THERMAL ANALYSIS

TGA 4000
Installation and Hardware Guide

PerkinElmer®

Release History

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09931396	A	September 2017

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

User Assistance
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Notices

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Contents

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 Online Help

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

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

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.*
I	***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	***Caution (Opgelet)*** *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.*

D

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.*

I

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

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 ***off*** position of the main power switch
	Indicates the ***on*** 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 retirees.

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)

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 0f Science, ICT, and Future Planning) EMC Registration requirements. This instrument is registered as a Class B instrument for residential and/or buiness use.

B급 기기 (가정용 방송통신기자재)
이 기기는 가정용(B급) 전자파적합기기로서 주
로 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

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

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

Space Requirements

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

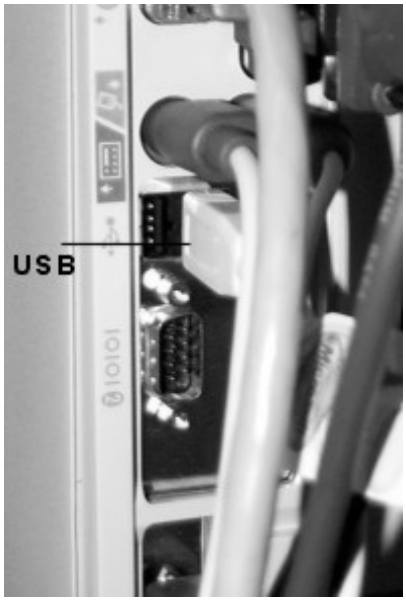
The only way to attach multiple instruments to your computer is via a universal serial bus. PerkinElmer no longer supports the Multiport RS-232 Card (P/N 0940-2018). Installation of the universal serial bus MUST be done before installing any analyzer.

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 0940-2020) and EdgePort/8 (P/N 0940-2019). 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:

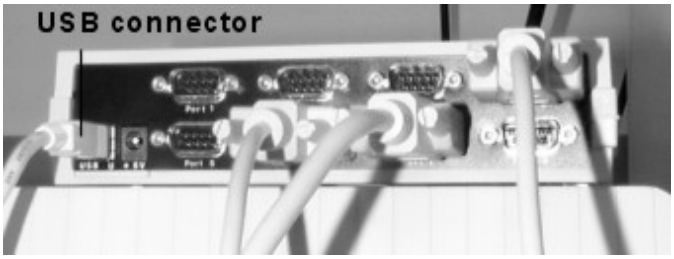
1. Attach one end of the USB cable to one of the USB connectors at the back of the computer.



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 TGA 4000

Safety Precautions for the TGA 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 when using the TGA 4000:

- Never switch off the computer until it has completed its normal shutdown procedure**.**
- Never press the Reset button on the computer if the Pyris software appears to malfunction. Press the CTRL–ALT–DEL keys simultaneously and select Task Manager. From the Task Manager, close the Pyris software.
- Never remove the outer instrument cover of the TGA 4000 without shutting the instrument down and disconnecting its power cord from the power source.
- Before connecting the TGA 4000 to the power outlet, check the voltage setting and fuse.
- The TGA 4000 requires a good earth ground that is common to the earth ground of the computer.
- Do not disconnect cables or tubes from the TGA 4000 while the instrument is on.
- Never open the furnace when it is at a high temperature.
- Always observe the proper startup and shutdown procedures with the TGA 4000 and all related instruments.
- 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.
- Use proper lifting posture when lifting the analyzer. The TGA 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

To remove the furnace lid, always use tweezers; the furnace might be hot.

AVERTISSEMENT

Pour retirer le couvercle du four, utilisez toujours une pince à épiler; le four pourrait être chaud.

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 TGA 4000. Operate the TGA 4000 in a fume hood.

AVERTISSEMENT

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

CAUTION

Do not expose the sample holder to mechanical stress. If no external cooling is applied, do not operate the TGA 4000 above 50 °C.

ATTENTION

Ne pas exposer le support de l'échantillon aux contraintes mécaniques. Si aucun refroidissement externe n'est appliqué, ne pas utiliser le TGA 4000 au-dessus de 50 ° C.

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

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

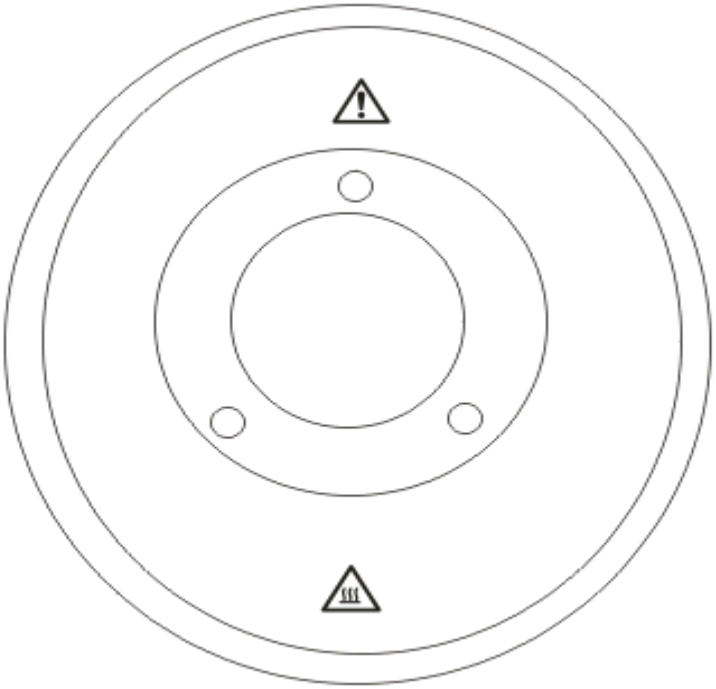
Exterior surfaces may be cleaned with a soft cloth, dampened with a mild detergent and water solution. Do not use abrasive cleaners or solvents.

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.).

TGA 4000 Warning Labels

Warning Labels on the Upper Ring Around the Furnace



WARNING

HOT SURFACE

AVERTISSEMENT

SURFACE CHAUDE

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

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

WARNING

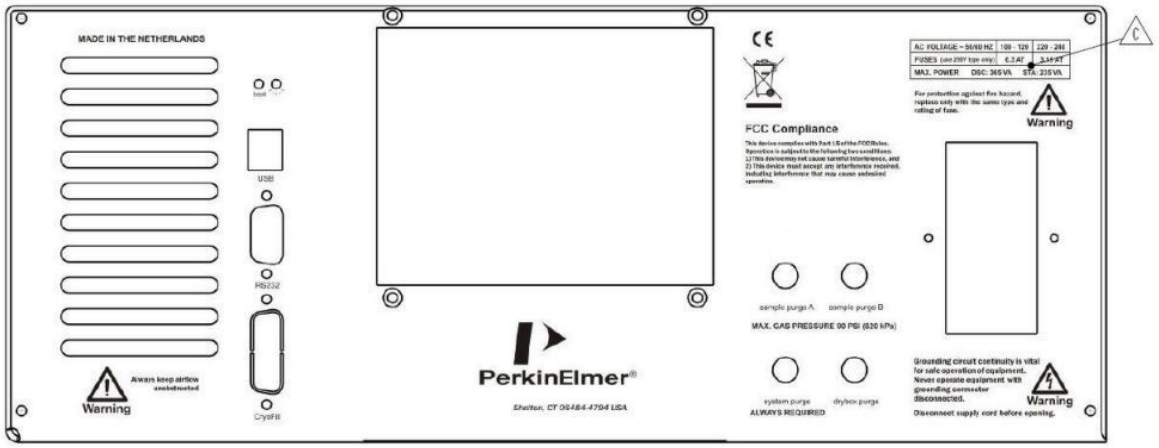
Toxic Gases – Fume Ventilation System

AVERTISSEMENT

Gaz toxiques - Système de ventilation des fumées

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.

Warning Labels on the Back of the TGA 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 TGA 4000

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

- Prepare the Laboratory (refer to page 18)
- Unpack the TGA 4000
- Set Up the TGA 4000 System Components
- Connect the TGA 4000 System Components
- Configure the TGA 4000
- Calibrate the TGA 4000

Unpack the TGA 4000

The TGA 4000 System consists of the following components:

- TGA 4000 Thermogravimetric Analyzer
- Spares and Accessory Kit
- Personal Computer
- Printer (Optional)
- Pyris
- Chiller (Optional)

The TGA 4000 comes set up to operate at voltages of 200–240 V. However, it can easily be set up to run at voltages of 100–120 V. Follow the instructions on page 38 to set your TGA 4000 to the proper voltage for your laboratory.

The TGA 4000 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, 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.

Examine the box for any damage. Check the contents of the Accessories kit against the packing list. If any part is damaged or missing, contact the carrier and PerkinElmer immediately.

Parts Included

The Spares and Accessories Kit contains the following parts:

Description	Part Number	Quantity
Level indicator	N5202088	1
Gas restrictor	01541498	1
Tweezers - flat forceps	09908400	1
Hose clamps	09920044	2
Fuse 6.3 A	09991662	2
Fuse 3.15 A	09991629	1
Magnet	L9003459	1
Sample pan kit	N5200040	1
Sample holder (3 pieces, 1 installed)	N5203020	2
Calibration standards kit	02190071	1
Alsiflex paper (5 pieces)	N5376819	1
Tubing adaptor, female 1/4 to 1/8	09903906	2
Calibration weight	N5200042	1
Ceramic furnace ring	N5376800	1

Also included with the TGA 4000 are:

Description	**Part Number**	**Quantity**
Tygon tubing for coolant	02506519	3 m
Teflon tubing for purge gas	02506483	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	09410042	1
Power cable	—	1

The procedure for unpacking the TGA 4000 is complete.

If you have purchased the AS 6000 Autosampler, this is installed at the factory.

Set Up the TGA 4000 System Components

Place the TGA 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 the system components listed below before switching on the TGA 4000:

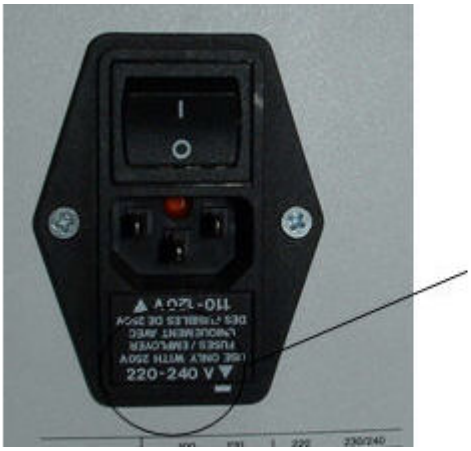
- Select the correct voltage
- Install the gases
- Install the cooling device
- Level the TGA 4000.

Select the Correct Voltage

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

It is configured and shipped with 220 V selected. Selection of the correct voltage for your lab can be done at the AC inlet plug at the rear of the TGA 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.



220-240V selected

To change the voltage:

WARNING

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

AVERTISSEMENT

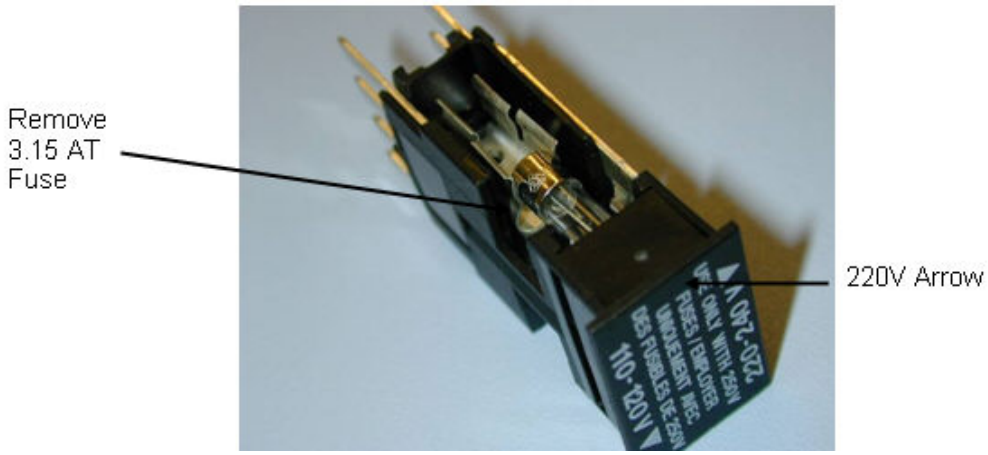
Assurez-vous que le cordon d'alimentation secteur est débranché de l'instrument avant de continuer.

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.

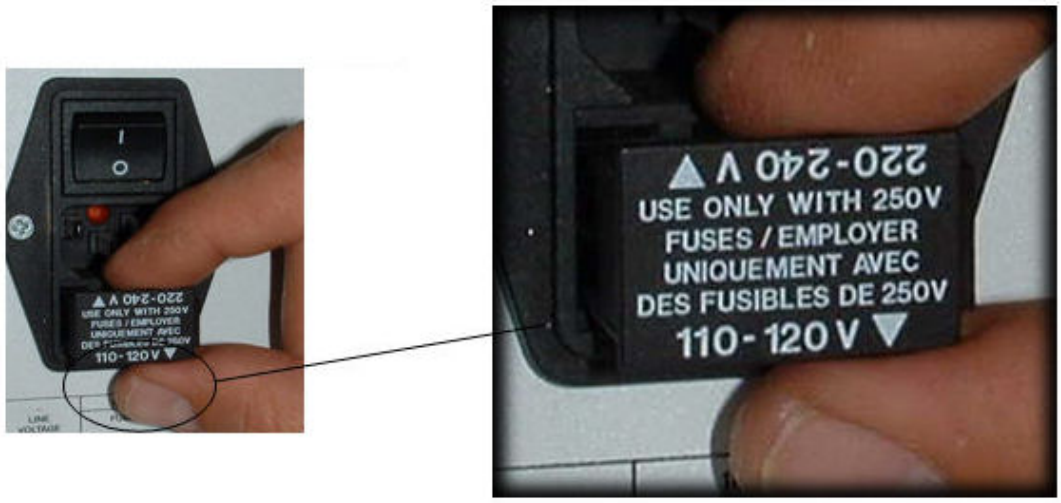


NOTE: In the figure above, we are showing you where the fuses go. The fuse that is installed is for 220V.

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 TGA 4000

The TGA 4000 has two sample purge gas inlets and a system purge gas inlet at the back of the analyzer. The system purge gas **must be a dry inert gas**; preferred gases are nitrogen and air (the latter only when measurements are performed in oxidative atmospheres). The dry inert gas flows through the balance chamber to maintain a constant environment, to prevent absorption or desorption of vapors, and to protect the balance against gaseous products that evolve from the samples. The system purge gas **must** be present in the balance chamber at all times. The system purge gas flow rate is controlled by a needle valve inside the analyzer, and is preset to 40 ml/min. The system has an equilibration time of more than 1 hour so you should leave this flow on permanently if the analyzer is used frequently.

NOTE: The TGA 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 0154-1498) or a Float Displacement Flow Meter (P/N 0290-1624) 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. The sample purge (reaction) gas enters the furnace directly below the sample and flows via the furnace wall to the sample. The reaction gas purges the sample holder/furnace area. Since the furnace and sample holder are ceramic, any noncorrosive gas can 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 flow rate should not exceed the flow rate of the system purge gas.

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 TGA 4000.

Connect the Purge Gas and System Purge Gas Lines to the TGA 4000

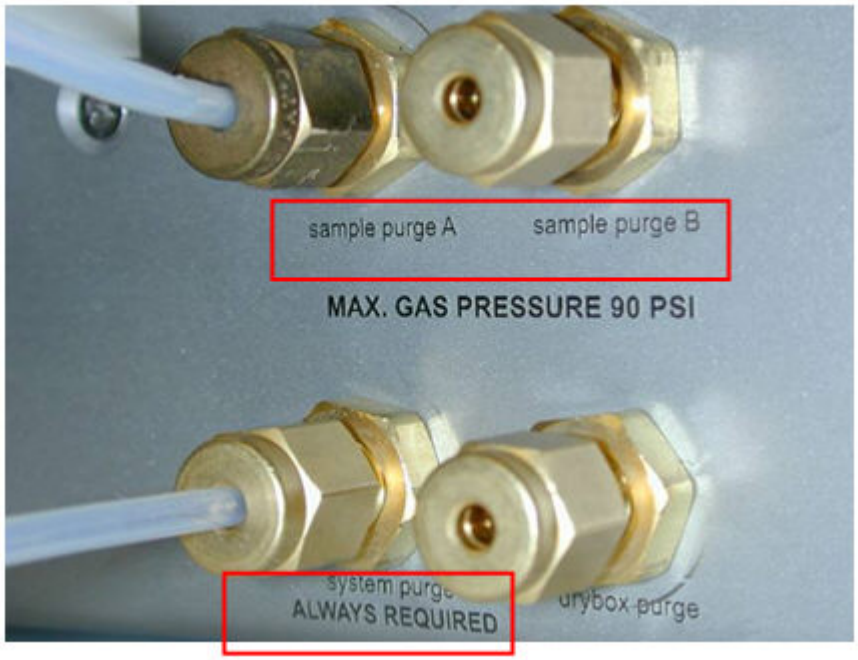
Perform the following procedure to connect your sample purge gas and system purge gas lines to the TGA 4000 analyzer regardless of the other components in the purge gas line. Assuming all the other components of the purge gas line are installed, you will now connect 1/8-in. Teflon tubing to the TGA 4000 using a female connector.

The TGA 4000 System Purge Gas for Balance (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 Sample 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 TGA 4000:



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

Connect the Cooling Device to the TGA 4000

The TGA 4000 has an integrated cooling system. The cooling inlet and outlet are located at the rear of the analyzer and are marked Cooling Liquid In and Cooling Liquid Out. The TGA 4000 is not used in subambient conditions; its temperature range is 15 °C–995 °C. Therefore, the only cooling devices available for the TGA 4000 are the Polyscience Chiller (P/N N537-0220/N537-0221, 120 V and 220 V, respectively) and circulating tap water. The coolant or tap water flows through the cooling jacket that is wrapped around the furnace. It is important that no condensation forms on the cooling jacket.

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 TGA 4000 as follows:

CAUTION	*An appropriate fluid must be used with the chiller. The fluid should be effective down to –20 °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 °C and 90 °C. For operation at –20 °C to 100 °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 ° 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 ° C et 90 ° C. Pour une utilisation de -20 ° C à 100 ° 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 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.*

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.

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 TGA 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 TGA 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 ± 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 TGA 4000.
2. Connect one hose to the Cooling Liquid In tube. Place a hose clamp around the hose and tube to secure the hose in place.
3. Connect the other end of this hose to the tap.
4. Connect the other hose to the Cooling Liquid Out tube. Place a hose clamp around the hose and tube to secure the hose in place.
5. Connect the other end of the hose to a suitable drain.

The temperature of the cooling liquid should not change more then 0.4 °C/hour in the case of tap water.

CAUTION	*The heat exchanger of the TGA 4000 will NOT accept pressure. Therefore, flow regulation should be done before the water enters the TGA 4000.*
ATTENTION	*L'échangeur de chaleur du TGA 4000 n'acceptera PAS la pression. Par conséquent, la régulation du débit devrait être effectuée avant que l'eau entre dans le TGA 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.*

Level the TGA 4000

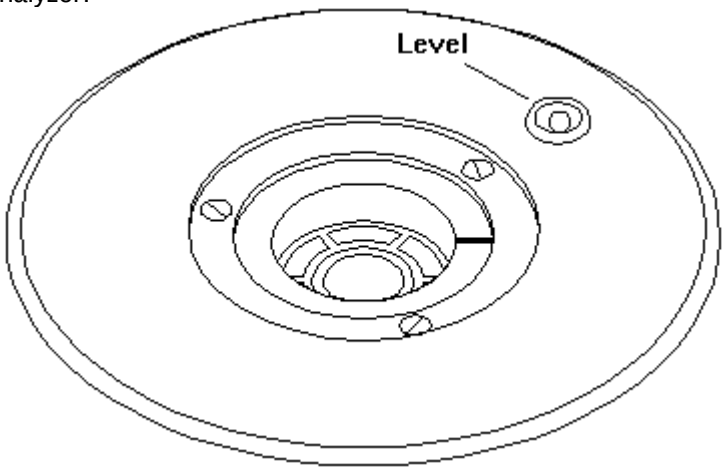
1. Lift the analyzer up at the front and turn each foot until it is screwed all the way into the bottom of the analyzer.

NOTE: The analyzer is heavy. When lifting it along the front edge, be sure your hand is firmly underneath it and can support the analyzer while you adjust the feet.

2. Position the analyzer in the exact spot on the bench where you will be using it.

3. Check whether the analyzer rocks on the bench, or if one foot is not touching the bench. If so, lift the analyzer and slightly turn the foot clockwise to extend it. Lower the analyzer to check that it does not rock. Once all of the feet are touching the bench, make sure that the analyzer is steady.

4. Place the level provided in the Spares kit on the furnace plate on the top of the analyzer.



5. Look at the level to see if the bubble is centered. If not, readjust the feet as in step 3 until the bubble is centered in the level.

   The TGA 4000 is level when the bubble is centered inside the ring.

6. Once the analyzer appears to be level, again make sure all of the feet are in contact with the bench and that the analyzer does not rock.

This procedure is complete.

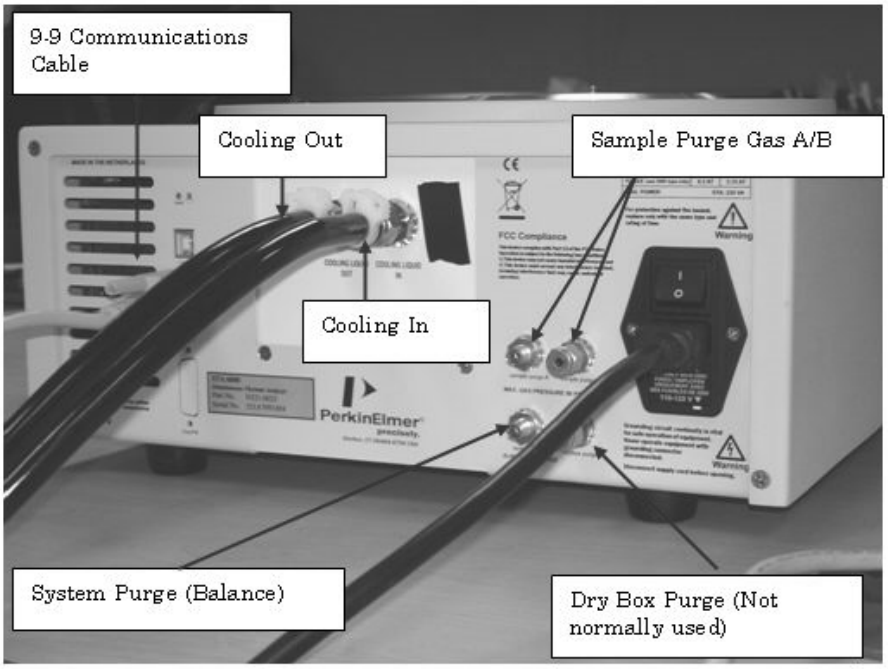
Connect the TGA 4000 System Components

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

Follow the steps below to connect the TGA 4000 to the computer.

CAUTION	*Make sure that the computer is off before plugging in the TGA 4000.*
ATTENTION	*Assurez-vous que l'ordinateur est éteint avant de brancher le TGA 4000.*

The image below shows the connections on the TGA 4000:

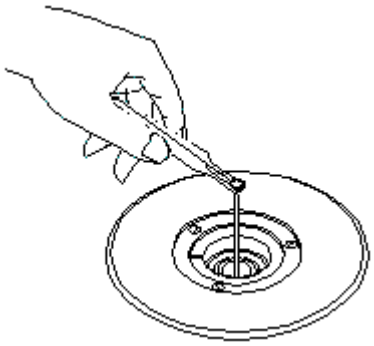


Make sure all components are turned off and unplugged before you begin.

1. Connect one end of the RS-232 cable to the back of the TGA 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, power on the system in the following order:
   - Computer
   - TGA 4000
   - Printer
6. Turn on the sample purge gas, if you are going to use it (recommended flow rate = 30 ml/min), the system purge gas (recommended flow rate = 40 ml/min), and the

water for the cooling system or any other accessories for the cooling device you are using.

7. Carefully grasp the sample holder with tweezers at the upper, shaped part.



8. Insert the alumina rod of the sample holder down through the center hole in the bottom of the furnace. Move it downward until you feel a slight resistance. Move the sample holder another 10 mm until you feel it stop.

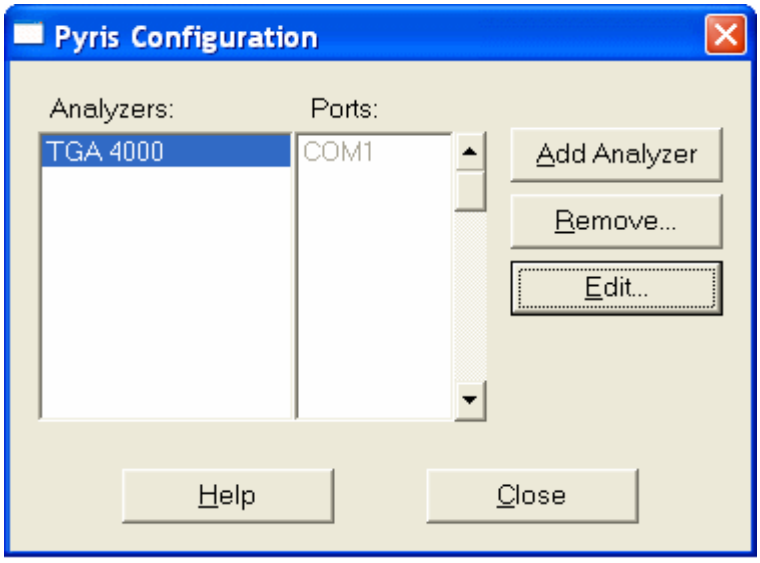
   DO NOT EXERT EXCESSIVE FORCE.

Configure the TGA 4000

The Pyris Software must be configured for the TGA 4000. This must be done before the TGA 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.

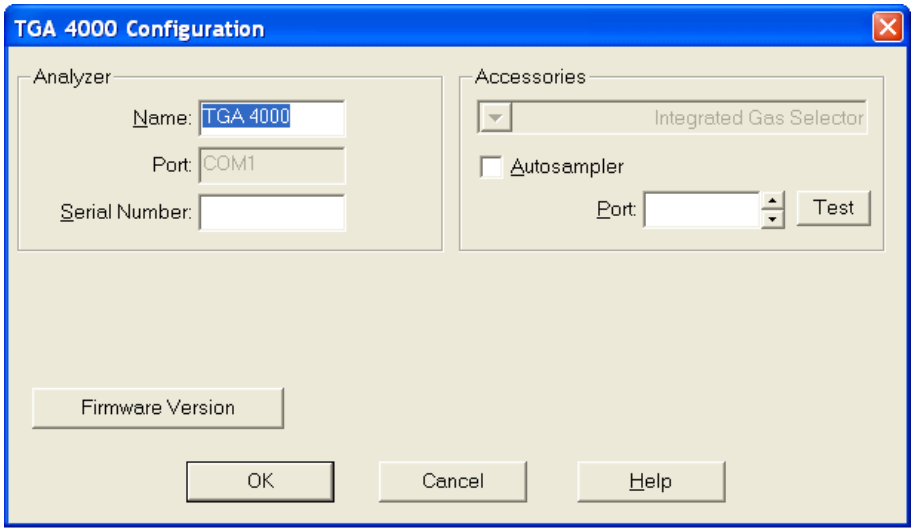


2. Select the **Add Analyzer** button.

   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 TGA 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 TGA 4000 in this dialog box. Select the accessories that are attached to the analyzer (for example, Autosampler).

5. Click **OK** to accept the selections.

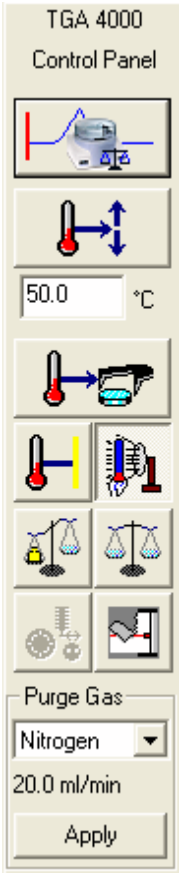
6. Click **OK** to exit the Configuration dialog box or select **Add Analyzer** to add another analyzer to the configuration. Remember that the analyzer must be connected to the communications port and switched on, otherwise, it will not be recognized by the Pyris software.

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

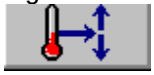
Starting the Pyris Manager

1. Select **Pyris Manager** from the Pyris Software group. The Pyris Manager is displayed on the screen.

2. Select **TGA 4000** from the Pyris Manager. The TGA 4000 control panel is displayed.



3. Now that the system is switched on, the TGA 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 Go To Temp button  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 installation of the TGA 4000 is complete. The analyzer is ready to be calibrated.

Calibrate the TGA 4000

The TGA 4000 Thermogravimetric Analyzer has been calibrated at the factory. However, it must be calibrated again before obtaining data from your samples. 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.

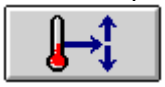
The TGA 4000 is shipped with the reference materials alumel, nickel, and iron for the temperature calibration and a 100 mg stainless steel sphere for the weight calibration in the Accessories kit.

The Calibration page in Pyris is accessed from the View menu of the Method Editor.
The three calibration routines should be performed in the order:

- Furnace
- Temperature
- Weight

Furnace Calibration

NOTE: The Furnace calibration must be performed **before** the Temperature calibration.

1. Make sure the sample holder is empty.
2. To verify thermocouple operation, program the temperature to 100 °C. Enter 100 in the Go To Temp entry field of the control panel and click the Go To Temp button  on the control panel.
3. Check that the current sample temperature displayed in the status panel is at or below the intended minimum calibration temperature. The easiest way to accomplish this is to click the Go To Load button  on the control panel.
4. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.
5. Select the Furnace tab in the Calibration window.
6. In the **Minimum** field, enter a minimum temperature at which the calibration will begin. This should be below your normal operating region.

NOTE: When selecting minimum and maximum temperatures for the furnace calibration, select the limits so that they encompass the temperature range in which you plan to operate. Maximum temperature minus minimum temperature must be greater than 100 °C or the furnace calibration will not be performed.

7. In the **Maximum** field, enter a maximum temperature at which the calibration will end. This should be above your normal operating region.

8. Click the **Begin Calibration** button.

   A message stating that the system defaults will be used for all temperature calibrations if you perform a furnace calibration is displayed. If you wish to continue, click **OK**. The Furnace calibration begins immediately. The Furnace Calibration dialog box is displayed showing the approximate time remaining in the calibration.

   You can stop the calibration, which takes about an hour, by clicking the **Stop Calibration** button.

9. When the calibration routine is finished, click the **Save and Apply** button in the Calibration window to send the new calibration values to the analyzer and save the calibration file.

10. Select the Temperature Calibration tab or the **Close** button to close the Calibration window and begin using the new calibration values.

Temperature Calibration

NOTE: You must perform a Furnace calibration **before** a Temperature calibration.

To calibrate the temperature of the TGA 4000, perform the following steps:

1. While in Instrument Viewer or Method Editor, select **Calibrate** from the View menu.

   The Calibration window is displayed.

2. Restore the default temperature calibration by selecting **Temperature** from the Restore menu (this step is optional).

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. Select **OK** to save the default file.

4. Click **Close** to close the Calibration window.

The TGA 4000 is calibrated by performing two runs each using two or three of the magnetic standards provided: alumel, nickel, and iron. These are supplied with the instrument. The runs should use the same conditions under which you would run your samples. This temperature calibration uses the Curie transition of the materials, that is, the point at which the magnetic properties disappear.

The first run of each reference material should use the lower scanning rate (for example, 5 °C/min) and the second run the higher scanning rate (for example, 50 °C/min). You must run at least two reference materials but you can use all three if desired. Therefore, you will do from four to six runs in order to perform a temperature calibration.

After you have completed the steps above, go on to the following steps to complete the temperature calibration:

5. From the Method Editor, select **Open Method** from the File menu and double-click **Alumel.t6m**, **Nickel.t6m**, or **Iron.t6m**, depending on the sample you are going to run.

6. Place an empty sample pan on the sample holder that is already in place in the furnace.

7. Place the furnace lid back on the analyzer.

8. Click the **Zero Weight** button on the control panel.

   The weight of the empty sample pan is displayed in the Zero field on the Sample Info page.

9. Remove the furnace lid. Without removing the pan, place a 1–2 mm length (approximately 20 mg) of reference material in the sample pan.

10. Place the furnace lid on the analyzer, position the plastic spacer and the magnet over the center of the lid, and click the **Sample Weight** button on the control panel.

    The weight appears in the Weight field in the Sample Info page.

11. Complete the other information on the Sample Info page.

12. To run Curie points, you must run the reference material under the same experimental conditions that you will run your samples. Check the purge gas type, coolants used, and the scanning rate.

    The first run of a reference material is performed at a scanning rate of 5 °C/min.

13. Start the run by clicking the **Start Method** button in the control panel.

14. When the run is finished, remove the sample pan and repeat steps 5 through 13 for the same reference material but at a scanning rate of 50 °C/min.

15. After the second run for one reference material, repeat steps 5 through 14 for the other reference material(s) you choose to use.

16. For each data file collected, open the Data Analysis window, select the data file, and calculate the Offset value by performing an Onset calculation at the end of the Curie point transition. Note the Onset result.

17. When all runs are complete and Onset calculations have been performed, select the **Calibrate** command in the View menu.

    The Calibration window appears.

18. Select the Temperature Calibration tab. Enter the **Operator** name. Enter the names of the reference materials used, the expected Onset values (provided in the documentation accompanying the reference materials), and the measured Onset results at the two scanning rates for each reference material.

19. Enter the scanning rates used in the **Rate 1** and **Rate 2** fields and click the appropriate option to indicate which references to use for the calibration: **1 and 2** or **1, 2, and 3**.

20. 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.

21. Click **Close** to close the Calibration window and begin using the new calibration values.

Weight Calibration

To perform a weight calibration of the TGA 4000:

1. While in Instrument Viewer or the Method Editor in the Pyris software, select **Calibrate** from the View menu.
2. Select the Weight tab in the Calibration window.
3. Enter the weight of the reference material in the **Ref. Weight** box.
   A 100 mg calibration weight is supplied with the instrument.
4. Click **Begin Calibration**.
5. To prepare for a zero weight reading, place a clean sample pan in the sample holder and then replace the furnace cover.
6. Click **OK** in the dialog box.
7. When the zero weight reading is stable in the Read Zero field, click **OK** in the dialog box.
8. Place the reference weight into the crucible. You can either first remove the crucible from the furnace and place the weight in it or place the weight with the crucible in place. Replace the furnace cover. Click **OK** in the dialog box.
9. When the weight reading is stable in the Read Value field, click **OK** in the dialog box.
   The calibration is complete.
10. Click **Save and Apply** to send the new calibration value to the analyzer and save the calibration file.

The TGA 4000 calibration is complete.

This completes the installation of the TGA 4000.

TGA 4000 Hardware

TGA 4000 Thermogravimetric Analyzer

The TGA 4000 Thermogravimetric Analyzer features reliability, robustness, low cost of operation, and ease of use, all of which make it an outstanding analyzer for quality control laboratories.

The TGA 4000 is controlled by Pyris software, installed on the computer to which the analyzer is connected. The TGA 4000 measures the weight of a substance as a function of its temperature or the time. Weight changes can be measured accurately, generating information on various processes in a sample such as dehydration, degradation, oxidation, etc. Also, since measurements can be performed in a well-defined atmosphere, the behavior of a sample in atmospheres other than air can be studied easily. The TGA 4000 can be used for component analysis (weight %), evaluation of stabilizers, loss on drying, proximate analysis, lubricating oil analysis, and oxidation studies. 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.

For more information on the TGA 4000, see the following topics:

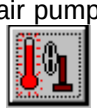
- Features of the TGA 4000
- Safety Precautions
- Sample Handling
- Calibration
- Maintenance
- TGA 4000 Part Numbers
- Autosampler

Features of the TGA 4000

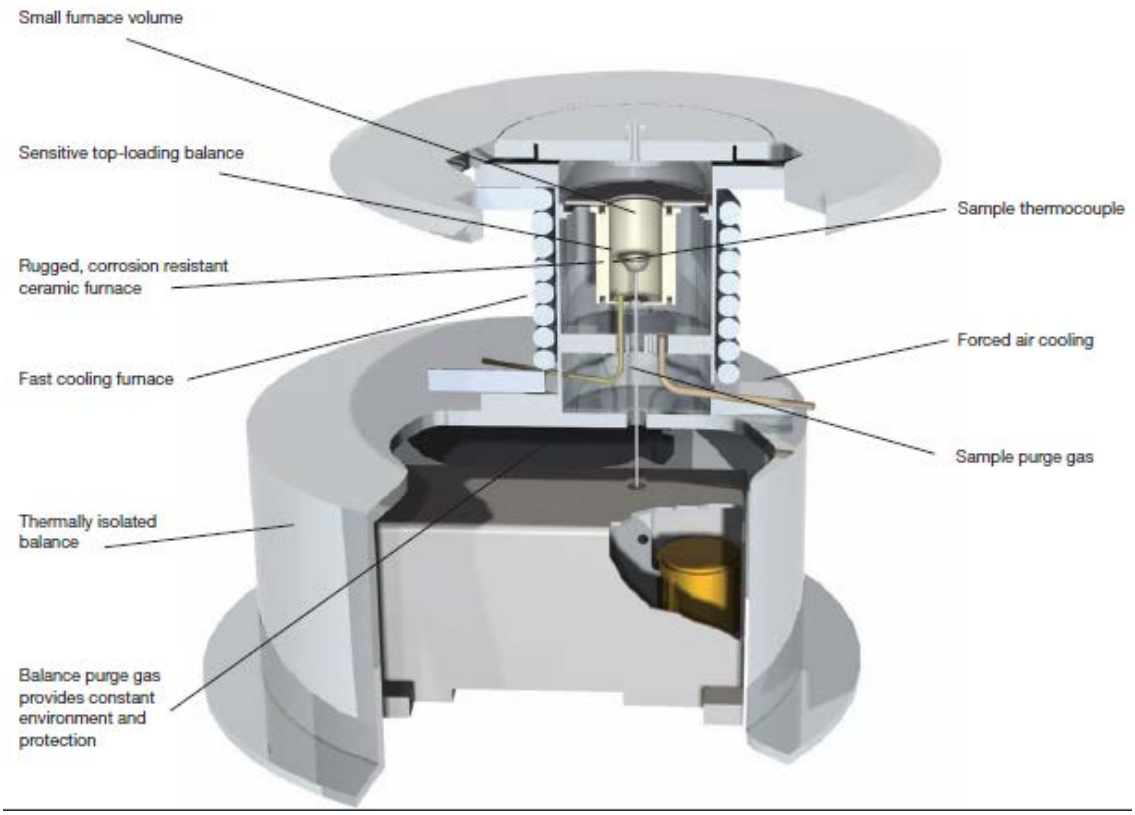
The TGA 400 has a high-performance balance and furnace for maximum accuracy and precision. The top-loading balance allows easy sample loading and unloading. The balance is protected from sample debris by a balance purge gases, and thick stainless steel wall act as a heat sink to thermally isolate the balance from the furnace. The TGA 4000 has a top-loading microbalance. The sample pan is placed onto the sample holder which connects to the microbalance via a very thin rod.

The furnace has a large isothermal zone, which keeps your samples at the same temperature as the furnace throughout a temperature program. This increases reproducibility of temperature measurements. The compact ceramic furnace is inert and corrosion resistance, enabling the use of a wide range of reactive gases. The built-in mass flow controller monitors and controls the purge gas flowrates and pressures, and can switch automatically between sample gases using Pyris software.

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. You can use various types of cooling liquids in the integrated cooling system. During operation, the cooling liquid must flow through the heat exchanger at a constant flow rate and the cooling liquid should be at a temperature such that there is no condensation inside the analyzer. The recommended cooling device is the Chiller (P/N N537-0220/N537-0221, 120 V and 220 V, respectively).

Forced air cooling is also possible. A small air pump is mounted inside the instrument and activated by clicking the Cooling Air button  on the control panel. Forced air and liquid cooling reduces cycle times, improving productivity.

Pyris supports the AS 6000 autosampler accessory. The autosampler allows testing of up to 45 samples. The unique two-piece carousel allows removal of one section while the other is in operation. Combined with the Pyris Player software, the runs are entirely automated. You can create play lists that contain multiple methods for analyzing one sample; multiple samples to be analyzed by one method, and many ways to perform postrun data analysis.



Heating

The basic temperature range of the TGA 4000 is from ambient to 995 °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 maximum heating rate at high temperatures is limited by the supply voltage, as shown in the following table:

Supply Voltage / V	**Maximum heating rate at 900 °C / °C min^{-1}**	**Maximum heating rate at 995 °C / °C min^{-1}**
180 or 90	42	0
200 or 100	134	26
220 or 110	200	118
230 or 115	200	138
240 or 120	200	143
250 or 125	200	145

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. The free heating rate of the TGA 4000 furnace has been limited to 200 °C/min.

Sample Handling

The TGA 4000 measures the change in weight of a sample as a function of temperature or time. The materials and techniques used to obtain data with a TGA 4000 are discussed below.

Sample Preparation

The thermogravimetric analyzer analyzes solid samples in powder, crystal, or granular form. Although quantitative accuracy will remain the same regardless of 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. Solid materials can be sliced into small pieces with a razor or knife.

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 sample holder. Three sample pans are provided in the Spares kit.

Other sample pans can also be used. The only requirement that the pans must meet is 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

It is important that the TGA 4000 is able to operate in various gas atmospheres. The change from one atmosphere to another should be rapid. The TGA 4000 has one purge gas inlet for the system purge gas inlet and two sample purge gas inlets at the back of the analyzer. The system purge gas should be a dry inert gas that flows through the microbalance 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 has a flow rate of 40 ml/min. Since the equilibration time of the analyzer is significant, it is recommended that the balance protective gas be present at all times.

You can control the atmosphere in which the sample is run by using a sample reactive or purge gas to displace or 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, always check the temperature calibration. A flow rate of 20–40 ml/min is recommended.

The reaction 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.

NOTE: The flow rate for the system purge gas should be equal to or greater than that for the sample purge gas.

The gas atmosphere should be pure (99.9% minimum), especially if you use nitrogen. There should be no trace of oxygen as this could lead to unwanted reactions. **The gas must be dry.**

The degradation byproducts from the sample leave the instrument directly via a small hole in the furnace cover. These byproducts could be harmful, so use adequate suction such 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. To prevent this, use the porous ceramic furnace cover to absorb the less volatile byproducts. When necessary, this ring should be changed.

Loading the Sample into the TGA 4000

Use the following procedure to load samples or remove samples:

1. Using tweezers, remove the stainless steel cover off of the top of the analyzer. Place it on the stainless steel ring surrounding the opening to the furnace.

2. Place the sample inside the sample pan.

3. With tweezers, carefully place the sample pan with sample onto the sample holder and make sure that the pan is centered.

4. Replace the lid.

Calibration

The furnace, temperature, and weight are calibrated by the service engineer upon installation. The TGA 4000's calibration should remain unchanged for quite some time, provided that there are no changes in the instrument's operating conditions. Even when the system is shut off, the calibration values are stored so that the next time the instrument is turned on, it will still be calibrated. Some of the conditions that could change either the temperature or the weight calibration of the TGA 4000 and require recalibration are as follows:

- If the operating temperature range of your experiments changes, recalibration may be necessary. Check the temperature calibration in the range of interest to determine if the current calibration is still valid.
- If the purge gas type or purge gas flow rate changes, the calibration should be checked for highest accuracy.
- If a new furnace is installed, the temperature calibration should be checked.
- If a new sample holder is installed, the system should be recalibrated.
- If a new sample thermocouple is installed or if the existing thermocouple has been disturbed, you should perform a temperature calibration.
- If the analyzer is moved or re-leveled, you should perform a weight calibration.

Maintenance

The TGA 4000 Thermogravimetric 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 balance protective gas and a purge gas. If the furnace should become contaminated, clean it as soon as possible.

Maintenance includes the following procedures:

- Cleaning the Furnace and Sample Holder
- Cleaning the Cover
- Changing the Sample Thermocouple

Cleaning the Furnace and Sample Holder

When the furnace and sample holder have become contaminated with reaction products, the best method of cleaning them is to heat the furnace up to 995 °C in an air or oxygen atmosphere for approximately 10 minutes. This should remove all products that can be oxidized.

CAUTION	*Do not touch the sample thermocouple (in the TGA 4000)..*
ATTENTION	*Ne pas toucher l'échantillon de thermocouple (dans le TGA 4000).*

CAUTION	*Do not remove the furnace lid when heating at high temperatures.*
ATTENTION	*Ne pas enlever le couvercle du four lorsqu'il chauffe à des températures élevées.*

If the sample holder 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 sample holder 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

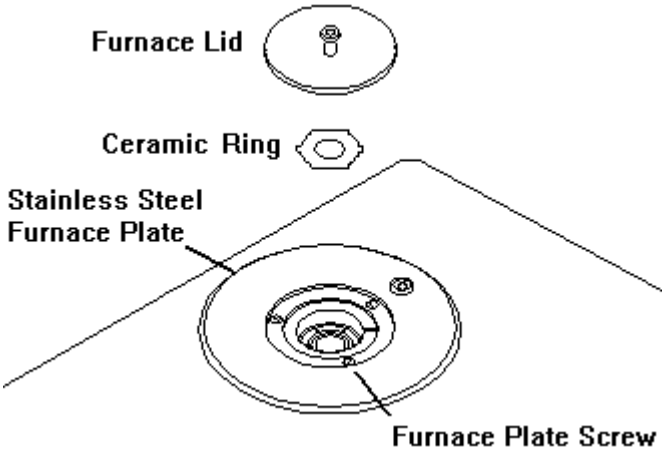
The reaction byproducts from the samples run inside the STA 6000, TGA 4000 and Pyris 6 TGA furnace and condense on cold spots of the instrument: the bottom side of the cover and the top inner part of the cooling jacket. The bottom side of the cover can be cleaned easily with a tissue and some solvent. This should be done on a regular basis. The top inner side of the cooling jacket can be cleaned in a similar manner. To keep the top of the cooling jacket clean, a ring of porous ceramic material is provided and should be positioned as seen in the figure displayed by clicking here. The ring should be replaced when necessary.

Changing the Sample Thermocouple

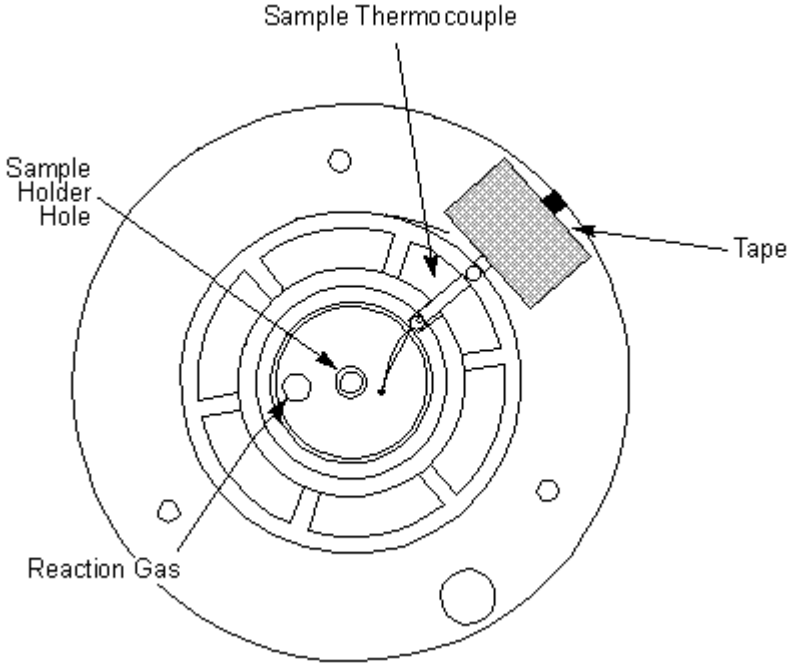
The sample thermocouple has a limited lifetime because of the range of temperatures and the high maximum temperature it withstands. Therefore, the TGA 4000 and Pyris 6 TGA are designed so that a user can change the sample thermocouple.

To remove the sample thermocouple, perform the following procedure:

1. Switch off the analyzer and the remove the power cord.
2. Remove the furnace lid.
3. Remove the porous ceramic ring.
4. Remove the sample holder.
5. Remove the three screws of the top stainless steel furnace plate and then remove the plate.

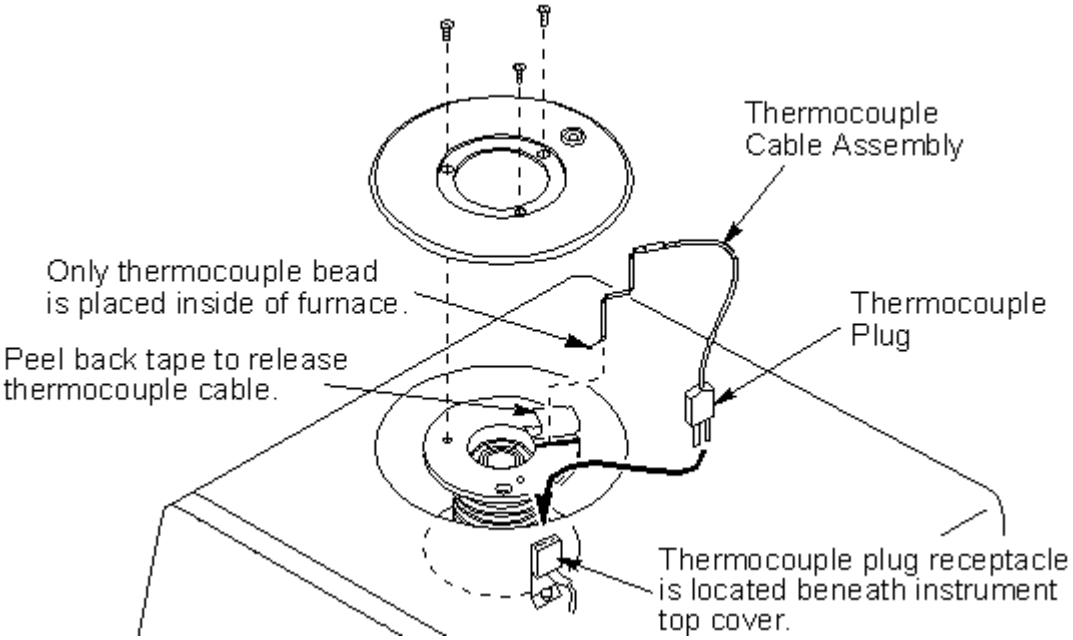


6. Lift the tape off the top of the furnace to free the thermocouple.



7. Unplug the yellow thermocouple plug.

8. While holding the plug, gently lift the thermocouple up and out of the furnace. Be careful not to bend the thermocouple. Note how the thermocouple is positioned in the furnace wall.



To install a new sample thermocouple, perform the following steps:

1. While holding the new thermocouple by its yellow plug, gently lower it into the furnace. Make sure that the lower vertical ceramic bead goes into the slit in the furnace wall. The two wires at the end of the thermocouple should be inside the furnace, near the center hole, but not over it. The horizontal bead at the top of the thermocouple should lie inside the groove at the top of the cooling jacket.

2. Connect the thermocouple plug with the connector at the side of the jacket.

3. Replace the tape covering the thermocouple at the top of the cooling jacket.

4. Replace the stainless steel furnace plate and replace the three screws.

5. Position the sample holder in the furnace.

6. Reposition the porous ceramic ring.

7. Place the furnace lid back on top of the analyzer.

8. Reconnect the power cord and switch on the analyzer.

Perform a temperature calibration.

TGA 4000 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 are in the Spares kit (P/N N520-0110).

Part No.	**Description**	**Quantity**
01541498	H restrictor	1
02190071 includes:	Calibration standards Kit	1
09988015	Alumel calibration standard	1
N5190869	Nickel calibration standard	1
09988018	Iron calibration standard	1
09909608	Calibration specification card	1
09908400	Tweezers – flat forceps	1
L9003459	Magnet (with plastic spacer)	1
N5200040	Ceramic sample pans	1
N5203020	Sample holder (3)	2 (+1 installed)
09903906	Female connector 1/8 in. to 1/4 in.	2
09920044	Hose clamps	2
N5200042	Calibration weight	1
N5376800	Ceramic furnace ring	1
09991629	Fuse 3.15 A slow blow	1
09991662	Fuse 6.3 A slow blow	2
N5376819	Alsiflex strips	5

Also included with the TGA 4000 are:

- Tygon tubing (P/N 0250-6519) for connection of liquid coolant
- Teflon tubing (P/N 0250-8059) for purge gas connection
- 5/16-in. pipe for Swagelok Cooling Liquid In and Out connectors
- RS-232 cable for communicating to the computer
- Power cable

Miscellaneous Part Numbers

Part Number	Description
N5370210	TGA 4000
N5370211	TGA 4000 with Autosampler
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

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
- Replace a Sample Thermocouple in a TGA

Safety Precautions for the AS 6000

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

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.

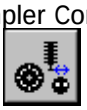
The procedure below is for the work cycle for DSC 4000/DSC 6000 for multiple samples using the reduced cycle time.

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

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 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 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

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 µL.

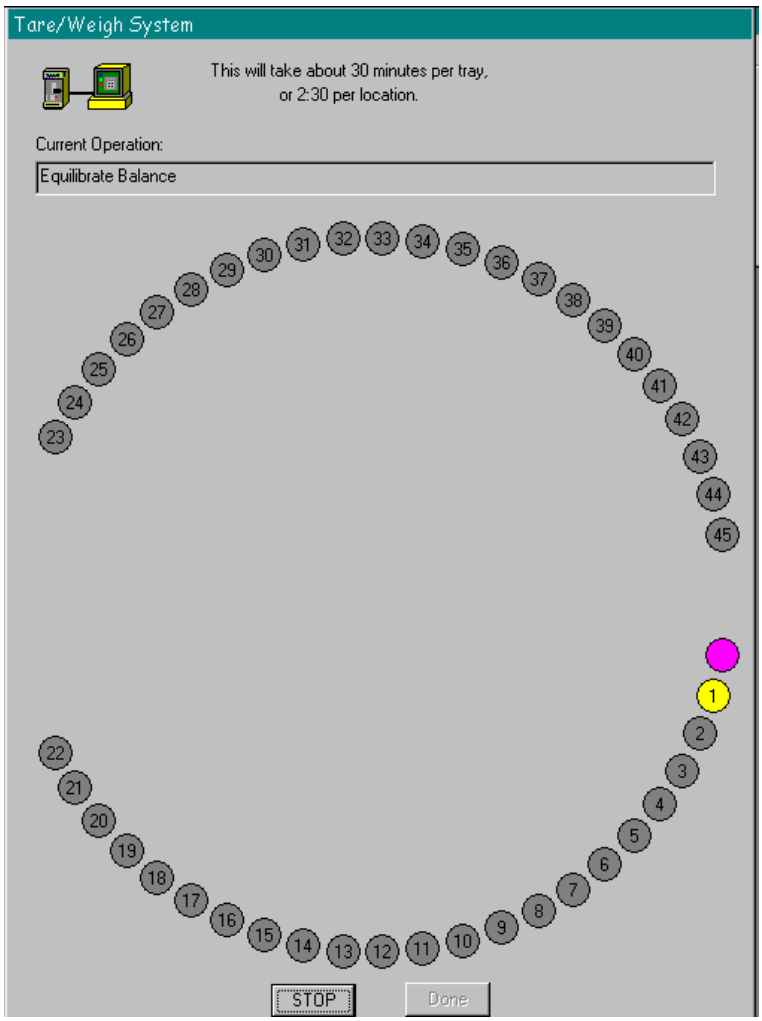
Sample Loading

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.

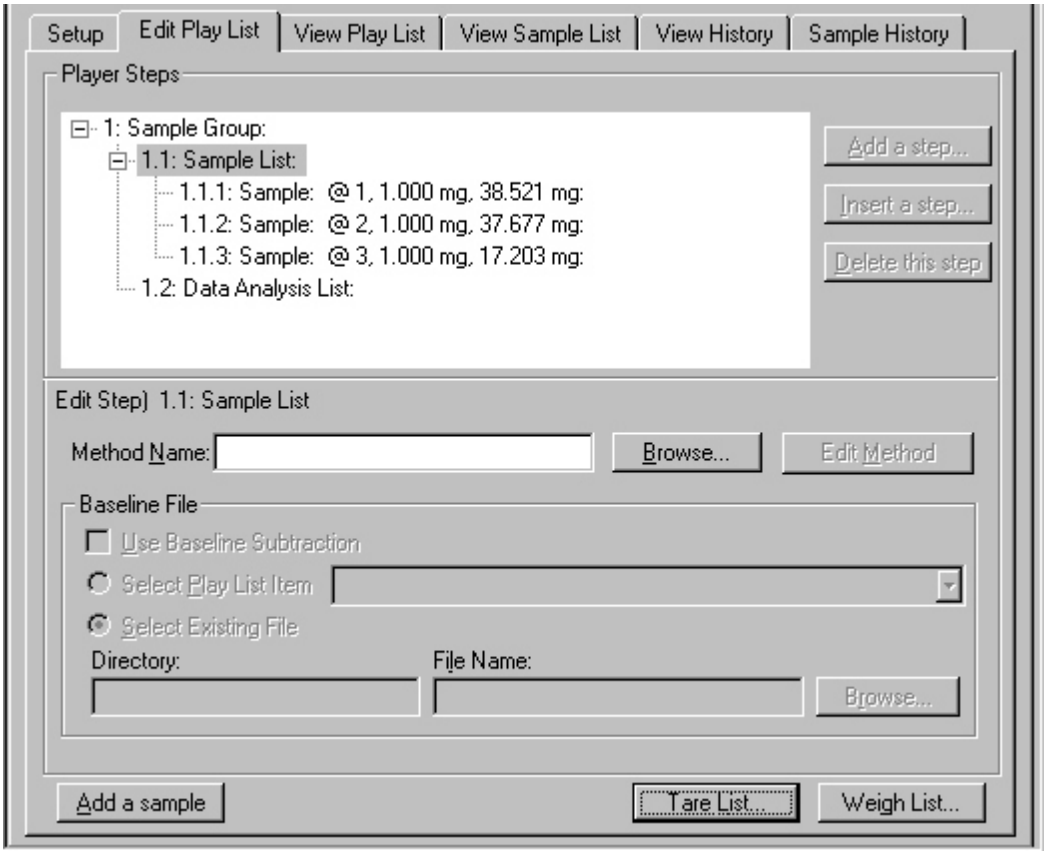
You can tare all of the crucibles in the tray using the Pyris Player Tare All feature. To do this your play list must contain a Sample Group. If you are creating a new play list, select Sample Group as the first entry. If you are using an existing play list, it should have a Sample Group in it in order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

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. Turn on the chiller and adjust according to its instructions.

4. Verify that the electrical and cable connections between the computer and the instrument and other components of the system are properly connected.

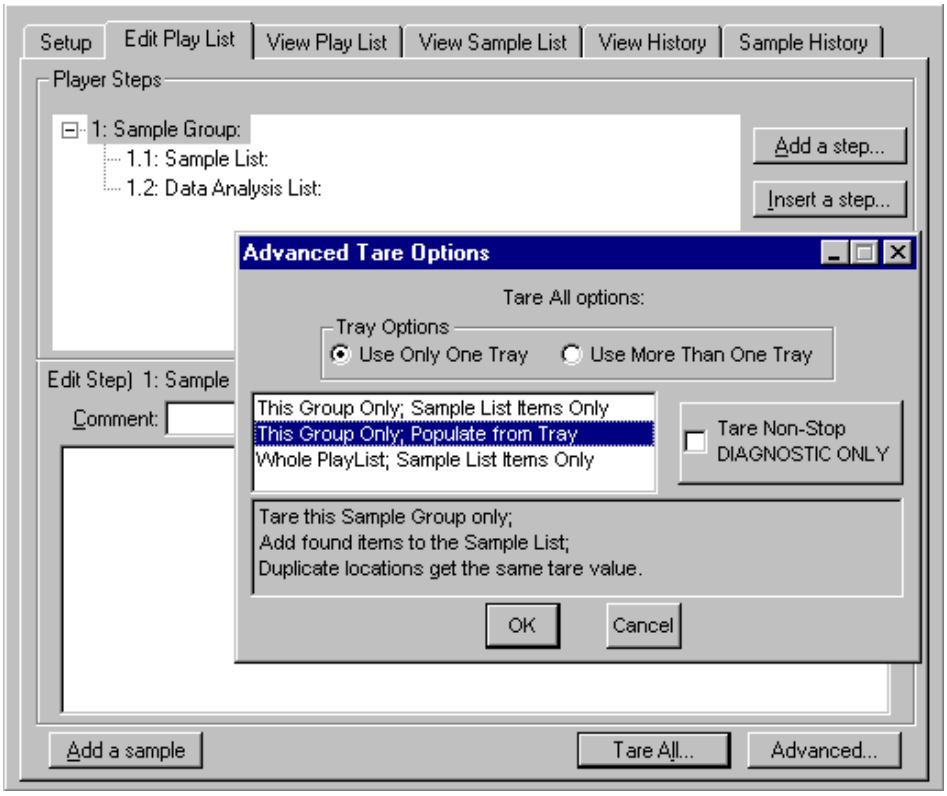
5. Turn on the power to the system components.

6. Prepare your samples.

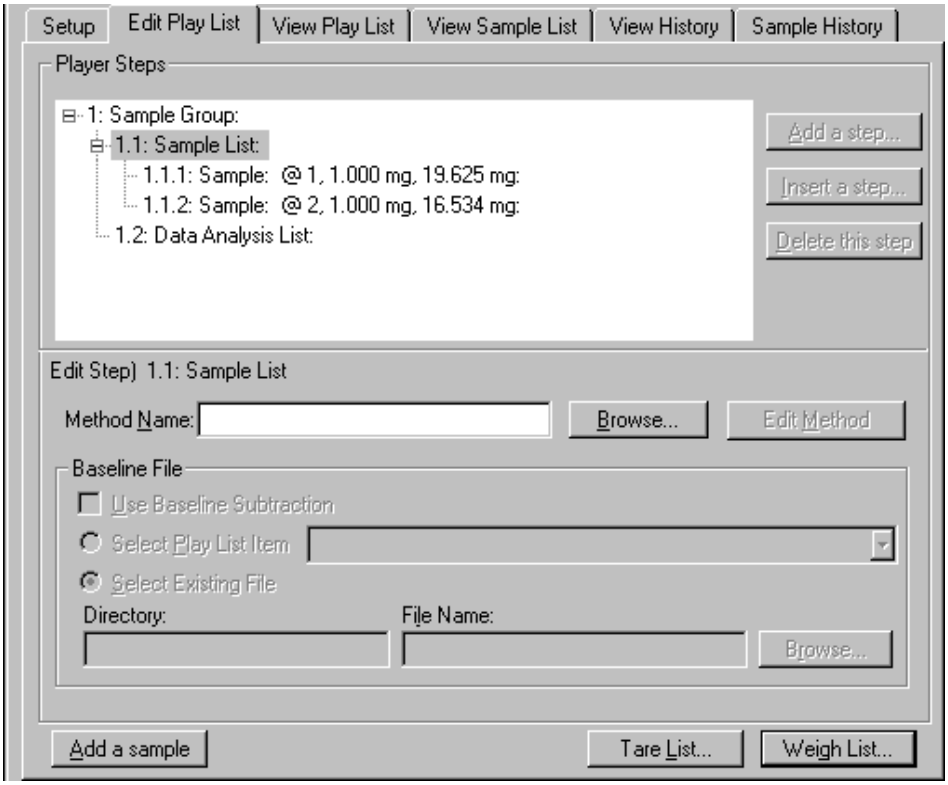
7. Start Pyris software and click the parent instrument's button in the Pyris Manager. Click the Pyris Player button on the toolbar:  . Either open an existing play list or create a new one.

   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**.

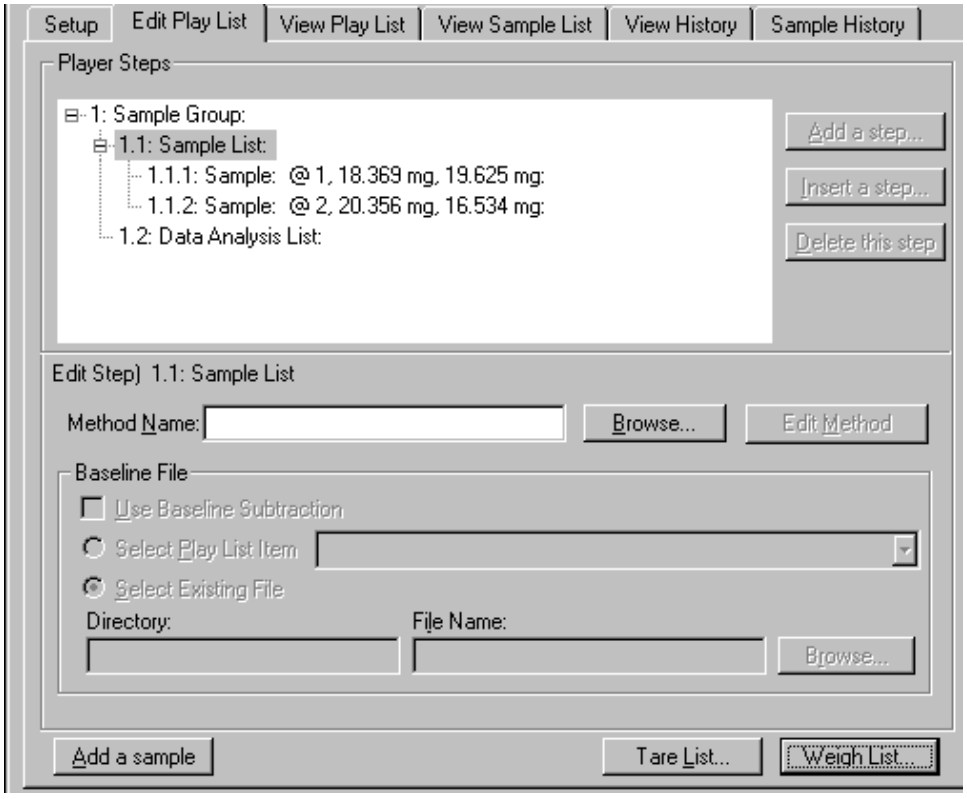


– Click **OK** and the Tare/Weigh System dialog box appears. The AS 6000 begins the procedure of taring the sample pans present on the sample trays. As it finds and tares each pan, it populates the Sample List and includes the tare weight for the sample pan in the Sample line. When taring is complete,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



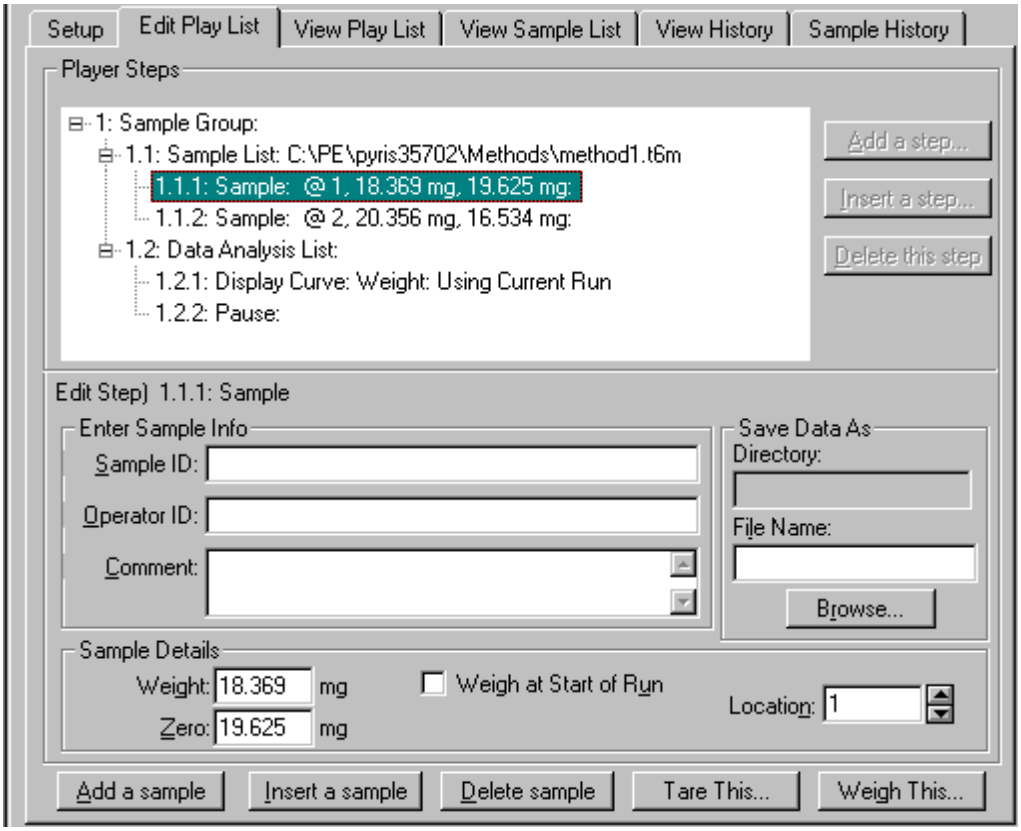
– Remove the sample trays from the autosampler, or leave them in place, to load the crucibles. If you removed the trays, carefully return them to the autosampler and make sure that the two knobs on the bottom of each tray engage the holes in the autosampler plate.

– 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.



8. Save the play list by selecting Save Player from the File menu.

9. 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.

Troubleshooting

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, power off the instrument and power 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.

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

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	Failure picking sample out of furnace Incorrect horizontal baseplate adjustment Incorrect horizontal calibration AS 6000 run was interrupted after putting a sample in furnace and restarting	Check if gripper fingers are bent. Replace gripper assembly Adjust baseplate Recalibrate the AS 6000 Reset parent instrument and setup link again
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	No detection of the gripper fingers because of direct sunlight at the light switches Gripper assembly displaced	Do not place instrument in direct sunlight Align gripper assembly

Error Messages from the TGA 4000

Error message ID	**Error**	**Possible Cause**	**Corrective Action**
ERR_DUE_TO_AUTOSAMPLER	AS 6000 entered fatal error state. This caused TGA 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 TGA and AS 6000 via I2C	I2C connection cable defective	Replace I2C cable

AS 6000 Autosampler Maintenance

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

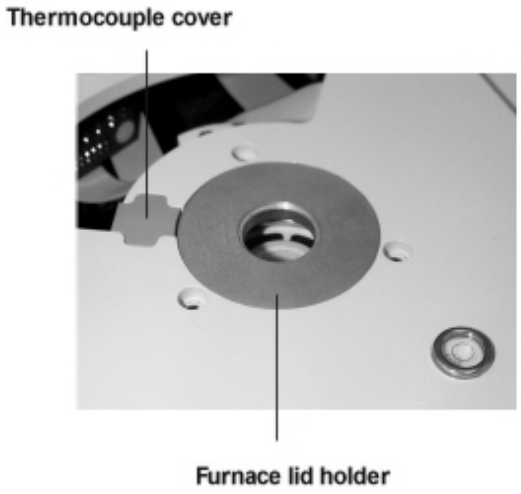
Il n'y a pas de pièces réparables par l'utilisateur à l'intérieur de l'échantillonneur automatique. Renvoyez tous les services à un ingénieur de service qualifié PerkinElmer Instruments.

Replace the Sample Thermocouple in a TGA 4000, Pyris 6 TGA or STA 6000 with AS 6000 Autosampler

1. Switch off the instrument.
2. Remove both sample trays.
3. Remove the four screws that attach the autosampler to the base plate.

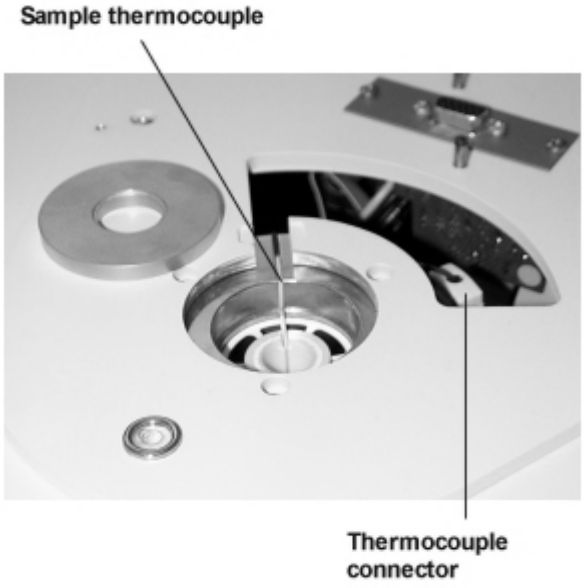
NOTE: Never remove the screws from the plate that is directly attached to the analyzer's cover. This plate is positioned with respect to the furnace. The positioning is performed by the service engineer upon installation.

4. Place your fingers in the indents in the sides of the base plate and lift the autosampler up and away from the instrument.
5. Remove the furnace lid and the lid holder.
6. Remove the base plate thermocouple cover.



7. Remove the alumina furnace cover disk.
8. Remove the alsiflex strip.
9. Remove the sample holder.

10. Remove the thermocouple by disconnecting the thermocouple connector and gently pulling the thermocouple out of the furnace.



11. Install the new thermocouple and replace the sample holder, alumina furnace cover disk, and the alsiflex strip.

12. Replace the lid holder and furnace lid.

13. Replace the base plate thermocouple cover.

14. Place the autosampler on top of the base plate, aligning the screw holes.

15. Attach the autosampler to the base plate with the four screws.

16. Place the sample trays on the autosampler with tray 1–22 toward the front and tray 23–45 on the rear.

After installing a new sample thermocouple, you will have to perform the gripper alignment procedure.