

# DSC 4000 Installation and Hardware Guide



## Release History

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

User Assistance  
PerkinElmer, Inc.  
710 Bridgeport Avenue  
Shelton, Connecticut 06484-4794  
U.S.A.

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# **Introduction**

## ***Pyris Installation***

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**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 analyzer.

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

**Safety and Regulatory**  
**Information**

## ***Symbols Used in This Manual***

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

UPPERCASE text, for example ENTER or ALT, refers to keys on the computer 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 **serious damage to the instrument** or other equipment. Details about these circumstances are in a box like this one.

**Caution (Achtung)**

Bedeutet, daß die genannte Anleitung genau befolgt werden muß, um einen **Geräteschaden** zu vermeiden.

**Caution (Bemærk)**

Dette betyder, at den nævnte vejledning skal overholdes nøje for at undgå en **beskadigelse af apparatet**.

**Caution (Advertencia)**

Utilizamos el término **CAUTION (ADVERTENCIA)** para advertir sobre situaciones que pueden provocar **averías graves en este equipo** o en otros. En los recuadros como éste se proporciona información sobre este tipo de circunstancias.

**Caution (Attention)**

Nous utilisons le terme **CAUTION (ATTENTION)** pour signaler les situations susceptibles de provoquer de **graves détériorations de l'instrument** ou d'autre matériel. Les détails sur ces circonstances figurent dans un encadré semblable à celui-ci.

**Caution (Attenzione)**

Con il termine **CAUTION (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.

**Caution (Opgelet)**

Betekent dat de genoemde handleiding nauwkeurig moet worden opgevolgd, om **beschadiging van het instrument** te voorkomen.

**Caution (Atenção)**

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.



**Warning (Warnung)**

Bedeutet, daß es bei Nichtbeachten der genannten Anweisung zu einer **Verletzung** des Benutzers kommen kann.



**Warning (Advarsel)**

Betyder, at brugeren kan blive **kvæstet**, hvis anvisningen ikke overholdes.



**Warning (Peligro)**

Utilizamos el término **WARNING (PELIGRO)** para informarle sobre situaciones que pueden provocar **daños personales** a usted o a otras personas. En los recuadros como éste se proporciona información sobre este tipo de circunstancias.



**Warning (Danger)**

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.



**Warning (Pericolo)**

Con il termine **WARNING (PERICOLO)** vengono segnalate situazioni che potrebbero provocare **incidenti alle persone**. Troverete informazioni su tali circostanze in un riquadro come questo.



**Warning (Waarschuwing)**

Betekent dat, wanneer de genoemde aanwijzing niet in acht wordt genomen, dit kan leiden tot **verwondingen** van de gebruiker.



**Warning (Aviso)**

Significa que a não observância da instrução referida poderá causar um **ferimento** ao usuário.

## Symbols Used on the Instruments



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:

	Indicates alternating current
	Indicates the primary protective grounding terminal
	Indicates the <i>off</i> position of the main power switch
	Indicates the <i>on</i> position of the main power switch

## Electrical Warnings



**WARNING**

*Connect the instrument to an AC line power outlet that has a protective ground connection. To ensure satisfactory and safe operation of the instrument, it is essential that the protective ground conductor (the green/yellow lead) of the line power cord is connected to true electrical ground. Any interruption of the protective ground conductor, inside or outside the instrument, or disconnection of the protective ground terminal may impair the protection provided by the instrument.*



**AVERTISSEMENT**

*Connectez l'instrument à une prise de courant de ligne AC qui a une connexion de terre de protection. Pour assurer un fonctionnement satisfaisant et sécurisé de l'instrument, il est essentiel que le conducteur de terre de protection (le fil vert / jaune) du cordon d'alimentation de la ligne soit connecté à une vraie terre électrique. Toute interruption du conducteur de terre de protection, à l'intérieur ou à l'extérieur de l'instrument, ou la déconnexion de la borne de terre de protection peut nuire à la protection fournie par l'instrument.*



**WARNING**

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



**AVERTISSEMENT**

*Ne pas utiliser l'instrument avec des couvertures ou des pièces retirées.*



**WARNING**

*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.*



**AVERTISSEMENT**

*N'essayez pas de faire 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 servir l'instrument.*



**WARNING**

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



**AVERTISSEMENT**

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

## ***Electromagnetic Compatibility (EMC)***

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### ***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 B instrument for residential and/or business use.

B급 기기 (가정용 방송통신기자재)

이 기기는 가정용(B급) 전자파적합기기로서 주로 가정에서 사용하는 것을 목적으로 하며, 모든 지역에서 사용할 수 있습니다.

### ***United States (FCC)***

This equipment has been tested and found to comply with the limits for a Class B 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 residential environment may cause harmful interference in which the user will be required to correct the interference at their own expense.

**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***

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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**



or



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. Requirements for waste collection, reuse, recycling, and recovery programs vary by regulatory authority at your location. Contact your local responsible body (for example, 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:**

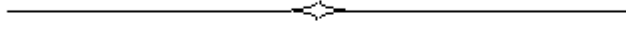
[www.perkinelmer.com/WEEE](http://www.perkinelmer.com/WEEE)

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.





# **Prepare the Laboratory**

## ***Prepare the Laboratory***

---

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

### ***Electrical 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.

#### ***Instrument Maximum Power Requirements***

Refer to the Safety Precautions for the instrument.

### ***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***

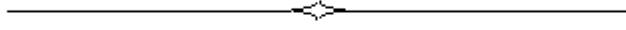
The recommended purge gas for all Thermal Analyzers at ambient temperatures is argon or nitrogen with a minimum purity of 99.9%. Other gases, such as air or oxygen, may also be used. Air or oxygen is recommended for the purge gas when performing oxidation studies (DSC analyses).

The purge gas for any instrument must be dry. Use a size 1A 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***

Refer to the Safety Precautions for the instrument.

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





# **Install Multiple Analyzers**

## ***Install Multiple Analyzers***

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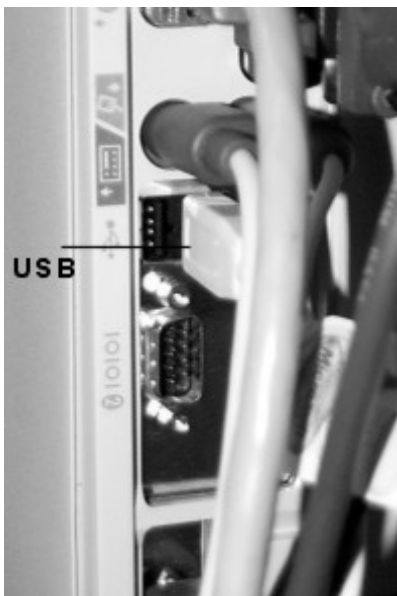
The only way to attach multiple instruments to your computer is via a universal serial bus. PerkinElmer no longer supports the Multiport RS-232 Card (P/N 09402018). Installation of the universal serial bus MUST be done before installing any analyzer.

A Pyris Series USB Multiport is an RS-232 module that uses the USB port on the PC and features plug-and-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:

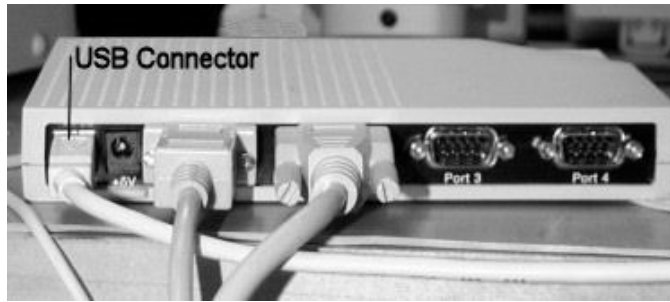
1. Attach one end of the USB cable to one of the USB connectors at the back of the computer.



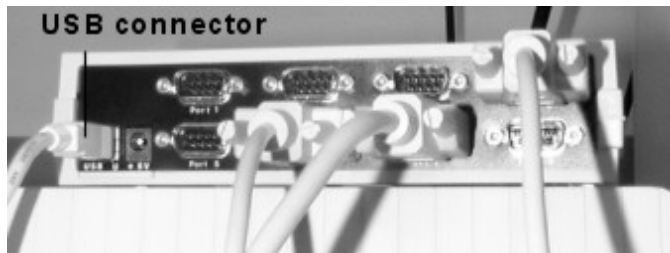


2. Attach the other end of the connector cable to the USB port on the EdgePort.

#### **EdgePort/4**

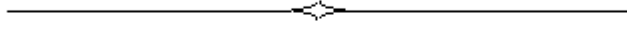


#### **EdgePort/8**



**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.



# **Install a DSC 4000**

## Safety Precautions for the DSC 4000



**WARNING**

*Be sure that all instrument operators read and understand the following precautions. It is advisable to post a copy of these precautions on or near the instrument itself.*



**AVERTISSEMENT**

*Assurez-vous que tous les opérateurs d'instruments lisent et comprennent les précautions suivantes. Il est conseillé de publier une copie de ces précautions sur ou près de l'instrument lui-même.*

The following precautions must be observed before and when using the DSC 4000 Differential Scanning Calorimeter:

- Before connecting the DSC 4000 to the main outlet, check the main voltage setting and fuse.
- The DSC 4000 requires a good earth ground that is common to the earth ground of the computer.
- Check that the power cord supplied is appropriate for your country and is undamaged before connecting it to the main voltage supply.
- Use proper lifting posture when lifting the analyzer. The DSC 4000 with autosampler weighs 19 kg. Lift the autosampler from underneath. Never attempt to lift the analyzer by the autosampler (if installed) or by any cables attached.
- When cleaning the instrument, consult PerkinElmer if there is any doubt about the compatibility of decontamination or cleaning agents with parts of the equipment or with material contained in it.



**WARNING**

*Never touch the outer and inner furnace lids. The temperature of the furnace can reach as high as 445 °C. Use tweezers to remove the lids.*



**AVERTISSEMENT**

*Ne touchez jamais les couvercles extérieurs et intérieurs du four. La température du four peut atteindre 445 °C. Utiliser une pince à épiler pour enlever les couvercles.*



**WARNING**

*Do not touch the inside of the furnace; it might be hot and internal pollution can cause loss of performance.*



**AVERTISSEMENT**

*Ne pas toucher l'intérieur du four; il pourrait être chaud et la pollution interne peut entraîner une perte de performance.*



**WARNING**

*Always ensure that there is adequate ventilation when operating the DSC 4000. Operate the DSC 4000 in a fume hood.*



**AVERTISSEMENT**

*Assurez-vous toujours qu'il existe une ventilation adéquate lorsque vous utilisez le DSC 4000. Faites fonctionner le DSC 4000 dans une hotte aspirante.*

**CAUTION**

*Do NOT expose the sensor surfaces to mechanical stress. If no external cooling is applied, do not operate the DSC 4000 above 50 °C. The DSC 4000 will automatically shut off when the inner temperature rises above 70 °C.*

**ATTENTION**

*NE PAS exposer les surfaces du capteur aux contraintes mécaniques. Si aucun refroidissement externe n'est appliqué, n'utilisez pas le DSC 4000 au-dessus de 50 ° C. Le DSC 4000 s'éteint automatiquement lorsque la température intérieure monte au-dessus de 70 ° C.*

**CAUTION**

*Do not exert excessive forces on the sensor disks.*

**ATTENTION**

*Ne pas exercer de forces excessives sur les disques du capteur.*

**CAUTION**

*Care must be taken when handling the outer furnace lid so its ceramic lining is not damaged.*

**ATTENTION**

*Des précautions doivent être prises lors de la manipulation du couvercle du four extérieur, de sorte que sa doublure en céramique n'est pas endommagée..*

- Avoid contamination of the furnace, use high-purity purge gases to purge any reaction products out of the furnace, and heat the furnace to temperatures above 430 °C ONLY when absolutely necessary.
- Check that the cooling liquid supply is circulating before starting a run.
- Ensure the purge gases are set to the recommended flow rates. A rate that is too high will disturb the inner furnace lid.
- Do not operate the instrument in a cold room. The ambient temperature and the temperature of the instrument should be between 10 °C and 30 °C.
- Never open the furnace when it is at an elevated temperature.

***Important Specifications***

Maximum power consumption	430 VA
Supply voltage	100–120 V or 220–240 V
Supply voltage frequency	50–60 Hz
Fuses	6.3 AT or 3.15 AT
Safe temperature range	10–40 °C
Dimensions L x H x D	38 x 17 x 41 cm (H 29 cm with autosampler)
Weight	17 kg (19 kg with autosampler)

## ***Decontamination and Cleaning***

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### ***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](http://www.perkinelmer.com/Content/technicalinfo/dts_instrumentdeconprocedure.pdf)

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

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

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.

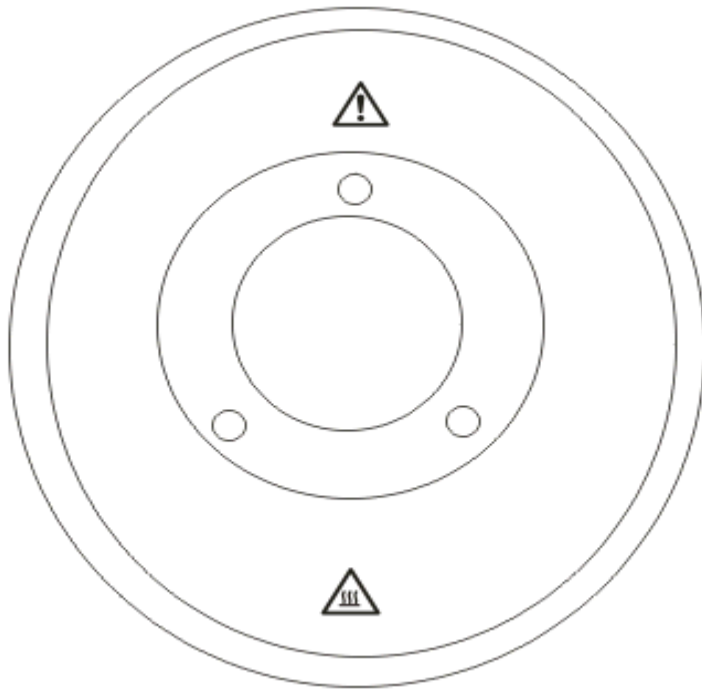
### ***General Laboratory Safety***

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.).

## ***DSC 4000 Warning Labels***

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### ***Warning Labels on the Upper Ring Around the Furnace***



**WARNING**

***HOT SURFACE***

*The furnace at the center of the ring may be hot.*



**AVERTISSEMENT**

***SURFACE CHAUDE***

*Le four au centre de l'anneau peut être chaud.*

**NOTE:** If you have an autosampler installed, this label is positioned on the autosampler.





**WARNING**

***Toxic Gases – Fume Ventilation System***

*Without adequate ventilation, potentially toxic vapors can build up in the laboratory. Your laboratory must have a reliable fume ventilation system before you use this instrument.*

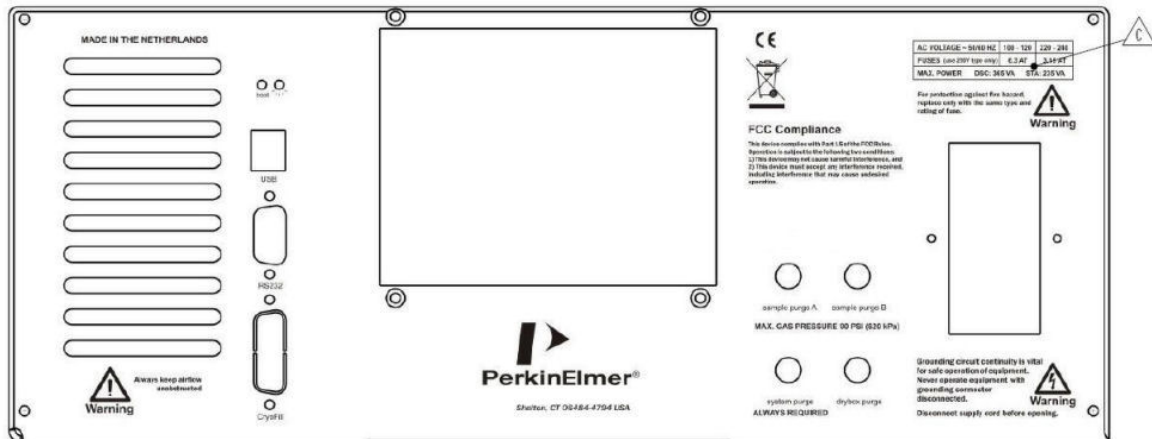


**AVERTISSEMENT**

***Gaz toxiques - Système de ventilation des fumées***

*Sans ventilation adéquate, des vapeurs potentiellement toxiques peuvent s'accumuler en laboratoire. Votre laboratoire doit disposer d'un système de ventilation fiable avant d'utiliser cet instrument.*

## Warning Labels on the Back of the DSC 4000



**WARNING**

***Always keep airflow unobstructed***

*To maintain adequate ventilation do not block the back of the instrument.*



**AVERTISSEMENT**

***Toujours garder le flux d'air dégagé***

*Pour maintenir une ventilation adéquate, ne bloquez pas l'arrière de l'instrument.*



**WARNING**

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



**AVERTISSEMENT**

***Pour la protection contre les risques d'incendie, remplacer uniquement par le même type et le même type de fusible.***



**WARNING**

*Grounding circuit continuity is vital for the safe operation of equipment. Never operate equipment with the grounding connector disconnected. Disconnect supply cord before operating.*



**AVERTISSEMENT**

*La continuité du circuit de mise à la terre est essentielle pour la sécurité de fonctionnement de l'équipement. N'utilisez jamais d'équipement avec le connecteur de mise à la terre déconnecté. Débranchez le cordon d'alimentation avant d'utiliser.*

## ***Install a DSC 4000***

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The installation procedure for the DSC 4000 consists of the following steps:

- Prepare the Laboratory (refer to page 19)
- Unpack the DSC 4000
- Set Up the DSC 4000 System Components
- Connect the DSC 4000 System Components
- Configure the DSC 4000
- Calibrate the DSC 4000

## ***Unpacking the DSC 4000***

---

The DSC 4000 System consists of the following components:

- DSC 4000
- Personal Computer
- Pyris Software
- Printer (optional)
- Intracooler 6P (optional)
- Portable Cooling Device (optional)
- Chiller (optional)
- Autosampler (optional)

The autosampler is an optional accessory that is used to automate the running of multiple samples. It is factory installed. The sample tray holds up to 45 samples. Runs can be sequenced and postrun data analyzed by using a play list which is created using the Pyris Player software.

The DSC 4000 comes wrapped in plastic and is surrounded by foam on all sides of the analyzer. The DSC 4000 weighs 17 kg (37 lb), or 19 kg (42 lb) with the autosampler installed. To unpack the analyzer, follow the steps below:

1. Remove the foam inserts from the box.
2. Remove the analyzer from the box.
3. Remove the foam from all sides, turning the analyzer on end as necessary.
4. Remove the tape and plastic from around the analyzer.
5. Remove the tape from the outer furnace lid and remove the lid. Place it to the side.
6. Remove the packing material from inside the furnace area.
7. Remove the tape from the connectors on the rear of the instrument.

When unpacking each box of your shipment, check all the parts included against the shipping list. If any part is damaged or missing, contact the delivery service and PerkinElmer immediately.

The DSC 4000 is shipped with a Spares Kit. If you have purchased the AS 6000 Autosampler, this is installed at the factory.

Also included with the DSC 4000 are:

<b>Description</b>	<b>Part Number</b>	<b>Quantity</b>
Tygon tubing for coolant	0250-6519	3 m
Teflon tubing for purge gas	0250-6483	6 m
5/16-in. pipe for Swagelok Cooling Liquid In and Out connectors	—	2
RS-232 cable	—	1
Male RJ45 to DB9 modem cable	0941-0042	1
Power cable	—	1

Place the analyzer carefully on the prepared bench. Unpack the remaining of the DSC 4000 and place them nearby. You will need these parts for the installation of the analyzer.

## ***Set Up the DSC 4000 System Components***

Place the DSC 4000 on your laboratory bench. Do not plug in the power cord until instructed. The computer and printer should already be on the bench and installed according to each item's instructions.

Be sure to set up all of the system components listed below before switching on the DSC 4000:

- Select the correct voltage
- Install the purge gases
- Install the cooling device

### ***Select the Correct Voltage for the DSC 4000***

The DSC 4000 accepts the following voltages: 100–120 V and 200–240 V (50/60 Hz).

It is configured and shipped to operate at 200–240 V. Selection of the correct voltage for your lab can be done at the AC inlet plug at the rear of the DSC 4000.

First check whether the voltage selection corresponds to the voltage in your laboratory. This can be seen easily: the voltage range with the text in an upright position and the arrow pointing at the small line represents the voltage that has been selected.



To select the correct voltage for the DSC 4000 in your laboratory:



**WARNING**

*Ensure that the mains power lead is disconnected from the instrument before proceeding.*



**AVERTISSEMENT**

*Assurez-vous que le cordon d'alimentation secteur est débranché de l'instrument avant de continuer.*

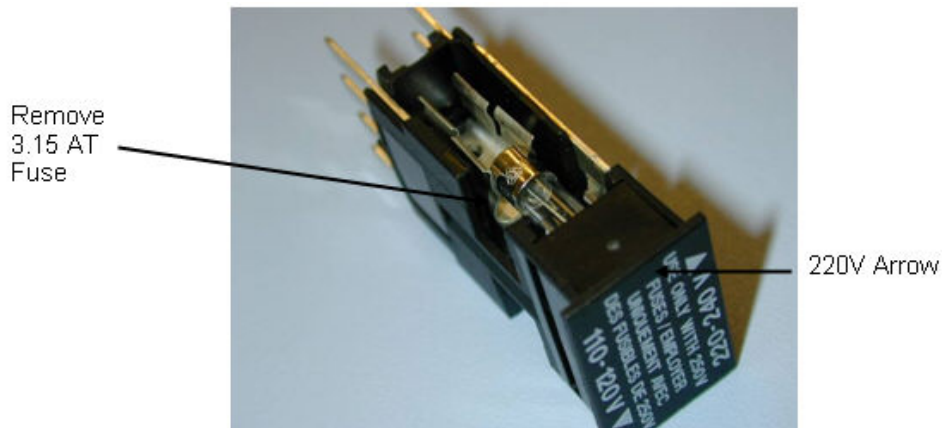
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1. Remove the fuse holder with a screw driver by gently prying it out of the compartment.



2. Once the holder is out far enough to hold onto, slide the fuse holder out of the slot.
3. Remove the fuse (3.15 A) from the 220 V position. This can be done using the point of a pencil to gently lift the fuse out of the holder.

**NOTE:** It may be necessary to move the fuse slightly towards the back of the fuse pack before prying it out of the holder.



**NOTE:** In the figure above, the fuse that is installed is for 220 V.

4. Insert the fuse (6.3 A) for 110 V (it goes into the right side when 110–120 V is in the upright (legible) position).

**NOTE:** Ensure that the head of the fuse makes contact at both ends of the fuse holder.





5. Reinsert the fuse holder.



### ***Connect the Purge Gas Supply to the DSC 4000***

The DSC 4000 has a system purge gas inlet and two sample purge gas inlets at the back of the analyzer. The system purge gas purges the outer portion of the sample holder/heat exchanger assembly with dry gas. This facility must ALWAYS be used when working with the analyzer. The dry gas, preferably dry nitrogen, prevents condensation of water vapor on the measuring cell. The system purge gas flow rate is controlled by a needle valve inside the analyzer, and is preset to 40 ml/min.

**NOTE:** The DSC 4000 analyzer has a mass flow controller built into the analyzer. However, if you have an autosampler and are using the dry box purge, this does not have a mass flow controller. Therefore, it is necessary to use either an H restrictor (P/N 01541498) or a Float Displacement Flow Meter (P/N 02901624) to obtain the proper flow rates. **The gas must be dry.** A flow rate of between 20 and 40 ml/min is recommended.

The sample purge gas inlets (A and B) are used to purge the furnace, and are part of the internal gas selector (which has two inlets and one outlet). Argon or nitrogen of 99.9% minimum purity is recommended for purging the sample area. Other gases such as air or oxygen may also be used. The flow rate of the sample purge gas is controlled using the software.

**NOTE:** **The gas must be dry.** A flow rate of between 20 and 40 ml/min is recommended.

The procedure for connecting the purge gas supply includes the following steps:

- Connect the purge gas line to the system gas supply.
- Install a filter dryer (optional).
- Connect the purge gas lines to the DSC 4000.

### ***Connect the Purge Gas and System Purge Gas Lines to the DSC 4000***

Perform the following procedure to connect your sample purge gas and system purge gas lines to the DSC 4000 analyzer regardless of the other components in the purge gas line.

Assuming that all the other components of the purge gas line are installed, you will now connect 1/8-in. Teflon tubing to the DSC 4000 using a female connector.

The DSC 4000 system purge gas (1 pc) and sample purge gas (2 pcs) connectors are mounted to the back of the analyzer.

1. Place the 1/8-in. Teflon tubing from your dry purge gas line onto the inlet labelled system purge gas ALWAYS REQUIRED.
2. Place the 1/8-in. Teflon tubing from your system purge gas line onto the inlet sample purge A.

If a second sample purge line is desired, connect it to sample purge B (1/8-in. Teflon tubing).

#### Connecting purge and system gas lines to the DSC 4000:



**NOTE:** The dry box purge inlet is only used when an autosampler is installed on the instrument.

### ***Connect the Cooling Device to the DSC 4000***

The DSC 4000 has an integrated cooling system that accepts various types of cold gases and liquids. In standard configuration, the cooling system operates from ambient to 445 °C. Normal tap water can be used for the coolant. The recommended cooling system for the range of -20 °C to 445 °C is the Polyscience Chiller (N537-0220/N5370221, 120 V and 220 V, respectively).

The Intracooler 2S for DSC 4000 is the cooling device for the range of -65 °C to 445 °C.

The Portable Cooling Device (PCD) can be used to quickly cool the DSC 4000 to ambient temperature, or to cool below room temperature for occasional subambient measurements. The PCD (P/N N5202068) can be filled with ice, ice water, liquid nitrogen or any other cooling mixtures.

**NOTE:** When operating the analyzer with a cooling device you **MUST** use a purge gas.

## Connecting to the Chiller

When using the chiller, connect the cooling supply to the DSC 4000 as follows:

**CAUTION**

*An appropriate fluid must be used with the chiller. The fluid should be effective down to  $-20^{\circ}\text{C}$ . Any lower than that would impair the conveyance power of the chiller's pump. Distilled water is recommended for operation at temperatures between  $15^{\circ}\text{C}$  and  $90^{\circ}\text{C}$ . For operation at  $-20^{\circ}\text{C}$  to  $100^{\circ}\text{C}$ , 50% ethylene glycol in water is recommended.*

**ATTENTION**

*Un fluide approprié doit être utilisé avec le refroidisseur. Le fluide devrait être efficace jusqu'à  $-20^{\circ}\text{C}$ . Tout inférieur à celui qui affecterait le pouvoir de transport de la pompe du refroidisseur. L'eau distillée est recommandée pour un fonctionnement à des températures comprises entre  $15^{\circ}\text{C}$  et  $90^{\circ}\text{C}$ . Pour une utilisation de  $-20^{\circ}\text{C}$  à  $100^{\circ}\text{C}$ , 50% d'éthylène glycol dans l'eau sont recommandés.*

**CAUTION**

*Ensure that the cooling air vents at the front and rear of the cooler are not blocked. Maintain a minimum space of 15–20 cm.*

**ATTENTION**

*Assurez-vous que les événements d'air de refroidissement à l'avant et à l'arrière du refroidisseur ne sont pas bloqués. Maintenir un espace minimum de 15-20 cm.*

1. Make sure that the chiller is switched off but that its power cord is plugged into a power supply.
2. Screw one of the barbed hose couplings shipped with the chiller into the OUTLET connection on the chiller unit, and tighten with a suitable wrench.
3. Couplings suitable for several different hose sizes are provided with the chiller.
4. Push a hose (compatible with the desired coolant) onto the OUTLET coupling and secure with a hose clamp.
5. Slide the other end of the hose over the metal tube labeled COOLING LIQUID IN on the thermal analyzer. Place a hose clamp around the hose and metal tube to secure the hose in place.
6. Screw a barbed hose coupling into the INLET connection on the chiller unit, and tighten with a suitable wrench.
7. Push a hose onto the INLET coupling and secure with a hose clamp.
8. Connect the other end of the hose to the COOLING LIQUID OUT connector on the analyzer. Place a hose clamp around the hose and the metal tube to secure the hose in place.

## Setting Up The Chiller

1. Fill the reservoir with coolant.  
Ensure that the cooling coils are completely covered. The maximum coolant level is 25 mm below the top of the reservoir.

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2. Switch the chiller on at the mains supply and at the switch on the rear of the control unit (NOT using the Power switch on the front of the control unit).
3. When switching on the chiller for the first time, select the display language by rotating the Select/Set control on the front panel, and then pressing to select the desired option. The chiller controller will continue with its start-up sequence and then display "Standby" when ready.
4. Set the safety set temperature on the controller using a flat screwdriver to rotate the control to the maximum temperature to which the bath should be heated.
5. Press the Power switch on the front panel.  
The pump will begin operating.
6. Top up the coolant reservoir to compensate for the fluid in the external circuit.
7. Rotate the Select/Set control on the front panel until the Pump/AutoTune menu is displayed.
8. Press the Select/Set control until the pump speed bar is highlighted.
9. Rotate the Select/Set control to adjust the pump speed, and press the control to accept the new setting.

**CAUTION**

*The heat exchanger of the DSC 4000 will NOT accept pressure. Use the lowest flow rate that provides sufficient cooling for your experiment to minimize the pressure in the system.*

**ATTENTION**

*L'échangeur de chaleur du DSC 4000 n'acceptera PAS la pression. Utilisez le débit le plus bas qui procure un refroidissement suffisant pour votre expérience afin de minimiser la pression dans le système.*

**CAUTION**

*It is essential to maintain a very constant flow rate of coolant. The liquid should be free of air bubbles. Changes in the liquid flow rate or the presence of air bubbles will reduce the quality of the measurement signal.*

**ATTENTION**

*Il est essentiel de maintenir un débit constant de liquide de refroidissement. Le liquide doit être exempt de bulles d'air. Des changements dans le débit de liquide ou la présence de bulles d'air réduiront la qualité du signal de mesure.*

### **Setting the Coolant Temperature**

When using a circulator, the temperature must be controlled to within 0.2 °C (set point  $\pm$  0.1 °C).

1. Press and release the Select/Set control on the front panel.  
The set point temperature value is highlighted.
2. Turn the control to set the temperature to the nearest whole degree Celsius.
3. Press the control again.

The degree fraction figures are highlighted.

4. Turn the control to select the desired fraction of a degree.
5. Press the control to accept the value.

### **Connecting to Tap Water**

When using tap water:

1. Make sure the tap water is conveniently located near the DSC 4000.
2. Connect a hose to the Cooling Liquid In connector. Secure with a clamp.
3. Connect the other end of this hose to the tap.
4. Connect the other hose to the Cooling Liquid Out connector. Secure with a clamp.
5. Connect the other end of the hose to a suitable drain.

The tap water must be continuously circulated through the heat exchanger with a low flow rate of 5–15 l/h during the operation of the analyzer. The temperature of the cooling liquid may not change more than 0.4 °C/hour in the case of tap water.

**CAUTION**

*The heat exchanger of the DSC 4000 will NOT accept pressure. Therefore, flow regulation should be done before the water enters the DSC 4000.*

**ATTENTION**

*L'échangeur de chaleur du DSC 4000 n'acceptera PAS la pression. Par conséquent, la régulation du débit devrait être effectuée avant que l'eau entre dans le DSC 4000.*

**CAUTION**

*It is essential to maintain a very constant flow rate of approximately 5 l/h (80–90 ml/min). The liquid should be free of air bubbles. Changes in the liquid flow rate or the presence of air bubbles will negatively influence the quality of the measurement signal.*

**ATTENTION**

*Il est essentiel de maintenir un débit très constant d'environ 5 l/h (80-90 ml/min). Le liquide doit être exempt de bulles d'air. Les changements dans le débit de liquide ou la présence de bulles d'air influenceront négativement la qualité du signal de mesure.*

### **Connecting to the Intracooler for the DSC 4000:**

**CAUTION**

*Frosting is possible on the rear of the DSC 4000. To prevent this problem the exhaust tube must be properly installed on the analyzer.*

**ATTENTION**

*Le givrage est possible à l'arrière du DSC 4000. Pour éviter ce problème, le tube d'échappement doit être correctement installé sur l'analyseur.*

Exhaust tube installation:

- For the KTS Kinetics Cooler use the entire piece as it does fit over the hose.
- For the PolyScience Intracooler use a piece that slips over the two chiller pipes, approximately 3.5 cm long.

Precautions for Intracoolers:

- Do not lift or move the unit by pulling the flexible line.
- Do not make any sharp bends in the flexible line.
- Do not block or restrict air flow to the vents on the unit.
- Do not operate the unit in temperatures above 32 °C.
- Do not operate at a line voltage outside of 110 V +/-10% or 220 V +/-10% when operating at 50/60 Hz.

## ***Connect the DSC 4000 System Components***

The computer and optional printer should already be connected together and the Pyris software already installed. To complete the connection of the system components, the DSC 4000 has to be connected to the computer.

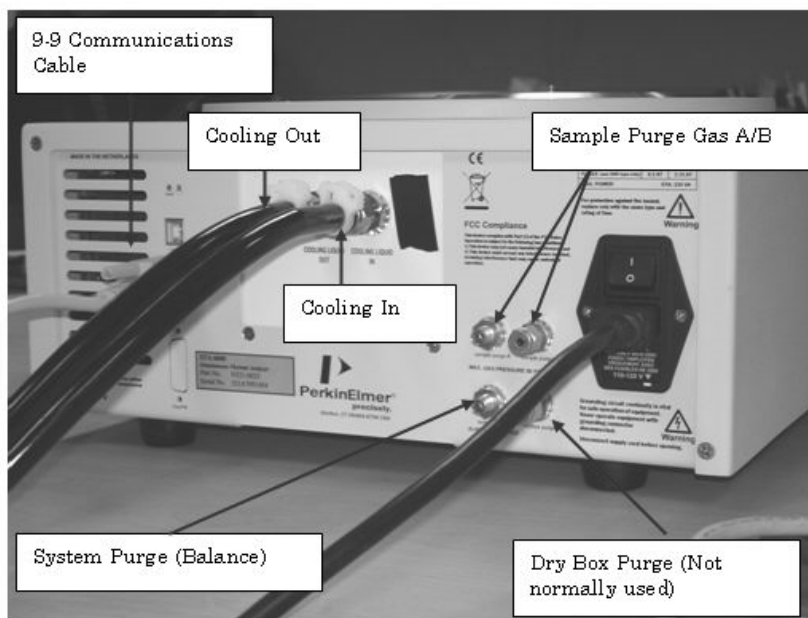
**CAUTION**

*Make sure that the computer is off before plugging in the DSC 4000.*

**ATTENTION**

*Assurez-vous que l'ordinateur est éteint avant de brancher le DSC 4000.*

The image below shows the connections on the DSC 4000:



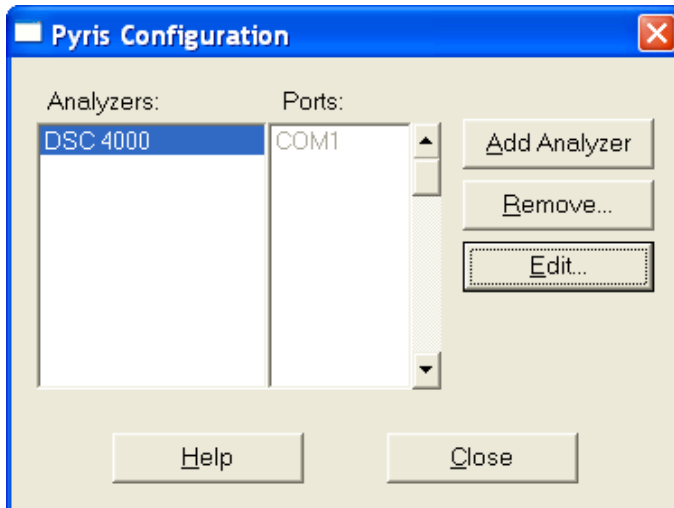
Make sure all components are turned off and unplugged before you begin.

1. Connect one end of the RS-232 cable to the back of the DSC 4000.
2. Connect the other end of the RS-232 cable to COM1 on the back of the computer.
3. Connect the printer and printer cable to the computer.
4. Connect the analyzer, printer and computer power cords to AC outlets.
5. Once the system connections are made, switch on the system in the following order:
  - Computer
  - DSC 4000
  - Printer
6. Turn on the purge gas and dry gas, the water for the DSC 4000 cooling system or any other accessories for the cooling device you are using.

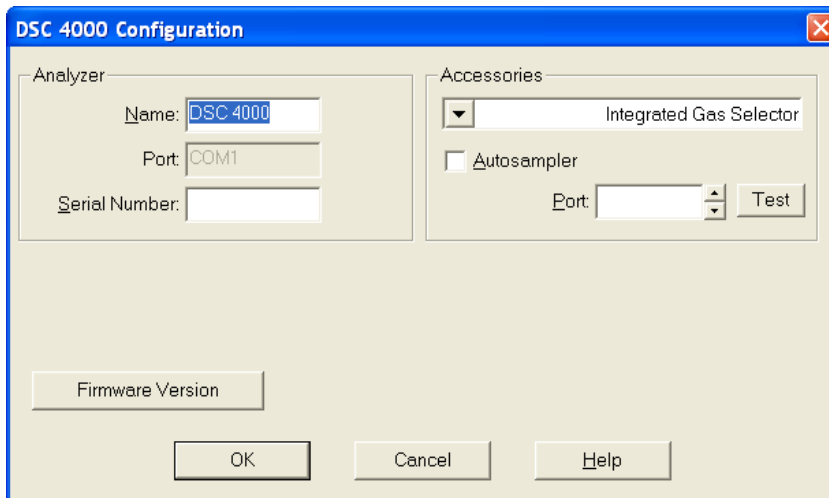
## Configure the DSC 4000

The Pyris software must be configured for the DSC 4000. This must be done before the DSC 4000 Application can be recognized.

1. Select **Pyris Config** from the Pyris group under PerkinElmer Applications in the Programs menu accessed from the Start button or select **Configure Analyzer** from the Pyris Manager Start button menu. The Pyris Configuration dialog box appears on the screen.



2. Click the **Add Analyzer** button. The Add Analyzer dialog box appears.
3. From the list of available ports, select the port to which you connected the analyzer. Remember that COM5 appears when an analyzer is connected to port 1 of an EdgePort USB. Click the **Add** button. The DSC 4000's Configuration dialog box is displayed.



4. The system detects the type of analyzer that is attached and displays the default name and other information for a DSC 4000 in this dialog box. Select the accessories that are attached to the analyzer.
5. Click **OK** to accept the selections.

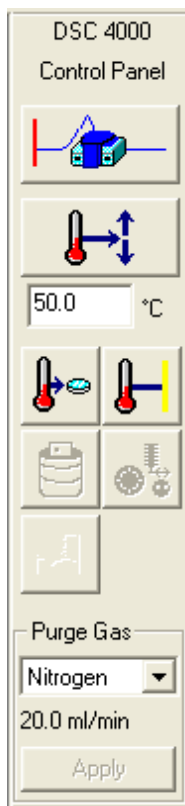



- Click **OK** to exit the Configuration dialog box or select **Add Analyzer** to add another analyzer to the configuration. Remember that the analyzer must be connected to the communications port and switched on; otherwise, it will not be recognized by the Pyris software.

Once the Pyris software has been configured for the DSC 4000, the DSC 4000 Application can be started from Pyris Manager.

## Starting the Pyris Manager

- Select **Pyris Manager** from the Pyris Software group in Programs on the Start menu. The Pyris Manager is displayed on the screen.
- Select **DSC 4000** from the Pyris Manager. The DSC 4000 control panel is displayed.



- Now that the system is switched on, the DSC 4000 should be conditioned before using for the first time (and when it has been switched off for long periods of time). This is to remove any water vapor that has been adsorbed to the furnace wall during transport and/or storage. To do this, enter 150 °C in the temperature field beneath the  on the control panel and click on the button. Alternatively, you can set up a method to heat to 150 °C at a heating rate of 5 °C/min. When the analyzer has cooled to ambient temperature, it is ready for normal use.

The configuration for the DSC 4000 is complete. The analyzer is ready to be calibrated.

## **Calibrate the DSC 4000**

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Upon installation, the DSC 4000 must be calibrated for temperature and heat flow before routine operation. Calibration is accomplished by running high-purity standards or reference materials with known temperature and energy transitions. The data obtained after running these materials (that is, temperature transitions and  $\Delta H$  values) are used in the Pyris calibration routine to automatically calibrate the analyzer. Once the analyzer is calibrated, it will remain calibrated, even when the system is switched off, unless there are major changes to the analyzer's operating conditions.

The DSC 4000 is shipped with a Spares Kit that contains reference materials. Additional reference materials can be ordered through [www.perkinelmer.com/supplies](http://www.perkinelmer.com/supplies). Indium and zinc are generally used for routine calibration.

1. Cut and weigh a small piece of each reference material (indium and zinc), each between 5 and 10 mg. Record the weights.  
The DSC 4000 Spares Kit includes precrimped, preweighed samples of indium and zinc (P/N N5190762). If you use these samples, be sure to use the small sample.
2. Encapsulate the reference material in a standard sample pan.  
To achieve the best temperature repeatability, samples should be flattened and placed in the center of the sample pan, then encapsulated.
3. Using tweezers, place the encapsulated sample into the sample area of the sample holder (left side); place an empty sample pan and lid into the reference side of the sample holder (right side).
4. Using tweezers, cover the furnace with the inner and the outer furnace covers.
5. To open the IndCal method from the Method Editor, select **File, Open Method**, and then choose **Indcal.d6m** from the Program Files\PerkinElmer\Pyris\Methods directory. You can also use the calibration method file for zinc called **Zinccal.d6m**.
6. In the Sample Info page, fill in the sample information. Enter the weight of the indium sample and the file name under which the data will be saved.
7. Click the Start Method button on the DSC 4000 control panel.  
The run starts; when completed, the file will be saved with the file name entered in the method.
8. After the run is complete, perform a Peak Area calculation, include the Onset temperature and note the Onset result.
9. Repeat steps 1–8 for each reference material you are using to calibrate the DSC 4000.  
Up to ten reference materials can be used to calibrate the temperature program of the analyzer.
10. From the Method Editor, select **Calibrate** from the View menu.
11. Select the Temperature tab.

12. Enter the name of the reference material used, the **Expected** Onset value, the **Measured** Onset value just measured for each reference material, and click the check box in the Use column.
13. Select the Heat Flow tab.
14. Enter the name of the reference material used (we recommend indium for this calibration), the **Expected**  $\Delta H$  value, the **Measured**  $\Delta H$  result just measured, and the **Weight** of the reference material used.
15. Click the **Save and Apply** button. In the Save As dialog box, select the default.d6c calibration file and click **OK**. Answer **Yes** to overwrite the existing file to save the new calibration values.
16. Click **Close** to exit Calibration and send the new calibration values to the DSC 4000.

The DSC 4000 calibration is complete.

This completes the installation of the DSC 4000.



# **DSC 4000 Hardware**

## ***DSC 4000***

---

The DSC 4000 Differential Scanning Calorimeter is a single-furnace, heat-flux DSC. Heat flow is determined by measuring a temperature difference over a very accurately known thermal resistance. This analyzer is used to characterize materials, design products, predict product performance, optimize processing conditions, and improve quality. The DSC 4000 system permits the direct calorimetric measurement, characterization, and analysis of thermal properties of materials. Under the control of the Pyris software on your PC, the DSC 4000 is programmed from an initial to a final temperature through transitions in the sample material such as melting, glass transitions, solid-state transitions, or crystallization. Usually the DSC 4000 is programmed to scan a temperature range at a linear rate for the study of these endothermic and exothermic reactions. Endotherms and exotherms can be shown as an upward or downward deviation from the baseline.

The DSC 4000 has an integrated cooling system which accepts various types of cold liquids. This allows operation from  $-100\text{ }^{\circ}\text{C}$  to  $445\text{ }^{\circ}\text{C}$ . The DSC 4000 can be used with a range of commercial circulating coolers, and for occasional subambient operation can be used with the Portable Cooling Device.

The DSC 4000 is also upgradeable to the DSC 6000.

### ***Furnace and Sensor Design***

A low-mass, alumina-coated aluminum furnace forms the heart of the DSC 4000. The furnace is designed for ruggedness and resistance to chemical corrosion. Because of its low mass, the furnace can heat and cool rapidly. A platinum resistance sensing device measures and controls the furnace temperature.

The atmosphere surrounding the sample is controlled very efficiently since the gas enters above the sample crucible. This is extremely important when performing oxidative stability experiments. The purge gas is also preheated resulting in minimal disturbance of the signal.

The sample is placed on a precisely machined sensor disk constructed of 90% nickel/10% chromium. This rugged and durable disk provides high thermal conductivity and a very low time constant for superior instrument response. Highly sensitive thermocouples measure the temperature at both the sample and reference positions. The DSC 4000 accommodates a wide range of PerkinElmer sample pans. Over two dozen different pan types are available.

## Cooling

The DSC 4000 incorporates a built-in cooling chamber that lies below and is attached to the furnace assembly. The chamber uses a simple in/out design which allows for the use of any noncorrosive circulating liquid or cooling gas. Cooling liquids include water and ethylene glycol.

For cooling with liquids, many commercial circulating coolers are available that can be interfaced to the DSC 4000. The Polyscience Chiller (P/N N5370220/N5370221, 120 V and 220 V, respectively) is the recommended cooling device for the range  $-20\text{ }^{\circ}\text{C}$  to  $445\text{ }^{\circ}\text{C}$ . The Intracooler (P/N N5374098/N5374099) is recommended for the range  $-65\text{ }^{\circ}\text{C}$  to  $445\text{ }^{\circ}\text{C}$ . The Intracooler accessory allows rapid cooling between sample runs, allowing fast sample turnaround. The Portable Cooling Device (PCD) can also be used to quickly cool the DSC 4000 to ambient temperature, or to cool below room temperature for occasional subambient measurements. The PCD (P/N N5202068) can be filled with ice, ice water, liquid nitrogen or any other cooling mixtures.



**WARNING**

*Do not attempt to service the DSC 4000. Service must be performed by a PerkinElmer Instruments Service Representative.*



**AVERTISSEMENT**

*N'essayez pas de réparer le DSC 4000. Le service doit être effectué par un représentant du service PerkinElmer Instruments.*

Other topics related to the DSC 4000 are:

- Safety Precautions
- Calibration
- Operating Variables and Sample Handling
- Subambient Operation
- Maintenance
- Part Numbers
- Autosampler

## ***Calibration of the DSC 4000***

---

The DSC 4000 has been calibrated at the factory for both temperature and heat flow. Under normal conditions, the analyzer does not need temperature recalibration. As a result of the use of high-precision Pt100 sensors, the temperature accuracy does not vary over the temperature range and remains constant for long periods of time.

We recommend that the temperature calibration and heat flow calibration be checked using the precrimped samples of indium and zinc before obtaining data. We also recommend that you perform calibrations in the following order:

- Temperature Calibration
- Heat Flow Calibration

### ***Temperature Calibration***

The DSC 4000 has been calibrated at the factory for temperature. Under normal conditions the DSC 4000 does not need temperature calibration. As a result of the use of high-precision sensors, the temperature accuracy does not vary over the temperature range and remains constant for long periods of time.

**NOTE:** It is recommended that a baseline correction be performed before temperature calibration.

At least two measured onset temperatures are necessary to calibrate the DSC 4000. The calibration materials selected must have at least a 50 °C temperature difference in their theoretical Onset temperatures. Indium and zinc are the recommended calibration materials. However, other reference materials can be used to calibrate the analyzer but they must be pure, for example, distilled water.

To calibrate the temperature of the DSC 4000:

1. While using the Instrument Viewer or the Method Editor in the Pyris software, select **Calibrate** from the View menu.  
The Calibration window is displayed.
2. Restore the default temperature calibration by selecting **Temperature** from the Restore menu. If you are going to perform a heat flow calibration, restore all default calibration values by selecting the **All** command.
3. Click the **Save and Apply** button in the Calibration window to send the default values to the analyzer and save the current calibration file. The Save As dialog box appears. Click **OK** to save the default file.
4. Select **Close** to close the Calibration window and begin using the new calibration values.


After you have performed these steps, go on to the following topics to complete the temperature calibration of the DSC 4000:

- Perform Indium and Zinc Runs
- Perform a Peak Area with Onset Calculation
- Perform a Temperature Calibration




## Perform Indium and Zinc Runs

In performing an indium and zinc run, you can use the precrimped, preweighed samples of indium and zinc or you can prepare your own samples for calibration. To perform the run, follow the steps below:

1. Place the sample pan containing indium or zinc in the sample area of the sample holder and place the reference sample pan in the reference area of the sample holder.
2. Using tweezers, cover the furnace with the inner and the outer furnace covers.
3. If not already displayed, select the Method Editor icon  from the toolbar.
4. From the File menu, select **Open Method**.  
The Open Method dialog box displays the methods available in the default methods directory.
5. Select the **IndCal.d6m** method if using indium or **ZincCal.d6m** if using zinc.
6. In the Sample Info page, fill in the sample information. Enter the weight of the indium or zinc sample and the file name under which the data file is to be saved.



7. Click the Start button  in the control panel to start the run.  
The run starts. When finished, the data file will be saved with the file name that you entered in the method.

After performing the indium and zinc runs, you must perform a peak area with onset calculation.

## Perform a Peak Area with Onset Calculation

For the indium and zinc runs that you performed, a Peak Area with Onset calculation is performed. The data file you just collected should be displayed in the Data Analysis window. Perform an onset calculation on the data as follows:

1. Select **Onset** from the Calc menu.  
The Onset Calculation dialog box appears.
2. Enter the Left Limit and the Right Limit. You can also click on the two red X's that appear on the curve and drag them to the desired left and right limits. The Left Limit and the Right Limit fields will be filled in on the dialog box.
3. Click the **Calculate** button.
4. Record the Onset value for temperature calibration and  $\Delta H$  for heat flow calibration.  
If you performing a heat flow calibration, you will need the  $\Delta H$  values.
5. Repeat steps 1–4 for the zinc calibration run.

Once you obtain the Onset values, you can perform a temperature calibration.

## Performing a Temperature Calibration

The measured Onset values for indium and zinc are used to calibrate the temperature of the DSC 4000, DSC 6000, Pyris 6 DSC and the Jade DSC. To perform the temperature calibration, perform the following steps:

1. With either the Instrument Viewer or the Method Editor as the active window, select **Calibrate** from the View menu.  
The Calibrate dialog box is displayed.
2. Select the Temperature tab.
3. Enter the name of the reference material that you used.
4. Enter the expected onset temperature for the reference material in the Exp. Onset field (for example, 156.60 for indium).
5. In the Meas. Onset field, enter the onset temperature that you obtained when you performed the peak area with onset calculation.
6. Enter the Method used for the calibration.
7. Select the check box in the Use column for each reference material used in the calibration.
8. Click the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the current calibration file.
9. Go to the Heat Flow calibration procedure or click **Close** to close the Calibration window and begin using the new calibration values.

## Heat Flow Calibration

It is recommended that indium be used when performing a heat flow calibration. However, the heat flow calibration can be performed using any one of the following materials:

Calibration Substance	Heat of Fusion (J/g)
Cyclopentane	8.60
Gallium	79.87
Indium	28.45
Lead	23.01
Tin	60.46
Zinc	108.37

1. While using the Instrument Viewer or the Method Editor, select **Calibrate** from the View menu.
2. Restore the default Heat Flow calibration by selecting **Heat Flow** from the Restore menu.  
If you performed a Temperature calibration just prior to starting a Heat Flow calibration and selected **All** from the Restore menu, then you do not need to restore the default Heat Flow calibration here.
3. Click **Save and Apply**.
4. Click **Close** in the Calibration window.
5. In Data Analysis, use the same data file obtained when you performed the indium run, or perform a run using another reference material.
6. Perform a Peak Area with Onset calculation and note the  $\Delta H$  result, or use the  $\Delta H$  result recorded for the reference material when you performed this step for temperature calibration.

The measured  $\Delta H$  value for indium is used to calibrate the heat flow of the DSC 4000. If you just performed the temperature calibration on a data file, the data file should still be displayed.

To perform the heat flow calibration, perform the following steps:

1. With either the Instrument Viewer or the Method Editor as the active window, select **Calibrate** from the View menu.  
The Calibrate dialog box is displayed.
2. Select the Heat Flow tab.
3. Enter the name of the reference material you used.
4. Enter the expected  $\Delta H$  for the reference material used (for example, 28.45 for indium).
5. Enter the measured  $\Delta H$  result.
6. Enter the **Weight** of the reference material used.
7. Enter the calibration method used.
8. Click the **Save and Apply** button in the Calibration window to send the new calibration value to the analyzer and save the current calibration file.
9. Click **Close** to close the Calibration window and begin using the new calibration values.

## ***Operating Variables and Sample Handling***

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The DSC 4000, DSC 6000, Pyris 6 DSC and the Jade DSC measure differential temperature and report the heat flow to the sample as it is heated, cooled, or held isothermally. The following topics discuss the materials and techniques used to obtain data with a DSC 4000, DSC 6000, Pyris 6 DSC or a Jade DSC.

### ***Temperature Range***

The temperature range for your analysis depends on the sample and the type of experiment you are performing. The Pyris 6 DSC can be used to analyze samples from a temperature of  $-120\text{ }^{\circ}\text{C}$  to  $200\text{ }^{\circ}\text{C}$  when used with cold nitrogen gas or from ambient to  $445\text{ }^{\circ}\text{C}$ . The DSC 4000 can be used to analyze samples from  $-65\text{ }^{\circ}\text{C}$  to  $445\text{ }^{\circ}\text{C}$  with the Intracooler with the PCD. The DSC 6000 and Jade DSC can be used to analyze samples from a temperature of  $-170\text{ }^{\circ}\text{C}$  to  $300\text{ }^{\circ}\text{C}$  when used with liquid nitrogen or from  $-65\text{ }^{\circ}\text{C}$  to  $445\text{ }^{\circ}\text{C}$  with the Intracooler (P/N N5374098/N5374099).

### ***Scanning Rate***

The DSC 4000, DSC 6000, Pyris 6 DSC and the Jade DSC allow heating rates of 0.1 to  $100\text{ }^{\circ}\text{C}/\text{min}$  in steps of  $0.1\text{ }^{\circ}\text{C}/\text{min}$ . Proper selection of heating rate will increase efficiency of your analysis at the desired sensitivity. Generally, slower heating rates improve the peak resolution while faster heating rates improve the apparent sensitivity. Heating rates greater than  $40\text{ }^{\circ}\text{C}/\text{min}$  are generally used for rapidly heating or cooling the analyzer to the beginning temperature of the next segment or to selected isothermal temperatures in the temperature program. Typical experimental heating rates range from 5 to  $20\text{ }^{\circ}\text{C}/\text{min}$ . Slower rates can be used for liquid crystals and purity analyses. Refer to the Help for other topics on operating variables and sample handling:

- Sample Preparation
- Sample Pans
- Sample Encapsulation
- Sample Atmosphere
- Loading the Sample into the DSC

## ***Subambient Operation of the DSC 4000***

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The DSC 4000 can be operated in the subambient mode. The Intracooler (N537-4098 / N5374099) is recommended for the range  $-65\text{ }^{\circ}\text{C}$  to  $445\text{ }^{\circ}\text{C}$ . The Portable Cooling Device (PCD) can be used to quickly cool the DSC 4000 to ambient temperature, or to cool below room temperature for occasional subambient measurements. The PCD (P/N N5202068) can be filled with ice, ice water, liquid nitrogen or any other cooling mixtures.

Subambient operation of the DSC 4000 is covered in the following topics in the Help:

- Precautions for Subambient Operation
- Operating the DSC 4000 in the Subambient Temperature Range
- Portable Cooling Device (PCD)

## ***DSC 4000 Maintenance***

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The DSC 4000 needs little routine maintenance other than proper treatment as a sensitive electromechanical device.

Since the **sensor disk** is the hottest component in the furnace area, it will usually remain clean and free of condensates. However, samples may ooze out of sample pans or be accidentally spilled onto the sensor disk. Such materials should be removed at low temperatures whenever possible by using tweezers or moistened cotton swabs.

Solvents should not be placed directly on the sensor. Moisten a cotton swab or a tissue with solvent; then clean the sensor or upper portion of the furnace wall with the swab.

Any residual sample that has carbonized may remain on the sensor disk after it has been cleaned with a solvent. To remove the residue, gently scrub the sensor with a fiberglass or nylon brush. Be careful not to exert excessive force when scrubbing the sensor since it can be damaged. The sensor must be as clean and flat as possible for proper operation.

To avoid contaminating the **furnace**, use a purge gas at proper flow rates. If the furnace becomes contaminated, clean it as soon as possible.

### ***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.

## ***DSC 4000 Part Numbers***

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Supplies, accessories, and replacement parts can be ordered directly from PerkinElmer. [www.perkinelmer.com/supplies](http://www.perkinelmer.com/supplies), PerkinElmer's web-based catalog service, offers a full selection of high-quality thermal analysis supplies.

To place an order, request a free catalog, or ask for information:

- If you are located within the U.S., call toll free at 1-800-762-4000, 8:30 a.m. – 7 p.m. EST. Your order will be shipped promptly, usually within 24 hours.
- If you are located outside the U.S., call your local PerkinElmer sales office.

or go on-line to [www.perkinelmer.com](http://www.perkinelmer.com).

Below is a list of the part numbers that are in the Spares kit (P/N N5200031).

<b>Part No.</b>	<b>Description</b>	<b>Quantity</b>
03190033	Indium calibrating reference material	1
03190036	Zinc calibrating reference material	1
09908400	Tweezers	1
01541498	H restrictor	1
N5190762	Kit – crimped indium/zinc calibrating reference materials	1
N5390123	Aluminum pans and covers (100)	1

Also included with the DSC 4000 are

- Tygon tubing (P/N 02506519) for connection of liquid coolant
- Hose clamps (P/N 09920044)
- Teflon tubing (P/N 02508059) for purge gas connection
- RS-232 cable for communicating to the computer
- Power cable

## **Miscellaneous Part Numbers**

<b>Part Number</b>	<b>Description</b>
N5370212	DSC 4000
N5370213	DSC 4000 with Autosampler
N5202068	Portable Cooling Device
N5202070	Mini drybox
09991662	Fuse for 100–120 VAC, 6.3 AT, 250 VAC, 5 x 20 mm
09991629	Fuse for 220–240 VAC, 3.15 AT, 250 VAC, 5 x 20 mm



# **AS 6000 Autosampler**

## AS 6000 Autosampler

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The AS 6000 autosampler accessory brings automation capability to the DSC 4000, DSC 6000, STA 6000 and TGA 4000 when used in conjunction with the Pyris software Player feature. It offers faster sampling rates than the AS 6 Autosampler.

**NOTE:** If you have an STA 6000, then you will need firmware for Pyris version 9.1 or later to operate the AS 6000 autosampler. Select **Programs > Pyris Software > Pyris User manuals > Pyris Installation manuals > Upgrading Pyris Software Installation.pdf** for more information.

- The autosampler is powered by the power supply of the parent instrument.
- The part of the autosampler that manipulates the sample pan is called the gripper. It comprises three fingers, 120° apart from each other. They extend out of the end of the gripper housing.
- There are only two ranges of motion for the gripper: the gripper arm moves up and down and rotates. This allows the gripper to access each sample pan in the trays, transfer a pan from the tray, through the furnace access hole in the base plate, to the furnace, and then return it to its position on the tray. Movement in these two directions also allows the gripper a sufficient range of motions to access the furnace lid(s) and move it onto and off of the furnace access hole.
- For the DSC 4000 and DSC 6000, you can use the existing robotic sample pans sealed with the Universal crimper press. All other standard sample pans (sealed and vented) can also be used. For the TGA 4000 and the STA 6000 the standard crucible (ceramic pan) is used.
- The AS 6000 communicates with the computer via the parent instrument's connection to the computer via the RS-232 port.
- The parent instrument communicates with the AS 6000 via an I2C bus.
- The autosampler operates over the full temperature ranges of the TGA 4000, STA 6000, DSC 4000 and DSC 6000.
- When power is supplied the three-finger gripper mechanism, the metal shrinks and the fingers open; when power is not supplied, the metal expands and the fingers close.
- The autosampler operates with a feedback system from the instrument. When a sample pan is located properly on the sensor the autosampler senses this and rises up out of the furnace.
- The light switch sensors by the furnace hole detect the presence of a sample pan or furnace lid being placed in the furnace hole. If the autosampler is supposed to be removing a lid, for example, and the gripper fails to pick up the lid, it is detected by the sensors and error message is displayed.
- The autosampler is fully automated and controlled by the software. The two-piece sample tray holds up to 45 samples for efficient unattended operation.
- One tray holds 22 samples and the other 23 samples. This difference means that you cannot mistakenly place one tray in the wrong position. The trays have recesses on the bottom which catch positioning pins in the base plate. The positioning pins for one tray are located in different places than those for the other tray, which also prevents one tray from being inserted into the wrong location.

The trays can be removed independently so that you can load one with fresh samples while the autosampler runs samples in the other. You could also add samples to the tray "in use" to replace samples that are completed by pausing the play list.

The Pyris Player feature allows for creation of sequences or play lists to operate the autosampler and instrument. During an autosampler sequence, you can perform multiple experiments, curve optimization, calculations, and printing. With Pyris Player, you can create Sample Groups that group similar analyses together to be run in any order. See the software Help for details.

The following topics discuss the AS 6000 autosampler in more detail:

- Safety Precautions
- How the Autosampler Works
- Autosampler Gripper Alignment
- Sample Handling
- Running a Play List
- Troubleshooting
- Maintenance
- Install a Reference Pan into a DSC

## **Safety Precautions for the AS 6000**

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The safety precautions to be followed when using the AS 6000 autosampler are the same as those for the parent instrument: STA 6000, DSC 4000, DSC 6000, or the TGA 4000. In addition, the following two precautions should be kept in mind:



**WARNING**

*To remove the furnace lid(s) of the parent instrument, always use tweezers. The furnace lid(s) might be hot.*



**AVERTISSEMENT**

*Pour retirer le (s) couvercle (s) du four de l'instrument parent, utilisez toujours une pince à épiler. Le (s) couvercle (s) du four pourrait être chaud.*

**CAUTION**

*Before switching on the parent instrument, make sure that the AS 6000 is properly attached to the base plate on the parent instrument.*

**ATTENTION**

*Avant d'allumer l'instrument parent, assurez-vous que l'AS 6000 est bien attaché à la plaque de base de l'instrument parent.*

## ***How the AS 6000 Autosampler Works***

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The autosampler has to be configured into the DSC 4000, the DSC 6000, the STA 6000 or the TGA 4000 via the Pyris Configuration dialog box in order for the software to recognize it. See the software Help for details.

The AS 6000 autosampler is computer controlled and fully automated. Information between the host computer with Pyris software and the AS 6000 is sent via the parent instrument's connection to the computer via the RS-232 port. Light switch sensors by the furnace access hole detect the presence or absence of a furnace lid or sample pan. An error message is generated and displayed in the software and on the LCD of the parent instrument if the sensors detect the presence of an object when there should not be one and vice versa.

**NOTE:** The instrument cannot detect if a sample pan is present in the furnace from a previous power on period. If you turn off the instrument with a sample pan in the furnace, you must remove the pan before loading another sample during the following power on session.

In general, you place samples in sample pans and load the pans in the sample trays. The sample trays are then placed on the base plate on the parent instrument. At the beginning of a run, the gripper arm is up all the way and is over the home position. The fingers are closed. The furnace lid (for the TGA 4000) or lids (for the DSC 4000 or DSC 6000) are on the lid holders in the furnace access hole. The gripper device is instructed via a command in the Pyris software to begin a session.

### ***Autosampler Work Cycle***

When you begin a sample run by starting a play list, the autosampler performs a typical work cycle. If you use the Sample Group instead of a regular play list, many steps are automatically added to the play list by the software. These additional steps are seen when you select View History. For example, if you wish to run two or more samples, you have to include a Return Sample line after the Start Method line for each sample before a Load Sample. When using Sample Group, the software adds that Return Sample line automatically when the play list is played back.

**NOTE:** Play lists created in previous versions of software are compatible with new versions of Pyris software. However, new play lists may not be compatible with older versions of Pyris, or with older instruments.

A typical work cycle for the autosampler on a DSC 4000 or a DSC 6000 and used with a play list is as follows:

**NOTE:** Before the start of a play list, the gripper should be over the home position and there should be no sample pan in the furnace. The lower furnace lid and the upper lid should be in place.

1. Start the play list. Your play list may have an explicit **Load Sample** command, or, if using a Sample Group, the entry Sample List implies Load Sample. Upon reaching this line in the list, the gripper swings around to remove the upper lid and place it on the ring.
2. The gripper then removes the lower lid and places it on the ring.
3. The gripper retrieves the sample pan and places it in the furnace.
4. The lower lid is placed within the furnace.

5. The upper lid is placed on the furnace.
6. The gripper returns to the home position.
7. The method runs.
8. At the end of the run, the system removes the upper lid and places it on the ring.
9. The system removes the lower lid and places it on the ring.
10. The sample pan is removed from the furnace and returned to its place in the tray.
11. The system places the lower lid back into the furnace.
12. The upper lid is placed back on the lid holder.
13. The gripper goes to the home position.
14. The data analysis list for the sample is performed.
15. If there is another sample in the list, the work cycle, starting with step 1, begins again. To remove the last sample from the furnace do a "Prepare Sample -Return Sample # #" as the last Play list step, where # # is the last sample loaded.

A typical work cycle for the autosampler on a TGA 4000 or an STA 6000 and used with a play list is as follows:

**NOTE:** Before the start of a play list, the gripper should be over the home position and there should be no sample pan in the furnace. The furnace lid should be in place on the lid holder over the furnace.

1. Start the play list. Your play list may have an explicit **Load Sample** command, or, if using a Sample Group, the entry Sample List implies Load Sample. Upon reaching this line in the list, the gripper swings around to remove the furnace lid and place it on the ring.
2. The gripper retrieves the specified sample pan and places it in the furnace.
3. The furnace lid is placed on the lid holder.
4. The gripper returns to the home position.
5. The method runs.
6. At the end of the run, and after the end condition temperature is achieved, the system removes the furnace lid and places it on the ring.
7. The sample pan is removed from the furnace and returned to its place in the tray.
8. The furnace lid is placed back on the lid holder.
9. The gripper goes to the home position.
10. The data analysis list for the sample is performed.
11. If there is another sample in the list, the work cycle, starting with step 1, begins again.

## **Reduced Time Cycle**

A reduced time cycle is available for the STA 6000, DSC 4000 and DSC 6000.

This feature is accomplished by using either a Sample Group or by doing successive Load Sample commands without an explicit Return Sample.

If you do this, the step(s) where the furnace lid(s) is (are) replaced is eliminated and the next sample is immediately loaded.

Below the procedure for the work cycle for DSC 4000/DSC 6000 for multiple samples using the reduced cycle time.

1. Start the play list. Your play list may have an explicit Load Sample command, or, if using a Sample Group, the entry Sample List implies Load Sample. Upon reaching this line in the list, the gripper swings around to remove the upper lid and place it on the ring.
2. The gripper then removes the lower lid and places it on the ring.
3. The gripper retrieves the sample pan and places it in the furnace.
4. The lower lid is placed within the furnace.
5. The upper lid is placed on the furnace.
6. The gripper returns to the home position.
7. The method runs.
8. The data analysis list for the sample is performed.
9. The system removes the upper lid and places it on the ring.
10. The system removes the lower lid and places it on the ring.
11. The sample pan is removed from the furnace and returned to its place in the tray.
12. The gripper retrieves the next sample pan and places it in the furnace.
13. The system places the lower lid back into the furnace.
14. The upper lid is placed back on the lid holder.
15. The gripper goes to the home position.

The procedure for the STA 6000/TGA 4000 is similar to the procedure above.

## ***AS 6000 Autosampler Gripper Alignment***

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The autosampler is aligned at the factory. However, if you install a reference pan into your DSC instrument or change the sample thermocouple of your TGA instrument, change the sensor, or if the gripper does not grasp the sample pans correctly, you should perform a gripper alignment procedure.

1. Access the procedure from the Autosampler Control dialog box which is displayed by



clicking on the Autosampler Control button on the control panel of your instrument. Click **Align** to open the Autosampler Alignment Wizard.

**NOTE:** When running an autosampler alignment, make sure that the temperature is 22 °C (ambient temperature). If the temperature is too high the grippers will not align.

2. The first wizard informs you that the upper furnace lid should be in place on top of the furnace access hole for either parent instrument. The lower furnace lid for the DSC 4000 or 6000 should be in its place on the ring. It is not necessary to have sample pans in positions 12 and 33. If you wish to use the previous calibration's values as a start for this calibration, click on the radio button. (The calibration values are stored in the firmware on the AS 6000 controller board.) Otherwise, the default values will be used to start. Click **Next** to continue.
3. The gripper moves from the home position to location 12. The buttons in the next wizard are used to move the gripper with respect to the dimple of location 12 so that the fingers are even around the dimple. You also have to adjust the height of the fingers above the tray surface. There should be only 0.1 mm between the tray and the finger. Use the buttons on the wizard to move the gripper up and down and left and right. When satisfied with the positioning, click **Next**. The gripper swings around to position 33.
4. Do the same thing for position 33 as you did for 12. Click **Next**.
5. The last wizard screen is displayed. Click **Finish** to exit from the Autosampler Alignment Wizard and return to the Autosampler Control dialog box.

**NOTE:** After you complete the alignment procedure check your purge gas. If the purge gas was turned off during the alignment procedure turn the purge gas back on.



## ***AS 6000 Sample Handling***

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### ***Sample Preparation***

See the sample preparation instructions for the parent instrument.

### ***Sample Pans***

The types of sample pans that you can use for the DSC 4000 and DSC 6000 with autosampler are all sizes of the standard robotics sample pans that are available. The sample pans must be sealed using the Universal Crimper press which comes with the system. The capacities for the sample pans are 10, 25, 30, and 50  $\mu\text{L}$ .

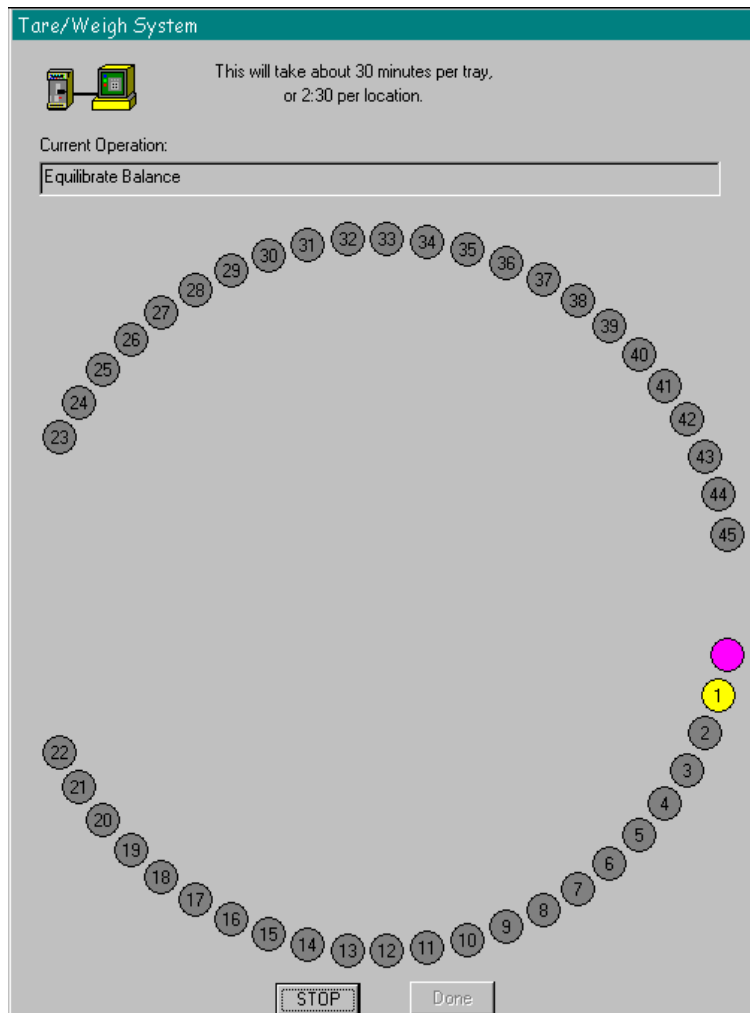
### ***Sample Loading***

Before loading the sample into the crucibles for the TGA 4000 or STA 4000, you will have to tare the crucibles, that is, have the system weigh them so the weight of the crucible is not included in the data. Load the empty crucibles into the sample trays. You can do this with the trays off of the autosampler or while they are still in place. After filling the locations with crucibles, carefully place the trays back onto the autosampler, making sure that the two "buttons" on the bottom side of each tray engage the holes in the autosampler. If you keep the trays on the autosampler, load the sample into the crucible while it is off the tray and then use tweezers to place the crucible onto the tray.

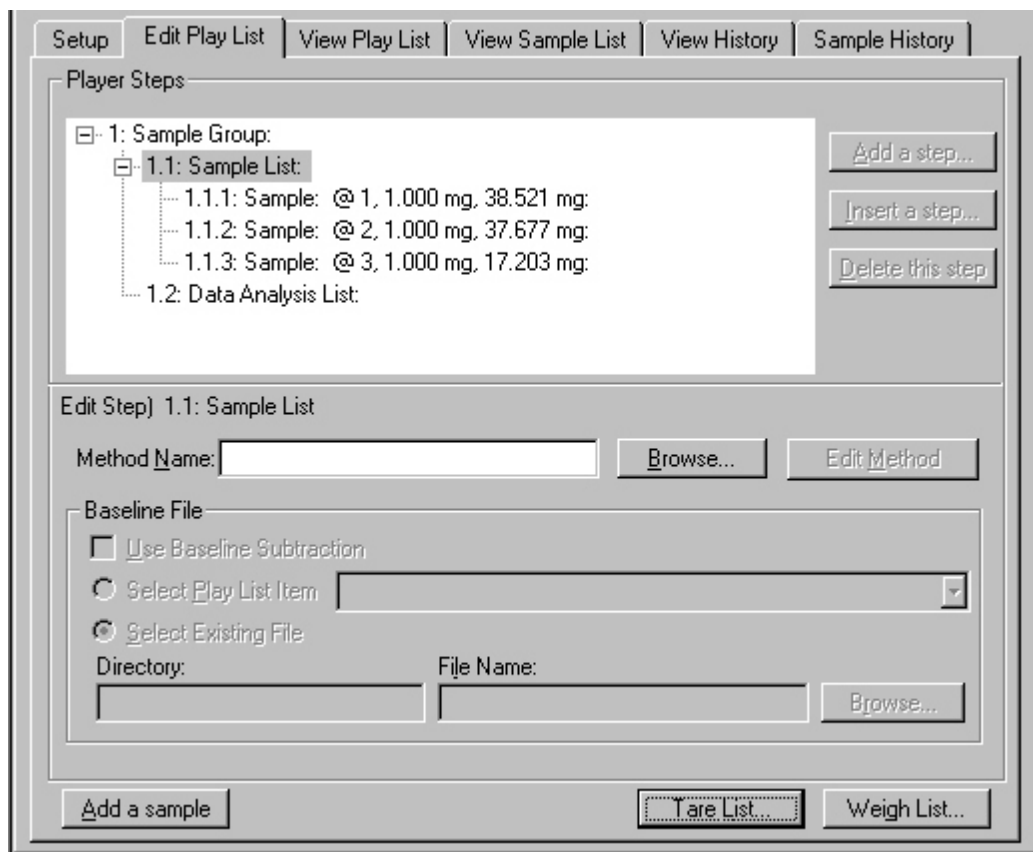
You can tare all of the crucibles in the tray using the Pyris Player Tare All feature. To do this your play list must contain a Sample Group. If you are creating a new play list, select Sample Group as the first entry. If you are using an existing play list, it should have a Sample Group in it in order to use the tare feature.

In a new play list, with Sample Group highlighted, click the **Tare All** button to display the Advanced Tare Options dialog box. If you choose **This Group Only; Populate from Tray**, the Sample List is filled with a line for each position in the trays that contains a crucible and the tare weight of the crucible is included. You can also add sample lines yourself and then select **This Group Only; Sample List Items Only** from the dialog box. Only the crucibles in the positions that you selected for your Sample List will be tared.

When you click the **OK** button in the Advanced Tare Options dialog box, the Tare/Weigh System screen appears:



The Sample List will now display the tare weights of the crucibles:



Once the empty crucibles are tared, remove each crucible and load the prepared sample into each one. Return the crucible to the correct position in the tray. Now the system can weigh all the samples before running the play list, or you can have each sample weighed at the beginning of its run. With Sample List highlighted, click the **Weigh List** button. The Tare/Weigh System dialog box appears. The system automatically starts the program to weigh each sample in the list. After the last sample, if there was a missing sample encountered, a message is displayed:

**The following samples could not be weighed. It is possible that the crucibles are empty.**

A list of samples follows. Click the dialog box's **Close** button to clear the box.

Click the **Done** button in the Tare/Weigh System window. The Sample List will now display the weights of each sample.

For efficient use of the autosampler and to increase sample throughput, you can use remove one tray after the samples have been run and replace the crucibles with empty ones while the samples in the other tray are being run.


## ***Running a Play List with the AS 6000***

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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 also a Sample Group. The Sample Group simplifies grouping like samples together (as you would have in a sample tray of the autosampler). These like samples use the same test method and data analysis as part of the Sample Group. 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.

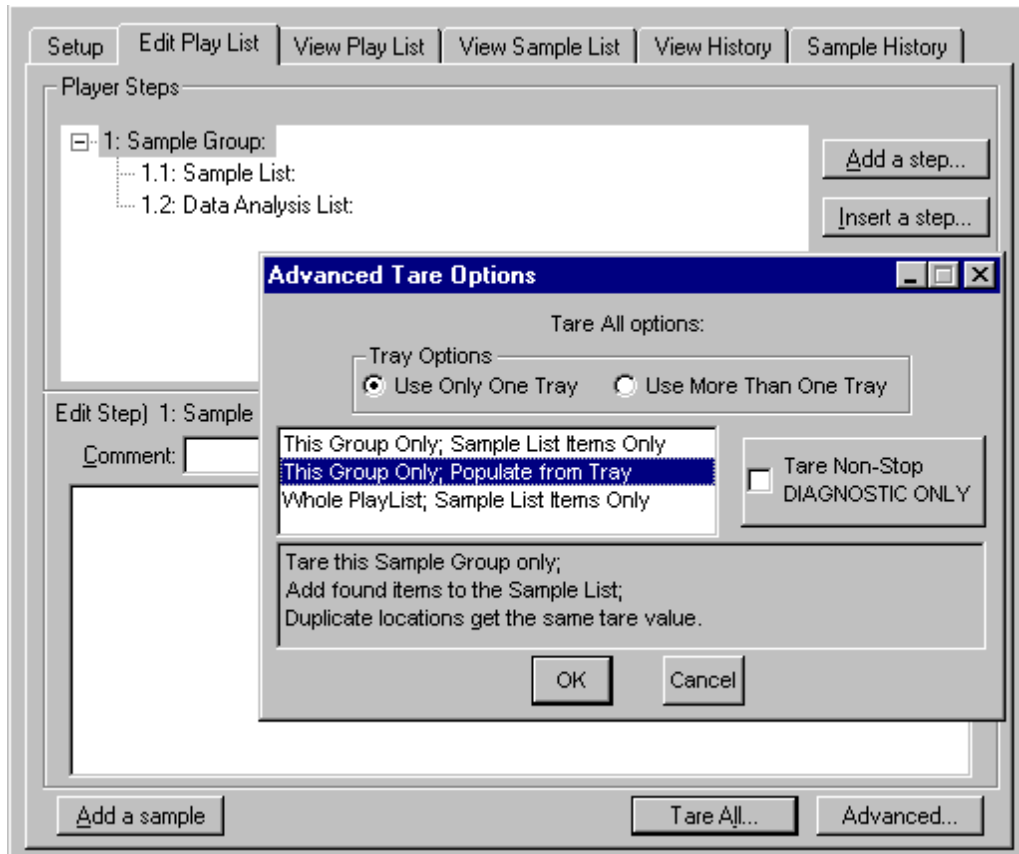
Before starting a run, perform the steps below:

**NOTE:** Pyris play lists created using previous version of Pyris software are usually compatible in later versions of Pyris. And play lists created for one instrument may be compatible with other instruments of the same type. However, play lists created in later versions of software may not be compatible with earlier versions of software.

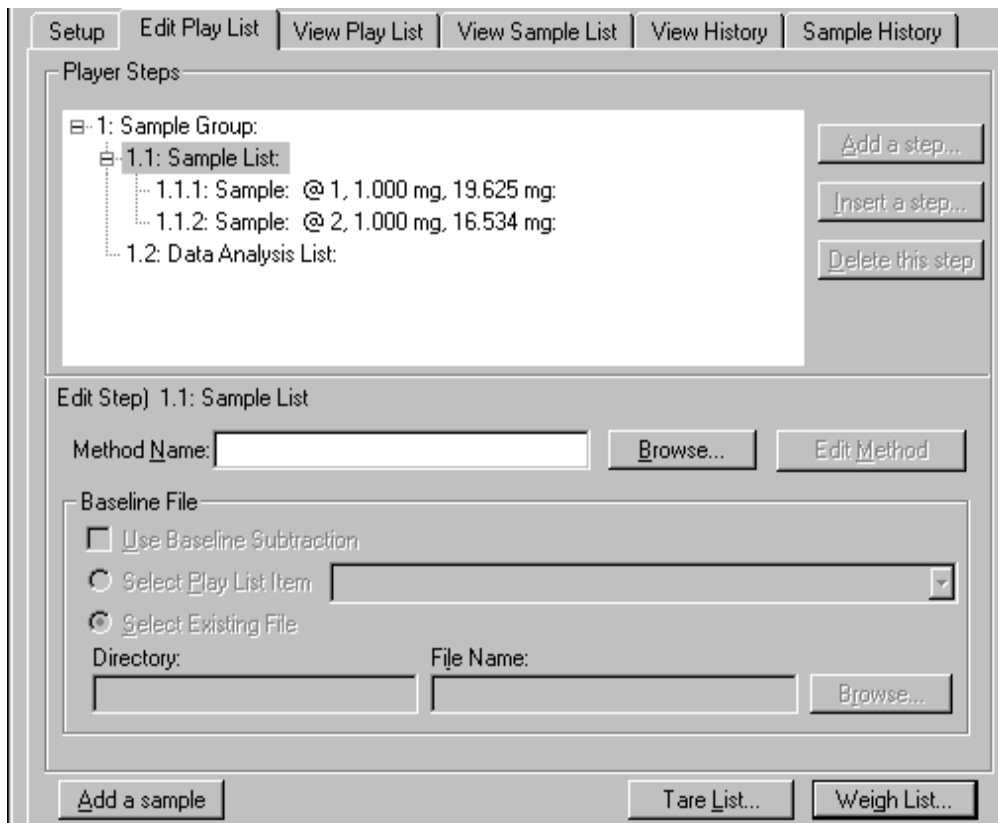
1. Review the safety and warning notes for the analyzer.
2. Verify that the purge gas (if you are using it) and cooling device tubes are properly connected at the rear of the instrument.
3. Turn on the gas and adjust the pressure.
4. Turn on the chiller and adjust according to its instructions.
5. Verify that the electrical and cable connections between the computer and the instrument and other components of the system are properly connected.
6. Turn on the power to the system components.
7. Prepare your samples.
8. Start Pyris software and click the parent instrument's button in the Pyris Manager. Click the Pyris Player button on the toolbar: . Either open an existing play list or create a new one.

There are many ways to use the play list with the autosampler. A quick way to create a play list for an STA 6000 or a TGA 4000 using the Sample Group feature is given below:

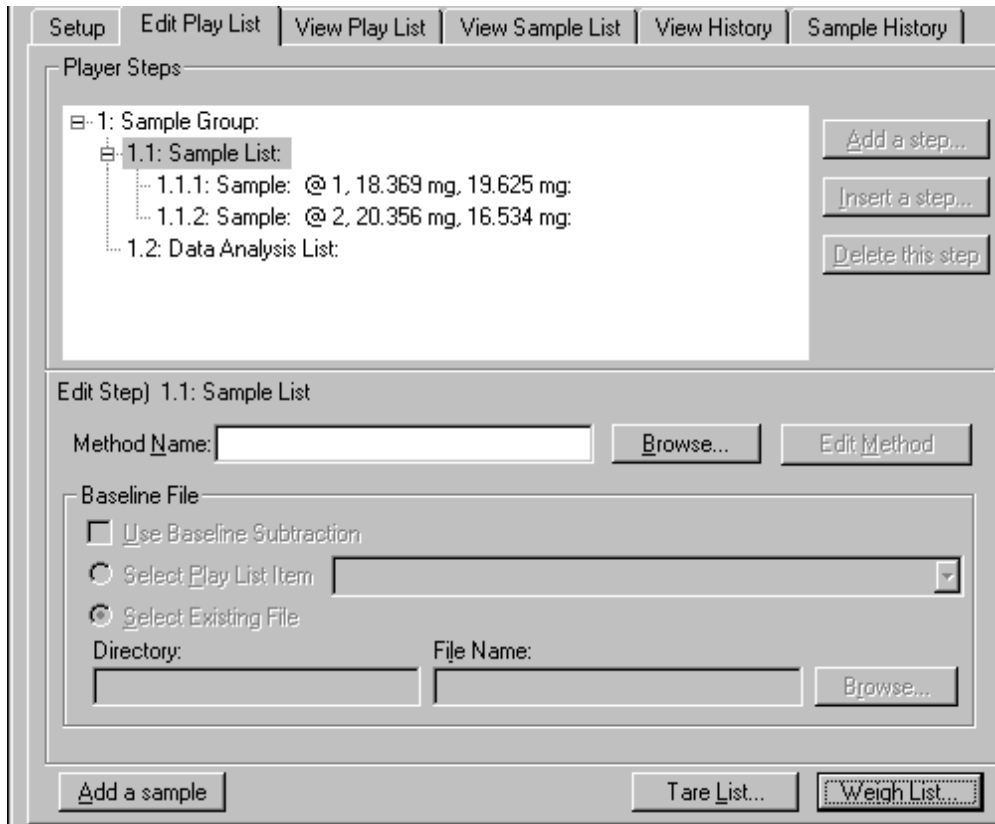
- Load empty sample pans into the locations you want to use in the sample tray. Create a new play list that contains only a Sample Group. Click the **Tare All** button. In the Advanced Tare Options dialog box, select **This Group Only; Populate from Tray**.



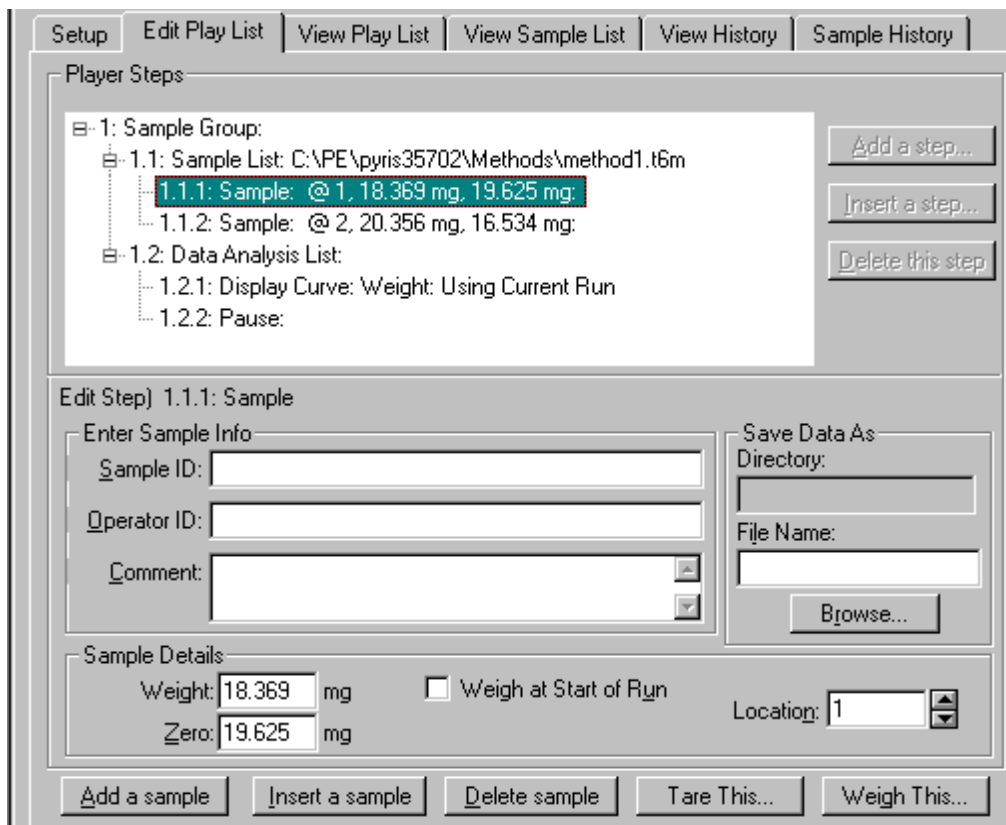
- Click **OK** and the Tare/Weigh System dialog box appears. The AS 6000 begins the procedure of taring the sample pans present on the sample trays. As it finds and tares each pan, it populates the Sample List and includes the tare weight for the sample pan in the Sample line. When taring is complete, click the **Done** button. The Sample List will be filled in.



- Remove the sample trays from the autosampler, or leave them in place, to load the crucibles. If you removed the trays, carefully return them to the autosampler and make sure that the two knobs on the bottom of each tray engage the holes in the autosampler plate.
- Now you can have the system weigh each sample in the list. You can also choose to have each sample weighed right before the run starts. To weigh all the samples before starting the play list, highlight the Sample List line. A message informing you that a method has not been selected is displayed. You can select the method after the samples have been weighed. Click **Weigh List**. The Tare/Weigh System screen appears and the system begins to weigh the samples listed in the Sample List. When finished, the weights are displayed, along with the tare weights, in each Sample line.



- Next you need to add some items to the Data Analysis List. If you have not entered a method for the Sample List, however, you cannot fill in the Data Analysis List. To enter a method, highlight the Sample List line. Type in the name of the method in the Method Name field or click the **Browse** button and find and select the method you want to use. Edit the method's program and initial state parameters by clicking the **Edit Method** button. When finished editing, close the Method Editor by clicking the window's **Close** button in the upper-right-hand corner.
- Click the Data Analysis List line and then **Add a step**. Select **Display Curve**. The default selection in the Edit Step area is **Use Current Run**. After adding the display of a curve, addition items are listed in the Player Step Options box when you add another step.



9. Save the play list by selecting Save Player from the File menu.
10. Start playback of the play list by clicking the Start at Top button or the Start at Current Item button on the Player toolbar. These are the first two buttons on the toolbar:



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

**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.

This is just one example of using a play list with the AS 6000 autosampler. The AS 6000 autosampler on a DSC 4000 or DSC 6000 does not have the Tare/Weigh feature, so the creation of a Sample Group is not automated. You will have to weigh the samples before placing them in the sample pans and crimping the pans. You have to enter the weights into the Sample Details area of the Edit Step: Sample section of the dialog box.

## ***Troubleshooting***

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When the AS 6000 autosampler malfunctions, in most cases it will generate an error message which is sent to the computer and is displayed in the Pyris software. The troubleshooting list below should be used if the AS 6000 malfunctions.

To continue normal operation with the parent instrument, switch off the instrument and switch it back on after 10 seconds. Depending on the error, normal operation may or may not be possible. If normal operation can not be resumed, the problem has to be resolved by a service engineer.

<b>Error message ID</b>	<b>Error</b>	<b>Possible Cause</b>	<b>Corrective Action</b>
ERR_NO_12VOLT	No +12 V power supply available (stepper motor power supply)	Overload condition in switched power supplies  12 V power supply not OK	Check stepper motor wires  Replace AS 6000 assembly  Replace controller PCB
ERR_HOR_STEP_BLOCKED	Horizontal stepper motor blocked	Blocked gear wheels  Blocked by external object	Check gear wheels  Remove external object
ERR_VER_STEP_BLOCKED	Vertical stepper motor blocked	Incorrect calibration  Blocked gear wheels  Blocked by external object  Inner lid of DSC misplaced	Recalibrate the AS 6000  Check gear wheels  Remove external object  Replace DSC inner lid
ERR_DUE_TO_PARENT	Parent instrument has entered fatal error state that results in the AS 6000 also entering error state	Error occurred in parent instrument	Check parent instrument
ERR_GRP_WIRE_BROKEN	Gripper wire is broken or no AS 6000 mechanics connected	Gripper wire is broken  AS 6000 connector not connected  AS 6000 cable not OK	Replace gripper assembly  Connect AS 6000 connector  Check AS 6000 cable



Error message ID	Error	Possible Cause	Corrective Action
ERR_GRP_NO_SAMPLE	<p>Gripper contains no sample when entering furnace. Light switch in state</p> <p>CST_FURN_PUT_SAMPLE</p> <p>during an automatic calibration</p>	<p>There was no sample pan at the reserved position on the sample tray</p>	<p>Place a sample on position 0 of the sample tray</p>
ERR_GRP_NOT_EMPTY	<p>Gripper still holds sample while moving OUT of furnace during state</p> <p>CST_FURN_PUT_SAMPLE</p> <p>or in state</p> <p>CST_PERFORM_TEST</p>	<p>Sample sticks to gripper fingers</p>	<p>Clean gripper tips</p> <p>Use clean sample pans</p>
ERR_LIGHT_SWITCH	<p>Light switches are not functioning or are blocked when the gripper is in raised position</p>	<p>Light switch is blocked</p> <p>Light switch in not OK</p> <p>AS 6000 cable is not OK</p>	<p>Remove object that blocks light switch</p> <p>Replace AS 6000 mechanics</p> <p>Check/replace AS 6000 connection board</p>
ERR_MICRO_SWITCH	<p>Microswitch is activated when gripper is NOT in reset state</p>	<p>Microswitch is not OK</p> <p>Microswitch wiring not OK</p> <p>AS 6000 cable not OK</p>	<p>Replace gripper assembly</p> <p>Check microswitch wiring</p> <p>Check/replace AS 6000 connection board</p>

Error message ID	Error	Possible Cause	Corrective Action
ERR_FURN_NOT_EMPTY	Furnace still contains a sample while trying to load a new one. This error can be generated only after loading at least one sample after power on, as the instrument cannot detect if a sample is present in the furnace from a previous power on period	<p>Failure picking sample out of furnace</p> <p>Incorrect horizontal baseplate adjustment</p> <p>Incorrect horizontal calibration</p> <p>AS 6000 run was interrupted after putting a sample in furnace and restarting</p>	<p>Check if gripper fingers are bent. Replace gripper assembly</p> <p>Adjust baseplate</p> <p>Recalibrate the AS 6000</p> <p>Reset parent instrument and setup link again</p>
ERR_NOT_AT_SENS	Sensor position not reached during autocalibrating the sensor position	Vertical stepper missed steps because it was blocked while running at slow speed. (During slow gripper speed, the stepper blocked detection is disabled.)	Check the DSC lower lid position
ERR_NO_ZERO_DETECT	Zero position is not detected	<p>No detection of the gripper fingers because of direct sunlight at the light switches</p> <p>Gripper assembly displaced</p>	<p>Do not place instrument in direct sunlight</p> <p>Align gripper assembly</p>

## **Error Messages from the DSC 4000**

<b>Error message ID</b>	<b>Error</b>	<b>Possible Cause</b>	<b>Corrective Action</b>
ERR_DUE_TO_AUTOSAMPLER	AS 6000 entered fatal error state. This caused DSC to enter fatal error state	Refer to AS 6000 error message	Refer to AS 6000 troubleshooting (above)
ERR_AUTOSAMPLER_RESPONS	Timeout on command. Response from command transmitted to AS 6000	I2C connection cable defective	Replace I2C cable
ERR_AUTOSAMPLER_I2C_WD	No link between DSC and AS 6000 via I2C	I2C connection cable defective	Replace I2C cable

## ***AS 6000 Autosampler Maintenance***

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The exterior surfaces of the AS 6000 may be cleaned with a soft cloth, dampened with a mild detergent and water solution.

The bushings of the gripper fingers should also be kept clean. Use a cotton swab dampened with a mild detergent and water solution to carefully wipe them clean. Use the same method to clean the two sample trays.

### ***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.



**WARNING**

*There are no user-serviceable parts inside the autosampler. Refer all servicing to a qualified PerkinElmer Instruments service engineer.*



**AVERTISSEMENT**

*Il n'y a pas de pièces réparables par l'utilisateur à l'intérieur de l'échantillonneur automatique. Renvoyez tous les services à un ingénieur de service qualifié PerkinElmer Instruments.*

## ***Install a Reference Pan in a DSC 4000, DSC 6000, Pyris 6 DSC or Jade DSC with AS 6000 Autosampler***

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When running samples with the DSC 4000, DSC 6000, Pyris 6 DSC or the Jade DSC it is recommended that a reference capsule be used in the furnace. The best reference material is an empty sample pan and lid of the same type in which the sample material is encapsulated. Because the autosampler and its base plate cover access to the reference pan area, they have to be removed first. The procedure to do this and place a reference pan inside is as follows:

1. Make sure that the power is off.
2. Remove both sample trays.
3. Remove the four screws that attach the autosampler to the base plate.

**NOTE:** Never remove the screws from the plate that is directly attached to the analyzer's cover. This plate is positioned with respect to the furnace. The positioning is performed by the service engineer upon installation.

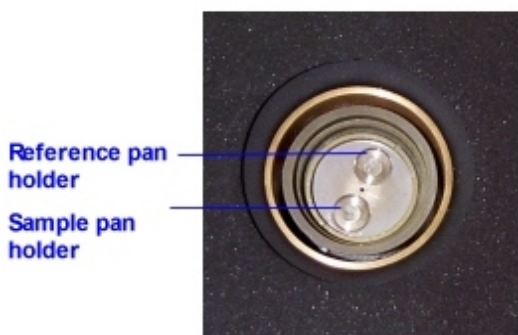
4. Place the tips of your fingers in the indents in the sides of the base plate and lift the autosampler to disengage the D connector and the two standoffs on the base plate.
5. Remove the upper furnace lid and the base plate lid holder.



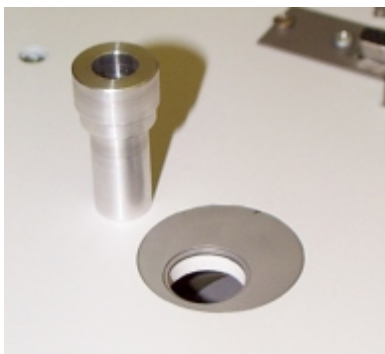
6. Remove the lower furnace lid and the lower furnace lid holder.



This exposes the reference pan position. The sample pan position is the one closer to the side of the instrument. The reference pan position is closer to the center of the instrument.



7. Place a reference pan on the reference position.
8. Replace the lower furnace lid holder.
9. Replace the base plate lid holder.
10. Use the furnace lid holder alignment tool to position the lower furnace lid holder properly with respect to the upper lid holder.



Make sure that the tool is all the way down.

11. Look down through the tool and make sure that the furnace is centered in the tool.
12. Carefully remove the alignment tool without disturbing the lower furnace lid holder.
13. Place the autosampler on top of the base plate, aligning the two holes near the D connector that engage the two standoffs on the base plate. Gently push the autosampler down to snap in place.
14. Attach the autosampler to the base plate with the four screws.
15. Place the sample trays on the autosampler with tray 1–22 toward the front and tray 23–45 on the rear.

After placing a reference pan within the instrument and replacing the autosampler, you should run through the gripper alignment procedure to make sure that the gripper aligns with the sample pans at locations 12 and 33.