# **DSC 8500**

# Installation and Hardware Guide



#### **Release History**

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Any comments about the documentation for this product should be addressed to:

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### **Pyris Installation**

*NOTE: : If you are going to install multiple analyzers, or just want to install a universal serial bus, see Install Multiple Analyzers before you begin to install an analyser.* 

This user's guide gives information on the installation of your PerkinElmer Thermal Analysis System, and details about the operation and maintenance of the hardware. This information can also be found in the Pyris Installation and Hardware Help provided with your Pyris software.

Information on the operation of the Pyris software can be found in the software Help. In general, the installation procedure consists of the following steps:

- Prepare the laboratory
- Unpack the thermal analysis system
- Set up the required system components (for example, purge gases, cooling supply, and analyzer-specific items)
- Connect the system components
- Configure the analyzer
- Calibrate the analyzer

# <u>Safety and Regulatory</u> <u>Information</u>

## Symbold Used in Online Help

The Pyris help files contain information and warnings that must be followed by the user to ensure safe operation and to maintain the instrument(s) in a safe condition.

Possible hazards that could harm the user or result in damage to the instrument are clearly stated at appropriate places throughout Help.

The following safety conventions are used throughout online Help:

Normal text is used to provide information and instructions.

**Bold** text refers to text that is displayed on the screen.

UPPERCASE text, for example ENTER or ALT, refers to keys on the PC keyboard. '+' is used to show that you have to press two keys at the same time, for example, ALT+F.

All eight digit numbers are PerkinElmer part numbers unless stated otherwise.

#### Notes, cautions and warnings

Three terms, in the following standard formats, are also used to highlight special circumstances and warnings.

**NOTE:** A note indicates additional, significant information that is provided with some procedures.

CAUTION	We use the term CAUTION to inform you about situations that could result in <b>serious damage to the instrument</b> or other equipment. Details about these circumstances are in a box like this one.
D	<b>Caution (Achtung)</b> Bedeutet, daß die genannte Anleitung genau befolgt werden muß, um einen <b>Geräteschaden</b> zu vermeiden.
DK	<i>Caution (Bemærk)</i> Dette betyder, at den nævnte vejledning skal overholdes nøje for at undgå en <b>beskadigelse af apparatet</b> .
E	<i>Caution (Advertencia)</i> <i>Utilizamos el término CAUTION (ADVERTENCIA) para advertir sobre</i> <i>situaciones que pueden provocar averías graves en este equipo</i> o en <i>otros. En recuadros éste se proporciona información sobre este tipo de</i> <i>circunstancias.</i>
F	<i>Caution (Attention)</i> Nous utilisons le terme <i>CAUTION</i> (ATTENTION) pour signaler les situations susceptibles de provoquer de graves détériorations de <i>l'instrument</i> ou d'autre matériel. Les détails sur ces circonstances figurent dans un encadré semblable à celui-ci.
	<i>Caution (Attenzione)</i> Con il termine <i>CAUTION</i> (ATTENZIONE) vengono segnalate situazioni che potrebbero arrecare gravi danni allo strumento o ad altra apparecchiatura. Troverete informazioni su tali circostanze in un riquadro come questo.
NL	<i>Caution (Opgelet)</i> Betekent dat de genoemde handleiding nauwkeurig moet worden opgevolgd, om <b>beschadiging van het instrument</b> te voorkomen.
P	<i>Caution (Atenção)</i> Significa que a instrução referida tem de ser respeitada para evitar a danificação do aparelho.

WARNING	We use the term WARNING to inform you about situations that could result in personal injury to yourself or other persons. Details about these circumstances are in a box like this one.
	<b>Warning (Warnung)</b> Bedeutet, daß es bei Nichtbeachten der genannten Anweisung zu einer Verletzung des Benutzers kommen kann.
DK	<b>Warning (Advarsel)</b> Betyder, at brugeren kan blive kvæstet, hvis anvisningen ikke overholdes.
E	<i>Warning (Peligro)</i> <i>Utilizamos el término WARNING (PELIGRO) para informarle sobre</i> <i>situaciones que pueden provocar daños personales a usted o a otras</i> <i>personas. En los recuadros como éste se proporciona información sobre</i> <i>este tipo de circunstancias.</i>
F	<i>Warning (Danger)</i> Nous utilisons la formule WARNING (DANGER) pour avertir des situations pouvant occasionner des dommages corporels à l'utilisateur ou à d'autres personnes. Les détails sur ces circonstances sont données dans un encadré semblable à celui ci.
	<i>Warning (Pericolo)</i> Con il termine WARNING (PERICOLO) vengono segnalate situazioni che potrebbero provocare incidenti alle persone. Troverete informazioni su tali circostanze in un riquadro come questo.
NL	<b>Warning (Waarschuwing)</b> Betekent dat, wanneer de genoemde aanwijzing niet in acht wordt genomen, dit kan leiden tot verwondingen van de gebruiker.
P	<b>Warning (Aviso)</b> Significa que a não observância da instrução referida poderá causar um ferimento ao usuário.

## Warning Signs on the Instrument



Caution, hot surface.

Attention surface chaude.



Caution, risk of electric shock.

Attention, risque d'électrocution.



#### Caution

Documentation must be consulted to determine the nature of the potential hazard and any actions which have to be taken.

Attention La documentation doit être consultée pour déterminer La nature du risque potentiel et des actions qui doit être pris.



Caution – Cold surface

Attention, Surface froide

The following additional graphic symbols used on the instrument:

$\sim$	Indicates alternating current
ŧ	Indicates the primary protective grounding terminal
0	Indicates the <i>off</i> position of the main power switch
	Indicates the <i>on</i> position of the main power switch

### Electrical Warnings





*Do not operate the instrument with any covers or parts removed.* 

*Ne faites pas fonctionner l'instrument avec des couvercles ou des pièces retirés.* 



Do not attempt to make adjustments, replacements, or repairs to this instrument except as described in this help file. Only a PerkinElmer service representative should be permitted to service the instrument.

N'essayez pas d'effectuer des ajustements, des remplacements ou des réparations à cet instrument, sauf comme décrit dans ce fichier d'aide. Seul un représentant du service PerkinElmer devrait être autorisé à réparer l'instrument.



Use only fuses with the required current rating and of the specified type for replacement.

N'utilisez que des fusibles avec la note de courant requise et du type spécifié pour le remplacement.

### General Warning

The CLN2 system of PerkinElmer's Differential Scanning Calorimeter emits a small amount of nitrogen during normal operations. If your laboratory is not properly ventilated, the level of oxygen in the laboratory may fall below the normal range. Please follow the applicable laboratory ventilation standards to ensure that an appropriate oxygen level is maintained.



### Europe

All information concerning EMC standards is in the Declaration of Conformity, and these standards may change as the European Union adds new requirements.

PerkinElmer instruments have been designed and manufactured, having regard to the state of the art, to ensure that:

- the electromagnetic disturbance generated does not exceed the level above which radio and telecommunications equipment or other equipment cannot operate as intended;
- it has a level of immunity to the electromagnetic disturbance to be expected in its intended use which allows it to operate without unacceptable degradation of its intended use.

### South Korea

This device complies with MSIP (Ministry Of Science, ICT, and Future Planning) EMC Registration requirements. This instrument is registered as a Class A instrument for buiness use only. Product seller and user should notice that this equipment is not for house hold use.

A급 기기 (업무용 방송통신기자재) 이 기기는 업무용(A급) 전자파적합기기로서 판 매자 또는 사용자는 이 점을 주의하시기 바라 며, 가정외의 지역에서 사용하는 것을 목적으 로 합니다.

### United States (FCC)

United States (FCC) This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a business/industrial/commercial environment is likely to cause harmful interference in which the user will be required to correct the interference at your own expense. Changes or modifications not expressly approved by the manufacturer could void your authority to operate the equipment in compliance with FCC rules.

**NOTE:** Changes or modifications not expressly approved by PerkinElmer could cause the instrument to violate FCC (U.S. Federal Communications Commission) emission regulations, and because of this violation could void the user's authority to operate this equipment.

### Electrical Safety

This analyzer conforms to IEC publication 61010-1 ("Safety requirements for electrical equipment for measurement, control and laboratory use") as it applies to IEC Class 1 (earthed) appliances, and therefore meets the requirements of the Low Voltage Directive 2006/95/EC.

### Pollution Degree 2

This product will operate safely in environments that contain nonconductive foreign matter up to Pollution Degree 2 in EN/IEC 61010-1.

Normally only non-conductive POLLUTION occurs. Occasionally, however, a temporary conductivity caused by condensation must be expected.

### WEEE Instructions for PerkinElmer Products



A label with a crossed-out wheeled bin symbol and a rectangular bar indicates that the product is covered by the Waste Electrical and Electronic Equipment (WEEE) Directive and is not to be disposed of as unsorted municipal waste. Any products marked with this symbol must be collected separately, according to the regulatory guidelines in your area.

The objectives of this program are to preserve, protect and improve the quality of the environment, protect human health, and utilize natural resources prudently and rationally. Specific treatment of WEEE is indispensable in order to avoid the dispersion of pollutants into the recycled material or waste stream. Such treatment is the most effective means of protecting the customer's environment.

Requirements for waste collection, reuse, recycling, and recovery programs vary by regulatory authority at your location. Contact your local responsible body (e.g., your laboratory manager) or authorized representative for information regarding applicable disposal regulations. Contact PerkinElmer at the web site listed below for information specific to PerkinElmer products.

Web address:

www.perkinelmer.com/WEEE

For Customer Care telephone numbers select "Contact us" on the web page.

Products from other manufacturers may also form a part of your PerkinElmer system. These other producers are directly responsible for the collection and processing of their own waste products under the terms of the WEEE Directive. Please contact these producers directly before discarding any of their products.

Consult the PerkinElmer web site (above) for producer names and web addresses.

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# **Preparing Your Laboratory**

## Introduction

The following sections describe requirements for your Thermal Analysis System. Make sure your laboratory meets all of the requirements before you try to install the system. You should step through the topics in the order presented below.

Before starting installation, please read the Safety and Regulatory Information.

- Electrical Requirements
- Environmental Requirements
- Purge Gas and Pneumatic Supply Requirements
- Space Requirements

#### Power Source

An independent power source should be provided for the system, including the computer. The power source should not be associated with heavy-duty equipment such as large motors, or with possible sources of high-frequency interference such as photocopying systems, discharge lamps, or radio transmitters. The power supply should be fused at a maximum of 20 A (120 V systems) or 16 A (200–240 V systems).

### Line Voltage

PerkinElmer analyzers and their associated instruments are designed to operate within a line voltage range of 10% of the nameplate voltage or other such voltage selected at installation to suit the particular country or region. (The range for 240 V systems is +6%, -10%.) The supply must be smooth, clean, earthed and free of transient voltages over 40 V.

The frequency range is  $\pm 1\%$  for 50 Hz and 60 Hz systems.

Voltage	100 -240VAC
Power	400VA
Frequency	50/60Hz

### Environmental Requirements

You must provide the following laboratory conditions for your Thermal Analysis System:

- A clean area, free from vibration and strong magnetic fields.
- An adequate and stable power source for all system components.
- The area must have a relative humidity of 20–75% (without condensation).
- For optimum performance, the temperature of the area should be between 10 °C and 35 °C (50 °F and 95 °F).
- The instrument will operate safely between 5 °C and 40 °C.
- Place the system components in an area that is not in direct sunlight or direct contact with heating and cooling ducts or units.
- The instruments are for indoor use only.
- The storage temperature is between 20 °C and 60 °C.
- The altitude limitation for the operation of this instrument is 2000 m.
- The altitude for storage of this instrument is 0–12000 m.
- The installation overvoltage category for all instruments is Category II.
- The pollution degree is 2 for all instruments. (This product will operate safely in environments that contain nonconductive foreign matter up to Pollution Degree 2 in EN/IEC 61010-1.)
- The instrument must be positioned so that the appliance coupler can be removed to completely disconnect the power from the instrument.
- *NOTE:* If the equipment is used in a manner not specified by PerkinElmer, the protection provided by the equipment may be impaired.

## Purge Gas and Pneumatic Supply

The recommended purge gas for all Thermal Analyzers at ambient temperatures is argon or nitrogen with a minimum purity of 99.9%. Other gases, such as air or oxygen, may also be used. Air or oxygen is recommended for the purge gas when performing oxidation studies (DSC analyses).

The purge gas for any instrument must be dry. Use a size 1 A cylinder equipped with a pressure regulator that has a shutoff valve at the outlet. The shutoff valve should have 1/4-in. NPT male threads on the outlet side for connection to the analyzer's purge gas line.

## Space Requirements

Length (front to back) = 62 cm (24.25 inches).

Width including Wand Holders = 53 cm (21 inches).

Width without Wand Holders = 46 cm (18 inches).

Height = 30 cm (12 inches) without autosampler, 51 cm (20 inches) with autosampler.

Once all of the above requirements are met, you can install your analyzer.

Install Multiple Analyzers

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## Installing Multiple Analyzers

The only way to attach multiple instruments to your computer is via a universal serial bus. PerkinElmer no longer supports the Multiport RS-232 Card (P/N 09402018). Installation of the universal serial bus MUST be done before installing any analyzer.

A Pyris Series USB Multiport is an RS-232 module that uses the USB port on the PC and features plugand-play intelligent connectivity. The USBs supported by PerkinElmer are the EdgePort/4 (P/N 09402020) and EdgePort/8 (P/N 09402019). They eliminate the need to install cards into dedicated computer slots and reconfigure the system. Computers with standard USB allow peripherals to be automatically configured as soon as they are physically attached without the need to reboot or run setup.

**NOTE:** Some PerkinElmer analyzers now have USB ports in addition to, or sometimes in place of, RS-232 ports. If you are working with these analyzers, you will need to use a USB hub device instead of the Edgeport to connect multiple analyzers to a single computer. A suitable 7-port USB hub is available (*P/N* HH10151008). Contact your PerkinElmer Service Representative for further information

To install an EdgePort:

1. Attach one end of the USB cable to one of the USB connectors at the back of the computer.



2. Attach the other end of the connector cable to the USB port on the EdgePort.

EdgePort/4



EdgePort/8



#### DO NOT connect any instruments to the EdgePort at this time.

If your computer is connected to the Internet, it will now download the software drivers for the EdgePort. Once this process is completed, you can install the individual analyzers by connecting them to the RS-232 ports on the EdgePort and configuring them in the Pyris software.



# Installing a DSC 8500

# Safety Precuations for the DCS 8500



The following precautions must be observed when using the DSC 8500:

- Never press the Reset button on the computer if the software appears to malfunction. Press the CTRL–ALT–DEL keys simultaneously and select **Task Manager**. End the Pyris Software task.
- Always use helium as the purge gas when liquid nitrogen is used in the subambient reservoir of the DSC 8500. At other times, use nitrogen, argon, oxygen, or air as the purge gas.
- Do not immerse the purge gas exit line in a liquid since the liquid may be drawn back into the sample holder.
- Always observe the precautions indicated for Intracoolers 2 and 3 cooling devices.
- Always observe the precautions indicated for operating the DSC 8500 with the CryoFill Liquid Nitrogen Cooling System.
- When operating the DSC 8500 in ambient mode, make sure that the Cover Heater is OFF.
- Only high-quality purge gases should be used with DSC 8500. Minimum purity of 99.9% is recommended. A high-quality filter dryer accessory is recommended for the removal of any moisture from the purge gases.
- Always observe the startup or shutdown procedures for Diamond DSC and all related instruments.
- Do **NOT** use aluminum sample pans above 600 °C. Since aluminum melts at 660 °C, the pans will alloy with and destroy the sample holders. Entering a temperature above 600 °C will cause the computer to display a dialog box with a cautionary message. Always make sure that you are not using aluminum pans in either the sample of reference furnace before extending the Diamond DSC temperature range above 600 °C.
- Always encapsulate sample standards, indium, tin, lead, and zinc standards in aluminum or graphite pans. These metals will alloy with gold, copper, or platinum pans.
- It is important that nothing fall down into the cavity surrounding the sample holders. If this happens, turn off the power **IMMEDIATELY** and call your PerkinElmer's Service Representative for instructions.

- Do not force the platinum sample holder covers to fit the sample holders and do not deform the covers in any way. The sample holder covers should fit easily and loosely into the holders. When required, reform the covers with the reforming tool.
- The CLN2 system of PerkinElmer's Differential Scanning Calorimeter emits a small amount of nitrogen during normal operations. If your laboratory is not properly ventilated, the level of oxygen in the laboratory may fall below the normal range. Please follow the applicable laboratory ventilation standards to ensure that an appropriate oxygen level is maintained.





Risk of injury through lifting. The instrument weighs approximately 30 kg with the autosampler (20 kg without the autosampler). Improper lifting can cause painful and sometimes permanent back injury. Use proper lifting procedures and/or mechanical lifting aids to lift or move the instrument.

Risque de blessures par levage. L'instrument pèse environ 30 kg avec l'auto-échantillonneur (20 kg sans l'échantillonneur automatique). Un levage inadéquat peut causer des blessures douloureuses et parfois permanentes au dos. Utilisez des procédures de levage appropriées et / ou des dispositifs de levage mécanique pour soulever ou déplacer l'instrument. 26 . DSC 8500 Installation and Hardware Guide

## Warning Labels on the DSC 8500

#### Front of Instrument: On the furnace surround:





**Caution – Hot surface** The furnaces and the furnace surround may be hot.

Attention-surface chaude Les fours et l'enceinte peuvent être chauds.



Caution – Cold surface The furnaces and the furnace surround may be cold. Attention - Surface froide Les fours et l'enceinte peuvent être froids.



Front of Instrument (on the open front lid):

#### Side of Instrument:



#### Back of Instrument:

#### Avertissement

Pour la protection contre les risques d'incendie, remplacezle uniquement par le même type et l'indice de fusible.



#### Warning

Do not operate the instrument with any covers or parts removed. Disconnect power supply whenever cover is removed.

#### Avertissement

Ne faites pas fonctionner l'instrument avec des couvercles ou des pièces retirés. Débranchez l'alimentation électrique lorsque le cache est enlevé.

Warning For protection against fire hazard, replace only with the same type and rating of fuse.

#### Warning

Disconnect supply cord before opening. Grounding circuit continuity is vital for safe operation of equipment. Never operate equipment with grounding conductor disconnected.

#### Autosampler:



#### Avertissement

Débranchez le cordon d'alimentation avant d'ouvrir le circuit de mise à la terre. La continuité est essentielle pour un fonctionnement sûr des équipements. N'utilisez jamais d'équipement avec le conducteur de mise à la terre déconnecté.

# **CAUTION** *The autosampler mechanism will activate during the instrument power up sequence regardless of the dust cover position.*

ATTENTION Le mécanisme d'échantillonnage automatique s'allume pendant la séquence de mise sous tension de l'instrument indépendamment de la position du cache-poussière.

# Installing a DSC 8500

The installation procedure for the DSC 8500 consists of the following steps:

- Prepare the Laboratory (refer to page 19)
- Unpack the DSC 8500
- Set Up the DSC 8500 System Components
- Connect the DSC 8500 System Components
- Configure the DSC 8500
- Optimize the Baseline for the DSC 8500
- Calibrate the DSC 8500

## Unpacking a DSC 8500

Each component of the DSC 8500 system is shipped in its own box:

- DSC 8500
- Personal Computer
- PYRIS Software for Window
- Printer (optional)
- CLN2 Cooling System (optional)
- Intracooler 2 or 3 (optional)
- Autosampler (optional)
- The circulating liquid chiller (optional)
- The Autosampler Dust Cover (optional)

The CLN2 Cooler System is an optional cooling system which allows the DSC 8500 to run in subambient mode, that is, at temperatures down to -180 °C.

The autosampler is an optional accessory that is used to automate the running of multiple samples. The sample tray holds up to 44 samples. Runs can be sequenced and post-run data analyzed by using a play list which is created using the Pyris Player software.

### Unpacking the DSC 8500

The DSC 8500 is shipped in a large box that contains the analyzer and a smaller box that contains the Spares Kit. If you have ordered the CLN2 Cooling System for your DSC 8500, there will be additional boxes for the accessories ordered.

- 1. Cut the straps around the box and open up the cardboard box.
- 2. Remove the two foam blocks to gain access to the analyzer.
- 3. The DSC 8500 weighs approximately 20 kg (44 lb). Two people are needed to lift it from the carton.



Use proper lifting posture when taking the instrument out of the shipping carton base. Make sure you bend your knees when lifting and securely hold on to the instrument as you lift.

*Utilisez une position de levage appropriée lorsque vous retirez l'instrument de la base du carton d'expédition. Assurez-vous de plier vos genoux lors du levage et de maintenir solidement l'instrument lorsque vous soulevez.* 

- 4. Place the analyzer carefully on the prepared bench.
- 5. Remove the remaining parts to the DSC 8500 and place them nearby. You will need these parts for the installation of the analyzer.
- 6. If you have purchased the CLN2 Cooling System for your DSC 8500, remove the parts from their box(es) and place them on a bench nearby.
## Unpacking the DSC 8500 Autosampler

The DSC 8500 autosampler is shipped in its own box. Unpack the autosampler as follows:

- 1. Cut the straps around the box and open up the cardboard box.
- 2. Open the outer box, and remove the four corner blocks.
- 3. Open the inner box and remove the two foam blocks, Accessories kit, and small box to gain access to the autosampler.
- 4. Use caution to lift the autosampler by placing hands under the baseplate to lift it out of the box.
- 5. Place the autosampler carefully on the prepared bench.
- 6. Put the foam pieces back into the small box and close it up. Place the small box back into the large box and put the foam corner pieces around it. Close the large box up and store in a safe place.

The procedure for unpacking the DSC 8500 is complete.

## Setting up the System Components for the DSC 8500

Place the DSC 8500 on your laboratory bench near the computer. Be sure the air intake and exhaust at the rear of the analyzer are not obstructed in any way. Do not plug in the power cord until instructed.

Be sure to set up all of the system components listed below before switching on the system.

- If required, select the correct voltage.
- Install the purge gases.
- Install the cooling devices.

If you purchased a DSC 8500 Autosampler, it must be installed by a PerkinElmer Service Engineer.

### Select the Correct Voltage for the DSC 8500

The DSC 8500 has a self selecting voltage system. It can be used throughout the range of 100 V–240 V.

### Install the Purge Gases in the DSC 8500

The DSC 8500 uses a rotary cover heater to eliminate frost build-up or condensation on the sample holder when the sample holder enclosure cover is opened, while samples are changed. The motor-driven covers are activated manually through the OPEN/CLOSE button on the front of the unit or through the instrument software.

The DSC 8500 has a built in mass-flow controller. The gas connectors are on the manifold on the rear of the instrument. The nitrogen purge gas lines are connected to the threaded tube connectors System Purge and Gas A. A third line connected to Gas B is optional.

The minimum pressure for the System Purge is 30 psi (207 kPa) and the maximum pressure is 45 psi (310 kPa). The minimum pressure on the Gas A and Gas B lines is 30 psi and the maximum pressure is 45 psi.

Two purge gas lines are used to purge the entire sample area. The furnace purge gas is split so that both the sample and reference furnaces are purged at the user-defined flow rate. We recommend using flow rates of 20–30 ml/min for your purge gas.

**NOTE:** Please note that the gas default setting is Gas A.

For the DSC 8500 the appropriate purge gases are nitrogen, helium or argon. The gas used in the system purge is usually nitrogen or argon.

CAUTIONAny other gases, other than nitrogen, helium or argon are used only at<br/>the discretion (and warranty risk) of the customer.ATTENTIONTous les autres gaz, autres que l'azote, l'hélium ou l'argon, ne sont<br/>utilisés qu'à la discrétion (et au risque de garantie) du client.

**NOTE:** Gas A should have nitrogen or argon connected. Gas B is for all gases. Reactive gases such as oxygen should be attached to Gas B. Helium should be attached to Gas B

All gases must have a filter/dryer in line.

**NOTE:** If the furnace cover is closed, at 30 psi, the total gas consumption will be 500 ml/min + the furnace purge gas flow rate. If the furnace cover is open, the total gas consumption will be 8000 ml/min + the furnace gas flow rate.



#### Diagram of the internal gas line layout

### Setting Up the Gas Supplies

This procedure is used to set up the purge gas cylinders with suitable regulators so that they can be connected to the purge gas line fittings used with DSC analyzers. We recommend the use of two gas cylinders for the DSC 8500, one is for the system purge gas and the other for the sample purge gas. Please note that the 859 kPa (125 psi) is maximum pressure to the filter/dryer.

- 1. Connect suitable regulators to the gas supplies that will be used to purge the system. The regulators should be equipped with shutoff valves at the outlet. The shutoff valves should have a 1/4-in. NPT male thread on the outlet side.
- 2. Wrap the threads of the shutoff valve fitting twice around clockwise with Teflon tape. Be sure to start wrapping one or two threads in from the end of the fitting so that the full width of the tape is on the threads.

3. Connect a reducing bushing to the taped shutoff valve fitting.



4. Remove the nuts from the 1/8-in. NPT male connector, then wrap the threads of the connector which will go into the bushing (installed in step 3 above) with Teflon tape, as described in step 2 above.

Do not wrap the threads of the 1/8-in. Swagelok side of the male connector.

5. Connect the taped end of the 1/8-in. NPT male connector to the reducing bushing.



6. Connect the 1/8-in. copper tubing (P/N N2410170) and nut to the end of the NPT male connector on the gas cylinder to be used for the sample purge



7. Repeat steps 1–7 to prepare the gas cylinder to be used for the AirShield purge using 1/4-in. Tygon tubing. Prepare the tank for the autosampler pneumatics and connect the green 1/4-in. tubing supplied with the autosampler.

You are now ready to connect the purge gas line to your analyzer.

### Connecting the Purge Lines

Perform this procedure to connect your sample purge gas line and the system purge line to the DSC 8500. Assuming all the other components of the purge gas lines are installed, you will now connect the 1/8-in. copper tubing from each gas cylinder to the back of the

DSC 8500 using female connectors.



- 1. Connect the 1/8-in. copper tubing and nut to the connector on the back of the analyzer labeled **Gas A**, **Gas B or System Purge**.
- 2. Connect copper tubing to the **Purge Gas Exhaust** connector and direct it to a fume hood. (This is optional and supplied by the customer.)

The purge gas installation is complete.

### Installing the Cooling Devices for the DSC 8500

There are several types of cooling devices available for use with the DSC 8500. The temperature range needed for your experiments will determine the coolant or cooling accessory you should use.

CAUTION	We recommend that you do not operate your DSC above 200 °C without using a cooling device.	
ATTENTION	Nous vous recommandons de ne pas utiliser votre DSC au-dessus de 200 ° C sans utiliser de dispositif de refroidissement.	

#### Intracooler 2:

The Intracooler 2 is a dual-stage, closed-loop circulating heat exchanger that automatically maintains a constant sample holder temperature of -65 °C.

#### Intracooler 3:

The Intracooler 3 is a three stage, closed-loop circulating heat exchanger that automatically maintains a constant sample holder temperature of -110 °C.

Intracooler 2	N5340039 (100 V, 50/60 Hz) N5340040 (120 V, 60 Hz)	
	N5340041 (230 V, 50 Hz)	
Intracooler 3	N5340043 (120 V, 60 Hz)	
	N5340044 (230 V, 50 Hz)	

#### CLN2 Cooler:

The CLN2 Cooler is a closed loop nitrogen chiller that automatically maintains a constant sample holder temperature (P/N N5340045 [100 V, 50/60 Hz] and P/N N5340046 [110–230 V, 50/60 Hz]).

#### Water Circulator:

The water circulator used is a Polyscience Chiller (P/N N5370220/1).

CAUTION	There are several precautions that must be followed before operating the analyzer with liquid nitrogen. You may want to print the precautions and display them by the analyzer for future use.
ATTENTION	Plusieurs précautions doivent être suivies avant d'utiliser l'analyseur avec de l'azote liquide. Vous pouvez imprimer les précautions et les afficher par l'analyseur pour une utilisation future.

The CLN2 system of PerkinElmer's Differential Scanning Calorimeter emits a small amount of nitrogen during normal operations. If your laboratory is not properly ventilated, the level of oxygen in the laboratory may fall below the normal range. Please follow your applicable laboratory ventilation standards to ensure that an appropriate oxygen level is maintained.



### Installing the CLN2 Cooler into the DSC 8500

Before installing the CLN2 cooler see the following procedures in the Help:

- Preparing for Liquid Nitrogen Operation
- Operating with Liquid Nitrogen

#### Before Installing a CLN2 Cooler

1. Place the CLN2 on the floor to the left of the bench where the DSC 8500 is located. Make sure that there is a 4-in. clearance near the vents of the Intracooler to provide air for cooling the unit.

For best results, the unit should be operated at a normal ambient temperature of 22 °C (74 °F). Higher temperature ambients will not permit the unit to reach its maximum low temperature. Never operate the CLN2 in temperatures above 32 °C (90 °F).

2. Shut down the DSC 8500 system and remove the line power.

To install a CLN2 cooler into the DSC 8500, follow the procedure below.

#### CLN2 Cooler Installation

- 1. Flip down the front looks cover to reveal the ambient plate.
- 2. Remove the four screws that hold the plate in place.



- 3. For the CLN2 cooler disconnect the inline connector located in the bottom left cavity. See the image below.
- 4. Remove the insulation. Please note that different coolers will have different types of insulation and the color can vary. See the image below.



#### Insulation

- 5. Loosen the four screws that connect the transfer line to the clamping collar. See the image below.
- 6. Have the DSC 8500 on the edge of the bench and carefully push it over the edge to gain access to the hole at the base. Through that access hole use a 3/4 inch socket wrench to loosen the bolt. See the image below.



7. Remove the clamping collar and remove the transfer line. See the image below.

- 8. Install the CLN2 cooler.
- 9. Before connecting up, apply a thin layer of Heatsink Compound (P/N 09987122) to the upper face of the expansion chamber, using the Grease Applicator tool (P/N N5347010). Then place the Insulator (P/N 03191720) on top of the expansion chamber and repeat the application of the Heatsink Compound to the Insulator.
- 10. Tighten the all screws and the expansion chamber securing bolt.
- 11. Return the insulation. Make sure that when you return the insulation clips go along the floor of the heat exchanger cavity and does not get stuck at the edge of the cavity. See the image below.



#### Insulation earthing clips

- 12. Return the ambient plate and flip the looks cover back into place.
- 13. Turn the DSC 8500 back on and allow the system to cool down and stabilize. This should take about one hour.

#### Reconnecting the DSC 8500 after CLN2 Cooler Installation

- 1. Plug in the line power for the DSC 8500.
- 2. Make sure that the REFRIGERATION switch on the CLN2 cooler is in the OFF (down) position. Plug the cooler into the mains power supply.
- 3. Set the purge gas flow rate to 20 psi (at the regulator).

This ends the procedure for installing the CLN2 cooler.

### Installing the Intracoolers 2 and 3 for the DSC 8500

To install an Intracooler 2 or 3 into the DSC 8500, follow the procedure below.

#### Before Installing an Intracooler

1. Place the Intracooler on the floor to the left of the bench where the DSC 8500 is located. Make sure that there is a 4-in. clearance near the vents of the Intracooler to provide air for cooling the unit.

For best results, the unit should be operated at a normal ambient temperature of 22 °C (74 °F). Higher temperature ambients will not permit the unit to reach its maximum low temperature. Operation above 35 °C (95 °F) is not recommended, and may damage the Intracooler.

2. Shut down the DSC 8500 system and remove the line power.

#### Intracooler Installation

- 1. Flip down the front looks cover to reveal the ambient plate.
- 2. Remove the four screws that hold the plate in place. See the following image.



3. For the Intercoolers 2 or 3 disconnect the inline electrical connector located in the bottom left cavity. See the following image..

4. Remove the insulation. Please note that different coolers will have different types of insulation and the color can vary. See the following image.



Insulation

- 5. Loosen the four screws that connect the transfer line to the clamping collar. See the following image.
- 6. Move the DSC 8500 to the edge of the bench and carefully push it over the edge to gain access to the hole at the base. Through that access hole use a 3/4 inch socket wrench to loosen the bolt and release the expansion chamber. See the following image.
- 7. Loosen and remove the clamping collar; then remove the transfer line. See the following image.



- 8. Install the Intracooler 2 or Intracooler 3.
- 9. Before connecting up, apply a thin layer of Heatsink Compound (P/N 09987122) to the upper face of the expansion chamber, using the Grease Applicator tool (P/N N5347010). Then place the Insulator (P/N 03191720) on top of the expansion chamber and repeat the application of the Heatsink Compound to the Insulator

Securing bolt

- 10. Tighten all the screws and the expansion chamber securing bolt.
- 11. Return the insulation. Make sure that when you return the insulation earthing clips go along the floor of the heat exchanger cavity and does not get stuck at the edge of the cavity. See the following image.



### Insulation earthing clips

- 12. Return the ambient plate and flip the looks cover back into place.
- 13. Turn the DSC 8500 back on and allow the system to cool down and stabilize. This should take about one hour.

#### Reconnecting the DSC 8500 after Intracooler Installation

- 1. Plug in the line power for the DSC 8500.
- 2. Make sure that the switch on the Intracooler is in the OFF (down) position. Plug the Intracooler into the mains power supply.

This ends the procedure for installing the intracooler.

## Installing a DSC 8500 Autosampler

Your DSC 8500 Autosampler is designed to be installed by a specially trained PerkinElmer Service Engineer. Do not try to perform this installation procedure.

## Connecting the System Components for the DSC 8500

The computer and optional printer should be connected together and the software already installed. To complete the connection of the system components, the DSC 8500 has to be connected to the computer. Follow the steps below to connect the DSC 8500 to the computer.



Follow the diagrams below showing the connections:



#### **Ethernet Connections for the DSC 8500**

Directly from Computer to Instrument:

- 12 foot (4 meter) crossover cable
- Instrument IP Address

Directly from Computer to multiple instruments:

- Standard ethernet cable from Computer to Switch Box
- Standard ethernet cables from Switch Box to Instruments
- Instrument IP Addresses

From Computer to Instrument via LAN:

- Standard ethernet cable from Computer to LAN
- Standard ethernet cable from LAN to Instrument
- LAN IP Address
- **NOTE:** The computer has an extra Network Interface Card so the computer can simultaneously communicate with the LAN and the instrument.
- **NOTE:** Double check the ethernet cable. Instrument(s) wait until the instrument and autosampler initialize completely. Turn on the purge gases.
  - 1. **Print this procedure** before switching off the computer, then follow the steps provided to make all connections.
  - 2. Connect one end of the ethernet cable to the port on the back of the DSC 8500 labeled Ethernet connection.
  - 3. Connect the other end of the cable to the first available communications port on the back of the computer.
  - 4. Plug in all power cords.
  - 5. If your PerkinElmer Service Engineer installed your autosampler, and they will connect the other end of the cable (P/N N5340203) from the autosampler to the DSC 8500.
  - 6. Once the system connections are met, switch on the system in the following order:
    - Computer
    - PYRIS software
    - Printer
  - 7. Begin cooling the analyzer.

## Configuring the DSC 8500

- 1. The Pyris software must be configured for the analyzer you are using. This must be done before the DSC 8500 Application can be recognized.
- 2. For details on how to configure your DSC 8500, refer to the software Help.

## Optimizing the Baseline for the DSC 8500

To optimize the baseline for the DSC 8500, refer to the software Help.

## Calibrating the DSC 8500

Calibration of the DSC for temperature and energy is accomplished by running high-purity standard and reference materials with known temperature and energy transitions. (PerkinElmer ships reference materials in the Spares kit with each analyzer.) The data obtained after running these materials are used in the calibration programs to automatically calibrate the DSC. Once the calibration is performed, the analyzer will be calibrated continuously, even when the system is turned off. Unless major analyzer condition changes are made, the DSC should remain calibrated.

### Calibration Precautions

The following precautions must be observed when the calibrating a DSC:

- Do not use aluminum pans above 600 °C since they will alloy with and contaminate the sample holders.
- All metal reference materials (indium, tin, lead, zinc) must be encapsulated in aluminum pans because they will alloy with gold or platinum pans.
- All inorganic reference materials (potassium sulfate, potassium chromate) should be encapsulated in gold pans.
- For the best temperature repeatability, the reference material should be flattened and placed in the center of the sample pan and then encapsulated. The sample pan should be placed in the center of the sample holder.
- When using subambient reference materials, it is recommended that they be encapsulated in volatile sample pans or autosampler pans for a tight, hermetic seal (P/N 0219-0062 and 0219-0080 and volatile sample sealer 02190061).
- When using liquid nitrogen or an Intracooler, always observe the precautions for liquid nitrogen use.

## When to Calibrate the DSC

Once you have performed the automatic calibration programs, the temperature and heat flow (energy) calibration of the DSC should remain unchanged for an extended period, provided there are no changes in the instrument operating conditions. (Periodic checks of the calibration typically show variations of <1% after many days.) There are a few conditions that will change either the temperature or the energy calibration and cause the need to recalibrate:

- If the operating temperature range of your experiments changes, recalibration may be necessary. Run a reference material in the new range to determine if the current calibration is still valid.
- If the Balance is adjusted, recalibration may be necessary.
- If the purge gas type or purge gas flow rate is changed, the calibration should be checked for highest accuracy.
- If the coolant or cooling accessory used with the DSC is changed, recalibration may be necessary.

- Since temperature calibration depends slightly on scan rate, final calibration should be performed at the scan rate that you will use for your experiments. As the scan rate is increased from the rate used for calibration, the transition temperature may change slightly. If you will be using a wide range of heating rates, or heating and cooling rates, it is better to calibrate at the slowest rate to be used.
- If the instrument has been turned off for a long time (that is, weeks or months), the instrument may appear to need recalibration. Condition the DSC by performing several heating and cooling runs with the sample holder empty and then check the calibration by running reference materials.

### DSC Calibration Routines

For information on calibrating the DSC see the software Help.

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# DSC 8500 Hardware

54. DSC 8500 Installation and Hardware Guide

## Overview of the DSC 8500

The DSC 8500 is a power-compensated differential scanning calorimeter. In power compensation DSC, the sample and reference material are each confined to a separate, self- contained calorimeter. In addition to the traditional benefits of the power compensation design, the DSC 8500 includes improved baseline and subambient performance, lower baseline noise, and higher measurement sensitivity. The DSC 8500 is connected directly to the computer containing Pyris software which controls the analyzer via temperature control programs. Using Pyris software, you can program the DSC 8500 to run from an initial to a final temperature through transitions in the sample material such as melting, glass transitions, solid-state transitions, or crystallization. The temperature range is scanned by changing the temperature at a linear rate in order to study these endothermic and exothermic reactions of samples.

Pyris software supports the DSC 8500 autosampler accessory. The autosampler allows testing of up to 96 samples. The autosampler is useful in laboratories that analyze many samples of the same or different types. Combined with the Pyris Player software, the runs are entirely automated. You can create "play lists" that contain multiple methods for analyzing one sample; multiple samples to be analyzed by one method, and many ways to perform post-run data analysis.

Other features of the DSC 8500 are:

- Platinum resistance thermometers, not thermocouples, are used for linear temperature measurement.
- Subambient to cryogenic temperatures are achieved through the use of a combined, vacuum jacketed, liquid nitrogen transfer line and heat exchanger assembly. The transfer line and heat exchanger assembly is attached to the bottom of the sample holder assembly in a way common to each DSC 8500 cooling accessory. Access to the attachment area is gained by opening the front hinged looks cover, removing the chassis front plate and the front section of the insulation piece if installed. Once the heat exchanger is installed the transfer line portion of the assembly is securely attached to the instrument internal chassis via a convenient clamp. The transfer line portion of the assembly that simultaneously transports liquid nitrogen droplets to the heat exchanger and removes nitrogen exhaust. The line remains free of frost or condensation and is safe to touch in operation.

The features of the DSC 8500 and autosampler are covered in the following topics:

- Safety Precautions
- Operating Variables and Sample Handling
- Subambient Operation
- DSC 8500 Sample Holder
- Maintenance
- Accessories
- Part Numbers
- Autosampler

## DSC 8500 Sample Holder

The DSC 8500 sample holder is on the top of the instrument and sits underneath the sample holder enclosure cover.



### Platinum - Iridium Sample Furnace

There are two low-mass platinum-iridium sample cells (furnaces) embedded in a large aluminum heatsink. The left furnace is used for encapsulated sample materials; the right furnace is used for reference materials. For example, the right furnace is typically an empty sample pan and lid or an empty sample container of the type used in the sample cell.

### Platinum Sample Holder Lid with Vent

Vented platinum lids are used to cover both the sample and the reference furnaces. These lids should be similarly oriented when they are placed in their respective furnace. The lids should fit snugly into the furnace but should not be forced. If a lid is bent or deformed, use the Sample Holder Cover Reforming Tool (P/N 03190030) to reform the lids. Whenever sample lids or sample materials are removed or placed into the instrument, make sure that the guard ring inserts are in place.

#### Sample Holder Cover Reforming Tool (P/N 0319-0030)

DSC performance is enhanced with sample holder covers that are formed properly and are not degraded by extended usage. This tool allows the analyst to reform the platinum covers to their original dimensions.

## DSC 8500 Operating Variables and Sampling Handling

In differential scanning calorimetry, the object of an experiment is to record the heat flow to or from a sample over a linearly changing temperature range or at a single isothermal temperature. The goal is to obtain accurate data in a minimum period of time. The following topics illustrate some of the techniques that can be used to obtain the most accurate DSC data in a minimum amount of time.

- Sample Preparation
- Sample Pans
- Sample Encapsulation
- Sample Size
- Sample Atmosphere
- Temperature Range
- Scanning Rate
- Sample Loading
- Specific Heat Analysis

## Sample Atmosphere for the DSC 8500

CAUTION	Always use helium as the purge gas when liquid nitrogen is used for subambient operation. Never leave the purge gas exit line immersed in a liquid.
ATTENTION	Utilisez toujours de l'hélium comme gaz de purge lorsque de l'azote liquide est utilisé pour un fonctionnement sous-ambiant. Ne laissez jamais la conduite de sortie de gaz de purge immergée dans un liquide.

You can control the atmosphere in which the encapsulated sample is run by using a purge gas to displace or introduce reactive gases into the sample furnaces. Recommended purge gases are air, nitrogen, argon, oxygen, and helium. When changing from one purge gas to another, always check the temperature calibration and energy calibration. Flow rates of 20–30 ml/min of purge gas are recommended. These flow pressures will be realized by inlet purge gas rates of 35–145 psi.

Argon or nitrogen of 99.9% minimum purity is recommended for purging the sample holders when operating at ambient temperatures and above. The gas must be dry. A size 1A cylinder equipped with a suitable regulator is recommended. The regulator should be equipped with a shutoff valve at the outlet. The shutoff valve should have a 1/4-in. NPT male thread on the outlet side for connection to the DSC's purge gas line. A purge gas flow restrictor should not be placed in the line as this is contained in the purge gas system within the instrument. You may begin your experiment with nitrogen purge gas and switch to a second gas (air or oxygen) via the Pyris software.

For degradation studies or for experiments on samples that offgas during a transition or reaction, a Flow Through Cover Kit is required on the DSC 8500. The Flow Through Cover permits the purge gas and volatiles to exit directly through the top of the DSC 8500 cover. The recommended flow rates of the purge gas are 40–50 ml/min programmable through the Pyris software. These flow rates will be realized by inlet purge gas pressures of 40–50 psi. Air or oxygen may be used as the purge gas.

Argon and nitrogen are the recommended purge gases. Helium is the recommended purge gas for liquid nitrogen subambient operation. Recommended flow rates of the purge gas are 20–30 ml/min. These flow rates will be realized by inlet purge gas pressures of 20–30 psi.

The DSC 8500 temperature ranges are as follows when purging with helium:

No Cooler = 20 °C to 200 °C Water Circulator = -10 °C to 480 °C Intracooler 2 = -80 °C to 410 °C Intracooler 3 = -130 °C to 360 °C CLN2 = -180 °C to 500 °C

CAUTION	Do not use aluminum sample pans when programming the DSC to run above 600 °C. Since aluminum melts at 660 °C, the pans will alloy with and destroy the sample holders. Always make sure that you are not using aluminum pans in either the sample or the reference cell when you want the temperature of the DSC to exceed 600 °C.	
ATTENTION	N'utilisez pas de casseroles en aluminium lorsque vous programmez le DSC pour dépasser 600 ° C. Puisque l'aluminium fond à 660 ° C, les casseroles alligent et détruisent les supports d'échantillons. Assurez-vous toujours que vous n'utilisez pas de poêles en aluminium dans l'échantillon ou la cellule de référence lorsque vous souhaitez que la température du DSC dépasse 600 ° C.	

The temperature range for your analysis depends on the sample and the applications of your results. The DSC 8500 can be used to analyze samples from a temperature of -170 °C to 750 °C. Isothermal analyses also can be performed at any selected temperature in the range of the instrument.

## Sample Loading for the DSC 8500

Before loading the sample into the DSC 8500 sample holder, the sample must undergo sample preparation and sample encapsulation.

In addition to encapsulating the sample, it is recommended that a reference capsule be used in the reference furnace during the DSC experiment. The best reference material is an empty sample pan and lid of the same type in which the sample material is encapsulated.

Load the sample and reference pans into the DSC 8500 sample holder as follows:

- 1. Rotate the cover back until it stops.
- 2. Note the orientation of the platinum sample holder covers. Remove them from the sample holders with tweezers.
- 3. Using tweezers, place the encapsulated sample in the left sample holder and put an empty pan and cover (reference) in the right sample holder.
- 4. Replace the sample holder covers back on the sample holders, with the same orientation as before they were removed. Maintaining the sample holder covers' orientation improves the baseline repeatability, especially at high temperatures.
- 5. Rotate the sample holder enclosure cover and lock it into place.
- **NOTE:** Try to load sample and calibration materials into the DSC 8500 when the furnace temperatures are at room temperature or higher. If you must open the sample holder enclosure cover, make sure that the Block Purge is on. Do not keep the cover open for more than a minute since frost will form in front of the cleats and around the Block Purge. If this should occur, use the acid brush provided to sweep away any frost, making sure to keep it away from the sample holders.

## DSC 8500 Subambient Operation

The DSC 8500 can be operated in subambient mode. PerkinElmer offers subambient accessories for the DSC 8500 to satisfy most temperature requirements. There are three cooling devices: Intracooler 2, Intracooler 3 and the CLN2 Cooler. These three options for DSC 8500 extend the temperature range down to -65 °C, -130 °C, and -170 °C, respectively. The sliding sample holder enclosure cover replaces the dry box on the DSC 8500. Subambient operation of DSC 8500 is comparable to ambient performance with respect to noise, resolution, baseline linearity, and repeatability.

To determine which subambient configuration you need, you must determine at which temperature you wish to operate.

Subambient operation of DSC 8500 is obtained by using either of the following modes:

- Intracoolers 2 and 3
- CLN2 Cooler

The CLN2 Cooling System must be installed initially by a PerkinElmer Service Representative. Subsequently, you can convert back to ambient operation or use an Intracooler.

## Maintenance of the DSC 8500

The DSC 8500 needs little routine maintenance other than proper treatment as a sensitive electromechanical device. Occasionally, the sample holder cups may become coated with sample residue and a cleaning procedure may be necessary. Use the clean routine on the control panel in an air environment. They can be wiped with a cotton swab moistened with isopropyl alcohol or an appropriate solvent.

### Cleaning and Decontamination

Before using any cleaning or decontamination methods except those specified by PerkinElmer, you should check with PerkinElmer that the proposed method will not damage the equipment.

The following topics contain information on maintaining your DSC 8500:

- Sample Holder Treatment
- Removing/Replacing the Sample Holder
- Cleaning the Sample Holder
- Cleaning the Sample Holder Cover
- Cleaning the Furnace
- Guard Ring Maintenance
- Wand Maintenance

The DSC 8500 has a wand pickup tool. The Vacupick 4 mm cup (Part No. N5341654) located at the end of the wand may need to be replaced.



If you notice that the wand no longer picks up the sample cups or lids contact your PerkinElmer Service representative to have that repaired.

## Cleaning of the DSC 8500 Sample Holder Cover

Over time the underside of the DSC 8500 sample holder cover, may need to be cleaned. Periodically check this area and, if necessary, clean it. Cleaning should be done at low temperatures whenever possible by using cotton swabs moistened with an appropriate solvent such as isopropyl alcohol. To clean the sample holder cover you must first remove the sample holder lid.

See the sample holder cover removal instructions below.

- 1. Remove the clear lid cover.
- 2. Press the button on the front of the DSC 8500 to open the sample holder cover.



3. Carefully remove the sample holder plate by following the lock/unlock symbols.



Lock/Unlock Symbols

4. Remove the plastic plate by removing the screw in the located in the center.



- 5. Both plates may need to be cleaned. Use cotton swabs moistened with an appropriate solvent such as isopropyl alcohol to clean the sample holder plate.
- 6. Once the sample holder plate is cleaned return the plastic plate, screw and sample holder plate.

### Accessories

Every day you can count on PerkinElmer to provide you with solutions that deliver reliable performance, control operating costs and maximize operational time. Our complete portfolio of consumables, parts, supplies, training and service helps you meet both routine and demanding measurement challenges. We invest heavily in testing and validating our products to ensure you receive guaranteed compatibility and performance-on-time, every time, for every instrument in your laboratory.

Supplies, replacement parts, and accessories can be ordered directly from PerkinElmer, using the part numbers quoted in the guides provided with the instrument.

See our website:

http://perkinelmer.com

PerkinElmer's catalog service offers a full selection of high-quality supplies.

To place an order for supplies and many replacement parts, request a free catalog, or ask for information:

If you are located within the U.S., call toll free 1-800-762-4000, 8 a.m. to 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.

If you are located outside of the U.S., call your local PerkinElmer sales or service office.

### DSC 8500 Part Numbers

#### Spares Kit (Part No. N5340519)

Part No.	Description	Quantity
02191268	Standard Sapphire Small	1
03190033	Indium DSC Calibration REF MAT	1
03190036	Zinc DSC Calibration Reference	1
04190299	Vent. Platinum Sample Covers	1
B0143263	Suction Manipulator (STD)	1
N5190762	Kit - DSC7 Sample Calibration	1
N5361078	Tweezers - PEARL	1
09908400	Tweezers - Flat Forceps	1
N5340140	Tool - Viper Wand Pickup	1
Part No.	Description	Quantity
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09988986	Linecord - 3C 18AW 2.29M 125V	1
09991629	Fuse - 3.15A 250V Metric SLO-BLO	2
09410061	Cable - Crossover CAT - 5E	1
N5370103	TA Filter Dryer Kit	1
02506515	Black Tygon Tubing	10
09903094	Insert	4
09903150	1/4 NPT MALE to 1/4 Tbg BRS	2
09903087	1/4 NPT MALE to 1/8 Tbg BRS	2
09903428	1/8 NPT MALE to 1/4 Tbg BRS	2
N5190462	Gas Split Kit	2
N2410170	Gas Line Assy (2) Copper	1

### CLN2 Cooling Accessory

Part No. N5340046 (100-240V, 50/60 Hz)

#### Intracooler 2

Part No. N5340039 (100 V, 50/60 Hz) Part No. N5340040 (120 V, 60 Hz) Part No. N5340041 (230 V, 50 Hz)

#### Intracooler 3

Part No. N5340043 (100–120 V, 60 Hz) Part No. N5340044 (220–230 V, 50 Hz) 68 . Product Title Hardware Guide

# DSC Autosampler

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The autosampler for the DSC 8500 is designed specifically for this instrument, and allows testing of up to 96 samples. Pyris software supports the DSC 8500 autosampler accessory. The autosampler is useful in laboratories that analyze many samples of the same or different types. Combined with the Pyris Player software, the runs are entirely automated. You can create "play lists" that contain multiple methods for analyzing one sample; multiple samples to be analyzed by one method, and many ways to perform post-run data analysis. The autosampler for the DSC 8500 also allows you to change reference pan during a run of multiple samples.

See the following topics for more information:

- How the autosampler works for the DSC 8500
- Warnings and Safety Information for the DSC 8500 autosampler
- Sample Handling
- Running a Play List
- Troubleshooting
- Maintenance

# How the Autosampler Works for the DSC 8500

# Working Space

The working space in the DSC 8500 autosampler is covered to protect the mechanical components and sample pans from dust. The transparent cover permits observation of the movements of the autosampler mechanism. An interlock can be activated using the Pyris software that pauses the autosampler if the cover is raised during operation. To restart the analysis, close the cover and press the AUTOSAMPLER PAUSE/RESUME button on the front of the analyzer.

# Sample Tray

The standard sample tray (P/N N5341583) holds 96 samples, arranged in an 8 x 12 array. The rows A–H and columns 1–12 are marked on the sample tray. The tray should lie flat on the sample mounting plate, located on the two pins.



Pyris automatically recognizes when a tray has been inserted. The Autosampler Status field will then display Tray inserted. If there is no tray inserted, the Autosampler Status field will display Tray missing. The sample tray can be removed, or samples loaded onto the tray while the analyzer (sample heating cycle) is running. To do this, press the PAUSE/RESUME button on the front of the analyzer. When you have replaced the tray and closed the cover, press the AUTOSAMPLER PAUSE/RESUME button on the front of the analyzer.



# Lid Positions

There are four positions on the sample tray mounting plate for sample and reference furnace lids: L1, L2, L3 and L4. Positions L1 and L2 are for sample furnace lids and positions L3 and L4 are for reference furnace lids.

There are two sizes of furnace lids. There is a standard furnace lid size (L1 and L3, sample and reference, respectively) and a larger diameter furnace lid size (L2 and L4, sample and reference, respectively). The larger diameter furnace lid is designed for use with tall pans.

## Trash Bin

A trash bin (P/N N5341586) is attached to the right side of the sample tray mounting plate. The Autosampler Control dialog box enables you to send unwanted sample or reference pans to the trash bin. You can also program the Pyris Player play list to send used samples to the trash bin.

## Autosampler Arm

The autosampler arm moves in three dimensions using three stepper motors. The X/Y-axes are used for positioning the pick-up tip over the sample tray or furnace area. The Z-axis is used for picking up the sample or reference pans and lids and unloading or loading them into the furnace.

The autosampler arm also functions as a sensor. During a typical work cycle, the autosampler uses the autosampler arm to check whether the sample tray position is empty and whether the furnace lid is positioned properly. An error is reported back to Pyris if there is a problem.

After a power failure, the autosampler will return to the home position.

# Autosampler Tip

The autosampler tip is secured magnetically to the sample arm, which allows for easy replacement.

The gold-plated brass autosampler tip is non- magnetic to aid the release of sample and reference pans.

If the autosampler tip becomes blocked, you can remove the tip and clean it with acetone. You can store additional autosampler tips (P/N N534-0185) in the holders on the left side of the sample tray mounting plate.



# Wand Pick-up Tool

The wand tool can be used to pick-up sample cups or lids. It uses the same vacuum pump as the autosampler, and so they cannot be used simultaneously. In normal operation, the wand is activated by picking up the tool, but if the autosampler is in operation the wand will not be activated until the Pyris player run is completed or paused, and the autosampler is in the home position. If the wand is in operation, the autosampler will pause until the wand is returned to its holder.

# Autosampler Start-up Procedure

When Pyris software starts, the Autosampler start-up procedure begins. You will be prompted to ensure that the system is ready for the procedure to run and asked if you wish to proceed with the start-up procedure. If you select **No**, you will still be able to use the autosampler.

The start-up procedure will take a few minutes, and includes a vacuum test. A progress bar provides information about the autosampler self-test. If an error occurs during the procedure, you will be asked if you would like to restart.

# Autosampler Alignment

The autosampler does not need to be aligned every time it is used. However, you should carry out an alignment if you change the accessory or sample tray.

To perform an alignment of the sample tray, sample lids and furnaces, click **Align Autosampler** on the Autosampler Control dialog box.

If an error occurs during the alignment, you will be asked if you would like to restart the procedure.

# Autosampler LED

The autosampler LED on the front of the analyzer provides information about the status of the instrument and autosampler.

LED display	Description
Solid Green	Ready for the next command.
Flashing Green	An action is in progress; for example, an alignment, picking up or replacing a pan, etc.
Solid Yellow	Paused. The Resume button on the front of the analyzer must be pressed to continue.
Flashing Yellow	Pause pending. The autosampler is under control by Pyris software.
Flashing Red	An error has occurred, but the autosampler can continue with the next action.
Solid Red	A severe error has occurred. The arm will return to the home position.

## Status Panel

If the Autosampler is selected as one of the parameters displayed in the Status Panel, it will display messages about the current status of the autosampler. Messages include: Empty sample tray, Tray missing, Tray inserted, Removing lid, Loading sample, Loading lid, Returning sample to tray, Pause and Ready.

# *Warnings and Safety Practices for the DSC 8500 Autosampler*



The following precautions must be observed when using the DSC 8500 Autosampler. These are in addition to the precautions that apply to the DSC 8500.



*Your DSC 8500 Autosampler is designed to be installed by a PerkinElmer Instruments Service Engineer. Do not try to perform any installation procedure beyond what is mentioned in this help file.* 

*Votre automateur DSC 8500 est conçu pour être installé par un ingénieur de service PerkinElmer Instruments. N'essayez pas d'effectuer une procédure d'installation au-delà de ce qui est mentionné dans ce fichier d'aide.* 



**AVERTISSEMENT** 

*Observe all local safety regulations when connecting the components of your DSC 8500 Autosampler system to each other and to the local electrical supply.* 

*Respectez toutes les règles de sécurité locales lors de la connexion des composants de votre système d'échantillonneur automatique DSC 8500 l'un à l'autre et à l'alimentation électrique locale.* 



*Observe all local safety regulations concerning the handling of gas cylinders when connecting the gas supply to the autosampler.* 

*Respectez toutes les réglementations de sécurité locales concernant la manipulation des bouteilles de gaz lors de la connexion de l'alimentation en gaz à l'automoteur.* 





CAUTION	<i>Never try to lift the DSC 8500 Autosampler by the autosampler arm as this could be damaged.</i>	
ATTENTION	N'essayez jamais de soulever l'échantillonneur automatique DSC 8500 par le bras auto-échantillonneur car cela pourrait être endommagé.	
CAUTION	Never try to lift the autosampler by the horizontal front panel. The tray could be damaged.	
ATTENTION	N'essayez jamais de soulever l'auto-échantillonneur par le panneau avant horizontal. Le plateau pourrait être endommagé.	
CAUTION	Make sure that all cables, tubes and tools are clear when lowering the autosampler cover down to be closed.	

# **ATTENTION** Assurez-vous que tous les câbles, tubes et outils sont clairs lors de l'abaissement du couvercle de l'automoteur pour être fermé.

# Sample Handling for the DSC 8500

Handling samples encompasses the following topics:

- Preparing Samples
- Loading samples in the DSC 8500
- Changing samples in the DSC 8500

## Preparing Samples for the DSC 8500 Autosampler

When preparing your samples for analysis in the DSC 8500 autosampler, take the following variables into consideration:

- Sample size
- Sample atmosphere
- Temperature range
- Scanning rate

In addition to these considerations, the autosampler has specific requirements for the choice of sample pan and cover and for the method of sample encapsulation.

#### Sample Pans

Sample pans and covers of various capacities and wall thicknesses are available from PerkinElmer. These pans and covers will resist the varying internal pressures of sample capsules without deformation.

CAUTION	Never touch the sample or the sample pan and cover with your bare fingers. Contamination from human body oils and greases can affect your results. Always use instrument wand, tweezers (Part Numbers: Flat Round Tip 09908400, or Flat Pointed Tip N5361078 or Hook 09908138) or the Standard Suction Manipulator (P/N B0143263) or the Special Suction Manipulator (P/N B0142512).
ATTENTION	Ne touchez jamais l'échantillon ou l'échantillon et couvrez-le avec vos doigts nus. La contamination des huiles corporelles et des graisses peut affecter vos résultats. Utilisez toujours la baguette instrumentale, les pinces (numéros de pièces: pointe plate plate 09908400 ou pointe plate à pointe N5361078 ou crochet 09908138) ou le manipulateur d'aspiration standard (P / N B0143263) ou le manipulateur d'aspiration spécial (P / N B0142512)

#### Sample Encapsulation

The Universal Crimper Press (P/N B0139005) is required for preparing samples for the autosampler.

## Loading Samples in the DSC 8500 Autosampler

Use the procedure below to load samples using the DSC 8500 autosampler and the Autosampler Control dialog box.

- **NOTE:** For details on how to run samples using the DSC 8500 and Pyris player, see Running a Play List in the DSC 8500 on page 80.
  - 1. Make sure that the analyzer is turned on.

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- 2. If you are using a purge gas, check that it is connected to the elbow connector properly.
- 3. Open the purge gas line and adjust the pressure.
- 4. Prepare your samples and references as described in Preparing Samples and Sample Preparation.
- Load your samples and references into the desired tray locations using the wand tool, tweezers, the Standard Suction Manipulator (P/N B0143263) or the Special Suction Manipulator (P/N B0142512).
- 6. Open the protective dust cover by lifting it up and back.
- 7. Place the furnace lids in positions L1, L2, L3 and L4, as required.
- 8. Insert the sample tray on to the sample tray mounting plate.
- **NOTE:** When the autosampler is in use, Pyris Player will not be able to run a method unless there is a tray in place.
  - 9. Close the protective dust cover.
  - 10. Start Pyris software for Windows and select the DSC 8500 analyzer.
  - 11. Click the Autosampler Control button in the control panel:
  - 12. In the Autosampler Control dialog box, click **Open Cover** to open the furnace cover.
  - 13. Enter the alphanumeric reference location and click Load Reference from Location.
  - 14. Select the appropriate radio button for the location of the reference lid (L3 or L4, standard or large) and then click **Load Reference Lid.**

**NOTE:** If you are going to perform high-precision specific heat, you must be very particular about the reference sample pan and the platinum lid used. You can use the Load Reference command in a play list to load a precisely measured reference sample pan from a specified position in the tray and then cover it with a precisely weighed platinum lid from the receptacle.

- 15. Enter the alphanumeric sample location and click **Load Sample from Location**.
- 16. Select the appropriate radio button for the location of the sample lid (L1 or L2, standard or large) and then click **Load Sample Lid**.

# Changing Samples in the DSC 8500 Autosampler

You may need to change samples in or add samples to the sample tray while a run is in progress. For example, you may want to run a play list that analyzes more than the maximum 96 samples (including reference) that the tray can hold. However, to prevent dust from entering the autosampler's working area, or if the DSC 8500 is in subambient mode, you should keep the protective cover closed as much as possible.

To change or add samples while a method step or a post-run data analysis step in the play list is being performed, you do not have to pause the play list. Use the AUTOSAMPLER PAUSE button on the front of the analyzer.

CAUTION	Do not try to change samples or add samples while the play list is executing a Load Sample, Return Sample, Load Reference, or Return Reference command.
ATTENTION	N'essayez pas de changer d'échantillons ou d'ajouter des échantillons pendant que la liste de lecture exécute une commande Load Sample, Return Sample, Load Reference ou Return Reference.

Use the following procedure to change samples in or add samples to the tray during a run:

- 1. Press the **AUTOSAMPLER PAUSE** button on the front of the analyzer.
- 2. Open the dust cover.
- 3. Remove the sample tray from the analyzer.
- 4. Insert a new tray, or replace the pans in the required tray positions and replace the tray.
- 5. Close the dust cover.
- 6. Press AUTOSAMPLER RESUME on the front of the analyzer.

**NOTE:** If you do not press RESUME, on completing the sample run the autosampler will remain paused.

# Running a Play List in the DSC 8500

Pyris Player is the backbone of Pyris Software for Windows automation. It was created with autosamplers in mind. In addition to the standard play list items - Load Sample, Run Method, Return Sample - there is a Sample Group. This simplifies grouping like samples together (as you would have in a sample tray of an autosampler) that use the same method and data analysis within a play list. A Sample Group consists of a Sample List and a Data Analysis List. A specific method is selected for the samples in the Sample List. The Data Analysis List allows you to access all necessary functions for data recall, curve manipulation, optimization and calculations for automatic data analysis. Display curve allows the last run data set to be recalled. Playback of the play list begins by clicking on the **Start** or **Start at Current Step** button on the Player toolbar. They are the two leftmost buttons on the Player toolbar:



*NOTE:* If you select the **Start at Current Step** button, the focused item must be a main- level item, that is, Prepare Sample, Data Analysis, or Sample Group. However, if in a Sample Group, you can start a play list if the current item is a Sample line.

Before starting a play list, perform the steps below:

- 1. Review the safety and warning notes in Warnings and Safety Practices.
- 2. Verify that the vacuum gas tubing for the autosampler and the DSC 8500 and the tubing for a cooling device, if applicable, are properly connected.
- 3. Verify that the electrical and cable connections between the autosampler and Pyris and all other cables are properly connected.
- 4. Turn on the purge gas for the DSC 8500; adjust the pressures.
- 5. Prepare your samples as described in Sample Preparation.
- 6. Load the sample and reference pans into the sample tray using the wand tool, tweezers, the Standard Suction Manipulator (P/N B0143263) or the Special Suction Manipulator (P/N B0142512).
- 7. Place the furnace lids in positions L1, L2, L3 and L4, as required.
- 8. Open the protective dust cover by lifting it up and back, and insert the sample tray on to the sample tray mounting plate.
- 9. Close the protective dust cover.
- 10. Start up Pyris software and either create a new play list or load an existing play list.
- 11. If using a subambient cooling device, follow the instructions in *Operating the DSC 8500 Using an Intracooler 2P or 3P* or *Operating the DSC 8500 Using Liquid Nitrogen*.
- 12. Start the play list. While the play list is running, you can, via the Pause Key in the Pyris Player:
  - a. Edit the entries in the play list that have not yet been executed.
  - b. View the progress of the run in the Instrument Viewer window.
  - c. Print a collapsed or expanded version of the play list or history.

- d. While in a Sample Group, you can continue to add, insert, or delete samples while the list if running, below the currently running line.
- **NOTE:** If you select Go To Load, Go To Temp, or Hold at Temp from the control panel while a play list is running, the current sample run will end and the playback of the play list ends.
- **NOTE:** If you are using the autosampler and it is paused while Pyris Player is running, Pyris Player will also pause until the Autosampler is resumed.
- **NOTE:** If **Use Initial Check (Playlist Only)** is selected on the Autosampler tab in the Preferences dialog box, the autosampler will check for the presence of pans in the sample and reference holders at the start of the run. Any pans or lids detected will be removed to the waste bin.

The View Play List page gives an expanded view of the Edit Play List page and provides a macro view of all the sequences.

The View Sample List page is a spreadsheet-style view of the samples of this play list. It includes all the sample information such as ID, method used, location, etc.

The View History page is a historical view of the completed steps of the play list and the Sample History page is a historical view of the samples run. It will notify you of all the successfully run commands, and any failed commands.

An example of a typical autosampler run cycle:

- 1. The temperature of the furnace goes to that set on the Autosampler tab of the Preference dialog box.
- 2. The furnace cover opens.
- 3. The autosampler removes the lid from the furnace and places it in the appropriate lid position.
- 4. The autosampler removes the pan from the furnace and places it in the appropriate sample tray location or the trash bin.
- 5. The furnace cover closes.
- 6. The autosampler returns to the home position.
- 7. The furnace cover opens.
- 8. The autosampler picks up next sample from the appropriate tray location and places it in the sample holder.
- 9. The autosampler picks up the appropriate lid from the lid position and places it on the holder.
- 10. The furnace cover closes.
- 11. The run starts.

# *Troubleshooting for the DSC 8500 Autosampler*

When there is a problem with the DSC 8500 Autosampler, a status message or code is displayed at the bottom of the Pyris window in the status bar and also in the status panel, if you have Autosampler Status selected for display. The error message will also appear in the History file of a play list run. The autosampler LED on the front of the analyzer also provides information about the status of the autosampler.

If an error occurs during the autosampler alignment procedure that causes the alignment to fail, an error message will be displayed, and the autosampler arm will return to the home position.

If an error, such as those listed below, occurs while you are running a play list, the play list will stop; you will have to restart the play list after resolving the problem. The History page can be used to determine what samples were run prior to the error.

To help resolve a problem causing the error message, the following topics give the possible causes and solutions for some of the more common errors that could occur while using the autosampler:

#### The autosampler is paused.

Possible Causes	Solutions
dust cover may have been opened to remove the sample	Check that the sample tray has been returned and that the autosampler cover is closed, and then press the <b>AUTOSAMPLER RESUME</b> button on the front of the analyzer.

#### The furnace rotary cover could not be closed.

Possible Causes	Solutions
There is an obstruction.	Check that the area near the furnace is free from obstruction. If this does not resolve the problem, contact your PerkinElmer Service Representative.

#### The furnace rotary cover could not be opened.

Possible Causes	Solutions
There may be a communication error.	Close the Pyris software. Turn off the analyzer/autosampler, and the switch it on again. Restart Pyris software and select the DSC 8500 from the Pyris Manager. If this procedure does not resolve the problem, contact your PerkinElmer Service Representative.

#### The sample/sample lid could not be found.

Possible Causes	Solutions
The sample pan or platinum cover is deformed or the sample pan cover was crimped asymmetrically, causing the arm to fail to pick up the pan.	When running a Pyris play list the play list will continue with the next sample, if it is loading a sample from the sample tray. To replace the sample: Open the protective cover and verify that the sample pan is deformed; replace it with a newly encapsulated sample. Close the protective cover. <b>NOTE:</b> See the Universal crimper for more information on how to crimp your samples. Pans with jagged edges may indicate that your crimper blades are blunt. Do not use deformed pans,
	or pans with jagged edges, with the autosampler. Restart the play list.
The location in the sample tray is empty.	When running a Pyris play list the play list will continue with the next sample.
The platinum lid is dropped when unloading a sample.	The autosampler will return to the home position. To replace the lid:
	Open the protective cover and replace the lid on the sample holder. Close the protective cover. Restart the play list.

#### Vacuum test failed/No vacuum/Low vacuum/Lost vacuum.

Possible Causes	Solutions
The autosampler tip is blocked.	Remove the tip and check for an obstruction. If necessary, replace with a new tip. The tip can be cleaned using acetone.
The filter inside the vacuum line (in the analyzer) is blocked.	Contact your PerkinElmer Service Representative.
The vacuum pump has failed.	Contact your PerkinElmer Service Representative.

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### Illegal contact during tip test or alignment.

Possible Causes	Solutions
There is an obstruction in the working space.	Check the working space for an obstruction. Ensure that the sample tray is kept free from tools, cables and loose samples.
	If there is no obstruction, contact your PerkinElmer Service Representative.

#### The sample tray is not located properly.

Possible Causes	Solution
The sample tray is not in position, or is not properly located on the locating pins.	Insert the sample tray, and ensure that it is aligned with the sensors.

#### The sample number is out of range.

Possible Causes	Solution
You have not entered an alphanumeric location reference, or a reference that is out of range, for Load Sample or Load Reference or for Tray Location field in the Autosampler Control dialog box.	Enter an alphanumeric number. The standard sample trays have rows A–H and columns 1–12.

# Maintenance for the DSC 8500 Autosampler

- The suction tip (P/N N5340185) on the autosampler arm may pick up debris over time. If it is dirty, it can be removed and cleaned with acetone.
- Clean the platinum lids with ether or alcohol on a cotton swab. Be sure that the lid is dry before using it again.

Observe the following guidelines when cleaning the autosampler:

- The exterior surfaces of the autosampler are resistant to diluted acids but not to concentrated acids.
- The protective cover is not resistant to organic solvents.
- To clean surfaces under the protective cover, that is, the working space, use only a cloth dampened with clear water to which a small amount of commercial household cleaning agent was added. Wipe these areas dry with a soft, dust-free cloth or tissue.
- With a medium soft brush, remove any dust that has accumulated in the folds of the robot arm bellow or in the sample tray holder.

# Cleaning and Decontamination

## Decontamination

Before using any cleaning or decontamination methods except those specified by PerkinElmer, users should check with PerkinElmer that the proposed method will not damage the equipment.

Customers wishing to return instrumentation and/or associated materials to PerkinElmer for repair, maintenance, warranty or trade-in purposes are advised that all returned goods must be certified as clean and free from contamination.

The customer's responsible body is required to follow the "Equipment Decontamination Procedure" and complete the "Certificate of Decontamination". These documents are available on the PerkinElmer public website:

http://www.perkinelmer.com/Content/technicalinfo/dts\_instrumentdeconprocedure.pdf

If you do not have access to the internet contact Customer Care:

Customer Care USA:	1-800-762-4000	(inside the USA)
(8:30 a.m. – 7 p.m. EST)	(+1) 203-925-4602	(outside the USA)
Customer Care Canada:	800-561-4646	
Customer Care EU:	0800 40 858	(Brussels)
	0800 90 66 42	(Monza)

If you are located outside of these regions, please call your local PerkinElmer sales office for more information.

# Cleaning the Instrument

Exterior surfaces may be cleaned with a soft cloth, dampened with a mild detergent and water solution. Do not use abrasive cleaners or solvents.

# General Laboratory Safety

Your laboratory should have all equipment ordinarily required for the safety of individuals working with chemicals (fire extinguishers, first-aid equipment, safety shower and eye-wash fountain, spill cleanup equipment, etc.).