DISCOVERY DSC 25P
PRESSURE CELL DIFFERENTIAL SCANNING CALORIMETER

Getting Started Guide

Revision B Issued November 2017

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
- TA Instruments Patents
- Other Trademarks
- TA Instruments End-User License Agreement
- TA Instruments Offices

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.

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.

Regulatory Compliance

Safety Standards

For Canada

CAN/CSA-C22.2 No. 61010-1-12 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-C22.2 No. 61010-2-010-15 Particular requirements for laboratory equipment for the heating of materials.

CAN/CSA-C22.2 No. 61010-2-081-15 Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes.

For European Economic Area

(In accordance with Council Directive 2014/35/EU of the European Parliament and of the Council of 26 February 2014 on the harmonization of the laws of the Member States relating to the making available on the market of electrical equipment designed for use within certain voltage limits.)

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

EN 61010-2-010:2014 Particular requirements for laboratory equipment for the heating of materials.

EN 61010-2-081:2015 Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes.

For United States

UL61010-1:2012 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

UL61010-2-010:2015 Particular requirements for laboratory equipment for the heating of materials.

UL 61010-2-081:2015 Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes.

Electromagnetic Compatibility Standards

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).

CAUTION: This equipment is not intended for use in residential environments and may not provide adequate protection to radio reception in such environments.

MISE EN GARDE: Cet appareil n'a pas destiné à être utilisé dans des environnements résidentiels et ne peut pas fournir une protection adéquate à la réception radio dans de tels environnements.

For the European Economic Area

EN61326-1; 2013. Class A conducted and radiated emissions. Table 1 Immunity: Basic Electromagnetic Environment.

Safety

Instrument Symbols

The following labels are displayed on the DSC 25P for your protection:

Symbol	Explanation
	This symbol appears on the top cover of the pressure vessel, indicating that the vessel may be under pressure and 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. Ce symbole apparaît sur le capot supérieur du récipient sous pression, indiquant que le navire peut être sous pression et que "l'utilisation incorrecte d'oxygène ou d'hydrogène peut endommager la cellule ou nuire à l'utilisateur". Voir l'AVER-TISSEMENT sur la page suivante Pour plus de détails.
	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. 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.

Please heed the warning labels and take the necessary precautions when dealing with these areas. The *Discovery DSC Getting Started Guide* and this *Discovery DSC 25P 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 DSC 25P

WARNING: If excessive amounts of hydrocarbons are present in the DSC 25P, energetic combustion could occur causing damage to the DSC 25P 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 DSC 25P 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 DSC 25P 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 25P, il peut se produire une combustion énergétique susceptible d'endommager la DSC 25P 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 DSC 25P 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 DSC 25P 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 DSC 25P and any of the following conditions apply to you.

- New installation of a DSC 25P
- Modification of supply lines, valves or gauges
- Sample was spilled in the DSC 25P
- DSC 25P has an “oily” smell
- DSC 25P has not been used recently.

You may ensure safe operation of your DSC 25P 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 DSC 25P:

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 (350°C) 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 (350 °C) 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 bâche 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 raccordements entre les éléments doivent être hermétiques et étanches.

If you have any questions about hydrogen use, contact the U.S. TA Instruments Thermal Support Helpline at http://www.tainstruments.com/support/applications/applications-hotline/

Contact our U.S. Thermal Support Helpline or your local TA Instruments Representative if you have any questions regarding the safe usage of the TA Instruments DSC 25P.

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.

Electrical Safety

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.

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 DSC 25P, cold surfaces 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.

Table of Contents

Chapter 1:

Introducing the Discovery DSC 25P

Overview

The DSC 25P utilizes a Discovery DSC cell design that is enclosed in a steel cylinder. It 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 DSC 25P cell can operate at elevated pressure or under vacuum. This ability to vary pressure as well as temperature provides the following capabilities:

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

Figure 1 Discovery DSC 25P.

The DSC 25P cell is mounted to a pressure manifold which uses two flow control valves to control the flow in and out of the pressure vessel and a three-way valve to select either control valve or to close the gas input. One control valve is used to regulate the flow into the pressure vessel and the other valve controls the purge flow through the cell itself. These valves are mounted to the front panel of the instrument. The pressure is measured by a transducer which is calibrated from 0-7MPa gauge (0-1000 psig) and a pressure relief valve rated for 1200 ±50 psig will vent overpressurization. As an additional safety measure, there is an analog dial pressure gauge mounted to the front panel next to the control valves. On the side panel there is a 1/8 inch compression fitting to connect the pressurizing gas using 1/8 inch outer diameter metal tubing. Next to the Gas In fitting is a Flow Out valve that is used to connect to an external flowmeter when per-

forming experiments with dynamic flow. A **PRESSURE RELEASE** valve on the right is used to vent the pressure at the end of the experiment or to vent excess pressure when setting the initial pressure at the start.

DSC 25P experiments are typically run by pressurizing the cell to a desired initial pressure and then closing the 3-way valve and running the experiment, allowing the pressure to change with the time/temperature profile of the experiment. The pressure is monitored continuously and can be viewed on both the touchscreen and TRIOS, and can be recorded in the data file. Use of an external flowmeter and the flow and purge control valves enable experiments to be run at constant pressure and with a dynamic flow over the sample during an experiment.

DSC 25P System Components

A functional DSC 25P system consists of the following components: the Discovery DSC 25P instrument, a pressure vessel, silver lids, and metal cell cover. An optional Quench Cooler for cooling the cell to subambient temperature is also available.

Pressure Vessel

The pressure vessel consists of a steel cylinder, lid, and three bolts that are used to secure the lid for use in pressure DSC experiments. The vessel can be pressurized up to 7 MPa gauge (1000 psig, 70 bar) or evacuated to run experiments under vacuum. The lid contains a rubber O-ring that creates the seal to the vessel; it must be intact and kept clean in order to maintain pressure within the vessel.

Figure 2 Pressure vessel (without lid).

Refer to “Pressurizing the Cell” for more information.

Silver Lids

Two silver lids are used to cover the cell body. The contact of the silver lids to the cell will influence the baseline shape, slope and offset. For optimal performance, they both must be kept clean and free of any scratches or any other physical damage that may occur from dropping the lid. Make sure that the lid and the cell body surface that it rests on are clean and free of dents or scratches (especially the inner lid).

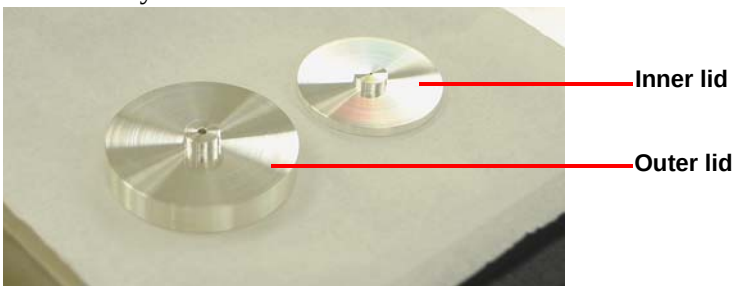

Figure 3 Inner and outer silver lids.

Metal Cell Cover

The cell body and lids are covered by a metal cover.

Figure 4 Metal cover.

Metal Tray

The metal tray located on the top of the DSC 25P can be used as a workspace and an area to store pans. Holding the tray by the knob on the top right corner, insert the tray into the tray cutout on top of the DSC 25P.

Figure 5 Metal tray.

Optional Quench Cooler

The optional Quench Cooler (P/N 970316.000) consists of an upper section that you can manually fill with coolants such as water/ice, dry ice or liquid nitrogen, and a lower section that rests in contact with the cooling flange. The cooler can be used to cool the cell to a sub ambient starting temperature as well as simply to expedite cooling the cell back to ambient after an experiment. The cooler does not remain in place during an experiment, after cooling to a sub ambient starting temperature, the cooler is quickly removed, the cell pressurized and the experiment is started. The temperature at which a stable heating rate can be achieved will depend on how quickly this can be accomplished. The lower temperature will be limited by the choice of coolant, the -130°C limit can only be reached using liquid nitrogen. When cooling the cell below ambient, the pressure vessel should be purged with a dry gas via the fill valve to prevent the formation of frost within the pressure vessel. Refer to “Installing/Uninstalling the Quench Cooler” for installation procedures.

Figure 6 Quench Cooler.

The Discovery DSC 25P User Interface

The Discovery DSC 25P includes an integrated user interface display for local operator control. The functions of the user interface change depending upon the view displayed. This section briefly describes the basic layout of these functions.

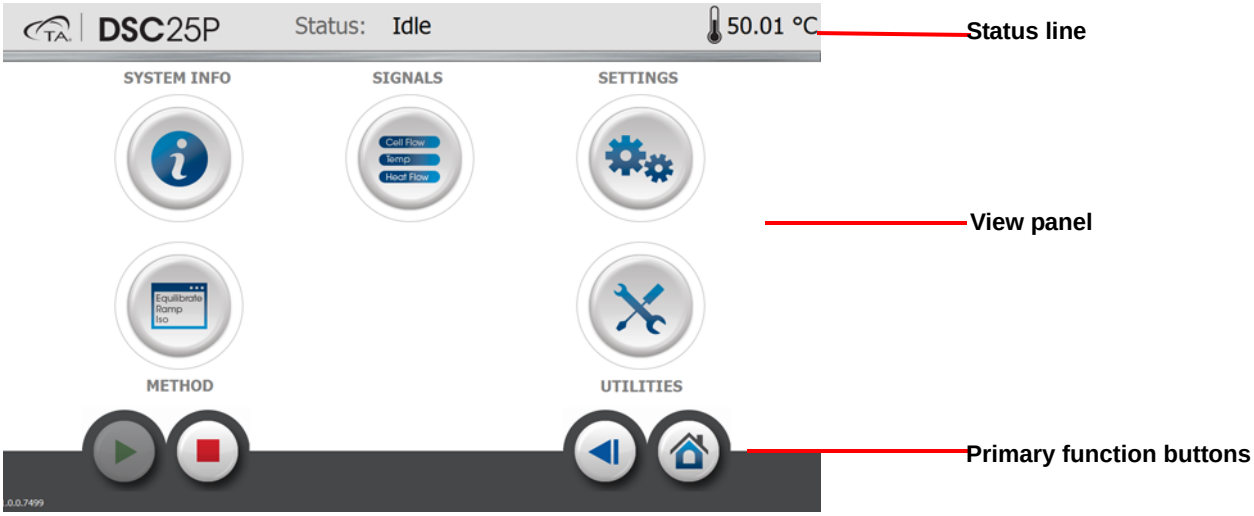

Figure 7 Discovery DSC 25P user interface display.

The instrument user interface has three basic sections:

- The status line along the top of the display indicates the current instrument status and sample temperature.
- View panel allows for real-time instrument status and associated actions.
- The primary function buttons allow for easy access of common functions.

Primary Function Buttons

Use the following buttons for the main functions of the instrument.

Table 1: Primary Function Buttons on the User Interface

Key Name	Description
Start 	Begins the currently-programmed experiment. This is the same function as **Start** on the instrument control software.
Stop 	If an experiment is running, this button ends the method normally, as though it had run to completion; i.e., the method-end conditions go into effect and the data that has been generated is saved. This is the same function as **Stop** on the instrument control software. If an experiment is not running (the instrument is in a standby or method-end state), the **Stop** button will halt any activity (Standby Temperature, etc.).
Back 	Returns to the previously used screen.
Home 	Returns to the Main screen.

View Panel

The view panel provides real-time instrument status and additional functionality pertinent to the selected operation. A list of available functions is described below.

Table 2: View Panel Functions on the User Interface

Button Name	Description
System Info 	Displays instrument information such as the serial number, IP configuration, and network configuration.
Signals 	User can select signals to display the real-time signal data generated directly from the instrument.
Settings 	Displays options for manual controls, such as Event and Standby Temperature.
Method 	Provides a summary of the sample and method information for the current run and allows the user to manually advance the method.
Utilities 	Displays system health and shut down button, as well as a real-time running plot of the Tzero® temperature and pressure signals.

Additional Function Buttons

Button Name	Description
Help 	The **Help** button can be found on the lower right side of some screens, and displays information regarding use of the currently displayed touchscreen.

DSC 25P Specifications

The table found below contains the technical specifications for the DSC 25P

Table 3: DSC 25P Technical Specifications

Item/Area	Specification
Dimensions	Depth: 26 in Width: 24 in Height: 19.5 in
Weight	35 kg (77 lbs)
Temperature Range	–130 to 550°C; 350°C with helium or hydrogen
Atmosphere	–100 kPa gauge to 7 MPa gauge (1 Pa to 7.1 MPa gauge absolute), constant pressure or constant volume
Dynamic gas purge	To 200 mL/min (cell flow rate at standard temperature and pressure)
Purge gases	Nitrogen, air, oxygen, carbon monoxide, carbon dioxide, hydrogen, helium, argon
Heat flow	T1 Heat Flow only

NOTE: Performance depends on the pressure and atmosphere selected.

Chapter 2:

Installing the Discovery DSC 25P

Unpacking/Repacking the DSC 25P Instrument

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

WARNING: Have an assistant help you unpack this unit. Do not attempt to do this alone.

AVERTISSEMENT: Faites-vous aider par une personne pour dépoter cet appareil. N'essayez pas de le faire tout seul.

Installing the Discovery DSC 25P System

Before shipment, the DSC 25P is inspected both electrically and mechanically so that it is ready for operation upon proper installation. Only limited instructions are given in this manual; consult the online help for additional information. Installation involves the following procedures:

- Inspecting the system for shipping damage and missing parts.
- Choosing a location for instrument installation.
- Connecting the TA Instruments controller and instrument to the router.
- Connecting the power cables.

It is recommended that you have your DSC 25P installed by a TA Instruments Service Representative; call for an installation appointment when you receive your instrument.

CAUTION: To avoid mistakes, read this entire chapter before you begin installation.

MISE EN GARDE: Pour éviter de commettre des erreurs, lisez tout le chapitre avant de commencer l'installation.

Inspecting the System

When you receive your Discovery DSC 25P system, look over the instrument and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

- If the instrument is damaged, notify the carrier and TA Instruments immediately.
- If the instrument is intact but parts are missing, contact TA Instruments.

Choosing a Location

Because of the sensitivity of DSC 25P experiments, it is important to choose a location for the instrument using the following guidelines. The Discovery DSC 25P system should be:

In

- A temperature-controlled area.
- A clean, vibration-free environment.
- An area with ample working and ventilation space.

On

- A stable, non-flammable work surface.

Near

- A properly grounded power outlet on a branch circuit of 20 amps maximum.
- Your TA Instruments thermal analysis controller (computer).
- Purge gas supplies with suitable regulators.

Away from

- Dusty environments.
- Exposure to direct sunlight.
- Direct air drafts (fans, room air ducts).
- Poorly ventilated areas.
- Noisy or mechanical vibrations.

NOTE: Allow free air to circulate around the instrument. Do not place equipment against walls or cabinets that might impede air flow. Leave at least 7.5 cm (3 in) clearance around the Discovery DSC 25P.

WARNING: For safety, position the equipment in a manner that allows access to the power cord for emergency disconnection.

AVERTISSEMENT: Par mesure de sécurité, placez l'équipement de sorte qu'il permette d'accéder facilement au cordon d'alimentation en cas de débranchement d'urgence.

CAUTION: Whenever plugging or unplugging power cords, handle them by the plugs, not by the cords.

MISE EN GARDE: Chaque fois que vous branchez ou débranchez les cordons d'alimentation, tenez-les par les fiches et non par les cordons.

WARNING: Protect power and communications cable paths. Do not create tripping hazards by laying the cables across access ways.

AVERTISSEMENT: Protégez les chemins de câble électriques et de câbles de télécommunication. Ne créez pas de risques de déclenchement en posant des câbles sur les voies d'accès.

Back Panel Connections

The Discovery DSC 25P back panel has ten ports; the table below provides a description of the function of each port.

Table 4: Discovery DSC 25P Back Panel

Port	Function
24 VDC OUT	May be used for certain accessories.
EVENT	This port is used for the RCS and Photocalorimeter Accessory (PCA) control.
USB 2.0 port	Provides communications for external accessories.
Micro USB 2.0 port	Provides communications for external accessories.
SD memory card slot	For TA Instruments Service use only.
CAN Communications Port	Provides communications with external accessories.
RS-232 Port	Provides communications with external accessories.
Ethernet	Provides communication between the instrument and the PC.
Audio Jack	External speaker connection.
Power Entry Module (power cord, switch, fuses)	Provides power and protective earthing to the system.

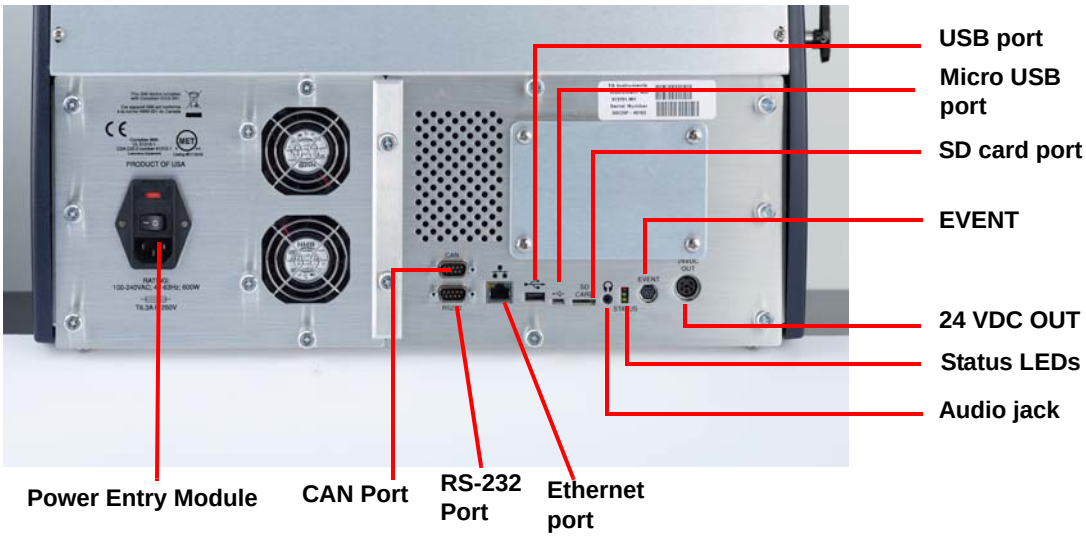

Figure 8 Discovery DSC 25P rear panel connections.

Side Panel

The side panel on the DSC 25P contains the **IN** port, Flow **OUT** valve, and **PRESSURE RELEASE** valve.

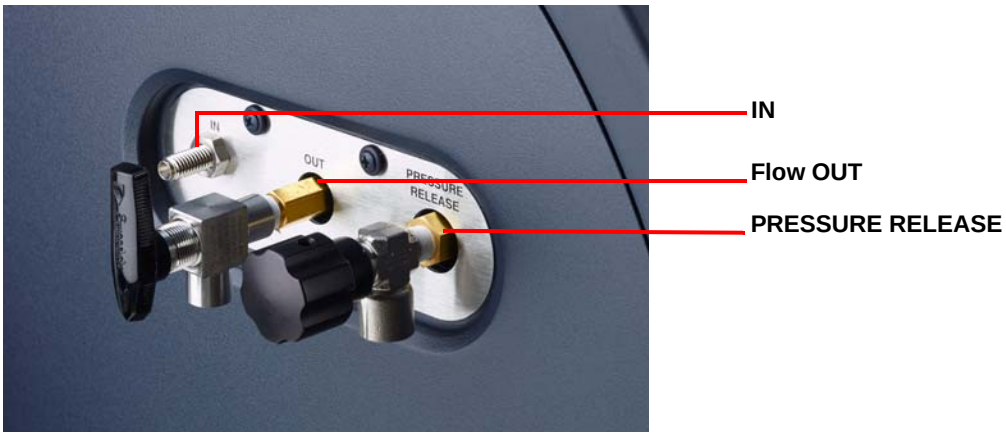

Figure 9 Side panel on the DSC 25P.

Front Panel

The front panel on the DSC 25P includes the **FILL** valve and **PURGE** valve, with a knob in between the two for valve selection (in the image below, the knob is set to **OFF**).

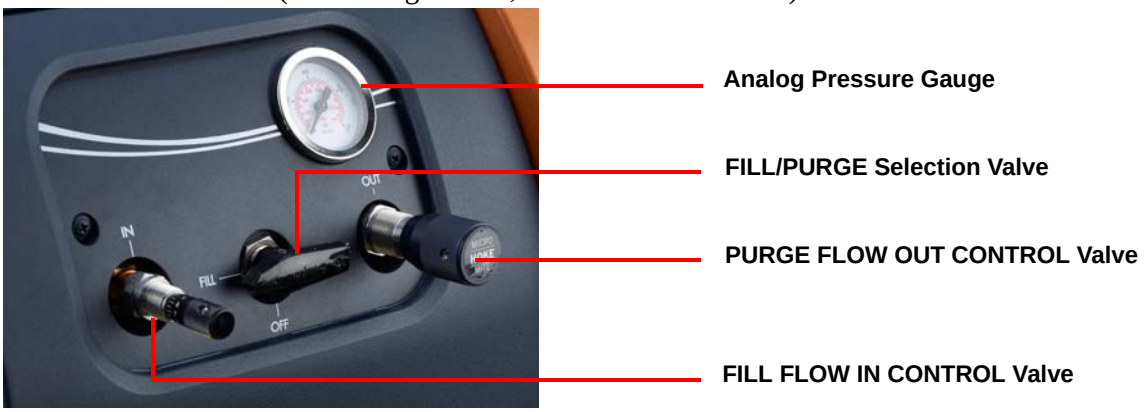

Figure 10 Front panel on the DSC 25P.

Setting Up System Communication

The instrument and controller must connect to an Ethernet router/switch for operation. The router can also be connected to a LAN to provide network storage and access, if desired.

Refer to TRIOS Software Installation Instructions for more details.

Connecting the Power Cable

NOTE: A <HAR>-marked (harmonized) power cable meeting the standards of the country of installation is required for the European Economic Area.

Install the power cable as follows:

1 Make sure the power switch is in the Off (**0**) position, as shown in Figure 11.

2 Plug the power cable into the power entry module (shown below).

Figure 11 Power entry module on the back panel of the instrument.

3 Plug the power cable into a properly grounded wall outlet on a branch circuit of 20 amps or less.

Installing/Uninstalling the Quench Cooler

When using water/ice, be especially careful not to spill the water when removing the cooling accessory. If water is spilled, then remove the cooler and pressure vessel and carefully dry the spill. If necessary, the cell can be dried by heating to and holding at 400°C for 120 min. at ambient pressure.

Use the following steps to install the Quench Cooler:

1 Using the **PRESSURE RELEASE** valve on the side panel of the DSC 25P, release the pressure slowly and completely so that the 3 pressure vessel bolts can be loosened.

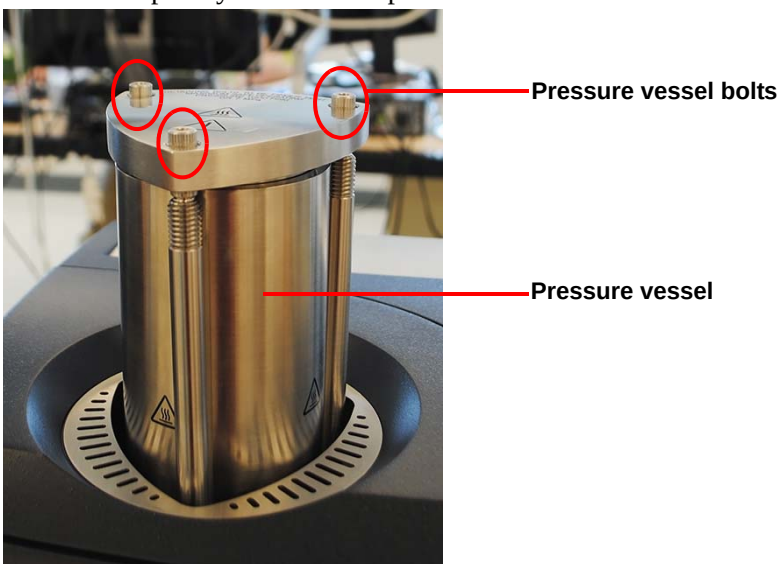

Figure 12 Pressure vessel with bolts loosened so that the lid can be removed.

2 Loosen the bolts and remove the pressure vessel lid.

Figure 13 Lid removed.

3 Remove the metal cell cover from inside of the vessel.

Figure 14 Remove cell cover.

4 Insert the cooler.

Simply reverse this procedure to remove the Quench Cooler.

Starting the Discovery DSC 25P System

The power switch is located at the back panel of the instrument. It is part of the assembly called the power entry module, which also contains the power cord connection and fuses. The power switch is used to turn the DSC 25P system on and off.

To power on the system:

1 Check the connection between the DSC 25P, Ethernet switch, and the controller. Make sure each component is plugged into the correct connection port.

2 Set the power switch to the ON (**I**) position.

3 After the proper power up sequence, the instrument user interface displays; this indicates that the instrument is ready for use.

NOTE: Allow the Discovery DSC 25P to warm up for 60 minutes before performing an experiment in order to allow time for the temperature-controlled measurement circuitry to stabilize.

Shutting Down the Discovery DSC 25P System

Before you decide to power down your system, consider the following:

- All of the components of your thermal analysis system are designed to be powered on for long periods.
- The electronics of the DSC 25P perform more reliably if power fluctuations caused by turning units on and off are minimized.

For these reasons, turning the system and its components on and off frequently is discouraged. Therefore, when you finish running an experiment on your instrument and wish to use the thermal analysis system for some other task, it is recommended that you leave the instrument on.

To ensure proper shutdown of the instrument, it is recommended that you execute a shutdown from the user interface or TRIOS software before turning off the power to the instrument. To shut down, perform one of the following options:

- Select **Shutdown** from the instrument user interface **Utilities** menu.
- Select **Shutdown** from the **Instrument** menu within the TRIOS software.

A confirmation message displays. Select **Yes** to continue. All communication to the instrument halts while the instrument saves any unsaved data. Once this procedure is complete, the instrument user interface screen goes blank (black), indicating that it is safe to turn off the power to the instrument.

To power down your system, set the power switch to the OFF (**O**) position.

Chapter 3:

Using and Maintaining the DSC 25P

Before You Begin

Please read this before using OXYGEN in the DSC 25P

WARNING: If excessive amounts of hydrocarbons are present in the DSC 25P, energetic combustion could occur causing damage to the DSC 25P 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 DSC 25P 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 DSC 25P 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 DSC 25P 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 25P, 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 jauge (3000 psig) et débarrassés d'hydrocarbures.

You should review the warnings above if you plan to use oxygen in the DSC 25P and any of the following conditions apply to you.

- New installation of a DSC 25P
- Modification of supply lines, valves or gauges
- Sample was spilled in the DSC 25P
- DSC 25P has an “oily” smell
- DSC 25P has not been used recently.

You may ensure safe operation of your DSC 25P 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:

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 bâche 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 DSC 25P 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 DSC 25P 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 jauge (3000 psig) et tous les raccordements entre les éléments doivent être hermétiques et étanches.

If you have any questions about hydrogen use, contact the U.S. TA Instruments Thermal Support Helpline at http://www.tainstruments.com/support/applications/applications-hotline/

Contact our U.S. Thermal Support Helpline or your local TA Instruments Representative if you have any questions regarding the safe usage of the TA Instruments DSC 25P.

Calibrating the DSC 25P

To obtain the best experimental results, you should calibrate the DSC 25P periodically if the purge gas, heating rate, and/or pressure is changed. These calibration experiments are performed and analyzed using TRIOS 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 DSC 25P 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.

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. When two or more temperature calibration standards are used, the highest and lowest melt temperatures should be outside the area of interest as a small discontinuity in heating rate will occur beyond those temperatures.

Pressure Calibration

Pressure calibration is an optional calibration procedure for the DSC 25P. It is based on comparing the pressure reading at two points, typically 1 atmosphere and another pressure selected by you, as indicated by the pressure reading on an external pressure gauge. The pressure transducer is already calibrated during manufacturing at TA Instruments, and–under normal circumstances–will not need to be recalibrated.

Running a Pressure DSC Experiment

Please read this before using OXYGEN in the DSC 25P

WARNING: If excessive amounts of hydrocarbons are present in the DSC 25P, energetic combustion could occur causing damage to the DSC 25P cell and possible injury to the operator. To help prevent these problems, follow these guidelines:

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(2) Cell Contamination: Remove the pressure housing and visually inspect the DSC 25P 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 DSC 25P 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 DSC 25P 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 25P, 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 jauge (3000 psig) et débarrassés d'hydrocarbures.

Please read this before using HYDROGEN in the Pressure DSC Cell:

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 bâche 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 DSC 25P 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 DSC 25P 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 jauge (3000 psig) et tous les raccordements entre les éléments doivent être hermétiques et étanches.

If you have any questions about hydrogen use, contact the U.S. TA Instruments Thermal Support Helpline at http://www.tainstruments.com/support/applications/applications-hotline/

Contact our U.S. Thermal Support Helpline or your local TA Instruments Representative if you have any questions regarding the safe usage of the TA Instruments DSC 25P.

WARNING: Any time you open 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 DSC 25P away from flammable materials.

AVERTISSEMENT: Chaque fois que vous ouvrez la vanne de décompression 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 DSC 25P à l'écart des matériaux inflammables.

Experimental Procedure

The DSC 25P 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 DSC 25P.

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 DSC 25P 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 36.
6. Enter experiment and procedure information, including both sample and instrument information. Follow the instructions for operating the DSC 25P found in this chapter.
7. Start the experiment. Do not exceed 7 MPa gauge (1000 psig) during the experiment. As a safety precaution, the experiment will be terminated above 7.6 MPa gauge (1100 psig). Keep this in mind when selecting an initial pressure, as the pressure is allowed to rise with temperature during typical pressure DSC experiments.
8. When the experiment is completed, release the pressure slowly using the **PRESSURE RELEASE** valve on the side panel of the DSC 25P before opening the cell. If the vessel is pressurized, you will not be able to turn the bolts to open the cell. If you can not turn the bolts, make sure the pressure has been fully released.

Selecting the Purge Gas

The DSC 25P 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 limit the experimental temperature conditions that can be achieved. The temperature for gas pressure is limited to 350°C for hydrogen and helium and 550°C for other gases (ex. Nitrogen).

NOTE: Upper limits may be limited at higher heating rates and higher pressure.

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 DSC 25P cell.

1 Close the **FILL/PURGE** valve to shut off the gas supply to the cell.

Figure 15 Fill/Purge valve set to **OFF**.

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 (shown below) from the top plate. Do not use tools to open or close the cell.

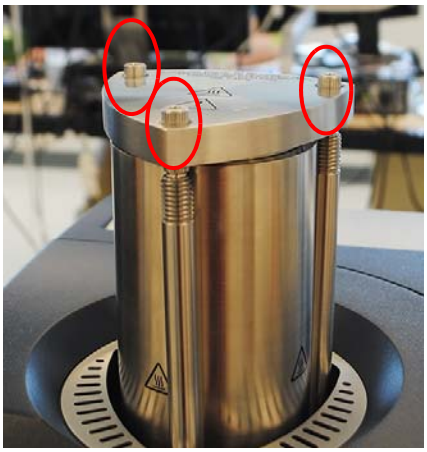

Figure 16 Thumbscrew bolts loosened.

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. Above 130°C thermal expansion prevents the bolts from unscrewing. (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 à dévisser les boulons à vis (frottement de boulon excessif), il est probable que la cellule est toujours sous une certaine pression ou la température de la cellule est trop élevée. Au-dessus de 130 ° C, la dilatation thermique empêche les boulons de se dévisser. (Ceci est une caractéristique de sécurité.) Vérifiez de nouveau les positions des soupapes comme décrit aux étapes 1 et 2 ou laissez la cellule refroidir à moins de 50 ° C (plus de 50 ° C est un risque de brûlure).

4 Remove the top plate, cell cover, and both inner and outer silver lids.

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 pans with a pinhole lid). See online help for details.

Figure 17 Pans loaded.

6 Replace the inner and outer silver lids, 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 sensors.

7 Uniformly finger-tighten the three thumbscrew bolts, making certain that the threads are fully engaged.

8 Control the pressure as directed (see “Controlling Cell Pressure” on page 38).

9 Verify the experiment and procedure information in TRIOS, including both sample and instrument information. Press the **Start** button on the instrument touch screen or TRIOS to begin the experiment.

Pressurizing the Cell

The DSC 25P can be run at internal pressures up to 7 MPa gauge (1000 psig) or under vacuum as low as 1 Pa (10-2 torr). The cell can be pressurized using either displacement or vacuum. On the instrument touch-screen, navigate to **Utilities** > **Diagnostics** > **Pressure** to view the pressure and Tzero® temperature real-time signals. This may be helpful when setting the desired pressure.

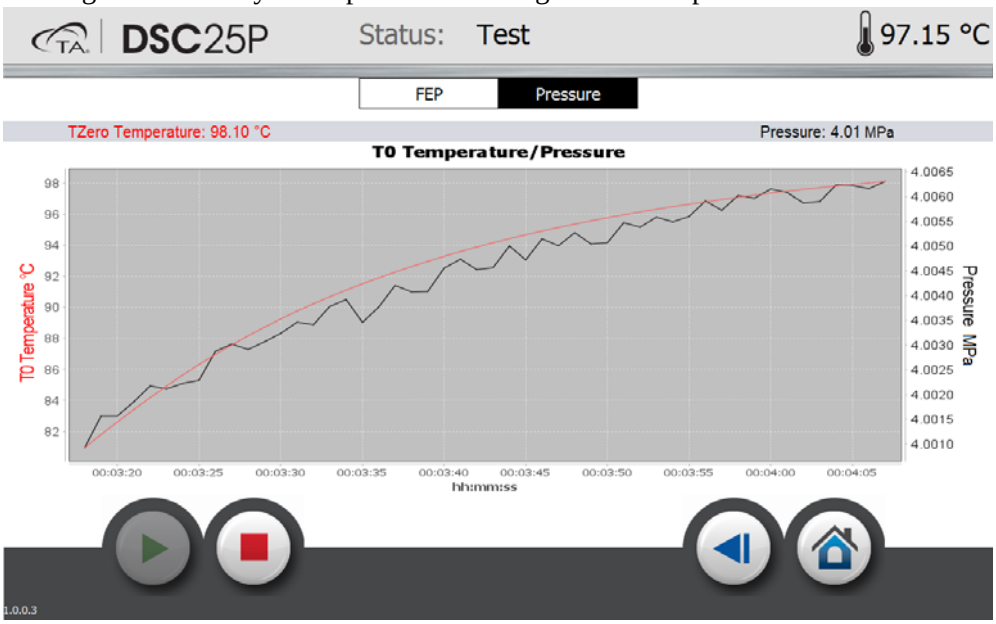

Figure 18 Pressure screen.

NOTE: Most Pressure DSC experiments are run by pressurizing the pressure vessel to a desired staring pressure, closing the 3-way valve and running the experiment. It is important to remember that when filling the pressure vessel that it was initially filled with atmospheric air. If you simply pressurize it and run it, all of the oxygen initially present in the vessel will remain and will potentially interact with your sample and, if the temperature exceeds 400°C, the materials of construction of the sensor.

It is strongly recommended that you follow the procedures described below, “Pressurizing By Displacement” and/or “Pressurizing By Evacuation” to preserve the integrity of your pressurizing gas. These procedures are particularly important if your experimental temperature exceeds 400°C, in which case it is recommended that you use an inert gas such as nitrogen or argon. Use of other gases are acceptable for periods of limited duration; however, you are advised to seek technical assistance, if necessary.

When using hydrogen gas, it is extremely important to remove oxygen from the pressure vessel before introducing hydrogen. In this situation, it is strongly recommended that “Pressurizing By Displacement” and/or “Pressurizing By Evacuation” procedures be followed very carefully. If using the “Pressurizing by Displacement” method, then it is recommended that you use helium as the displacement gas prior to introducing hydrogen.

NOTE: The pressure transducer in the DSC 25P cell is calibrated up to 7MPa gauge. 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 Figure 9 and Figure 10 for the locations of the appropriate valves.

1 Close all three valves on the cell: the **FILL FLOW INCONTROL** valve, the **PURGE FLOW OUT CONTROL** valve, and the **PRESSURE RELEASE** valve. Do not overtighten the **FILL FLOW IN CONTROL** and **PURGE FLOW OUT CONTROL** valves.

2 Set the **FILL/PURGE** valve to **FILL**.

3 Set the output regulator on the source gas cylinder to the maximum initial pressure of the experiment.

NOTE: If the cell is to be operated at constant volume, do not exceed 7 MPa gauge (1000 psig).

4 Slowly open the **FILL FLOW INCONTROL** valve and allow gas to fill the cell to about 4 MPa gauge (580 psig).

5 Close the **FILL FLOW IN CONTROL** valve, then slowly open the **PRESSURE RELEASE** valve and allow the pressure to return to 0.1 MPa gauge.

6 Close the **PRESSURE RELEASE** valve.

7 Open the **FILL FLOW IN CONTROL** valve and allow the pressure to build to the desired level.

This procedure can be repeated as necessary if samples sensitive to atmospheric contamination are being evaluated.

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

Pressurizing By Evacuation

Refer to Figure 9 and Figure 10 for the locations of the appropriate valves.

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.

1 Close all three valves on the cell: the **FILL FLOW IN CONTROL** valve, the **PURGE FLOW OUT CONTROL** valve, and the **PRESSURE RELEASE** valve. Do not overtighten the **FILL FLOW IN CONTROL** and **PURGE FLOW OUT CONTROL** valves.

2 Set the **FILL/PURGE** 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.

3 Start the vacuum pump and open the **PRESSURE RELEASE** valve. Then slowly open the **FILL** valve to evacuate the gas in the cell. Monitor the head pressure of the vacuum pump while the gas flows out of the cell. Adjust this pressure with the **FILL FLOW IN CONTROL** valve. Do not allow the head pressure to exceed the manufacturer's limits for the pump.

4 Allow the pressure to stabilize at the desired level.

5 Close the **PRESSURE RELEASE** valve first, then shut off the vacuum pump and open the **FILL FLOW IN CONTROL** 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 pressurized the DSC 25P to the pressure required for your experiment. Guidelines for operation at constant volume, constant pressure, and dynamic pressure are given here.

Operation at Constant Volume

Constant Volume is the most commonly used type of pressure DSC experiment. The cell is pressurized to an initial pressure and then the valves are closed and the experiment is started. Since the volume is constant as the cell temperature increases the pressure will also increase. The pressure increase will be a function of the gas temperature, which will not necessarily be the same as the cell temperature reported by the instrument. The amount that the pressure increases during an experiment will depend on the time/temperature profile of the experiment as well as the conditions of previous experiments since the pressure vessel temperature may be hotter in subsequent runs.

When running constant volume experiments remember that the actual pressure at a given point during the experiment will not be the same as the initial pressure. The pressure signal is available in the data file so that it can be measured at any point of interest during the experiment.

Do not start the experiment at an initial pressure that is high enough so that after heating during the experiment the pressure exceeds 7 MPa gauge (1000 psi). If the pressure exceeds 7.6 MPa gauge (1100 psig) then the experiment will terminate. A pressure relief valve, rated for 8.3 ± 0.3 MPa gauge (1200 ± 50 psig), will vent excess pressure if necessary. The pressure relief valve will reseat itself after venting and all components are pressure tested to 24.8 MPa gauge (3600 psig).

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 jauge (1000 psig) à température ambiante. NE dépassez PAS cette valeur.

Operation at Constant Pressure

Refer to Figure 9 and Figure 10 for the locations of the appropriate valves.

NOTE: In constant pressure operation, the cell is initially pressurized to a desired value. A slight bleed through the **Flow OUT** valve is then created and measured using an external flowmeter. This arrangement allows the increase in pressure, which occurs as the temperature is increased, to be bled off. This mode of operation depends on the user-supplied high pressure cylinder regulator to deliver the pressure set by the user while bleeding off the excess gas through the **Flow OUT** valve. Verify that the regulator is adequate for this task.

1. Close all three valves on the cell: the **FILL FLOW INCONTROL** valve, the **PURGE FLOW OUT CONTROL** valve, and the **PRESSURE RELEASE** valve. Do not overtighten the **FILL FLOW IN CONTROL** and **PURGE FLOW OUT CONTROL** valves.
2. Set the source gas regulator at the desired operating pressure.

3 Check the connections of the unrestricted flowmeter at the **Flow OUT** valve on the cell.

4 Turn the **FILL/PURGE** valve to **FILL**.

5 Slowly open the **FILL FLOW IN CONTROL** valve on the cell until it is fully open. Wait for the internal cell pressure, as indicated on the instrument display, to stabilize at the desired operating pressure.

6 Slowly open the **PURGE FLOW OUT CONTROL** valve.

7 Using the **PURGE FLOW OUT CONTROL** 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.

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.

Operation with Dynamic Pressure (Fixed Purge Rate)

Refer to Figure 9 and Figure 10 for the locations of the appropriate valves.

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 **FILL FLOW IN CONTROL** and **PURGE FLOW OUT CONTROL** valves. This arrangement maintains a constant exhaust flow rate. An unrestricted flow meter is required at the **Flow OUT** fitting for operation in this mode.

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 **FILL/PURGE** valve is set to **FILL**. An unrestricted flowmeter is required at the **Flow OUT** fitting for operation in this mode.

1 Close all three valves on the cell: the **FILL FLOW IN CONTROL** valve, the **PURGE FLOW OUT CONTROL** valve, and the **PRESSURE RELEASE** valve. Do not overtighten the **FILL FLOW IN CONTROL** and **PURGE FLOW OUT CONTROL** valves. Set the regulator at the source gas cylinder to an appropriate pressure.

2 Slowly open the **FILL FLOW IN CONTROL** valve.

3 Slowly open the **PURGE FLOW OUT CONTROL** valve. Wait for the flow measured at the flowmeter to stabilize. If finer flow adjustment is desired, a high pressure capable metering flow valve may be connected between the **Flow OUT** port and the flowmeter.

4 Adjust the **PURGE FLOW OUT CONTROL** valve until the flowmeter indicates the desired value (typically about 50 mL/min). If the flow rate is too low with the **PURGE FLOW OUT CONTROL** valve fully opened, check the position of the **FILL FLOW IN CONTROL** valve. Carefully open the **FILL FLOW IN CONTROL** valve further if necessary. If this does not raise the flow to the desired rate, the source gas pressure must be adjusted.

5 To readjust the source gas pressure, close both control valves, then repeat this procedure from step 1.

6 Wait until the pressure and flow rate are at the desired values. Turn the **FILL/PURGE** valve to **PURGE**.

NOTE: 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

Refer to Figure 9 and Figure 10 for the locations of the appropriate valves.

After a DSC 25P 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 détende 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

Refer to Figure 9 and Figure 10 for the locations of the appropriate valves.

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 sensor 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 while the cell is pressurized. 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 TRIOS 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 DSC 25P 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 T-zero cell liquid nitrogen cans, an optional accessory available from TA Instruments.

Refer to Figure 9 and Figure 10 for the locations of the appropriate valves.

1 Close the **FILL FLOW 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 lids.

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. Replace the inner silver lid.

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 outer 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 sensors.

NOTE: The lowest recommended temperature to achieve stable performance with the DSC 25P 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 40).

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. DSC 25P 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 cell 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 550°C, whichever is lower. Use a heating rate of 20°C/min. Ramp to 550°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 cell looks clean, 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).

Replacement Parts

The table below lists the replacement parts for the DSC 25P that are available from TA Instruments. See the list of offices when ordering parts.

Table 5: Fuses, Cords, and Cables*

Part Number	Description
201969.001	Fuse 6.3-amp time delay, 250 V (T 6.3A H 250V)
251470.010	Ethernet cable (10 foot, shielded)
253827.000	Power cord, 120 V North America
920223.902	Event cable

*Contact your local TA Instruments representative for information on non-US style power cords.

Table 6: Lids and Accessories

Part Number	Description
271580.001	Curved tweezers
970916.001	Lid, upper high pressure
973706.001	Lid, inner high pressure
972581.901	Cell Conditioning Kit
910824.001	Cell cleaning brush
970347.901	Quench Cooling Can

Table 7: Spare Parts

Part Number	Description
202813.039	O-ring pressure cylinder seal
973474.001	Manual tray
973592.001	Lid placement plug

Table 8: Tzero Sample Encapsulation Press

Part Number	Description
901600.901	Tzero sample press kit (includes the press, the .901, .903, and .904 die sets given below, and 1 box each of Tzero aluminum pans (PN 901683.901) and Tzero aluminum lids (PN 901671.901)
901608.901	Die set for standard series pans (green)
901608.902	Die set for standard aluminum hermetic pans (white)
901608.903	Die set for Tzero series aluminum pans (black)
901608.904	Die set for Tzero series aluminum hermetic pans and hermetic alodined pans (blue)
901608.905	Die set for Tzero series high volume pans (yellow)

Table 9: Tzero Series Sample Pans and Lids

Part Number	Description
901670.901	Tzero aluminum pans (pkg of 100)
901671.901	Tzero aluminum lids (pkg of 100)
901683.901	Tzero hermetic pans (pkg of 100)
901684.901	Tzero hermetic lids (pkg of 100)
901697.901	Tzero hermetic alodined pans (pkg of 100)
901698.901	Tzero hermetic alodined lids (pkg of 100)

Table 10: Standard Series Pans and Lids

Part Number	Description
900578.901	Platinum pans (pkg of 10)
900786.901	Aluminum pans (pkg of 200)
900779.901	Aluminum lids (pkg of 200)
900793.901	Aluminum pans, hermetic (pkg of 200)
900794.901	Aluminum pans, hermetic (pkg of 200)
900860.901	Aluminum hermetic lids with pinhole (pkg of 50)
900796.901	Coated aluminum pans, hermetic (pkg of 200)
900790.901	Coated aluminum lids, hermetic (pkg of 200)
900870.901	Aluminum pans for SFI sample (pkg of 200)
900867.901	Copper pans (pkg of 200)
900866.901	Gold pans (pkg of 10)
900868.901	Gold lids (pkg of 10)
900871.901	Gold pans, hermetic (pkg of 10)
900872.901	Gold lids, hermetic (pkg of 10)
900874.901	Graphite pans (pkg of 10)
900873.901	Graphite lids (pkg of 10)
900825.901	High volume pan kit (includes metal bell jar, lid, dies set, pans, lids, seals)
900824.901	High volume pan die set
900825.902	High volume pans, lids & seals (100 of each)
900808.901	High pressure pan kit (includes metal bell jar, lid, dies set, crimping tools, reusable SST capsules [5] & seals [20])
900814.901	High pressure capsule seals (pkg of 20)
900815.901	High pressure SST capsules (pkg of 5)

Table 11: Calibration/Reference Materials

Part Number	Description
915060.901	DSC / DTA temperature calibration kit
915061.901	Replacement certified indium reference material for above kit
900902.901	Indium calibration material
900910.901	Tin calibration material
900907.901	Zinc calibration material
970345.901	Tzero sapphire calibration kit
970370.901	MDSC® sapphire calibration kit
915079.901	Sapphire specific heat material
899096.901	Anisic acid (1 g)
899097.901	Biphenyl (1 g)
900319.901	DSC oxidative stability calibration kit