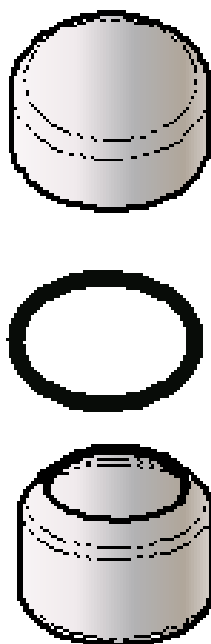


Discovery and Q Series DSC

High Volume Pan Kit



P/N 900825.901

Getting Started Guide



Notice

The material contained in this manual, and in the online help for the software used to support this instrument, is believed adequate for the intended use of the instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. TA Instruments also reserves the right to revise this document and to make changes without notice.

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Introduction

Important: TA Instruments Manual Supplement

Please click the [TA Manual Supplement](#) link to access the following important information supplemental to this Getting Started Guide:

- TA Instruments Trademarks
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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

NOTE: A NOTE highlights important information about equipment or procedures.

CAUTION: A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Regulatory Compliance

Safety Standards

For European Economic Area

The high volume pans have been evaluated to the requirements of the standards below and found to fall below the pressure times volume specification of 200 kPa L which is used to determine applicability. Calculated value = 0.285 kPa L.

EN 61010 Third Edition

EN 61010-2-010:2015 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For United States

UL61010-1:2012 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

UL61010-2-010:2015 Particular requirements for laboratory equipment for the heating of materials

For Canada

CAN/CSA-C22.2 No. 61010-1-12 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-C22.2 No. 61010-2-010-04 Particular requirements for laboratory equipment for the heating of materials.

Safety

WARNING: High volume pans are designed for evaluation of materials under self-generated atmospheres at pressures up to 250°C and pressures up to 3.8 MPa gauge (550 psig). As such, they are not intended to be a replacement for the pressure DSC cell, which is designed to evaluate materials under high pressures of specific purge gas (usually inert or reactive). Rather, the pans are primarily intended for the evaluation of materials for which suppression of volatilization of water or solvent (or sublimation) and a larger volume of sample are required to obtain good heat flow results.

With a 75 µL (nominal) aqueous sample,* these capsules can be used to about 250°C before the upper pressure limit is exceeded. However, since other samples may build up pressure more rapidly on heating and, more important, since the final rupture (failure) of the capsules at the upper pressure limit could result in damage to the DSC cell, it is strongly recommended that you use caution in deciding whether or not to evaluate a material in the high pressure capsules. Highly energetic materials such as pyrotechnics should never be run in these capsules.

*Do not exceed sample volume capacity (100 µL) of the pan (i.e., do not fill pan completely with sample).

WARNING: Do not touch the inside of the cell, or lean over it when inserting or removing a pan. Do not remove the pans at the end of an experiment until the pans have cooled to room temperature. Even then, handle the cooled pans with care. Gases produced during decomposition reactions at high temperature may not condense when cooled; therefore, the pan may remain under pressure at ambient temperature.


WARNING: Properly sealed pans may release pressure rapidly when internal pressure exceeds pan-seal capability. These pans should not be used for analysis of thermally unstable or explosive materials.

The high volume pans can be used with the Discovery DSC, Q2000/Q1000, or Q200/Q100 models configured with a Finned Air Cooling System (FACS), RCS, or LNCS. The AutoLid must be in position during experiments. For the DSC Q20/Q10 instrument and Discovery instruments without an Autosampler, the manual lid and safety clamp must be in position during experiments.

CAUTION: Properly sealed pans can withstand internal pressures up to 3.8 MPa gauge (550 psi), with temperatures up to 250°C. Improperly sealed pans can leak before reaching this pressure.

Instrument Symbols

The following labels are displayed for your protection:

Symbol	Explanation
	This symbol indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface.

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *DSC High Volume Pan Kit* manual contains cautions and warnings that must be followed for your own safety.

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Chapter 1:

Cleaning and Preparing the Pans

Cleaning the Pans

CAUTION: Handle the pan lid, pan bottom, O-ring, sample, and assembled pans with tweezers or other suitable tool. The body oils from your hands can give erroneous data.

Like the other pans provided for use with TA Instruments DSC systems, the high volume pans are manufactured to high quality standards. For most applications, these pans can be used as received; however, if the pans are used for high sensitivity experiments, an additional cleaning process is recommended before use. This procedure is taken from Appendix A of ASTM standard E1858, Test Method for Oxidative Induction Time of Hydrocarbons by Differential Scanning Calorimeters.

Follow the procedure given here to clean TA Instruments DSC High Pressure Capsules:

- 1 Place a maximum of 20 pans in a 250-mL Erlenmeyer flask that has been fitted with a glass stopper.
- 2 Add approximately 150 mL of reagent grade toluene (enough to cover the pans).
- 3 Swirl the flask, containing the capsules and toluene, for 0.5 to 2.0 minutes.
- 4 Let the flask stand for at least 1 minute.
- 5 Decant the water out of the flask.
- 6 Add approximately 150 mL of toluene (enough to cover the pans).
- 7 Swirl the flask, containing the pans and toluene, for 0.5 to 2.0 minutes.
- 8 Let the flask stand for at least 1 minute.
- 9 Decant the toluene out of the flask.
- 10 Repeat steps 2 through 5.
- 11 Add approximately 150 mL of reagent grade acetone after the second toluene wash.
- 12 Swirl the flask, containing the pans and acetone, for 0.5 to 2.0 minutes.
- 13 Let the flask stand for at least 1 minute.
- 14 Decant the acetone out of the flask.
- 15 Repeat steps 11 through 14.

NOTE: This procedure is best performed in a fume hood.

- 16 Rotate the flask—so that no pans adhere to the bottom or sides of the flask—as you flow nitrogen at 200 to 200 mL/min over the wet pans to drive off the excess solvent. This should take approximately 5 to 6 minutes.
- 17 Return the cleaned capsules to their storage container, and record the date they were cleaned.

Using the Tzero® Press to Encapsulate Samples

The following section describes the procedures for sealing samples using the Tzero DSC sample press.

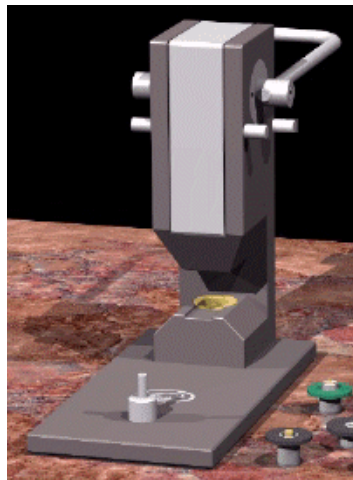


Figure 1 Tzero press.

WARNING: Do not carry the Tzero press by the handle!

The high volume pans require the use of the yellow die set shown in the figure below.



Figure 2 Upper die (left) and lower die (right).

There is one lower yellow die (right) and one upper yellow die (left). Both yellow dies must be used to seal the high volume pans.



Figure 3 High volume pan, o-ring, and lid.

Follow these basic instructions to use the Tzero sample press and the high volume pans:

- 1 Select the desired sample pan, matching lid, and o-ring, and obtain the matching die set. The die sets have colored rings and the pan/lid boxes have matching colored labels.

CAUTION: Handle the pan lid, pan bottom, O-ring, sample, and assembled pans with tweezers or another suitable tool. The body oils from your hands can give erroneous data. Ensure that any and all surfaces that the pans are placed on are clean and free of any possible contaminants.

- 2 Weigh the empty pan, o-ring, and lid.

NOTE: Only the Discovery DSC, Q2000, and Q1000 use the weight of the pan and lid (in Advanced Tzero mode) to improve the resolution and sensitivity of your heat flow measurement. If you carefully measure and enter the empty sample and reference pan weights into the software, the effect of the difference between the sample and reference pan weights will be compensated for and will improve your heat flow measurement. It is highly recommended that you take the extra time to do so. Users of the other Q Series, earlier DSC instruments, and the DSC25 do not need to perform this step.

- 3 Use tweezers to place the o-ring in the lid and push it all the way to the top of the lid.
- 4 Prepare the sample and carefully place the sample in the pan. If you are using a powder or granular sample, spread it evenly in the pan. Do not allow the sample to spill onto the lip of the pan.
- 5 Weigh the filled sample pan and lid and determine the sample weight. It is important to control the amount of sample you are using. Refer to additional material in the training courses for optimal sample size considerations.
- 6 Place the filled sample pan in the lower die and position the matching lid in place.



Figure 4

- 7 Push the press handle back and place the upper yellow die into the press. The upper die slides in easily and a magnet will hold it in place.
- 8 Place the lower yellow die with the prepared sample pan and lid into the press. Position the die with the raised platform to the rear of the press as shown in the figure below. Rotate the die slightly from side to side to ensure that it seats properly.

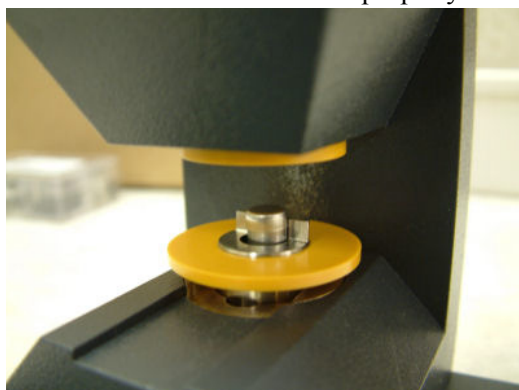


Figure 5 Position the die in the press.

- 9 Pull the handle forward until it stops to form the edges of the sealing surface prior to finishing the seal.

CAUTION: Make sure that you hold the handle firmly throughout the entire crimping procedure!

10 Push the handle back again and remove the lower die and pan.

Using the Blue Press to Encapsulate High Volume Pans

The first step when preparing high volume pans for sealing in the TA Instruments blue sample press is to set up the press for that pan type.

Setting Up the Blue Press for Sealing High Volume Pans

The TA Instruments blue Sample Encapsulating Press is used to seal samples in the DSC sample pans. The procedure below describes the steps required to convert a press that is currently set up for non-hermetic or hermetic pan sealing to the setup required for high volume pan sealing. (When you first receive your press it arrives pre-configured for sealing non-hermetic [crimped] pans.)

Refer to the figure below to identify the parts as you perform the following steps.

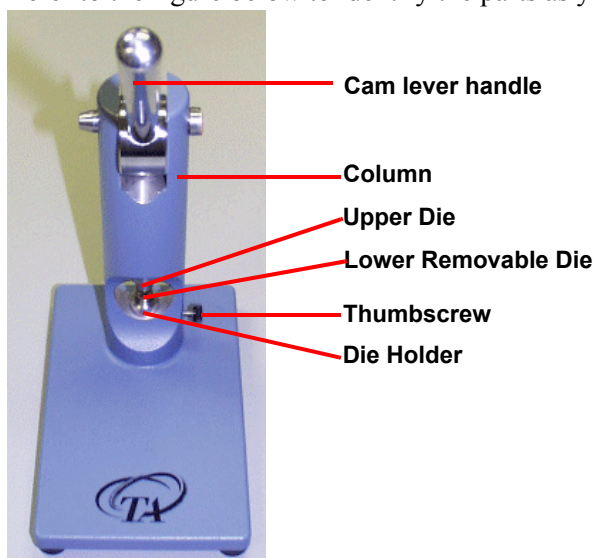


Figure 6 Blue press.

- 1 Remove the lower non-hermetic or hermetic die as follows:
 - a Raise the cam lever handle, then loosen the thumbscrew on the column of the sample press, which locks the lower die in place.
 - b Turn the press over so that the bottom is accessible.

- c Lower (loosen) the die holder by turning the base screw on the bottom of the press counterclockwise.



Figure 7 Base screw.

- d Return the press to its upright position, then lift the lower non-hermetic or hermetic die to remove it from the die holder.

- 2 Place the lower high volume die into the die holder with the larger end facing up.

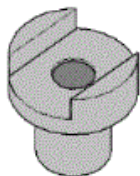


Figure 8 Lower high volume die.

- 3 ***When changing from either hermetic or non-hermetic setup to high volume sealing:*** Since the same upper die is used for both hermetic and non-hermetic pans, the procedure for converting to the upper high volume die is the same regardless of the current press setup. To change the upper die, slide the upper high volume die onto the extended portion of the upper hermetic/non-hermetic die and tighten the setscrew.
- 4 Adjust the position of the lower die relative to the upper die as follows:
- a Pull the sample press cam lever handle all the way down until it rests on the column.
 - b Turn the press over so that the bottom is accessible.
 - c Raise the lower die by turning the base screw clockwise until the lower die meets the upper die and the cam lever handle is horizontal.
 - d Return the press to its upright position and tighten the thumbscrew on the column to lock the die holder in place.

Sealing High Volume Pans

- 1 Before using the sample encapsulating press, ensure that it is set up for high volume pans. See [“Setting Up the Blue Press for Sealing High Volume Pans” on page 11](#)
- 2 Practice sealing a few sample pans to become familiar with this procedure before encapsulating your samples.
- 3 If quantitative work will be done, weigh the sample pan, lid, and o-ring and record the value.

NOTE: When doing quantitative work, use tweezers to handle the sample pan, lid, and o-ring. Touching them with your fingers could leave residue that could affect your results.

NOTE: For most DSC experiments, the parameter of primary interest is sample weight. This can either be obtained by weighing the pan and lid and then weighing the pan and lid again once the sample has been encapsulated, or by taring out the weight of the pan and lid on a microbalance before weighing the encapsulated sample. If T4P heat flow is being collected, however, the former approach is required because the pan/lid weights for sample and reference pans must be known to correct for pan-sensor interactions.

- 4 Place the sample in the pan. Make sure no sample gets on the lip of the pan where the o-ring will rest. (Since high volume pans are primarily used to evaluate liquid samples, a micropipette is an effective way to introduce sample into the pan without contaminating the lip.)
- 5 Place the pan on the indentation in the bottom die of the sample press.
- 6 Push the o-ring all the way down inside the lid. Place the lid on the pan.

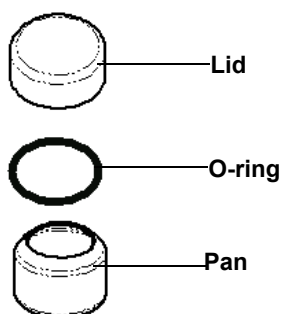


Figure 9 Lid, o-ring, pan.

- 7 Pull the sample press lever forward until the two die sections meet.
- 8 Raise the lever and remove the pan with tweezers.
- 9 Inspect the pan. The bottom of the pan should be smooth and the o-ring should not be visible.
- 10 For quantitative work, weigh the sealed pan containing the sample and determine the sample weight by subtracting the weight of the empty pan, lid, and o-ring (determined in step 3).
- 11 Follow steps 5 through 9 to prepare an empty reference pan. The same care should be taken with the reference pan as you did with the sample pan. (If heat capacity imbalance is undesirable in the final results, an equivalent volume of “inert” solvent can be sealed in the reference pan. For example, when evaluating weak transitions in aqueous biological samples, sealing an equivalent volume of water or buffer in the reference pan will result in flatter baselines and may make interpretation of the results easier.)

Weighing the Sample

Before you can enter the sample size in your experimental parameters information, you will need to weigh the sample.

NOTE: Try to keep the sample mass as low as practical.

When handling the sealed pan, be sure to hold it level, especially when using liquid and viscous samples. The sample will then remain in the pan, and will not adhere to the underside of the lid.

Weigh the capsule with the seal and sample. Determine the sample weight by subtracting the weight of the empty capsule (found in step 1) from the total weight:

$$\text{Weight}_{\text{step 8}} - \text{Weight}_{\text{step 1}} = \text{Sample weight}$$

The high volume pan is now ready to run in your DSC cell.

Chapter 2:

Calibration and Maintenance

Calibrating the DSC

In order to get accurate experimental results, your DSC system should be recalibrated for cell constant and temperature using the sealed high pressure capsules. Follow these directions for calibration. Refer to the appropriate section for your instrument.

All Discovery DSC Instrument Models

Follow the directions in the online help for detailed instructions on calibrating your Discovery DSC system.

Q200/Q100/Q20A or Q2000/Q1000 Instruments

- 1 Use an empty sealed capsule as a reference. Place it in the cell.
- 2 Weigh an 8 to 10 mg sample of indium.
- 3 Place a thin layer of alumina (about 20 mg) in the sample capsule. Then place the indium sample on top of the alumina.

NOTE: Normally DSC heat flow and temperature calibration are accomplished by evaluating the calibration material (usually indium) under the same experimental conditions as the subsequent sample materials. With the increased resolution of the Q DSC T4 heat flow, however, the calibration conditions must be changed slightly to account for the small indium sample mass relative to the large mass of the low thermal conductivity stainless steel high pressure capsule. The addition of alumina helps counter balance that difference and provides a calibration better suited to the kinds of material run in these capsules.

No alumina is necessary for T1 heat flow calibration.

- 4 Seal the pan.
- 5 Close the AutoLid before proceeding. This step is very important for safety reasons.
- 6 Use the recommended calibration methods to calibrate the system (refer to the DSC and the instrument control online documentation for information). Make sure that calibration of the Tzero cell resistance and capacitance values are performed.

NOTE: Return the instrument to standard mode before running your experiments.

Q20/Q10 Instruments

- 1 Use an empty sealed capsule as a reference. Place it in the cell.
- 2 Weigh an 8 to 10 mg sample of indium.

- 3 Seal the capsule and place it inside the cell.
- 4 Install the safety clamp on the manual lid as follows:
 - a Unscrew the knob from the top of the manual lid.
 - b Place the hole in the middle of the safety clamp over the exposed screw.
 - c Screw the knob back onto the manual lid and tighten.
- 5 Squeeze the tabs on the safety clamp toward the center (as shown in the figure below) while lowering the manual lid onto the cell.



Figure 10 Squeeze tabs.

IMPORTANT: Make sure the hooks at either end are fully engaged in the vent holes as shown. Pull up slightly on the lid to make sure it is secure

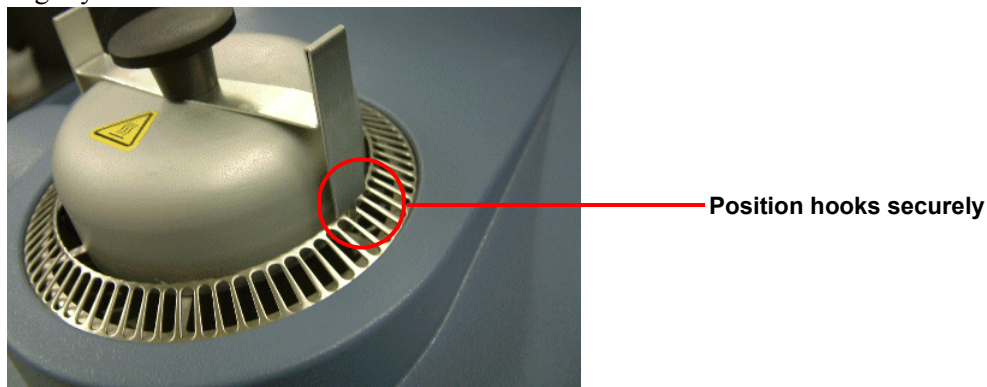


Figure 11 Position hooks securely.

WARNING: Do not attempt to run the DSC Q20/ Q10 using the high volume pans unless the safety clamp is securely in place as shown.

- 6 Use the recommended calibration methods to calibrate the system. The Calibration Wizard can be used to conveniently calibrate the DSC Q20/Q10. Only T1 heat flow calibration is possible on the Q20/Q10.
- 7 Return the instrument to standard mode.

Preparing the Instrument

No special preparation of the instrument is needed when the Discovery DSC, DSC Q2000/Q1000, or Q200/Q100 is loaded with a high volume pan. But, you must make sure that the AutoLid or manual lid is closed at all times when running an experiment with a high volume pan loaded.

When the DSC Q20/Q10 is loaded with a high volume pan, you must make sure the safety clamp is securely installed on the manual lid before beginning the experiments using high volume pans.

Running Experiments

After you calibrate the DSC using the High Volume Pan and return the instrument to standard mode, load the sample and reference capsules into the DSC cell. Refer to your instrument's online help for detailed information on running experiments.

WARNING: DO NOT EXCEED 250°C when using the high volume pans. Properly sealed pans will explode when internal pressure exceeds pan-seal pressure capability.

Discovery DSC, DSC Q200/Q100, and Q2000/Q1000 Instruments: Be sure to close the AutoLid before beginning the experiments using high volume pans.

Discovery DSC without Autosampler and DSC Q20/Q10 Instruments: Make sure the safety clamp is securely installed on the manual lid before beginning the experiments using high volume pans.

When using the DSC high volume pans to evaluate larger samples with high heat capacities (e.g., dilute aqueous protein solutions), it may be beneficial to add “inert” material (e.g., water) to the reference pan. This will balance the heat capacities, producing a flatter baseline and allowing weak transitions of interest to be observed.

WARNING: Do not touch the inside of the cell or lean over it when inserting or removing a pan. Do not remove the pans at the end of an experiment until the pans have cooled to room temperature. Even then, handle the cooled pans with care. Gases produced during decomposition reactions at high temperature may not condense when cooled; therefore, the pan may remain under pressure at ambient temperature.

Maintaining the Sample Press

Wipe the Sample Encapsulating Press clean with a soft cloth that has been dampened with a dilute laboratory detergent solution when needed.

Specifications

Table 1 contains the technical specifications for High Volume Pans and seals.

Table 1: Specifications for High Volume Pans and Seals

Item	Specification
Pressure capability	3.8 MPa gauge (550 psig)
Temperature limit aqueous solutions	250°C
Pan Volume Material	100 µL (max) 302 SST
O-ring material	Viton

Replacement Parts

When ordering replacement parts for the high volume pans, use the following part numbers.

Table 2: High Volume Pan Parts

Part Number	Description
900825.902	DSC High Volume Pan Kit (includes 100 pans, 100 lids, and 100 o-rings)
900824.901	Die set for DSC High Volume Pans
900906.901	Aluminum Oxide (Alumina) Reference Material