperature of the DSC cell/cooling accessor, i.e., -80 to 555 C. These temperatures should bracket the region where sample transitions are expected to occur, and they should be

4b
1
<b>10</b>

previous themal history. The heating again. The known the evaluated during the subsqu compared directly. The temp heat/cool/heat experiments accessory and heating/cool more details.	en the material is cooled a hermal history imparted du uent reheating) allows sim perature range, which car , depends on the type of ling rate selected. Press t	at a known rate before uring cooling (and ilar materials to be h be used for pans, cooling he HELP button for
Enter the desired parameter	5:	
Start temperature	✓ Use current 35.00	ີີ
Heating rate:	10.000	°C/min
Upper temperature:	250.00	°C
Cooling rate:	5.000	− °C/min
	Labore	-

far enough below or above the transitions to establish a stable baseline. You can choose to start your experiment from one of the following choices:

- 1st. Current Temperature-the temperature of the DSC cell at the current moment (usually ambient temperature, 35°C). In this case, the ramped heating begins immediately on starting the experiment
- 2nd. Another Temperature—you choose the specific temperature desired (default is 50°C). In this case, the system equilibrates at the chosen temperature before ramped heating begins
- Upper Temperature: To obtain the intended benefits from a heat/cool/heat experiment, the upper ii. temperature chosen should be higher than the transition that "erases" previous thermal history. This is typically 10 to 15°C above the glass transition or melting peak. However, the upper temperature should also be low enough to prevent the onset of decomposition
- iii. Lower Temperature: The lowest temperature achieved during the cooling portion of the experiment should be below any transitions of interest
- iv. Heating Rate & Cooling Rate: The acceptable range of heating/cooling rates for conventional DSC is 0.01 to 100°C/minute. This range depends on a number of variables including the temperature range covered and the presence of a cooling accessory. Faster heating/cooling rates generally increase sensitivity particularly for thermal events such as the glass transition. They also obviously shorten the time of analysis. Slower heating/cooling rates generally provide better resolution (separation of closely spaced thermal events). Heating and cooling rates of 5 to 10°C/minute are a good starting point for most materials
- Additional Conditions: There are additional experimental conditions that can be changed to optimize ٧. specific situations. These are accessed by pressing on the 'Advanced Parameters' and 'Post-Test Conditions' buttons. For most experiments, the system default values shown are recommended



c. 'DSC Cyclic' experiments are used to assess changes in the material as it is exposed to a series of heating/cooling cycles (figure 4c). Multiphase materials (e.g., emulsifiers) often change internally (become less stable) during thermal cycling

4c	

This experiment is designed to heat and cool a material multiple times (cycles) between an upper and lower temperature. These repetitive temperature cycles provide information about the stability of multiphase materials. The temperature range which can be used for cyclic experiments depends on the type of pans, cooling acessory, and ramp rate selected. Press HELP for more details

	Enter the desired parameters	B:	
	Start temperature	-80.00	°C
	Ramp rate:	20.000	°C/min
0.2	Final temperature:	400.00	r
	Number of cycles	6	
	Advanced Paramete	rs P	ost-Test Conditions

i. Start Temperature: The actual starting, and upper temperatures chosen for a

> specific experiment must be within the usable temperature of the DSC cell/cooling accessor, i.e., -80 to 555 C. These temperatures should bracket the region where sample transitions are expected to occur, and they should be far enough below or above the transitions to establish a stable baseline. When selecting the final temperature, you need to consider the sample that will be analyzed and the scope of your experiment.

- ii. Heat/Cooling (Ramp) Rate: The acceptable range of ramp rates for conventional DSC is 0.01 to 100°C/minute. This range depends on a number of variables, including the temperature range covered and the presence of a cooling accessory. Faster ramp rates generally increase sensitivity particularly for thermal events such as the glass transition. They also obviously shorten the time of analysis. Slower ramp rates generally provide better resolution (separation of closely spaced thermal events). The heating and cooling in cyclic experiments are usually the same (3 to 5°C/minute)
- iii. Number of Cycles: The number of cycles required to assess stability or reversible transitions, such as melting and recrystallization, varies based on material. Several cycles (3 to 5) usually provide a good indication of stability or ensure that no changes occur in the events observed
- Additional Conditions: There are additional experimental conditions that can be changed to optimize iv. specific situations. These are accessed by pressing on the 'Advanced Parameters' and 'Post-Test

Conditions' buttons. For most experiments, the system default values shown are recommended

d. 'DSC Isothermal' experiments provide an indication of a material's stability at elevated temperature (figure 4d). The purge gas, which surrounds the material, remains



This experiment is designed to ra temperature and then hold at th- or until a specific event (e.g., ox changing purge gas at the isoth mass flow cooling accessory is i stopping the experiment when th available. Press the HELP butto	apidly heat the ma at temperature for idation) occurs. Ti ermal temperature nstalled. The optione desired heat flo n for more details.	terial to an elevated a specific amount of time ne option of automatically is available When a in of automatically w event occurs is also
Enter the desired parameters:		
Start temperature	200.00	°C
Switch to Gas 2		
Isothermal time:	100.00	min
End experiment when	mW > 0.5	50

Advanced Parameters



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Post-Test Conditions

constant or is switched at the elevated temperature to generate a sample-atmosphere interaction (e.g., oxidative stability)

- i. Isothermal (Start) Temperature: The acceptable range for the equilibration temperature in an isothermal experiment is cooler dependent. Hypothetically only isothermal temperatures within the operating range of the selected cooler can be used, i.e., -80 to 550 C. However, prolonged experiments near the upper temperature limit for a specific cooler could affect the lifetime of the unit. CAUTION: We recommend that you do not use the RCS when running isothermal experiments above 400°C. Damage to the unit can occur if used at high temperatures for extended periods
- ii. (Optional) Switch to Gas 2 (requires a Mass Flow Controller): The option of switching gas after achieving the equilibration temperature provides the ability to measure material characteristics that are based on not only temperature, but also on the atmosphere around the material, e.g., stability in oxygen (resistance to oxidative degradation)
- iii. Isothermal Time: The isothermal time should be set to a value that is greater than required to measure the event of interest. If the time required is unknown, a large value should be entered.
- iv. End Experiment When: No analysis time will be wasted provided a proper "End Experiment" condition is set. DSC isothermal experiments are usually stopped based on detecting heat flows (mW/minute) greater than a "threshold" value. This threshold should be large enough to ensure that a real event is occurring and that the extrapolated onset can be determined. 0.5 mW/minute is a good initial choice. Check the End Experiment When box and select the desired setting to use this option
- Additional Conditions: There are additional experimental conditions that can be changed to optimize ٧. specific situations. These are accessed using Advanced Parameters and Post-Test Conditions. For most experiments, the system default values shown are recommended
- 5. Press on the 'Next' button and check the overview report to ensure that all parameters entered are correct
- 6. Press on the 'Next' button and provide the sample specific information (figure 6)
  - a. 'Sample Name' is the name of your sample (not the file name)
  - b. 'Sample Size' is the mass of your sample in mg. The recorded signal (mW) will be normalized against the mass of your sample and signal will be reported in mW/mg units

6 mer	<ul> <li>Sample Informati</li> <li>Sample Name</li> <li>Sample Size</li> <li>Comments</li> </ul>	on Sapphire 2.918 mg Pan No. 25 📩 Ref. 0 🛨	]
	Data File	\\DSC-B172\Data\IMSERC\Sapphire.001	
Koy	−Auto Analysis — □ <u>A</u> utoanalyze		
	Analysis Macro		~

c. 'Pan No' is the position number of your sample in the autosampler tray



- d. 'Ref' is the reference position of the empty crucible in the autosampler tray. Leave this number as '0' since an empty Aluminum crucible is permanently mounted on the reference position in the cell
- e. 'Comments' is optional where you can provide additional information about your sample
- 'Data File' is the path of where your data will be saved. You should save under your PI's folder located in f. '\\DSC-B172\Data\'
- g. 'Auto Analysis' leave it unselected
- 7. Press on the 'Next' button and provide any additional notes (figure 7)
  - a. 'Pan Type' currently available is 'Tzero Aluminum'
  - b. 'Mass Flow Control Settings' are set to '#1 -Nitrogen' at 50 mL/min 'Flow Rate'
- 8. Press on the 'Next' button, and select 'Append Run'

TED	Operator Me	
	Pan Type Tzero Aluminum	•
	Extended Text	^
		~
	Mass Flow Control Settings	
100	Sample #1 - Nitrogen 💌 Flow F	Rate 50 mL/min

- 9. In no additional edits to the procedure are needed, keep appending more runs in case you have multiple samples
- 10. If you need to modify or save (highly recommended) the parameters you provided, see section 'C. Modify a procedure'
- 11. Start your measurement by following the information under the section 'D. Start a measurement'

### B. CREATE A PROCEDURE USING THE EXPERIMENTAL VIEW PAGE

This method is recommended when you have some basic experience with the software, and you like to create a procedure from the pre-programmed templates:

- 1. Press on the 'Procedure' tab in the panel located in the middle of the main window (figure 1)
- 2. Select the desired experiment from the list of available pre-programmed templates listed under 'Test' (figure 2). Most of the options in the list are the same with the corresponding option available under the 'Experimental Wizard'. See section 'A. Create a procedure using the Experimental Wizard' for details

	t/Cool/Heat		<b>.</b> 2
Description Mate prev agai	rial is heated at a linea ious thermal history, the n.	r rate to an elevated ter en cooled at a linear rate	mperature to eras e before heating
1ethod			
Start temperature	Vse current		Advanced.
	35.00	°C	Post Test
Heating rate	35.00	°C °C/min	Post Test
Heating rate Jpper temperature	35.00 10.000 250.00	°C °C/min °C	Post Test



- 3. Enter the desired parameters. When you have finished setting up this procedure, you can enter more information by selecting the 'Notes' and 'Summary' tabs for this run
- 4. Press on the 'Apply' button when finished to save these changes or the 'Append' button to save the changes to this run and add another run to the end of the sequence
- 5. Start your measurement by following the information under the section 'D. Start a measurement'

#### **C. MODIFY A PROCEDURE**

This method is recommended when you have some basic experience with the software, and you would like to edit a procedure.

- 1. Press on the 'Procedure' tab in the panel located in the middle of the main window (figure 1)
- 2. Under the 'Test' drop down menu, select 'Custom'
- 3. If the procedure you would like to modify is not loaded, press on the 'Load' icon (figure 3)
- 4. Modify the desired parameters listed under each segment in the 'Method' panel by doublepressing on the values you need to edit
- 5. In case you need to add more segments, press on the 'Editor' button to access all commands
- 6. When done with all segments, press on the 'Apply'

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ethod ame	l  Heat/Cool/Heat		Editor
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ethod ame # 1 2 3	Heat/Cool/Heat Segment Description <sup>↓~</sup> Ramp 10.000 °C/min to 250.00 °C ✓ Mark end of cycle 0 <sup>↓~</sup> Ramp 5.000 °C/min to -90.00 °C	^	Editor Advanced Post Test.
ethoc ame # 1 2 3 4	Heat/Cool/Heat Segment Description <sup>↓</sup> Ramp 10.000 °C/min to 250.00 °C ✓ Mark end of cycle 0 <sup>↓</sup> Ramp 5.000 °C/min to -90.00 °C ✓ Mark end of cycle 0	^	Editor Advanced Post Test.

- 7. Save your procedure by pressing on the 'Save' icon (figure 7). Saving a procedure is highly recommended, especially if you plan on reusing the temperature profile for other samples
- 8. Start your measurement by following the information under the section 'D. Start a measurement'





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#### **D. START A MEASUREMENT**

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Once the sample measurement procedure is set, you can start the measurement(s). You can append, delete, or pause runs listed in the queue while the instrument is measuring other samples. In case of an error/warning message, please check the 'Troubleshooting' section.

- Ensure that nitrogen gas flow is on by checking the 'Sample Purge Flow' value on the signal's table at the bottom right corner of the main window (figure 1). Flow should be at around 50 mL/min when the gas is on and at 0 mL/min when the flow is off. To turn the flow on, use the 'dsc\_gas\_valve' script:
  - a. Look at the windows taskbar to see if the script is running (circled in figure 1a). If script is not running, double press on the 'dsc\_gas\_valve' icon on the desktop
  - Press anywhere on the large red area around the 'OFF' label and wait for a couple of seconds (figure 1b)
  - c. You should see a green colored window now with the label 'ON' (figure 1c) and the flow should read around 50 mL/min. If window is green (ON) and flow is still zero, please see a staff
  - d. Minimize the 'dsc\_gas\_valve'. Closing the script will leave the valve still open
- 2. Start the cooling device by going to the 'Control' menu, and selecting 'Event', and 'On'
- 3. Wait for around 5 minutes until the 'Flange Temperature' value goes below -60 C (figure 3)
- Start the measurement by pressing on the 'Run' green button at the top right corner of the main window.
   Alternatively, under the 'Control' menu, press on 'Start'
- 5. Measurement will start after a few seconds, and you'll be able to see some data plotted on the graph of the main window
- At the end of the measurement, sample will be returned to the original numbered position in the autosampler.
   The cooling device will turn automatically off when the last sample in the queue is measured
- 7. With the cooling device off, after a few minutes, the 'Flange Temperature' will be near room temperature. Select the 'dsc\_gas\_valve' script and turn off the gas flow. Turning off the gas while the device is still cold (flange temperature below 10 C) might create icing problems in the cooling device and potentially an instrument damage. *Please turn off the gas flow only when the flange temperature is near room temperature*



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### **PUBLICATION**

#### A. EXPERIMENTAL SECTION

#### Modify the text below according to the setup and conditions you used during the measurement:

"Differential Scanning Calorimetry (DSC) measurements were performed with a TA Q200 instrument. XX mg of sample YY were placed in an aluminum crucible that was cold sealed with a press. Sample was measured under ultra-high purity nitrogen gas (50 ml/min) and an empty aluminum was used as a reference. Baseline signal, collected with no crucibles loaded in the cell, was subtracted automatically from the raw data. Temperature was increased at a rate of XX C/min from X to Y."

#### **B. ACKNOWLEDGEMENT**

"This work made use of the IMSERC X- facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-2025633), and Northwestern University."





### TROUBLESHOOTING

#### A. THE COMPUTER SCREEN WILL NOT TURN ON

Begin your reservation in NUcore to initiate access to the instrument

#### **B. COMPUTER REQUIRES LOGIN AND A PASSWORD**

The default 'DSC' user account should be logged in. In case the computer was restarted, the password for the 'DSC' account is . See 'Default instrument status' section for more details

#### C. ACQUISITION SOFTWARE REQUIRES LOGIN AND A PASSWORD

Please contact a staff member if the acquisition software requires login credentials

#### **D. GAS FLOW RELATED ERROR**

In case you receive the following error message 'Sample purge gas can not reach specified flow rate. (656)', ensure that the gas valve is turned on and the 'Sample Purge Flow' is around 50 mL/min (figure D). If gas flow is at 0 mL/min, follow step #1 under the section 'D. Start a measurement'

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	39.96 °C
Heat Flow	0.138 mW
Set Point Temp	40.00 °C
Heater Power	38.130 W
Flange Temperature	-62.49 °C
Heater Temperature	39.55 °C
Sample Purge Flow	49.99 mL/min

#### E. COOLING CAPACITY RELATED WARNING

In case you receive the following warning message 'A cooling ramp specified may exceed cooling capacity. Run continuing. (724)', your measurement will continue and there is no action required. This message is due to the target temperature being lower than the base temperature of the instrument which is represented as 'Flange Temperature' (see table in figure D). Most likely, the lowest temperature that will be recorded in your data file will be near the 'Flange Temperature' rather than the nominal temperature listed in your temperature profile.

### F. THERE IS AN ERROR OR PROBLEM WITH THE INSTRUMENT NOT ADDRESSED UNDER THIS **TROUBLESHOOTING SECTION**

If there is an error or problem with the instrument which is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue





- 2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html add place the 'Stop' sign near the instrument computer. 'Stop' signs are located on the shelf above the computers in BG51 and online at the link above. Email or talk to a staff member
- 3. Email or talk to a staff member





REVISIONS		
v1.07 2021/09/22	٠	Troubleshooting section updated
	•	Training video files transferred from Box to OneDrive
v1.05	٠	Information about using the new software control of the nitrogen valve was added
2021/08/08	•	More pictures related to sample prep and mounting were added
v1.02 2021/06/30	٠	Release of original version of the user manual for the acquisition software Advantage 5.5.24

