STA 8000 Installation and Hardware Guide



Part Number	Release	Publication Date
09931395	В	March 2018

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

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Or email http://www.perkinelmer.com/contactus/

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Introduction

Pyris Installation

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

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

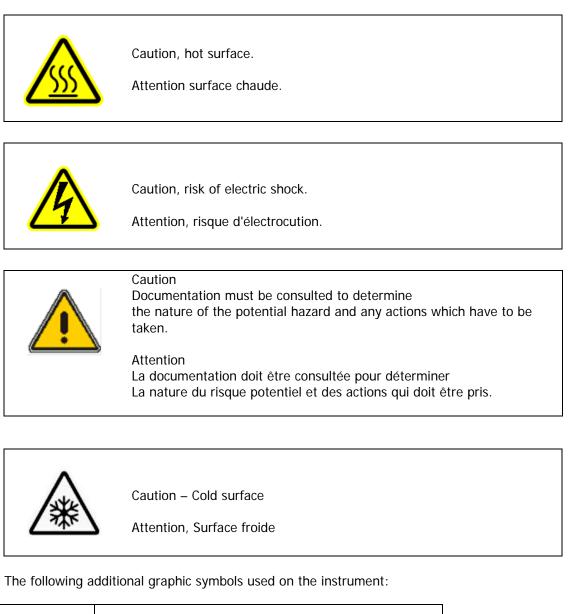
Three terms, in the following standard formats, are also used to highlight special circumstances and warnings.

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.
D	Caution (Achtung) Bedeutet, daß die genannte Anleitung genau befolgt werden muß, um einen Geräteschaden zu vermeiden.
DK	Caution (Bemærk) Dette betyder, at den nævnte vejledning skal overholdes nøje for at undgå en beskadigelse af apparatet .
E	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.
F	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.
NL	<i>Caution (Opgelet)</i> Betekent dat de genoemde handleiding nauwkeurig moet worden opgevolgd, om beschadiging van het instrument te voorkomen.
P	Caution (Atenção) Significa que a instrução referida tem de ser respeitada para evitar a danificação do aparelho .

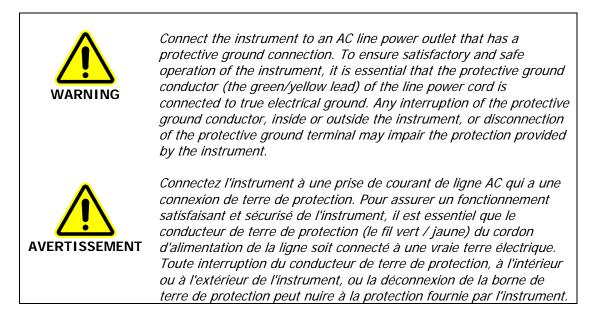
	We use the term WARNING to inform you about situations that could result in personal injury to yourself or other persons. Details about these circumstances are in a box like this one.
	Warning (Warnung) Bedeutet, daß es bei Nichtbeachten der genannten Anweisung zu einer Verletzung des Benutzers kommen kann.
DK	Warning (Advarsel) Betyder, at brugeren kan blive kvæstet , hvis anvisningen ikke overholdes.
E	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.
F	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.
NL	Warning (Waarschuwing) Betekent dat, wanneer de genoemde aanwijzing niet in acht wordt genomen, dit kan leiden tot verwondingen van de gebruiker.
P	Warning (Aviso) Significa que a não observância da instrução referida poderá causar um ferimento ao usuário.

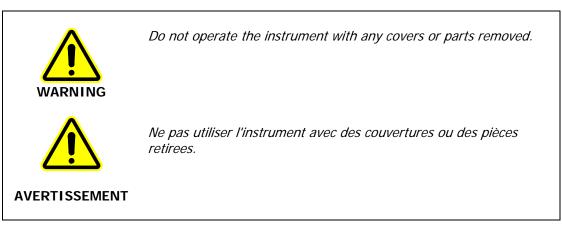
Symbols Used on the Instruments



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

Electrical Warnings







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

N'essayez pas 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.



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

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

AVERTISSEMENT

Electromagnetic Compatibility (EMC)

Europe

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

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

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

South Korea

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

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

United States (FCC)

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

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

Electrical Safety

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

Pollution Degree 2

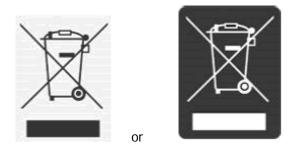
This product will operate safely in environments that contain nonconductive foreign matter up to Pollution Degree 2 in EN/IEC 61010-1.

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

Use of Hazardous Substances

This product meets the requirements of the Restriction of Hazardous Substances Directive 2011/65/EU.

WEEE Instructions for PerkinElmer Products



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

The objectives of this program are to preserve, protect and improve the quality of the environment, protect human health, and utilize natural resources prudently 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

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.

Safety and Regulatory Information . 19

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). The furnace in the STA 6000/8000 must be protected from condensation, for example, by using a dry box.
- For optimum performance, the temperature of the area should be between 10 °C and 35 °C (50 °F and 95 °F).

- 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.

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Install Multiple Analyzers

Install Multiple Analyzers

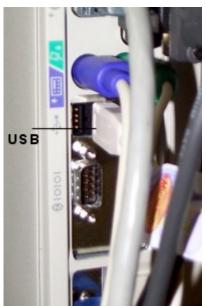
The only way to attach multiple instruments to your computer is via a universal serial bus. PerkinElmer no longer supports the Multiport RS-232 Card (P/N 09402018). Installation of the universal serial bus MUST be done before installing any analyzer.

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

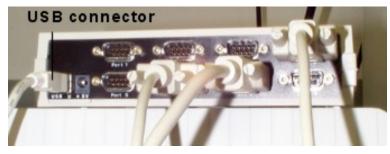
1. Attach one end of the USB cable to one of the USB connectors at the back of the computer.



 Attach the other end of the connector cable to the USB port on the EdgePort. EdgePort/4



EdgePort/8



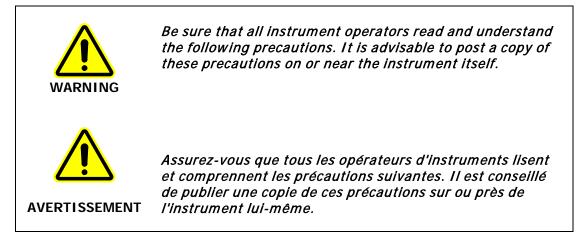
DO NOT connect any instruments to the EdgePort at this time.

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.

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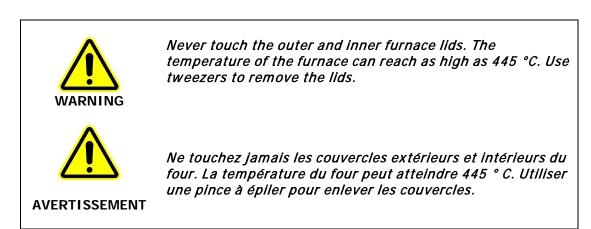
Install an STA 8000

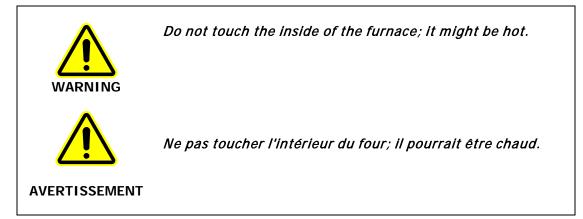
Safety Precautions for the STA 8000

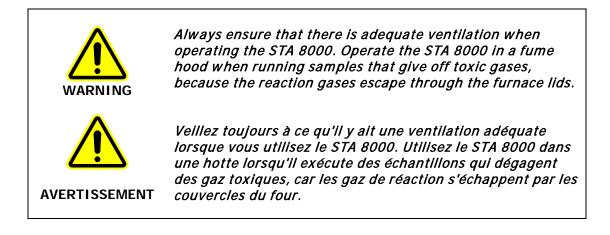


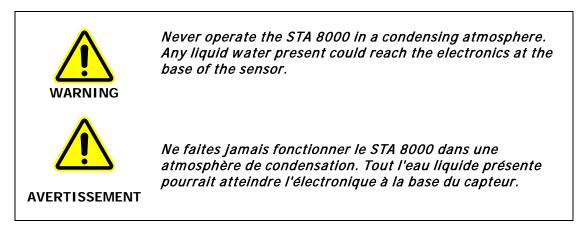
The following precautions must be observed before and during use of the STA 8000:

- Before connecting the STA 8000 to the main outlet, check the main voltage setting and fuse.
- The STA 8000 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 STA 8000 weighs 16 kg. Never attempt to lift the analyzer with 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.









CAUTION	Do NOT expose the sensor surfaces to mechanical stress. If no external cooling is applied, do not operate the STA 8000 above 50 °C.
ATTENTION	NE PAS exposer les surfaces du capteur aux contraintes mécaniques. Si aucun refroidissement externe n'est appliqué, ne pas utiliser le STA 8000 au-dessus de 50 °.

CAUTIONDo not exert excessive forces on the sensor disks.ATTENTIONNe pas exercer de forces excessives sur les disques du capteur.

Important Specifications

Maximum power consumption	450 W
Supply voltage	100-240VAC (+5%, -10%)
Supply voltage frequency	50–60 Hz
Fuses	T6.3A (250VAC type only)
Safe temperature range	10–40 °C
Dimensions L x H x D	36 x 20 x 35 cm
Weight	16 kg

Decontamination and Cleaning

Decontamination

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

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

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

http://www.perkinelmer.com/Content/technicalinfo/dts_instrumentdeconprocedure.pdf

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

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

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

Cleaning the Instrument

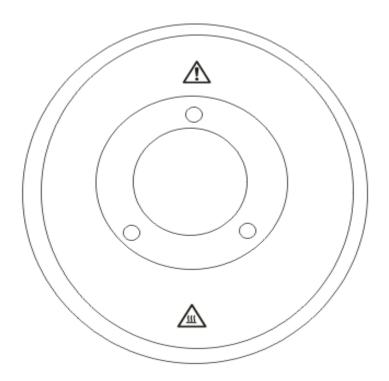
Exterior surfaces may be cleaned with a soft cloth, dampened with a mild detergent and water solution. Do not use abrasive cleaners or solvents.

General Laboratory Safety

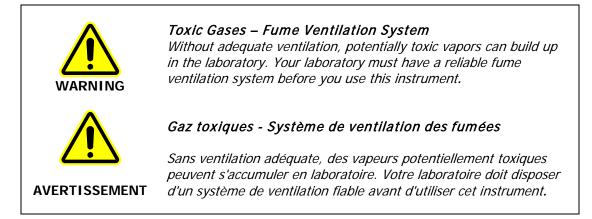
Your laboratory should have all equipment ordinarily required for the safety of individuals working with chemicals (fire extinguishers, first-aid equipment, safety shower and eye-wash fountain, spill cleanup equipment, etc.).

STA 8000 Warning Labels

Warning Labels on the Upper Ring Around the Furnace

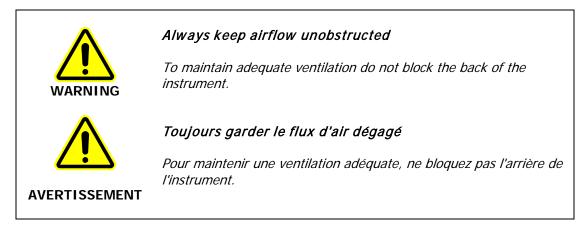


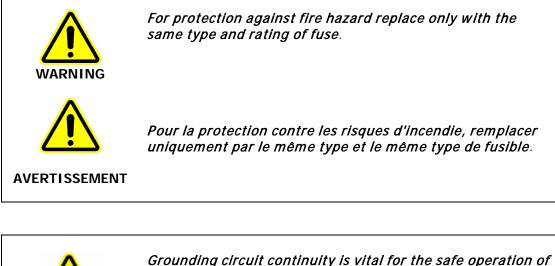


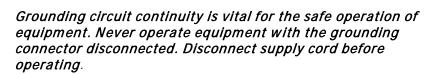


Warning Labels on the Back of the STA 8000











WARNING

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.

Installing an STA 8000

The installation procedure for the STA 8000 consists of the following steps:

- Prepare the Laboratory (refer to page 22)
- Unpack the STA 8000
- Set Up the STA 8000 System Components
- Connect the STA 8000 System Components
- Configure the STA 8000
- Calibrate the STA 8000

Unpacking the STA 8000

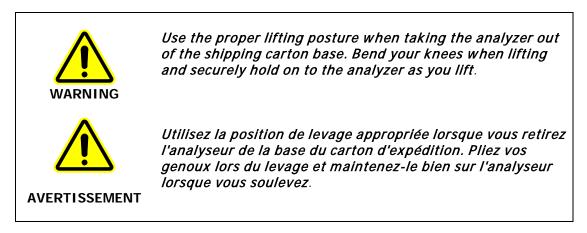
The STA 8000 system consists of the following components:

- STA 8000
- Personal Computer
- Printer (Optional)
- Chiller (Optional)

The STA 8000 is able to operate using mains supply voltages of 100–120 VAC or 200–240 VAC. The analyzer automatically adjusts for the supplied voltage; no manual adjustments are needed.

The STA 8000 comes wrapped in plastic and is surrounded by foam on all sides of the analyzer. 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, resting the analyzer on its left or right side 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.

It is recommended that you save the shipping foam should the analyzer require shipment to another facility in the future.

Parts Included

The STA 8000 comes in two boxes and consists of the following components:

- STA 8000
- Pyris software
- Spares and Accessories Kit

The Spares and Accessories Kit contains the following parts:

Item	Part Number	Quantity
Sensor	N5202007	1
Indium calibration standard	03190033	1
Aluminum calibration standard	N5380057	1
Gold calibration standard	N5380058	1
Palladium calibration standard	N5380071	1
Aluminum oxide powder (1 oz bottle)	04190197	1
Tweezers (for sensor installation and loading pans)	09908400	1
Tweezers (for sensor removal)	N5202093	1
Calibration weight	N5200042	1
Ceramic sample pans (pack of 3)	N5200040	2
Ceramic lids (pack of 6)	N5202032	1
Tubing adaptor (1/8 in. to 1/4 in.)	09903906	1
Gas restrictor	01541498	1

Also included with the STA 8000 are:

- Tygon tubing for connection of liquid coolant to the cooling chamber.
- Hose clamps.
- Teflon tubing for purge gas connection.
- Nickel-plated 5/16 in. tubing (2 pieces) for use on Swageloks on the cooling liquid connections.
- Level indicator.
- RS-232 cable for communicating to the computer.
- Spare fuses.
- Power cord.
- CD containing calibration information.

The procedure for unpacking the STA 8000 is complete.

Set Up the STA 8000 System Components

Place the STA 8000 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 instrument's instructions.

Be sure to set up all of the system components listed below before configuring the STA 8000:

- Install the purge gases
- Remove the shipping/alignment tool
- Install the sensor
- Install the cooling device

Connect the Purge Gas Supply to the STA 8000

The STA 8000 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 sensor/heat exchanger assembly with dry gas. This facility must ALWAYS be used when working with the analyzer. The purge gas, preferably dry nitrogen, prevents condensation of water vapor on the measuring cell. The system purge gas flow is controlled by a needle valve inside the analyzer, and is preset to a flow rate of 45 ml/min.

NOTE: The gas pressure must be regulated by the user to a maximum of 90 psi (6.2 bar). A pressure of 30–45 psi (2–3 bar) is suitable for most experiments.

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 Pyris software, and can be between 0 and 200 ml/min.

NOTE: The gas must be dry. A flow rate of 20-40 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 and system purge gas lines to the STA 8000.

For more information on the purge gases, see *Sample Handling* on page 57.

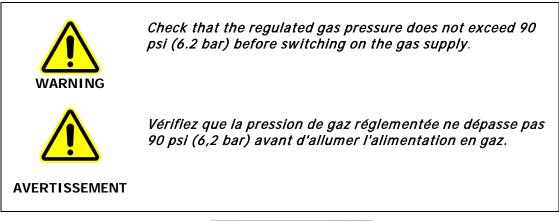
Connect the Purge Gas and System Purge Gas Lines to the STA 8000

Perform the following procedure to connect your sample purge gas and system purge gas lines to the STA 8000 analyzer regardless of the other components in the purge gas line. Assuming all the other components of the purge gas line are installed, you will now connect 1/8 in. Teflon tubing to the STA 8000 using a female connector.

The STA 8000 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 ALWAYS REQUIRED.
- 2. Place the 1/8 in. Teflon tubing from your Sample Purge Gas line onto the inlet labelled sample purge A.

If required, a second sample purge line (1/8 in. Teflon tubing) can be connected to the purge B inlet.





When using the STA 8000 in environments of high relative humidity, a mini dry box (P/N N5202070) is recommended to prevent condensation. This acts simply to protect the furnace from condensation, and is not connected to the dry box purge inlet.

NOTE: The dry box purge inlet is non-functional in the STA 8000.

Removal of the Shipping/Alignment Tool

The STA 8000 is shipped with a tool inserted through the furnace into the balance mechanism to minimize vibration during transportation. This tool is also used to align the system after replacing a furnace or balance. It is important that you keep this tool in a safe place. It is also recommended that you save the shipping foam should the analyzer require shipment to another facility in the future.

- 1. Remove the furnace cover and the foam inserts.
- 2. Grip the upper part of the shipping/alignment tool using flat tweezers and carefully lift it straight upwards and out of the furnace.

To replace the shipping/alignment tool:

Insert the tool into the hole in the bottom of the furnace.
 The flat plate must be pointing towards the front of the analyzer.

To prepare the analyzer for shipment:

- 1. Position the two-part foam insert on top of the shipping/alignment tool within the furnace.
- 2. Place the cylindrical foam insert on top of the first foam insert.
- 3. Replace the furnace cover and tape it shut.

Install the Sensor

- 1. Switch on the STA 8000.
- 2. Ensure that the shipping/alignment tool is removed.

CAUTION	The STA 8000 sensor is a fragile part due to the breakable ceramics used, and must be handled with care. Until the sensor is placed in the analyzer, it should be kept in its box.
ATTENTION	Le capteur STA 8000 est une partie fragile en raison de la céramique cassable utilisée et doit être manipulé avec précaution. Jusqu'à ce que le capteur soit placé dans l'analyseur, il doit être conservé dans sa boîte

- Remove the sensor from its box.
 Avoid touching it with bare hands.
- 4. Using the pointed tweezers provided (P/N 09908400), grip the sensor by the thicker area at the top of the rod.



5. Position the sensor just above the hole in the bottom of the furnace.

The three electrical contacts must point towards the front of the analyzer.

- Carefully lower the sensor into the hole, keeping it in an upright position.
 Do not rub the electrical contacts against the furnace insulation.
- 7. When the sensor encounters resistance, release the rod.
- 8. Press the top of the rod GENTLY with the tweezers and push the sensor downward a further 2–3 mm until it does not go any further.

As the sensor makes contact with the balance mechanism, a beep will sound.

CAUTION Do not push down on any part of the sensor except the top of the rod.

ATTENTION *Ne poussez pas sur n'importe quelle partie du capteur, sauf le haut de la tige.*



To remove the sensor:

Grip the sensor at the top of the rod with tweezers, and pull it straight upwards and out of the furnace.

Two short beeps will sound.

NOTE: Always place the sensor in its box if it is not going to be put back in the instrument immediately. Replace the shipping/alignment tool if the analyzer is to be moved.

Connect the Cooling Device to the STA 8000

The STA 8000 has an integrated cooling jacket that accepts various types of cold gases and liquids. The recommended cooling system is the Polyscience Chiller, which operates between –20 and 200 °C. The recommended temperature range of the coolant is 15–25 °C.

CAUTIONLow temperature cooling should only be used with caution on the STA
8000, as any condensation in the furnace area could damage the
electronic circuits.ATTENTION

Le refroidissement à basse température ne doit être utilisé qu'avec prudence sur le STA 8000, car toute condensation dans la zone du four pourrait endommager les circuits électroniques.

NOTE: A mini dry box accessory (P/N N5202070) is available which sits on top of the furnace and is recommended to help prevent condensation forming when the furnace is at low temperature in a humid atmosphere.



Connecting to the Chiller

NOTE: An appropriate fluid must be used with the chiller. Distilled water is recommended for operation at temperatures between 10 °C and 90 °C. Alternatively, normal tap water can be used. For operation at –20 °C to 100 °C, 50% ethylene glycol in water is recommended.

CAUTIONEnsure that the cooling air vents at the front and rear of the cooler are
not blocked. Maintain a minimum space of 15–20 cm.ATTENTIONAssurez-vous que les évents 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.

When using the chiller, connect the cooling supply to the STA 8000 as follows:

1. Make sure that the chiller is switched off but that its power cord is plugged into a power supply.

- 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.
 Couplings suitable for several different hose sizes are provided with the chiller.
- 3. Push a hose (compatible with the desired coolant) onto the OUTLET coupling and secure with a hose clamp.
- 4. Slide the other end of the hose over the metal tube labelled COOLING LIQUID IN on the thermal analyzer.

Place a hose clamp around the hose and metal tube to secure the hose in place.

- 5. Screw a barbed hose coupling into the INLET connection on the chiller unit, and tighten with a suitable wrench.
- 6. Push a hose onto the INLET coupling and secure with a hose clamp.
- 7. 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

- 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.
- 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).
- 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 control unit will continue with its start-up sequence and then display "Standby" when ready.
- Set the Safety Set Point on the control unit using a flat screwdriver to rotate the control to the maximum temperature to which the bath should be heated.
 To ensure that the analyzer performs correctly, we recommend a maximum temperature of 45 °C for the cooling supply.
- 5. Press the Power switch on the front panel. The pump will begin operating.
- 6. Top up the coolant reservoir as needed 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 cooling jacket of the STA 8000 will NOT accept pressure. Use the lowest flow rate that provides sufficient cooling for your experiment to minimize the pressure in the system.
ATTENTION	La gaine de refroidissement du STA 8000 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.

NOTE: 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.

Setting the Coolant Temperature

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

- 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. A value of 20 °C is recommended.
- 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 STA 8000.
- 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 cooling inlet and outlet are located at the rear of the analyzer and are correspondingly marked Cooling Liquid In and Cooling Liquid Out. In order to connect the cooling supply to the STA 8000, connect the black tubing supplied to the inlet and outlet (outer diameter is 8 mm). When using tap water, connect the inlet to the tap and the outlet to a suitable drain. When using a circulator, connect the inlet of the STA 8000 to the outlet of the circulator, and the outlet of the STA 8000 to the inlet of the circulator.

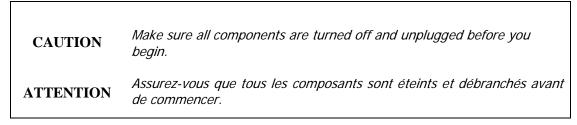
When using tap water, the temperature should not change more than 0.4 °C/hour.

Γ

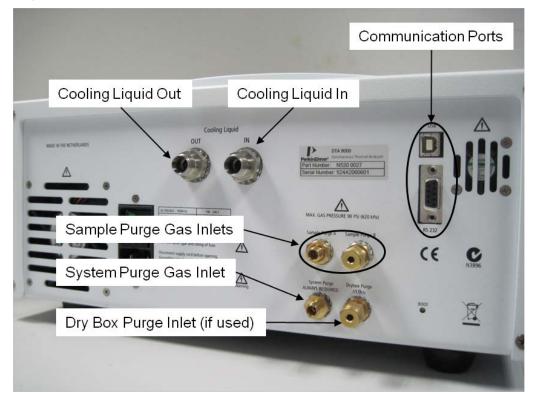
CAUTIO	N The cooling jacket of the STA 8000 will NOT accept pressure. Use the lowest flow rate that provides sufficient cooling for your experiment to minimize the pressure in the system.
ATTENT	ON La gaine de refroidissement du STA 8000 n'acceptera PAS la pression. Utilisez le débit le plus bas qui procure un refroidissement suffisant pour votre expérience afin de minimiser la pression dans le système.
free of	ssential to maintain a very constant flow rate of water. The liquid should be air bubbles. Changes in the liquid flow rate or the presence of air bubbles will the quality of the measurement signal.

Connect the STA 8000 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 STA 8000 has to be connected to the computer.



The image below shows the connections on the STA 8000:



- 1. Connect one end of the RS-232 cable to the back of the STA 8000.
- 2. Connect the other end of the RS-232 cable to COM1 on the back of the computer.
- 3. Connect the printer cable to the USB port.
- 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
 - STA 8000
 - Printer
- 6. Turn on the purge gas supply, and the water for the STA 8000 cooling system or any other accessories for the cooling device you are using.

Configure the STA 8000

The Pyris software must be configured for the STA 8000. This must be done before the STA 8000 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.

Pyris Configuration		×
Analyzers: STA 8000	Ports:	▲ <u>Add Analyzer</u> <u>R</u> emove <u>E</u> dit
<u>H</u> elp		Close

- Click the Add Analyzer button. The Add Analyzer dialog box appears.
- 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 Converter.
- 4. Click the Add button.

The STA 8000 Configuration dialog box is displayed. The system detects the type of analyzer that is attached and displays the default name and other information for an STA 8000 in this dialog box.

- 5. Select the accessories that are attached to the analyzer.
- 6. Click **OK** to accept the selections.
- 7. 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.

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Once Pyris Software has been configured for the STA 8000, the STA 8000 Application can be started from Pyris Manager.

Starting the Pyris Manager

- 1. Select **Pyris Manager** from the Pyris Software group in Programs on the Start menu. The Pyris Manager is displayed on the screen.
- 2. Select **STA 8000** from the Pyris Manager. The STA 8000 control panel is displayed.
- 3. Heat the sensor to approximately 150 °C to burn off any excess moisture.

Enter 150 in the box beneath the Go To Temp button ______ on the control panel and click the button.

4. After 10–15 minutes, cool the analyzer to ambient temperature.

The configuration for the STA 8000 is complete. The analyzer is ready to be calibrated.

Calibration of the STA 8000

The STA 8000 has been calibrated at the factory for both temperature and heat flow. 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. Under normal conditions, the only calibrations required at installation are the weight calibration and baseline optimization. The weight calibration should also be performed if the analyzer is physically moved, and the baseline optimization should be repeated whenever there is a substantial change in chiller temperature or purge gas flow rate, or when the sensor is replaced.

We do recommend that the temperature and heat flow calibrations are verified using the samples of indium and gold (which are provided in the Spares and Accessory Kit) before obtaining data. When installing a new analyzer, carry out these steps in order:

- 1. Weight Calibration and Verification
- 2. DTA Baseline Optimization
- 3. Temperature Verification
- 4. Heat Flow Verification

For general guidance on loading samples into the analyzer, see Sample Handling on page 57.

Weight Calibration

- 1. Once all the system components are installed and the system purge and coolant flow have been turned on, leave the instrument for 4–8 hours to fully equilibrate and stabilize the balance to the laboratory environment.
- 2. Place a ceramic pan on each platform of the sensor.
- 3. Open the calibration routine and click **Start Calibration**.
- 4. Enter the value of the calibration weight.Use a Class '0' 100 mg weight or the weight provided with the STA 8000.
- 5. Click **Run Calibration** and follow the instructions on the screen.
- 6. At the end of the calibration, save and close the calibration.
- 7. Remove the weight.

Weight Verification

- 1. After the balance has stabilized and the weight has been calibrated, tare an empty pan, place the calibration reference weight provided into the sample pan and record the weight displayed in the status panel.
- 2. Verify that the weight reading is in good agreement with the certified value of the calibration reference weight.

DTA Baseline Optimization

This optimizes the Differential Thermal Analyser scanning baseline between two temperatures selected by the user. The resultant DTA curve is fitted to an equation that flattens the baseline. The process takes 1–2 hours.

In the DTA Baseline Optimization screen, enter the Start Temperature, End Temperature and the Scan Rate, and then click Run Calibration.

Select start and end temperatures which include the range you will use for your measurements. It is important to choose a start temperature below that which you will use in experiments to ensure that the baseline optimization covers the full range of temperatures needed. Use a scan rate that you plan to use for most analyses; suggested values are 10-20 °C/min.

TG Drift Optimization

This process corrects for the variation in weight measurement that occurs as the temperature of the balance alters during an experiment. The balance temperature is affected by the cooling device used with the analyzer. We recommend that you carry out the TG Drift Optimization when the instrument is first installed. Subsequently, it should only be repeated if you change the cooling system.

In the DTA Baseline Optimization screen, click the box marked Perform TG drift optimization (~8 hours).

The optimization routine will be performed using a predefined method (not the same method as the DTA Baseline Optimization). Since the procedure takes approximately 8 hours to complete, we recommend that you run it overnight. If the option is selected, Pyris will run the TG Drift Optimization followed automatically by the DTA Baseline Optimization.

Temperature and Heat Flow Verification

During normal use, it is usually only necessary to verify that the existing calibration gives accurate data by running a test with a standard and checking that the temperature and heat flow measurements agree with the accepted values. An example of a suitable procedure is given below.

NOTE: The recommended calibration standards for temperature in the STA 8000 are indium, aluminum and gold. The user can substitute these for different standards using the calibration wizard in the Pyris software. Palladium should be used instead of gold for experiments where the temperature exceeds 1300 °C. The recommended calibration standards for heat flow are indium and gold/palladium. Refer to the Pyris software Help for more information.

- 1. Place empty sample pans onto both platforms of the sensor, and tare the weights.
- 2. Place a piece of the standard (10–20 mg) into the sample pan (on the left side of the sensor) and record the weight.

The weight can also be entered manually in the Method Editor.

3. Create and run a method to heat the sample in excess of its melting temperature at a scan rate of 20 °C/min.

- 4. After the data have been collected, calculate the onset temperature and heat flow for the melting peak as follows:
 - Click to open Data Analysis, and open the files of the experiments run using the standards.
 - Perform a peak area and onset calculation by clicking Calc > Peak Area and selecting the Onset box.
 - Move the X markers on the chart into position at the beginning and end of the melt peak and click **Calculate.** If the peak does not show a straight leading edge (which makes it difficult to accurately measure the onset temperature), rerun the method after equilibrating at the start temperature.
 - Record the results for each standard.
- 5. Verify that the melting temperature and heat flow data are in good agreement with the values in the table below.
- 6. Remove the reference sample for future use.

The recommended standard materials have the following values for melting temperature and heat flow (from *CRC Handbook of Chemistry and Physics*, 81st edition, 2000, p.12-197):

Standard	Melting Temperature (°C)	Energy (Heat Flow) (J/g)
Indium	156.6	28.6
Aluminum	660.3	399.9
Gold	1064.2	64.6
Palladium	1554.9	157.3

If the results for the standards give temperature or heat flow data that do not correspond to these values, then a new calibration must be performed. Refer to the Pyris software Help for further details.

The installation of the STA 8000 is now complete.

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STA 8000 Hardware

STA 8000 Hardware Overview

The STA 8000 Simultaneous Thermal Analyzer offers performance, reliability and productivity you can depend on. Designed with routine and research applications in mind, the STA 8000 applies leading edge sensor technology to yield higher accuracy and quality results.

The STA 8000 is controlled by PerkinElmer's Pyris software on the computer to which the analyzer is connected. The information it provides is essential to industries such as plastics and polymers; automotive; semiconductors and electronics; adhesives; paints and coatings; fuels; ceramics, clays, and soil; food; pharmaceuticals; and medical devices and equipment.

The STA 8000 has an integrated cooling jacket, in which you can use various types of cooling liquids with temperatures higher than 15 °C (non-condensing). During operation, the cooling liquid flow rate should be between 0.5 and 1 l/min. The cooling liquid should be at a temperature such that there is no condensation inside the analyzer. The recommended cooling device is the Polyscience Chiller (P/N N5370220/N537-0221 for 120 V and 220 V respectively).

The STA 8000 has a top-loading microbalance. There are two platforms that connect to the microbalance via the sensor. When seen from the front of the analyzer, the right-hand platform is for a reference pan, and the left-hand platform is for the sample pan. The top-loading balance has many advantages over a hangdown wire design. With this design, however, placement of the sample in the sample pan and of the sample pan on the platform is very important. The microbalance is very sensitive to sample positioning.

The furnace has a large isothermal zone. The sample and reference positions in the STA 8000 are approximately in the middle of the isothermal zone. This increases reproducibility of temperature measurements.

The rapid cooling rate of the furnace is achieved by mounting the furnace in a liquid-cooled jacket. To reduce the heating power at high temperatures, the cooling jacket is nickel-plated. Also, there is forced air cooling. A small air pump is mounted inside the instrument and activated by clicking the Cooling Air button on the control panel. Liquid cooling is also used to keep the temperature of the balance housing constant. The cooling jacket is mounted on and thermally insulated from the balance housing. Temperature gradients are eliminated as much as possible.

For more information on the STA 8000, refer to the topics below:

- Sample Handling
- Heating
- STA 8000 Maintenance
- STA 8000 Part Numbers

Sample Handling

The STA 8000 measures the change in temperature (relative to a reference) and weight of a sample as a function of temperature and/or time. The materials and techniques used to obtain data with an STA 8000 are discussed below.

CAUTION ATTENTION	Check that the cooling liquid supply is circulating before starting a run. Vérifiez que l'alimentation en liquide de refroidissement est en circulation avant de commencer une course.
CAUTION	Ensure the purge gases are set to the recommended flow rates. A rate that is too high will disturb the inner furnace lid. Assurez-vous que les gaz de purge sont réglés sur les débits recommandés. Un taux trop élevé perturbera le couvercle intérieur du four.
CAUTION ATTENTION	Do not open the furnace when it is at an elevated temperature (>100 °C). Ne pas ouvrir le four lorsqu'il est à une température élevée (> 100 °C).

Sample Preparation

The instrument analyzes solid samples in powder, crystal, or granular form. Although quantitative weight loss accuracy is mostly unaffected by sample shape, the qualitative appearance of a run may be affected by the sample configuration. The best sample form for optimum performance is powder or fine granules. Bulk solids can be sliced into small pieces with a razor or knife. For precise work, such as calibrating the instrument using metal standards, melting and then cooling the sample in the pan before running the test improves thermal contact and gives more repeatable results. A sample weight of 10–20 mg is recommended for most experiments, but the balance can weigh up to 1500 mg, although this must include the weight of the ceramic pans (approximately 200 mg).

To improve the quality of the data further, cover the sample in a layer of aluminum oxide (P/N 04190197) within the sample pan. Place a similar quantity inside the reference pan so that the levels of the material are approximately the same. The aluminum oxide equalizes the reflectivity of the two pans, which reduces the effect of the sample's surface emissivity on the heat flow data.

Alternatively, you can place ceramic lids (N520-2032) on to the sample and reference pans to equalize their reflectivities without the use of the aluminum oxide powder. This approach also makes it easier to determine the sample weight, because you can measure the empty weight of the pan and lid before adding the sample.

Sample Pans

The preferred sample pan is the self-centering, thin-walled, ceramic sample pan provided with the instrument. The sample centers itself in the pan and the pan centers itself on the platform of the sensor, which optimizes the sample weight data obtained by the STA 8000. Three sample pans are provided in the spares kit.

Other sample pans can also be used. The only requirements that the pans must meet are that they do not react or melt within the temperature range of interest, and that the sample does not form alloys with the material of the sample pan.

Sample Atmosphere

The STA 8000 is able to operate in various gas atmospheres. There is one system purge gas inlet and two sample purge gas inlets at the back of the analyzer.

The system purge gas should be a dry, relatively inert gas that flows through the microbalance chamber and exits through the sample chamber. This keeps the environment of the balance constant, prevents absorption or desorption of vapors, and protects the balance against gaseous products evolving from the samples. The system purge gas flow rate is fixed at 45 ml/min. To prevent delays due to equilibration of the analyzer, we strongly recommend that the system purge gas is present at all times.

You can control the atmosphere in which the sample is run by using a sample purge gas to augment the system purge, or to introduce reactive gases into the sample furnace. Recommended purge gases are air, nitrogen, argon, oxygen, and helium. When changing from one purge gas to another or changing flow rates, we recommend that you check the temperature calibration. A sample purge flow rate of 20–40 ml/min is recommended (controlled by the Pyris software). A purge gas inlet pressure of 2–3 bar (30–45 psi) is suitable, with a maximum pressure of 6.2 bar (90 psi).

The sample purge gas enters the furnace/sample area directly, just below the sample, and flows via the furnace wall to the sample. Thus, dead volume can be low, resulting in a small gas change time constant. The time constant depends on the flow rate.

The gas atmosphere should be pure (99.9% minimum), especially if you use nitrogen. If the analysis calls for an inert purge, there should be no trace of oxygen because this could lead to unwanted reactions. **The gas must be dry.** A size 1A cylinder equipped with a suitable regulator is recommended.

The degradation byproducts from the sample leave the instrument directly via a small hole in the furnace cover. These byproducts could be harmful so you should use adequate protection such as placing the instrument in a fume hood. There is some condensation of less volatile products on the cold spots of the analyzer: the lower side of the cover and the upper inner side of the cooling jacket. These spots can be cleaned easily with a suitable solvent. Condensation may also occur on the top inner side of the cooling jacket.

Loading Samples into the STA 8000

NOTE: Good quality results require uniform thermal contact between the pans and the platforms. Avoid spilling sample materials onto the platforms.

Use the following procedure to load samples into an STA 8000:

- 1. Ensure that the furnace is at a stable temperature below 100 °C.
- 2. Click the Pause Temperature Control button on the control panel



This prevents problems with temperature control when the furnace tries to compensate for rapid changes in heat capacity during sample loading and unloading. If the function is operating correctly, a short beep is heard every four seconds and the thermometer in the icon becomes blue in color. If the temperature is above 100 °C or the status of the instrument is not At Temp, this button is unavailable.

- 3. Remove the furnace lid using tweezers.
- 4. Using tweezers, place empty pans on both the reference (right) and sample (left) platforms.

Ensure that the pans are upright and centered on the platforms.

5. Replace the furnace lid and wait for the weight and temperature to stabilize.



- Remove the furnace lid, and place the sample in the sample pan on the left platform. It may be easier to remove the pan first to prevent sample being spilled into the furnace.
- 8. Replace the furnace lid and wait for the weight and temperature to stabilize.
- 9. Click the Sample Weight button
- 10. Click the Pause Temperature Control button again to restart normal temperature control.

Two short beeps will be heard, followed by silence.

CAUTION	Take exceptional care not to drop sample pans or sample material into the furnace. If this should happen, stop the experiment immediately and cool the furnace.
ATTENTION	Prenez des soins exceptionnels pour ne pas laisser tomber les poêles d'échantillons ou les échantillons dans le four. Si cela se produit, arrêtez l'expérience immédiatement et refroidissez le four.

See the software Help for more details of the control panel functions.

Heating

The basic temperature range of the STA 8000 is from ambient to 1600 °C. Since the system is liquid-cooled, it is possible, in principle, to begin a run at subambient temperatures. However, condensation of water vapor on the cooling tubes around the furnace could occur and should be avoided. Reducing condensation is the user's responsibility.

The heating rate of the STA 8000 depends on the temperature range, as follows:

- Ambient to 1000 °C: maximum 100 °C/min
- 1000 to 1600 °C: maximum 25 °C/min

The heating rate can be set in 0.1 °C/min increments. In general, lower heating rates lead to better separation of transitions. For most experiments, a heating rate of 20 °C/min is optimal.

STA 8000 Maintenance

The STA 8000 analyzer needs little routine maintenance other than giving it the proper treatment of a sensitive electromechanical device. Avoid contamination of the furnace by always using a system purge gas and, ideally, a sample purge gas. If the furnace should become contaminated, clean it as soon as possible (refer to *Cleaning and Decontamination* on page 33 for more information).

When cleaning the analyzer, use the following procedures:

- Cleaning the Furnace and Sensor
- Cleaning the Cover

Cleaning the Furnace and Sensor

When the furnace and sensor have become contaminated with reaction products, the best method of cleaning them is to heat the furnace up to 1400 °C in an air or oxygen atmosphere for approximately 10 minutes. This should remove all products that can be oxidized.

CAUTIONDo not remove the furnace lid when heating at high temperatures.ATTENTIONNe pas enlever le couvercle du four lorsqu'il chauffe à des températures
élevées.

If the sensor becomes contaminated, remove it from the furnace and clean it *gently* with a small brush.

When a sample has dropped into the furnace, remove the sensor first, if present, and then remove the sample material, preferably with a vacuum. Take care that material does not fall into the hole for the sensor.

Cleaning the Cover

The reaction byproducts from the samples run inside the STA 8000 furnace and condense on cold spots of the instrument: the bottom side of the cover and the top inner part of the cooling jacket. The bottom side of the cover can be cleaned easily with a tissue and some solvent. This should be done on a regular basis. The top inner side of the cooling jacket can be cleaned in a similar manner.

STA 8000 Part Numbers

Supplies, accessories, and replacement parts can be ordered directly from PerkinElmer. *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.

Below is a list of the part numbers that tools, spare parts and materials for the STA 8000:

Part Number	Description
N5202007	Sensor
N5200040	Ceramic sample pans (pack of 3)
N5202032	Ceramic lids (pack of 6)
03190033	Indium calibration standard
N5380057	Aluminum calibration standard
N5380058	Gold calibration standard
N5380071	Palladium calibration standard
03190034	Tin calibration standard
03190035	Lead calibration standard
03190036	Zinc calibration standard
N5370438	Silver calibration standard
04190197	Aluminum oxide (1 oz bottle)
09908400	Tweezers (flat-tipped)
N5202093	Tweezers (flat, serrated)
N5361078	Tweezers (narrow-tipped)
01541498	Gas restrictor

Part Number	Description
02506519	Tygon tubing (5 m)
02506483	Teflon tubing (6 m)
09903906	Connector (1/8 in. to 1/4 in.)
N5202088	Level indicator
N5200042	Calibration weight

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