DSC1
Differential Scanning Calorimetery

Version 1.1.1

SMIF Team

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Contents

1 Introduction

The DSC2500 determines the temperature and heat flow of materials as a function of time and temperature. Provides data on endothermic (heat absorption) and exothermic (heat production) processes during phase transitions, melting, oxidation, and other kinetic and thermodynamic processes. It can also be used to measure and quantify the heat capacity of a material. Important features are:

- This DSC is equipped with the **R**efrigerated **C**ooling **S**ystem **RCS 90**. This operation ranges from $-90°C$ to $550°C$. The instrument last calibration was on Nov 09/2023 using a temperature ramp from $-90°C$ to $400°C$.
- The robotic arm (autosampler) automatically load the sample and reference pans and can process up to 53 samples + 1 reference
- The system is operated with TRIOS software. The user needs to design a sequence .

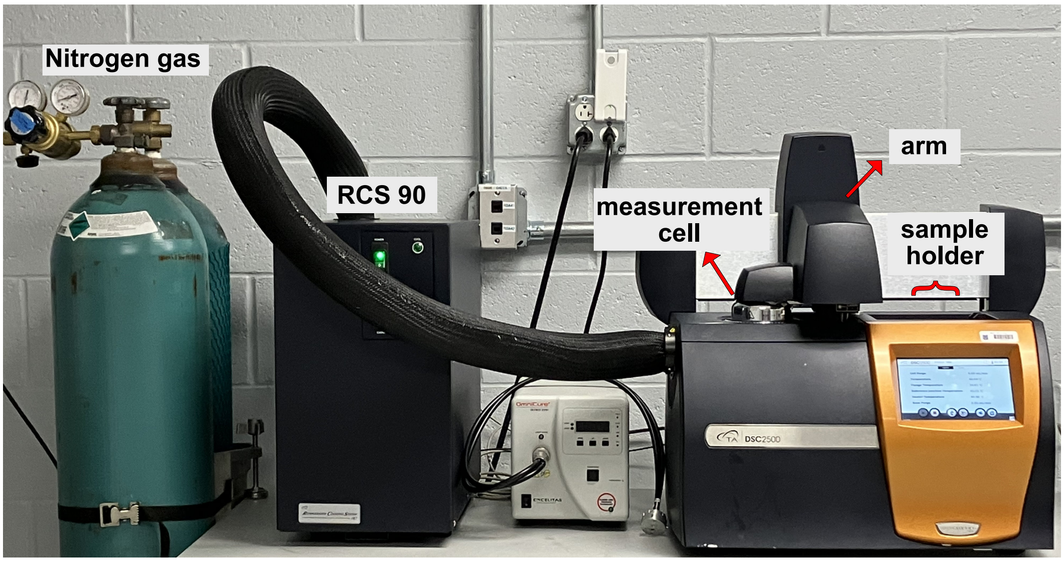

Figure 1: DSC2500 at the Soft Materials Lab, in SMIF

1.1 DSC2500 display

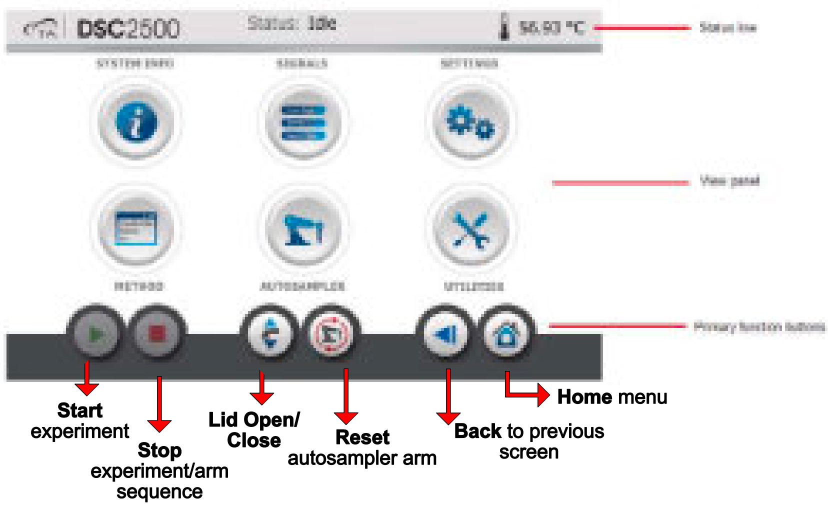

Figure 2: DSC2500 display panel, allows control of the DSC as well as monitoring relevant parameters

2 Basic steps quick check

The general outline for DSC experiments include:

1. Open Nitrogen valve
2. Selecting sample pan / lid system.
3. Preparing sample.
4. Configuring the autosampler or manually loading the DSC cell.
5. Designing the experimental run or sequence of runs using the TRIOS software.
6. Running the experiment or sequence.
7. Shut down RCS 90
8. Wait until the Flange temperature reaches 20°C, then close nitrogen valve

3 System Startup

1. **Open Nitrogen valve** and verify there is sufficient gas to last the duration of your measurements. The **Gas Delivery Module (GDM)** light in the Status Bar will turn green (fig. 3, red circle) and the base purge signal will indicate approximately 406 ml/min of flow (fig. 3, panel highlighted by a yellow rectangle).

2. Verify that the DSC2500 instrument is turned on and the RCS 90 cooler is both turned on and switched to **Event** mode.

3. Open TRIOS software and connect to the DSC2500 instrument.

4. Turn the RCS 90 **ON** by cliking the command in the **Temperature** subsection of the Control Panel window.The flange temperature will approach $-90°$C approximately 15 minutes after the RCS 90 is switched on.

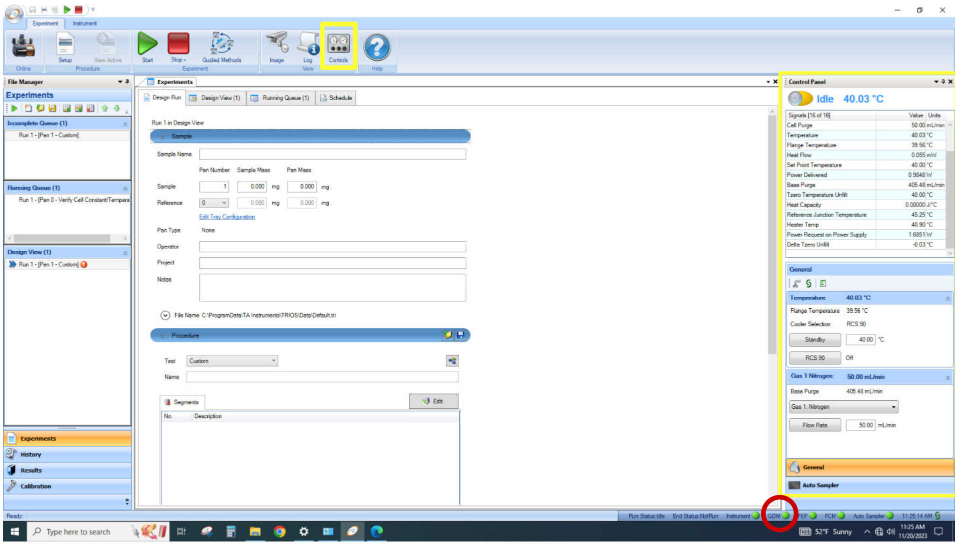

Figure 3: ***Trios*** interface. The control panel can be called clicking the **Controls** button in the top menu. The Control window is located at the right of the screen

4 Define Standby Temperature: Control Panel

The control panel can be called clicking the **Controls** button in the top menu. The Control window is located at the right of the screen

4.1 Set Temperature

Although the experiment temperature si defined in the test procedure, it is often desirable to set the temperature prior to starting the experiments. To access go to the tab **GeneralTemperature** in the **Control panel** (fig. 3).

- Enter the desired value in the field to the right of **Standby**, then click **Standby**

4.2 Gas source and flow rate

The system you're using only has **Nitrogen gas**. The system must be operated with a **Flow rate of 50 mL/min**

5 Preparing the Sample

This instrument uses Tzero pans, Tzero lids, and Tzero hermetic lids. The sample pan is loaded with a sample of a known mass. The optimal sample weight is dependent on the type of measurement being conducted (see Appendix A).Typical numbers ranges between 5mg to 20 mg. Pans are sealed using the Tzero press.

1. Determine the appropriate sample mass for your DSC experiment(see fig. 4 for reference).

Determining Sample Size

Types of Measurement	Sample Size	Heating Rate (°C/min)
Glass transition	10 to 20	10 to 20
Melting point	2 to 10	5 to 10
Kinetics (Borchardt and Daniels)	5 to 10	5 to 20
Kinetics (ASTM)	2 to 5*	0.5 to 20
Heat capacity	10 to 20	20**
Purity	1 to 3	0.5 to 1
Crystallinity	5 to 10	5 to 10
Oxidative stability	5 to 10	5 to 10
MDSC	2 to 10	1 to 5

* Use larger sample sizes at slower heating rates, and smaller sample sizes at higher rates.

** Except for Modulated DSC®, maximum 5°C/min.

Figure 4: Table for detemining sample size.

2. Determine the appropriate pan, lid and press die set. (see Pan / Die Selection table)

3. Install appropriate die set into the Tzero press.

4. Use the balance and record the mass of the sample and of the Tzero pans.

5. Spread the sample as evenly and as flat as possible into the sample pan. Non-flat samples may need to be pressed using the cupped upper die available in the BLACK and GREEN die sets.

6. Using tweezers position the lid onto the pan, and place the assembled pan into the lower die.

7. Press handle to crimp the lid onto the pan. No significant resistance should be encountered during pres operation.

8. Use the pan ejection pin to remove the sealed pan from the lower die.

9. Place the closed sample pan into any available position in the autosampler tray. Some experiments, such as isothermal oxidation, and specimens require an open pan. When using open pans, the risk of DSC cell contamination may increase.

6 Setting experimental sequence in TRIOS

In this section we give an overview on how to create "runs". For more information refer to appendix B and the resources on SMIF/DSC website.
Each **Runs** is requires defining a**sample** and a **procedure**. We recommend creating the runs in the **Design** view.

1. If available, Open a previously **Saved Sequence file**. Go to “Experiment” tab. Access “Design View”.

2. load a sequence clicking "Load Sequence File" (fig 5).

3. Create a sequence by clicking "Create New Runs" in the Design View (fig 5)

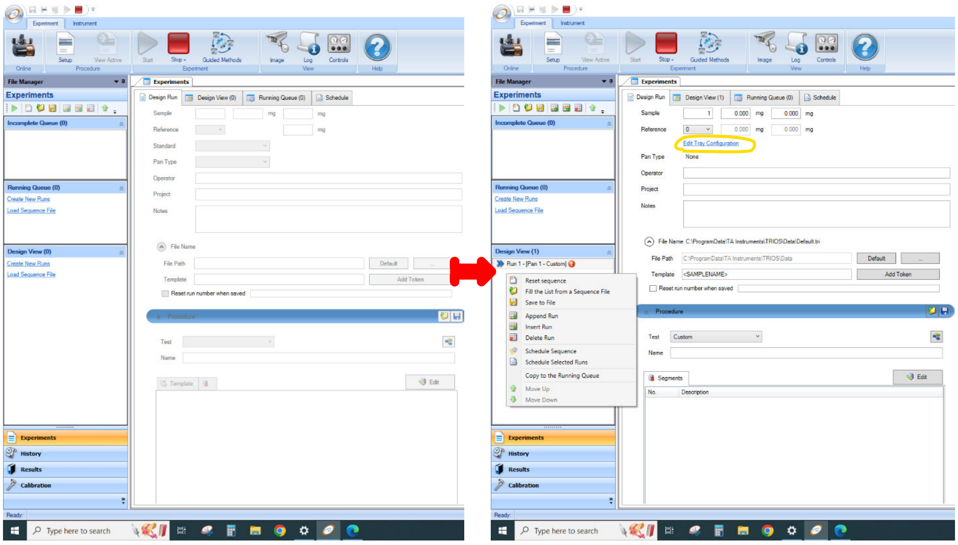

Figure 5: Create runs in the "Design View". You can create multiple runs in the window, for that right click-> "Append Run". Move it to the "Running Queue" by selecting the run and doing right click-> "Copy to the Running Queue"

4. Enter the sample information.

   - Enter the sample name.
   - Enter the sample pan number, sample mass, and the pan mass.

5. Enter the reference information.

   - Press the “Edit Tray Configuration” (fig 5, yellow circle). A graphic with the tray will appear (fig 6). Select the numbers you will use by clicking. Report the "Sample Mass", the "Pan Mass" and the "Pan Type" for each tray number.
   - Select the reference pan number from the drop down list. Note:Pan number 0 is for manual loading of the pan. Use it if you don’t want to use the autosampler arm.

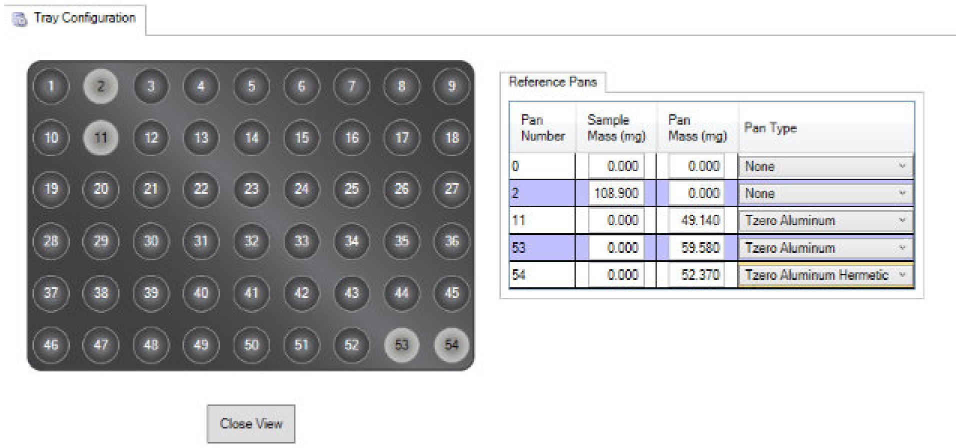

Figure 6: configure the tray according to the Pan Numbers you will be using. Report mass and pan type for each pan number in use

6. Define the Operator, Project and Note fields.
7. Define the DSC procedure.
   - Using the test drop-down list you can select a pre-programmed test template or, by selecting "Custom", create your own method.
   - Optionally name the test
   - Edit the segments to define the specific experimental conditions of your DSC experiment.
8. In the "Design View" sub-panel of the File Manager Panel, right-click on the currently designed run, and select "Copy to the Running Queue" (fig. 5).
9. To begin executing runs located in the running queue, press the green start button at the top panel of TRIOS.

Runs may be inserted or appended into the running queue from the "Design View" window, previously completed runs, or saved custom templates.
it is possible to modify the currently running method by interacting with the "Running Method List" sub-panel of the "Control Panel"

7 Saving Your Data

7.1 Save Procedure and data

1. Go to the Design view or in the Running Queue.

2. Click Save sequence

3. Locate your folder and save.

4. Export data to Excell:

    - Select the run.
    - click in the ***TA*** button at the top left of the screen
    - select "Export to Excell"

8 Shut Down

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

1. Turn off the RCS 90 cooler from the **Temperature** subsection of the Control Panel window. Alternatively, you can set this step in the **Experiment Sequence** by defining an **Event** step at the end of the run.

2. If changed during the course of your experiment, set the **Standby Temperature** to 20°C.

3. When the **Flange Temperature** reaches 40°C, close the Nitrogen gas valve.

4. Exit the TRIOS software, Leave DSC and RCS 90 powered on

A Tzero Pans: how to prepare samples

Selecting a Sample Pan

In this topic

In an ideal DSC experiment, the sample material is placed directly on the DSC sensor to obtain optimum heat transfer. However, that is not practical due to potential contamination of the sensor. Therefore, samples are placed in the sample pans for analysis.

Selection of the appropriate DSC sample pan and lid is a very important step in the method development process. The results obtained from the DSC can be affected by the sample pan. Proper method development is addressed in training courses available from TA Instruments.

TA Instruments offers various types of DSC pans for use in your experiments. Use the following guidelines to select a sample pan material and configuration that meets the temperature and pressure range, composition, and reactivity requirements of your experiment.

Selecting a Sample Pan for the Tzero Press

There are two series of DSC pans available for use with the Tzero® Press. The Tzero Series sample pans and lids are manufactured using an advanced process to higher design specifications and their use typically results in improved resolution, sensitivity, and enthalpy and Cp repeatability compared to the Standard Series sample pans and lids. The Standard Series is suitable for general-purpose use and provide a balance between performance and cost.

Within each series, the following pan types are currently available. Factors to consider when choosing a pan type are described briefly below.

Tzero Series Aluminum Pans

- Tzero Aluminum
- Tzero Low-Mass Aluminum
- Tzero Aluminum Hermetic
- Tzero Aluminum Hermetic Alodined
- Tzero Aluminum Pin Hole Hermetic

Standard Series Aluminum Pans

- Standard Aluminum
- Standard Aluminum Hermetic
- Standard Aluminum Hermetic Alodined
- Standard Aluminum Pin Hole Hermetic

Specialty Pans

- High Volume
- High Pressure Capsule
- High Temperature / High Pressure Capsule

Tzero Pan Types

There are three types of Tzero pans available: the Tzero Pan (PN 901683.901), Tzero Alodined Pan (PN 901697.901), and the Tzero Low-Mass Pan (PN 901670.901).

There are also three types of lids available: the Tzero Lid (PN 901671.901), Tzero Hermetic Lid (PN 901684.901), and Tzero Hermetic Alodined Lid (PN 901698.901).

These pans and lids are used to make three main pan types, which are described below: Tzero Aluminum, Tzero Hermetic Aluminum, and Tzero Low-Mass Aluminum.

- The Tzero Aluminum Pan is formed from the combination of the Tzero Pan and the Tzero Lid. It is used for typical DSC applications where there is not a need for hermetic sealing. This pan type has a capacity in volume of ca. 20 µL. These pans are prepared using the black die set for the Tzero press. Choose **Tzero Aluminum** as the pan type in TRIOS software when using this pan.
- The Tzero Hermetic Aluminum Pan is formed from the combination of the Tzero Pan and the Tzero Hermetic Aluminum Lid. This is a high-volume pan (ca. 40 µL) and is ideal when hermetic sealing is required, or when volatiles need to be contained. (Note that care should be taken when using liquid samples, as the presence of liquid may compromise the cold-weld hermetic sealing mechanism). This pan type is sealed using the blue die set for the Tzero press. Choose **Tzero Aluminum Hermetic** as the pan type in TRIOS software when using this pan. **NOTE**: A modified lid, which contains a pinhole of specific known diameter (75 micron, P/N 901685.901) is available for vapor pressure studies with the Tzero pans.
- For samples which may evolve large amounts of water (e.g., food), it is recommended to use the Tzero Hermetic Aluminum Alodined Pan. At higher temperatures and pressure, water can react with aluminum. This exothermic reaction may manifest in your measurement data. An inorganic alodine treatment applied to the Tzero aluminum hermetic pans eliminates the interference caused by the water in the sample. (Note that care should be taken when using liquid samples, as the presence of liquid may compromise the cold-weld hermetic sealing mechanism). This pan type is sealed using the blue die set for the Tzero press. Choose **Tzero Alodined Aluminum Hermetic** as the pan type in TRIOS software when using this pan.
- The Tzero Low-Mass Aluminum Pan is formed from the combination of the Tzero Low-Mass Pan and the Tzero lid. This is a specialty pan type, which is ideal for high-sensitivity applications on small amounts of material (<10 mg). This pan type is sealed using the black die set for the Tzero press. Choose **Tzero Aluminum** or **Tzero Low Mass Aluminum** as the pan type in TRIOS software when using this pan. Note that Tzero Hermetic Lids should not be used with Tzero Low-Mass Pans.

The various pan type configurations are summarized in the following table:

Pan Type Configurations

Pan Type	Pan	Lid	Die Set	Software Pan Type	Application
Tzero Aluminum	Tzero Pan	Tzero Lid	Black	Tzero Aluminum	DSC/MDSC applications
Tzero Hermetic Aluminum	Tzero Pan	Tzero Hermetic Lid	Blue	Tzero Hermetic Aluminum	DSC applications which require hermetic seals
Tzero Hermetic Aluminum Alodined	Tzero Alodined Pan	Tzero Hermetic Alodined Lid	Blue	Tzero Hermetic Aluminum Alodined	DSC applications which require hermetic seals and may evolve water
Tzero Low-Mass Aluminum	Tzero Low-Mass Pan	Tzero Lid	Black	Tzero Aluminum Low Mass	High-sensitivity for low mass of sample

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Tzero Aluminum Pan (Tzero Pan and Tzero Lid)

- PN 901683.901 Tzero Pans (Pkg/100)
- PN 901671.901 Tzero Aluminum Lids (Pkg/100)

The Tzero Aluminum Pan is an excellent choice for the majority of applications where elimination of volatiles is not an issue. The performance of the Tzero pan is exceptional due to the precise flatness of the pan bottom and the contact of the lid to the upper surface of the sample.

The Tzero Pan and Tzero Lid packages are labeled with a black/blue stripe and a solid black stripe, respectively. The Tzero Press includes three dies (shown below) with a black ring that are easily recognized and are used with these pans and lids. The lower die is readily distinguished from the upper dies by a larger outer diameter compared to the upper dies.

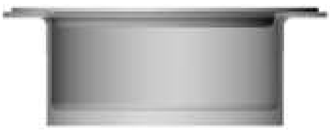
The design of the Tzero Lid and Tzero Press helps ensure that the pan bottom remains flat after sample loading. The lower die has a flat surface to support the pan bottom and minimize deformation from the sample when pressing the lid in place. You may choose either of two upper dies—one is flat and the other concave. The flat die is typically the first choice so that the sample-to-pan and sample-to-lid contact can be maximized. However, use of the concave die may be advantageous for irregularly shaped samples, as the lid will tend to conform to the sample better.

When using the Tzero Aluminum Pan, the maximum volume is 20 µL. The maximum height of the sample should be 1.0 mm (0.039 in) or less in order for the lid to properly engage the pan. The average mass of the pan and lid is about 49.7 mg with a typical variability of 0.75%. Advanced Tzero technology available with the Discovery DSC minimizes the impact of the higher mass, while the superior flatness of the pan bottom improves the sensitivity, resolution, and enthalpy and Cp repeatability of the heat flow measurement to levels never before achieved in routine practice.

The Tzero Pans and Lids are made of high purity aluminum and do not contain any intentional alloying elements. They are formed into their precisely designed shapes by an impact extrusion process that uses high pressure and a series of progressive dies to flow the aluminum into the precise shape of the tooling. Reliable adherence to stringent design specifications is achieved using this process. The Tzero Press and Pans represent the new state of the art in DSC sample preparation tools.

Tzero Hermetic Aluminum Pan (Tzero Pan and Tzero Hermetic Lid)

- PN 901683.901 Tzero Pans (Pkg/100)
- PN 901684.901 Tzero Hermetic Lids (Pkg/100)



The Tzero Hermetic Aluminum Pan is recommended for applications whenever volatiles generated by the sample during the experiment interfere with the transitions of interest. The Tzero Hermetic Aluminum Pan is not typically recommended for liquid samples, as the presence of liquid may compromise the cold-weld hermetic sealing mechanism.

The performance of the Tzero Hermetic Aluminum Pan is outstanding due to the precise flatness of the pan bottom and the design of the upper lid and sealing mechanism.

The Tzero Pan and Tzero Hermetic Lid packages are labeled with a black/blue stripe and a solid blue stripe, respectively. The Tzero Press kit contains a set of two dies (shown below) with a blue ring that are used with these pans and lids. The lower die is easily distinguished from the upper die by a larger outer diameter.

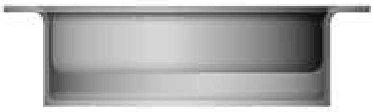
The Tzero Hermetic Lid should be positioned with the concave side down. The lid is readily sealed to the Tzero Pan by a cold welding process that uses pressure applied by the upper die in the Tzero Press to join the two aluminum surfaces together. The sealing process does not exert any force on the bottom of the pan and the pan bottom remains flat. The pressure seal formed will typically hold 2-3 atmospheres of internal pressure. When pressurized above 2 atmospheres, the lid will deform outward, with the bottom of the Tzero Pan in contact with the DSC sensor remaining flat.

When using the Tzero Hermetic Aluminum Pan the maximum volume is 40 µL. The maximum height of the sample should be less than 1.9 mm (0.075 in) in order for the lid to properly position on the sealing surface of the pan. The average mass of the pan and lid is 51.5 mg with a typical variability of 0.75 %. Advanced Tzero technology available with the Discovery DSC minimizes the impact of the higher mass, while the superior flatness of the pan bottom improves the sensitivity, resolution, and enthalpy and Cp repeatability of the heat flow measurement to levels never before achieved in routine practice.

The Tzero Pans and Tzero Hermetic Lids are made of high purity aluminum and do not contain any intentional alloying elements. They are formed into their precisely designed shapes by an impact extrusion process that uses high pressure and a series of progressive dies to flow the aluminum into the precise shape of the tooling. Reliable adherence to stringent design specifications is achieved using this process. The Tzero Press and Pans represent the new state of the art in DSC sample preparation tools.

Tzero Low-Mass Aluminum Pan (Tzero Low-Mass Pan and Tzero Lid)

- PN 901670.901 Tzero Low-Mass Pans (Pkg/100)
- PN 901671.901 Tzero Lids (Pkg/100)

The Tzero Low-Mass Aluminum Pan is intended for smaller sized samples that may benefit from the lower mass of the pan. The performance of the Tzero Low Mass pan is exceptional due to the precise flatness of the pan bottom and the contact of the lid to the upper surface of the sample.

The Tzero Low-Mass Pan and Tzero Lid packages are labeled with solid black stripes. The Tzero Press includes three dies (shown below) with a black ring that are easily recognized and are used with these pans and lids. The lower die is readily distinguished from the upper dies by a larger outer diameter compared to the upper dies.

The design of the Tzero Low Mass Lid and Tzero Press helps ensure that the pan bottom remains flat after sample loading. The lower die has a flat surface to support the pan bottom and minimize deformation from the sample when pressing the lid in place. The user may choose either of two upper dies; one is flat and the other concave. The flat die is typically the first choice so that the sample to pan and sample to lid contact can be maximized. However, use of the concave die may be advantageous for irregularly shaped samples, as the lid will tend to conform to the sample better.

The maximum volume of the Tzero Low-Mass Pan is 10 µL. The maximum height of the sample should be 0.51 mm (0.020 inches) or less in order for the lid to properly engage the pan. The average mass of the pan and lid is about 28.4 mg with a typical variability of 0.60%. Advanced Tzero support available with the Discovery DSC and Q2000 and the superior flatness of the pan bottom improves the sensitivity, resolution and enthalpy repeatability of the heat flow measurement.

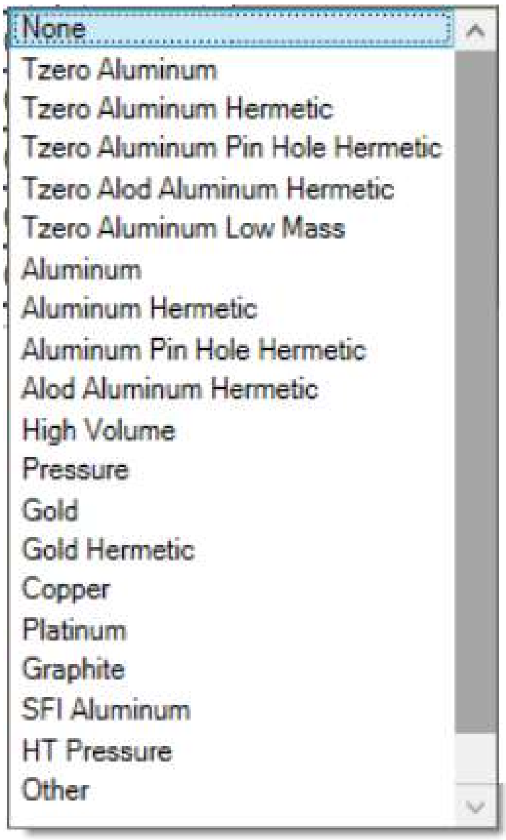
The Tzero Pans and Lids are made of high purity aluminum and do not contain any intentional alloying elements. They are formed into their precisely designed shapes by an impact extrusion process that uses high pressure and a series of progressive dies to flow the aluminum into the precise shape of the tooling. Reliable adherence to stringent design specifications is achieved using this process. The Tzero Press and Pans represent the new state of the art in DSC sample preparation tools.

Pan Resistance Correction

The Discovery DSC provides a more accurate measurement of true sample heat flow by accounting for heat flow within the DSC cell sensor as well as between the sensor and the sample pan, and between the pans themselves. The resultant sample heat flow is designated as T4P heat flow.

The heat flow between the sensor and sample pan is primarily affected by the sample pan thermal resistance, which is primarily a function of the pan material (thermal conductivity) and temperature, assuming that both the pan and sensor are perfectly flat surfaces and in intimate contact. This assumption, however, is not true, even if careful experimental procedure is followed when preparing samples. Therefore, the thermal resistance of the thin layer of purge gas between the pan and sensor must also be considered.

TA Instruments has included in both TRIOS and the Q Series™ Thermal Advantage software fixed values for both pan and purge gas thermal resistances. Available pan choices include aluminum, hermetic aluminum, hermetic aluminum alodined, hermetic pin hole, gold, hermetic gold, copper, platinum, graphite, SFI aluminum, large volume, low mass, pressure, and "other." Purge gas choices include air, nitrogen, oxygen, helium, and outside purge. When running experiments in Advanced Tzero mode T4P, select the pan type and purge gas that will be used. If you are using a pan type and/or purge gas that is not among the available choices listed, select "other."



Standard Pans

The three types of pans within the Standard Series serve similar purposes. The main factor to consider when choosing one of these three pan types is whether the sample will evolve volatiles and/or water.

- The Standard Aluminum pans (PN 900786.901) are the common first choice. These pans use lids (PN 900779.901) that are pressed down into contact with the sample and essentially encapsulate the sample in aluminum and thereby maximize the quality of the heat flow measurement. However, these types of lids are not sealed to prevent the loss of volatiles present in or generated by the sample during the experiment. Often, if these volatiles are allowed to evaporate, the endothermic heat flow associated with the vaporization obscures the transitions of interest. In some cases, the sample itself can flow out of these pans at higher temperatures and contaminate the cell. In the TRIOS software, select **Aluminum** as the pan type when using this pan.
- The Standard Aluminum Hermetic pans (PN 900793.901) have a different type of lid (PN 900794.901) and sealing mechanism that is able to hold the pressure buildup due to volatiles. This pressure can reach up to 2-3 atmospheres before the sealed pan releases the pressure. In many applications, the buildup and retention of the self-generated atmosphere by the sample improves the resolution of the transitions of interest. In the TRIOS software, select **Aluminum Hermetic** as the pan type when using this pan.

NOTE: Care should be taken when using liquid samples, as the presence of liquid may compromise the cold-weld hermetic sealing mechanism.

- The Standard Aluminum Hermetic Alodined pans (pan PN 900796.901 and lid PN 900790.901), are used when samples have high water content. At higher temperatures and pressure, water can react with the aluminum used to make these pans. This exothermic reaction can in some cases interfere with your measurement. An inorganic alodine treatment applied to the aluminum hermetic pans eliminates the interference caused by the water in the sample. In the TRIOS software, select **Alodined Aluminum Hermetic** as the pan type when using this pan.

NOTE: Care should be taken when using liquid samples, as the presence of liquid may compromise the cold-weld hermetic sealing mechanism.

Crimped and Hermetic Sample Pans

In an ideal DSC experiment, the sample material would be placed directly on the DSC sensor to obtain optimum heat transfer. However, that is not practical due to potential contamination of the sensor. Therefore, samples are placed in sample pans for analysis. The table below shows the common types of crimped and hermetic sample pans and their recommended temperature ranges.

NOTE: The useable temperature range could be reduced by the cooling accessory being used.

Crimped and Hermetic Sample Pans

Sample Pan	Usable Temperature Range (°C)	Part Number (pan/lid)	TRIOS Software Selection
Tzero and Standard Aluminum*	–180 to 600	900786.901 / 900779.901	Aluminum
Aluminum hermetic* [to 300 kPa (3 atm) internal pressure]	–180 to 600	900793.901 / 900794.901	Aluminum Hermetic
Alodined aluminum hermetic* [to 300 kPa (3 atm) internal pressure]	–180 to 200	900796.901 / 900790.901	Alodined Aluminum Hermetic
Gold*	–180 to 725	900866.901 / 900868.901	Gold
Gold hermetic* [to 600 kPa (6 atm) internal pressure]	–180 to 725	900871.901 / 900872.901	Gold Hermetic
Copper*[+]	–180 to 725	900867.901	Copper
Platinum[+]	–180 to 725	900578.901	Platinum
SFI Aluminum (Solid Fat Index)[+]	–180 to 600	900870.901	SFI Aluminum
Graphite	–180 to 725	900874.901 / 900873.901	Graphite

* Indicates pans designed for use with the DSC Autosampler and requires the Autosampler Sample Pan Tray PN 971126.90.

[+] No lid available.

Crimped and Open Pans

- Standard Series pans are designed to be run either open (no lid) or with a lid (crimped) on top of the sample. The lid is usually used to flatten the sample against the bottom of the pan for improved thermal contact. Without a lid, they provide the opportunity for good sample–atmosphere (purge gas) interaction. These pans are available in aluminum, gold, copper, platinum, and graphite. (Note that the copper, platinum, and SFI pans have no lids.)
- Aluminum pans are used for most experiments unless temperatures above 600°C are of interest for your sample or the sample reacts with aluminum.
- Copper pans are widely used for oxidative stability experiments, particularly for polymeric materials used as wire and cable coatings.
- Graphite pans are used for evaluating materials that might alloy with the various metal pans. Graphite pans should be used with a non-oxidizing purge gas (such as nitrogen).
- SFI (Solid Fat Index) pans are designed specifically for evaluating edible fats and oils. They have a recessed rim around the bottom of the pan to prevent "wicking" due to surface tension.

Hermetic Pans

- Hermetic pans are designed for evaluation of materials under self-generated atmosphere (modest pressure). Hermetic pans are almost always run sealed and are valuable for studies of volatile liquids (including vapor pressure studies), materials that sublime, and materials that generate corrosive or condensable gases.

Care should be taken when using liquid samples, as the presence of liquid may compromise the cold-weld hermetic sealing mechanism.

- Aluminum hermetic pans (sealable to 2–3 atm. of internal pressure) are used for most samples. Alodined aluminum pans are used to evaluate samples that react with "untreated" aluminum (e.g.,, aqueous biological materials).

Care should be taken when using liquid samples, as the presence of liquid may compromise the cold-weld hermetic sealing mechanism.

- A modified lid, which contains a pinhole of specific known diameter (75 micron, PN 900860.901), is available for vapor pressure studies in the aluminum hermetic pans.
- Gold hermetic pans are designed for evaluating materials under higher, self-generated pressures (up to 6 atm.) and temperatures (up to 725°C).
- Standard Hermetic pans, however, do provide a poorer thermal contact between the sample, pan and disk. This fact, plus the added mass of the hermetic pans and covers, leads to a slight loss of resolution compared to the crimped pans. The calorimetric accuracy is not affected, only the time constant of the system and the resolution of the measurement.

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High Volume, High Pressure, and High Temperature/High Pressure Pans

These pans are "specialty" pans designed for specific applications. The table below shows the temperature and pressure ranges appropriate for each type of pan. For additional information, refer to the Discovery Series and Q Series DSC High Pressure Pan Kit & High Temperature/High Pressure Pan Kit Getting Started Guide and the High Volume Pan Kit Operator's Guide.

Specialty Pans

<table>
<tr><th>Sample Pan</th><th>Usable Temperature Range (°C)</th><th>Usable Pressure Range (psi)</th><th>Part Number</th><th>TRIOS Software Selection</th></tr>
<tr><td>High volume pans*[+]</td><td>–100 to 250</td><td>575 (37 atm)</td><td>900825.902 (pans, lids, seals)</td><td>High Volume</td></tr>
<tr><td>High pressure pans[~]</td><td>Ambient to 300</td><td>1450 (100 atm)</td><td>900815.901 (pans and lids)
900814.901 (seals)</td><td>Pressure Capsule</td></tr>
<tr><td>High temperature / high pressure pans[~]</td><td>Ambient to 550</td><td><table><tr><th>Temperature (°C)</th><th>Pressure (MPa)</th></tr><tr><td>≤ 380</td><td>20</td></tr><tr><td>400</td><td>17</td></tr><tr><td>450</td><td>12</td></tr><tr><td>500</td><td>8</td></tr><tr><td>550</td><td>6</td></tr></table></td><td>900812.901 (pans, lids)
900811.901 (seals)</td><td>HT Pressure Capsule</td></tr>
</table>

[+]Use the yellow die set and Tzero press for closing.

[~]Special closing tool is required.

Additional Information About the Specialty Pans

- High volume pans are stainless steel, disposable pans designed for evaluating liquid samples up to 75 µL. They are particularly useful for measuring weak transitions in aqueous-based biological and food samples. The pans can be sealed to withstand modest pressures so that these materials can be evaluated under self-generated atmospheres. Although the material of construction (stainless steel) will withstand exposure to temperatures above 250°C, it is not recommended that these pans be used above this temperature because rupture of the pan at that point could damage the DSC cell.
- High pressure pans and high temperature/high pressure pans are stainless steel pans sealable to withstand up to 1450/2175 psi internal pressure and 300/550°C maximum temperature. They are specifically designed for pressure studies (evaluation of materials under self-generated atmospheres) and have a gold-plated copper rupture disk, which releases pressure gradually when it fails so that no DSC cell damage occurs. High pressure pans can be reused; seals are for one time use only.

B Experimental Sequence: 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	The **Abort** segment skips over the next segment when specified limit conditions are met. • 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. NOTE: The **Abort** segment is generally followed by a **Ramp** or **Isothermal** segment. Example (DSC): 1. Equilibrate at 200°C 2. Abort next segment if mW>1 3. Isothermal for 100 min
Air Cool (Discovery DSC only)	The **Air Cool** segment controls the internal cooling solenoid valve connected to compressed air (air cool feature). Example: Air Cool: On
Data	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. Example: Data Storage: On
Equilibrate	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. Example: Equilibrate at 200°C
Event 1 / **Event 2**	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. Example: Event 1: On
Heater PID	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. Example:

	P= 35 I= 70 D=2
Increment	The **Increment** segment raises or lowers the temperature in a controlled step, lets the temperature equilibrate, then begins the next segment. Example: Increment by 5°C
Initial Temperature (Discovery DSC only)	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. Example: Initial Temperature 200°C
Isothermal	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. Example: Isothermal for 10 min
Jump	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. Example: Jump to 200°C
Mark End	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. Example: Mark end of cycle 0
Mass Flow	The **Mass Flow** segment alters the rate of flow of the selected gas when an instrument is equipped with a Gas Delivery Module (GDM). Example: Mass Flow 50 mL/min
Modulate Temperature	*Available for Modulated Instruments Only*: 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. Example: Modulate temperature amplitude 1°C period 60 seconds
Ramp	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. Example: Ramp 10°C/min to 200°C
Repeat	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. Example: 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	The **Repeat Until** segment repeats of group of one or more segments within a method until the specified final temperature is reached or passed.

	Example: 1. Equilibrate at 50°C 2. Isothermal for 5 min 3. Increment 10°C 4. Repeat segment 2 until 200°C
Sample Interval	The **Sample Interval** segment allows you to define or change the rate at which data is to be collected (in seconds per point). Example: Sample Interval 2 sec /pt
Select Gas	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. Example: Select Gas 1
Shutter	*Applicable to Discovery DSC PCA accessory only:* 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. Example: Isothermal for 0.5 min Shutter: Open Isothermal for 2 min Shutter: Closed Isothermal for 0.5 min
Step	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. Example: Step 5°C for 2 min to 200°C

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