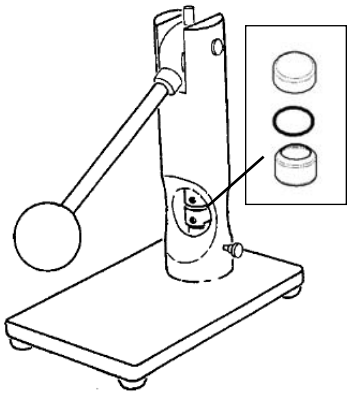
DSC
High Volume
Pan Kit



Operator's Guide

PN 900826.001 Rev. J
Issued Febraury 2004

109 Lukens Drive
New Castle, DE 19720

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(continued on next page)

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Method and Apparatus for Modulated Differential Analysis (MDSC®) describes proprietary technology patented by TA Instruments Waters—LLC (U.S. Patent Nos. 5,224,775; 5,248,199; 5,346,306; 5,439,291. Additional Patent Nos. CA 2,089,225; JP 2,966,691 and 3,299,575; and BE, DE, EP, GB, IT, NL 0559362).

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Method and Apparatus for Performing Localized Thermal and Sub-Surface Imaging by Scanning Thermal Microscopy describes proprietary technology patented by TA Instruments Waters—LLC (U.S. Patent Nos. 6,095,679 and 6,491,425. Additional Patent No. JP 3,229,329).

Modulated-Temperature Thermomechanical Analysis describes proprietary technology patented by TA Instruments—Waters LLC (U.S. Patent No. 6,007,240).

(continued on next page)

Method and Apparatus for High Resolution Analysis of the Composition of a Material describes proprietary technology patented by TA Instruments—Waters LLC (U.S. Patent No. 5,368,391 and 5,165,792. Additional Patent Nos. CA 2,051,578 and DE, EP, FR, GB, IT 0494492).

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Dynamic and Thermal Mechanical Analyzer Having an Optical Encoder with Diffraction Grating and a Linear Permanent Magnet Motor describes proprietary technology patented by TA Instruments—Waters LLC (U.S. Patent Nos. 5,710,426 and 5,915,283).

Heater Control Evaporation of Cryogenic Fluids for Cooling Thermal Analysis Instruments describes proprietary technology patented by TA Instruments Waters—LLC (U.S. Patent No. 5,973,299).

Differential Scanning Calorimeter describes proprietary technology patented by TA Instruments Waters—LLC (U.S. Patent No. 5,842,788).

Method and Apparatus for Performing Chemical Analysis Using Imaging by Scanning Thermal Microscopy describes proprietary technology patented by TA Instruments Waters—LLC (U.S. Patent No. 6,405,137).

Thermogravimetric Apparatus describes proprietary technology patented by TA Instruments—Waters LLC (U.S. Patent No. 5,321,719).

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Method and Apparatus for High Spatial Resolution Spectroscopic Microscopy describes proprietary technology patented by TA Instruments Waters—LLC (U.S. Patent No. 6,260,997).

Method and Apparatus for Localized Mechano-Thermal Analysis Combined with Scanning Probe Microscopy describes proprietary technology patented by TA Instruments Waters—LLC (U.S. Patent No. 6,200,022 and GB Patent No. 2,332,949).

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Thermal Analysis Assembly with Distributed Resistance and Integral Flange for Mounting Various Cooling Devices describes proprietary technology patented by TA Instruments Waters—LLC (U.S. Patent No. 6,523,998).

Liquid Nitrogen Cooling System describes proprietary technology patented by TA Instruments Waters—LLC (U.S. Patent No. 6,578,367).

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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.

Table of Contents

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 the Sample Encapsulating Press. Be sure to read the following section on safety carefully before proceeding further.

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 at 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 Q1000 or Q100 models configured with a Finned Air Cooling System (FACS), RCS, or LNCS. The AutoLid must be in position during experiments. For the DSC Q10 instrument, the manual lid and safety clamp must be in position during experiments.**

***DSC 2XXX Series*: Be sure to use the following supplied safety devices when running any experiments with these high volume pans on the DSC 2000 series 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 2XXX Series 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 reagent grade xylene (enough to cover the pans).
7. Swirl the flask, containing the pans and xylene, for 0.5 to 2.0 minutes.
8. Let the flask stand for at least 1 minute.
9. Decant the xylene out of the flask.

10. Repeat steps 2 through 5.

11. Add approximately 150 mL of reagent grade acetone after the second xylene 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.

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.

Preparing a Pan

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 Figure 1 and follow the instructions in this section to prepare sample pans for the DSC high volume pan experiments.

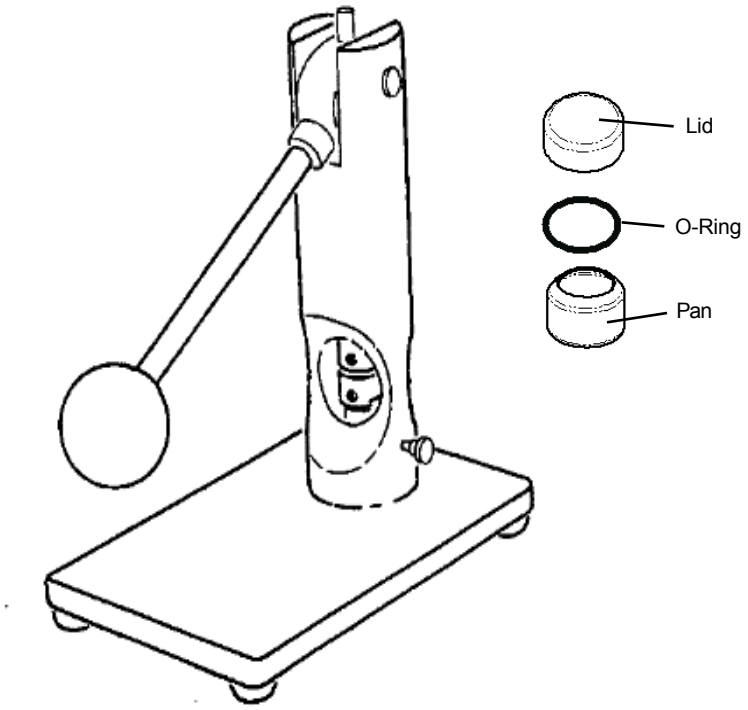


Figure 1
Sample Encapsulating Press and
DSC High Volume Pans

Preparing the Sample Press

The DSC high volume pans can be sealed using the Sample Encapsulating Press, which is used for other types of DSC pans.

The Sample Encapsulating Press is shipped with the upper nonhermetic die installed. To set up the press to make high volume sample pans, proceed as follows:

1. Loosen the lower setscrew on the upper die set. Remove the bottom section of the upper die (leave the top section in place).

2. Loosen the thumbscrew on the column of the Sample Press (see Figure 2).

3. Lower the lower die holder by turning the base screw on the bottom of the press counterclockwise.

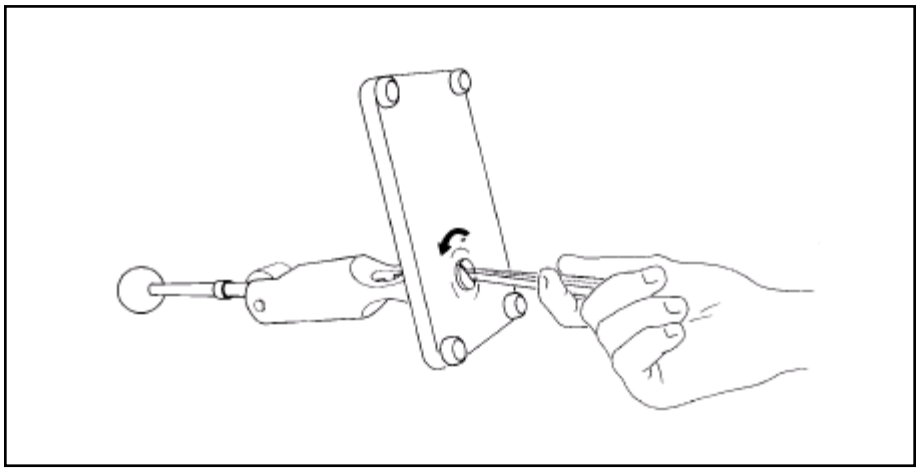


Figure 2
Lowering the Base Screw

4. Lift the lower die and remove it from the die holder.

5. Place the lower high volume pan die (Figure 3) into the lower die holder.

6. Place the upper high volume pan die into the press under the existing upper die (there will now be two upper die sections in place).

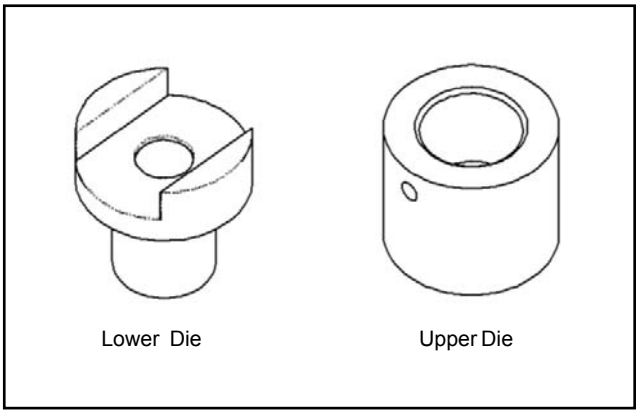


Figure 3
The DSC High Volume Pan Dies

7. Tighten the set screw on the top die section. No adjustment is required.

Loading and Sealing the Pans

After the Sample Encapsulating Press has been set up with the appropriate dies installed according to the previous instructions, follow these steps to load and seal the high volume pans.

1. Weigh the high volume pan, lid, and O-ring before placing the sample inside to get the total mass.
2. Push the O-ring all the way down into the lid.
3. Load the sample into the pan.
4. Place the pan on the indentation in the bottom die of the sample press.
5. Place the high volume lid on the pan with the sample.

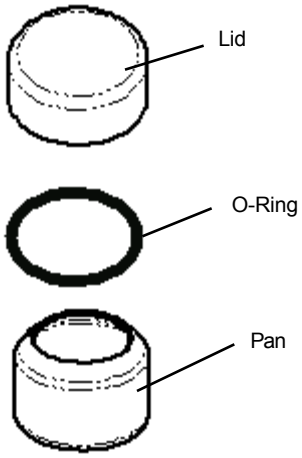


Figure 4
DSC High Volume Lid, Pan, and O-Ring

6. Pull the sample press lever down until the two die sections meet. The pan is now sealed and the O-ring should not be visible.

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 2XXX 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 (2XXX series DSC's) and lids (all DSC's). 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.

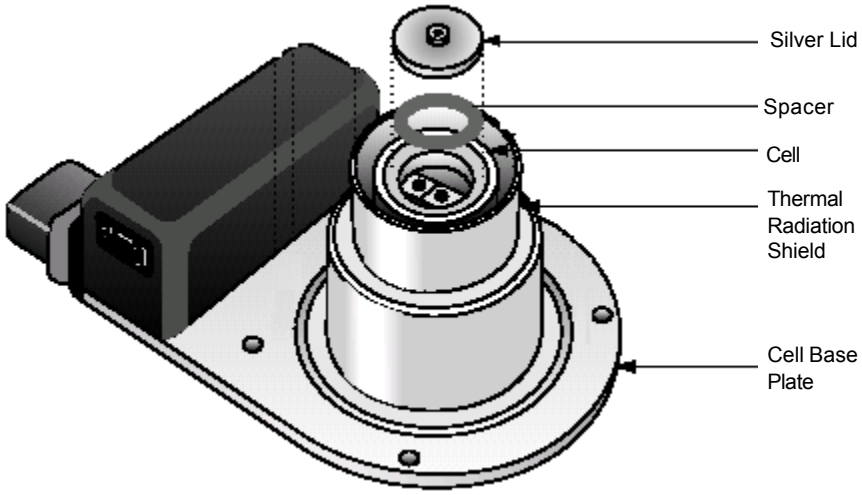


Figure 5
Installing the Spacer in the Cell (DSC 2920 Shown)

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 2XXX Series 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 Figure 7) or the hold-down knobs for the DSC 2920 (see Figure 8). 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 11 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 Q100 or 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.

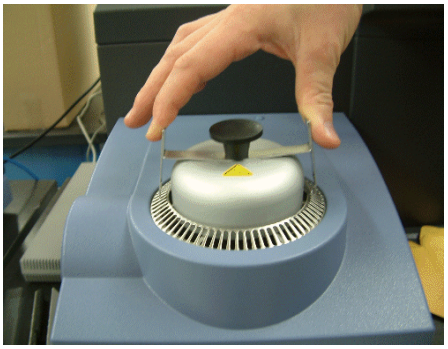
6. Use the recommended calibration methods to calibrate the system (refer to the DSC and the instrument control online documentation for information). Make sure that calibration of the Tzero cell resistance and capacitance values are performed.

   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 Q10 Instrument

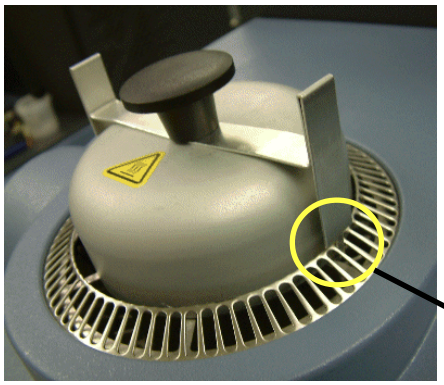
1. Use an empty sealed pan as a reference. Place it in the cell.
2. Weigh an 8-mg to 10-mg sample of indium.
3. Seal the pan and place it inside the cell.
4. Install the safety clamp on the manual lid as follows:
   a. Unscrew the knob from the top of the manual lid.
   b. Place the hole in the middle 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 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 Q10. Only T1 heat flow calibration is possible on the Q10.
7. Return the instrument to standard mode.



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 2XXX Series Instruments: **Be sure to use the supplied safety devices, as shown in Figures 6 through 9 on pages 26 through 28, when running any experiments or calibrating with the high volume pans.**

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

DSC 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 2000 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 Figure 6, then secure it using either the bracket (2010) or hold-down knobs (2910 and 2920). See Figures 7 and 8.

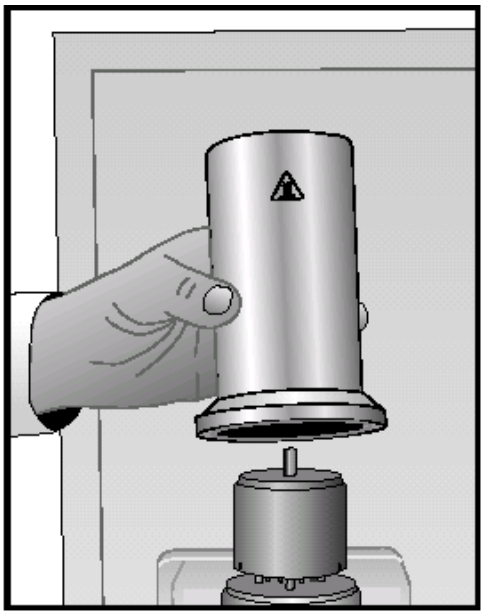


Figure 6
Using the Metal Bell Jar with the DSC 2920, 2910, or 2010 Cell

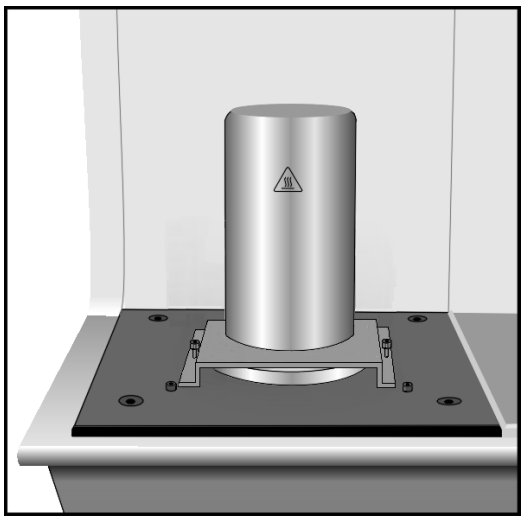


Figure 7
Securing the Metal Bell Jar on
the DSC 2010 Using the Bracket

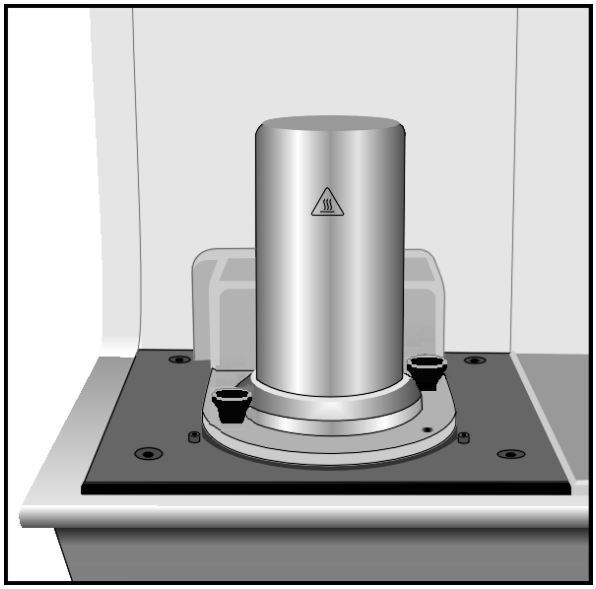


Figure 8
Securing the Metal Bell Jar on the DSC
2920 (or 2910) Using the Hold-Down Knobs

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 Figure 9 below.

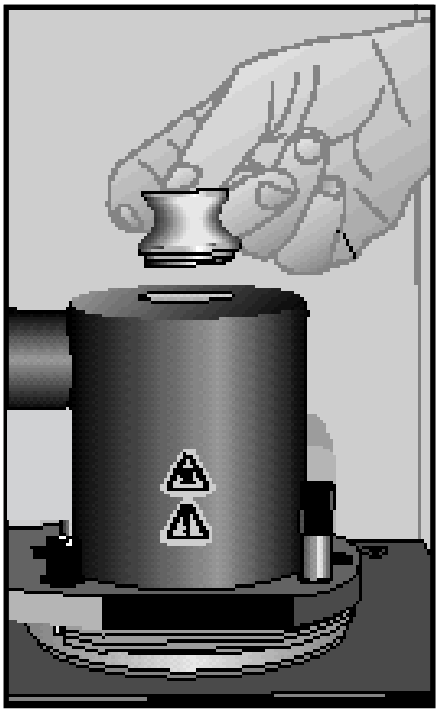


Figure 9
Using the Safety Lid with the DSC
Cell and the Heat Exchanger

Preparing the DSC Q Series Instruments

No special preparation of the instrument is needed when the DSC Q1000 or 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 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 24 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	100 µL (max)
Material	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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