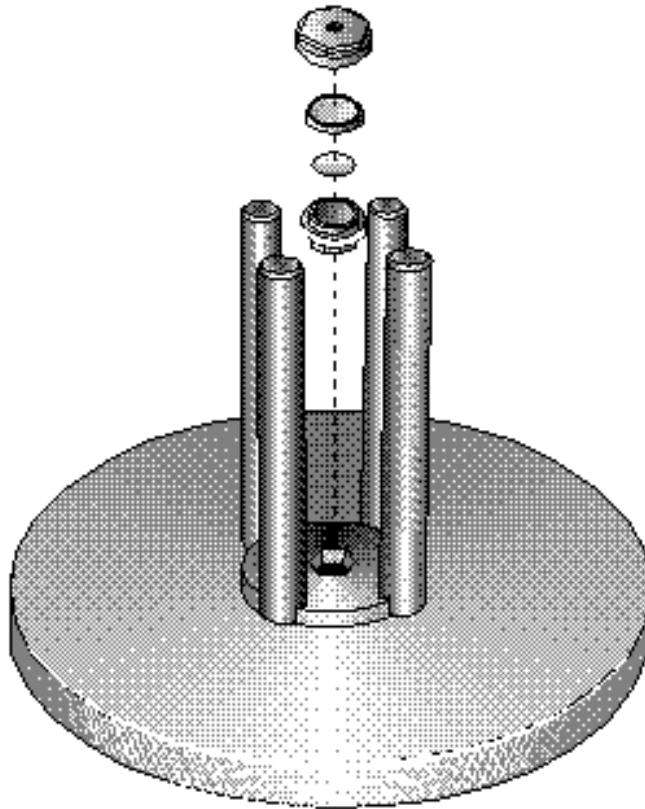


# Discovery and Q Series DSC

## High Pressure Capsule Kit



P/N 900808.901

## 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**

EN 61010-1:2010 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

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

## Safety

**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:** 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 capsules should not be used for analysis of thermally unstable or explosive materials.


**DSC Q Series or Discovery DSC™:** The high pressure capsule can be used with the Discovery DSC, Q2000/Q1000, or Q200/Q100/Q20A models configured with a Finned Air Cooling System (FACS), RCS, or LNCS. The AutoLid/Autosampler cell cover must be in position during experiments. For the Discovery DSC without Autosampler and the DSC Q20/Q10 instrument, the manual lid and safety clamp must be in position during experiments.

**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.

### 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 Pressure Capsule Kit* manual contains cautions and warnings that must be followed for your own safety.

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# Chapter 1:

## Cleaning, Preparing, and Opening the Capsule

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### *Cleaning the 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.

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 toluene (enough to cover the capsules).
- 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 toluene out of the flask.
- 6 Repeat steps 1 through 5.
- 7 Add approximately 150 mL of reagent grade acetone after the second toluene 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.

## Summary of High Pressure Capsule Safety Devices

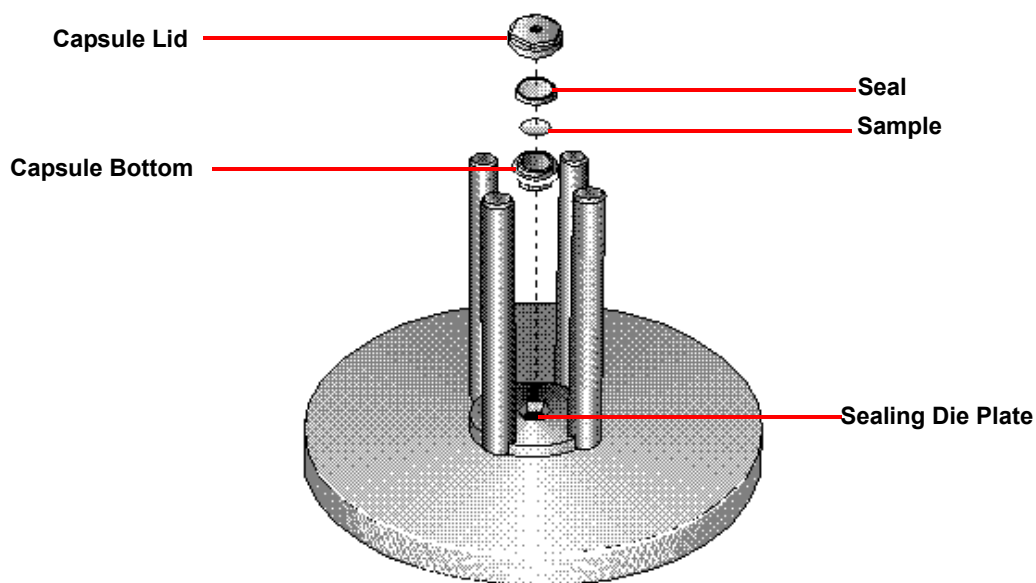
**Table 1: High Pressure Capsule Safety Devices**

Instrument	High Pressure Capsule Safety Device
Discovery DSC Q2000/Q1000 Q200/Q100/Q20A	AutoLid/Autosampler
Discovery DSC without Autosampler Q20/Q10	Manual Lid with Safety Clamp

### *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.



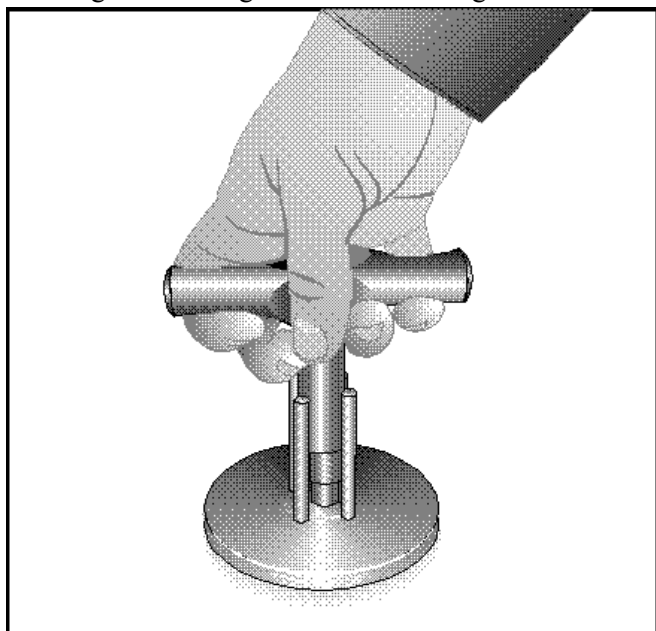
## Sealing and Weighing the Sample

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.



**Figure 2** Using the Sealing tool.

- 6 Turn the sealing tool clockwise, while applying light pressure, until the sealing tool slips—you will hear a click.
- 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.**

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

#### **Q20/Q10 Instruments**

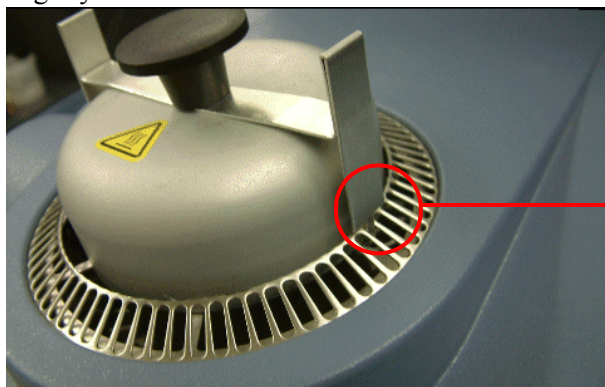
- 1 Use an empty sealed capsule as a reference. Place it in the cell.
- 2 Weigh an 8 to 10 mg sample of indium.

- 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 3** 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



**Position hooks securely**

**Figure 4** Position hooks securely.

**WARNING: Do not attempt to run the DSC Q20/ 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 Q20/Q10. Only T1 heat flow calibration is possible on the Q20/Q10.
- 7 Return the instrument to standard mode.

## 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. Refer to your instrument's online help for detailed information on running experiments.

## 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 2: Specifications for High Pressure Capsule and Seals**

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

## *Replacement Parts*

**Table 3: High Pressure Capsule Parts**

<b>Part Number</b>	<b>Description</b>
900808.901	DSC High Pressure Capsule Kit
900814.901	DSC High Pressure Capsule Seals (qty 20)
900815.901	DSC High Pressure Capsules (qty 5)