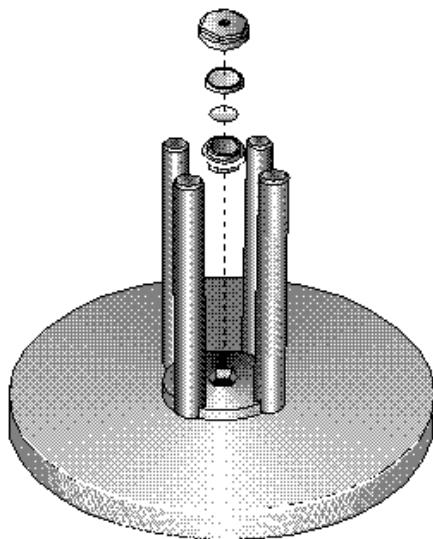


DSC High Pressure Capsule Kit



Operator's Guide

PN 900813.001 Rev. G
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New Castle, DE 19720

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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).

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WARNING: These capsules should not be used for analysis of thermally unstable or explosive materials. 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 pressure sample capsules for use in the DSC cell. Be sure to read the following section on safety carefully before proceeding further.

Safety

The high pressure capsules 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 x volume product that 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-Pressure Capsule 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 following notes, cautions, and warnings are provided to prevent accident and ensure safe laboratory practice. Read this section, beginning on the next page, carefully before using the materials provided in this kit, and follow all instructions. For protection, wear safety glasses at all times.



WARNING: High pressure capsules are designed for evaluation of materials *under self-generated atmospheres* at pressures up to 10 MPa (1450 psi). 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 capsules are primarily intended for the evaluation of materials for which suppression of volatilization of water or solvent (or sublimation) is required to obtain good heat flow results.



WARNING (continued): With a 10- μ L (nominal) aqueous sample,* these capsules can be used to about 300°C before the upper 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 (35 μ L) of the capsule (i.e., do not fill capsule completely with sample).



WARNING: Do not touch the inside of the cell, or lean over it when inserting or removing a capsule. Handle cooled capsules with care. Gases produced during decomposition reactions at high temperatures may not condense when cooled; therefore, the capsule may remain under pressure at ambient temperature.



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

DSC Q Series: The high pressure capsule 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 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 with the high pressure capsules.



WARNING: 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 pressure capsules. There are no concerns regarding the use of the LNCA (Liquid Nitrogen Cooling Accessory) heat exchanger with the DSC high pressure capsules.



WARNING: The DSC high pressure capsules 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 Pressure Capsule Kit.



CAUTION: Properly sealed capsules can withstand internal pressures up to 10 MPa (1450 psi), with temperatures up to 300°C. Improperly sealed capsules can leak before reaching this pressure.



CAUTION: Heating rates should be 10°C/minute or less because of the high heat capacity of the capsule. Keep the sample mass as low as practical.

Cleaning the Capsules



CAUTION: Handle the capsule lid, capsule bottom, seal, sample, and assembled caps, covers, and sealing disks 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 pressure capsules are manufactured to high quality standards, including cleaning to remove contaminants that might be present from the manufacturing process. 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 capsules in a 250-mL Erlenmeyer flask that has been fitted with a glass stopper.
2. Add approximately 150 mL of reagent grade xylene (enough to cover the capsules).
3. Swirl the flask, containing the capsules and xylene, for 0.5 to 2.0 minutes.
4. Let the flask stand for at least 1 minute.
5. Decant the xylene out of the flask.
6. Repeat steps 1 through 5.
7. Add approximately 150 mL of reagent grade acetone after the second xylene wash.
8. Swirl the flask, containing the pans and acetone, for 0.5 to 2.0 minutes.
9. Let the flask stand for at least 1 minute.
10. Decant the acetone out of the flask.

11. Repeat steps 7 through 10.
12. Rotate the flask—so that no capsules adhere to the bottom or sides of the flask—as you flow nitrogen at 150 to 200 mL/min over the wet capsules to drive off the excess solvent. This should take approximately 5 to 6 minutes.
13. Return the cleaned capsules to their storage container, and record the date they were cleaned.

Preparing a Capsule



CAUTION: Handle the capsule lid, capsule bottom, seal, sample, and assembled caps, covers, and sealing disks with tweezers or other 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 capsules for the DSC high pressure capsule experiments.

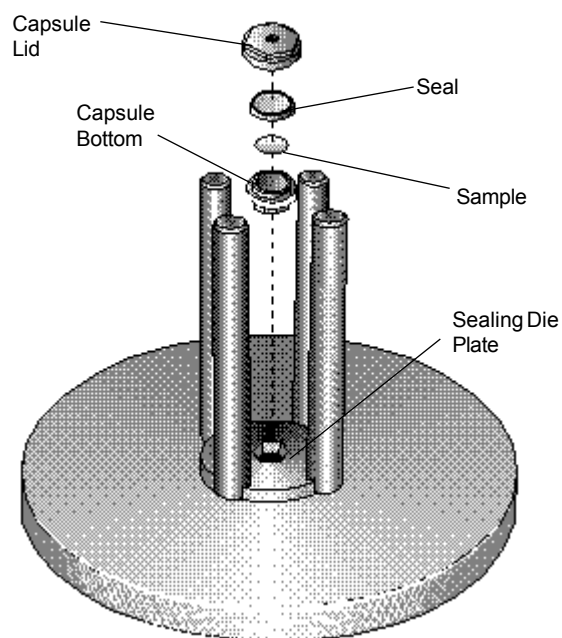


Figure 1
Parts of DSC High Pressure Capsule Sealing Assembly

Sealing and 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..

To weigh samples, follow these directions:

1. Obtain the total weight of the capsule bottom, capsule lid, and seal.
2. Using tweezers, place a capsule bottom in the hexagonal hole of the sealing plate (see Figure 1).
3. Place the sample carefully in the capsule bottom; then place the seal, cup downward, on the capsule (see Figure 1).
4. Using tweezers, place the capsule lid over the seal.
5. Align the sealing tool over the hexagonal lid as shown in Figure 2 here.
6. Turn the sealing tool clockwise, while applying light pressure, until the sealing tool slips—you will hear a click.

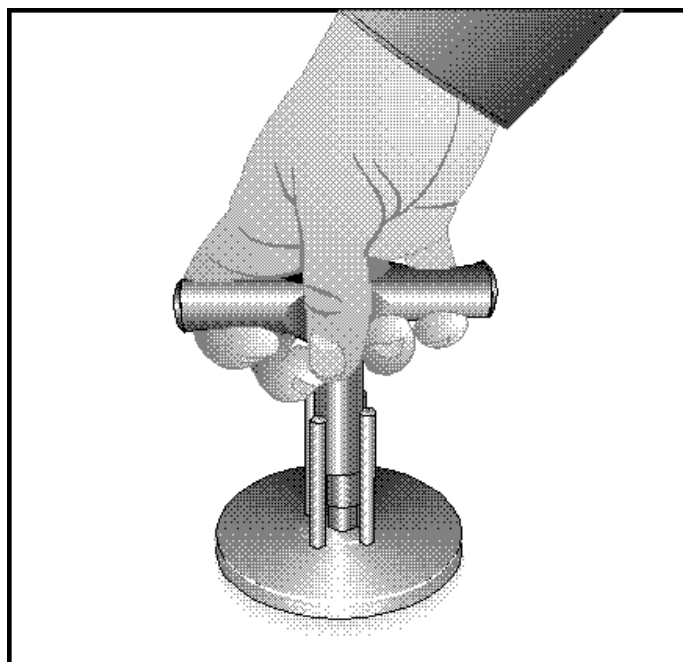


Figure 2
Using the Sealing Tool

7. Remove the sealing tool from the sealing die plate, and take out the sealed capsule.

NOTE: When handling the sealed capsule, 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.

8. 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 pressure capsule is now ready to run in your DSC cell.

Opening the Capsule

To open the capsule, reverse the procedures listed previously for sealing. This allows you to remove the sample and reuse the capsule. Seals must be discarded after use.



WARNING: High pressure capsules may retain pressure after material decomposition. Make sure the capsule is at room temperature before opening it. Open the capsule slowly and carefully to allow the seal to open and vent the pressure.

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.

DSC 2XXX Series Instruments

1. Use an empty sealed capsule as a reference. Place it in the cell.
2. Seal an 8-mg to 10-mg indium metal sample in a high pressure capsule and place it in the cell.
3. Install the metal bell jar for cell without a heat exchanger or the safety lid for cell using a heat exchanger.



WARNING: Properly sealed capsules may release pressure rapidly when internal pressure exceeds capsule-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 pressure capsules.

Make sure that you read the Warnings beginning on page 8 before proceeding.

4. Use standard calibration methods to calibrate the system. If additional temperature calibration points are required, make sure that you use the high pressure capsule when gathering data.
5. Return the instrument to standard mode before running your experiments.

DSC Q100 or Q1000 Instruments

1. Use an empty sealed capsule as a reference. Place it in the cell.
2. Weigh an 8-mg 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 capsule and place it inside the cell.
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.

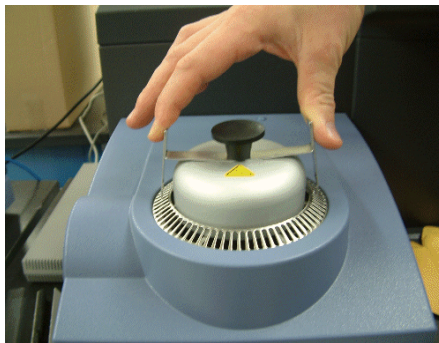
7. Return the instrument to standard mode before running your experiments.

DSC Q10 Instrument

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

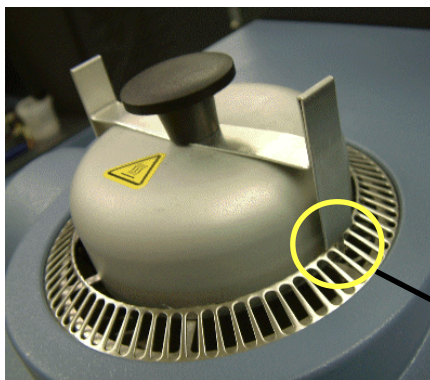
5. Squeeze the tabs on the safety clamp toward the center (as shown in the figure here) while you lower the manual lid onto the cell.

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 pressure capsules 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.



Position
Hooks
Securely

Running Experiments

After you calibrate the DSC using the high pressure capsule and return the instrument to standard mode, load the sample and reference capsules into the DSC cell.



WARNING: Properly sealed capsules may release pressure rapidly when internal pressure exceeds capsule-seal pressure capability.

DSC 2XXX Series Instruments: Be sure to use the supplied safety devices, as shown in Figures 3 and 4, when running any experiments or calibrating with the high pressure capsules.

DSC Q100 and Q1000 Instruments: Be sure to close the AutoLid before beginning the experiments using high pressure capsules.

DSC Q10 Instruments: Make sure the safety clamp is securely installed on the manual lid before beginning the experiments using high pressure capsules.



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



WARNING: Make sure that you read the Warnings beginning on page 8 before proceeding.

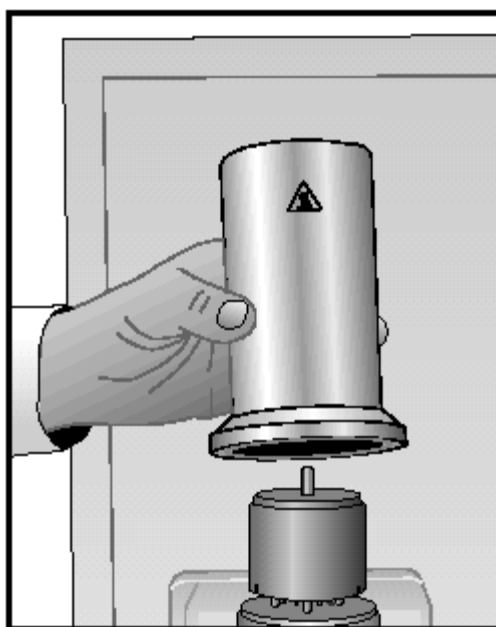


Figure 3
Using the Metal Bell Jar with the 2XXX DSC Cell

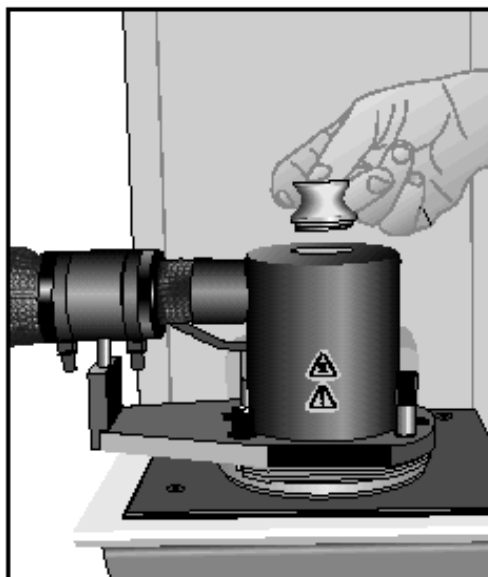


Figure 4
Using the Safety Lid with the 2XXX DSC Cell and the Heat Exchanger

Maintaining the Capsules and Sealing Tool

The capsule bottom and lid are normally reusable after being run on the instrument. Open the capsule as directed, remove the sample, and clean the capsule after each use. The seal must be replaced after each use of the capsule.

Stainless Steel Capsules

The stainless steel capsules can react differently from other types of capsules; they:

- May discolor and distort at high temperatures.
- May be cleaned with a solvent that does not affect the steel.

Sealing Tool Maintenance

Wipe the sealing tool and sealing die plate clean with a soft cloth that has been dampened with a dilute laboratory detergent solution.

The hexagonal hole in the sealing die plate should be periodically checked for debris from the capsules. Use a soft tissue or cloth to clean the sealing die plate as needed.

Specifications

Table 1 contains the technical specifications for High Pressure Capsules and seals.

Table 1
High Pressure Capsule Specifications

Pressure capability	10 MPa (1450 psi)
Temperature limit for aqueous solutions	300°C
Capsule Volume	35 μ L (max)
Material	440A SST
Seal material	Gold-plated copper

Replacement Parts

Table 2 contains the replacement parts for High Pressure Capsules and seals.

Table 2
List of DSC High Pressure Capsule Parts

Part Number	Description
900814.901	DSC High Pressure Capsule Seals (qty 20)
900815.901	DSC High Pressure Capsules (qty 5)

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