

DSC1
Differential Scanning Calorimetry:
TA Instrument Help (partial)

December 4, 2023

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1 Create Runs

Overview of Creating Runs and Run Sequences

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Overview

Each Discovery experiment, or "run," consists of a set of user-defined **Sample** and **Procedure** experimental parameters. The **Sample** parameters include info such as sample name, pan type, etc. The **Procedure** parameters include the mode, the custom method or predefined template, and other experimental information. For instructions on defining these parameters, refer to the Defining Experimental Parameters Help topic applicable to your instrument.

Often, more than one run is performed in thermal analysis, especially when an Autosampler is present on the instrument. This collection of multiple runs is known as a "run sequence". Individual runs and run sequences are created in either the **Design View** or **Running Queue** (see the [Sequence View Types](#) section below for information). Both of these views contain the same basic tools for creating, modifying, and saving runs. However, runs are only executed from the Running Queue. Once an experiment completes, the experimental data is copied to the user's designated directory and the experimental information is recorded in the instrument [History View](#). If any experiment faults due to an error at the start of the test, the run is moved to the **Incomplete** container.



Single Run Form vs. Table View

To view and configure runs, access the "Single Run" form located in the **Design Run** view shown above or **Queued Run** view, depending on the selected run in the File Manager **Experiment** list. Alternatively, access the "Table View" in the **Design View** experimental window shown below or the **Running Queue** window. Both of these views display a limited number of experimental parameters.

- The Single Run form provides complete access to all the experimental parameters associated with the run and is used to create a full Procedure. See [Creating Run Sequences](#) for more information.
- The Table View provides a summary of all the runs in a sequence, allowing for a quick review of key parameters and for copying of run information from one experiment to another. In this view, the experimental information can be arranged in a spreadsheet-style table view, with each row representing a single run. Columns containing the specific experimental information are customizable.

See [Using Table View](#) for more information.

| Sample Name | Pan Number | Sample Mass | Pan Mass | Reference Pan | Reference Sample Mass | Reference Pan Mass | Pan Type | Operator | Project | Comments | File Path | Test |
|-------------|------------|-------------|----------|---------------|-----------------------|--------------------|----------------|----------------|---------|----------|--|------------|
| Sample A | 1 | 10.000 | 54.000 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample A.tn | Isothermal |
| Sample B | 2 | 5.000 | 53.500 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample B.tn | Isothermal |
| Sample C | 3 | 1.900 | 55.000 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample C.tn | Isothermal |
| Sample D | 4 | 7.300 | 54.250 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample D.tn | Isothermal |
| Sample E | 5 | 8.300 | 53.650 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample E.tn | Isothermal |
| Sample F | 6 | 2.750 | 54.500 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample F.tn | Isothermal |
| Sample G | 7 | 4.800 | 54.100 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample G.tn | Isothermal |
| Sample H | 8 | 3.450 | 55.100 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample H.tn | Isothermal |
| Sample I | 9 | 2.860 | 52.160 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample I.tn | Isothermal |
| Sample J | 10 | 4.950 | 54.600 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample J.tn | Isothermal |
| Sample K | 11 | 6.400 | 56.000 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample K.tn | Isothermal |
| Sample L | 12 | 3.300 | 52.500 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample L.tn | Isothermal |
| Sample M | 13 | 9.100 | 53.780 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample M.tn | Isothermal |
| Sample N | 14 | 5.750 | 52.600 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample N.tn | Isothermal |
| Sample O | 15 | 6.770 | 54.320 | 37 | 0.000 | 45.050 | Tzero Aluminum | TA Instruments | TRIOS | DSC2500 | C:\ProgramData\TA Instruments\TRIOS\Data\Sample O.tn | Isothermal |

Sequence View Types

The following sections describe the different ways to access the sequence views. These sequence views are selectable from the File Manager **Experiment** panel.

Design View

Design View (0)

The **Design View** contains the tools needed to create sequences of runs. The Design View tools are available both online (when connected to an instrument, performing a run, etc.) and offline (without any connection to the instrument needed). Each individual Design View can contain a series of runs (a sequence) defined by the user. The user can create as many different Design Views sequences as desired.

The Design View sequences are not executed directly from the Design View window. Once a series of runs is arranged into a sequence, copy the sequence over to the Running Queue for execution (from within the File Manager **Experiment** view, drag the runs from the Design View to the Running Queue), or save as a **Sequence file** for future recall. Or, since [multiple Design Views](#) can be open at the same time, your sequence can simply remain in the Design View until you are ready to use or edit it. Note that sequences in the Design View are never deleted unless manually done so by the user.

Running Queue

Running Queue

The **Running Queue** is the programmed queue of experiments that are run when the **Start** button is clicked. The Running Queue is the only sequence view from which you can actually execute experiments. The run at the top of the queue is always the next run to be executed. When the run completes, it is deleted from the queue, a data file for that run is created, and an entry containing all the run information is created in the [History View](#). Each run then moves up in the queue and the next run is executed, and so on. This process continues until the Running Queue is depleted.

Experiments can be set up in the Running Queue with all the same functionality as a Design View, but it is important to keep in mind that when the sequence completes, the queue is empty (as all runs in the sequence are deleted upon execution). To avoid the accidental deletion of a sequence, it is recommended that sequences be created in the Design View and then exported to the Running Queue for execution. Note that sequences in the Design View are never deleted unless manually done so by the user.


Incomplete

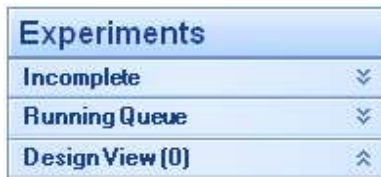
Incomplete

The **Incomplete** view collects unfinished runs; a run may fail to complete, for example, as a result of the purge gas running out or some other external issue. In this case, the suspect run moves from the Running Queue into the Incomplete view, and the rest of the run sequence continues. The operator can then perform the desired action for these Incomplete runs (either delete the run or send it back to the Running Queue).

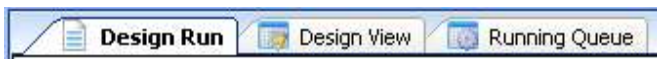
Switching Sequence Views

Switch between the various sequence view types by performing either of the two options described below.

- Using the File Manager **Experiments** panel: Select the arrow  to display the run list for the selected view.



- By selecting the experimental window tab: Select the desired document by clicking on the appropriate tab in the experiment window.



The image below shows the various sequence view types and experimental windows as they appear in TRIOS software. The experimental parameters that appear in the experiment window depend upon the run selected in the File Manager **Experiments** panel on the left. Therefore, using the image below as an example, clicking **Run 1** or **Run 2** within the **Design View** in the File Manager displays the parameters for that respective run in the experimental window. The parameters for each individual run can then be reviewed or edited in Single Run form (**Design Run** experiment window, shown below) or Table View (**Design View** experimental window). However, clicking the **Running Queue** tab from the experiment window enables the **Running Queue** sequence view as the active sequence view in the File Manager and also the Single Run form (Queued Run) associated with the selected run in the Running Queue list. At the top of the Single Run form is text showing the associated run.

File Manager

Experiments

Incomplete Queue (0)

Running Queue (1)

Design View (2)

Run 1 - [Pan 1 - Custom]

Run 2 - [Pan 2 - Custom]

Experiments

History

Results

Calibration

Experiments

Design Run

Design View (2)

Running Queue (1)

Schedule

Run 1 in Design View

Sample

Sample Name

| | Pan Number | Sample Mass | Pan Mass |
|-----------|------------|-------------|----------|
| Sample | 1 | 0.000 mg | 0.000 mg |
| Reference | 0 | 0.000 mg | 0.000 mg |

[Edit Tray Configuration](#)

Pan Type: None

Operator

Project

Notes

File Name: C:\ProgramData\TA Instruments\TRIOS\Data\Default(1).tri

Procedure

Test: Custom

Name

Segments

| No. | Description |
|-----|-------------|
|-----|-------------|

Advanced

Load Window

Use Standby Temperature

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

2 Define Experimental Parameters

Defining Experimental Parameters

In this topic

[Entering Sample Information](#)

[Defining a DSC Procedure](#)

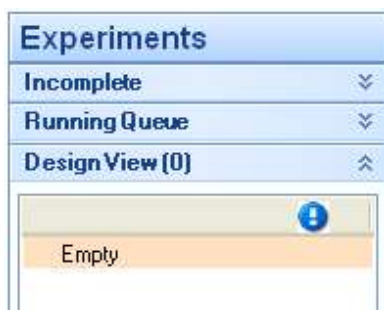
[Saving a Procedure to File](#)

[Opening a Saved Procedure File](#)

Creating DSC experiments requires entering the sample information and defining a procedure. Experiments are created in either the **Design View** or within the **Running Queue**. In either case, the information below applies.

To set up a new run, click **Setup**  from the **Experiment** tab. This automatically enables the **Design Run** experiment window as the active window.

If no runs are currently set up, then **Empty** appears within the File Manager - **Experiments** list (as shown below). Click **Empty** to create a new run. If runs already appear in the list, you can append a new run to the list. See [Creating Sequences](#) for more information.



Follow the instructions below to enter the experiment parameters (**Sample** and **Procedure** information).

Entering Sample Information

The **Sample** window defines the sample parameters. If the sample parameters are not visible, click the **Sample** bar to expand the contents of this selection.

Design Run | Design View (2) | Running Queue (1) | Schedule

Run 1 in Design View

Sample

Sample Name

| | Pan Number | Sample Mass | | Pan Mass | |
|-----------|--------------------------------|------------------------------------|----|------------------------------------|----|
| Sample | <input type="text" value="1"/> | <input type="text" value="0.000"/> | mg | <input type="text" value="0.000"/> | mg |
| Reference | <input type="text" value="0"/> | <input type="text" value="0.000"/> | mg | <input type="text" value="0.000"/> | mg |

[Edit Tray Configuration](#)

Pan Type


Operator


Project

Notes

File Name C:\ProgramData\TA Instruments\TRIOS\Data\Default(1).tri

1. Enter the **Sample Name**.
2. Enter the **Sample** pan number, sample mass, and pan mass.

 NOTE: Setting the sample pan value to **0** (zero) implies manual loading of the pan, and the Autosampler will not attempt to load the pan.

-  NOTES for T4P Heat Flow selection:
- In the Pan Mass fields, enter the weight of the sample pan and reference pan to use the weights for Advanced Tzero calculations. [Pan correction](#) is factored into the calculations.
 - Selecting a Pan Type (other than "none") without inputting a Pan Mass will generate incorrect data.

3. Select the **Reference** pan, which includes the type of pan that will be used in the experiment. Click the down arrow to display a drop-down list of Reference pans, and then select the desired pan for the experiment. A different pan type can be selected for each reference pan number; click **Edit Tray Configuration** to change the pan type and make your selection from the drop-down menu. **Pan Mass** and **Sample Size** can also be set for the desired Reference pan in the Edit Tray Configuration mode (shown in the image below on the right). See [Selecting a Sample Pan](#) for more information.

Sample C mg mg

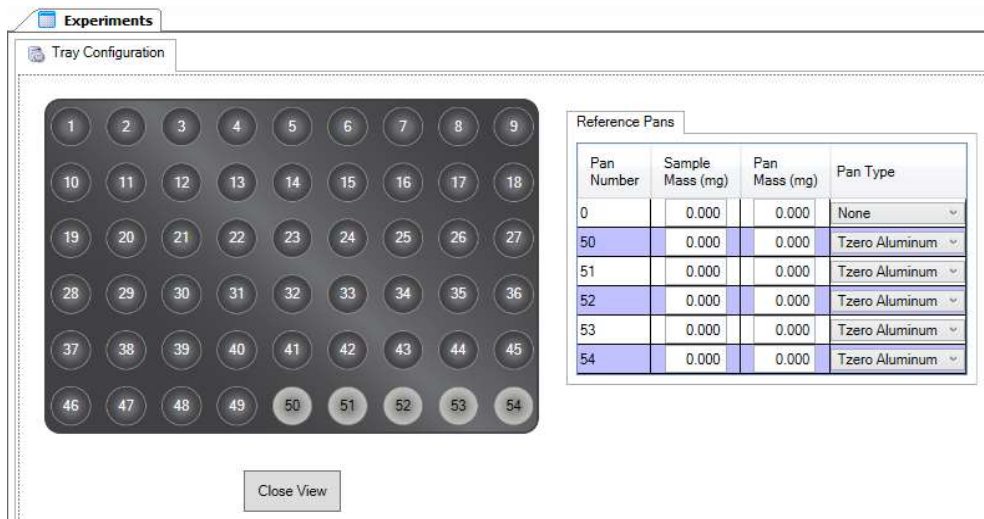
Reference mg

Pan Type

Operator

Project

[Edit Tray Configuration](#)



4. Enter the **Operator** name.
5. Enter the **Project** name, if desired.
6. Enter any comments into the **Notes** field. For example, enter additional text to help describe the experiment, sample treatment, or experimental conditions.
7. Verify the **File Name**. TRIOS is designed to make data file naming easy through the use of tokens. File names are created based on the experimental parameters defined for the test (for example, sample name, procedure, operator, and instrument). TRIOS uses the default file directory and file name template specified in the [TRIOS Options](#). If you wish to override the defaults for this experiment, click the down arrow located to the left of **File Name** to expand the selection and modify the desired parameter(s). The default values are used when either the file path or template are blank.

File Name C:\ProgramData\TA Instruments\TRIOS\Data\Default(1).tri

Path C:\ProgramData\TA Instruments\TRIOS\Data Default ...

Template <SAMPLENAME> Add Token

Reset run number when saved

- Default Path: Use this field to select the file save directory.
- Template: Enter the desired file name or click **Add Token** to set up a file name schema to be used in creation of the file name.
 - File name templates can be specified using any combination of text and tokens.
 - To add a token, click **Add Token**. Select the desired token from the list of available options. Click **Insert** to add to template. Multiple tokens can be used:
 - holder** (specified pan)
 - instrumenttype**
 - instrumentserialnumber**
 - operator, project, procedure, samplename** (as defined for experiment on Sample information parameters)
 - instrumentname** (as defined by operator through instrument setup or user interface)
 - date**
 - runnumber** (internal counter; incremented each run)
- In addition, tokens can be used to specify subdirectories (for example, **samplename**>). If the specified directory doesn't exist it will be created at run start.
- **Reset run number when saved**: Select to have the run number token reset back to one, if used in the file name template.

Defining a DSC 25/250/2500 Procedure

Click the **Procedure** bar to reveal the Procedure parameters. The current procedure displays, as shown below.

Procedure

Test: Custom

Name:

| No. | Description |
|-----|-------------|
|-----|-------------|

Advanced

Load Window

Use Standby Temperature

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

Parameters

An experimental Procedure consists of following parameters:

- **Test:** You can choose to use one of the available pre-preprogrammed test types (templates), or you can choose a custom test and create your own method.
- **Name:** A procedure name used to describe the test (this field is optional).
- **Method / Custom:** A series of one or more segments used to define the experimental conditions of the test. Depending on the **Test** type selected (**Custom** or a [pre-defined template](#)), the method parameters are represented as an [editable custom method](#) or a template, respectively.
- **Advanced Settings:** Defines the specific temperature conditions to be used between experiments.

⬆️ Advanced

Load Window

Use Standby Temperature

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

- **Analysis and Reporting** options: Select to apply an [Analysis template](#) or a [Report template](#) to your results, or to **Send results to a Control Chart** (see [Control Chart](#) for more information).

⬆️ Analysis and Reporting

Analysis Template

Report Template

Send Results to a Control Chart

Basic Steps for Setting Up a Procedure

The basic steps for setting up a Procedure are described below:

1. Select the test (**Custom** in the example above) from the drop-down list. Based on the information selected, the parameters for that test type are displayed. The lists contain both [pre-programmed tests](#) (test templates) and a Custom option. Based on the information selected, the parameters for that test type are displayed. The available mode is **Standard**, which is used to generate the standard (non-modulated) data.
2. Input a procedure **Name**, if desired.
3. Enter the desired method segments. For additional information, refer to [Creating Custom Methods](#).
4. Access the **Advanced** parameters by clicking the arrow next to **Advanced**.
 - **Load Window:** Select to use the current standby temperature or define a specific temperature range to load the pans at the start of the test.
 - **End of Test:**
 - Select the **Discard pan in waste bin at end of text** option to have the Autosampler arm place the pan in the waste bin at the end of the run. If this is not selected, the arm will return the pan to its previous position on the sample tray at the end of the run.
 - Select the **Use Standby Temperature** option to use the current standby temperature; uncheck this option to define a specific temperature range to unload the pans at the end of the test.
5. Select the [Analysis and Reporting](#) options, if desired.
6. If desired, click the **Save** button on the **Procedure** bar to save the procedure for future use.



NOTE: Experimental information can also be imported from the **History View**, the **Incomplete** run view, and from a saved data file.

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Saving a Procedure to File

Clicking **Save** saves the **Procedure** parameters described above. To save the current procedure to file for future recall:

1. Access the Experiment **Procedure** section.
2. Click **Save Procedure**.



3. Locate the desired folder for your method files.
4. Enter the name for the file. The default extension is **.tprc**.
5. Click **Save**.

Opening a Saved Procedure File

To open a saved Procedure file:

1. Access the Experiment **Procedure** section.
2. Click **Load Procedure** .



3. Locate the desired folder containing the method files. Select a file from the list.
4. Click **Open**.

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3 Segments Available

Available Method Segments


A segment is a pre-programmed series of instructions used in a method. Several segments can be linked together to create the desired method. The available segment list varies depending on the instrument and optional accessories installed.

See also:

[Creating a Custom Method](#)

For details on a specific segment, see the table below:

Available Segments

| Segment | Description |
|---|---|
| Abort | <p>The Abort segment skips over the next segment when specified limit conditions are met.</p> <ul style="list-style-type: none"> If the limit is reached at the beginning of a segment, then that segment is skipped and method execution continues with the next segment. If the limit is reached during the execution of a segment, then the remaining portion of the segment is skipped. <p> NOTE: The Abort segment is generally followed by a Ramp or Isothermal segment.</p> <p>Example (DSC):</p> <ol style="list-style-type: none"> Equilibrate at 200°C Abort next segment if mW>1 Isothermal for 100 min |
| Air Cool (Discovery DSC only) | <p>The Air Cool segment controls the internal cooling solenoid valve connected to compressed air (air cool feature).</p> <p>Example: Air Cool: On</p> |
| Data | <p>The Data segment controls data collection during the experiment. If a Data segment is not used, data storage is automatically initiated by the first Ramp, Isothermal, or Step segment that appears in the method.</p> <p>Example: Data Storage: On</p> |
| Equilibrate | <p>The Equilibrate segment heats or cools the furnace to the defined temperature, stabilizes the furnace at that temperature, then continues to the next segment. This segment does not automatically start data collection.</p> <p>Example: Equilibrate at 200°C</p> |
| Event 1 / Event 2 | <p>The Event segment controls the external event relay through the event jack on the back of the instrument. This is used to synchronize control of additional hardware through the method.</p> <p>Example: Event 1: On</p> |
| Heater PID | <p>The Heater PID segment changes the performance of the instrument furnace during the execution of a thermal method. PID stands for Proportional, Integral, and Derivative, the three modes of traditional temperature control. The Heater PID segment specifies the control coefficients for each mode of temperature control. This segment is only maintained during the current method. At the end of the method, the Heater PID values are reset to the default values.</p> <p>Example:</p> |

| | |
|--|--|
| | P= 35 I= 70 D=2 |
| Increment | <p>The Increment segment raises or lowers the temperature in a controlled step, lets the temperature equilibrate, then begins the next segment.</p> <p>Example: Increment by 5°C</p> |
| Initial Temperature (Discovery DSC only) | <p>The Initial Temperature segment heats or cools the furnace to the defined temperature, stabilizes the furnace at that temperature, then holds the temperature until the experiment is continued by clicking OK on the TRIOS dialog box, or by selecting Start on the instrument display or instrument keypad. This segment does not automatically start data collection.</p> <p>Example: Initial Temperature 200°C</p> |
| Isothermal | <p>The Isothermal segment holds the sample at the current temperature (as programmed by the previous segment) for a defined period of time. This segment automatically turns on data collection, except when preceded by a Data OFF segment.</p> <p>Example: Isothermal for 10 min</p> |
| Jump | <p>The Jump segment instantly changes the set point temperature, causing ballistic changes in the sample temperature. This segment then allows the immediate execution of the next segment (which is usually the Isothermal segment). Note that large temperature overshoots may result from the use of this segment. This segment does not automatically start data collection.</p> <p>Example: Jump to 200°C</p> |
| Mark End | <p>The Mark End segment places a marker in the data for use by the data analysis programs. In general, markers provide quick parsing of data to separate experimental segments (i.e., the heat-cool cycle). This segment is available but not necessary for TRIOS.</p> <p>Example: Mark end of cycle 0</p> |
| Mass Flow | <p>The Mass Flow segment alters the rate of flow of the selected gas when an instrument is equipped with a Gas Delivery Module (GDM).</p> <p>Example: Mass Flow 50 mL/min</p> |
| Modulate Temperature | <p><i>Available for Modulated Instruments Only:</i> This segment allows you to enter the modulation temperature amplitude and period (frequency) parameters that will be used with subsequent ramp or isothermal segments. Data collection begins after two modulation cycles.</p> <p>Example: Modulate temperature amplitude 1°C period 60 seconds</p> |
| Ramp | <p>The Ramp segment heats or cools the sample at a fixed rate until it reaches the specified temperature, producing a linear plot of temperature versus time. This segment automatically turns on data collection, except when preceded by a Data OFF segment.</p> <p>Example: Ramp 10°C/min to 200°C</p> |
| Repeat | <p>The Repeat segment does exactly what the name implies: it repeats a group of one or more segments within a method for the number of times specified.</p> <p>Example:</p> <ol style="list-style-type: none"> 1. Ramp 5°C/min to 200°C 2. Ramp 5°C/min to 50°C 3. Repeat segment 1 for 2 times |
| Repeat Until | <p>The Repeat Until segment repeats of group of one or more segments within a method until the specified final temperature is reached or passed.</p> |

| | |
|------------------------|--|
| | <p>Example:</p> <ol style="list-style-type: none"> 1. Equilibrate at 50°C 2. Isothermal for 5 min 3. Increment 10°C 4. Repeat segment 2 until 200°C |
| Sample Interval | <p>The Sample Interval segment allows you to define or change the rate at which data is to be collected (in seconds per point).</p> <p>Example: Sample Interval 2 sec /pt</p> |
| Select Gas | <p>The Select Gas segment controls the switching of gas between Gas 1 and Gas 2 for an instrument with a GDM installed. This segment is used to synchronize gas switching at a specific time or temperature in an experiment.</p> <p>Example: Select Gas 1</p> |
| Shutter | <p><i>Applicable to Discovery DSC PCA accessory only:</i> The Shutter segment controls the PCA UV shutter through the USB port on the back of the DSC using an RS232 to USB interface board.</p> <p>Example: Isothermal for 0.5 min Shutter: Open Isothermal for 2 min Shutter: Closed Isothermal for 0.5 min</p> |
| Step | <p>The Step segment causes the temperature to jump a specified number of degrees at a specified time interval until a final temperature is reached. This segment automatically turns on data collection, except when preceded by a Data OFF segment.</p> <p>Example: Step 5°C for 2 min to 200°C</p> |

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4 Available Procedures

Available Procedures (Tests)

Predefined Test Templates

Select from the following links for more information:


 [Standard: Ramp](#)

 [Modulated: Conventional](#)

 [Standard: Heat/Cool/Heat](#)

 [Modulated: Heat Only](#)

 [Standard: Cyclic](#)

 [Modulated: Quasi Isothermal](#)

 [Standard: Isothermal](#)

Calibration Tests

Select from the following links for more information:

 [Discovery DSC Calibration Overview](#)

 [Calibrating Tzero](#)

 [Calibrating T1 Baseline](#)

 [Calibrating Enthalpy \(Cell Constant\) and Temperature](#)

 [Calibrating Direct Heat Capacity](#)

 [Calibrating Reversing Heat Capacity](#)

See Also:

[Basic Steps Needed to Start a DSC Experiment](#)

[Overview of Creating Runs and Run Sequences](#)

[Defining DSC Experimental Parameters](#)

5 Procedure: Creating a Custom Method

Creating Custom Methods

In this topic

[Overview](#)

[Creating a Custom Method](#)

Overview

A procedure consists of the following parameters:

- Test type
- Method (template or custom)
- Advanced settings

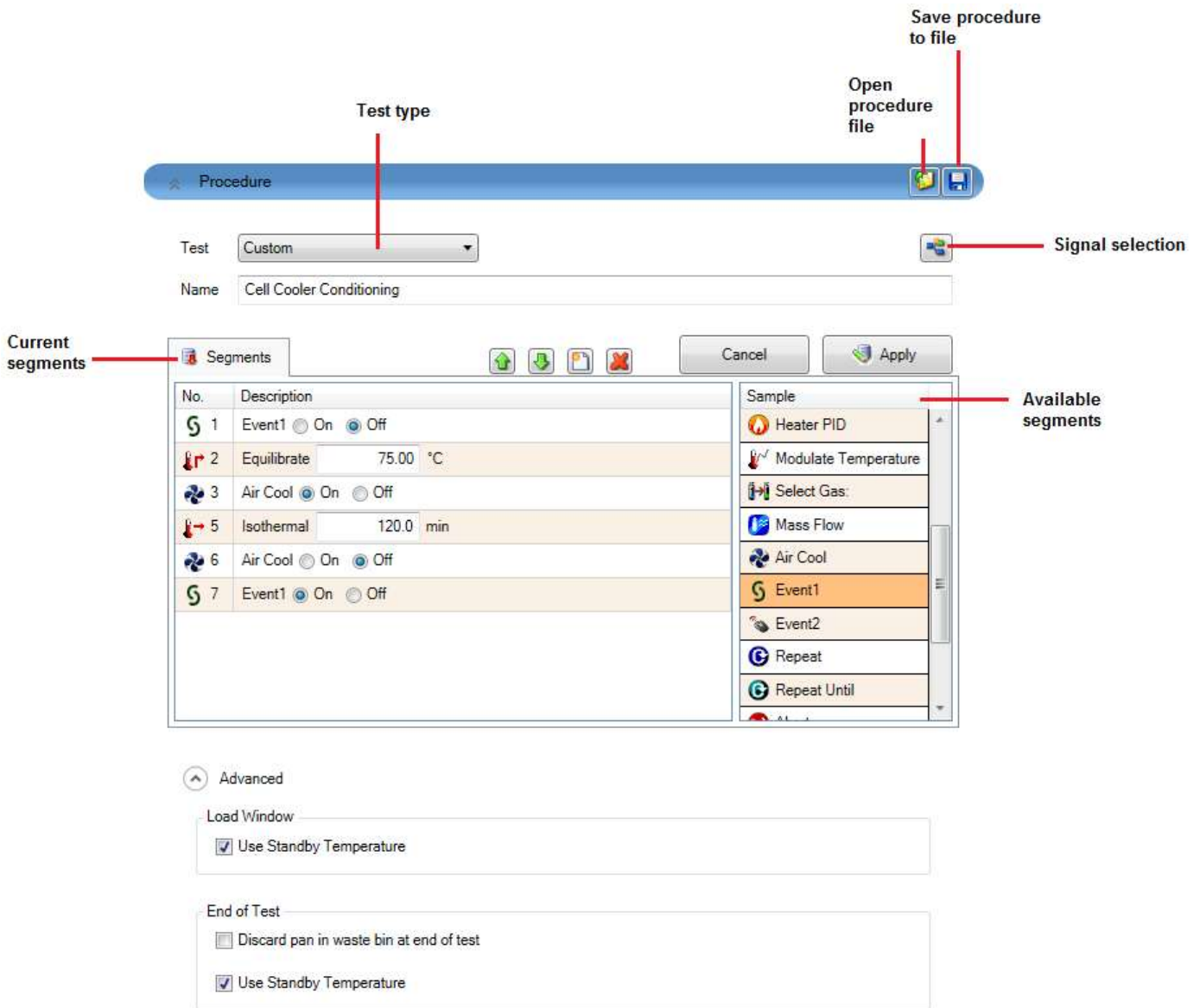
There are two options when creating procedures. You can choose to use one of the available [pre-programmed templates](#), or you can choose a **Custom** test and create your own method using the [available method segments](#). Both options use method segments to program an experiment, but the representation of the segments and their parameters is different.





This help topic describes how to create a custom method.

Creating a Custom Method

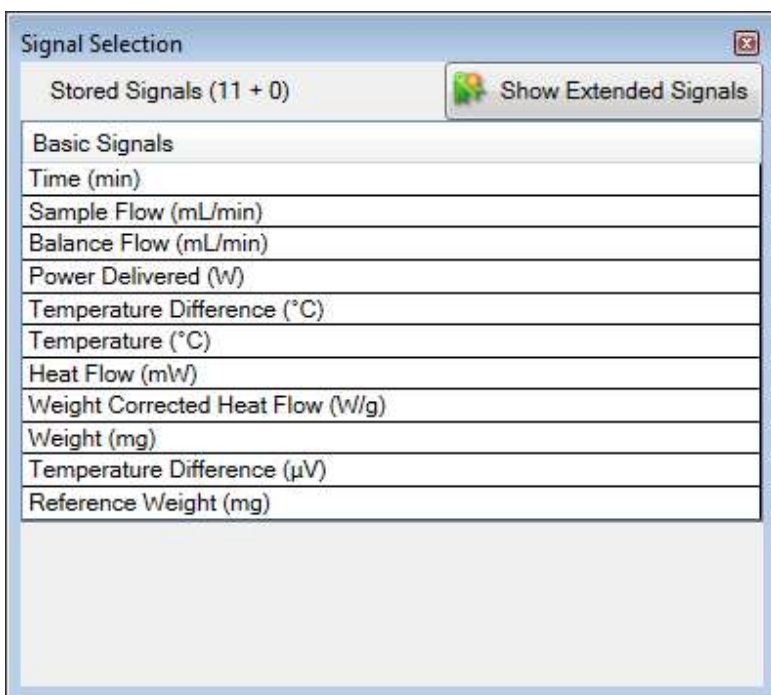
The following instructions assume that a run (or series of runs) have already been created.

1. Access the Experiment **Procedure** section.
2. Select **Custom** test from the list of available templates.
3. Click **Edit** to access the **Method Edit** functions. A list of available method segments is displayed.



-  Move segment up
-  Move segment down
-  New method, clears method segments
-  Remove method segment

4. Click **New** to clear the current method segment contents.
5. Add the desired segment from the available segment list by one of the following methods:
 - Double-clicking on the segment to be added.
 - Use the drag-and-drop method to move the segment: Hold down the mouse button while moving the cursor to the desired position in the list of numbered segments.
6. Make the desired changes to the segment parameters by either entering the desired parameter or choosing from the drop-down list of available options. Use the **TAB** key or mouse to move between segment fields.
7. Repeat steps 6 and 7 until all of the desired segments are added to the method.
8. Click **Signal selection** to view the stored signals. Click **Show Extended Signals** to add additional signals to the stored selection.



9. Click **Apply** when finished.
10. Verify/set the desired **Advanced Options**. See [Defining DSC Experimental Parameters](#) for Advanced Options information for your particular instrument.
11. Save this procedure if you plan to use it again in the future.

Method Editor Functions

| Function | Description |
|---------------------------------------|--|
| Adding, inserting, or moving segments | You can use the drag-and-drop technique to add, move, or insert segments. Drag the desired segment from the available method segment list to the method description. |
| Removing segments | Select the desired segment(s) to remove. <ul style="list-style-type: none"> • Click Remove Segment. • Right-click in Method and select Delete. • Click Delete from computer keyboard |
| Reordering segments | Select the desired segment(s) to move. <ul style="list-style-type: none"> • Hold down the left mouse button while you move the pointer to the desired segment location. Then release the button. The segment will be moved to your chosen location. • Use the Move Up / Move Down buttons to move to the desired position. |
| Editing segment parameters | Make the desired changes by either entering the desired parameter or choosing from the drop-down list of options available for that segment. |
| Copying and pasting segments | <ol style="list-style-type: none"> 1. Highlight the desired segment. 2. Right-click and select Copy from the pop-up menu. Select multiple segments at once by holding down the |

Ctrl key while highlighting the segments, then release the key.

3. Highlight the desired location within the segment description list and select **Paste** from the right-click pop-up menu.

For information on opening a saved procedure file or saving a procedure to the file, refer to the help topic [Defining DSC Experimental Parameters](#).

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6 Procedure: Standard Isothermal

Setting Up a Standard Isothermal Procedure

In this topic

[Test setup](#)

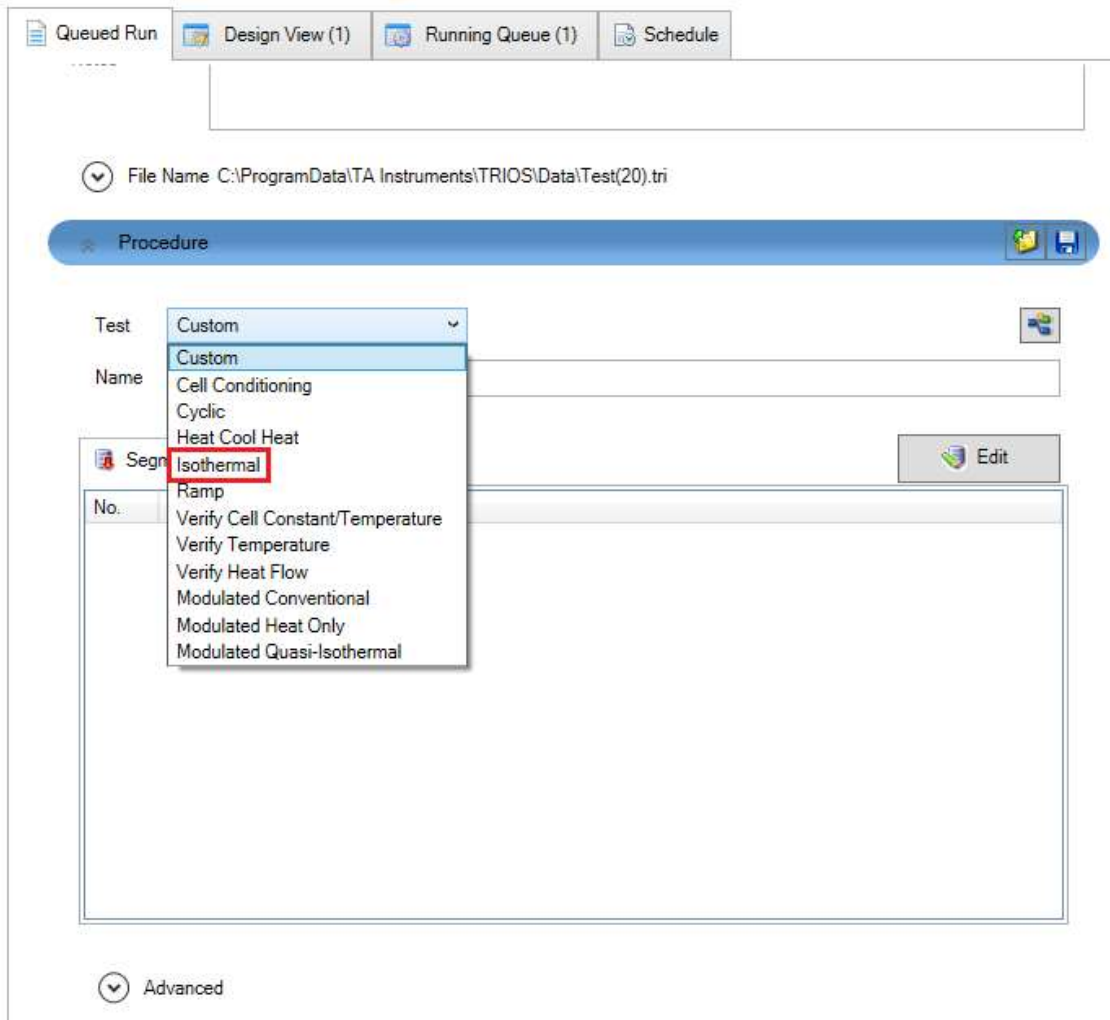
[Advanced](#)

[Analysis and Reporting](#)

DSC isothermal experiments provide an indication of a material's stability at an elevated temperature. The purge gas, which surrounds the material, remains constant or is switched at the elevated temperature to generate a sample-atmosphere interaction (for example, oxidative stability).

Test Setup

To select this test, click **Experiments > Design Run** in the File Manager. In the **Procedure** window, select **Isothermal** from the **Test** drop-down list (shown below).



Choose the following parameters after selecting the Standard Isothermal procedure:

1. **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 (for example, -120 to 550°C for the [RCS](#)). However, prolonged experiments near the upper temperature limit for a specific cooler could affect the lifetime of the unit. Therefore, use of the [Finned Air Cooling System \(FACS\)](#) is recommended for isothermal work above 350°C . In addition, isothermal evaluations above 600°C should be performed with an inert purge gas. Standard test methods exist where the equilibration temperature is specified. For example, polyolefin oxidative stability analysis studies are specified at 200°C based on ASTM Method D3895-07.

2. **Switch to Gas 2:** 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 (for example, stability in oxygen [resistance to oxidative degradation]).
3. **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.
4. **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) 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 is a good initial choice. Check the **End experiment when** box and select the desired setting to use this option.

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Advanced

The **Advanced** menu contains additional experimental conditions that can be modified to optimize testing for specific situations. For most experiments, the system default values shown are recommended.

⬆ Advanced

Load Window

Use Standby Temperature

Use °C to °C

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

Use °C to °C

Selected Calibrations

Use default calibrations

CalibrationSet11/21/19 ▾

CalibrationSet11/21/19

CalibrationSet9/21/19

CalibrationSet7/21/19

1. **Load Window** (applicable for Autosampler instruments only): This parameter is used to specify temperature range used to load the sample for the specified run. The experiment will not start until this condition is met. Select between the following options:
 - Check the **Use Standby Temperature** option to use the standby temperature (as defined on the [General Control panel](#)).
 - Uncheck this option to define a specific temperature range for load.
2. **End of Test** options:
 - For DSC Autosamplers: Check **Discard pan in waste bin at end of test** to have the Autosampler arm place the pan in the waste bin at the end of the run. If this is not selected, the arm will return the pan to its previous position on the sample tray at the end of the run.
 - **Use Standby Temperature:** This parameter is used to specify the temperature used to unload the sample for the specified run.
 - ◆ Check this option to use the standby temperature (as defined on the [General Control panel](#)).

- ❖ Uncheck this option to define a specific temperature range for unload. If desired, enter the delay time that the instrument will delay after reaching the unload temperature range before unloading the sample. A delay time of 2 minutes should be used when an experiment ends with the cell at subambient temperature. This allows time for the cell lids and cover to warm in order to prevent moisture from condensing on cold components.

3. **Selected Calibrations:** This option allows you to either use the default calibrations or select a specific set of calibrations to use for calculations on the file. For information on creating a Calibration Set, see the topic [Using the Calibration Data Window](#).

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Analysis and Reporting

⤴ Analysis and Reporting

Analysis Template

Report Template

Send Results to a Control Chart

- **Analysis Template:** To apply an existing analysis template, select **Analysis Template** and then browse for the template file.
- **Reporting:** To apply an existing report template, select Reporting and then browse for the template file.
- **Send Results to Control Chart:** Select **Send Results to a Control Chart** to send the results to an existing Control Chart document. For more information see [Control Charts](#).

7 Procedure: Standard Ramp

Setting Up a Standard Ramp Procedure

In this topic

[Test setup](#)

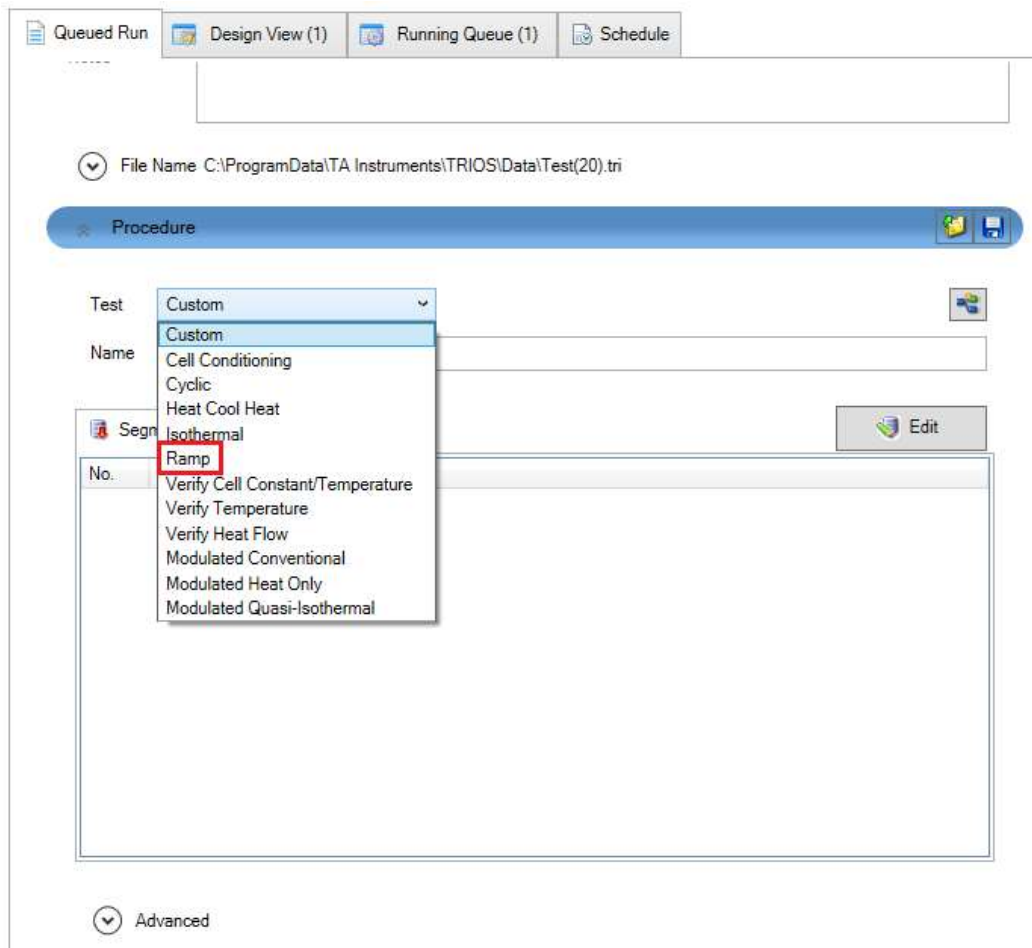
[Advanced](#)

[Analysis and Reporting](#)

DSC ramp experiments heat or cool the material at a constant rate. 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."

Test Setup

To select this test, click **Experiments > Design Run** in the File Manager. In the **Procedure** window, select **Ramp** from the **Test** drop-down list (shown below).



Choose the following parameters after selecting the Standard Ramp procedure:

1. **Start** and **Final Temperature**: The actual start and final temperatures chosen for a specific experiment must be within the usable temperature range 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 they should be far enough below or above the transitions to establish a stable baseline. Be sure to stay below the decomposition temperature of the sample. (Note that the decomposition temperature depends on the heating rate).
 - **Use current**: Check this box to use 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.

- Unselect **Use current** to choose the specific temperature desired. In this case, the system equilibrates at the chosen temperature before ramped heating begins.
 - The full temperature range of analysis for the DSC cell is -180 to 725°C , but may be attenuated depending on the type of sample pan, lid type, and cooling accessory used. (Only inert gases such as nitrogen can be used above 600°C .) To evaluate materials below room temperature or to run controlled-cooling experiments, the DSC cell requires an active cooling source. Typically, this is provided by a liquid nitrogen cooling system, LN2P (usable from -150 to 550°C), or a refrigerated cooling accessory, RCS (usable from -120 to 550°C). In addition, there are multiple types of DSC pans available. All of them are acceptable for use to 725°C except for aluminum pans, which should only be used to 600°C .
2. **Heating (Ramp) Rate:** The acceptable range of heating/cooling rates for conventional DSC is 0.01 to $200^{\circ}\text{C}/\text{minute}$. This range depends on a number of variables including the temperature range covered and the type of 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 rates of 10 to $20^{\circ}\text{C}/\text{minute}$ and cooling rates of 5 to $10^{\circ}\text{C}/\text{minute}$ are a good starting point for most materials.

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Advanced

The **Advanced** menu contains additional experimental conditions that can be modified to optimize testing for specific situations. For most experiments, the system default values shown are recommended.

Advanced

Load Window

Use Standby Temperature

Use $^{\circ}\text{C}$ to $^{\circ}\text{C}$

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

Use $^{\circ}\text{C}$ to $^{\circ}\text{C}$

Selected Calibrations

Use default calibrations

CalibrationSet11/21/19

CalibrationSet11/21/19

CalibrationSet9/21/19

CalibrationSet7/21/19

1. **Load Window** (applicable for Autosampler instruments only): This parameter is used to specify temperature range used to load the sample for the specified run. The experiment will not start until this condition is met. Select between the following options:
 - Check the **Use Standby Temperature** option to use the standby temperature (as defined on the **General Control** panel).
 - Uncheck this option to define a specific temperature range for load.
2. **End of Test** options:
 - For DSC Autosamplers: Check **Discard pan in waste bin at end of test** to have the Autosampler arm place the pan in the waste bin at the end of the run. If this is not selected, the arm will return the pan to its previous

position on the sample tray at the end of the run.

- **Use Standby Temperature:** This parameter is used to specify the temperature used to unload the sample for the specified run.
 - ❖ Check this option to use the standby temperature (as defined on the **General** Control panel).
 - ❖ Uncheck this option to define a specific temperature range for unload. If desired, enter the delay time that the instrument will delay after reaching the unload temperature range before unloading the sample. A delay time of 2 minutes should be used when an experiment ends with the cell at subambient temperature. This allows time for the cell lids and cover to warm in order to prevent moisture from condensing on cold components.
- 3. **Selected Calibrations:** This option allows you to either use the default calibrations or select a specific set of calibrations to use for calculations on the file. For information on creating a Calibration Set, see the topic [Using the Calibration Data Window](#).

Analysis and Reporting

⬆ Analysis and Reporting

Analysis Template

Report Template

Send Results to a Control Chart



- **Analysis Template:** To apply an existing analysis template, select **Analysis Template** and then browse for the template file.
- **Reporting:** To apply an existing report template, select Reporting and then browse for the template file.
- **Send Results to Control Chart:** Select **Send Results to a Control Chart** to send the results to an existing Control Chart document. For more information see [Control Charts](#).

8 Procedure: Standard Heat/Cool/Heat

Setting Up a Standard Heat/Cool/Heat Procedure

In this topic

[Test setup](#)

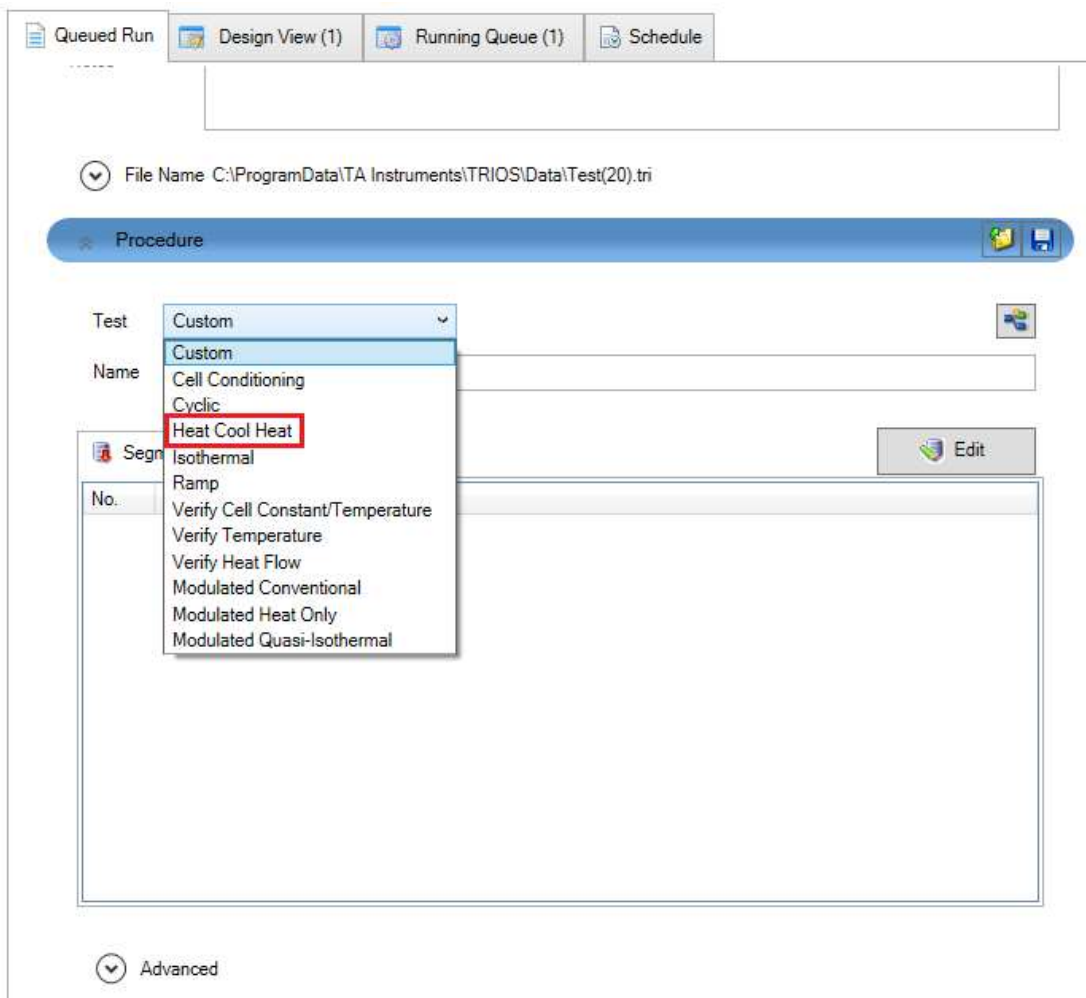
[Advanced](#)

[Analysis and Reporting](#)

DSC heat/cool/heat experiments are designed to erase previous thermal history by heating the material above a transition (for example, glass transition or melting), where relaxation or molecular rearrangement can occur, then cooling at a known rate before heating again. 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 (molecular weight), rather than previous thermal history effects.

Test Setup

To select this test, click **Experiments > Design Run** in the File Manager. In the **Procedure** window, select **Heat Cool Heat** from the **Test** drop-down list (shown below).



Choose the following parameters after selecting the Standard Heat/Cool/Heat procedure:

1. **Start Temperature:** The actual start and final temperatures chosen for a specific experiment must be within the usable temperature range 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 they should be far enough below or above the transitions to establish a stable baseline. Be sure to stay below the decomposition temperature of the sample. (Note that the decomposition temperature depends on the heating rate).

- **Use current:** Check this box to use 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.
 - Unselect **Use current** to choose the specific temperature desired. In this case, the system equilibrates at the chosen temperature before ramped heating begins.
 - The full temperature range of analysis for the DSC cell is –180 to 725°C, but may be attenuated depending on the type of sample pan, lid type, and cooling accessory used. (NOTE: Only inert gases such as nitrogen can be used above 600°C.) To evaluate materials below room temperature or to run controlled-cooling experiments, the DSC cell requires an active cooling source. Typically, this is provided by a liquid nitrogen cooling system, [LN2P](#) (usable from –150 to 550°C; not yet available for Discovery DSC models 25/250/2500), or a refrigerated cooling accessory, [RCS](#) (usable from –120 to 550°C). In addition, there are multiple types of DSC pans available. All of them are acceptable for use to 725°C except for aluminum pans, which should only be used to 600°C.
2. **Heating Rate** and **Cooling Rate:** The acceptable range of heating/cooling rates for conventional DSC is 0.01 to 200°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 rates of 10 to 20°C/minute and cooling rates of 5 to 10°C/minute are a good starting point for most materials.
 3. **Upper Temperature:** To obtain the intended benefits from a heat/cool/heat experiment, the chosen upper temperature 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.
 4. **Lower Temperature:** The lowest temperature achieved during the cooling portion of the experiment should be below any transitions of interest.

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Advanced

The **Advanced** menu contains additional experimental conditions that can be modified to optimize testing for specific situations. For most experiments, the system default values shown are recommended.

⬆ Advanced

Load Window

Use Standby Temperature

Use °C to °C

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

Use °C to °C

Selected Calibrations

Use default calibrations

CalibrationSet11/21/19 ▾

CalibrationSet11/21/19

CalibrationSet9/21/19

CalibrationSet7/21/19

1. **Load Window** (applicable for Autosampler instruments only): This parameter is used to specify temperature range used to load the sample for the specified run. The experiment will not start until this condition is met. Select between

the following options:

- Check the **Use Standby Temperature** option to use the standby temperature (as defined on the [General Control panel](#)).
- Uncheck this option to define a specific temperature range for load.

2. **End of Test** options:

- For DSC Autosamplers: Check **Discard pan in waste bin at end of test** to have the Autosampler arm place the pan in the waste bin at the end of the run. If this is not selected, the arm will return the pan to its previous position on the sample tray at the end of the run.
- **Use Standby Temperature:** This parameter is used to specify the temperature used to load the sample for the specified run.
 - ❖ Check this option to use the standby temperature (as defined on the [General Control panel](#)).
 - ❖ Uncheck this option to define a specific temperature range for unload. If desired, enter the delay time that the instrument will delay after reaching the unload temperature range before unloading the sample. A delay time of 2 minutes should be used when an experiment ends with the cell at subambient temperature. This allows time for the cell lids and cover to warm in order to prevent moisture from condensing on cold components.

3. **Selected Calibrations:** This option allows you to either use the default calibrations or select a specific set of calibrations to use for calculations on the file. For information on creating a Calibration Set, see the topic [Using the Calibration Data Window](#).

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Analysis and Reporting

⬆ Analysis and Reporting

| | | |
|--|----------------------|------------------------------------|
| <input type="checkbox"/> Analysis Template | <input type="text"/> | <input type="button" value="..."/> |
| <input type="checkbox"/> Report Template | <input type="text"/> | <input type="button" value="..."/> |
| <input type="checkbox"/> Send Results to a Control Chart | <input type="text"/> | <input type="button" value="..."/> |

- **Analysis Template:** To apply an existing analysis template, select **Analysis Template** and then browse for the template file.
- **Reporting:** To apply an existing report template, select Reporting and then browse for the template file.
- **Send Results to Control Chart:** Select **Send Results to a Control Chart** to send the results to an existing Control Chart document. For more information see [Control Charts](#).

9 Procedure: Standard Cyclic

Setting Up a Standard Cyclic Procedure

In this topic

[Test setup](#)

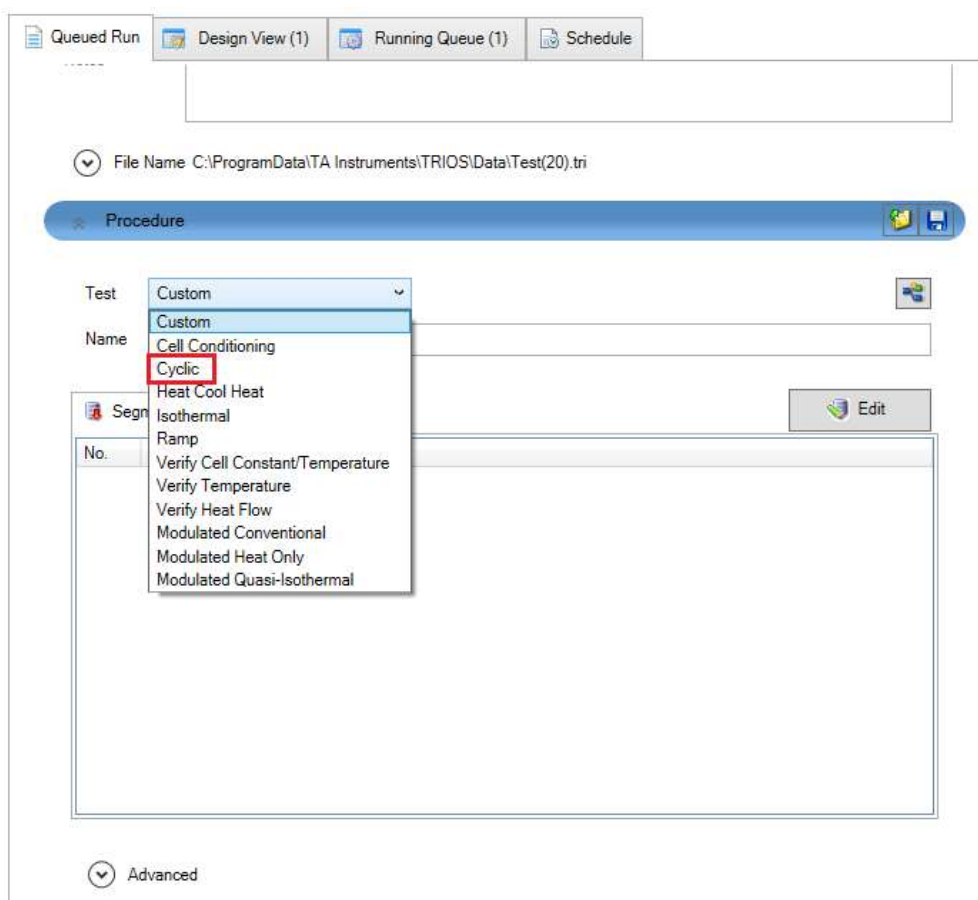
[Advanced](#)

[Analysis and Reporting](#)

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

Test Setup

To select this test, click **Experiments > Design Run** in the File Manager. In the **Procedure** window, select **Cyclic** from the **Test** drop-down list (shown below).



Choose the following parameters after selecting the Standard Cyclic procedure:



NOTE: The full temperature range of analysis for the DSC cell is -180 to 725°C , but may be attenuated depending on the type of sample pan, lid type, and cooling accessory used. (Only inert gases such as nitrogen can be used above 600°C .) To evaluate materials below room temperature or to run controlled-cooling experiments, the DSC cell requires an active cooling source. Typically, this is provided by a liquid nitrogen cooling system, [LN2P](#) (usable from -150 to 550°C ; not yet available for Discovery DSC models 25/250/2500), or a refrigerated cooling accessory, [RCS](#) (usable from -120 to 550°C). In addition, there are multiple types of DSC pans available. All of them are acceptable for use to 725°C except for aluminum pans, which should only be used to 600°C .

1. **Lower Temperature:** The lowest temperature achieved during the cooling portion of the experiment should be below any transitions of interest.

2. **Heating Rate:** The acceptable range of heating/cooling rates for conventional DSC is 0.01 to 200°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 rates of 10 to 20°C/minute and cooling rates of 5 to 10°C/minute are a good starting point for most materials.
3. **Upper Temperature:** The chosen upper temperature 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.
4. **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.

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Advanced

The **Advanced** menu contains additional experimental conditions that can be modified to optimize testing for specific situations. For most experiments, the system default values shown are recommended.

⬆ Advanced

Load Window

Use Standby Temperature

Use °C to °C

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

Use °C to °C

Selected Calibrations

Use default calibrations

CalibrationSet11/21/19 ▾

CalibrationSet11/21/19

CalibrationSet9/21/19

CalibrationSet7/21/19

1. **Load Window** (applicable for Autosampler instruments only): This parameter is used to specify temperature range used to load the sample for the specified run. The experiment will not start until this condition is met. Select between the following options:
 - Check the **Use Standby Temperature** option to use the standby temperature (as defined on the [General Control panel](#)).
 - Uncheck this option to define a specific temperature range for load.
2. **End of Test** options:
 - For DSC Autosamplers: Check **Discard pan in waste bin at end of test** to have the Autosampler arm place the pan in the waste bin at the end of the run. If this is not selected, the arm will return the pan to its previous position on the sample tray at the end of the run.
 - **Use Standby Temperature:** This parameter is used to specify the temperature used to unload the sample for the specified run.

- ❖ Check this option to use the standby temperature (as defined on the [General Control panel](#)).
- ❖ Uncheck this option to define a specific temperature range for unload. If desired, enter the delay time that the instrument will delay after reaching the unload temperature range before unloading the sample. A delay time of 2 minutes should be used when an experiment ends with the cell at subambient temperature. This allows time for the cell lids and cover to warm in order to prevent moisture from condensing on cold components.

3. **Selected Calibrations:** This option allows you to either use the default calibrations or select a specific set of calibrations to use for calculations on the file. For information on creating a Calibration Set, see the topic [Using the Calibration Data Window](#).

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Analysis and Reporting

⬆ Analysis and Reporting

Analysis Template

Report Template

Send Results to a Control Chart

- **Analysis Template:** To apply an existing analysis template, select **Analysis Template** and then browse for the template file.
- **Reporting:** To apply an existing report template, select Reporting and then browse for the template file.
- **Send Results to Control Chart:** Select **Send Results to a Control Chart** to send the results to an existing Control Chart document. For more information see [Control Charts](#).

10 Procedure: Modulated Heat only

Setting Up a Modulated DSC[®] Heat Only Procedure

In this topic

[Test setup](#)

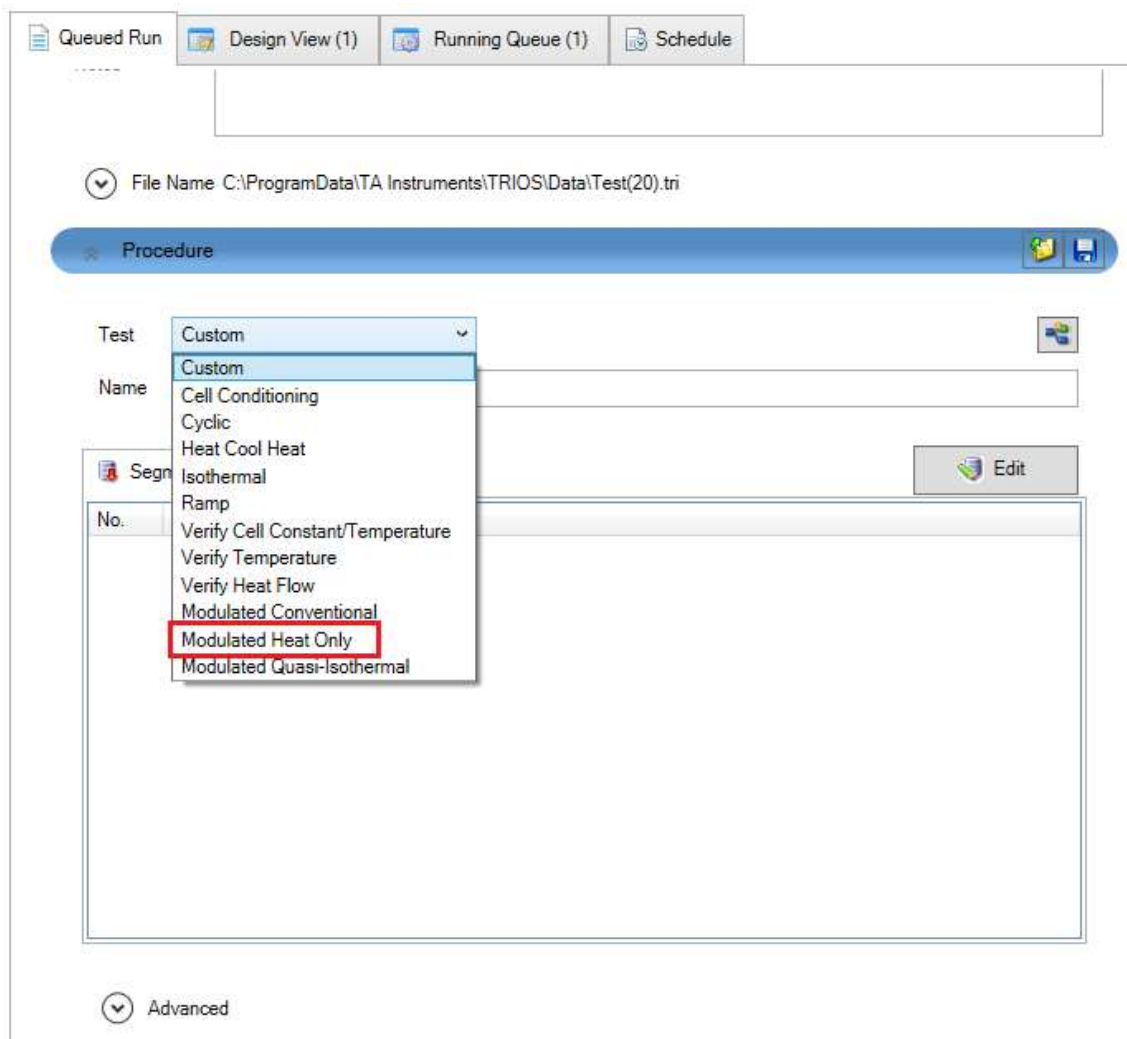
[Advanced](#)

[Analysis and Reporting](#)

Three variables are adjusted to obtain the desired MDSC[®] temperature profile—the underlying heating, modulation period, and temperature amplitude of modulation. When studying melting events, it is beneficial to adjust these variables so that the minimum instantaneous heating rate achieved is 0°C/minute (isothermal). By not cooling the material at any time during the modulation, the possibility for "artificially" affecting any observed crystallization phenomena is eliminated. More importantly, when the instantaneous heating rate is zero, there is no heat flow associated with heat capacity and, therefore, any heat flow observed at the top of the raw modulated heat flow signal must be the result of kinetic phenomena.

Test Setup

To select this test, click **Experiments > Design Run** in the File Manager. In the **Procedure** window, select **Modulated Heat Only** from the **Test** drop-down list (shown below).



Choose the following parameters after selecting the MDSC Heat Only procedure:

1. **Modulation Period:** Use a modulation period of 40 seconds. Longer periods (60 seconds) are recommended for polymer samples greater than 15 mg.

2. **Ramp Rate:** Select an underlying heating rate of 5°C/minute or less, if required to get a minimum of four modulation cycles over the width of the melting peak (measure at half-height).
3. **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 725°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.

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Advanced

The **Advanced** menu contains additional experimental conditions that can be modified to optimize testing for specific situations. For most experiments, the system default values shown are recommended.

Advanced

Load Window

Use Standby Temperature

Use °C to °C

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

Use °C to °C

Selected Calibrations

Use default calibrations

CalibrationSet11/21/19

CalibrationSet11/21/19

CalibrationSet9/21/19

CalibrationSet7/21/19

1. **Load Window** (applicable for Autosampler instruments only): This parameter is used to specify temperature range used to load the sample for the specified run. The experiment will not start until this condition is met. Select between the following options:
 - Check the **Use Standby Temperature** option to use the standby temperature (as defined on the [General Control panel](#)).
 - Uncheck this option to define a specific temperature range for load.
2. **End of Test** options:
 - For DSC Autosamplers: Check **Discard pan in waste bin at end of test** to have the Autosampler arm place the pan in the waste bin at the end of the run. If this is not selected, the arm will return the pan to its previous position on the sample tray at the end of the run.
 - **Use Standby Temperature:** This parameter is used to specify the temperature used to load the sample for the specified run.
 - ❖ Check this option to use the standby temperature (as defined on the [General Control panel](#)).
 - ❖ Uncheck this option to define a specific temperature range for unload. If needed, enter the delay time that the instrument will wait after reaching the unload temperature range before unloading the sample. A delay time of 2 minutes (120 S) should be used when an experiment ends with the cell at subambient

temperature. This allows time for the cell lids and cover to warm in order to prevent moisture from condensing on cold components.

3. **Selected Calibrations:** This option allows you to either use the default calibrations or select a specific set of calibrations to use for calculations on the file. For information on creating a Calibration Set, see the topic [Using the Calibration Data Window](#).

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Analysis and Reporting

 Analysis and Reporting

Analysis Template



Report Template



Send Results to a Control Chart



- **Analysis Template:** To apply an existing analysis template, select **Analysis Template** and then browse for the template file.
- **Reporting:** To apply an existing report template, select Reporting and then browse for the template file.
- **Send Results to Control Chart:** Select **Send Results to a Control Chart** to send the results to an existing Control Chart document. For more information see [Control Charts](#).

11 Procedure: Modulated DSC Quasi Isothermal

Setting Up a Modulated DSC[®] Quasi Isothermal Procedure

In this topic

[Test setup](#)

[Advanced](#)

[Analysis and Reporting](#)

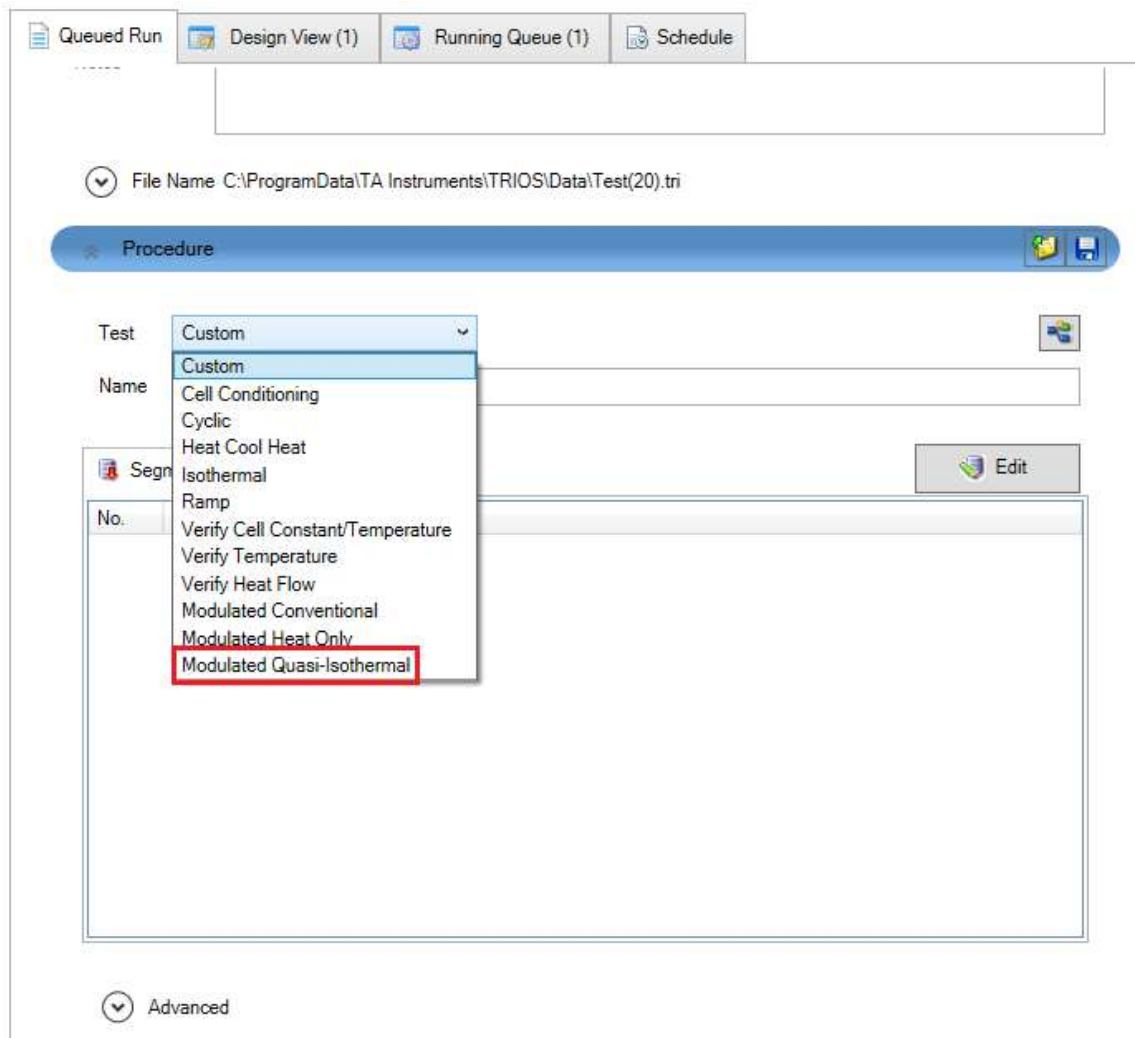
In quasi-isothermal MDSC experiments, the underlying heating rate is zero. However, by selecting a modulation temperature amplitude and period, the material is still exposed to an instantaneous heating rate.

Two variables are adjusted to obtain the desired complex MDSC temperature profile: the modulation period and temperature amplitude of modulation.

Generally, comparable results are obtained for underlying heating rates between 1 and 5°C/minute. However, for evaluation of time-dependent events such as glass transitions, the results obtained vary slightly with heating or cooling rate. Therefore, to obtain the most thermodynamically correct value for the glass transition temperature as well as the heat capacity change during the transition, the underlying heating rate should be set to zero (isothermal). A measurement of heat capacity can still be made under isothermal conditions based on the instantaneous heating rate generated by the modulation.

Test Setup

To select this test, click **Experiments > Design Run** in the File Manager. In the **Procedure** window, select **Modulated Quasi-Isothermal** from the **Test** drop-down list (shown below).



Choose the following parameters after selecting the MDSC Quasi-Isothermal procedure:

1. **Modulate Temperature Amplitude ±:** A typical temperature amplitude (e.g., +/-0.5°C) should be used to generate the modulation.
2. **Modulation Period:** A typical modulation period (e.g., 60 seconds) should be used to generate the modulation.
3. **Isothermal 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 (e.g., -90 to 550°C for the RCS). However, prolonged experiments near the upper temperature limit for a specific cooler could affect the lifetime of the unit. Therefore, use of the [Finned Air Cooling System \(FACS\)](#) is recommended for isothermal work above 350°C. In addition, isothermal evaluations above 600°C should be performed with an inert purge gas.
4. **Isothermal Time:** A stabilization time (e.g., 10 minutes) is allotted at each temperature to ensure the equilibrium heat capacity value is obtained.
5. **Temperature Increment:** A series of isothermal steps (e.g., 1°C increments) is run, which covers the transition and 10°C above it.
6. **Number of Increments:** Select the number of temperature increments desired. The default is 20.

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Advanced

The **Advanced** menu contains additional experimental conditions that can be modified to optimize testing for specific situations. For most experiments, the system default values shown are recommended.

Advanced

Load Window

Use Standby Temperature

Use °C to °C

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

Use °C to °C

Selected Calibrations

Use default calibrations

CalibrationSet11/21/19 ▼

CalibrationSet11/21/19

CalibrationSet9/21/19

CalibrationSet7/21/19

1. **Load Window** (applicable for Autosampler instruments only): This parameter is used to specify temperature range used to load the sample for the specified run. The experiment will not start until this condition is met. Select between the following options:
 - Check the **Use Standby Temperature** option to use the standby temperature (as defined on the [General Control panel](#)).
 - Uncheck this option to define a specific temperature range for load.
2. **End of Test** options:

- For DSC Autosamplers: Check **Discard pan in waste bin at end of test** to have the Autosampler arm place the pan in the waste bin at the end of the run. If this is not selected, the arm will return the pan to its previous position on the sample tray at the end of the run.
 - **Use Standby Temperature:** This parameter is used to specify the temperature used to load the sample for the specified run.
 - ❖ Check this option to use the standby temperature (as defined on the [General Control panel](#)).
 - ❖ Uncheck this option to define a specific temperature range for unload. If needed, enter the delay time that the instrument will wait after reaching the unload temperature range before unloading the sample. A delay time of 2 minutes (120 S) should be used when an experiment ends with the cell at subambient temperature. This allows time for the cell lids and cover to warm in order to prevent moisture from condensing on cold components.
3. **Selected Calibrations:** This option allows you to either use the default calibrations or select a specific set of calibrations to use for calculations on the file. For information on creating a Calibration Set, see the topic [Using the Calibration Data Window](#).

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Analysis and Reporting

⬆ Analysis and Reporting

Analysis Template

Report Template

Send Results to a Control Chart

- **Analysis Template:** To apply an existing analysis template, select **Analysis Template** and then browse for the template file.
- **Reporting:** To apply an existing report template, select Reporting and then browse for the template file.
- **Send Results to Control Chart:** Select **Send Results to a Control Chart** to send the results to an existing Control Chart document. For more information see [Control Charts](#).

12 Procedure: Modulated DSC Conventional Procedure

Setting Up a Modulated DSC[®] Conventional Procedure

In this topic

[Test setup](#)

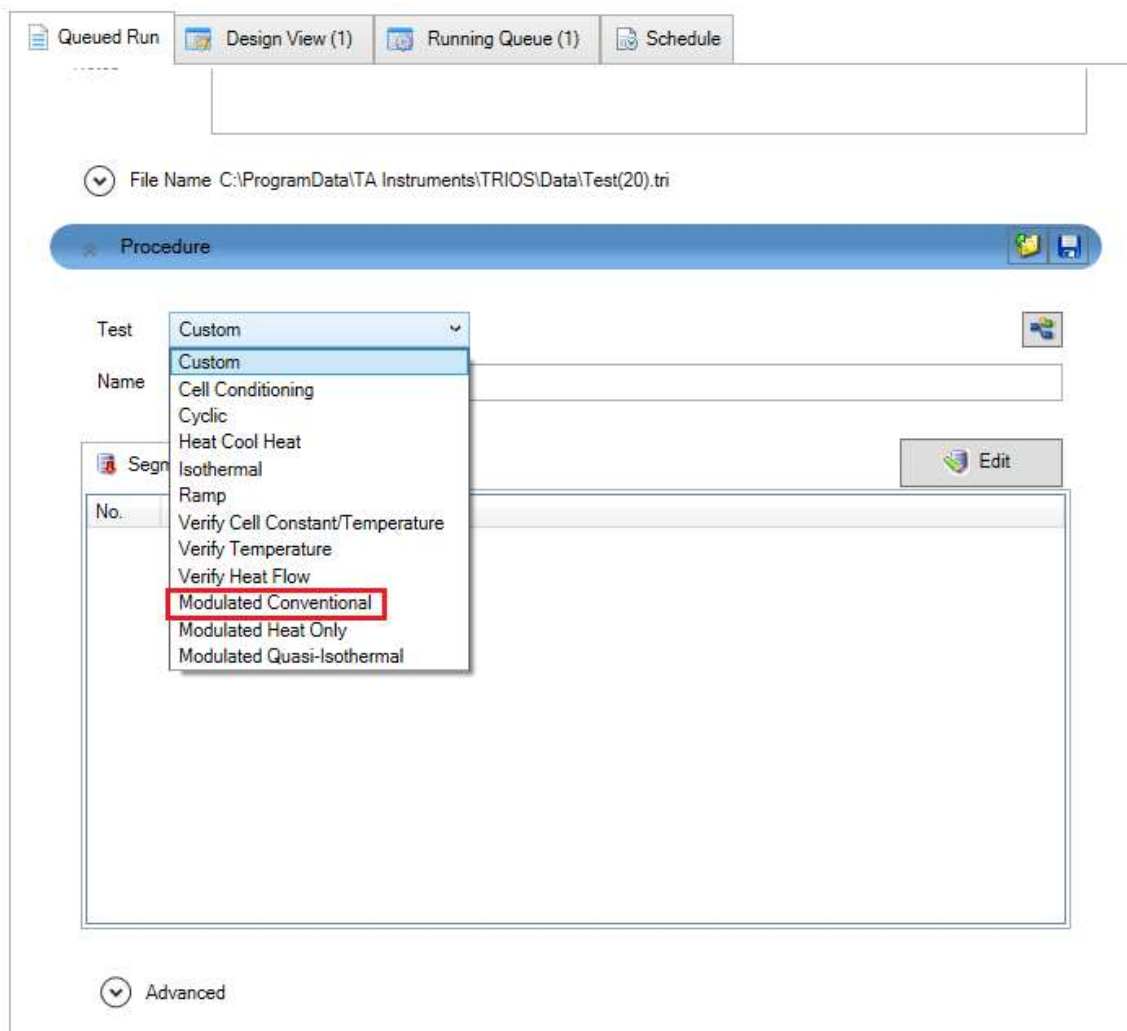
[Advanced](#)

[Analysis and Reporting](#)

The actual complex temperature/time profile in Modulated DSC[®] (MDSC) depends on three variables—underlying heating/cooling rate, modulation period, and modulation temperature amplitude. In conventional MDSC experiments, all three variables are operator-selected for best results and the material is evaluated during heating over a temperature range.

Test Setup

To select this test, click **Experiments > Design Run** in the File Manager. In the **Procedure** window, select **Modulated Conventional** from the **Test** drop-down list (shown below).



Choose the following parameters after selecting the MDSC Conventional procedure:

1. **Modulate Temperature Amplitude \pm :** 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.

2. **Modulation Period:** Select a modulation period of 20 to 200 seconds. For most materials in crimped pans, 40 to 60 seconds is recommended.
3. **Ramp Rate:** Select an underlying heating/cooling rate of 1 to 5°C/minute. Make sure that the rate chosen provides at least 4 complete temperature modulations (oscillations) over the temperature range of each transition studied.
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 725°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.

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Advanced

The **Advanced** menu contains additional experimental conditions that can be modified to optimize testing for specific situations. For most experiments, the system default values shown are recommended.

Advanced

Load Window

Use Standby Temperature

Use °C to °C

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

Use °C to °C

Selected Calibrations

Use default calibrations

CalibrationSet11/21/19

CalibrationSet11/21/19

CalibrationSet9/21/19

CalibrationSet7/21/19

1. **Load Window** (applicable for Autosampler instruments only): This parameter is used to specify temperature range used to load the sample for the specified run. The experiment will not start until this condition is met. Select between the following options:
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Analysis and Reporting

⬆ Analysis and Reporting

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|--|----------------------|------------------------------------|
| <input type="checkbox"/> Analysis Template | <input type="text"/> | <input type="button" value="..."/> |
| <input type="checkbox"/> Report Template | <input type="text"/> | <input type="button" value="..."/> |
| <input type="checkbox"/> Send Results to a Control Chart | <input type="text"/> | <input type="button" value="..."/> |

- **Analysis Template:** To apply an existing analysis template, select **Analysis Template** and then browse for the template file.
- **Reporting:** To apply an existing report template, select Reporting and then browse for the template file.
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