

DSC

Differential Scanning Calorimeter



Q SeriesTM
Getting Started Guide

Revision N
Issued January 2007



Notices

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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Important: TA Instruments Manual Supplement

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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions.

A NOTE highlights important information about equipment or procedures.



A **CAUTION** emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.



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

Regulatory Compliance

Safety Standards

For Canada:

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

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

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

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

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

For the United States:

UL61010A-1 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

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

Electromagnetic Compatibility Standards

For Australia and New Zealand:

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

For Canada:

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

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

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

For the United States:

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

Safety



CAUTION: 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.

Instrument Symbols

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

Symbol	Explanation
	Indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface.
	<p>This symbol on the rear access panel indicates that you must unplug the instrument <i>before</i> doing any maintenance or repair work; voltages exceeding 120/240 Vac are present in this system.</p> <p>If you are not trained in electrical procedures, do not remove the cabinet covers unless specifically instructed to do so in the manual. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.</p>

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *DSC Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

Electrical Safety



CAUTION: The hold-down thumbscrews that hold the DSC Q2000/Q1000 cell in place ensures proper lid alignment and electrical connections. You cannot run experiments without them. If they are not fully in place, the instrument may not be able to apply power to the cell and will not operate.

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.



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

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

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

Handling Liquid Nitrogen

Some of the cooling accessories use the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. Because of its low temperature [-195°C (-319°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.

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.



WARNING: Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent the Liquid Nitrogen Cooling System (LNCS) container in confined spaces.

Do not enter confined spaces where high concentrations of nitrogen gas may be present unless the area is well ventilated.

The warning above applies to the use of liquid nitrogen. Oxygen depletion sensors are sometimes utilized where liquid nitrogen is in use.

Thermal Safety

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.

Lifting the Instrument

The 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.

Chapter 1

Introducing the DSC

Overview

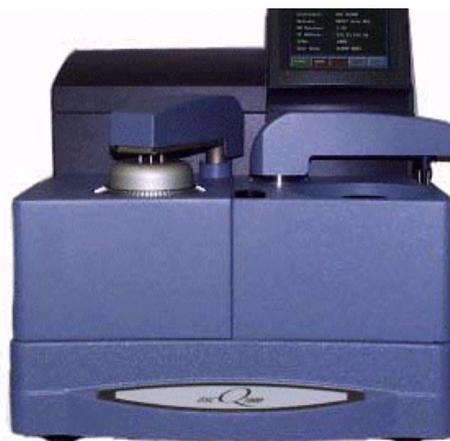
The Differential Scanning Calorimeter (DSC) determines the temperature and heat flow associated with material transitions as a function of time and temperature. It also provides quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes of materials during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

The DSC instrument works in conjunction with a controller and associated software to make up a thermal analysis system.

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 constants
- Stores experimental data
- Runs data analysis programs.

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



DSC with Autosampler and Finned Air Cooling System

DSC System Components

A functional DSC system has three major components: the instrument itself, which contains the system electronics; the cell, which monitors differential heat flow and temperature; and a cooling accessory. The cooling accessory selected depends on the temperature range desired for your experiments.

DSC Cell

In a "heat flux" DSC, the sample material, encapsulated in a pan, and an empty reference pan sit on a thermoelectric disk surrounded by a furnace. As the temperature of the furnace is changed (usually by heating at a linear rate), heat is transferred to the sample and reference through the thermoelectric disk. The differential heat flow to the sample and reference is measured by area thermocouples 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 thermoelectric disk



This simple relationship, however, does not take into account extraneous heat flow within the sensor or between the sensor and sample pan. The TA Instruments Q Series DSC's are specifically designed to account for those latter heat flows.

The cell sensor consists of a constantan body with separate raised platforms to hold the sample and reference. The platforms are connected to the heating block (base) by thin-walled tubes that create thermal resistances between the platforms and the base. Area detectors (thermocouples) on the underside of each platform measure the temperature of the sample and reference. A third thermocouple measures the temperature at the base. The 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) is

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

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 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 modified to account for pan heat flow effects.

TA Instruments supplies the following Q Series DSC's.

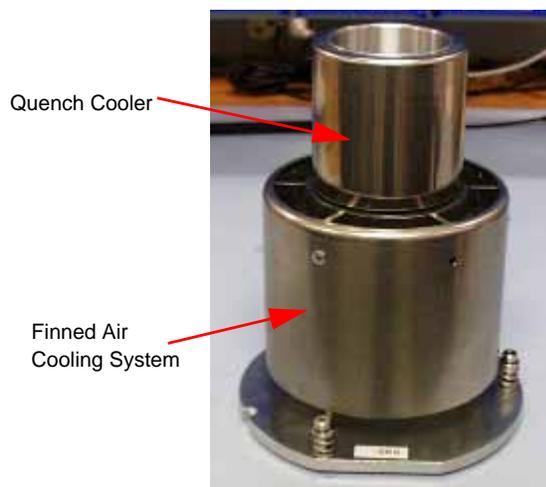
- The Q20, Q10, and Auto Q20 instruments provide conventional (single-term) heat flow (commonly designated as "T1 heat flow") only.
- The Q200 and Q100 provide not only the single-term (T1) heat flow, but also the expanded four-term heat flow (designated as "T4 heat flow"), which accounts for the cell resistances and capacitances.
- The Q2000 and Q1000 provide T1 and T4 heat flows, as well as a third heat flow (designated as "T4P heat flow"), which accounts for pan effects.

Cooling Accessories

The 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. To install the cooling accessories, see Chapter 2. **For proper operation, the correct cooler type must be selected through the instrument control software, see the online documentation 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 in the cell.



Quench Cool
Accessory for FACS

Quench Cool Accessory for FACS

This accessory 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 quench cool accessory 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 inaccurate.

Refrigerated Cooling System (RCS)

The Refrigerated Cooling Systems (RCS) are used to perform DSC cooling experiments. There are two different models offered by TA Instruments—the RCS 90 and the RCS 40. Both 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.



CAUTION: The RCS coolers are not compatible with the accessory outlets on the back of the instrument due to power requirements.

- The RCS 90 (shown in the figure to the right) consists of a two-stage, cascade, vapor compression refrigeration system with an attached cooling head. See the figure to the right. The RCS 90 can be used for experiments requiring cooling within an operating range of -90°C to 550°C . The maximum rate of cooling depends on the temperature range of your experiment.
- The RCS 40 (shown in the figure below) consists of a single-stage vapor compression refrigeration system with an attached cooling head. See the figure below. The RCS 40 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.



RCS 90 with DSC Q1000



CAUTION: We recommend that you do not use the RCS when running isothermal experiments above 400°C . Damage to the unit can occur if used at high temperatures for extended periods.

Quench Cooler



Stand-Alone
Quench
Cooler

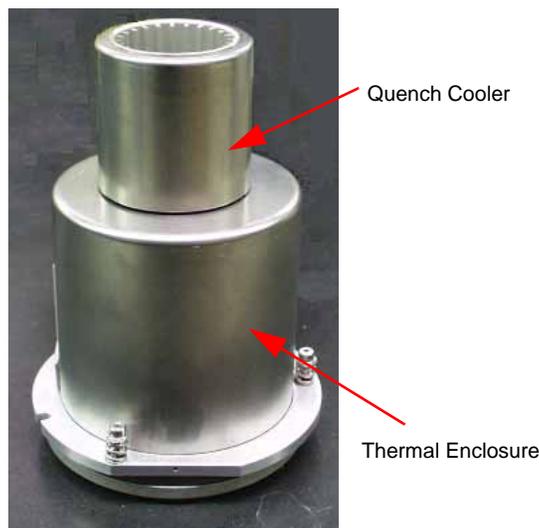
The Quench Cooler is designed for rapidly cooling the DSC cell to a lower temperature before initiating programmed heating experiments. The DSC Quench Cooler fits over the standard DSC cell and has a reservoir into which you can place coolant to cool the cell. The temperature when using the Quench Cooler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

Before you initiate a heating experiment, you will need to remove the Quench Cooler and put the enclosure lid back in place.

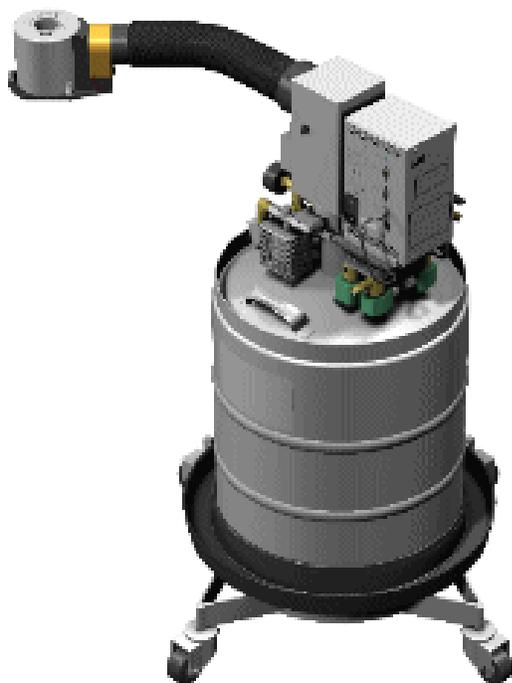
If the Quench Cooler is used for temperatures below -100°C , then helium must be used as the purge gas.

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 manual lids and therefore cannot be used with the Autosampler. The AutoLid is disabled ("parked") during Quench Cooler operation.



Q10 Stand-Alone
Quench Cooler



Liquid Nitrogen Cooling System (LNCS)

The LNCS (Liquid Nitrogen Cooling System) is a universal cooling accessory for use with TA Instruments Analyzers. It can be used with all DSC Q Series models.

The Liquid Nitrogen Cooling System (LNCS) allows automatic and continuous temperature control within the range of -180°C to 550°C . The LNCS tank is pressurized to deliver the liquid nitrogen to the heat exchanger, which in turn cools the cell.

The LNCS (shown here) can be autofilled in your laboratory using the DSC touch screen or through the instrument control software. This requires a low pressure 170 kPa gauge maximum (25 psig) bulk storage tank to be located within 1.8 m (6 feet) of the LNCS. See the *LNCS Getting Started Guide* for further information.

The DSC Q2000/Q200 Touch Screen

The DSC Q2000 and Q200 instruments have a built-in integrated display and keypad in the form of a touch screen for local operator control. The functions on the screen change depending upon the menu you are using. This section briefly describes the basic layout of these functions.

The *status line* along the top of the display shows the current instrument status, run selection, and temperature.

At the bottom of the screen is a set of keys that are used for the primary instrument functions. See the table below for a description of each key.

The functions in the middle of the touch screen will vary depending on the screen displayed.



Q2000/Q200 Display

Primary Function Keys

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

Key Name	Description
Start	Begins the experiment. This is the same function as Start on the instrument control software. Start automatically loads the sample pan and closes the furnace, if necessary, before beginning the experiment.
Stop	<p>If an experiment is running, this key 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.</p> <p>If an experiment is not running (the instrument is in a standby or method-end state), the Stop key will halt any activity (air cool, all mechanical motion, etc.).</p> <p>If an Autosampler sequence is in progress, Stop will halt the sequence.</p>
Control	Displays a list of the control command functions. These are used to control the instrument actions such as furnace movement, sample loading/unloading, taring, etc. Items can be selected from the icons or from the drop-down menu. Select Apply to initiate the command. See the next page for more details on this screen.

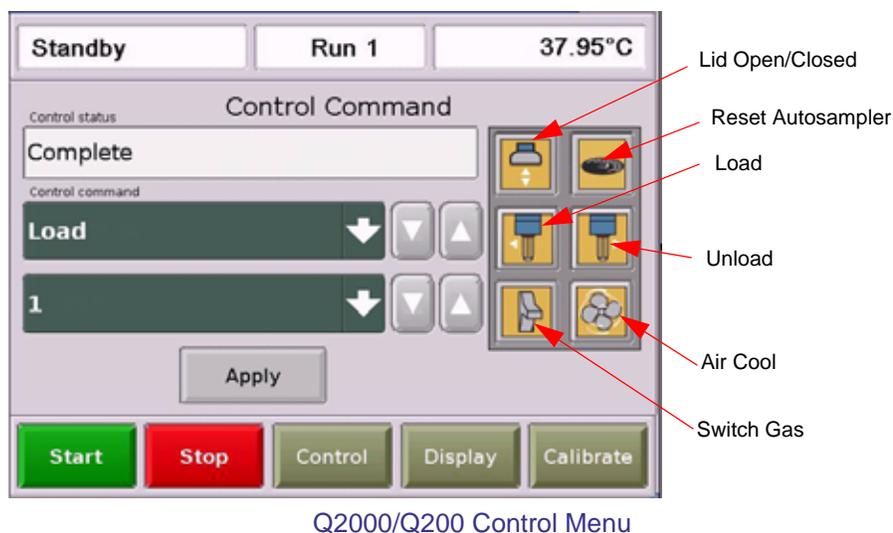
(table continued)

Display	Accesses the display screen, which displays the signals from the instrument such as signal display, real-time plot, instrument information, etc.
Calibrate	Displays the calibration functions available for this instrument. Functions such as Autosampler and touch screen calibration can be accessed using this key.

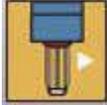
Control Menu

The Control Menu (see the figure to the right) is accessed by touching the **Control** key at the bottom of the touch screen. A brief description of each control command is provided in the table below.

NOTE: Most of the commands shown are not available during an active experiment.



Select the desired function either from the drop-down list of Control Commands or by pressing the icon. Then press Apply to initiate the action.

Control Command	Description
LID OPEN/CLOSED 	Toggles between opening and closing the AutoLid on the cell.
RESET AUTOSAMPLER 	Clears all commands, instructs the unit to locate all home positions, and returns the Autosampler to the park position. Also instructs the Autosampler to "forget" the current lid and sample pan locations.
UNLOAD 	Unloads the pan from the cell to the Autosampler tray or waste bin.
LOAD  <i>(table continued)</i>	Loads the selected pan from the Autosampler tray to the cell.

Control Command	Description
UNLOAD REFERENCE	Unloads the reference pan from the cell to the Autosampler tray or waste bin.
SWITCH GAS	Toggles between purge Gas #1 and Gas #2.
AIR COOL 	Toggles the air cool function on or off. This is the same function as Air Cool on the instrument control software.
PARK AUTOSAMPLER	Sends the autosampler tray to the park position, which is set off to the right and below the home position.
GO TO STANDBY TEMPERATURE	When using an RCS or LNCS, this option will cool to the standby temperature selected in the instrument control program.
LNCS	Automatically fills the LNCS with liquid nitrogen from the holding tank when an experiment is not in progress.
RCS	Toggles the RCS (Refrigerated Cooling System) between on and off, depending on the state of the RCS when you press the key.
RESET SAVED PARAMETERS	Resets the saved instrument parameters and resets the instrument.
SHUTDOWN 	Shuts down and resets the instrument.

Display Touch Screen Options

The Display Options are accessed by touching the Display key at the bottom of the touch screen. The keys shown in the figure to the right are displayed.

A brief description of the function of each key is provided in the table below.



Q2000/Q200 Display Screen

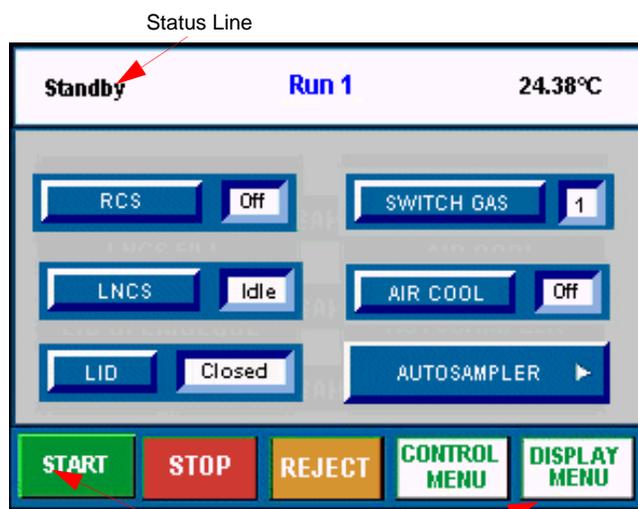
Key Name		Description
SEGMENTS		Accesses the experimental method that is currently being used for this experiment.
INFORMATION		Displays instrument information such as the software version, options, and the IP address.
STATUS		Displays the three main signals indicating the current status of the experiment.
SIGNALS		Displays the real-time signal data that comes directly from the instrument. The signals displayed here are customized through the instrument control software by accessing Tools/Instrument Preferences.
PLOT		Displays a time-based plot of data as it is received from the instrument during experiments.
SCREEN SAVER		Allows you to choose a screen saver for the touch screen.
HOME		Returns to the opening window.

The DSC Q1000/Q100 Touch Screens

The DSC Q1000 and Q100 instruments have a built-in integrated display and keypad in the form of a touch screen for local operator control. The functions shown on the screen change depending upon the menu you are using. This section briefly describes the functions of the keys shown on the touch screen displays.

The *status line* along the top of the display (see the figure to the right) shows the current instrument status, current run number, and temperature.

At the bottom of the screen is a set of five keys that are used for the primary instrument functions. These keys are available to you regardless of the menu selected. See the next section for an explanation of the *primary function keys*.



NOTE: Experiment information and instrument constants are entered from the controller keyboard, not the instrument touch screen.

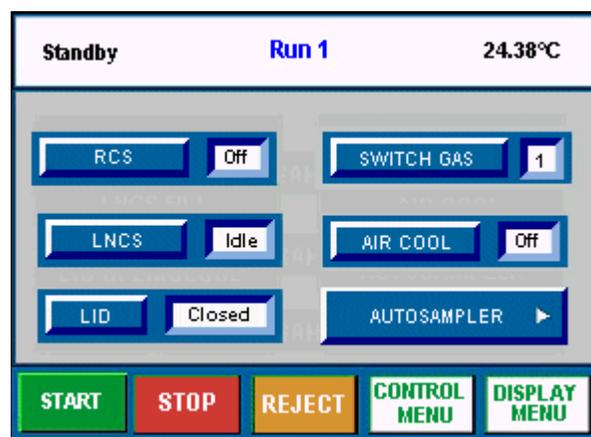
Primary Function Keys

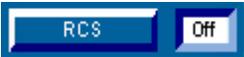
This set of keys, found at the bottom of the touch screen, are used to perform the basic functions of the instrument and to access the two main screens. See the table below for details.

Key Name	Description
	Begins the experiment. This is the same function as Start on the instrument control software.
	If an experiment is running, this key ends the method normally, as though it had run to completion; <i>i.e.</i> , 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 key will halt any activity (air cool, all mechanical motion, <i>etc.</i>).
	If an experiment is running, REJECT ends the method. The the method-end conditions go into effect just as if the method had run to completion. However, the data that has been generated is <i>discarded</i> . This is the same function as Reject on the instrument control software.
	Displays the Control Menu touch screen keys. These are used to control certain instrument actions.
	Accesses the Display Menu screen, which is used to select the desired display option.

DSC Control Menu Keys

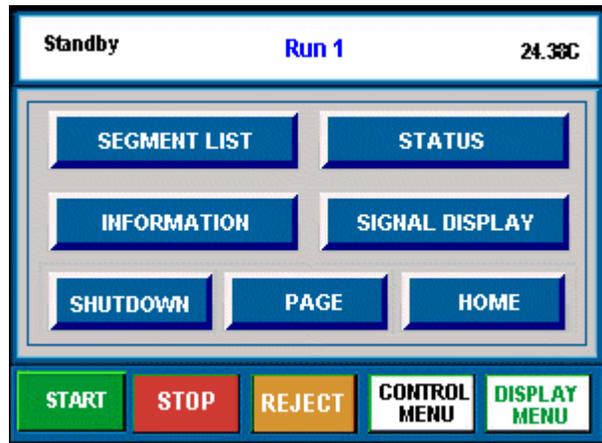
The Control Menu is accessed by touching the Control Menu key at the bottom of the touch screen. The keys shown in the figure here are displayed. A brief description of the function of each key is provided in the table below. The available keys will vary based on the selected coolers and installed accessories.



Key Name	Description
	Toggles the RCS (Refrigerated Cooling System) between on and off, depending on the state of the RCS when you press the key.
	Toggles between purge Gas #1 and Gas #2. See page 35 for information on gases to be used with the DSC.
	Automatically fills the LNCS with liquid nitrogen from the holding tank when an experiment is not in progress.
	Toggles the air cool function on or off. This is the same function as Air Cool on the instrument control software.
	Toggles between opening and closing the AutoLid on the cell.
	Takes you to the Autosampler touch screen display where you can calibrate the Autosampler and perform related functions. See the online help for information.

Display Menu Keys

The Display Menu is accessed by touching the DISPLAY MENU key at the bottom of the touch screen. The menu shown in the figure here will be displayed. A brief description of the function of each key is provided in the table below.



Key Name	Description
	Accesses the experimental procedure that is currently being used and highlights the active segment.
	Displays the three main signals indicating the current status of the experiment.
	Displays instrument information such as the software version, options, and the IP (Internet Protocol) address.
	Displays the real-time signal data that comes directly from the instrument. The signals displayed here are customized through the instrument control software.
	Ensures proper shutdown of the instrument before turning off the power.
	Beeps the controller that is connected to the instrument.
	Returns to the opening window.

DSC Auto Q20/Q20/Q10 Keypad

The DSC Auto Q20, Q20, and Q10 instruments do not have a touch screen for communication; these instruments use a keypad shown below.



The DSC instrument keypads, shown here, contain keys that control local operations at the instrument (experiment starting and stopping). Experiment information and instrument parameters are entered using the instrument control software.

The table below explains the functions of the instrument keys:

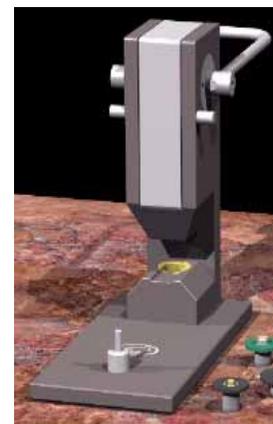
Key	Description
Start Key 	This key begins the experiment. This is the same function as Start on the instrument control software.
Stop Key 	If an experiment is running, this key ends the method normally, as though it had run to completion; <i>i.e.</i> , the post test conditions go into effect and the data that has been generated is saved. This is the same function as Stop on the instrument control software.
Open/Close Key 	<i>(Available on Auto Q20 Only.)</i> Toggles between opening and closing the DSC Auto Q20 Autolid.

Options and Accessories

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

Tzero Sample Encapsulating Press

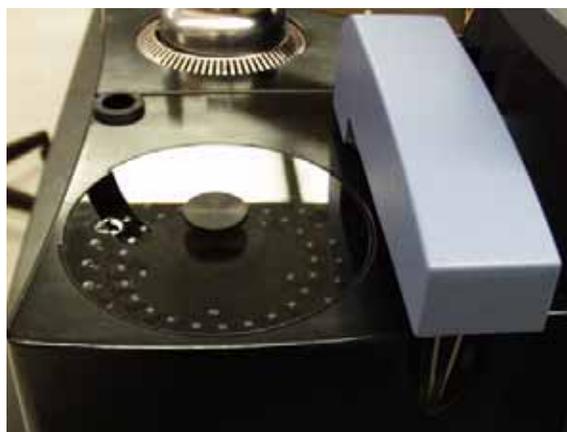
The TA Instruments Q Series Tzero Sample Encapsulating Press is used to prepare encapsulated samples for DSC experiments. It comes with die sets for hermetic and non-hermetic sealing. The online documentation provides details about using this accessory.



DSC Autosampler

The DSC Autosampler, also known as the Auto DSC, is standard on the TA Instruments' DSC Q2000 and Q1000, and an optional accessory to the DSC Q200, Q100, and Q20.

The Autosampler is a four-axis robotic device that automatically loads sample and reference pans to and from the Differential Scanning Calorimeter (DSC) measurement cell. Once the Autosampler loads the pans and the cell covers are in place, the controller takes over and runs the preprogrammed thermal analysis experiments. After the experiment is complete, the instrument uncovers the cell the Autosampler then unloads the pans. The unattended process can be preprogrammed to run up to 50 experiments.



Autosampler Tray and Arm

Photocalorimeter Accessory (PCA)



The Photocalorimeter Accessory (PCA) is used with the TA Instruments Q2000, Q1000, Q200, and Q100 Differential Scanning Calorimeters. This accessory allows samples placed inside the DSC cell to be irradiated with ultraviolet or visible light. When the samples (usually photopolymers) react to the light, heat is released (*i.e.*, an exothermic reaction occurs). This heat is measured and used to study the relative reactivity and/or kinetics of reaction. The reactions studied are typically rapid and results are obtained in less than 15 minutes.

DSC Pressure Cell

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

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

The Pressure DSC cell has two gas flow control valves, a three-way valve, a pressure gauge, a pressure release valve, and gas pressure fittings on the side. The cell has a pressure transducer which continuously measures actual sample pressure and stores it in the data file.

This cell is optional for the Q2000 and Q1000. It comes standard on the Q20P and Q10P.



Instrument Specifications

The tables found on the following pages contain the technical specifications for the DSC. The specifications apply to all models, except where noted.

DSC Instrument Characteristics

Dimensions (Q2000, Q200, Q1000 & Q100)	Depth 56 cm (22 in.) Width 46 cm (18 in.) Height 48 cm (19 in.)
Dimensions (Auto Q20/Q20/Q10)	Depth 56 cm (22 in.) Width 46 cm (18 in.) Height 41 cm (16 in.)
Weight (without Autosampler) Weight (with Autosampler) Weight of cell alone Weight of Autosampler alone Weight of transformer	25 kg (54 lbs) 29 kg (65 lbs) 1.6 kg (3.5 lbs) 4.5 kg (10 lbs) 8.6 kg (19 lbs)
Power	120 Vac, 47–63 Hz, 500 W (4.5 amps) standard 230 Vac, 47–63 Hz, 500 W (2.25 amps) if configured with a voltage configuration unit
Accessory Outlets	Power: 120 V, 47–63 Hz, 400 W each Compatible accessories: LNCS, DPC
Operating Environment Conditions	Temperature: 15–30 °C Relative Humidity: 5–80 % (non-condensing) Installation Category II Pollution Degree 2 Maximum Altitude: 2000 m
Temperature Range: with FACS with Quench Cooler with RCS 90 with RCS 40 with LNCS	Room temperature to 725 °C* -180 °C to 550 °C** -90 to 550 °C -40 to 400 °C -180 to 550 °C
<p>* You must use an inert atmosphere above 600 °C. ** Helium purge gas must be used for temperatures below -100 °C</p> <p style="text-align: right;"><i>(table continued)</i></p>	

Sample size	0.5 to 100 mg (nominal)
Sample volume	10 mm ³ in hermetic pans
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 volume	3.4 mL

Chapter 2

Installing the DSC

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 documentation associated with the instrument control software. You may wish to retain all of the shipping hardware, the plywood, 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.

Installing the Instrument

Before shipment, the DSC instrument 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 documentation for additional information. Installation involves the following procedures:

- Inspecting the system for shipping damage and missing parts
- Connecting the DSC to the TA Instruments controller
- Connecting cables and gas lines
- Installing a transformer, if needed, for 230 Vac power

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.

Inspecting the System

When you receive your DSC, 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 DSC should be:

In ... a temperature-controlled area.
... a clean, vibration-free environment.
... an area with ample working and ventilation space.

On... a stable work surface.

*Near...*a power outlet (120 Vac, 50 or 60 Hz, 15 amps, or 230 Vac, 50 or 60 Hz, 10 amps if configured with a step down transformer).
...your TA Instruments thermal analysis controller.
...compressed lab air and purge gas supplies with suitable regulators and flowmeters, if a mass flow controller (MFC) is not installed in the instrument.

*Away from...*dusty environments.
... exposure to direct sunlight.
... direct air drafts (fans, room air ducts).
... poorly ventilated areas.
... noisy or mechanical vibrations.



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

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

- 1 External Event Off**
 - 2 Equilibrate at 75°C**
 - 3 Isothermal for 120.00 min.**
-

Connecting Cables and Lines

To connect the cables and gas lines, you will need access to the DSC instrument's rear panel. All directional descriptions are written on the assumption that you are facing the back of the instrument.

NOTE: Connect all cables before connecting the power cords to outlets. Tighten the thumb-screws on all computer cables.



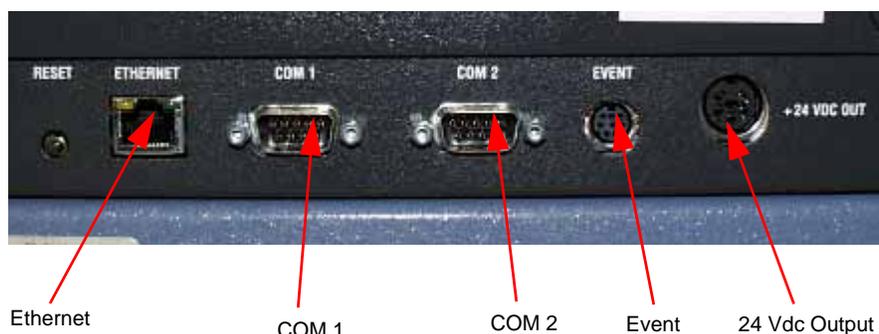
CAUTION: Whenever plugging or unplugging power cords, handle them by the plugs, not by the cords.



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

Ports

The DSC has nine ports that are located on the back of the instrument. The following table provides a description of function of each port. Refer to this list when connecting cables and lines.



Ethernet

COM 1

COM 2

Event

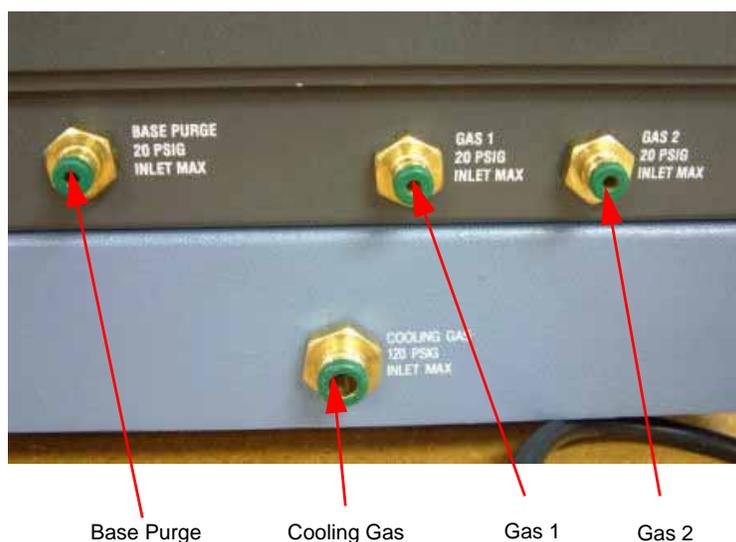
24 Vdc Output

Five Ports on Left Rear of DSC

Port	Function
Ethernet	Provides communication capabilities.
Com 1	Standard RS232 level signals.
Com 2	Accessory port. Standard RS232 level signals. Used for LNCS communications.
Event	Capable of the following functions: general purpose relay contact closure, or general purpose input 4 – 24 Vdc for external synchronization. This port is used for the RCS and Differential Photocalorimeter (DPC) control.
24 Vdc output	May be used for certain cooling accessories.
Base Purge	Used as an inlet port for nitrogen purge with the LNCS, QCA, and RCS cooling accessories.

(table continued)

Port	Function
Gas 1	Gas inlet port controlled by the Mass Flow Controller when installed. Used for the primary sample purge gas.
Gas 2	Gas inlet port. Used, when needed, for the secondary sample purge gas (if equipped with MFC).
Cooling Gas	Provides air for the Finned Air Cooling System (FACS) and nitrogen for the Refrigerated Cooling System (RCS).



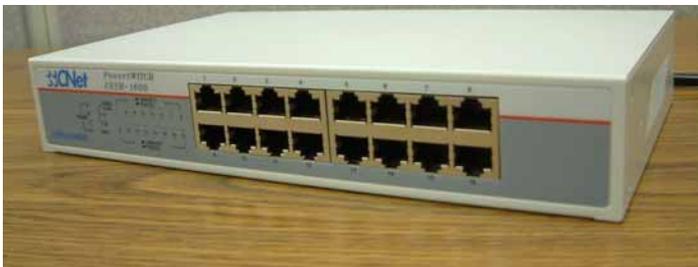
Four Ports on the Right Rear of the DSC

Ethernet Switch Setup

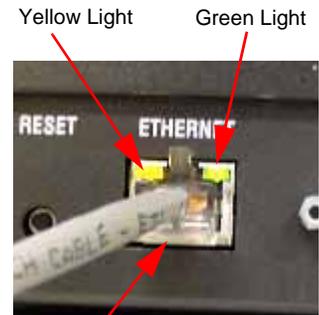
In order to connect the instrument to a network, you will need to make the necessary cable connections as described below. The instrument and controller will be connected to an Ethernet switch. In addition, there are instructions for connecting the controller to a LAN.

Connecting the Instrument to the Switch

1. Locate the Ethernet port on the left rear of the instrument (shown in the figure to the right).
2. Connect one end of the Ethernet cable into the instrument's Ethernet port.
3. Connect the other end of the Ethernet cable to one of the network ports on the Ethernet switch (shown in the figure below).



Ethernet Switch



Ethernet Connection

4. Check the configuration switches, located on the back panel. They must be set to off, or the up position, for the controller to communicate to the instruments.
5. Check the Ethernet port on the rear of the instrument. If communication between the instrument and the switch has been properly established, a solid green light and flashing yellow light will appear at the port.
6. Follow the directions in the next section to connect the controller to the Ethernet switch.



Configuration Switches

Connecting the Controller to the Switch

1. Locate the Ethernet port on the back of the computer.
2. Plug one end of the Ethernet cable into the computer's Ethernet port (shown in the figure to the right).
3. Connect the other end of the cable to one of the network ports on the switch.
4. Check the Ethernet port on the rear of the computer. If communication between the computer and the switch has been properly established, a solid green light and flashing yellow light will appear at the port.
5. Follow the directions in the next section to connect the controller to a LAN for networking capabilities.

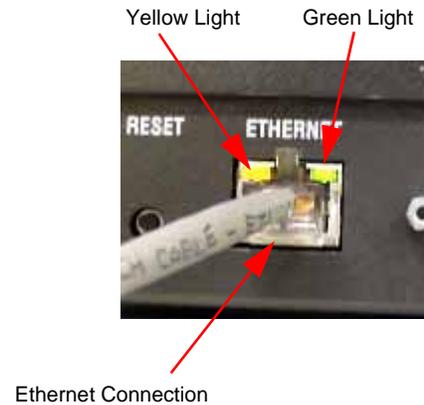


Computer Ethernet Port

Connecting the Controller to a LAN

Before you can connect the controller to a LAN, you will need to have already installed a network interface card into the computer.

1. Locate the second Ethernet port on the back of the computer.
2. Plug one end of the Ethernet cable into the computer's Ethernet port.
3. Plug the other end into the LAN.
4. Check the Ethernet port on the rear of the computer. If communication between the computer and the LAN has been properly established, a solid green light and flashing yellow light will appear at the port.



Purge Lines

You can control the sample atmosphere during DSC experiments by connecting purge gases to the system. The DSC instruments are equipped with a mass flow controller (MFC) to control the flow rate of the gas. Up to two different gases may be connected to the instrument to facilitate gas switching. Follow these instructions to connect the purge lines. Refer to the figure below to locate the purge lines.



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

1. Locate the Gas 1 port. The Gas 1 port is used to purge the sample area.
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 using 1/8-inch O.D. tubing. Teflon® TFE tubing is recommended and is supplied in the instrument shipping accessory kit. If desired, connect a secondary gas to the Gas 2 port.



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

For older Q100 or Q10 instruments that may not have an MFC, it is important to maintain the proper flow rates by using a flowmeter that is connected to each of the purge fittings (Gas 1 and Gas 2) on the back of the DSC.

4. Make sure that the pressure of your purge gas source is regulated between 100 and 140 kPa gauge (15 and 20 psig).
5. Specify the connected gas on the Instrument Preferences/MFC Page using the instrument control software.
6. Set the flow rate to the recommended value of 50 mL per minute for your experiments on the Notes Page of the Experiment View. Click Apply to save the changes.

NOTE: If you are using laboratory purge, rather than bottled purge, it is highly recommended that you install an external drier and a five- μ m filter.



CAUTION: Corrosive gases cannot be used with this instrument.



WARNING: Use of an explosive gas as a purge gas is dangerous and is not recommended for this instrument. For a list of the purge gases that can be used with the DSC instrument, see Chapter 1.

Cooling Gas Line

The Cooling Gas port is used with the Finned Air Cooling System (FACS), Refrigerated Cooling System (RCS), or the Liquid Nitrogen Cooling System (LNCS). The FACS requires a source of compressed air, while the RCS and LNCS require a source of dry nitrogen for the cooling gas line. Follow these instructions to connect the appropriate cooling gas line.

1. Locate the fitting, a 1/4-inch compression fitting or 1/4-inch Legris fitting on the right rear of the DSC, marked with a 830 kPa gauge (120 psig) warning label.
2. Make sure your gas source is regulated between 170 to 830 kPa gauge (25 to 120 psig) when using the FACS. For details on the pressure required for the RCS and LNCS, see the appropriate *Getting Started Guide*.



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

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

Base Purge Line

A base purge is required when the Quench Cooling Accessory (QCA), Refrigerated Cooling System (RCS), or the Liquid Nitrogen Cooling System (LNCS) are used.

1. Locate the Base Purge port.
2. Make sure that the pressure of your gas source is regulated between 100 and 140 kPa gauge (15 and 20 psig). Dry nitrogen is the recommended gas.
3. Connect the gas line to the Base Purge port using 1/8-inch O.D. tubing. Teflon® TFE tubing is recommended. An orifice in the instrument will automatically regulate the flow rate (300 to 350 mL/min) for proper operation.

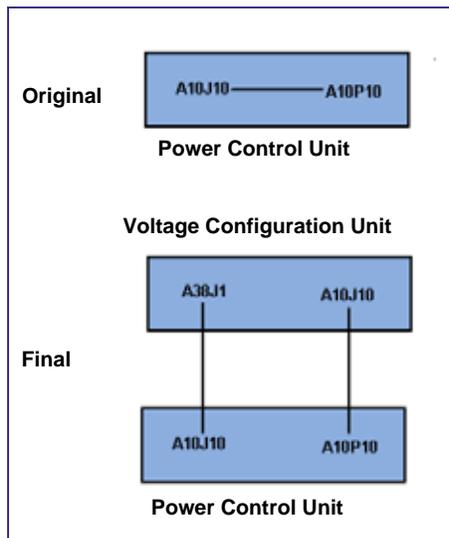
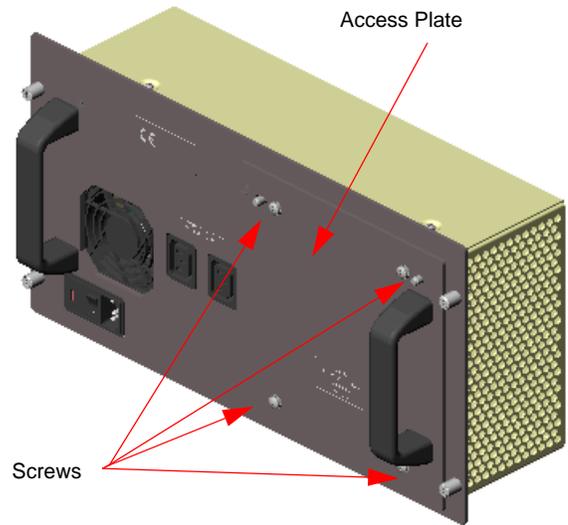
Voltage Configuration Unit

A voltage configuration unit is required if you use 230 Vac, rather than 120 Vac. Follow these steps to install the transformer in the Power Control Unit (PCU):

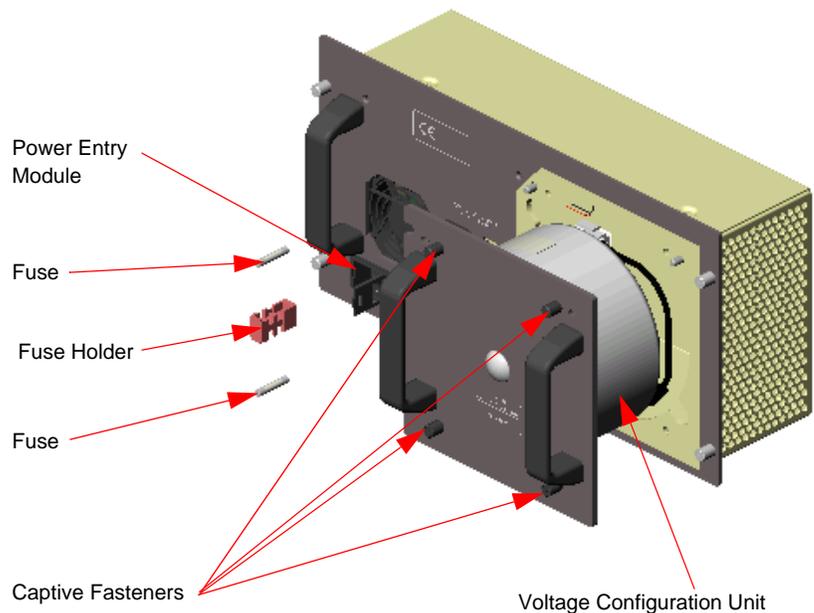


WARNING: High voltages are present in this instrument as indicated by the  label. Be sure to unplug the instrument before performing these instructions. See the WARNING on page 9.

1. Remove the contents from the shipping box and verify that all of the components are present.
2. Remove the access plate located on the rear of the instrument by removing the four (4) screws that secure it in place. See the figure to the right.
3. Disconnect the A10J10 connector from A10P10 located inside the PCU. Now connect the A10J10 connector on the voltage configuration unit to A10P10 located inside the PCU. Then connect A10J10 located inside the PCU to A38J1 on the anti-surge subassembly. See the diagram below for clarification.



4. Install the subassembly into the PCU and tighten the four (4) captive fasteners to secure it.
5. Remove the fuse holder from the power entry module and replace the 10 amp fuses with 6.3 amp fuses, which are supplied in the kit. Discard the 10 amp fuses. See the figure to the right.



Power Switch

The power switch is located at the rear of the instrument. It is part of the assembly called the *power entry module*, which also contains the power cable connection and fuses. The power switch is used to turn the instrument on and off.

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 DSC POWER switch is in the Off (0) position.
2. Plug the power cable into the DSC power entry module.



CAUTION: Before plugging the DSC power cable into the wall outlet, make sure the instrument is compatible with the line voltage. Check the label on the back of the unit to verify the voltage.

3. Plug the power cable into the wall outlet.



Starting the DSC

1. Check all connections between the DSC and the controller. Make sure each component is plugged into the correct connector.
2. Set the instrument power switch to the ON (1) position.

After the proper power up sequence, the TA Instruments logo will be displayed on the touch screen or the green light on the keypad will be lit, this indicates that the instrument is ready for use.

NOTE: Allow the DSC to warm up for at least 30 minutes before performing an experiment.

Shutting Down the DSC

Before you decide to power down your instrument, 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 and the controller 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 select **Control/Shutdown Instrument** from the Instrument Control menu or select **SHUTDOWN** from the instrument. A confirmation message will be displayed. Select OK (touchscreen) or Shutdown (Instrument Control) to proceed. All communication to the instrument will be halted while the instrument saves data to the flash screen. Once this procedure is complete, the instrument will post a message indicating that it is safe to turn off the power to the instrument or reset the instrument.

To power down your instrument set the power switch to the OFF (0) position.

Installing the DSC Cooling Accessories

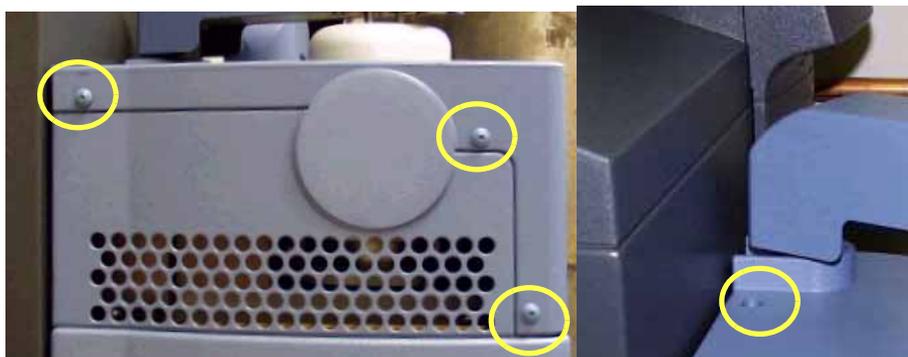
The DSC operates in conjunction with one of several cooling accessories. Your choice of cooling accessories depends on the temperature range that you wish to use for your experiments. This section provides the information that you need to install the Finned Air Cooling System and Quench Coolers. For proper operation, the correct cooler type must be selected through the instrument control software. See the online documentation for details.

NOTE: For information on installing the RCS 90, RCS 40, and LNCS consult the appropriate manual or the online help.

Installing the Finned Air Cooling System

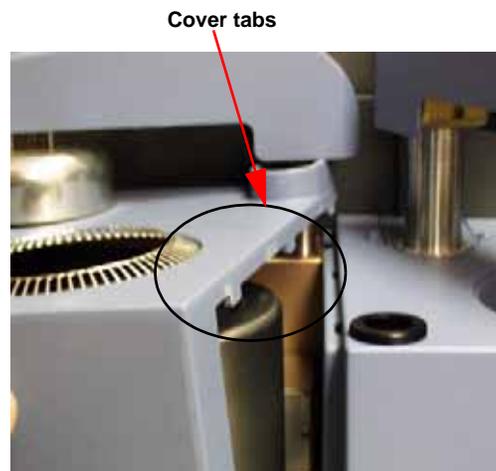
The Finned Air Cooling System (FACS) is an optional accessory that can be used with heating experiments to provide a heat sink for optimum performance. To install the FACS follow these instructions:

1. Remove the lid(s). If you do not have an AutoLid on your instrument, manually remove the lids. If your instrument has an AutoLid, select the **Control/Lid/Open** function to raise the Autolid from the cell and cause it to move out of the way to its home position.
2. Pull the plug on the side of the unit cover out to remove it. Then remove the screws attaching the cell cover to the unit cover (circled in the figure below). Three screws are located on the side (Q2000/Q1000) and one is located on the top. Retain the screws.



3. If your instrument has an Autosampler installed, lift up the cover to release the tabs (shown here) and pull the cover towards you to remove it fully being careful not to damage the cell lids.

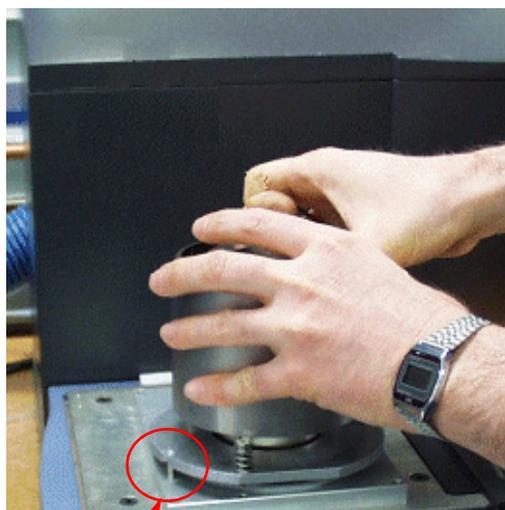
If you do not have an Autosampler, you will have to remove additional screws to release the cover. Then pull the cover towards you to remove it fully.



- Place the Finned Cooler over the cell and align the notch in the plate with the alignment pin as seen in the figure to the right.
- Obtain a long 5/32-inch hexagonal (Allen) wrench from the accessory kit.
- Insert the tip of the wrench into any one of the three captive screws in the FACS plate while holding onto the cooler. (See the figure below.) You will need to exert downward pressure on the wrench while you tighten the screw a few turns. DO NOT fully tighten yet.



- Repeat step 6 for the two remaining captive screws. After you have started each screw, go back and tighten down all three screws until you feel them touch the bottom. Do not over tighten.



Notch and Alignment Pin

- Slide the cover back over the cell and replace the screws removed originally. Push the plug back into place on the side of the cover.
- Select the correct cooler type on the Instrument Preferences/Cooler Page of the DSC instrument control software.

You are now ready to use the Finned Air Cooling System. (This cooler requires a pressurized air source connected to the Cooling Gas fitting.)

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 Finned Air Cooling System (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 Finned Air Cooling System is already installed. (Follow the directions beginning on page 38 if you need to install the FACS first.)

- Remove the lid(s). If you do not have an AutoLid on your instrument, manually remove the lids. If your instrument has an AutoLid, select the **Control/Lid/Open** function to raise the Autolid from the cell and cause it to move out of the way.
- Slide the cooler into place into the top of the Finned Air Cooling System (FACS) using gloves to handle it, if you have already poured liquid nitrogen into it as shown in the figure here.



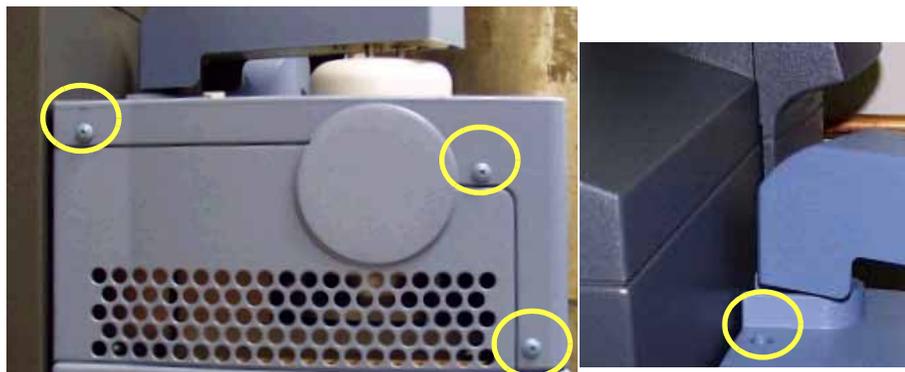
NOTE: When using the FACS quench cooler make sure that the cooling accessory identified in the instrument control program is the FACS and not the quench cooler. If the wrong cooler is identified, your results may not be as expected.

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. Remove the lid(s). If you do not have an AutoLid on your instrument, manually remove the lids. If your instrument has an AutoLid, select the **Control/Lid/Open** function to raise the Autolid from the cell and cause it to move out of the way to its home position.

2. Pull the plug on the side of the unit cover out to remove it. Then remove the screws attaching the cell cover to the unit cover (circled in the figure to the right). Three screws are located on the side (Q2000/Q1000) and one is located on the top. Retain the screws.



3. If your instrument has an Autosampler installed, lift up the cover to release the tabs (shown in the figure below) and pull the cover towards you to remove it fully.



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

4. Place the thermal enclosure over the cell and align the notch in the plate with the alignment pin (shown in the figure below right).

5. Obtain a long 3/32-inch hexagonal (Allen) wrench from the accessory kit.

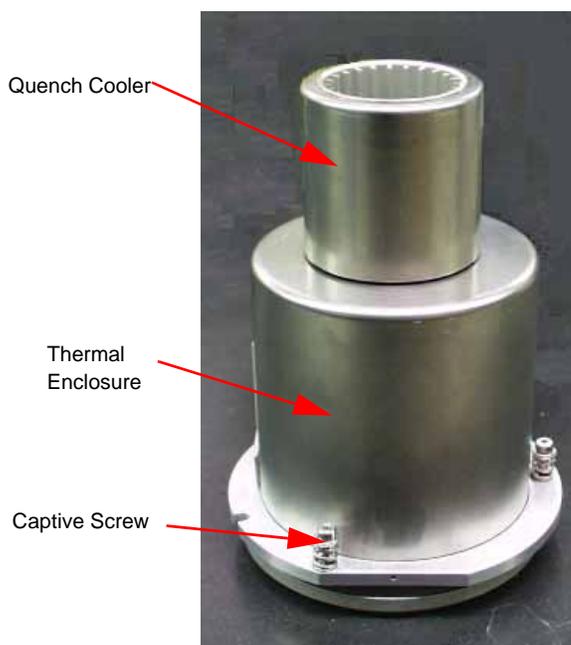
6. Insert the tip of the wrench into any one of the three captive screws in the thermal enclosure plate while holding onto it. You will need to exert downward pressure on the wrench while you tighten the screw a few turns. DO NOT fully tighten yet.



Alignment Pin

7. Repeat step 6 for the two remaining captive screws. After you have started each screw, go back and tighten down all three screws until you feel them touch the bottom. Do not over tighten.
8. Slide the cover back over the cell and replace the screws removed originally. Push the plug back into place on the side of the cover.
9. Connect the base purge line. The system is now ready to receive the quench cooler.
10. Select the correct cooler type on the **Instrument Preferences/Cooler Page** of the DSC instrument control software. The AutoLid and Autosampler, if applicable, are both disabled when using the Quench Cooler.
11. Install the two inner lids over the cell when running experiments. Then slide the quench cooler into the top of the thermal enclosure using gloves to handle it, if you have already poured liquid nitrogen into it.

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



"Stand-Alone" Quench Cooler and Cooling Head

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 Manual Lids

When you receive your DSC Q20 or Q10 instrument the three lids will be 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. Each time you place a sample and reference pan in the cell you must cover the cell with the lids before beginning a run.

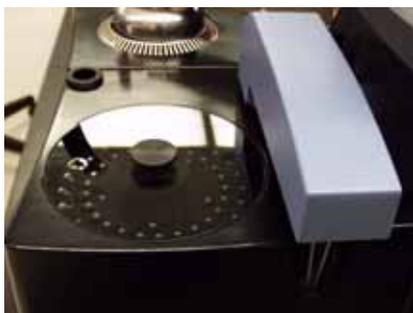
Install the smallest (inner) lid first, then the medium (outer) lid, and lastly the large (upper) lid on the top. Then you can begin your experiment.

Installing the Autosampler Tray and Dust Cover

When you receive your DSC, equipped with an Autosampler, the sample tray and dust cover will be 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.

Grasp the tray by the center knob and lower it into the opening positioning the grooves in the underside of the tray so that they fit into the appropriate position. The figure to the right illustrates the grooves and tabs.

Load the samples onto the tray. After the tray is loaded and installed, cover them with the clear plastic dust cover. Align the tray to fit into the predetermined slots so that the opening is properly positioned over the first sample pan to allow for proper operation of the Autosampler.



Chapter 3

Use, Maintenance, & Diagnostics

Using the 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 program; 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 TA 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 procedures carefully.

Before You Begin

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

- Made all necessary cable connections from the DSC to the computer
- Connected all gas lines
- Powered up the unit
- Connected any desired accessories
- Connected the instrument with the controller
- Become familiar with controller operations
- Calibrated the DSC, if necessary.

Calibrating the DSC

To obtain accurate experimental results you should calibrate the 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 instrument model and heat flow selection. These experiments will be performed using the instrument control software. See the chart below for a general guide to types of calibration. For details on how to perform that calibration, refer to the online documentation accessed through the instrument control 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.

Types of Calibration for the DSC

DSC Model	Tzero*	Cell Constant	Temperature	Baseline	Pressure	Heat Capacity**	
						MDSC	Q2000 Q1000
Auto Q20/Q20/Q10		✓	✓	✓			
Q200/Q100	Heat Capacity calibration is NOT performed in cal mode and is not required except when making absolute measurements of heat capacity.						
Heat Flow T4	✓	✓	✓			✓	
Heat Flow T1		✓	✓	✓			
Q2000/Q1000 with standard cell	Heat Capacity calibration is NOT performed in cal mode and is not required except when making absolute measurements of heat capacity.						
Heat Flow T4P	✓	✓	✓			✓	✓
Heat Flow T4	✓	✓	✓			✓	✓
Heat Flow T1		✓	✓	✓			
DSC with pressure cell	Heat Capacity calibration is NOT performed in cal mode and is not required except when making absolute measurements of heat capacity.						
		✓	✓	✓	✓		

* Tzero™ describes proprietary technology invented by Robert Danley of TA Instruments—Waters LLC and patented by TA Instruments—Waters LLC (U.S. Patent No. 6,431,747).

** Heat Capacity calibration is NOT performed in cal mode and is not required except when making absolute measurements of heat capacity.

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
- Purge gas change
- Cooling device or accessory (FACS, LNCS, RCS, or Quench Cooling Can) change.

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

- Heat flow selection change (T4P, T4, etc.)
- Pressure (in PDSC experiments) change.

Calibration is performed in the instrument's calibration mode, which is accessed through the controller. (This does not apply to heat capacity calibration, which is performed in both the standard and MDSC modes, where applicable).

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

TzeroTM

The DSC Tzero calibration requires two experiments. The first experiment is done without samples or pans (baseline); the second is performed with large (approximately 95 mg) sapphire disks (without pans) on both the sample and reference positions. Both experiments use the same method beginning with a cell preheat followed by an equilibration at an initial temperature, holding isothermal for five minutes, heating at constant rate to a final temperature and holding isothermal for 5 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.

Tzero calibration is used with the DSC Q2000 and Q1000 for Heat Flow T4 and T4P, and with the DSC Q200 and Q100 for Heat Flow T4.

NOTE: The "heat flow" signal should be selected and used to measure the heat of fusion or enthalpy of sharp melting transitions such as obtained with Indium.

Baseline Slope and Offset Calibration

The baseline slope and offset calibration involves heating an empty cell through the entire temperature range expected in subsequent experiments, holding isothermal at the temperature limits. The calibration program is used to calculate the slope and offset values needed to flatten the baseline and zero the heat flow signal. This calibration is used in the DSC Auto Q20, Q20, and Q10 and when the selected heat flow is T1 for the other DSC models.

Enthalpy (Cell) Constant Calibration

This calibration is based on a run in which a standard metal (e.g., indium) is heated through its melting transition. The calculated heat of fusion is compared to the theoretical value. The cell constant is the ratio between these two values. The onset slope, or thermal resistance, is a measure of the suppression of temperature rise that occurs in a melting sample in relation to the thermocouple. Theoretically, a standard sample should melt at a constant 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 a run in which a temperature standard (e.g., indium) is heated through its melting transition. The extrapolated onset of 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 three or more standards, the temperature is corrected 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 (Q2000/Q1000) Calibration

Heat capacity can be obtained in a single experiment with the DSC Q2000/Q1000. Heat capacity calibration is required if it is desirable to measure the absolute value of the sample's heat capacity. The calibration uses a standard material (such as sapphire) with a known heat capacity at a specific temperature of interest. Before performing the calibration experiment, the previously used heat capacity calibration values need to be manually set to the default value of 1.0. Calibration is typically done at a heating or cooling rate of 10 to 20 °C/min. Since this calibration (measurement) uses the absolute value of the heat flow signal ($C_p = \text{Heat Flow Rate}/\text{Heating Rate}$) to calculate the sample's heat capacity, the first segment of the method should be a "Zero Heat Flow" segment at a temperature in the middle of the temperature range for the experiment. This ensures that any drift in the heat flow signal, since the last calibration, is corrected.

At the end of the experiment the heat capacity calibration constant is calculated by dividing the theoretical value of heat capacity by the measured value at a specific temperature. The calculated constant is then manually entered into the instrument control software, where it is automatically applied to future experiments.

Heat Capacity (MDSC[®]) Calibration

MDSC heat capacity calibration is only required if it is desirable to measure the absolute value of the sample's heat capacity or when using periods less than or equal to 40 seconds. The calibration uses a standard material (such as sapphire) with a known heat capacity at a specific temperature of interest. Before performing the calibration experiment, the previously used calibration value needs to be manually set for both Heat Capacity and Reversing Heat Capacity to the default values of 1.0. 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.

At the end of the experiment the calibration constants for the Total and Reversing Heat Capacity signals are manually calculated by dividing the theoretical value of heat capacity, at the desired temperature, by the measured value. The constants are then manually entered into the instrument control software, where they are automatically applied to future experiments. See the online help for more detailed information.

Pressure Calibration

Pressure calibration is an optional calibration procedure for the Pressure DSC cells. It is based on comparing the pressure reading at two points, typically one atmosphere and another pressure selected by you, to the pressure reading on an external pressure gauge.

Autosampler Calibration

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

Once the Autosampler is correctly calibrated, you may need to recalibrate it only when you replace the DSC cell. Periodic checks are the best way of determining when the Autosampler needs recalibration. For more complete information on calibrating the Autosampler, see the online documentation.

Q2000/Q200/Auto Q20 Autosampler Calibration

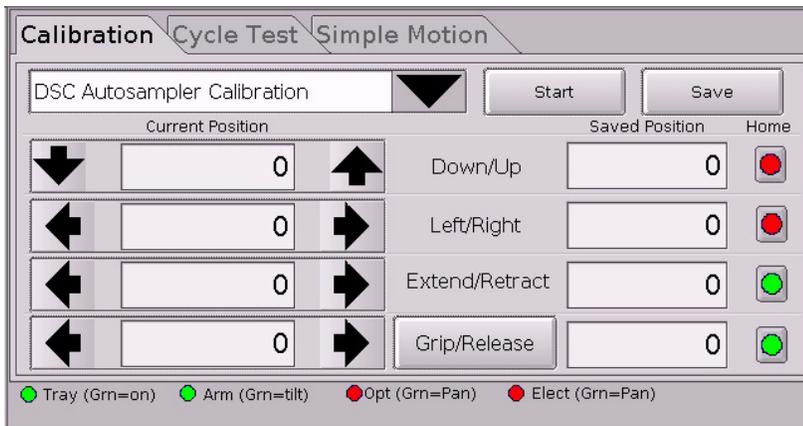
To perform Autosampler calibration follow these steps:

1. Access the Autosampler calibration functions via the touch screen or the instrument control menu. Refer to the section that applies.

- **Autosampler Calibration via the Touch Screen**

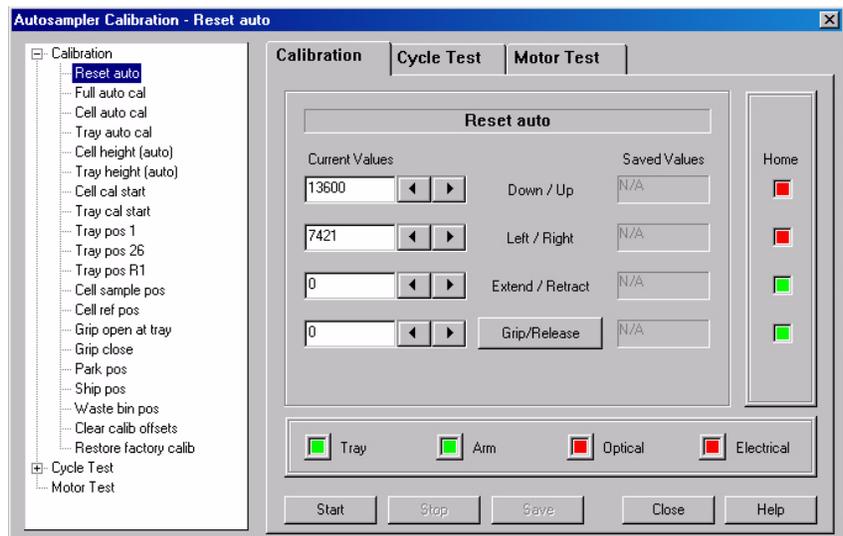
- a. Touch the **Calibrate** button at the bottom of the touch screen. The screen shown in the figure to the right will be displayed.
- b. Press the **Autosampler** button.
- c. Touch the **Calibration** tab, if it is not already selected. The screen shown on the next page is displayed.





- **Autosampler Calibration via Instrument Control**

- Select **Calibrate/ Autosampler** from the menu. The window shown in the figure to the right will be displayed.
- Click the plus (+) sign to display the types of calibration available.



2. Select the desired type of calibration from the list available.

- To calibrate both the sample tray and cell areas select **Full auto cal.**
- To calibrate the sample tray only select **Tray auto cal.**
- To calibrate the cell areas only select **Cell auto cal.**
- If you need to perform more refined calibrations, select any of the manual calibration functions.

See the online help for detailed information.

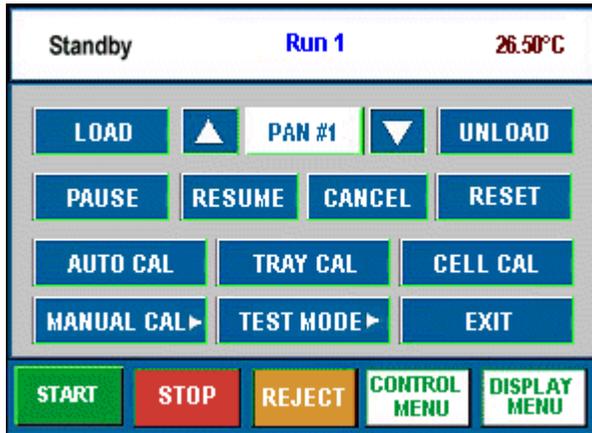
3. Make sure you press the **Save** button when you have finished the desired calibration function.

After calibration is complete, you can perform a **Cycle Test** to test the calibrated positions.

Q1000/Q100 Autosampler Calibration

To perform Autosampler calibration from the touch screen follow the instructions below.

1. Touch the **AUTOSAMPLER** button on the **CONTROL MENU** touch screen. The screen shown in the figure to the right will be displayed.
2. Select the desired type of calibration from the buttons available.
 - To calibrate both the sample tray and cell areas select **AUTO CAL**.
 - To calibrate the sample tray only select **TRAY CAL**.
 - To calibrate the cell areas only select **CELL CAL**.
 - If you need to perform more refined calibrations, select **MANUAL CAL** to access more calibration functions.



See the online help for detailed information.

3. Make sure you save the calibrations when you have finished the desired calibration function.

After calibration is complete, you can perform a **Cycle Test** to test the calibrated positions.

Running a DSC Experiment



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

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

- 1 External Event Off
 - 2 Equilibrate at 75°C
 - 3 Isothermal for 120.00 min.
-

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.

- Attaching and setting up external accessories as required (e.g., purge gas, cooling accessory). If you are planning to run subambient experiments with the LNCS, use helium as the purge gas. If you are using the LNCS for rapid cooling above ambient (*i.e.*, isothermal crystallization), then nitrogen may be used as a purge gas. If you are using the RCS 90 or RCS 40 for cooling, nitrogen may be used as a purge gas.

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.

- Selecting and preparing 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.
- Loading the sample pan (and a similarly prepared empty reference pan) into the cell or onto the Autosampler tray.
- Entering experiment and procedure information through the TA controller, this includes both sample and instrument information.
- Starting the experiment.

Loading the Sample

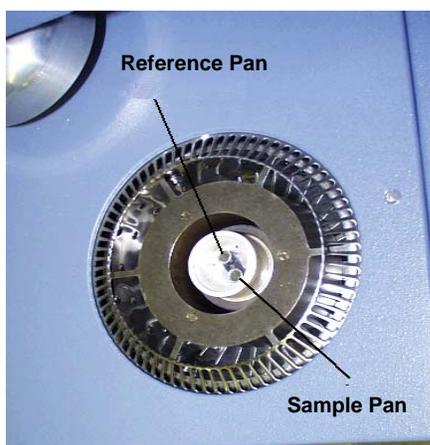
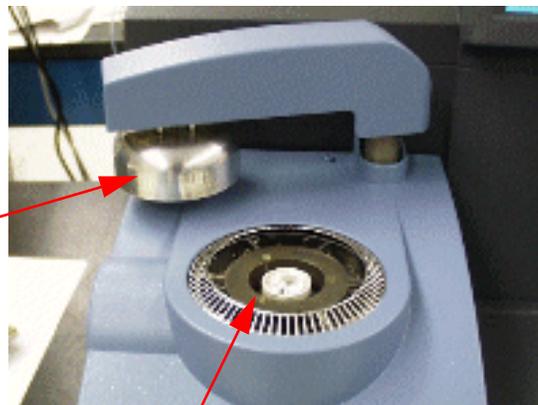
Once the sample pan has been prepared and the sample information has been recorded, you are ready to load the sample pan into the DSC Cell. If you have an Autosampler, the pans will automatically be loaded when you run an Autosampler sequence.



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.

Load the sample pan into the cell as follows:

1. Remove the lids. If the DSC has an autolid assembly, touch the LID OPEN key on the DSC Control Menu touch screen to raise the lid from the cell. See the figure to the right.
2. Carefully place the sample pan on the front right raised platform and the reference pan on the rear left platform (shown in the figure below). Centering the pans will give more accurate results.



3. Cover the cell. If the DSC has an autolid assembly, touch the LID CLOSED key on the DSC Control Menu touch screen to return the lid to the cell.

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.

NOTE: Once the experiment is started, operations are best performed at the computer keyboard. The DSC is very sensitive to motion and might pick up the vibration caused by touching a key on the instrument touch screen.

Start the experiment by selecting **Start** on the instrument control software, touching the **START** key on the instrument touch screen, or by pressing the **Start** key on the keypad (Auto Q20/Q20/Q10). 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, touching the **STOP** key on the instrument touch screen, or by pressing the **STOP** key on the keypad (Auto Q20/Q20/Q10).

Another function that stops the experiment is **REJECT**. However, the **Reject** function discards all of the data from the experiment; the **Stop** function saves any data collected up to the point at which the experiment was stopped.

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 documentation installed with the instrument control software for further information.



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

Cleaning the Touch Screen

You can clean the DSC touch screen as often as you like. The touch screen should be cleaned with a household liquid glass cleaner and soft cloth. Wet the cloth, not the touch screen with the glass cleaner, and then wipe off the touch screen and surrounding surfaces.



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

Cleaning a Contaminated Cell

Baseline anomalies are often the sign of a contaminated cell. DSC and PDSC cells must be cleaned properly to maintain satisfactory operation. Scraping the contamination off is not recommended.

If your baseline appears to show sample contamination, try the following recommended cleaning procedure. The procedure varies slightly depending upon the type of cell (QDSC Standard Cell or QDSC Pressure Cell) and the type of cooler that is installed. For instructions on cleaning the QDSC Pressure Cell refer to the online help or the manual for the QPDSC.

Start by removing any pans from the cell.

Cleaning a Contaminated QDSC Standard Cell

- Determine that the anomalies seen in the baseline are actually caused by contamination, and not a faulty Tzero™ calibration. Contamination is normally manifested in very sharp, distinct, and sometimes noisy, peaks in an empty baseline (no pans) run. If the baseline run shows shifts or step changes in the baseline, large startup events, or slope, these are more likely caused by a faulty Tzero calibration and no amount of cleaning will correct the problem.
- If you have determined that the anomalies in the baseline are in fact caused by contamination, select a solvent that is most appropriate for dissolving the contaminant. Note that in most cases and organic solvent such as acetone or methanol may be necessary, but an aqueous solvent might also work for some contaminants.
- Dampen the end of a small cotton swab with the chosen solvent. Avoid saturating the swab to prevent the solvent from dripping into the Tzero thermocouple tube.

- Gently dissolve the contaminants with the swab, starting at the outside of the silver housing within the sample chamber and working inwards toward the Tzero thermocouple. Repeat the process until the contaminants have been removed. Do not rub hard with the swab, especially at the Tzero thermocouple.
- Dry the interior of the cell with a clean dry swab.
- Heat the cell to 200°C in Nitrogen at approximately 50 ml/min, and hold for 5 minutes to remove all traces of solvent.
- Verify by running an empty baseline run. Always store all available Diagnostic Signals when running verification baseline runs. If the anomalies are gone, the baseline shape or slope is not acceptable, than Tzero calibration may be necessary. If the anomalies are still there, but are less evident, repeat the above steps and re-verify the baseline.
- If you are unable to remove the anomalies using the above procedure, then you will need to perform additional cell cleaning by heating the cell to a high temperature in an oxidizing atmosphere. This procedure should only be done as a last resort.
- Begin by connecting the air purge (See also: Connecting Purge Gas Lines), and then follow the steps below depending on which cooler is installed.

NOTE: Cleaning of the cell is performed with the lids off to prevent the lid gas exit tube from becoming plugged by condensing off-gases. If your instrument is equipped with an autolid mechanism you will need to open the cell manually at the start of the method. You can open the cell by selecting **LID** on the Control Menu touch screen or by selecting **Lids/Open** from the Control menu.

Burning Off Contamination in a DSC Cell with a FACS Installed

- Begin cleaning by heating the cell with an air purge to 50°C above the highest operating temperature or 600°C, whichever is lower, without pans. Use a heating rate of 20°C per minute. The Ramp test can be used for this method. Volatile contaminants can be removed at lower temperatures; decomposed contaminants must be oxidized above 550°C.
- After cool-down, lightly brush out the cell with a small fiberglass brush (included in the DSC accessory kit). Then remove the residue from the cell using clean compressed air.
- Run a baseline (empty cell). If there is an improvement relative to the baseline before “burn off” but the baseline is still unacceptable, repeat the procedure above.

Burning Off Contamination in a DSC Cell with an RCS or LNCS

- Begin cleaning by heating the cell with an air purge to 50°C above the highest operating temperature (400°C maximum for the RCS 40) or 550°C, whichever is lower, without pans with the cooling accessory ON. Use a heating rate of 20°C per minute. The Ramp test can be used for this method. Volatile contaminants can be removed at lower temperatures; decomposed contaminants must be oxidized above 550°C.



WARNING: Do not exceed 100°C with the RCS cooling head installed and the RCS power off. Serious damage and/or injury could occur.



CAUTION: We recommend that you do not use the RCS 90 when running isothermal experiments above 400°C. Damage to the unit can occur if used at high temperatures for extended periods. The RCS 40 cannot be heated above 400°C.

- After cool-down, lightly brush out the cell with a small fiberglass brush (included in the DSC accessory kit). Then remove the residue from the cell using clean compressed air.
- Run a baseline (empty cell). If there is an improvement relative to the baseline before “burn off” but the baseline is still unacceptable, repeat the procedure above. If you need to hold longer at the higher temperature to remove the contamination, you will need to remove the RCS or LNCS heat exchanger before conducting this experiment. You will also need to temporarily change the selected cooler type to FACS on the Instrument Preferences/Cooler Page. (Don’t forget to reselect the correct cooler type after performing this experiment.)

NOTE: When removing the RCS/LNCS heat exchanger from the cell, it is recommended that the RCS or LNCS be allowed to warm to room temperature after turning it “off” before removing the heat exchanger. That will minimize the chance for moisture condensation/contamination and will avoid the possibility of damaging the connector hose by moving while cold (and brittle).

- Removing the heat exchanger requires that the DSC-RCS/LNCS system be reconditioned and recalibrated after reassembled on the cell.
- Minor baseline anomalies due to sample contamination that remain after cleaning may be compensated for by a new Tzero calibration. They cannot be compensated for by a Q20/Q10 baseline calibration.

If the baseline problem remains, it is probably not due to contamination; the cell may need to be replaced (contact your TA Instruments service representative).

- Once the baseline is acceptable, return to normal operation.

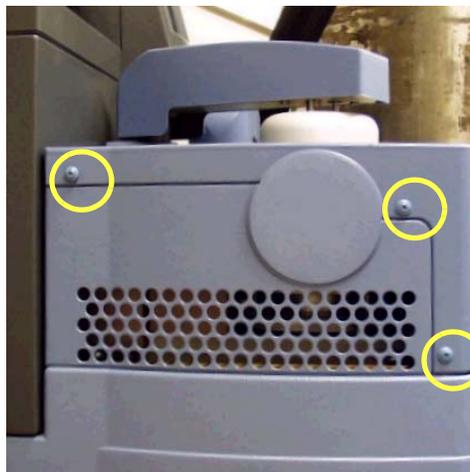
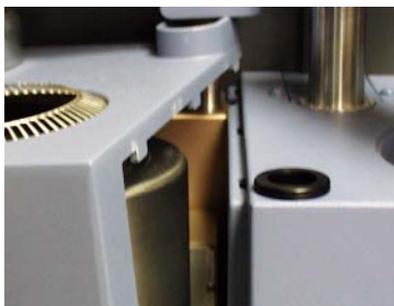
Removing a DSC Q2000/Q1000 Cell

Should you need to remove the DSC Q2000/Q1000 cell from the instrument for any reason, follow these steps:

1. Select the **Control/Lid/Open** function to raise the Autolid from the cell and cause it to move out of the way to its home position.
2. Pull the plug on the side of the unit cover out to remove it. Then remove the screws attaching the cell cover to the unit cover (circled in the figure to the right). Three screws are located on the side and one is located on the top. Retain the screws.

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

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



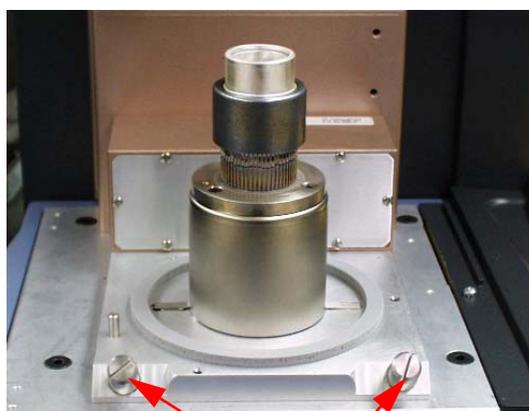
4. If there is a cooling accessory installed, make sure that it has reached room temperature, then remove it. See Chapter 2 and reverse the installation instructions for that cooler.
5. Remove the two thumbscrews at the front of the cell base as shown in the figure to the right.
6. Make sure the cell has reached room temperature, then slide out the cell.

Installing a Cell

To install a cell on the Q2000/Q1000, slide the cell into place fully engaging the connectors.

Tighten the thumbscrews equally until they cannot be tightened further — do not force.

Then reverse the directions given in the previous section to reinstall the cover.



Replacing Fuses



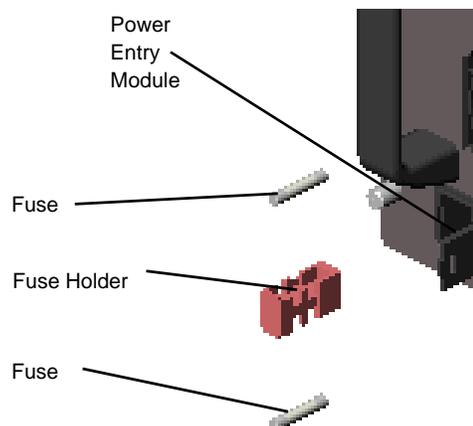
WARNING: Always unplug the instrument before you examine or replace the fuses.

Replacing the DSC Fuses

The 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 fuses that you can replace yourself are the fuses located in the power entry module located at the rear of the instrument. To check or change these fuses:

1. Turn the instrument off and remove the power cord.
2. Insert a small screwdriver at the edge of the power entry module door and pry it open.
3. Insert the screwdriver on the edge of the fuse holder to pull it out of the instrument.
4. Remove old fuses and replace the fuses only with the type and rating indicated on the instrument's rear panel.
5. Place fuse holder back into opening and push the door shut.



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 DSC that are available from TA Instruments. See the tables below when ordering parts.

Fuses, Cords, and Cables

Part Number	Description
205221.001	Fuse (6.3 amp, 250 V)
205221.002	Fuse (10 amp, 250 V)
205224.035	Fuse (0.75 amp, 250V Slo Blo) for 230V/50Hz RCS
205224.039	Fuse (1.0 amp, 250V Slo Blo) for 120V/60Hz RCS
251470.025	Ethernet cable (7.7m [25 foot], shielded)
253827.000	Power cord 120 V(Q10, Q100, Q1000, RCS)
270469.001	Power cord 220 V (RCS)
920223.901	Event cable

DSC Cells, Lids, and Accessories

Part Number	Description
910824.001	Cell cleaning brush
970020.901	Removable cell (Q2000/Q1000)
970520.901	Fixed Cell (Q200 / Q100 / Q20 / Q10 / AQ20) - Service installation required
970045.901	Removable cell thumb screw (Q200/Q1000)
970132.001	Cell plate gasket
970464.001	Upper lid (AutoLid II - Q200, Q20000)
970442.001	Inner lid (Manual - Q20, AutoLid II - Q200, Q2000)
970283.001	Outer manual lid (Q10, Q20)
970284.901	Manual cell cover (Q10, Q20)
970291.001	Inner lid (AutoLid I - Q100, Q1000)
970292.901	Auto cell cover (AutoLid I - Q100, Q1000, AutoLid II - Q200, Q2000)
970294.001	Auto lid clamp (AutoLid I - Q100, Q1000)
970295.001	Auto lid clamp spring (AutoLid I - Q100, Q1000)
<i>(table continued)</i>	

Part Number	Description
970520.901	Q10, Q100 Fixed cell - Service installation required.
970003.901	Mass Flow Controller Upgrade Kit for Q10/Q100 - Includes Field Service installation
271580.001	Curved tweezers
271244.001	Auto lid bias spring (AutoLid I - Q100, Q1000, AutoLid II - Q200, Q2000)
970830.901	AutoLid II Upgrade for Q1000/Q100

Autosampler Accessories

Part Number	Description
971126.901	Autosampler Sample Pan Tray
971012.901	Autosampler Dust Cover
971081.901	Autosampler Cell Calibration Fixture
971092.001	Autosampler Sample Waste Bin Container

Tzero Sample Encapsulation Press

Part Number	Description
901600.901	Tzero Sample Press Kit (Includes the press, the 3 die sets given below, and 1 box each of Tzero Aluminum pans (PN 901670.901) and Tzero Aluminum Lids (PN 901671.901))
901608.901	Replacement Die Set for Standard Series Pans
901608.903	Replacement Die Set for Tzero Series Aluminum Pans
901608.904	Replacement Die Set for Tzero Series Hermetic Pans and Tzero Series Hermetic Alodined Pans

DSC Sample Pans, Lids, and Accessories

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)

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)
	<i>(continued)</i>

Standard Series Pans and Lids

Part Number	Description
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)

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

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