DISCOVERY DSC™
DIFFERENTIAL SCANNING CALORIMETER

Getting Started Guide for DSC2500, DSC250, DSC25

Revision A Issued April 2016

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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159 Lukens Drive
New Castle, DE 19720

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
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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.

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. 61010-2-010-04 Particular requirements for laboratory equipment for the heating of materials.

CAN/CSA-C22.2 No. 61010-2-081-04 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 Australia and New Zealand

AS/NZS CISPR11:2011 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radio frequency equipment.

For Canada

ICES-001 Issue 4 June 2006 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

For the European Economic Area

(In accordance with Council Directive 2004/30/EC of the European Parliament and of the Council of 26 February 2014 on the harmonization of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1:2013 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements. Emissions: Meets Class A requirements per CISPR 11. Immunity: Per Table 2 General electromagnetic environment, 3V/m EM environment.

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.

Safety

WARNING: The operator of this instrument is advised that if the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.

AVERTISSEMENT: L'utilisateur de cet instrument est prévenu qu'en cas d'utilisation contraire aux indications du manuel, la protection offerte par l'équipement peut être altérée.

Instrument Symbols

The following labels are displayed on the Discovery DSC™ system for your protection:

Symbol	Explanation
	This symbol indicates that you should read this Getting Started Guide for important safety information. This guide contains important warnings and cautions related to the installation, operation, and safety of the Discovery DSC system. 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. Ce symbole indique que vous devez lire entièrement ce guide de démarrage pour obtenir d'importantes informations relatives à sécurité. Ce guide contient d'importants avertissements et mises en garde relatifs à l'installation, à l'utilisation et à la sécurité du système Discovery DSC. 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 TA Instruments ou tout autre personnel d'entretien qualifié.
	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 those parts of the instrument. The *Discovery DSC Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

Electrical Safety

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

DANGER: Risk of electric shock. 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.

DANGER: Risque de choc électrique. 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: Protective earthing is provided through the mains power cord. Use of a grounded mains power outlet is required.

AVERTISSEMENT: Mise à la terre de protection est assurée par le cordon d'alimentation secteur. Utilisation d'une prise d'alimentation secteur terre est nécessaire.

Handling Liquid Nitrogen

Some of the cooling accessories use the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. Because of its low temperature (–196°C [–321°F)]), liquid nitrogen will burn the skin. When you work with liquid nitrogen, use the following precautions:

WARNING: Liquid nitrogen boils rapidly when exposed to room temperature. Be certain that areas where liquid nitrogen is used are well ventilated to prevent displacement of oxygen in the air.

AVERTISSEMENT: L'azote liquide bout rapidement lorsqu'il est exposé à la température ambiante. Assurez-vous que les zones où l'azote liquide est utilisé sont bien aérées pour éviter le déplacement de l'oxygène dans l'air.

1. Wear goggles or a face shield, gloves large enough to be removed easily, and a rubber apron. For extra protection, wear high-topped, sturdy shoes, and leave your pant legs outside the tops.
2. Transfer the liquid slowly to prevent thermal shock to the equipment. Use containers that have satisfactory low-temperature properties. Ensure that closed containers have vents to relieve pressure.
3. The purity of liquid nitrogen decreases when exposed to air. If the liquid in a container has been open to the atmosphere for a prolonged period, analyze the remaining liquid before using it for any purpose where high oxygen content could be dangerous.

Thermal Safety

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

DANGER: Les surfaces de cellule peuvent être assez chaudes pour vous brûler la peau pendant l'analyse d'un échantillon. Si vous effectuez un essai à basse température sur le DSC, le froid peut également provoquer des blessures. Après avoir effectué un type d'expérience quelconque, vous devez laisser la cellule DSC revenir à la température ambiante avant de toucher les surfaces internes de la cellule.

Chemical Safety

WARNING: Do not use hydrogen or any other explosive gas in the DSC.

AVERTISSEMENT: N'utilisez pas d'hydrogène ou tout autre gaz explosif dans le DSC.

WARNING: Oxygen can be used as a purge gas in the DSC. However, the cell must be kept clean so that volatile hydrocarbons, which might combust, are removed. Maximum pressure for oxygen shall be no greater than 34 kPa gauge (5 psig).

AVERTISSEMENT: L'oxygène peut être utilisé comme gaz de drainage dans le DSC. Toutefois la cellule doit rester propre pour que les hydrocarbures volatils, qui peuvent brûler, soient éliminés. Pression maximale d'oxygène ne doit pas être supérieure à 34 kPa (5 psig).

WARNING: If you are routinely evaluating materials in the DSC that lose a large amount of volatile hydrocarbons (e.g., lubricating oils), clean the DSC cell more frequently to prevent dangerous buildup of debris in the furnace.

AVERTISSEMENT: Si vous évaluez systématiquement les équipements du DSC qui perdent une grande quantité d'hydrocarbures volatils (p.ex., les huiles de graissage), nettoyez la cellule du DSC plus souvent pour éviter l'accumulation dangereuse des débris dans le four.

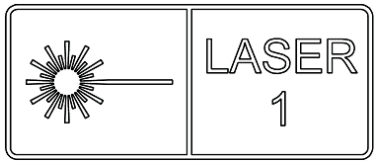
WARNING: If you are using samples that may emit harmful gases, vent the gases by placing the instrument near an exhaust.

AVERTISSEMENT: Si vous utilisez des échantillons qui émettent des gaz nocifs, ventilez les gaz en plaçant l'instrument près d'un échappement.

Laser Safety

This instrument is a Class 1 Laser product. Do not stare into the beam. Exempt laser product.

Class 1 Laser Products are accompanied by the following label:



This product complies with IEC 60825-1, with FDA performance standards for laser products.

21 CRF 1040.10 except for deviation pursuant to Laser Notice No.50.

Lifting the Instrument

The Discovery DSC™ is a fairly heavy instrument. In order to avoid injury, particularly to the back, please follow this advice:

WARNING: Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

AVERTISSEMENT: Demandez à deux personnes de soulever et/ou de porter l'instrument. L'instrument est trop lourd pour qu'une seule personne le manipule en toute sécurité.

Table of Contents

Chapter 1:

Introducing the Discovery DSC™ 2500, DSC250, DSC25

Overview

The Discovery Differential Scanning Calorimeter determines the temperature and heat flow associated with materials as a function of time and temperature. It also provides quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat production) processes of materials during physical transitions that are caused by phase changes, melting, oxidation, and other kinetic and thermodynamic processes. It can also be used to measure and quantify the heat capacity of a material. This information helps the scientist or engineer identify processing and end-use performance.

Figure 1 Discovery DSC2500 shown.

Your controller is a computer that performs the following functions:

- Provides an interface between you and the analysis instruments.
- Enables you to set up experiments and enter necessary information.
- Stores experimental data.
- Runs instrument control and data analysis program (TRIOS software).

NOTE: For technical reference information, theory of operation, and other information associated with the DSC not found in this manual, see the online help associated with the instrument control software.

DSC System Components

A functional Discovery DSC™ system has two major components: the Discovery DSC instrument, which includes the DSC cell used for monitoring differential heat flow and temperature and a gas delivery module; and a cooling accessory. The cooling accessory selected depends on the temperature range desired for your experiments.

Discovery DSC

The Discovery DSC instrument consists of a cell module, gas delivery module, and optional Autosampler. These components are described below.

Discovery DSC Cell

In a “heat flux” DSC, the sample material (encapsulated in a pan) and an empty reference pan are placed on a thermoelectric sensor surrounded by a furnace. The DSC cell module is shown below.

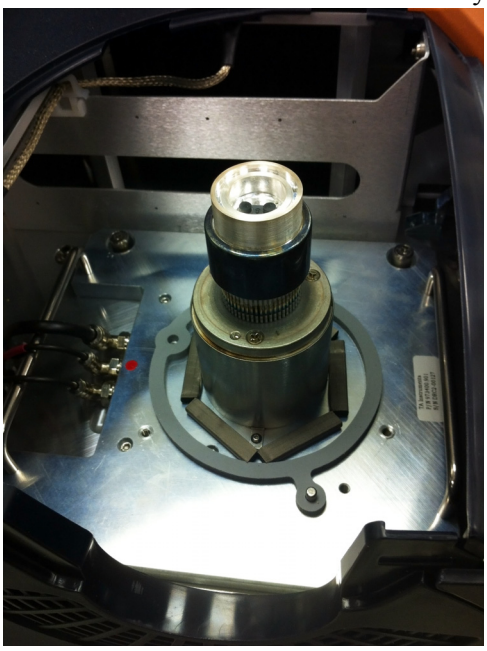


Figure 2 DSC cell module.

As the temperature of the furnace is changed (usually by heating at a linear rate), heat is transferred to and from the sample and reference through the thermoelectric sensor. The differential heat flow to the sample and reference is measured by a thermocouple device using the thermal equivalent of Ohm's Law.

$$q = \frac{\Delta T}{R}$$

where:

q = sample heat flow
ΔT = temperature difference between sample and reference
R= resistance of the sensor

This simple relationship, however, does not take into account extraneous heat flow within the sensor. The TA Instruments DSC is specifically designed to account for those latter heat flows.

The cell sensor consists of a constantan transducer with separate chromel disk welded to the surface under the sample platform. Chromel wires are welded to the Chromel disks to generate the delta-T signal. A third thermocouple measures the temperature at the base and one of those wires with the sample platform wire generates the delta-T zero signal. The T4 equation below shows the thermal network model which represents this cell arrangement, and the resultant heat flow expression that describes this cell arrangement (designated the T_0 [Tzero®] cell):

$$q = - \frac{\Delta T}{R_r} + \Delta T_0 \left(\frac{R_r - R_s}{R_r R_s} \right) + \left(C_r - C_s \right) \frac{dT_s}{dt} - C_r \frac{d\Delta T}{dt}$$

where:

ΔT = measured sample temperature (T_s) minus measured reference temperature (T_r)

ΔT_0 = measured base temperature of sensor minus measured sample temperature ($T_0 - T_s$)

T_0 = temperature for control

R_r = reference sensor thermal resistance

R_s = sample sensor thermal resistance

C_r = reference sensor heat capacity

C_s = sample sensor heat capacity

The first term in this expression is the equivalent of the conventional single-term DSC heat flow expression. The second and third terms account for differences between the sample and reference sensor resistances and capacitances respectively. These terms have their largest impact during regions of the thermal curve where the heat capacity of the sample is the predominant contributor to heat flow. The fourth term accounts for the difference in heating rate between the sample and reference. This term has its largest impact during enthalpic events (e.g., melting). This equation can be further adapted to account for pan heat flow effects T4P.

The DSC can deliver any of three separate heat flow signals, including:

- The conventional (single-term) heat flow (commonly designated as “T1 heat flow”).
- The expanded four-term heat flow (designated as “T4 heat flow”), which accounts for the cell resistances and capacitances and the heating rate difference between the sample and reference.
- A third heat flow (designated as “T4P heat flow”), which also accounts for pan effects.

Gas Delivery Module

The Discovery DSC™ gas delivery module controls the flow and delivery of purge gas to the Discovery DSC cell. It is based on a manifold design, and is capable of switching between two different purge gases at any point during or between experiments. It also measures the base purge flow, which prevents frosting when cooling accessories are used, and switches on/off the cooling gas flow.

DSC Autosampler

The Autosampler (shown below) is standard on the TA Instruments Discovery DSC2500, and optional for the Discovery DSC25 and DSC250.

Figure 3 Discovery DSC2500 with Autosampler.

The Autosampler is a four-axis robotic device that automatically loads sample and reference pans between the DSC measurement cell and the Autosampler tray. Once the Autosampler loads the pans and the cell covers are in place, the controller takes over and runs the pre-programmed thermal analysis experiments. After the experiment is complete and the cell returns to standby temperature, the instrument uncovers the cell and the Autosampler then unloads the pans. The unattended process can be pre-programmed to run up to 53 samples with one reference pan.

WARNING: Do not place your hands in the path of the Autosampler when it is in motion. Physical injury may occur.

AVERTISSEMENT: Ne placez pas vos doigts sur le chemin de l'échantillonneur automatique lorsqu'il est en mouvement. Des blessures physiques peuvent se produire.

Cooling Accessories

The Discovery DSC operates in conjunction with one of the cooling accessories listed below. Your choice of cooling accessories depends on the temperature range that you wish to use for your experiments. For information on installing the cooling accessories, see Chapter 2. For proper operation, the correct cooler type must be selected through the instrument control software; see the online help for details.

Finned Air Cooling System (FACS)

The Finned Air Cooling System (FACS) allows operation from ambient to 725°C, using flowing air as the coolant. For installation instructions, see Chapter 2. You can use the optional Quench Cool accessory with the FACS (see next section) if you want to return the temperature of the cell to ambient more quickly. This cooler is not intended for use at temperatures below ambient due to the potential for frost formation on the fins.

Figure 4 Finned Air Cooling System (FACS).

Quench Cooling Accessory (QCA) for FACS

The Quench Cooling Accessory (QCA) is placed inside the FACS and has a reservoir into which you can place coolant to return the cell to ambient more quickly. The rate of temperature drop depends on the coolant used (ice and water, dry ice, or liquid nitrogen).

NOTE: When using the FACS QCA, make sure that the cooling accessory identified in the instrument control program is the FACS and **not** the Quench Cooler. If the wrong cooler is identified, your results will be affected.

NOTE: Before you initiate a heating experiment, remove the Quench Cooler and put the manual silver lid back in place.

Stand-Alone Quench Cooler

The stand-alone Quench Cooler is designed for rapidly cooling the DSC cell to a lower temperature before initiating programmed heating experiments. The DSC Quench Cooler, along with a thermal enclosure, fits over the standard DSC cell and has a reservoir into which you can place coolant to cool the cell. The temperature range when using the Quench Cooler is –180 to 550°C. The actual range depends on the coolant (ice and water, dry ice, or liquid nitrogen).

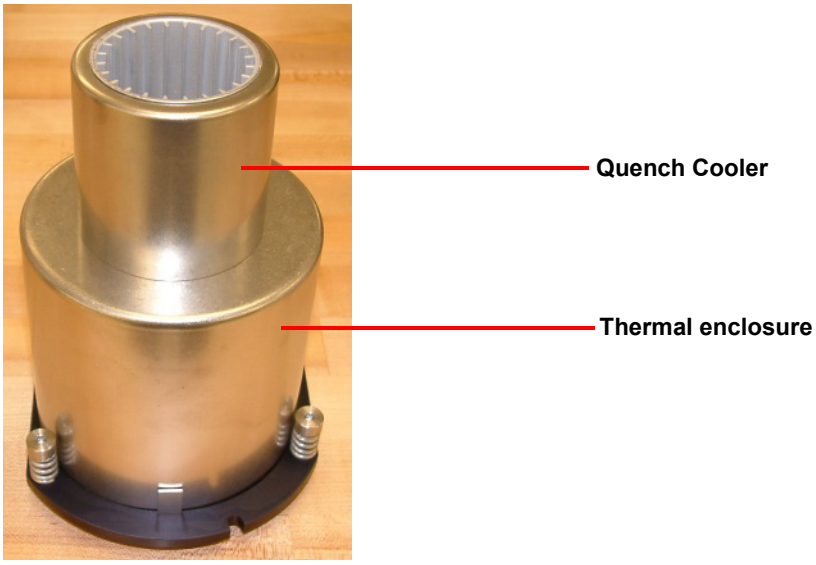


Figure 5 Stand-alone Quench Cooler with thermal enclosure.

NOTE: The Quench Cooler cannot be used to perform a Tzero calibration of the DSC. Therefore, only T1 heat flow is available when using the Quench Cooler.

NOTE: The Quench Cooler requires the manual silver lid provided in the Discovery DSC™ kit, and therefore cannot be used with the Autosampler. The DSC Autosampler is disabled during Quench Cooler operation.

Refrigerated Cooling System (RCS)

The Refrigerated Cooling Systems (RCS) are used to perform DSC cooling experiments. There are three different models offered by TA Instruments: the new RCS120, the RCS90, and the RCS40. All three models have a cooling head, which is made up of an internal heat exchanger, anti-condensate heaters, and various other components. The cooling head fits over the DSC cell.

- The RCS120 is a three stage, vapor compression refrigeration system with an attached cooling head. The operating range of the RCS120 is -120 to 400°C.
- The RCS90 consists of a two-stage, cascade, vapor compression refrigeration system with an attached cooling head. The RCS90 can be used for experiments requiring cooling within an operating range of –90 to 550°C. The maximum rate of cooling depends on the temperature range of your experiment.
- The RCS40 consists of a single-stage vapor compression refrigeration system with an attached cooling head. The RCS40 can be used for experiments requiring cooling within an operating range of –40°C to 400°C. The maximum rate of cooling depends on the temperature range of your experiment.

CAUTION: We recommend that you do not use the RCS40 when running long isothermal experiments above 325°C. Damage to the unit can occur if used at high temperatures for extended periods. Contact TA Instruments for more information.

MISE EN GARDE: Nous recommandons de ne pas utiliser le RCS40 lorsque vous effectuez de longues expériences isothermes au-dessus de 325°C. L'appareil peut subir des dégâts s'il est utilisé à des températures élevées sur de longues périodes. Contactez TA Instruments pour plus de renseignements.

The Discovery DSC™ User Interface

The Discovery DSC 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 6 Discovery DSC user interface display (DSC2500 shown).

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.
- The primary function buttons allow for easy access of common functions.
- View panel allows for real-time instrument status and associated actions.

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. **Start** automatically loads the selected sample pan and closes the furnace, if necessary, before beginning the experiment.
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 (air cool, all mechanical motion, etc.). If an Autosampler sequence is in progress, **Stop** will halt the sequence.
Lid Open/Close 	Toggles between opening and closing the lid on the DSC cell.
Reset 	Returns the Autosampler to the Home position and eliminates all records of pans.
Back 	Returns you to the previous page used
Home 	Returns you 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, Air Cool, Ship, and Standby Temperature.
Autosampler 	Displays a graphical representation of the Autosampler tray, indicating the status of the active sequence (e.g., which pan positions have been configured in the active sequence and which runs are active or pending). In addition, this panel provides access to other Autosampler operations such as calibration functions and utilities (manually loading pans).
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.

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 touch-screen.
Autosampler Stop 	Displayed while the Autosampler is in motion, pressing this button will stop the Autosampler, and can be used instead of the **Stop** button.

Instrument Specifications

The tables found below contain the technical specifications for the Discovery DSC™

Table 3: Discovery DSC Technical Specifications

Item/Area	Specifications
Dimensions	Depth 51 cm (20 in) Width 53 cm (21 in) Height 53 cm (21 in)
Weight (with Autosampler and FACS)	32 kg (70 lbs)
Mains Power	100–240 VAC, 50/60 Hz, 600 W Safety ground per local regulations 20 amp branch circuit maximum
Operating environmental conditions	Temperature: 15 to 35°C Relative humidity: 5 to 80% (non-condensing) at 31°C, decreasing linearly to 67% at 35°C. Installation Category II Pollution Degree 2 Maximum altitude: 2000 m The degree of protection for this instrument according to IEC 529 is IP20.
Temperature range with: FACS RCS120 RCS90* RCS40** Quench Cooling Accessory (without FACS) Quench Cooler for FACS	 Ambient to 725°C –120 to 400°C –90 to 550°C –40 to 400°C –180 to 550°C Ambient to 725°C
Sample pans	Various open or hermetically sealed (standard and Tzero series)
Purge gases	Recommended: air, argon, helium, nitrogen, or oxygen
Typical purge flow rate	50 mL/min cell purge 300 mL/min base purge (always flowing when Gas 1 is connected)
Cell volume	3.4 mL
Air Cool Gas***	~10 L/min at 20 psig

WARNING: Protective earthing is provided through the mains power cord. Use of a grounded mains power outlet is required.

AVERTISSEMENT: Mise à la terre de protection est assurée par le cordon d'alimentation secteur. Utilisation d'une prise d'alimentation secteur terre est nécessaire.

*Extended temperature isothermals above 400°C may cause the RCS to shut down; the software will detect the shut-down and stop the run. This is done by monitoring the flange temperature.

**While performance during heating is not affected, during cooling the cooler is able to maintain cooling rate linearity only below 325°C.

***Air cool can also be used when selected to operate when the cell cover is opened while operating with an active cooling accessory to prevent moisture from entering the cell/cooler. In this case, the air cool inlet will be connected to a dry nitrogen source.

CAUTION: When using the coated lid, the upper temperature of the instrument is limited to 400°C. Operation above this temperature can damage the coated lid. If temperatures higher than 400°C are required, make sure to install the standard silver lid prior to starting the experiment. Recalibration may be required when switching between the two lids.

MISE EN GARDE: Lors de l'utilisation du couvercle revêtu, la température supérieure de l'instrument est limitée à 400°C. Toute utilisation à une température supérieure à celle-ci peut endommager le couvercle revêtu. S'il est nécessaire de travailler à des températures supérieures à 400°C, assurez-vous d'installer un couvercle en argent standard avant de commencer l'expérience. Le réétalonnage peut s'avérer nécessaire en cas de changement de couvercle.

CAUTION: Holding the Discovery DSC™ cell at high temperatures for extended periods of time may cause sticking of the silver lid. If this occurs, please contact TA Instruments for assistance in removing the lid.

MISE EN GARDE: Le maintien de la cellule Discovery à de hautes températures pendant de longues périodes de temps peut engluer le couvercle en argent. Si cette situation se produit, contactez TA Instruments pour obtenir de l'aide pour le retrait du couvercle.

Options and Accessories

Several additional pieces of equipment are available from TA Instruments to be used with the Discovery DSC™. A brief description of each one is below. For more information refer to the online documentation.

Tzero® Sample Encapsulating Press

The TA Instruments Tzero sample encapsulating press is used to prepare encapsulated samples for DSC experiments. It comes with die sets for hermetic and non-hermetic sealing. The online documentation provides details about using this accessory.

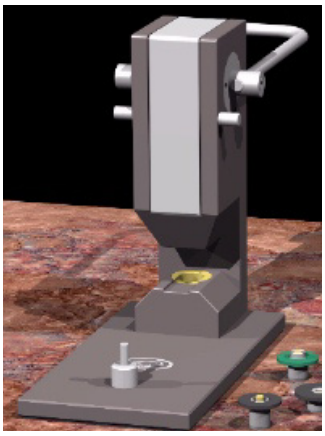


Figure 7 Tzero sample encapsulating press.

Chapter 2:

Installing the Discovery DSC™ System

Unpacking/Repacking the DSC

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 System

Before shipment, the DSC 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 gas lines to the Discovery DSC gas delivery module.
- Connecting the power cables.
- Installing a cooler option.

It is recommended that you have your DSC 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 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 experiments, it is important to choose a location for the instrument using the following guidelines. The Discovery DSC 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).
- Compressed lab air and 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.

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.

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

Table 4: Discovery DSC Back Panel

Port	Function
GAS 2	Gas inlet port controlled by the gas delivery module. Used, when needed, for the secondary sample purge gas.
GAS 1	Gas inlet port controlled by the gas delivery module. Used for the primary sample purge gas and base purge gas – use Nitrogen only.
COOLING GAS	Provides air for the Finned Air Cooling System (FACS), and nitrogen for the Refrigerated Cooling System (RCS) to prevent condensation when the lid is open.
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.

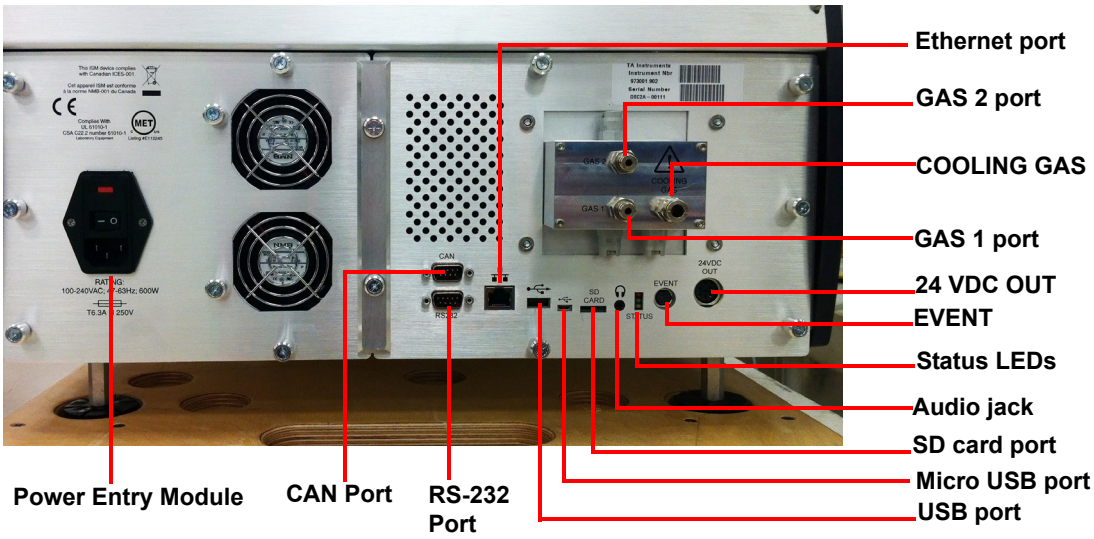


Figure 8 Discovery DSC rear panel connections.

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 Lines to the Gas Delivery Module

It is important to accurately control the atmosphere of the Discovery DSC cell during the experiment. The Discovery DSC is equipped with a gas delivery module (GDM) to control the flow rate of the gas used to purge the DSC cell. Two different gases may be connected to the instrument to facilitate gas switching. Follow these instructions to connect the purge lines.

CAUTION: Do not use any liquid in the purge lines.

MISE EN GARDE: N'utilisez aucun liquide dans les conduites de gaz de drainage.

Purge Gas Lines

1 Locate the **Gas 1** port on the back panel of the Discovery DSC. The **Gas 1** port is used to purge the sample area, and is also used as a base purge. As such, an inert gas such as nitrogen must be used in this port–not helium or air. A base purge is required when a sub-ambient cooling accessory is used.

2 Locate the **Gas 2** port. The **Gas 2** port is also used to purge the sample area and is used when a purge gas other than **Gas 1** is desired or when gas switching during an experiment is needed.

3 Connect the primary gas line to the **Gas 1** port.

4 If desired, connect a secondary gas to the **Gas 2** port.

WARNING: If connecting oxygen, it should ONLY be connected to the GAS 2 port. Maximum pressure for oxygen shall be no greater than 34 kPa gauge (5 psig).

AVERTISSEMENT: Tout raccordement de l'oxygène doit s'effectuer UNIQUEMENT sur l'orifice à GAZ 2. Pression maximale d'oxygène ne doit pas être supérieure à 34 kPa (5 psig).

NOTE: The flow rate is controlled through settings chosen using the instrument control software.

5 Make sure that the pressure of your purge gas source is regulated to 140 kPa gauge (20 psig), 34 kPa gauge (5 psig) for oxygen.

CAUTION: The Gas 1 and Gas 2 lines feeds into a pressure relief valves that are set to 170 kPa gauge (25 psig). The source pressure setting should not go above this value.

MISE EN GARDE: Les conduites de gaz 1 et 2 alimentent une soupape de détente de pression réglée à 170 kPa (pression manométrique) (25 psig). Le réglage de la pression à la source ne doit pas dépasser cette valeur.

6 Specify the connected gas on the **Discovery DSC** > **General** page of the TRIOS **Options**.

7 Set the flow rate to the recommended value of 50 mL/min for your experiments through the control panel options within TRIOS Instrument Control Software.

NOTE: If you are using a “house” laboratory supply rather than bottled gas, it is highly recommended that you install an external drier and a 5-µm filter.

CAUTION: Corrosive gases cannot be used with this instrument.

MISE EN GARDE: Des gaz corrosifs peuvent être utilisés avec cet instrument.

WARNING: Use of flammable or explosive gas as a purge gas is dangerous and is not recommended for this instrument.

AVERTISSEMENT: L'utilisation d'un gaz inflammable ou explosif comme gaz de drainage est dangereuse et n'est pas recommandée pour cet instrument.

Cooling Gas Line

The **Cooling Gas** port is used with the Finned Air Cooling System (FACS) and sub-ambient coolers. The FACS requires a source of compressed air, while sub-ambient coolers require a source of dry nitrogen in order to prevent condensation when the cell cover is open. Follow these instructions to connect the appropriate cooling gas line.

1 Locate the **Cooling Gas** fitting on the back panel of the Discovery DSC, marked with a 172 kPa gauge (25 psig) warning label.

2 Make sure your gas source is regulated to 138 kPa gauge (20 psig) when using the FACS. Excessive tubing length between the pressure regulator and the Cooling Gas inlet will result in reduced cooling capacity. For details on the pressure required for the RCS, see the appropriate Getting Started Guide.

CAUTION: The Cooling Gas line feeds into a pressure relief valve that is set to 170 kPa gauge (25 psig). The source pressure setting should not go above this value.

MISE EN GARDE: La conduite du gaz de refroidissement alimente une soupape de détente de pression réglée à 170 kPa pression manométrique (25 psig). Le réglage de la pression à la source ne doit pas dépasser cette valeur.

3 Connect the 1/4-inch O.D. tubing from the gas source (compressed air for the FACS or dry nitrogen for the sub-ambient coolers) to the **Cooling Gas** fitting.

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 9.

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

Figure 9 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.

Starting the Discovery DSC 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 system on and off.

To power on the system:

1 Check the connection between the DSC, 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 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 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 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 (**0**) position.

Installing the DSC Cooling Accessories

The DSC operates in conjunction with one of several cooling accessories. Your choice of cooling accessories depends on the temperature range required for your experiments. This section provides the information needed to install the FACS and Quench Coolers. For proper operation, the correct cooler type must be selected through the instrument control software. See the online help for details.

NOTE: For information on installing the RCS90 and/or RCS40, consult the appropriate manual or the online help.

Installing the Finned Air Cooling System (FACS)

Follow the instructions below to install the FACS onto the Discovery DSC.

WARNING: When installed, the cell ring restricts airflow to the FACS. As a result, the cell ring may reach an unsafe handling temperature, posing a burn risk to the Discovery DSC operator.

AVERTISSEMENT: Une fois installé, l'anneau de la cellule restreint l'écoulement de l'air vers le FACS. Par conséquent, l'anneau de la cellule peut atteindre une température dangereuse pour la manipulation, présentant ainsi un risque de brûlure pour l'opérateur de Discovery DSC.

1 Ensure that the cell is at a temperature where contact with the cell will not cause a burn.

2 If an Autosampler is present, it must be moved away from the cell by pressing Lid Open/Close on the touch screen or on the controller

WARNING: Do not place your hands in the path of the Autosampler when it is in motion. Physical injury may occur.

AVERTISSEMENT: Ne placez pas vos doigts sur le chemin de l'échantillonneur automatique lorsqu'il est en mouvement. Des blessures physiques peuvent se produire.

3 Carefully lift up and pull the top cover off of the Discovery DSC.

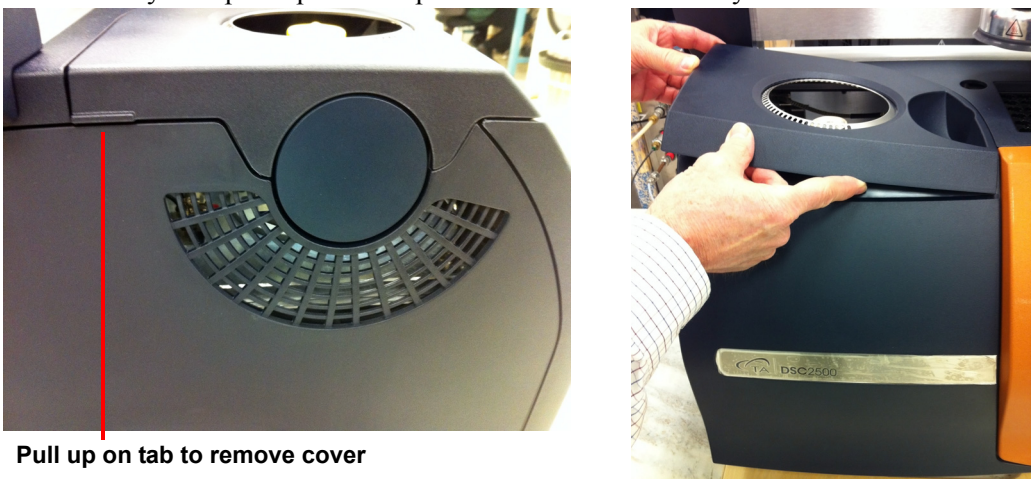


Figure 10 Remove cover.

4 Place the FACS over the cell, aligning the orientation hole in the FACS with the pin on the instrument cell plate.

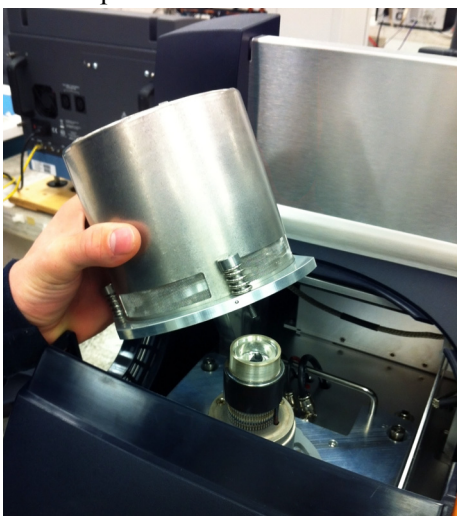


Figure 11 Placing the FACS over the cell.

5 Obtain a long 5/32-inch hexagonal (Allen) wrench from the accessory kit.

6 Insert the tip of the wrench into any one of the three captive screws in the FACS plate and begin to tighten (3 to 4 turns). DO NOT fully tighten yet.

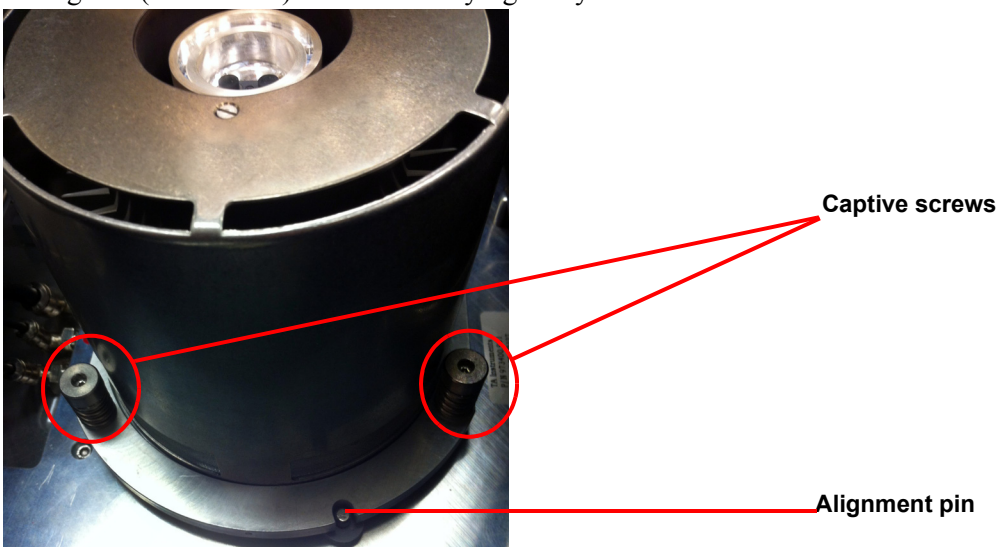


Figure 12 Securing the FACS to the cell (2 out of 3 screws shown).

7 Repeat step 6 for the two remaining captive screws. After you have started each screw, go back and tighten down all three screws (3 to 4 turns at a time) until you feel them touch the bottom. Do not over tighten.

8 Replace the top cover of the instrument and snap it into place.

Figure 13 FACS fully installed.

9 Select the correct cooler type on the **Discovery DSC Cooler** setting within the TRIOS software **Options** window.

10 You are now ready to use the FACS. This cooler requires a pressurized air source connected to the **Cooling Gas** fitting.

Installing the Refrigerated Cooling System (RCS)

1 Connect the heater jacket harness to the instrument.

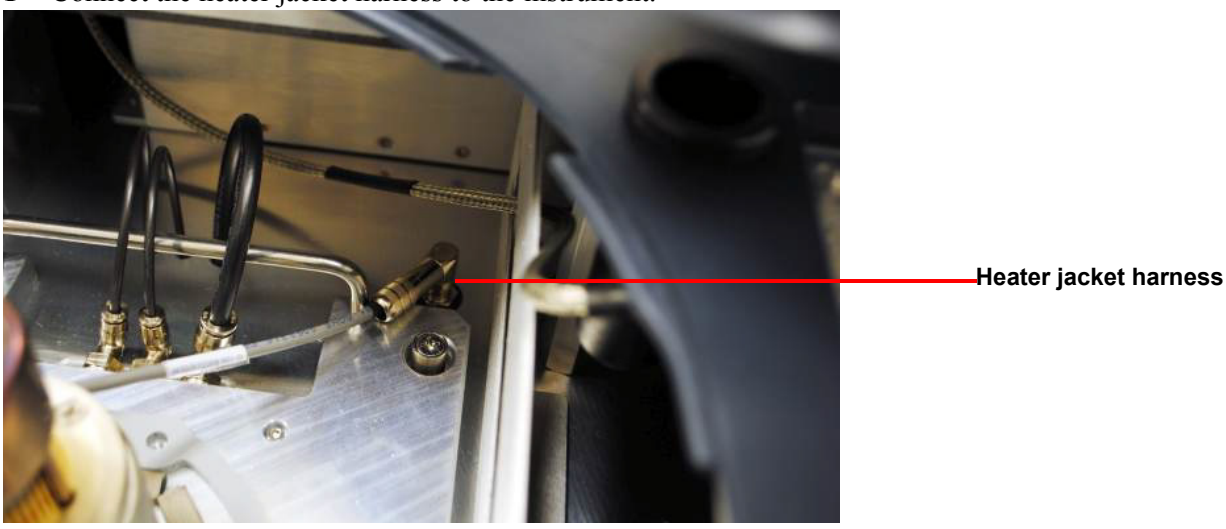


Figure 14 Connect heater jacket harness.

2 Open the lid.

3 Position the cooling head by aligning it with the pin in the instrument cell plate.

4 Secure the cooler by tightening the three captive screws. First engage all screws with three turns and then continue to tighten each screw three turns each in sequence until the cooling head is fully secure. Do not overtighten screws.

5 Connect the two conductor cables exiting the rear of the cooling head base to the heater jacket harness.

Figure 15

6 Unscrew the 2 screws holding the DSC cooler plug in place, and then remove the plug from the cover.

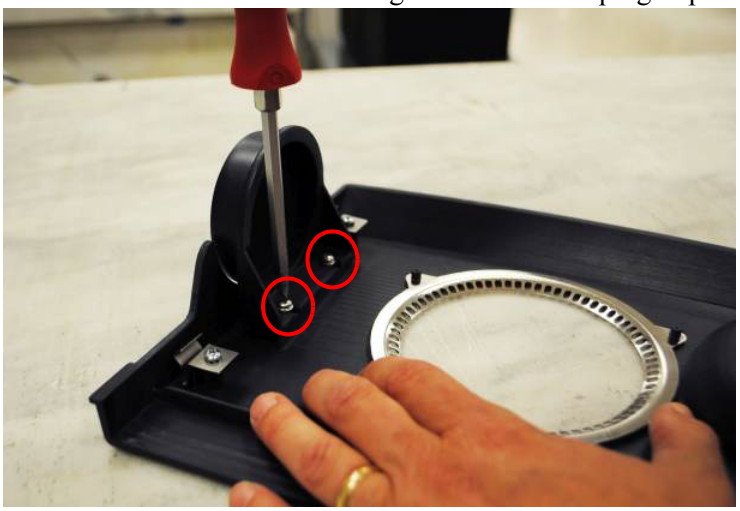


Figure 16 Remove cooler plug.

7 Snap the DSC instrument cover back into place by inserting the tabs on the right side of the cover and then snapping the left side into position.

Figure 17 Cover installed.

Installing the Quench Coolers

Installing a Quench Cooler is slightly different depending on whether you are using the “stand-alone” variety or the type to be used with the FACS. See the instructions below that apply to the desired type of Quench Cooler.

Quench Cool Accessory for FACS

The FACS Quench Cooler is used to expedite the process of returning the cell to ambient temperature at the end of a run. (This Quench Cooler is not intended to perform subambient experiments.) The following instructions are written on the assumption that the FACS is already installed. If you need to install the FACS, refer to “Installing the Finned Air Cooling System (FACS)” on page 32.

1 Open the DSC lid to raise the lid from the cell and cause it to move out of the way.

2 Slide the cooler into place into the top of the FACS.

NOTE: When using the FACS Quench Cooler, make sure that the cooling accessory identified in the instrument control program is the **FACS** and not the **Quench Cooler**. If the wrong cooler is identified, your results may not be as expected.

NOTE: If an Autosampler is in use, be sure to remove the Quench Cooler can before loading a sample and closing the cell cover.

Stand-Alone Quench Cooler

The stand-alone Quench Cooler requires the use of a thermal enclosure. Follow these instructions to install both the thermal enclosure and the Quench Cooler on your instrument. There should be no FACS installed on your instrument.

1 Ensure that the cell is at a temperature where contact with the cell will not cause a burn.

2 Press the **Lid** button on the DSC User Interface to open the lid on your instrument.

WARNING: Do not place your hands in the path of the Autosampler when it is in motion. Physical injury may occur.

AVERTISSEMENT: Ne placez pas vos doigts sur le chemin de l'échantillonneur automatique lorsqu'il est en mouvement. Des blessures physiques peuvent se produire.

3 Carefully lift up and pull the top cover away from the Discovery DSC.

4 Place the thermal enclosure over the cell and align the notch in the plate with the alignment pin.

5 Obtain a long 5/32-inch hexagonal (Allen) wrench from the accessory kit.

6 Insert the tip of the wrench into any one of the three captive screws in the thermal enclosure plate and begin to tighten (3 to 4 turns at a time). DO NOT fully tighten yet.

7 Repeat step 6 for the two remaining captive screws. After you have started each screw, go back and tighten down all three screws (3 to 4 turns at a time) until you feel them touch the bottom. Do not over tighten.

8 Slide the top cover back over the instrument.

9 Connect the an inert dry gas to the **GAS 1** port. The system is now ready to receive the Quench Cooler.

10 Select the correct cooler type on the **Discovery DSC Cooler** setting within the TRIOS software **Options** window. The AutoLid and Autosampler are both disabled when using the Quench Cooler.

11 Manually install the standard silver lid when running experiments. Then slide the Quench Cooler into the top of the thermal enclosure.

NOTE: The Quench Cooler may only be used to obtain **Heat Flow T1**.

Figure 18 Stand-alone Quench Cooler and thermal enclosure fully installed.

Installation of Additional Items

Several additional items can be found in the accessory box that is shipped with your instrument. While your TA Instruments Representative may install many of these for you, it is important to know how to install them yourself.

Installing the Autosampler Tray

When you receive your DSC, the sample tray is shipped in the accessory box, separate from the instrument. After unpacking the instrument and installing the instrument completely (see instructions in this chapter), you will be ready to run samples using the Autosampler.

WARNING: Do not place your hands in the path of the Autosampler when it is in motion. Physical injury may occur.

AVERTISSEMENT: Ne placez pas vos doigts sur le chemin de l'échantillonneur automatique lorsqu'il est en mouvement. Des blessures physiques peuvent se produire.

Installing the Lid (DSC2500 only)

The Discovery DSC2500 is shipped with two lids: a coated lid for operation up to 400°C, and a silver lid for operation up to 725°C. After unpacking the instrument and installing the instrument completely (see instructions in this chapter), you are ready to install a lid and run samples. Note that if the silver lid is used for high-temperature experiments, the cell should be redressed using the conditioning tool (included in the Conditioning Tool Kit) before reverting to the coated lid for experiments.

Refer to TRIOS software Online Help for full details on selecting and switching DSC lids.

DANGER: The cell surfaces can be hot enough to burn the skin during a sample run. If you are conducting a subambient test on the DSC, cold could also cause injury. After running any type of experiment, you must allow the DSC lid to return to the standby temperature before installing or replacing the lid.

DANGER: Les surfaces de cellule peuvent être assez chaudes pour vous brûler la peau pendant l'analyse d'un échantillon. Si vous effectuez un essai à basse température sur le DSC, le froid peut également provoquer des blessures. Après avoir effectué un type d'expérience quelconque, vous devez laisser la DSC lid revenir à la température ambiante avant d'installer ou de remplacer le couvercle.

1 Ensure that the instrument is not running a method and that the lid is at a safe temperature to handle.

2 Obtain the lid you wish to install and locate the notch on the top side of the lid. See the figure below.

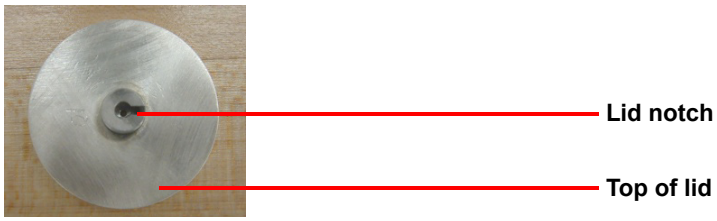


Figure 19 Top side of lid and lid notch.

3 Gently grasp the sides of the AutoLid cover and slowly lift it to expose more of the lid pin.

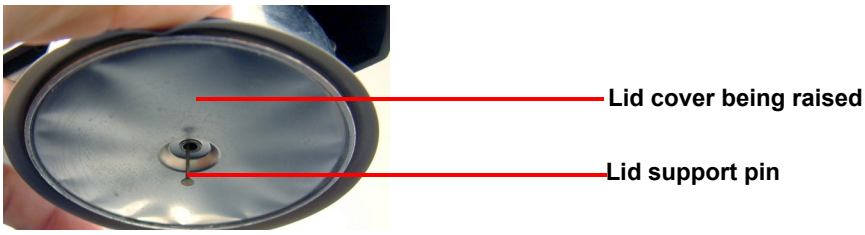


Figure 20 Lifting the insulated cover to expose the lid support pin.

4 While still holding the insulated cover, install the lid by aligning the notch on the top side of the lid with the lid support pin.

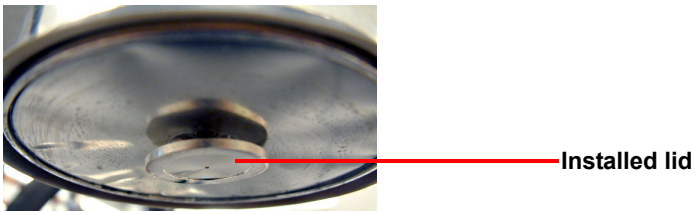


Figure 21 Lid properly installed on the lid support pin.

5 Specify the installed lid type through the **Discovery DSC** > **General** page of the TRIOS **Options**.

Chapter 3:

Use, Maintenance, & Diagnostics

Using the Discovery DSC™

All of your DSC experiments will have the following general outline. In some cases, not all of these steps will be performed. The majority of these steps are performed using the instrument control software. The instructions needed to perform these actions can be found in the online help in the instrument control software; therefore, they will not all be covered in detail here.

- Calibrating the instrument
- Selecting the pan type and material
- Preparing the sample
- Creating or choosing the test procedure and entering sample and instrument information through the instrument control software
- Setting the purge gas flow rate
- Loading the sample and closing the cell lid
- Starting the experiment

To obtain accurate results, follow these procedures carefully.

Before You Begin

Before you set up an experiment, ensure that the Discovery DSC system and the controller have been installed properly. Make sure you have:

- Connected the instrument and the controller to an Ethernet router/switch
- Connected all gas lines
- Connected any desired accessories
- Powered up the unit
- Become familiar with controller operations
- Calibrated the Discovery DSC, if necessary

Calibrating the Discovery DSC™

To obtain accurate experimental results, you should calibrate the Discovery DSC cell when you first install it. For the best results, you should recalibrate periodically. The calibration procedures that you need to perform will vary depending on the heat flow selection. These experiments will be performed using the instrument control software. For details on how to perform each calibration, refer to the online help accessed through TRIOS software.

NOTE: Please make sure that you run your experiments with the same gas that you used to calibrate the system. For example, if you calibrate using nitrogen, make your runs with nitrogen.

Perform calibration runs that encompass the temperature range you plan to use in your experiments. We recommend calibration over the widest temperature range; if your subsequent experiments cover a wider range, you may wish to recalibrate.

For optimum experimental results, you will need to perform all DSC calibrations whenever you change one of the following parameters:

- First use of a new cell
- Installation of new lid
- Purge gas change
- Cooling device or accessory change

Calibration consists of several different types of procedures that are described briefly below. For more details on performing each type of calibration, refer to the instructions in online help.

T1 Baseline Calibration (Discovery DSC2500, DSC250, DSC25)

The baseline slope and offset calibration involves heating an empty cell through the entire temperature range expected in subsequent experiments. The calibration program is used to calculate the slope and offset values needed to flatten the baseline and zero the heat flow signal. Baseline Slope is a calibration constant that accounts for the baseline slope of an empty cell. The baseline slope is calculated from data gathered as an empty cell is heated through the temperature range expected in subsequent experiments. Default is 0.00. Baseline Offset is a calibration factor that shifts the curve up or down along the Y-axis to balance the heat flow at the sample and reference thermocouple positions. The baseline offset is calculated from data gathered as an empty cell is heated through the temperature range expected in subsequent experiments.

Tzero® Calibration

The DSC Tzero calibration requires two experiments. The first experiment is done without samples or pans (baseline); the second is performed with large (approximately 100 mg) sapphire disks (without pans) on both the sample and reference positions. Both experiments use the same method, beginning with an equilibration at an initial temperature, holding isothermal for ten minutes, heating at constant rate to a final temperature, and holding isothermal for ten minutes. The range of temperatures should be at least as broad as the desired experimental range. Tzero calibration should be done at relatively high heating rates (such as 20°C/min) in order to obtain the most accurate calibration of the sensor thermal capacitance and resistance values. Rates of less than 10°C/min are not recommended for Tzero calibration.

NOTE: Tzero calibration is not available for the Discovery DSC25.

Enthalpy (Cell) Constant Calibration

This calibration is based on a run in which a reference material (e.g., indium) is heated through its melting transition. The measured 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 temperature. 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 an experiment in which a reference material (e.g., indium) is heated through its melting transition. The extrapolated onset of the recorded melting point of this reference material is compared to the known melting point, and the difference is recorded 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 three or more standards, the temperature correction is modelled by a cubic spline fit. The multiple-point temperature calibration is more accurate than the one-point calibration if absolute temperature measurements are required over a broad (>300°C) temperature range.

If multipoint temperature calibration is used, it is recommended that you choose standards that will bracket the desired measurement temperature range. This will ensure optimal calibration and performance.

Heat Capacity Calibration (Discovery DSC2500 only)

Heat capacity can be obtained in a single experiment with the Discovery DSC™. Heat capacity calibration is required to optimize the accuracy of the sample's heat capacity. The calibration uses a sapphire standard material with a known heat capacity at a specific temperature of interest. Calibration is typically done at a heating or cooling rate of 10 to 20°C/min.

The heat capacity calibration curve is calculated by dividing the theoretical value of heat capacity by the measured value at a specific temperature. The resultant calibration data curve, as a function of temperature, is sent to the instrument and applied to subsequent measurements.

Heat Capacity (MDSC®) Calibration

MDSC heat capacity calibration is only required to optimize the accuracy of the sample's heat capacity or when using periods less than or equal to 40 seconds. The calibration uses a sapphire standard material with a known heat capacity at a specific temperature of interest. Except for heating rate, the calibration experiment should be run under similar conditions (pan type, modulation amplitude, and period) to those that will be used for subsequent samples. The heating rate can be set to a nominal value of 5°C/min or the calibration experiment can be performed isothermally if you are calibrating just the reversing heat capacity.

The Reversing Heat Capacity calibration curve is calculated by dividing the theoretical value of heat capacity, as a function of temperature, by the measured value. The resultant calibration data, as a function of temperature, is sent to the instrument over the entire temperature range of the calibration experiment. See online help for more detailed information.

Autosampler Calibration

The DSC Autosampler may require recalibration to refine pan placement and pickup capabilities. Autosampler calibration involves calibrating the closed cell position, sample loading position, and sample tray position. These procedures have been automated for ease-of-use operation. You can choose to calibrate all positions by using the calibration functions from the user interface or from the instrument control program.

Once the Autosampler is correctly calibrated, recalibration is generally required only when you replace the DSC cell. Periodic checks are the best way of determining when the Autosampler needs recalibration.

Autosampler calibration is accessed through the instrument user interface functions. Consult online help for detailed instructions.

Running a Discovery DSC™ Experiment

Experimental Procedure

All of your DSC experiments will have the following general outline. In some cases, not all of these steps will be performed. See the instrument control software online help for anything not covered in this manual.

1 Attach and set up external accessories as required (e.g., purge gas, cooling accessory).

NOTE: Please make sure that you run your experiments with the same gas used during calibration of the system.

2 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 online help.

3 Load the sample pan (and a similarly prepared empty reference pan) into the cell or onto the Autosampler tray.

4 Enter experiment and procedure information through the TA Instruments TRIOS Software; this includes both sample and instrument information.

5 Start the experiment.

Loading the Sample

Once the sample pan has been prepared and the sample information has been recorded, you are ready to load the sample pan into the DSC cell. The pans will automatically be loaded into the cell by the Autosampler when you run an Autosampler sequence.

WARNING: If the cell has just been used, the components of the cell could be very hot. As a safe-operating practice, use the tweezers whenever handling the cell or pans within the cell.

AVERTISSEMENT: Si la cellule vient juste d'être utilisée, ses composants peuvent être très chauds. Par mesure de sécurité, utilisez des brucelles lorsque vous manipulez la cellule ou les bassins contenus dans la cellule.

Starting an Experiment

Before you start the experiment, ensure that the DSC is connected with the controller and you have entered all necessary information through the instrument control software.

Start the experiment by selecting **Start** on the instrument control software or touching the **START** button on the instrument user interface. When you start the instrument, the system automatically runs the experiment to completion.

Stopping an Experiment

If for some reason you need to discontinue the experiment, you can stop it at any point by selecting **Stop** through the instrument control software or touching the **STOP** button on the instrument user interface.

Maintaining the Instrument

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 help installed with the instrument control software for further information.

DANGER: Because of the high voltages in this instrument, untrained personnel must not attempt to test or repair any electrical circuits.

DANGER: 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 TA Instruments, check with TA Instruments that the proposed method will not damage the instrument.

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

Cleaning the User Interface Screen

You can clean the DSC user interface screen as often as you like. The instrument should first be turned off in order to prevent activating the touch screen switches, which can result in unexpected instrument behavior. The user interface screen should be cleaned with a household liquid glass cleaner and soft cloth. Wet the cloth, not the user interface screen with the glass cleaner, and then wipe off the screen and surrounding surfaces.

WARNING: Do not use harsh chemicals, abrasive cleansers, steel wool, or any rough materials to clean the user interface screen, as you may scratch the surface and degrade its properties.

AVERTISSEMENT: N'utilisez pas de produits chimiques agressifs, de nettoyants abrasifs, de la laine d'acier ou tout autre matériau rugueux pour nettoyer l'écran de l'interface utilisateur, car vous pourriez égratigner sa surface et dégrader ses propriétés.

Replacing Fuses

WARNING: Always unplug the instrument before you examine or replace the fuses.

AVERTISSEMENT: Débranchez toujours l'instrument avant d'examiner ou de remplacer les fusibles.

The Discovery DSC™ contains internal fuses that are not user serviceable. If any of the internal fuses blows, a hazard may exist. Call your TA Instruments service representative.

The only customer-replaceable fuses are the fuses located in the fuse holder located on the back panel of the instrument. To check or change these fuses:

1 Turn the instrument off and remove the power cord.

2 The instrument power entry module has two standard fuse holders built in. Use a flat-blade screwdriver to remove the fuse carriers.

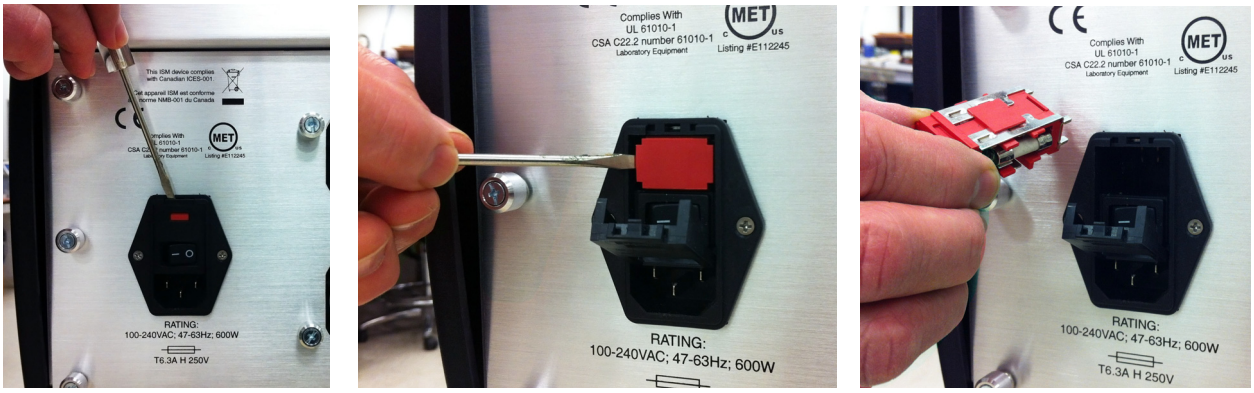


Figure 22 Use a screw driver to open the fuse cover and remove the fuse holder.

3 Remove old fuses and replace the fuses only with the type and rating indicated on the instrument's rear panel.

4 Place the fuse carrier back into the fuse holder and snap the cover in place.

NOTE: For information on replacing the RCS fuses or for maintenance needed for the various coolers, refer to the appropriate manual or the online help associated with the DSC instrument control software.

Replacement Parts

Replacement parts for the Discovery DSC™ are listed below. Refer to the tables below 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
920223.901	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
972628.001	Lid (coated)
972628.002	Lid (standard Silver)
973252.001	Lid (manual)
972581.901	Cell Conditioning Kit
910824.001	Cell cleaning brush
972633.001	Lid support pin

Table 7: Manual Instrument Accessories (Non-Autosampler)

Part Number	Description
973474.001	Sample tray (manual)
973061.903	Cover (manual)
973252.001	Lid (manual, Silver)
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

Table 12: Autosampler

Part Number	Description
973475.001	Autosampler tray