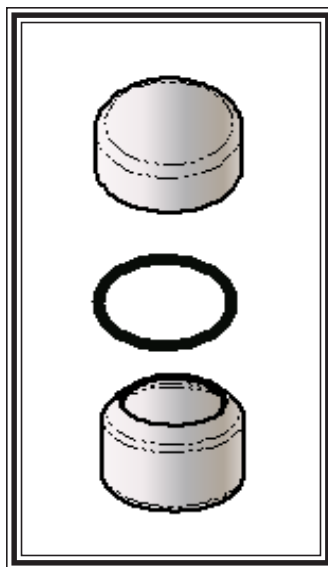


DSC High Volume Pan Kit



Operator's Guide

PN 900826.001 Rev. K
Issued February 2007



Notice

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WARNING: These pans should not be used for analysis of thermally unstable or explosive materials. The high volume pans are not designed to be used with the DSC Dual Sample Cell. You must always use extreme care for your own and others' safety when handling materials that can decompose violently.

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Overview

This booklet describes the procedures needed to prepare high volume sample pans for use in the DSC cell. To seal the high volume pans, you will use one of TA Instruments Sample Encapsulating Presses. There are three types that can be used: The gray Tzero press, the blue press, or the beige press. Refer to the section for the press you are using. Be sure to read the following section on safety carefully before proceeding further.

- Using the Tzero Press - see page 10
- Using the Blue or Beige Press - see page 12

Safety

The high volume pans have been evaluated as to European Union requirements in EN61010-1/1993 + A2/1995 and EN61010-2-010/1994 and have been found to fall below the pressure (200 kPa L) times volume (0.285 kPa L) calculation, which is used to determine applicability.

Safety Label



The label shown to the left is displayed on the metal bell jar that comes with the DSC High Volume Pan Kit for your protection. This label indicates that a hot surface may be present. Take care not to touch this area or to allow any material that may melt or burn to come in contact with this hot surface.

The notes, cautions, and warnings on the following pages are provided to prevent accidents and ensure safe laboratory practice. Read this entire section carefully before using the materials provided in this kit, and follow all instructions. For protection, wear safety glasses at all times.



WARNING: Do not use the DSC high volume pans in the DSC dual sample cell.



WARNING: High volume pans are designed for evaluation of materials *under self-generated atmospheres* at temperatures 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 a 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 pans 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 pans 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 volume pans. Highly energetic materials, such as pyrotechnics, should never be run in these pans.

*** 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.

DSC Q Series: The high volume pans can be used with the 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, the manual lid and safety clamp must be in position during experiments.

DSC 2920/2910/2010: Be sure to use the following supplied safety devices when running any experiments with these high volume pans on these instruments: For cells without a heat exchanger—Metal bell jar and hold-down bracket [DSC 2010] or metal bell jar and hold-down knobs [DSC 2910 and 2920]. For all cells (DSC 2010, 2910, or 2920) with an RCS or LNCA heat exchanger—a safety lid.

NOTE: The safety lid provided will not work for 2920/2910/2010 RCS (Refrigerated Cooling System) heat exchangers that have been modified with a “prototype” heat transfer sleeve. RCS units with serial numbers lower than 1641 may contain this prototype sleeve. Contact our service department at (302) 427-4050 or your local TA Instruments Representative for further details on how to modify those RCS units for use with high volume pans.



WARNING: The DSC high volume pans should not be used with an older-style DSC Mechanical Cooling Accessory (MCA). The MCA (PN 990460.901/.902), which was discontinued as a product in 1995, does not work with the safety lid included in the DSC High Volume Pan Kit.



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

Cleaning the Pans



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.

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 volume pans:

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 water (enough to cover the pans).
3. Swirl the flask, containing the pans and water, 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 150 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 pans 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 shown in the figure to the right.



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.



Upper Die

Lower Die

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. The figure to the right shows the 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.



High Volume Pan, O-Ring, and Lid



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 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 and earlier DSC instruments 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.

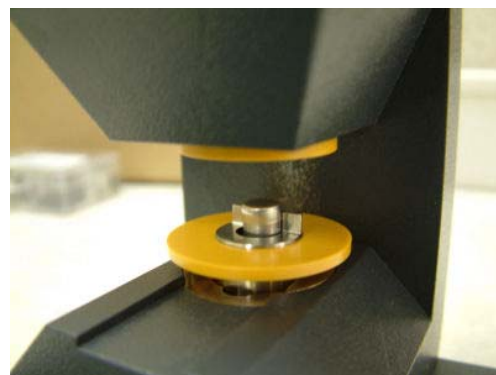
5. Weigh the filled sample pan and lid and determine the sample weight. It is important to control the amount of sample you are using. Refer to additional material in the training courses for optimal sample size considerations.

6. Place the filled sample pan in the lower die and position the matching lid in place.



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 to the right. Rotate the die slightly from side to side to ensure that it seats properly.



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.

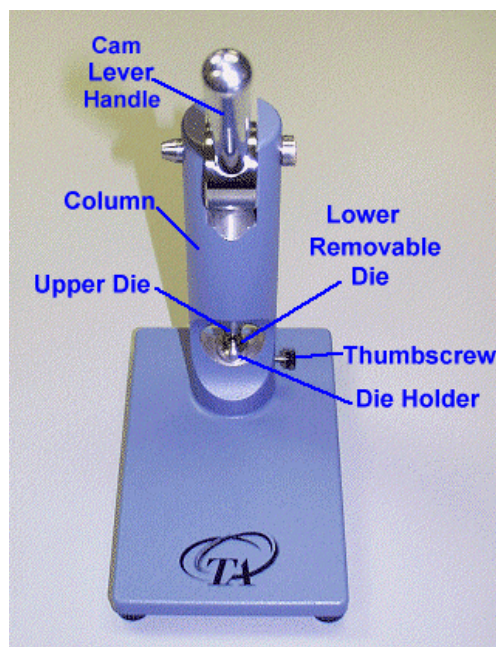
Using the Blue or Beige Press to Encapsulate High Volume Pans

The first step when preparing high volume pans for sealing in the TA Instruments blue or beige sample presses is to set up the press for that pan type using the instructions found in the section appropriate to your press.

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 nonhermetic or hermetic pan sealing to the setup required for high volume pan sealing. [When you first receive your press it arrives preconfigured for sealing nonhermetic (crimped) pans.]

NOTE: The pans sealed with this press may be used in either a Q Series or 2900 series DSC instrument.



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

1. Remove the lower nonhermetic 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 as seen in the figure to the right.
 - d. Return the press to its upright position, then lift the lower nonhermetic or hermetic die to remove it from the die holder.
2. Place the lower high volume die (see figure to the right) into the die holder with the larger end facing up.
3. **When changing from either hermetic or nonhermetic setup to high volume sealing:** Since the same upper die is used for both hermetic and nonhermetic 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/nonhermetic 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.

Setting Up the Beige Press for Sealing High Volume Pans

The TA Instruments beige Sample Encapsulating Press (shown here) 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 hermetic or nonhermetic pan sealing to the setup required for high volume sealing. [When you first receive your press it arrives preconfigured for sealing nonhermetic (crimped) pans.]

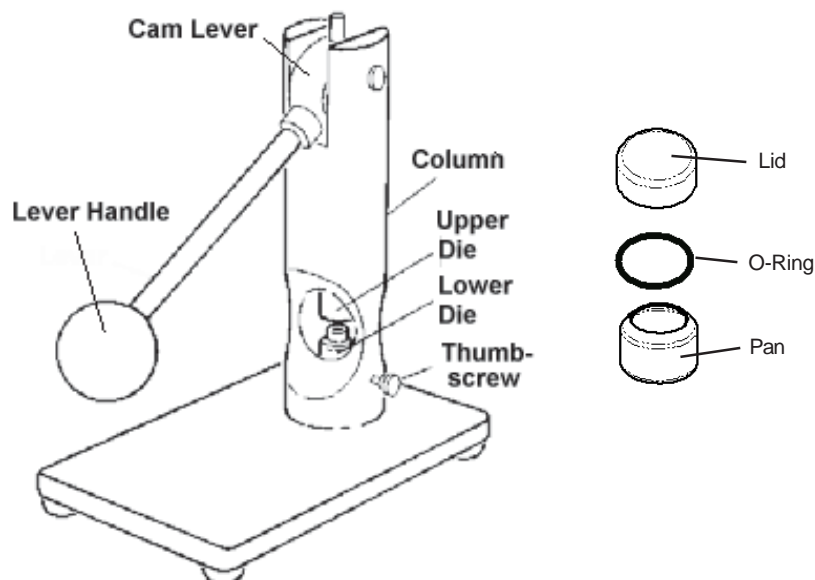


CAUTION: Handle the pan lid, pan bottom, O-ring, sample, and assembled pans with tweezers or another suitable tool. The body oils from your hands can give erroneous data.

Refer to the figures and follow the instructions in this section to prepare sample pans for the DSC high volume pan experiments.

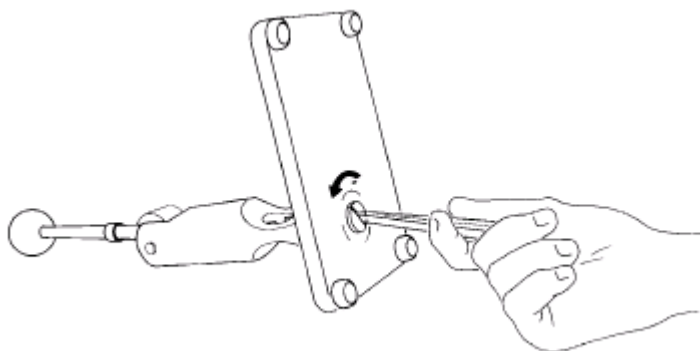
NOTE: The pans sealed with this press may be used in either a Q Series or 2900 series DSC instrument.

Refer to the figure below to identify the parts.



1. Remove the lower nonhermetic 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 as seen in the figure to the right.
- d. Return the press to its upright position, then lift the lower nonhermetic or hermetic die to remove it from the die holder.

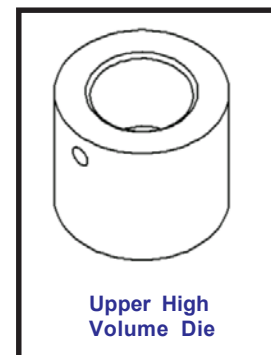


2. Place the lower high volume die (see figure below) into the die holder with the larger end facing up.



3. Mount the high volume upper die as follows:

- a. **From a press setup for hermetic pans:** Slide the high volume upper die onto the extended portion of the upper hermetic die and tighten the setscrew.
- b. **From a press setup for nonhermetic pans:** Remove the upper nonhermetic die by loosening the setscrew holding it to the extended portion of the upper hermetic die. Then slide the high volume upper die on in its place. 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 Volumes Pans with the Blue or Beige Press

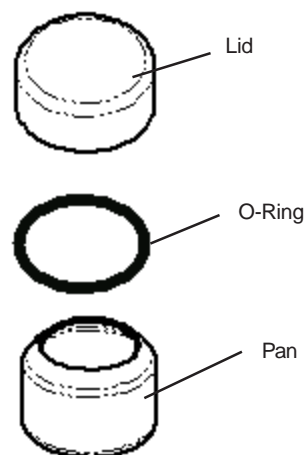
The procedure for preparing high volume sample pans is the same for both the beige and blue sample presses.

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" or "Setting Up the Beige Press for Sealing High Volume Pans."
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.
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 pan with the seal and sample. Determine the sample weight by subtracting the weight of the empty pan, lid, and O-ring (found in step 1 on the previous page) from the total weight:

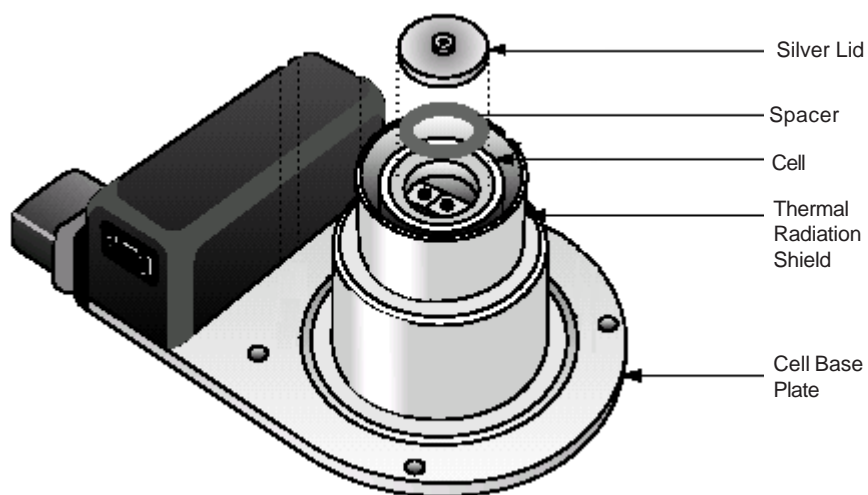
$$\text{Weight}_{\text{with sample}} - \text{Weight}_{\text{without sample}} = \text{Sample weight}$$

The high volume pan is now ready to run in your DSC cell. See the instructions on the next page to prepare the cell.

Preparing the 2920/2910/2010 Cell

Before calibrating or running an experiment using the sealed high volume pans, you will need to perform a simple step to prepare the DSC cell.

First remove the glass cover and lids. Then, place the supplied spacer down inside the open cell as shown in the figure below (DSC 2920 cell shown). The spacer should rest on the rim inside the cell. The silver spacer must be used to ensure that the top of the high volume pan does not come in contact with the lid. If the silver spacer is not used, sensitivity will be degraded and the ability to detect weak transitions will be impaired. After installation of the spacer, you can proceed with the calibration instructions in the next section.



Installing the Spacer in the Cell (DSC 2920 Shown)

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 volume pans. Follow these directions for calibration. Refer to the appropriate section for your instrument.

DSC 2920/2910/2010 Instruments

1. Use an empty sealed pan as a reference.
2. Seal an 8-mg to 10-mg indium metal sample in a high volume pan.
3. Place the pans inside the cell.
4. Perform the following steps:
 - a. *With a cooling accessory and a heat exchanger:* Install the safety lid.
 - b. *Without a heat exchanger:* Install the metal bell jar over the cell. Then install the appropriate safety device for your instrument—the hold-down bracket for the DSC 2010 (see page 23) or the hold-down knobs for the DSC 2920 (see page 23). This will ensure that the metal bell jar provides the desired safety enclosure.



WARNING: Properly sealed pans may release pressure rapidly when internal pressure exceeds pan-seal pressure capability. Be sure to use the supplied safety devices (metal bell jar for cells without a heat exchanger, or safety lid for cells using a heat exchanger) when running any experiments or calibrating with the high volume pans. Make sure that you read the Warnings on page 8 before proceeding, if you are using a DSC cell with an RCS heat exchanger or with a Mechanical Cooling Accessory.

5. Use the recommended calibration methods to calibrate the system (refer to the DSC and the instrument control online documentation for information). If additional temperature calibration points are required, make sure that you use the high volume pan when gathering data.
6. Return the instrument to standard mode before running your experiments.

DSC Q200/Q100 or Q2000/Q1000 Instruments

1. Use an empty sealed pan as a reference.
2. Weigh an 8-mg to 10-mg sample of indium.
3. Place a thin layer of alumina (about 20 mg) in the sample pan. 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 volume pan. The addition of alumina helps counter balance that difference and provides a calibration better suited to the kinds of material run in these pans.

No alumina is necessary for T1 heat flow calibration.

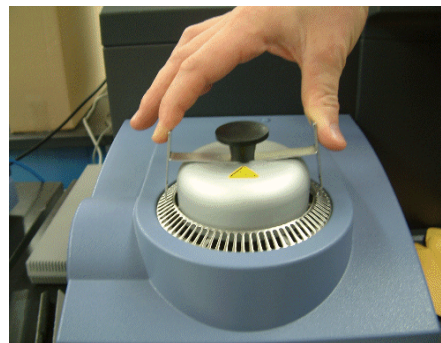
4. Seal the pan.
5. Close the AutoLid before proceeding. This step is very important for safety reasons.
6. Use the recommended calibration methods to calibrate the system (refer to the DSC and the instrument control online documentation for information). Make sure that calibration of the Tzero cell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

NOTE: The Calibration Wizard can be used to conveniently calibrate the DSC Q Series instruments.

7. Return the instrument to standard mode before running your experiments.

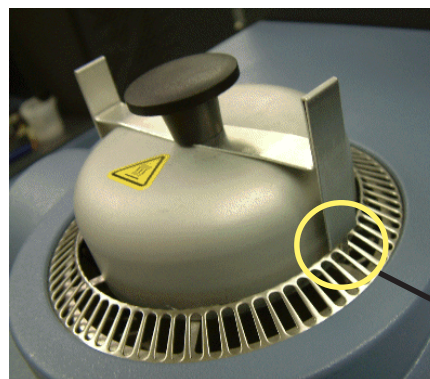
DSC Q20/Q10 Instrument

1. Use an empty sealed pan as a reference. Place it in the cell.
2. Weigh an 8-mg to 10-mg sample of indium.
3. Seal the pan 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 here) while you lower the manual lid onto the cell. **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.



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.



Position
Hooks
Securely

Running Experiments

After you calibrate the DSC using the high volume pan and return the instrument to standard mode, load the sample and reference pans into the DSC cell.



WARNING: DO NOT EXCEED 250°C when using the high volume pans.

Properly sealed pans may release pressure rapidly when internal pressure exceeds pan-seal pressure capability.

DSC 2920/2910/201 Instruments: Be sure to use the supplied safety devices, as shown in the figures on the next page, when running any experiments or calibrating with the high volume pans.

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

DSC Q20/Q10 Instruments: Make sure the safety clamp is securely installed on the manual lid before beginning the experiments using high volume pans.



WARNING: Do not use the high volume pans in a DSC dual sample cell. The high volume pans are not designed to be used with that cell.



WARNING: Make sure that you read the Warning on page 11 before proceeding, if you are using a DSC cell with an RCS heat exchanger or Mechanical Cooling Accessory.

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.

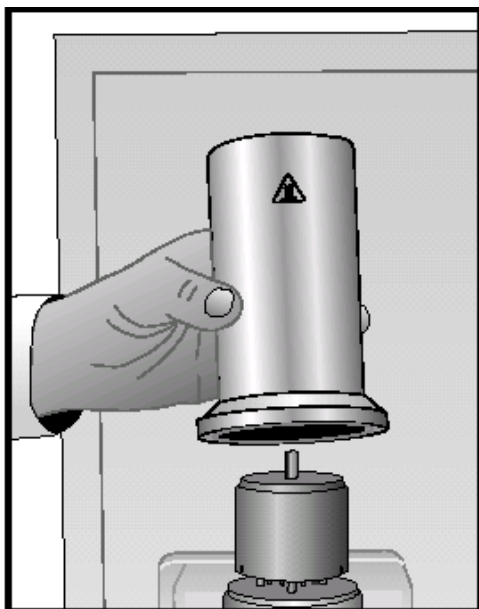


WARNING: Do not touch the inside of the cell or lean over it when inserting or removing a pan. Do not remove the pans at the end of an experiment until the pans have cooled to room temperature. Even then, handle the cooled pans with care. Gases produced during decomposition reactions at high temperature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

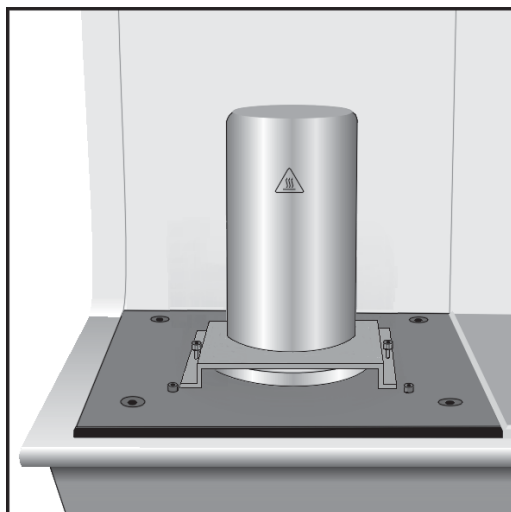
Preparing the DSC 2920/2910/2010 Instruments

Without Heat Exchanger

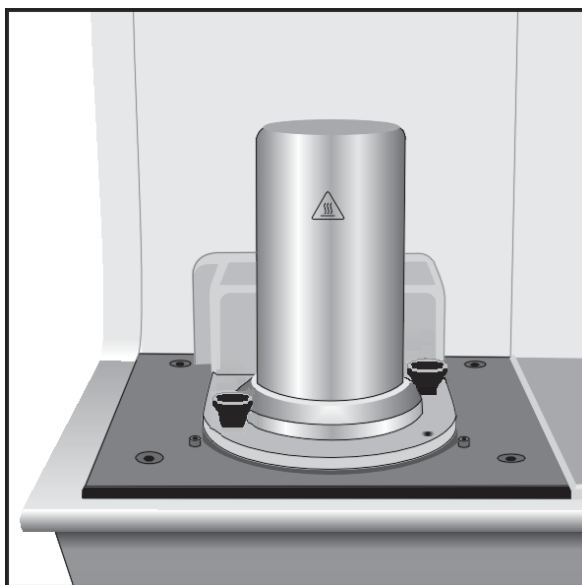
When using the DSC without a heat exchanger you must install the metal bell jar on the DSC 2920, 2910, or 2010 instruments as shown in the figures below, then secure it using either the bracket (2010) or hold-down knobs (2910 and 2920).



Using the Metal Bell Jar with the DSC
2920, 2910, or 2010 Cell



Securing the Metal Bell Jar on
the DSC 2010 Using the Bracket



Securing the Metal Bell Jar on the DSC
2920 (or 2910) Using the Hold-Down Knobs

With a Heat Exchanger

When using the DSC with an RCS or LNCA heat exchanger you must install the safety lid on the heat exchanger as shown in the figure below.



Using the Safety Lid with the DSC
Cell and the Heat Exchanger

Preparing the DSC Q Series Instruments

No special preparation of the instrument is needed when the 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. See page 21 for instructions.

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
High Volume Pan Specifications

Pressure capability	3.8 MPa gauge (550 psig)
Temperature limit for 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
List of DSC 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

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