

# Q Series

## Pressure Cell Differential Scanning Calorimeter



## 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.

**UNE MISE EN GARDE** met l'accent sur une procédure susceptible d'endommager l'équipement ou de causer la perte des données si elle n'est pas correctement suivie.

|                                                                                   |                                                                                                                                                                                                                                                                                               |
|-----------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
|  | <p><b>A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.</b></p> <p><b>Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.</b></p> |
|-----------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|

## Regulatory Compliance

### For Canada

CAN/CSA-22.2 No. 1010.1-92 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

CAN/CSA-22.2 No. 1010.2.010-94 Particular requirements for laboratory equipment for the heating of materials + Amendments.

### For European Economic Area

(In accordance with Council Directive 73/23/EEC of 19 February 1973 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN61010-1: 1993 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN61010-2-010: 1994 Particular requirements for laboratory equipment for the heating of materials + Amendments.

### For United States

UL3101-1 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

IEC 1010-2-010: 1992 Particular requirements for laboratory equipment for the heating of materials + Amendments.

# **Electromagnetic Compatibility Standards**

## **For Australia and New Zealand**

AS/NZS 2064: 1997 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radio frequency equipment.

## **For Canada**

ICES-001 Issue 3 March 7, 1998 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

## **For the European Economic Area**

(In accordance with Council Directive 89/336/EEC of 3 May 1989 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1: 1997 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements + Amendments. Emissions: Meets Class A requirements (Table 3). Immunity: Meets performance criteria A for noncontinuous operation.




## **For the United States**

CFR Title 47 Telecommunication Chapter I Federal Communications Commission, Part 15 Radio frequency devices (FCC regulation pertaining to radio frequency emissions).

## Safety

### Instrument Symbols

The following label is displayed on the LNCS for your protection:

| Symbol                                                                              | Explanation                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
|-------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
|    | <p>This symbol appears on the right side of the cell stating that "improper use of oxygen or hydrogen can result in damage to the cell or harm to the user." See the WARNING on the next page for details.</p> <p>Ce symbole s'affiche à droite de la cellule et indique que « l'utilisation inappropriée de l'oxygène ou de l'hydrogène peut provoquer des dégâts à la cellule ou des dommages à l'utilisateur ». Voir l'AVERTISSEMENT à la page suivante pour plus de détails.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
|    | <p>This symbol on the rear access panel indicates that you must unplug the instrument before doing any maintenance or repair work; voltages exceeding 120/240 Vac are present in this system.</p> <p>If you are not trained in electrical procedures, do not remove the cabinet covers unless specifically instructed to do so in the manual. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.</p> <p>Ce symbole apposé à l'arrière du panneau d'accès indique vous devez débrancher l'instrument avant d'effectuer des travaux de maintenance ou de réparation ; présence de tensions excédant 120 VCA dans ce circuit.</p> <p>Présence de tensions élevées dans cet instrument/accessoire. Si vous n'êtes pas formé aux procédures électriques, ne déposez pas les couvercles de l'armoire sauf indications spécifiques contenues dans le manuel. La maintenance et la réparation des pièces internes doivent être effectuées uniquement par le personnel d'entretien qualifié de TA Instruments.</p> |
|  | <p>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.</p> <p>Ce symbole indique la présence possible d'une surface chaude. Prenez soin de ne pas toucher cette zone ou de laisser un matériau susceptible de fondre ou de brûler entrer en contact avec cette surface chaude.</p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |

Please heed the warning labels and take the necessary precautions when dealing with these areas. The *DSC Getting Started Guide* and this *Pressure Cell Getting Started Guide* contain cautions and warnings that must be followed for your own safety.

## *Important Safety Information*

Please read this before using oxygen in the Pressure Cell DSC



**WARNING:** If excessive amounts of hydrocarbons are present in the Pressure DSC (PDSC), energetic combustion could occur causing damage to the Pressure DSC cell and possible injury to the operator. To help prevent these problems, follow these guidelines:

(1) **Clean Supply Lines:** The oxygen supply lines, valves, gauges, and regulators must all be free of hydrocarbons and rated for oxygen service. Check with your supplier if you are uncertain whether a component is rated for oxygen service. If the inside of the tubing smells “oily” or has liquid or a black carbon residue in it, hydrocarbons may be present. Consult with your compressed gas suppliers for a cleaning procedure.

(2) **Cell Contamination:** Remove the pressure housing and visually inspect the Pressure DSC cell for oil or other organic contamination. The entire oxygen pressure system must be free of hydrocarbons. If there is a possibility of hydrocarbon contamination (spilled samples, oily residue, oily smell, carbon black, etc.) in your Pressure DSC cell, immediately discontinue use. Contact TA Instruments Service at (302) 427-4050 to schedule a safety inspection, or for additional information.

(3) **Check all Supply Tubing.** All tubing connecting your Pressure DSC cell to other devices (oxygen cylinder, gauges, valves, regulators, etc.) should be 0.125-in. O.D. All plumbing, valves, gauges, and regulators must be rated for high pressure service to 21 MPa gauge (3000 psig) and be free of hydrocarbons.



**AVERTISSEMENT:** S'il y a des quantités excessives d'hydrocarbures dans le DSC à pression (PDSC), il peut se produire une combustion énergétique susceptible d'endommager la cellule DSC à pression et de blesser l'opérateur. Pour éviter ces problèmes, respectez les directives ci-dessous:

(1) **nettoyez les conduites d'alimentation:** Les conduites d'alimentation en oxygène, les vannes, jauges et régulateurs doivent tous être débarrassés d'hydrocarbures et conçus pour la fourniture d'oxygène. Vérifiez auprès de votre fournisseur si vous n'êtes pas certain qu'un composant est approprié pour la fourniture de l'oxygène. Si l'intérieur de la tuyauterie a une odeur « d'huile » ou contient un liquide ou un résidu de carbone noir, il est possible qu'elle contienne des hydrocarbures. Consultez vos fournisseurs de gaz comprimé pour une procédure de nettoyage.

(2) **Contamination de la cellule:** Déposez le boîtier à pression et inspectez visuellement la cellule DSC à pression pour y rechercher de l'huile ou d'autres contaminants organiques. Tout le système à pression d'oxygène doit être débarrassé d'hydrocarbures. S'il existe une possibilité de contamination aux hydrocarbures (déversement d'échantillons, résidu huileux, odeur d'huile, carbone noir, etc.) dans votre cellule DSC à pression, arrêtez immédiatement de l'utiliser. Contactez le service TA Instruments au (302) 427-4050 pour programmer une inspection de sécurité ou pour obtenir d'autres renseignements supplémentaires.

(3) **Vérifiez toute la tuyauterie d'alimentation.** Toute la tuyauterie reliant votre cellule DSC pression à d'autres dispositifs (bouteille d'oxygène, jauges, vannes, régulateurs, etc.) doit mesurer 0,125 po. de diamètre extérieur. Toute la plomberie, les vannes, jauges et régulateurs doivent être conçus pour fournir une haute pression manométrique de 21MPa (3000 psig) et débarrassés d'hydrocarbures.

You should review the warnings on the previous page if you plan to use oxygen in the PDSC and any of the following conditions apply to you.

- New installation of a PDSC
- Modification of supply lines, valves or gauges
- Sample was spilled in the PDSC
- PDSC has an “oily” smell
- PDSC has not been used recently.

You may ensure safe operation of your Pressure DSC if you follow the important safety instructions and warnings as directed throughout this section and the entire manual.



**Please read this before using hydrogen in the Pressure DSC Cell:**

DO NOT use hydrogen or any other highly flammable gas with the Q Series™ **standard** DSC cells.



**WARNING:** Hydrogen gas should be used with extreme care. It is highly flammable when exposed to flame or oxidizing materials. [The Sax Safety Handbook, Dangerous Properties of Industrial Materials, indicates that the lower explosion limit (LEL) under ambient conditions for hydrogen is 4.1% in air. Care should be taken to keep the concentration below this value.] When using hydrogen in the Pressure DSC cell, the cell should be initially purged thoroughly with helium before introducing hydrogen.

**AVERTISSEMENT:** Le gaz d'hydrogène doit être utilisé avec une extrême prudence. Il est hautement inflammable lorsqu'il est exposé aux flammes ou aux matières comburantes. [Le manuel de sécurité Sax, Propriétés dangereuses des matières industrielles, indique que la limite inférieure d'explosion (LIE) de l'hydrogène dans les conditions ambiantes est de 4,1% dans l'air. Prenez soin de garder la concentration en dessous de cette valeur.] Lorsque vous utilisez de l'hydrogène dans la cellule DSC à pression, drainez d'abord la cellule à fond avec de l'hélium avant d'introduire l'hydrogène.

**CAUTION:** When hydrogen or helium is used as a pressurizing gas, the maximum temperature or maximum heating rate may be less than the specification.

**MISE EN GARDE:** Lorsque de l'hydrogène ou de l'hélium est utilisé comme gaz de pressurisation, la température maximale ou la vitesse de chauffage maximale peut être inférieure à la spécification.



**WARNING:** At the end of the experiment, the cell should be vented into an exhaust hood and repurged with helium prior to opening the pressure container.

**AVERTISSEMENT:** À la fin de l'expérience, aérez la cellule dans une bache d'échappement et drainez-la encore à l'hélium avant d'ouvrir le récipient sous pression.



**WARNING:** Check all supply tubing. All tubing connecting your Pressure DSC cell to other devices (such as hydrogen cylinders, gauges, regulators, etc.) should be 0.125 in. O.D. All plumbing, valves, gauges, and regulators must be rated for high pressure service to 21 MPa gauge (3000 psig) and all connections between items should be tight and leak-free.

**AVERTISSEMENT:** Vérifiez toute la tuyauterie d'alimentation. Toute la tuyauterie reliant votre cellule DSC à pression à d'autres dispositifs (tels que les bouteilles d'oxygène, jauges, régulateurs, etc.) doit mesurer 0,125 po. de diamètre extérieur. Toute la plomberie, les vannes, jauges et régulateurs doivent être conçus pour fournir une haute pression manométrique de 21 MPa (3000 psig) et tous les raccords entre les éléments doivent être hermétiques et étanches.

If you have any questions about hydrogen use, contact the U.S. TA Instruments Applications Hotline at (302) 427-4070.

Contact our U.S. Applications Hotline at (302) 427-4070 or your local TA Instruments Representative if you have any questions regarding the safe usage of the TA Instruments DSC Pressure Cells.

## ***Chemical Safety***

Use only the purge gases listed in Chapter 1. Use of other gases could cause damage to the instrument or injury to the operator.

**CAUTION: Do not remove the white, fibrous insulation from inside the cell cover.**

**MISE EN GARDE: Ne retirez pas l'isolant en fibre blanche à l'intérieur du couvercle de la cellule.**

## ***Electrical Safety***

**CAUTION: The hold-down thumbscrews that hold the DSC Pressure cell in place ensures proper mating with the electrical connections. You cannot run experiments without them. If they are not fully in place, the instrument may not be able to apply power to the cell and will not operate.**

**MISE EN GARDE: Les vis de serre-flan qui fixent la cellule DSC sous pression en place assure la jonction appropriée avec les connexions électriques . Vous ne pouvez pas faire des expériences sans eux. Si ils ne sont pas entièrement en place, l'instrument peut ne pas être en mesure d'appliquer la tension électrique à la cellule et ne fonctionnera pas.**

You must unplug the instrument before doing any maintenance or repair work; voltages as high as 120/240 Vac are present in this system.



**WARNING: High voltages are present in this instrument. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.**

**AVERTISSEMENT: Présence de tensions élevées dans cet instrument. La maintenance et la réparation des pièces internes doivent être effectuées uniquement par TA Instruments ou tout autre personnel d'entretien qualifié.**



**WARNING:** Drying out the Pressure DSC cell may be needed, if it has been exposed to humid conditions. It is important to be certain that the instrument ground is adequately connected to the facilities ground for safe operation.

Run the "Cell/Cooler Conditioning" test template to dry out the cell:

- 1 Ramp at 10 °C/min to 400 °C
- 2 Isothermal for 120 min.

**AVERTISSEMENT:** Le séchage de la cellule DSC à pression peut s'avérer nécessaire, si elle a été exposée à l'humidité. Il est important de s'assurer que la mise à la terre de l'instrument est correctement connectée à la mise à la terre des installations pour une utilisation sécurisée.

Effectuez le modèle d'essai « Conditionnement de la cellule/du refroidisseur » pour sécher la cellule.

- 1 Augmentation de 10 °C/min à 400 °C
- 2 Isotherme pendant 120 min.

### *Thermal Safety*

The cell surfaces can be hot enough to burn the skin during a sample run. If you are conducting a subambient test using the Pressure DSC cell, cold could also cause injury. After running any type of experiment, you must allow the cell to return to room temperature before you touch the inner cell surfaces.

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# Chapter 1:

## Introducing the DSC Pressure Cell

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### Overview

The Pressure DSC (PDSC) cell (shown to the right) is a DSC cell enclosed in a steel cylinder that can be pressurized to 7 MPa gauge (1000 psig). In addition to performing most of the same basic measurements as a standard DSC cell, the PDSC cell can operate at elevated pressure or under vacuum. This ability to vary pressure as well as temperature provides the following:

- Resolution of overlapping peaks
- Determination of heats of vaporization and vapor pressure
- Reaction rates in controlled atmospheres
- Studies of pressure-sensitive reactions

The Pressure DSC cell has two gas flow control valves, a three-way valve, a pressure gauge, a pressure release valve, and gas pressure fittings on the side. An 8.3 MPa gauge (1200 psig) pressure relief valve and a pressure transducer are contained in the base of the cell.

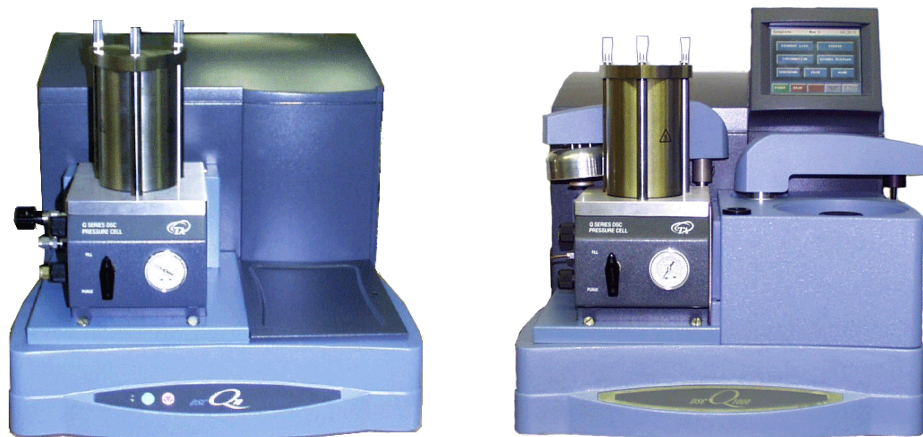
The Pressure DSC cell uses a constantan (thermoelectric) disc as a primary heat transfer element producing Heat Flow T1. A silver heating block, capped with a vented silver lid, encloses the constantan disc. The selected sample and an inert reference are placed in pans that sit on raised portions of the disc. Heat is transferred through the constantan disc to both the sample and the reference pans. Differential heat flow to the sample and reference are monitored by the CHROMEL®-constantan area thermocouples. The thermocouples are formed at the junctions of the constantan disc and the CHROMEL® wafers welded to the underside of the two raised portions of the disc. CHROMEL® and ALUMEL® wires are connected to the sample CHROMEL® wafer at the thermocouple junction to measure sample temperature. An ALUMEL® wire is welded to the reference wafer for thermal balance. The furnace control thermocouple is CHROMEL®-constantan.



Figure 1

Purge gas, entering the heating block through an inlet in the Pressure DSC cell's base, is preheated to block temperature by circulation before entering the sample chamber through the purge gas inlet. Gas exits through the vent hole in the silver lid.

The Pressure DSC cell is available as an optional operator-replaceable cell for the DSC Q2000/ Q1000 (shown in Figure 2, right). It is also available as part of an integrated DSC Q20P/Q10P system (shown in Figure 2, left).



**Figure 2** Left: DSC Q20P/Q10P; Right: Q2000/Q1000 with Pressure Cell

## *Pressure Cell Specifications*

The table found below contains the technical specifications for the PDSC

**Table 1: PDSC Technical Specifications**

| Item/Area               | Specification                                                                                                    |
|-------------------------|------------------------------------------------------------------------------------------------------------------|
| Dimensions of Cell Only | Depth: 23.6 cm (9.3 in)<br>Width: 22.4 cm (8.8 in)<br>Height: 33.3 cm (13.1 in)                                  |
| Weight of Cell Only     | 10.5 kg (23 lbs)                                                                                                 |
| Temperature Range       | -130 to 725°C                                                                                                    |
| Atmosphere              | –100 kPa gauge to 7 MPa gauge (1 Pa to 7 MPa absolute), constant pressure or constant volume                     |
| Dynamic gas purge       | To 200 mL/min (cell flow rate)                                                                                   |
| Purge gases             | Nitrogen, air, oxygen, carbon monoxide, carbon dioxide, hydrogen, helium, argon                                  |
| Heat flow               | T1 Heat Flow only                                                                                                |
| Compatibility           | Available as an interchangeable cell for DSC Q2000/Q1000. Available also as part of the dedicated DSC Q20P/Q10P. |

Performance depends on the pressure and atmosphere selected.



# Chapter 2:

## Installing the Pressure Cell

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### *Unpacking/Repacking the DSC Pressure Cell*

The instructions needed to unpack and repack the instrument are found as separate unpacking instructions in the shipping box and in the online documentation associated with the instrument control software. You may wish to retain all of the shipping hardware and boxes in the event you wish to repack and ship your cell.

### *Installing the DSC Pressure Cell*

To install the Pressure DSC (PDSC) Cell, follow these instructions and refer to the accompanying figures.

Because the PDSC is available as an interchangeable cell for DSC Q2000/Q1000 or as a dedicated cell installed on the DSC Q20P/Q10P, this section covers both configurations.

**CAUTION:** Do not remove the white, fibrous insulation from inside the cell cover.

**MISE EN GARDE:** Ne retirez pas l'isolant en fibre blanche à l'intérieur du couvercle de la cellule.

### **Preparing for Cell Installation**

Before the Pressure DSC cell can be installed the cell base must be fully exposed. Follow the instructions that apply to your specific DSC instrument to remove the covers.

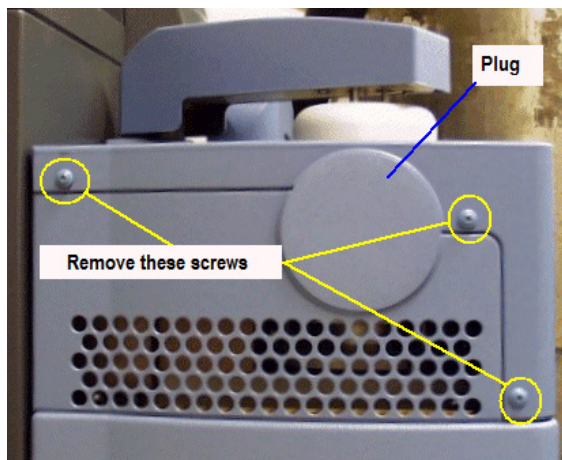
### *Removing the DSC Q2000/Q1000 Standard Cell & Covers*

To remove the DSC Q2000/Q1000 standard cell from the instrument before installing the Pressure DSC cell, follow these steps:

- 1 Select the Control/Lid/Open function to raise the AutoLid from the cell and cause it to move out of the way to its home position. If the DSC is equipped with an Autosampler, press the HOME key on the Autosampler touch screen to move it to the "home" position.

**NOTE:** The Pressure DSC cell is not compatible with the DSC Q2000/Q1000 AutoLid or Autosampler. These items must be disabled so that they remain out of the way (parked) during pressure cell experiments. The software automatically recognizes when a Pressure DSC cell is mounted and disables these two items.

- 2 Pull the plug on the side of the unit cover out to remove it. Then remove the Phillips head screws attaching the cell cover to the unit cover. Three screws are located on the side and one is located on the top. Retain the screws.

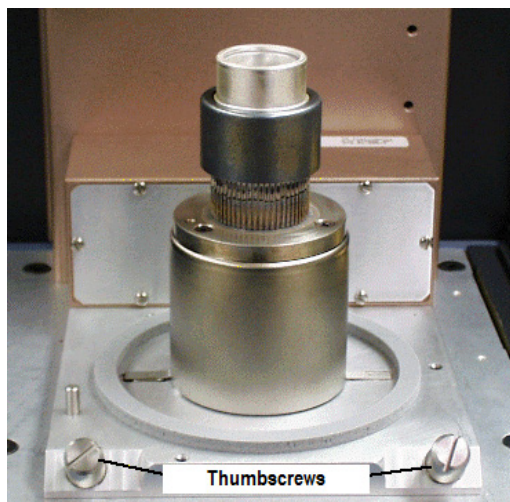


**Figure 3** Remove screws.

- 3 If your instrument has an Autosampler installed, lift up the cover to release the tabs and pull the cover towards you to remove it fully.

If you do not have an Autosampler, you will have to remove additional screws to release the cover. Then pull the cover towards you to remove it fully.

- 4 If there is a cooling accessory installed, make sure that it has reached room temperature, then remove it. For instructions on removing a cooling accessory, reverse the instructions for installing the cooling accessory, which can be found in the Getting Started Guide for that accessory.
- 5 Remove the two thumbscrews at the front of the cell base as shown in the figure below.



**Figure 4** Remove thumbscrews.

- 6 Make sure the cell has reached room temperature, then slide out the cell.
- 7 Install the PDSC cell on the DSC as directed on page 20, then install the dress cover as directed on page 19.

## ***Removing the DSC Q20P/Q10P Cell Dress Covers***

The TA Instruments Q20P/Q10P is a dedicated pressure DSC system. However, it has been designed so that the operator can readily replace an old (damaged, nonfunctional) Pressure DSC cell with a new one, should the need arise. This replacement is accomplished by removing the cell dress covers and old cell as described in this section and then installing a new cell as described in the next section.

Follow these directions:

- 1 Use a Phillips head screwdriver to remove the single screw holding the top piece of the cell dress cover in place behind the cell



**Figure 5** Screw on top piece of PDSC cell cover.

- 2 Slide the top piece up and remove it.
- 3 Slide the bottom piece of the cell dress cover out and remove it as shown in the figure below.

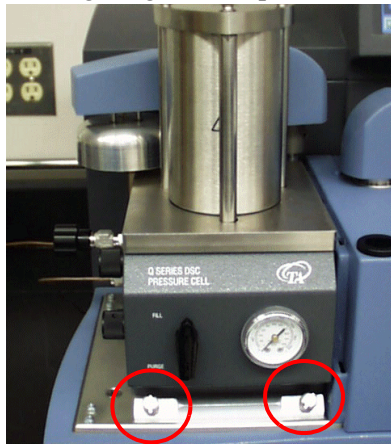


**Figure 6** Thumbscrews.

- 4 Make sure that the cell is at ambient pressure (i.e., is not pressurized). Disconnect all tubing from the IN and OUT ports.
- 5 Remove the two thumbscrews at the front of the cell base (shown in [Figure 6](#)).
- 6 Make sure the cell is at room temperature. Then slide out the cell.
- 7 Install the new PDSC cell as directed in the next section.

## *Installing the PDSC Cell (Q2000/Q1000 or Q20/Q10P)*

- 1 Hold the PDSC and slide it onto the baseplate. The back of the cell should touch the connector housing on the DSC base.
- 2 Install the two hold-down thumbscrews, then turn them clockwise. The hold-down screws need only be finger tight to keep the PDSC stable.



**Figure 7** Hold down thumbscrews.

- 3 Connect a sufficient length of 0.125-inch tubing from a pressure regulator on your pressurized gas source to the IN port on the side of the PDSC Cell as shown below. The gas (nitrogen, air, oxygen, argon, etc.) should be pressure-regulated up to 7 MPa gauge (1000 psig).



**Figure 8** IN port.



**WARNING:** If hydrogen is used, the Pressure DSC cell must be filled in a way that ensures no oxygen is present at elevated temperatures.

**AVERTISSEMENT:** Si vous utilisez de l'hydrogène, remplissez la cellule DSC à pression de façon à vous assurer qu'il n'y a pas d'oxygène à des températures élevées.

**CAUTION:** If oxygen is used, be certain to use fittings, gauges, and tubing that are oxygen-rated.

**MISE EN GARDE:** Si l'oxygène est utilisé, être certain d'utiliser des raccords, des jauges, et des tubes appropriés à l'utilisation de l'oxygène.



The regulator you choose should have two gauges: one to monitor source pressure and one to monitor the regulator output pressure. The regulator should be rated to withstand the source pressure; its output should cover the experimental range up to 7 MPa gauge (1000 psig).



**WARNING: DO NOT connect the PDSC directly to a pressurized gas source without using an appropriate regulator.**

**AVERTISSEMENT: NE raccordez PAS la cellule PDSC directement à la source de gaz sous pression sans utiliser un régulateur approprié.**



**WARNING: The tubing must be of sufficient strength to withstand the pressure to be used in your experiments.**

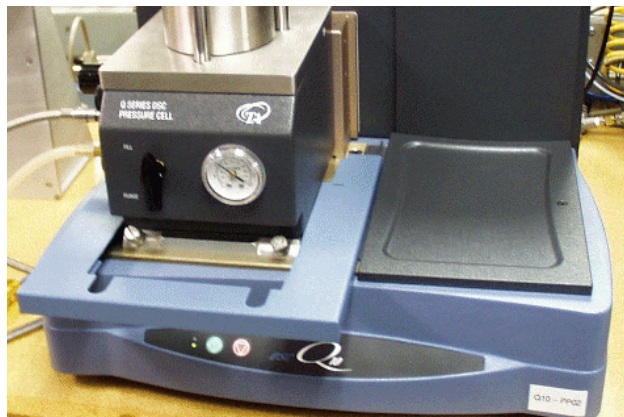
**AVERTISSEMENT: La tuyauterie doit être suffisamment solide pour résister à la pression qui sera utilisée pour vos expériences.**

- 4 Install the cell cover as directed below, then calibrate the Pressure DSC cell after installation. See [Chapter 3](#) for calibration information.

## *Installing the Dress Cover on the Pressure Cell (Q2000/Q1000 or Q20P/Q10P)*

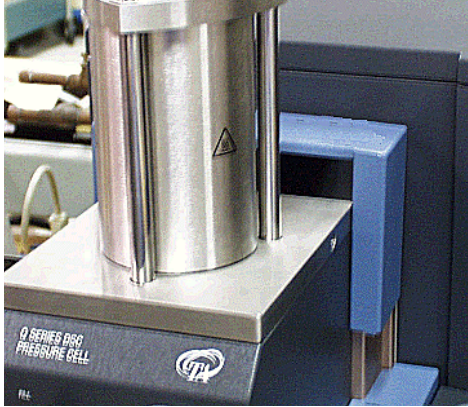
A special dress cover for the pressure cell can be placed on the cell after the PDSC cell is installed as directed earlier in this chapter. The dress covers used for the Q20P/Q10P are slightly different from that used for the Q2000/Q1000. The Q2000/Q1000 dress cover is modified to accommodate the presence of the AutoLid and Autosampler on the Q2000/Q1000. However both dress cover types can be installed following the same procedures below. (The Q20P/Q10P is shown in the figures.)

- 1 Slide the bottom piece of the cell dress cover onto the cell base as shown in the figure below. Push it back until it touches the back of the unit.



**Figure 9** Installing the dress cover bottom piece.

- 2 Slide the top piece down over the cell connector as seen in the figure below and match up the tabs in the top piece with the slots in bottom piece.



**Figure 10** Installing the dress cover top piece.

- 3 Use a Phillips head screwdriver to install the single screw holding the top piece of the cell dress cover in place.

# Chapter 3:

## Using and Maintaining the Pressure Cell

---

### *Before You Begin*

Please read this before using oxygen in the Pressure Cell DSC



**WARNING:** If excessive amounts of hydrocarbons are present in the Pressure DSC (PDSC), energetic combustion could occur causing damage to the Pressure DSC cell and possible injury to the operator. To help prevent these problems, follow these guidelines:

(1) **Clean Supply Lines:** The oxygen supply lines, valves, gauges, and regulators must all be free of hydrocarbons and rated for oxygen service. Check with your supplier if you are uncertain whether a component is rated for oxygen service. If the inside of the tubing smells “oily” or has liquid or a black carbon residue in it, hydrocarbons may be present. Consult with your compressed gas suppliers for a cleaning procedure.

(2) **Cell Contamination:** Remove the pressure housing and visually inspect the Pressure DSC cell for oil or other organic contamination. The entire oxygen pressure system must be free of hydrocarbons. If there is a possibility of hydrocarbon contamination (spilled samples, oily residue, oily smell, carbon black, etc.) in your Pressure DSC cell, immediately discontinue use. Contact TA Instruments Service at (302) 427-4050 to schedule a safety inspection, or for additional information.

(3) **Check all Supply Tubing.** All tubing connecting your Pressure DSC cell to other devices (oxygen cylinder, gauges, valves, regulators, etc.) should be 0.125-in. O.D. All plumbing, valves, gauges, and regulators must be rated for high pressure service to 21 MPa gauge (3000 psig) and be free of hydrocarbons.



**AVERTISSEMENT:** S'il y a des quantités excessives d'hydrocarbures dans le DSC à pression (PDSC), il peut se produire une combustion énergétique susceptible d'endommager la cellule DSC à pression et de blesser l'opérateur. Pour éviter ces problèmes, respectez les directives ci-dessous:

(1) **nettoyez les conduites d'alimentation:** Les conduites d'alimentation en oxygène, les vannes, jauges et régulateurs doivent tous être débarrassés d'hydrocarbures et conçus pour la fourniture d'oxygène. Vérifiez auprès de votre fournisseur si vous n'êtes pas certain qu'un composant est approprié pour la fourniture de l'oxygène. Si l'intérieur de la tuyauterie a une odeur « d'huile » ou contient un liquide ou un résidu de carbone noir, il est possible qu'elle contienne des hydrocarbures. Consultez vos fournisseurs de gaz comprimé pour une procédure de nettoyage.

(2) **Contamination de la cellule:** Déposez le boîtier à pression et inspectez visuellement la cellule DSC à pression pour y rechercher de l'huile ou d'autres contaminants organiques. Tout le système à pression d'oxygène doit être débarrassé d'hydrocarbures. S'il existe une possibilité de contamination aux hydrocarbures (déversement d'échantillons, résidu huileux, odeur d'huile, carbone noir, etc.) dans votre cellule DSC à pression, arrêtez immédiatement de l'utiliser. Contactez le service TA Instruments au (302) 427-4050 pour programmer une inspection de sécurité ou pour obtenir d'autres renseignements supplémentaires.

(3) **Vérifiez toute la tuyauterie d'alimentation.** Toute la tuyauterie reliant votre cellule DSC pression à d'autres dispositifs (bouteille d'oxygène, jauges, vannes, régulateurs, etc.) doit mesurer 0,125 po. de diamètre extérieur. Toute la plomberie, les vannes, jauges et régulateurs doivent être conçus pour fournir une haute pression manométrique de 21MPa (3000 psig) et débarrassés d'hydrocarbures.

You should review the warnings above if you plan to use oxygen in the PDSC and any of the following conditions apply to you.

- New installation of a PDSC
- Modification of supply lines, valves or gauges
- Sample was spilled in the PDSC
- PDSC has an “oily” smell
- PDSC has not been used recently.

You may ensure safe operation of your Pressure DSC if you follow the important safety instructions and warnings as directed throughout this section and the entire manual.



**Please read this before using hydrogen in the Pressure DSC Cell:**

DO NOT use hydrogen or any other highly flammable gas with the Q Series™ **standard** DSC cells.



**WARNING:** Hydrogen gas should be used with extreme care. It is highly flammable when exposed to flame or oxidizing materials. [The Sax Safety Handbook, Dangerous Properties of Industrial Materials, indicates that the lower explosion limit (LEL) under ambient conditions for hydrogen is 4.1% in air. Care should be taken to keep the concentration below this value.] When using hydrogen in the Pressure DSC cell, the cell should be initially purged thoroughly with helium before introducing hydrogen.

**AVERTISSEMENT:** Le gaz d'hydrogène doit être utilisé avec une extrême prudence. Il est hautement inflammable lorsqu'il est exposé aux flammes ou aux matières comburantes. [Le manuel de sécurité Sax, Propriétés dangereuses des matières industrielles, indique que la limite inférieure d'explosion (LIE) de l'hydrogène dans les conditions ambiantes est de 4,1% dans l'air. Prenez soin de garder la concentration en dessous de cette valeur.] Lorsque vous utilisez de l'hydrogène dans la cellule DSC à pression, drainez d'abord la cellule à fond avec de l'hélium avant d'introduire l'hydrogène.



**WARNING:** At the end of the experiment, the cell should be vented into an exhaust hood and repurged with helium prior to opening the pressure container.

**AVERTISSEMENT:** À la fin de l'expérience, aérez la cellule dans une bache d'échappement et drainez-la encore à l'hélium avant d'ouvrir le récipient sous pression.



**WARNING:** Check all supply tubing. All tubing connecting your Pressure DSC cell to other devices (such as hydrogen cylinders, gauges, regulators, etc.) should be 0.125 in. O.D. All plumbing, valves, gauges, and regulators must be rated for high pressure service to 21 MPa gauge (3000 psig) and all connections between items should be tight and leak-free.

**AVERTISSEMENT:** Vérifiez toute la tuyauterie d'alimentation. Toute la tuyauterie reliant votre cellule DSC à pression à d'autres dispositifs (tels que les bouteilles d'oxygène, jauges, régulateurs, etc.) doit mesurer 0,125 po. de diamètre extérieur. Toute la plomberie, les vannes, jauges et régulateurs doivent être conçus pour fournir une haute pression manométrique de 21 MPa (3000 psig) et tous les raccords entre les éléments doivent être hermétiques et étanches.

If you have any questions about hydrogen use, contact the U.S. TA Instruments Applications Hotline at (302) 427-4070.

Contact our U.S. Applications Hotline at (302) 427-4070 or your local TA Instruments Representative if you have any questions regarding the safe usage of the TA Instruments DSC Pressure Cells.

# *Calibrating the Pressure Cell*

To obtain the best experimental results, you should calibrate the Pressure DSC cell when you first install it and periodically thereafter, if the purge gas, heating rate, and/or pressure is changed. These calibration experiments are performed and analyzed using the instrument control software. For details on how to perform each calibration, refer to the online help documentation accessed through the Help menu or by clicking the Help button in the instrument control software.

## **Baseline Slope and Offset Calibration**

The heat flow measured using the PDSC is T1 heat flow. A baseline slope and offset calibration is performed, which involves heating an empty cell through the entire temperature range expected in subsequent experiments. This calibration is used to calculate the slope and offset values needed to flatten the baseline and zero the heat flow signal.

## **Enthalpy (Cell) Constant Calibration**

This calibration is based on a run in which a standard metal (e.g., indium) is heated through its melting transition. The calculated heat of fusion is compared to the theoretical value. The cell constant is the ratio between these two values.

The onset slope, or thermal resistance, is a measure of the suppression of temperature rise that occurs in a melting sample in relation to the thermocouple. Theoretically, a standard sample should melt at a constant heating rate. As it melts and draws more heat, a temperature difference develops between the sample and the sample thermocouple. The thermal resistance between these two points is calculated as the onset slope of the heat flow versus temperature curve on the front of the melting peak. The onset value is used for kinetic and purity calculations to correct for this thermal resistance.

## **Temperature Calibration**

Temperature calibration is based on a run in which a temperature standard (e.g., indium) is heated through its melting transition. The recorded melting point of this standard is compared to the known melting point, and the difference is calculated for temperature calibration. The same file used for the cell constant calibration can be used for this calibration.

In addition, you can use up to four other standards to calibrate temperature. If you use one pair of known and observed points, the entire curve is essentially offset, or shifted, to the actual melting point. If you use multiple standards, the temperature is corrected by a cubic spline fit. The multiple-point temperature calibration is more accurate than the one-point calibration.

## **Pressure Calibration**

Pressure calibration is an optional calibration procedure for the Pressure DSC cells. It is based on comparing the pressure reading at two points, typically 1 atmosphere and another pressure selected by you, to the pressure reading on an external pressure gauge. See the online help with your instrument control software for information.

## Running a Pressure DSC Experiment



**WARNING:** Any time you open the OUT or Pressure Release valve during operation, you may be applying full pressure to the external lines or components (e.g., flowmeter), which may not be able to withstand full pressure. If you have a vacuum connected to the cell, the pressure would be reversed back into the cell, which may not be able to withstand an abrupt change in pressure. This could seriously damage the cell. Keep the Pressure DSC Cell away from flammable materials.

**AVERTISSEMENT:** Chaque fois que vous ouvrez la vanne de décompression ou OUT pendant le fonctionnement, vous risquez d'appliquer la pleine pression aux lignes ou accessoires externes (par exemple, un débitmètre), qui peuvent ne pas être en mesure de résister à la pression complète. Si vous avez une pompe à vide reliée à la cellule, la pression sera imposée dans la cellule, qui peut ne pas être en mesure de résister à un changement brusque de pression. Cela pourrait sérieusement endommager la cellule. Maintenir la pression DSC cellule à l'écart des matériaux inflammables.

### Experimental Procedure

The Pressure DSC can be used for heating ramp and isothermal experiments. Pressure experiments may be conducted at constant volume, constant pressure, dynamic pressure (fixed purge rate) or under vacuum. (See information later in this chapter.) The following sections describe those experimental conditions that are unique for the Pressure DSC cell.

- 1 Select the purge gas (see [“Selecting the Purge Gas”](#)).
- 2 Attach and set up external accessories as required (e.g., flowmeter).
- 3 Select and prepare a sample. This involves preparing a sample of the appropriate size and weight, selecting the pan type and material, and encapsulating the sample in the pan. For details refer to the online documentation.
- 4 Open the Pressure DSC cell and load the sample pan (and a similarly prepared empty reference pan) into the cell.
- 5 Close the cell and pressurize as directed. See [“Pressurizing the Cell” on page 30](#).
- 6 Enter experiment and procedure information through the TA controller, this includes both sample and instrument information. Follow the instructions for operating the Pressure DSC cell found in this chapter.
- 7 Start the experiment. When the experiment is completed, release the pressure before opening the cell (see page 35).

### Selecting the Purge Gas

The Pressure DSC cell is designed for use of purge gases such as nitrogen, air, oxygen, hydrogen, argon, helium, dry carbon dioxide, carbon monoxide, as well as other gases compatible with the materials of construction (stainless steel, copper, constantan, silver, CHROMEL®, ALUMEL®). The gas of choice will depend on the specific application. However, there are several other considerations which could also affect selection of a specific purge gas. These considerations are:

- **Safety:** Reactive gases such as oxygen, hydrogen, and carbon monoxide can present an explosion or health hazard, if handled improperly. See additional warnings located throughout this manual for more information.
- **Thermal Conductivity:** The high thermal conductivity of gases like hydrogen and helium may limit the experimental temperature conditions that can be achieved. The table below provides a rough indication of those limitations.

**Table 2: Gas/Temperature Pressure Limits**

| <b>Gas</b>                                        | <b>Pressure MPa gauge (psig)</b> |                |                  |                  |                  |                   |
|---------------------------------------------------|----------------------------------|----------------|------------------|------------------|------------------|-------------------|
|                                                   | <b>°C/min</b>                    | <b>Ambient</b> | <b>1.4 (200)</b> | <b>2.8 (400)</b> | <b>5.6 (800)</b> | <b>7.0 (1000)</b> |
| Nitrogen                                          | 100                              | 385°C          | —                | —                | —                | 275°C             |
|                                                   | 50                               | 720°C          | 600°C            | —                | —                | —                 |
|                                                   | 20                               | 720°C          | 720°C            | 720°C            | 600°C            | —                 |
|                                                   | 10                               | 720°C          | 720°C            | 720°C            | —                | 655°C             |
| Helium                                            | 100                              | 255°C          | —                | —                | —                | —                 |
|                                                   | 50                               | 465°C          | 450°C            | —                | —                | 360°C             |
|                                                   | 20                               | —              | 570°C            | 535°C            | —                | 455°C             |
|                                                   | 10                               | —              | 605°C            | —                | 510°C            | 490°C             |
| Hydrogen                                          | 100                              | —              | 215°C            | 195°C            | 160°C            | 155°C             |
|                                                   | 50                               | —              | 370°C            | 325°C            | 290°C            | 265°C             |
|                                                   | 20                               | —              | 480°C            | 435°C            | 340°C            | 300°C             |
|                                                   | 10                               | —              | 540°C            | 495°C            | 425°C            | 300°C             |
| NOTE: Dashes (—) represent values not determined. |                                  |                |                  |                  |                  |                   |

## Loading a Pressure DSC Sample

Once you have prepared the sample pan and entered all necessary pre-experiment data, you are ready to load the sample pan into the PDSC cell. The PDSC cell should already be installed on the DSC before you load the sample (see [Chapter 2](#) for installation instructions).

- 1 Close the IN control valve to shut off the gas supply to the cell.

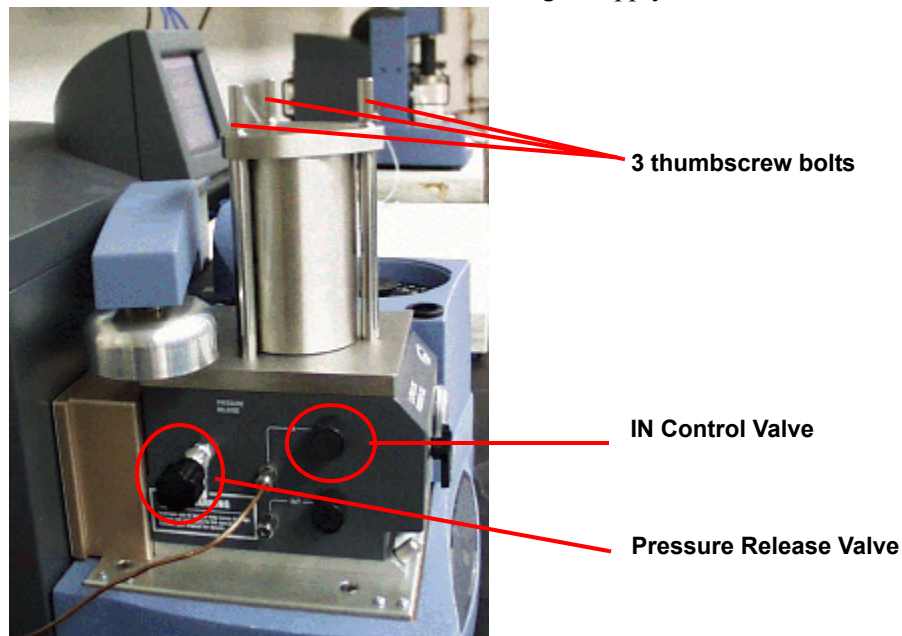


Figure 11

- 2 Slowly open the Pressure Release valve and leave it open to ensure that the cell is at ambient pressure.
- 3 Unscrew the three thumbscrew bolts (see [Figure 11](#)) from the top plate. Do not use tools to open or close the cell.

**CAUTION:** If you have difficulty unscrewing the thumbscrew bolts (excessive bolt friction), it is likely that the cell is still under some pressure or the cell temperature is too high. (This is a safety feature.) Recheck the valve positions as described in steps 1 and 2 or allow the cell to cool to less than 50 °C (greater than 50 °C is a burn hazard).

**MISE EN GARDE:** Si vous avez des difficultés pour dévisser manuellement les boulons de vis (friction trop forte), il est probable que la cellule est toujours sous une certaine pression ou la température de la cellule est trop élevée (protection de sécurité). Vérifier la position de la vanne comme décrit dans les étapes 1 et 2 ou permettre à la cellule de refroidir à moins de 50 °C (plus de 50 °C représente un risque de brûlure).

- 4 Remove the top plate, cell cover, and silver lid.

**CAUTION:** If the cell has just been used, these components could be very hot. As a safe operating practice, use leather gloves when handling the top plate, and use tweezers whenever handling the metal cell cover or silver lid.

**MISE EN GARDE: Si la cellule vient d'être utilisée , ces composants pourraient être très chaud. Pour une meilleure sécurité, utiliser des gants de cuir lors de la manipulation de la plaque supérieure, et utiliser des pinces de manutention pour le couvercle de la cellule en métal ou le couvercle d'argent.**

- 5 Load the sample and reference pans with the sample in the front and the reference in the back. Most pressure experiments are run with open sample pans to ensure optimum sample-purge interactions. Other pan configurations are used for specific determinations (e.g., for vapor pressure measurements using hermetic pans with a pinhole). See online help for details.
- 6 Replace the silver lid, cell cover, and top plate. Push the top plate down as far as it will go, taking care not to damage the O-ring or jar the cell, which could cause the pans to move off the dimples.
- 7 Uniformly finger-tighten the three thumbscrew bolts, making certain that the threads are fully engaged.
- 8 Close the Pressure DSC cell and control the pressure as directed (see [“Controlling Cell Pressure” on page 32](#)).
- 9 Enter experiment and procedure information through the TA controller, this includes both sample and instrument information.
- 10 Start the experiment. When the experiment is completed, slowly release the pressure before opening the cell (see page 35).

## Pressurizing the Cell

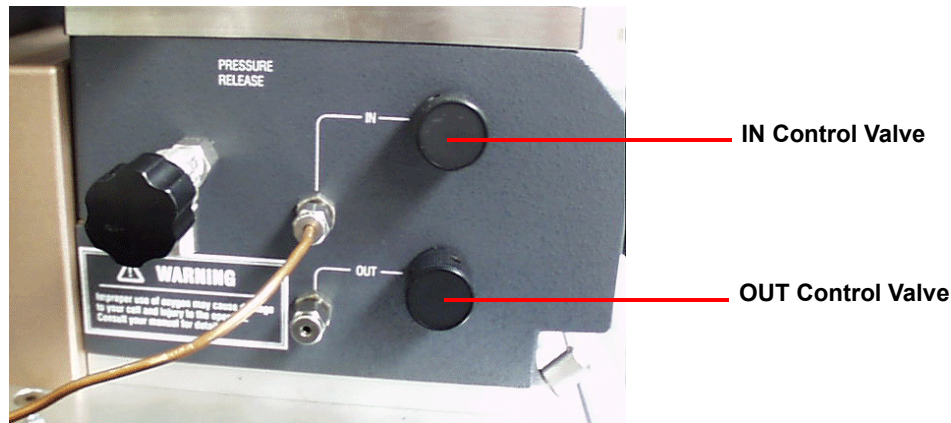
The Pressure DSC can be run at internal pressures up to 7 MPa gauge (1000 psig) or under vacuum as low as 1 Pa (10<sup>-2</sup> torr). The cell can be pressurized using either displacement or vacuum.

**NOTE:** The pressure transducer in the QPDSC cell is a 7MPa gauge full scale device. The transducer is capable of measuring vacuum, indicated as negative kPa gauge, however the offset and resolution of this device limit its usefulness under high vacuum conditions. An external gauge must be used to accurately measure high vacuum conditions.

### *Pressurizing By Displacement*

Refer to the figures for the appropriate valves.

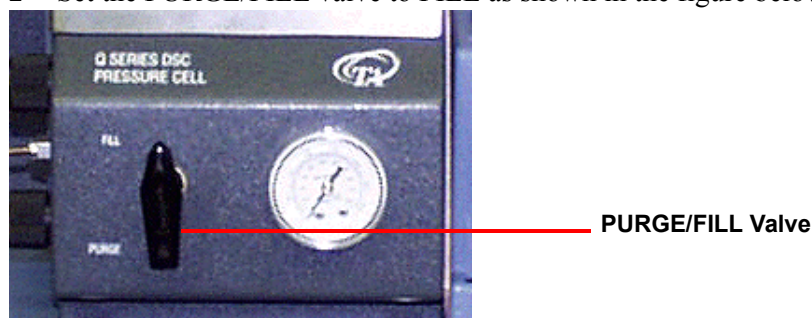
- 1 Close the IN control valve and close the OUT control valve.



**Figure 12**



- 2 Set the PURGE/FILL valve to FILL as shown in the figure below.



**Figure 13**

**NOTE:** The FILL position should be used anytime that the cell is being pressurized. This includes whether the gas is being controlled at static conditions or with a dynamic gas flow. The PURGE position should only be used at ambient pressure when desiring a dynamic purge.

- 3 Set the output regulator on the source gas cylinder to the maximum initial pressure of the experiment. If the cell is to be operated at constant volume, do not exceed 7 MPa gauge (1000 psig).
- 4 Slowly open the IN control valve and allow gas to fill the cell to about 2 MPa gauge (300 psig).
- 5 Close the IN control valve, then slowly open the Pressure Release valve and allow the pressure to return to ambient.
- 6 Close the Pressure Release valve.
- 7 Open the IN valve and allow the pressure to build to the desired level.

**NOTE:** When running isothermal experiments where it is desirable to convert to the gas of interest after reaching temperature (e.g., oxidative stability evaluations), the cell should be initially pressurized at room temperature with an inert gas (e.g., nitrogen) to a pressure slightly below the desired final pressure. Then thermal pressurization by displacement process should be used (one empty and fill sequence only) to introduce the gas of interest. The gas of interest should be introduced at the pressure of interest, which will be slightly above the pressure of the inert gas being replaced.

### ***Pressurizing By Evacuation***

- 1 Attach a vacuum pump and hose to the Pressure Release valve outlet. Insert a gauge for measuring pump head pressure into the hose using a tee fitting.
- 2 Close all three valves on the cell: OUT, IN, and Pressure Release.
- 3 Set the PURGE/FILL valve to FILL.

**NOTE:** The FILL position should be used anytime that the cell is being pressurized. This includes whether the gas is being controlled at static conditions or with a dynamic gas flow. The PURGE position should only be used at ambient pressure when desiring a dynamic purge.

- 4 Start the vacuum pump and open the Pressure Release valve. Then slowly open the IN valve to introduce source gas into the cell. Monitor the head pressure of the vacuum pump while the gas flows through the cell. Adjust this pressure with the IN valve. Do not allow the head pressure to exceed the manufacturer's limits for the pump.
- 5 Allow the gas to flow through the cell for several minutes.

- 6 Close the Pressure Release valve first, then shut off the vacuum pump and open the IN valve, allowing the pressure to build to the desired level.

**NOTE:** When working with hydrogen, the evacuation approach is preferred to ensure that all oxygen is removed from the cell before hydrogen is introduced.

## Controlling Cell Pressure

Before you begin your experiment, make sure you have charged (filled) the Pressure DSC Cell to the pressure required for your experiment. Guidelines for operation at constant volume, constant pressure, and dynamic pressure are given here. Refer to the figure to the right for the location of the PURGE/FILL valve. As you perform experiments, the pressure will be stored in the data file.

**NOTE:** During an experiment, if the pressure transducer indicates a pressure greater than 7.6 MPa gauge (1100 psig), the experiment will be terminated.

### *Operation at Constant Volume*

**NOTE:** In constant volume operation, the cell is initially pressurized to a desired value and then all entrance/exit ports are closed. The actual cell pressure during the experiment will increase as the temperature rises. The cell pressure signal is displayed as a real-time signal and, if selected, can also be saved as a data file signal.

After pressurizing the cell, check that all three cell valves are closed, that the cell is near the desired pressure, and the PURGE/FILL valve is set to FILL. Use the cell pressure shown on the instrument display to determine the internal pressure of the cell.

If the cell pressure is lower than the desired starting pressure, use the IN valve to raise it. If the cell pressure is too high, use the OUT valve to lower it. However, use the IN and OUT valves conservatively; there is a lag in the reading of any pressure gauge, and if the valves are opened too rapidly or too far, the final pressure will overshoot or undershoot the desired starting pressure.



**WARNING: The maximum permissible starting pressure for constant volume operation is less than 7.0 MPa gauge (1000 psig) at room temperature. DO NOT exceed this value.**

**AVERTISSEMENT: La pression de démarrage admissible maximale pour un fonctionnement à volume constant est inférieure à une pression manométrique de 7,0 MPa (1000 psig) à température ambiante. NE dépassez PAS cette valeur.**

Once the final pressure is achieved, close all cell valves.

### *Operation at Constant Pressure*

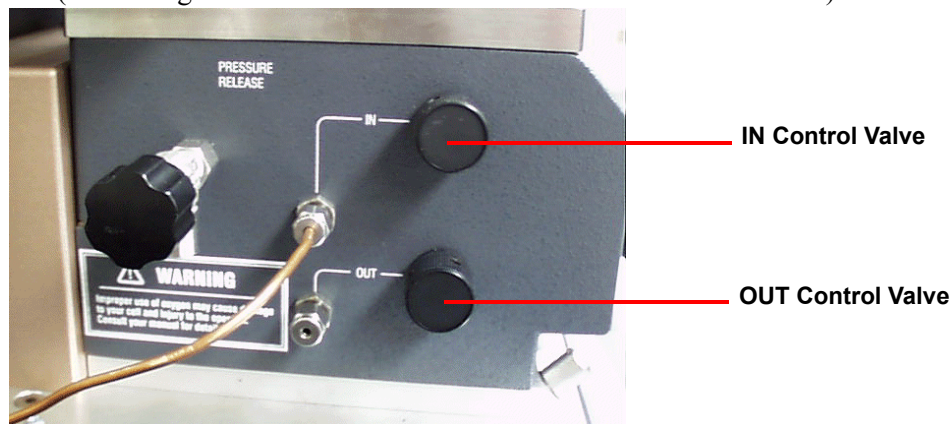
**NOTE:** In constant pressure operation, the cell is initially pressurized to a desired value. A slight bleed through the OUT valve is then created using a flowmeter. This arrangement allows the increase in pressure, which occurs as the temperature is increased, to be bled off.

For work at constant pressure, a flowmeter is required at the OUT valve to allow bleed-off of excess pressure.



After initial pressurization, ensure that all three cell valves are closed and the cell is near the desired pressure with the PURGE/FILL valve set to FILL.

- 1 Set the source gas regulator at the desired operating pressure.
- 2 Check the connections of the unrestricted flowmeter at the OUT fitting on the cell. Close the OUT valve. (See the figure below for the locations of the IN and OUT valves.)



**Figure 14**

- 3 Slowly open the IN valve on the cell. Wait for the internal cell pressure, as indicated on the instrument display, to stabilize at the desired operating pressure.
- 4 Slowly open the OUT valve.
- 5 Using the OUT valve, adjust the flow through the unrestricted flowmeter to 1 L/min. Gas should vent from the cell. Wait for the internal cell pressure to stabilize at the desired operating pressure.
- 6 Turn the PURGE/FILL valve to FILL.

|  |                                                                                                                                                                                                                                                                                                                           |
|--|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
|  | <p><b>WARNING: Do not place any restrictions in the line after the flowmeter. A restricted line will cause the flowmeter to become pressurized.</b></p> <p><b>AVERTISSEMENT: Ne placez aucune restriction dans la conduite après le débitmètre. Une conduite restreinte entraîne la pressurisation du débitmètre.</b></p> |
|--|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|

### ***Operation with Dynamic Pressure (Fixed Purge Rate)***

**NOTE:** In dynamic pressure (constant flow) operation, the cell is initially pressurized to a desired value. A dynamic purge is then established by opening both the IN and OUT valves. This arrangement maintains a constant exhaust flow rate.

After initial pressurization of the cell, ensure that all three cell valves are closed, that the cell is near the desired pressure, and that the PURGE/FILL valve is set to FILL. An unrestricted flowmeter is required at the OUT fitting for operation in this mode.

- 1 Set the regulator at the source gas cylinder to an appropriate pressure.
- 2 Slowly open the IN valve.
- 3 Slowly open the OUT valve. Wait for the flow measured at the flowmeter to stabilize. If finer flow adjustment is desired, a metering flow valve may be connected between the OUT port and the flowmeter.

- 4 Adjust the OUT valve until the flowmeter indicates the desired value (typically about 50 mL/min). If the flow rate is too low with the OUT valve fully opened, check the position of the IN valve. Carefully open the IN valve further if necessary. If this does not raise the flow to the desired rate, the source gas pressure must be adjusted. IN Control Valve OUT Control Valve
- 5 To readjust the source gas pressure, close all three valves, then repeat this procedure from step 1.
- 6 Wait until the pressure and flow rate are at the desired values. Turn the PURGE/FILL valve to FILL.

Because a flowmeter in this position is venting to the atmosphere, be sure to take the pressure differential into account when calculating flow rate over the sample at an elevated pressure.



**WARNING: Do not place any restrictions in the line after the flowmeter. A restricted line will cause the flowmeter to become pressurized.**

**AVERTISSEMENT: Ne placez aucune restriction dans la conduite après le débitmètre. Une conduite restreinte entraîne la pressurisation du débitmètre.**

## Releasing Cell Pressure

After a PDSC run is complete, slowly release the cell pressure by opening the Pressure Release valve.



**WARNING:** The exhaust gas from the Pressure Release valve may be hot enough to cause burns, fires, or damage to materials.

**AVERTISSEMENT:** Le gaz d'échappement issu du DÉTendeur DE PRESSION peut être assez chaud pour provoquer des brûlures, des incendies ou des dégâts sur le matériel.

**CAUTION:** Rapid release of pressure can cause damage to the cell.

**MISE EN GARDE:** Une détente rapide de la pression peut endommager la cellule.



**WARNING:** When running with hydrogen as the purge gas, the cell should be vented into an exhaust hood and then repurged with an inert gas (helium) prior to opening the pressure container.

**AVERTISSEMENT:** Lors d'une mesure avec de l'hydrogène en tant que gaz de purge, la cellule doit être dépressurisée dans une hotte aspirante puis purgée avec un gaz inerte (hélium) avant d'ouvrir le conteneur de pression.

## Operating Under Vacuum

To operate the Pressure DSC under vacuum, connect a vacuum system to the Pressure Release valve, and leave the two other valves closed.

To maintain normal sensitivity and resolution under vacuum, you may need to use a thermally conductive material (preferably a paste) between the constantan disc and the pans. Silicone heat-sink greases (Dow Corning type 340 or equivalent) work very well. Silicone high-vacuum greases may also be used. These should not be used at temperatures over 200°C.

## Operating in Modulated Mode

Modulated DSC® experiments can be run in the Q2000/Q1000 Pressure DSC cell while the cell is pressurized. (The MDSC® option is not available for the Q20P/Q10P instrument.) This allows pressurized MDSC® data files to be obtained.

Experimental parameters (e.g., modulation period, modulation amplitude) are selected using the same criteria described in the Advantage Q Series™ software online help for conventional ambient pressure DSC. Note, however, that the acceptable range for those parameters will vary depending on the nature and pressure of the purge gas. To achieve meaningful results in Modulated DSC mode, experiments should be conducted at temperatures above 100°C.

## Operating at Subambient Temperatures

The Pressure DSC cell is normally used for experiments at or above ambient temperatures; however, it can be used for subambient experiments provided you follow the instructions below. This requires the use of the quench cooling can, PN 900674.901, an optional accessory available from TA Instruments.

- 1 Close the IN control valve to shut off the gas supply to the cell.
- 2 Slowly open the Pressure Release valve and leave it open to ensure that the cell is at ambient pressure.
- 3 Unscrew the three thumbscrew bolts from the top plate. Do not use tools to open or close the cell.

**CAUTION: If you have difficulty unscrewing the thumbscrew bolts (excessive bolt friction), it is likely that the cell is still under some pressure or the cell temperature is too high. (This is a safety feature.) Recheck the valve positions as described in steps 1 and 2 or allow the cell to cool to less than 50°C (greater than 50° is a burn hazard).**

**MISE EN GARDE: Si vous avez des difficultés pour dévisser manuellement les boulons de vis (friction trop forte), il est probable que la cellule est toujours sous une certaine pression ou la température de la cellule est trop élevé (protection de sécurité). Vérifier la position de la vanne comme décrit dans les étapes 1 et 2 ou permettre à la cellule de refroidir à moins de 50 ° C (plus de 50 ° C représente un risque de brûlure).**

- 4 Remove the top plate, cell cover, and silver lid.

**CAUTION: If the cell has just been used, these components could be very hot. As a safe operating practice, use leather gloves when handling the top plate, and use tweezers whenever handling the metal cell cover or silver lid.**

**MISE EN GARDE: Si la cellule vient d'être utilisée, ces composants pourraient être très chaud. Pour une meilleure sécurité, utiliser des gants de cuir lors de la manipulation de la plaque supérieure, et utiliser des pinces de manutention pour le couvercle de la cellule en métal ou le couvercle d'argent.**

- 5 Load the sample and reference pans with the sample in the front and the reference in the back.
- 6 Place the quench cooling can in the top of the Pressure DSC cell and add liquid nitrogen or other suitable coolant (e.g., dry ice, etc.). See the cautions and warnings regarding liquid nitrogen safety in the front of this manual.

**CAUTION: DO NOT pour liquid nitrogen directly into the Pressure DSC cell as you may damage the cell.**

**MISE EN GARDE: Ne pas verser de l'azote liquide directement dans la cellule de DSC sous pression car vous pourriez endommager la cellule.**

- 7 When the cell has reached the desired low temperature, remove the quench cooling can, then replace the silver lid, cell cover, and top plate. Push the top plate down as far as it will go, taking care not to damage the O-ring or jar the cell, which could cause the pans to move off the dimples.

**NOTE:** The lowest recommended temperature to achieve stable performance with the DSC pressure cell is -130°C.

- 8 Uniformly finger-tighten the three thumbscrew bolts, making certain that the threads are fully engaged.
- 9 Pressurize the cell and start the experiment. When the experiment is completed, slowly release the pressure before opening the cell (see page 35).

## *Maintenance and Diagnostics*

The primary maintenance procedures described in this section are the customer's responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel. Consult the online documentation installed with the instrument control software for further information.



**WARNING: Because of the high voltages in the DSC, untrained personnel must not attempt to test or repair any electrical circuits.**

**AVERTISSEMENT: À cause de la présence de tensions élevées dans cet instrument, le personnel non formé ne doit pas essayer de tester ou de réparer les circuits électriques.**

**CAUTION:** Before using any cleaning or decontamination method except those recommended by the manufacturer, users should check with the manufacturer that the proposed method will not damage the equipment.

**MISE EN GARDE:** Avant d'utiliser une méthode de nettoyage ou de décontamination autre que celle recommandée par le fabricant, vérifiez auprès de le fabricant que la méthode proposée n'endommagera pas l'instrument.

### **Cleaning a Contaminated Pressure Cell**

A poor baseline is often the sign of a contaminated cell. PDSC cells must be cleaned properly to maintain satisfactory operation. Scraping the contamination off the cell's constantan disc is not recommended because the disc is very thin (about 0.1 mm, or 0.004 inches), and if the disc deforms, the baseline may be affected.

If your baseline appears to show sample contamination, try the following recommended cleaning procedure:

- 1 Remove any pans from the cell.
- 2 Connect the air purge.
- 3 Lightly brush out the cell with a small fiberglass eraser (included in the DSC accessory kit).
- 4 Clean the disk with air.

**NOTE:** Be sure to wear safety glasses or goggles when cleaning the cell with air.

- 5 Begin cleaning by heating the cell with an air purge to 50 °C above the highest operating temperature or 600 °C, whichever is lower. Use a heating rate of 20 °C/min. Ramp to 600 °C, then hold isothermally for 15 minutes.
- 6 After the cell has cooled down, repeat steps 3 and 4.

- 7 Run the experiment again and compare the baselines. If there is a marked improvement but the baseline is still unacceptable, the contaminant probably oxidized and reduced to an inert ash. Run the experiment again and check for further improvement.
- 8 Once the baseline is acceptable, return to normal operation.

If the constantan disc looks clean and is not bent or cracked, but the baseline problem remains, it is probably not due to contamination; the cell may need to be replaced (contact your TA Instruments service representative).

## Lapping the Silver Lid and Ring

If sinusoidal baseline noise is observed in a PDSC heat flow curve obtained under pressure, the silver lid and gas ring (the silver ledge on which the lid sits) may have become slightly warped and should be smoothed with the lapping tool before the next run. The lapping tool is provided with the PDSC cell.

- 1 Place the silver lid, handle side up, on a piece of fine (600 grit) emery paper backed by a flat, smooth surface, and move the lid in a figure-eight motion until any deformed areas are smoothed.
- 2 To smooth the gas ring, attach a piece of abrasive paper (400 grit) to the lapping tool with the double-sided tape provided. Trim the paper to the size of the tool using scissors or a razor knife. Rotate the lapping tool (PN 008837.001) back and forth on the ring with light-to-moderate pressure. Clean afterward with a fiberglass brush and a light air blast.

**NOTE:** Be sure to wear safety glasses or goggles when cleaning the cell with air.

## *Replacement Parts*

The table below lists the replacement parts for the DSC that are available from TA Instruments. See the list of offices when ordering parts.

**Table 3: PDSC Replacement Parts**

| <b>Part Number</b> | <b>Description</b>                  |
|--------------------|-------------------------------------|
| 202813.039         | O-ring, PDSC Pressure Cylinder Seal |
| 008837.001         | Gas ring lapping tool               |
| 900969.001         | Silver PDSC lid                     |
| 900601.901         | PDSC insulation                     |
| 900674.901         | DSC Quench Cooling Can              |
| 970800.901         | Replacement PDSC Cell               |