IMSERC User Manual for TA DSC2500

CONTENTS

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/about-policies.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-faq.html#data for details about data access.

SOFTWARE

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

- For registered IMSERC users, the 'Trios' software can be downloaded from 'imsercdata.northwestern.edu' under the folder 'public/TA'. Software is available for Windows only. Please follow instructions under 'Data Access' at http://imserc.northwestern.edu/about-general-faq.html#data on how to connect to the 'public' folder
- Remotely via NUWorkspace which is operating system independent. After logging in with your netID credentials, please launch the 'IMSERC' workspace which contains the thermal analysis software
- 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 -90 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
2. The default 'DSC' user account should be logged in. In case the computer was restarted, the password for the 'DSC' account is (see hardcopy by the instrument)
3. Acquisition software (Trios) 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

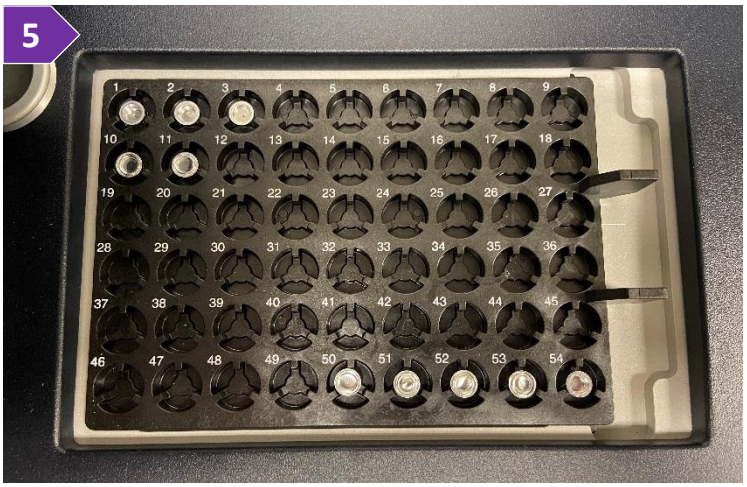
PREPARING AND LOADING A SAMPLE INTO THE INSTRUMENT

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 (circled in figure 1) located in the middle drawer in the cabinet under the DSC instrument. Always use tweezers to handle the pan and lid as contact with skin my affect the DSC measurement

2. Weigh your sample by using the microbalance. Watch the 'microbalance' video for a visual demonstration on 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
3. Remove the pan with the sample from the microbalance, place them into the blue stab of the press, and seal your sample. Watch the 'dsc-sealing-al-crucible' 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. Load your sealed crucible into the autosampler tray and note the number of the sample position (figure 5). Sample positions from 46 to 54 are used for standards and you should not use those positions for samples



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 a few ways for creating a new measurement procedure:

- Creating from scratch a new procedure, or
- Loading an existing measurement file and copying the procedure, or
- Loading a template and modifying it accordingly

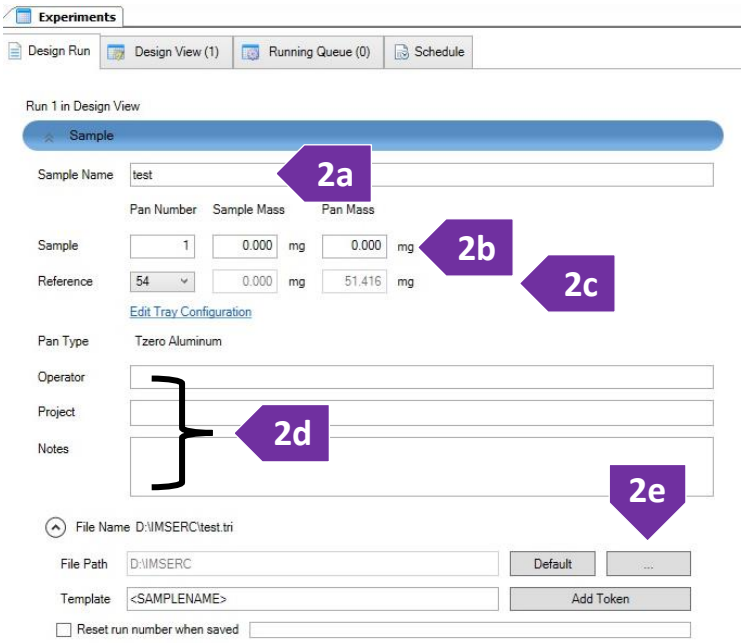
Please read the detailed instructions below based on the measurement type of your choice.

A. CREATE A NEW PROCEDURE STEP-BY-STEP

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. Press on the 'Experiments' tab on the bottom left of the file manager portion of the 'Trios' software and select 'Create New Runs' under the 'Design View' (figure 1). Here we will be able to build a procedure, and ultimately add it to the 'Running Queue'
2. To set the main configuration parameters of your measurement, on the 'Design Run/View' tab of the 'Experiments' window, you need to provide (figure 2):
   a. 'Sample Name' is the name you would like to provide for this specific sample (figure 2a)
   b. 'Pan Number' is the position in the autosampler that this pan has been placed (figure 2b). Note that pan and empty crucible must be pre-tared using the software. See the 'Loading a sample into the instrument' section for more details

c. 'Reference' must be selected and accurately represent the type of crucible + lid combination that in on the autosampler (figure 2c). Please refer to the 'References.txt' file under the 'Procedures' folder for the current assignment of reference crucibles loaded on the autosampler. Select the appropriate 'Pan Number' from the drop-down menu



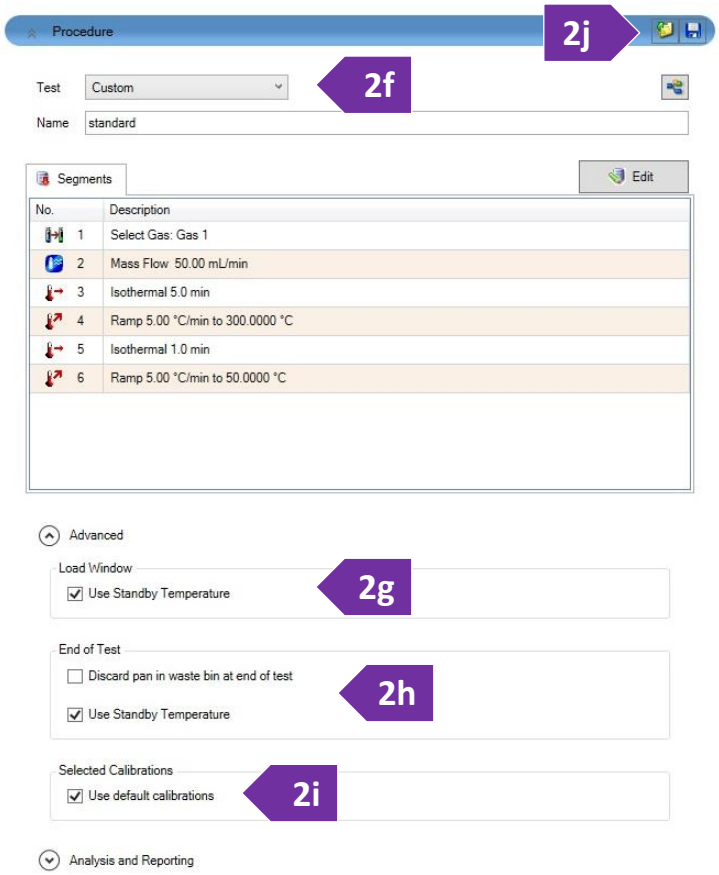
d. (optional) Provide your name (as the operator of this measurement) a project title, or notes. These will all be carried over in the final collection file but are not necessary to start a measurement (figure 2d)

e. 'File Path' is the folder that you would like to save your data. Press the three dots next to the file path field, find your PI's folder on the D drive, and then your own folder (example D:\PI'sLastName\YourName) (figure 2e) Leave the template to '<SAMPLENAME>' this will make the filename the same as you have described at the sample name. Leave the 'Reset run number when saved' unchecked.

f. 'Test' field under the Procedure tab is where we can select from a set of pre-made procedures or create your own. The preset procedures include 'Heat and Hold', 'Ramp', and 'stepwise Isothermal'. It is recommended to either make your own custom procedure or start with a template and then configure it to your needs. Check the 'Procedures' folder for examples of templates.

- To start with a pre-made procedure, simply select whichever pre-made procedure you would like, click on the segments tab, and then change the test type to 'custom'. This will allow a box that says 'Edit' to appear in the upper right-hand corner of the 'segments' field, and then add/take away segments that you do/do not want (figure 2f)
- It is highly recommended to include segments that explicitly define the type of gas and the mass flow of the gas since the user before you might have used completely different gas environments. Add an isothermal step after setting gases to allow for the system to stabilize. Ensure that the right type of gas is connected to the line used by your procedure by following the instructions at the 'Change type of gas' section

- A list of all the available 'Method Segments' is listed in Appendix A. When done with edits, press on the 'Apply' button. Additionally, under the 'Procedures' folders, you'll find a list of available templates for various procedures



g. Under the 'Advanced' header under the 'Procedure' tab, the box labeled 'Start Experiment After Weight Stabilization' should be checked. This will endure that the weight of the sample has stabilized before the measurement starts (figure 2g). In case your sample is sensitive and for example absorbs or releases moisture over time, you might want to uncheck the 'Start Experiment After Weight Stabilization' option

h. 'End of Test' header shows a box labeled 'Use Standby Temperature' which should be checked (figure 2h). If you do not need your measured crucible + sample back, check the 'Discard pan in waste bin at the end of test' option and the system will put your sample in the built-in waste bin

i. 'Selected Calibrations' shows a checkbox that states 'Use Default Calibrations'. This option should also be checked, and will use the calibrations that have been saved to the instrument (figure 2i)

j. (optional) Once all the parameters have been selected, you have the option to save the procedure, by clicking on the 'Save Procedure' Icon (figure 2j). Procedure is also permanently saved in your file which you could reload for future measurements

k. Next is to add the run(s) to the running Queue. Right click the run(s) you would like to queue for collection. Do this by selecting all the run(s) of interest, right clicking, and selecting 'Copy to the Running Queue'. You will now see the list of run(s) that you created in the design view, in the 'Running Queue'

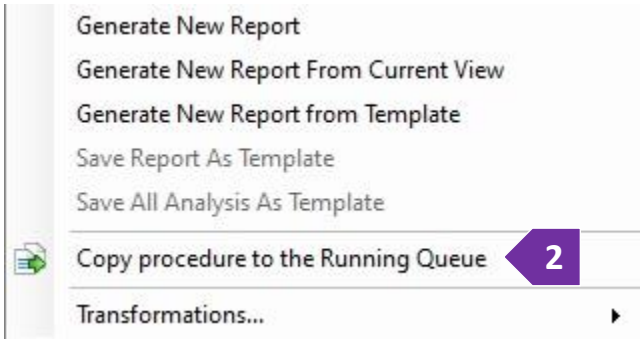
3. Repeat step #2 for each sample by pressing on the 'Append' icon or selecting the append option after right pressing on the 'Design View' or 'Running Queue' area

4. Once all the runs have been added to the running queue, press the green Start/Play button at the top of the experiment window of 'Trios'. All the runs in the 'Running Queue' tab will be collected in the order that they appear in the list. While the instrument/measurements are running, you could append new runs
5. Start your measurement by following the information under the section 'D. Start a measurement'

B. REPEAT A DSC MEASUREMENT USING AN EXISTING PROCEDURE

To either repeat or load an existing procedure follow the steps below:

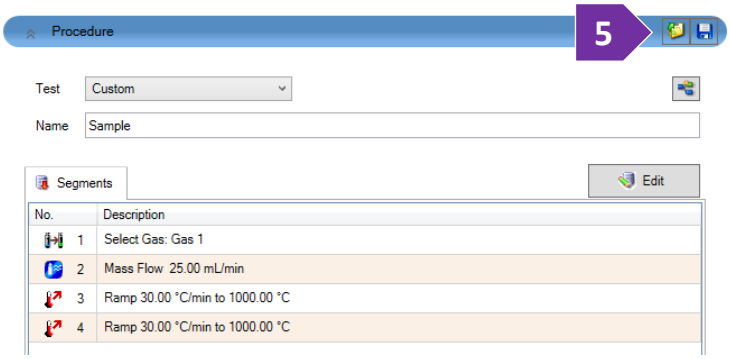
1. Double press on the TRI file that contains the procedure you'd like to reuse. File will be loaded on 'Trios' and data will be shown under the 'Results' tab
2. Right press on the sample name on the lefthanded side and select 'Copy procedure to the Running Queue' that is located towards the end of the menu (figure 2)



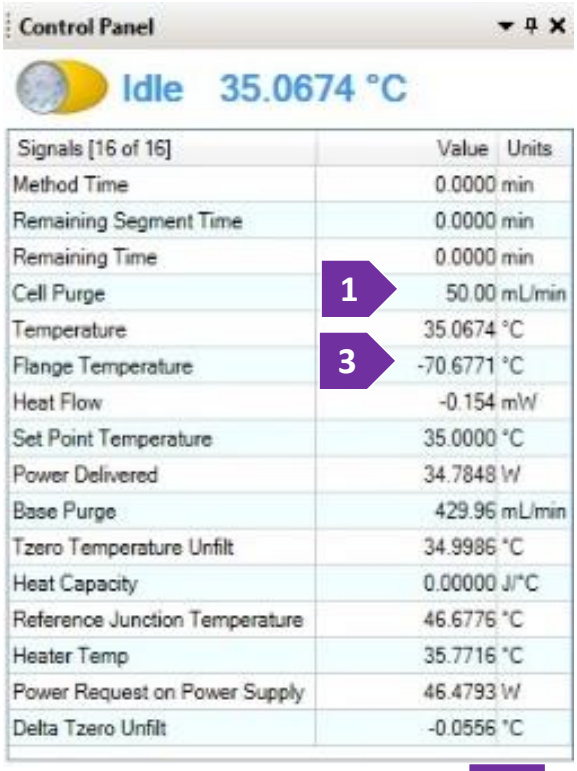
3. A new run is created using the existing procedure. Ensure that you have provided a new file name for the new run

Alternatively, you could:

4. Create a new blank run by adding/appending a new run in the queue
5. Load an exported procedure by pressing on the 'Load' button on the 'Procedure' ribbon (figure 5)

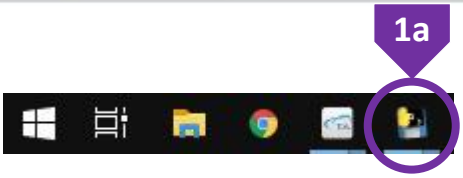


C. START A MEASUREMENT

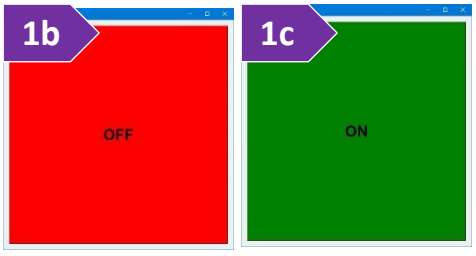


Once the sample measurement procedure is set, you can start the measurement(s). You can append, delete, or pause runs listed in the queue while the instrument is measuring other samples. In case of an error/warning message, please check the 'Troubleshooting' section.

1. Ensure that nitrogen gas flow is on by checking the 'Cell Purge' value on the signal's table at the top 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



   b. 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)
4. 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



6. 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***

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 DSC2500 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

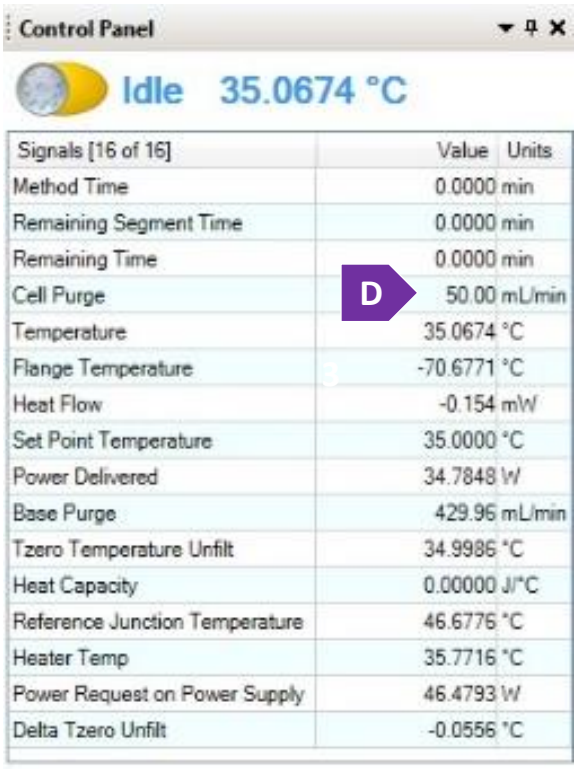
The default 'DSC' user account should be logged in. In case the computer was restarted, the password for the 'DSC' account is . See 'Default instrument status' section for more details

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'



Signals [16 of 16]	Value	Units
Method Time	0.0000	min
Remaining Segment Time	0.0000	min
Remaining Time	0.0000	min
Cell Purge	50.00	mL/min
Temperature	35.0674	°C
Flange Temperature	-70.6771	°C
Heat Flow	-0.154	mW
Set Point Temperature	35.0000	°C
Power Delivered	34.7848	W
Base Purge	429.96	mL/min
Tzero Temperature Unfilt	34.9986	°C
Heat Capacity	0.00000	J/°C
Reference Junction Temperature	46.6776	°C
Heater Temp	35.7716	°C
Power Request on Power Supply	46.4793	W
Delta Tzero Unfilt	-0.0556	°C

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

v1.08 2023/05/30	• Release of original version of the user manual for the acquisition software Trios 5.7.0.56