TGA 8000

Installation and Hardware Guide



2. TGA 8000 Installation and Hardware Guide

Release History

Part Number	Release	Released Date	Minimum Software Version	
09931345	В	August 2017	Release 12.0	

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

User Assistance PerkinElmer, Inc. 710 Bridgeport Avenue Shelton, CT 06484

Or emailed to: info@perkinelmer.com

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Safety and Regulatory Information

Symbols Used in this Manual

The Pyris installation and hardware guide contains 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 this manual.

The following safety conventions are used throughout:



We use the term **WARNING** to inform you about situations that could result in **personal injury** if precautions are not followed.

Nous utilisons le terme **ATTENTION** pour vous informer de situations pouvant entraîner des dommages graves à l'instrument ou à d'autres équipements.



We use the term **CAUTION** to inform you about situations that could result in serious damage to the instrument or other equipment.

Nous utilisons le terme **AVERTISSEMENT** pour vous informer de situations pouvant entraîner des blessures corporelles si les précautions ne sont pas respectées.

Symbols Used on the Instrument

The symbols that appear on the instruments discussed in this installation are as follows:



CAUTION - Risk of Danger

Sections in this manual must be consulted to determine the nature of the potential hazard and any actions which have to be taken.

AVERTISSEMENT: Consulter les documents d'accompagnement.

If there is a hazard of heat or electric shock, the following warning symbols will appear on the instrument:



This symbol indicates the danger of burns, if precautions are not followed, due to the presence of heat in this area of the instrument. There may also be hot surfaces that can be easily touched.



AVERTISSEMENT: Surface chaude.

This symbol means **CAUTION** – Cold surface.

Ce symbole signifie **ATTENTION** – surface froide.



This symbol indicates the danger of electric shock, if precautions are not followed, due to the presence of high voltage.

AVERTISSEMENT: Risque de choc électrique.

Additional graphic symbols used on the instrument are the following:



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



CAUTION: The protection provided by this equipment may be impaired if the equipment is used in a manner not specified by PerkinElmer.

ATTENTION: La protection fournie par cet équipement risque d'être moins efficace si l'équipement fait l'objet d'une utilisation différente de celle mentionnée par PerkinElmer.



CAUTION: 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.

ATTENTION: Raccordez l'instrument à une prise secteur ayant une connexion de terre de protection. Pour assurer un fonctionnement satisfaisant et sûr de l'instrument, il est essentiel que le conducteur de terre de protection (le conducteur vert / jaune) du cordon d'alimentation secteur soit connecté à une vraie masse 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.



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

ATTENTION: Ne pas utiliser l'instrument avec des couvercles ou des pièces enlevés.



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

ATTENTION: N'essayez pas d'effectuer des réglages, des remplacements ou des réparations sur cet appareil, sauf comme décrit dans ce manuel. Seul un représentant de service PerkinElmer devrait être autorisé à réparer l'instrument.



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

ATTENTION: N'utiliser que des fusibles avec le courant nominal requis et du type spécifié pour le remplacement.

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.

Electrical Safety Guidelines

Service on electrical components should be performed only by a qualified PerkinElmer service engineer.

Be sure the power cord is the correct one for your laboratory. The TGA 8000 power cord must be rated at or better than 100/120 VAC at 15 A or 150 AC at 10 A. The line cord used must meet the National Safety Agency's guidelines for the particular country.



Fuses

NOTE: Service on electrical components should be performed only by a qualified PerkinElmer service engineer. The fuses in the power entry module can be changed by the user.

Safety Precautions for the TGA 8000



CAUTION: The protection provided by this equipment may be impaired if the equipment is used in a manner not specified by PerkinElmer.

ATTENTION: La protection fournie par cet équipement risque d'être moins efficace si l'équipement fait l'objet d'une utilisation différente de celle mentionnée par PerkinElmer.



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 de l'instrument lisent et comprennent les précautions suivantes. Il est conseillé de poster une copie de ces précautions sur ou près de l'instrument lui-même.



WARNING: The glass parts on the TGA 8000 are fragile and may be subject to breakage if not handled carefully.

AVERTISSEMENT: Les pièces en verre du TGA 8000 sont fragiles et risquent d'être cassées si elles ne sont pas manipulées avec soin.

The following precautions must be observed when using the TGA 8000 Thermogravimetric Analyzer:

• Never turn the computer off until the following message appears:

It is now safe to shut off your computer.

- Never press the Reset button on the computer if the Pyris software appears to malfunction. Press the Ctrl-Alt-Del keys simultaneously and select the Task Manager. From the Task Manager, close the Pyris software.
- Never remove the outer instrument cover without shutting down the instrument and disconnecting its power cord from the power source.
- Only high-quality purge gases should be used. Minimum purity of 99.9% is recommended. A high-quality filter dryer accessory is recommended for the removal of any moisture from the purge gases.
- Always observe the proper startup and shutdown procedures with the TGA 8000 and all related instruments.
- Always maintain a positive purge flow (20 100 cc/min) through the system gas chamber at all times. An inert gas such as nitrogen or argon is recommended as a balance purge.
- Do not raise the furnace when the Pyrex furnace tube has been removed. This will cause severe damage to the furnace and other components of the analyzer.
- Prior to performing any experiment or calibration procedure, be sure that the thermocouple currently installed is functioning properly. The thermocouple can be checked by programming the TGA 8000 to 100°C and checking the temperature displayed in the status panel. The temperature should be close to 100°C if the instrument is calibrated.

- To prolong the life of the standard furnace, it is recommended that the automatic furnace cleaning procedure be performed routinely such as every 5 to 10 runs.
 Pyrolysis products should be burned off after every analysis that involves pyrolysis of a sample in the absence of air or oxygen.
- Always close the furnace door before attempting to run an analysis. When the TGA 8000 standard furnace is heating, keep the protective plastic shield in the down position. This is for **non-autosampler** instruments. This will protect you from accidentally touching the furnace when it is hot.
- Do not attempt to move the TGA 8000 while it is on. Wait until the power is off and the furnace cooling fan has stopped spinning before moving the instrument.
- Do not remove cables from the analyzer while it is on.
- Do not touch the opening on the furnace lift mechanism or insert objects into the opening.
- Do not use hydrogen gas with the TGA 8000 since it is highly explosive.
- 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.

Specifications

Maximum power consumption Rated Power	300W
Supply voltage	100 – 240VAC
Supply voltage frequency	50/60Hz
Fuses Glass Cartridge, 0.213 "Dia x 0.886" L (5.40mm x 22.50)	6.3AT
Safe temperature range	10 – 40°C
Dimensions L x H x D	45.8 cm (18-in) x 48.3 cm (19-in) x 45.8 cm (18-in)
Weight	26kg with autosampler (57lbs)

Warnings and Labels

Warning Label on the Upper Ring





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 au bon fonctionnement de l'équipement. Ne jamais faire fonctionner l'équipement avec le connecteur de mise à la terre débranché. Débrancher le cordon d'alimentation avant de l'utiliser.

Warning Label behind the Furnace





CAUTION – HOT SURFACE which indicates that the furnace at the center of the ring may be hot.

ATTENTION - SURFACE CHAUD qui indique que le four au centre de l'anneau peut être chaud.

Warning Labels on the Back of the TGA 8000





WARNING: Always keep airflow unobstructed. To maintain adequate ventilation, do not block the back of the instrument.

AVERTISSEMENT: Toujours garder l'écoulement d'air dégagé. Pour maintenir une ventilation adéquate, ne pas bloquer le dos 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 la même classe 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 au bon fonctionnement de l'équipement. Ne jamais faire fonctionner l'équipement avec le connecteur de mise à la terre débranché. Débrancher le cordon d'alimentation avant de l'utiliser.



WARNING: Toxic Gases – Fume Ventilation System

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

AVERTISSEMENT: Gaz toxiques - Système de ventilation des fumées Sans ventilation adéquate, des vapeurs potentiellement toxiques peuvent s'accumuler en laboratoire. Votre laboratoire doit disposer d'un système fiable de ventilation des fumées avant d'utiliser cet instrument.

Environmental Requirements for the TGA 8000

The TGA 8000 has been designed for indoor use only and should not be operated in an explosive environment. The TGA 8000 operates most efficiently under the following conditions:

- Ambient temperature range of 10°C 35°C (50°F 95°F). The instrument will remain safe when operated between 5°C and 40°C (41°F to 104°F).
- Ambient relative humidity between 20% and 80% without condensation.
- Altitude no more than 2000 m.
- Clean area free of dust, smoke, vibration, and corrosive fumes.
- On a bench area out of direct sunlight.
- Away from heating or cooling units or ducts.
- The instrument must be positioned so the power switch on the rear panel can be easily reached by the operator.
- There must be an adequate and stable power source for all system components.
- The TGA 8000 is a highly sensitive, precision laboratory instrument. The environment in which it is used must be free of radio frequency interference sources which may affect the performance of the instrument. If performance is affected by RF fields, the instrument should be reoriented or relocated, or separated by a greater distance from the interfering source. Consult PerkinElmer for additional assistance.



WARNING: If the heating of materials could lead to the liberation of hazardous gases, the use of a fume extraction system will be required.

AVERTISSEMENT: Si le chauffage des matériaux peut entraîner la libération de gaz dangereux, il sera nécessaire d'utiliser un système d'extraction des fumées.



WARNING: There may be possible hazards of explosion, implosion, or the release of toxic or flammable gases arising from the materials being heated.

AVERTISSEMENT: Il peut y avoir des risques d'explosion, d'implosion ou de dégagement de gaz toxiques ou inflammables provenant des matières chauffées.



WARNING: Do not mount the instrument of a surface of flammable material.

AVERTISSEMENT: Ne pas monter l'instrument d'une surface de matériau inflammable.



CAUTION: Before using any cleaning or decontamination method, except that recommended in this guide, the user should check with PerkinElmer Instruments that the proposed method will not damage the instrument.

ATTENTION: Avant d'utiliser toute méthode de nettoyage ou de décontamination, à l'exception de celle recommandée dans ce guide, l'utilisateur doit vérifier auprès de PerkinElmer Instruments que la méthode proposée n'endommagera pas l'instrument.

Hazardous Chemical Warnings

Before using any chemicals or solvents with the instrument, you should be thoroughly familiar with all hazards and safe handling practices. Observe the manufacturer's recommendations for use, storage, and disposal. These recommendations are normally provided in the material safety data sheets (MSDS) supplied with the solvents.



We use the term **CAUTION** to inform you about situations that could result in serious damage to the instrument or other equipment.

Nous utilisons le terme **AVERTISSEMENT** pour vous informer de situations pouvant entraîner des blessures corporelles si les précautions ne sont pas respectées.



WARNING: Some chemicals used with this instrument may be hazardous or may become hazardous after completion of an analysis. The responsible body (e.g., Lab Manager) must take the necessary precautions to ensure that the surrounding workplace and instrument operators are not exposed to hazardous levels of toxic substances as defined in the applicable Material Safety Data Sheets or OSHA, ACGIH, or COSHH documents. Venting for fumes and disposal of waste must be in accordance with all national, state, and local health and safety regulations and laws.

AVERTISSEMENT: Certains produits chimiques utilisés avec cet instrument peuvent être dangereux ou risquent de devenir dangereux une fois l'analyse terminée. L'organisme responsable (p. Ex., Le responsable du laboratoire) doit prendre les précautions nécessaires pour s'assurer que le lieu de travail environnant et les exploitants d'instruments ne sont pas exposés à des niveaux dangereux de substances toxiques tels que définis dans les fiches de données de sécurité ou les documents OSHA, ACGIH ou COSHH. L'évacuation des fumées et l'élimination des déchets doivent être conformes à toutes les réglementations et lois nationales, nationales et locales en matière de santé et de sécurité.

Some definitions of terms used above are:

- **Responsible body:** "Individual or group responsible for the use and maintenance of equipment, and for ensuring that operators are adequately trained" [per IEC 1010-1, Amendment 2].
- **Operator:** "Person operating equipment for its intended purpose" [per IEC 1010-1, Amendment 2].
- **OSHA:** Occupational Safety and Health Administration (United States)
- **ACGIH:** American Conference of Governmental Industrial Hygienists
- **COSHH:** Control of Substances Hazardous to Health (United Kingdom)

Installation Category

This instrument is able to withstand transient overvoltage according to Installation Category II as defined in IEC 1010-1.

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.

South Korea

A급 기기 (업무용 정보통신기기)

이 기기는 업무용으로 전자파적합등록을 한 기기이오니 판매자 또는 사용자는 이 점을 주의하시기 바라며, 만약 잘못판매 또는 구입하였을 때에는 가정용으로 교환하시기 바랍니다.

Ingress Protection

The TGA 8000 is rated IP20 as defined in IEC 60529.

Storage Conditions

The TGA 8000 may be stored under the following conditions:

- ambient temperature from -20°C to +60°C (-4°F to +140°F)
- ambient relative humidity from 20% to 80% noncondensing
- altitude in the range of 0 12,000 m

WEEE Instructions for PerkinElmer Products



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

The objectives of this program are to preserve, protect and improve the quality of the environment, protect human health, and utilize natural resources prudently Requirements for waste collection, reuse, recycling, and recovery programs vary by regulatory authority at your location. Contact your local responsible body (for example, your laboratory manager) or authorized representative for information regarding applicable disposal regulations. Contact PerkinElmer at the web site listed below for information specific to PerkinElmer products.

Web address:

http://www.perkinelmer.com/WEEE

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

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

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

Prepare the Laboratory

Overview

Thermogravimetry is a branch of thermal analysis that examines the mass change of a sample as function of temperature or time as it is subjected to a controlled temperature program in a controlled atmosphere. Not all thermal events cause a change in the sample mass (for example, melting, crystallization, glass transition), but there are some important exceptions such as desorption, absorption, oxidation, decomposition. Thermogravimetry characterizes the decomposition and thermal stability of materials under a variety of conditions and examines the kinetics of the physico-chemical processes occurring in the sample.

The TGA 8000 is a computer-controlled analyzer that has been designed to take advantage of the enhanced features of Pyris Software. It is made up of two major components: a microbalance and a furnace element.

About the Microbalance

The microbalance of the TGA 8000 operates as a high gain electromechanical servo system that permits detection of weight changes as small as $0.1 \ \mu$ g, with a maximum capacity of 1300 mg. The null balance design uses a servo-controlled torque motor to automatically compensate for weight changes in the sample material. When a sample is placed in a sample pan and the pan is on the hangdown wire, the beam that supports the sample pan deflects. A beam position detector measures the deflection with an optical sensor and uses current to return the beam to its original position. The amount of current necessary to maintain the system in the "null" state is directly proportional to the weight change in the sample. The current is amplified and filtered and then displayed in the display panel of the instrument in mg.

About the Furnace

The furnace allows operation of the TGA 8000 from subambient to 1200°C. The furnace uses a microfurnace that has a low thermal mass for quick cooling and equilibration. Accurate program temperature control reduces overshoot to 0.2°C at 100°C/minute which results in stepwise analysis precision. The sample temperature is sensed by a chromel–alumel thermocouple.

Features

Some of the features of the TGA 8000 are:

- 1. Faster furnace cool-down with the cooling fan above the furnace instead of underneath. This creates positive upward ventilation of the furnace during cool-down.
- 2. iPad control.
- 3. The sample gas interface has been repositioned for easier access.
- 4. Controlled balance environment gives the TGA 8000 long-term ordinate stability. The weighing balance is housed in a thermally-isolated enclosure that eliminates thermal gradient effects.
- 5. An anti-convection iris prevents decomposition products and heat from the furnace from backstreaming into the balance chamber.
- 6. The "quick release" furnace tube makes furnace tube cleanup easy. The furnace tube is attached to the furnace base by a clamp.
- 7. Temperature control of the segmented furnace is optimized by the heater/sensor furnace technology.

- 8. The removable quartz furnace chamber sleeve regulates furnace convection by reducing the effective furnace volume. The reduced volume also enhances gas switching time because there is less furnace volume to displace.
- 9. The antistatic device creates an ion stream which is an invisible curtain of charged particles that surrounds the sample loading area. This minimizes static cling between the sample pan and the furnace wall during sample loading and static drift during analysis. Loading fine, powdered, or static-sensitive samples is no longer a problem. The device can be controlled from the control panel in the software.

In addition to these features, there is an autosampler designed for both the R&D laboratory and the quality control/quality assurance environment. The autosampler increases the speed of analysis by being able to hold up to 48 samples ready for analysis. The autosampler enables samples to be prepared and run during the same shift. Using the Pyris Player feature of Pyris Software, you can create play lists that will run the samples in the autosampler tray and analyze the data automatically.

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. You can get from one topic to the next by either returning to this topic or by clicking on a "continue" hotspot at the end of the topic.

Operating Variables in the TGA 8000

In thermogravimetric analysis, the object of an experiment is to record the change in weight of a sample as function of temperature or time using a constant heating rate or a more complex temperature program. Below are some techniques that can be used to obtain the most accurate data in a minimum amount of time with a TGA 8000.

Sample Preparation

The TGA 8000 can analyze solid or liquid samples. Solid samples can be in the form of film, crystal, or grains. In some cases, you may want to chop or grind the sample to create a large surface area that is exposed to the purge gas atmosphere. In other cases, larger chunks or sections of sample may be placed directly into the sample pan or crucible for analysis.

Sample Size

The size of sample that can be analyzed ranges from <1 mg to 400 – 500 mg. In most cases, the typical sample size for TGA analysis is 2 – 50 mg. The type of transition or reaction that you expect to occur in the sample should dictate the amount of sample used. For example, in the case of polymer decomposition studies, sample sizes of 2 – 15 mg are recommended since very large weight loss values are associated with this type of experiment. If there will be a very small weight loss such as with water loss or solvent evaporation, larger sample sizes should be used. It is not uncommon to use samples as large as 40 - 50 mg.

Temperature Range

The temperature used for your experiment will depend on the type of samples to be run as well as the specific application performed. The standard furnace operates from subambient to 1200° C with a temperature precision of $\pm 2^{\circ}$ C. This furnace has broad applications in the polymer, pharmaceutical, and organic chemical industries.

Because of the unique design of the TGA 8000, you can perform controlled heating or cooling experiments as well as constant temperature experiments as a function of time. You can even perform multiple-step experiments using any combination of heating, cooling, or isothermal segments, thereby allowing the performance of virtually any thermogravimetric

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application. In addition, there is also an AutoStepwise Scan available in the method program. This scan heats the sample rapidly between weight loss regions, and slows down or stops heating during rapid weight loss regions.

Scanning Rate

Scanning rates range from 0.1°C/min to 500°C/min for the standard furnace. The exact scanning rate used will depend on the experiment and the end result you are trying to achieve. For example, most TGA experiments are performed at heating rates of 5°C/min to 50°C/min. However, there may be times when you want to heat or cool rapidly to a selected temperature and then hold isothermally or scan a controlled rate. In such cases, very fast heating or cooling rates (i.e., 100°C/min or 200°C/min) are typically used to quickly increase or decrease the sample temperature.

Sample Atmosphere



WARNING: Due to the highly explosive nature of hydrogen, it is recommended that it not be used with this instrument.

AVERTISSEMENT: En raison de la nature très explosive de l'hydrogène, il est recommandé de ne pas l'utiliser avec cet instrument.

The atmosphere to which the sample is exposed is carefully controlled by the selection of the sample purge or sample gas and flow rate. Recommended sample gases are nitrogen, argon, helium, carbon dioxide, air, oxygen, or other inert or reactive gases, as long as they do not react with the furnace materials, thermocouple, or other analyzer components in which gases come into contact. Analyses are done at normal pressure or at reduced pressures.

In addition, sample gases may be switched at any point during an analysis, for example, the pyrolosis of a polymer occurs while in an inert atmosphere (Sample Gas A) then switching to an O2 or Air to oxidize the remaining components (Gas B), by using the internal gas switching capabilities which accurately control the flow rates of both gasses.

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

Cooling Device

Chiller: -20°C and above

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: TGA 8000: 100 - 240 +/- 10%, 50/60 Hz. +/- 1%

Purge Gas and Pneumatic Supply Requirements

Minimally, the TGA 8000 requires a System Purge gas, also known as the balance purge. This gas must be clean and dry. System gas is controlled by a mass flow controller resident in the analyzer. Pressure, between 2-3bar is applied at the connector at the rear of the analyzer labeled "System Gas". Typically this is an inert, non-reactive gas i.e. N_2 , Ar, or He. Gases such as Air or O_2 can also be used.

A Sample Purge gas is not a requirement to operate the TGA, however it is recommended. There are two inputs, A and B for the sample gas which are also controlled by a separate mass flow controller, allowing for gas switching experiments. Typically, "Sample Gas A" is the same gas that is purging the "System" (Balance) and "Sample Gas B" would be Air or O2. Switching gases at elevated temperatures facilitates the burning of the sample and aids in the cleaning of the furnace and sample pans.

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.

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



CAUTION: Maximum pressure should not exceed that listed in the chart below.

ATTENTION: La pression maximale ne doit pas dépasser celle indiquée dans le tableau ci-dessous.

Gas	Pressure	Flow	Purity (Minimum)
System Gas: Argon, nitrogen, helium, air or oxygen	29- 43.5 psi (2- 3 bar)	0-200 cc/min	99.95
Sample Gas: Argon, nitrogen, helium, air or oxygen	29- 43.5 psi (2- 3 bar)	0-100 cc/min	99.95
Reactive Gas: Argon, nitrogen, helium, air or oxygen	Unpressurized	0-100 cc/min (Controlled with restrictor at the source gas)	99.95

Install a Filter Dryer

The filter dryer is an optional accessory that is used to remove moisture, carbon dioxide, and other impurities from your purge gas or any other gas used with a particular analyzer. Refer to Ordering Spares and Accessories, or contact your local PerkinElmer sales representative for assistance when ordering this accessory.

Space Requirements

Prepare a clean bench space large enough to accommodate all system components and accessories. Click on the appropriate analyzer below to display the dimensions of a component. For computer and printer dimensions, refer to the manufacturers' instructions that were included with them.

NOTE: Before the instrument is installed, the area around, under and behind the instrument's planned location is to be cleared of any dirt and dust to prevent their entry into the instrument's interior, which could adversely affect performance.

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

Order Spares and Accessories

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

Installing the TGA 8000

TGA 8000 Installation Summary

The installation procedure for the TGA 8000 comprises the following steps:

- Prepare the Laboratory
- Unpack the TGA 8000
- Set Up the TGA 8000 System Components
- Connect the TGA 8000 System Components
- Configure the TGA 8000
- Install the Furnace Hangdown Wire and Balance Tare Weight
- Calibrate the TGA 8000
- Install the Cooling Jacket Accessory or the Accupik Accessory (if applicable)

Instructions for installing a TGA 8000 with an autosampler are included where appropriate.

Unpack the TGA 8000

Each component of the TGA 8000 system comes in its own box. The system components are:

- TGA 8000
- Personal Computer
- Printer (optional)
- Installation Kit containing the Spares Kit



CAUTION: Take care when unpacking the TGA. Some of the boxes packed with it contain fragile parts that can break easily.

ATTENTION: Faites attention lors du déballage du TGA. Certaines des boîtes emballées avec elle contiennent des pièces fragiles qui peuvent se casser facilement.

The autosampler is used to automate the running of samples. If you ordered a TGA with an autosampler, it is shipped with the autosampler already installed.

When you unpack the box, check all the parts included against the shipping list. If any part is damaged or missing, contact the shipping company and PerkinElmer immediately.

The TGA is shipped in a large box that contains the analyzer and a box that contains the Install Kit (P/N N5320120). In the Install Kit is the Spares Kit (P/ N N5320026).

To unpack the TGA main box:

1. Place the instrument carton on the floor in front of the bench on which the instrument will be placed. Open the box.



2. Remove the box from the packing, then remove the autosampler tray and furnace door and set them aside.



- **NOTE:** The furnace shield is shipped in the inverted position on the furnace door.
 - 3. Unscrew and remove the furnace shield from the furnace door, invert it then secure it on the furnace door.
 - 4. Remove the top foam piece that secures the instrument in the box.
 - 5. Prepare to lift the instrument out of the carton.



WARNING: Since the instrument weighs 26 kg (57 lb), **two people** are needed to safely do this. The bench should be nearby. **DO NOT** lift the unit by the top round dome. This dome protects the balance mechanism and is not screwed down.

AVERTISSEMENT: Puisque l'appareil pèse 26 kg (57 lb), **deux personnes** sont nécessaires pour le faire en toute sécurité. Le banc doit être à proximité. **NE soulevez PAS** l'appareil par le dôme supérieur. Ce dôme protège le mécanisme de l'équilibre et n'est pas vissé.

- 6. One person should put one hand underneath the bottom of the instrument and the other hand beneath the front side. The other person should place one hand underneath the top of the instrument and the other hand underneath the back side. Lift the instrument simultaneously.
- 7. Place the analyzer carefully on the prepared bench.
- 8. Remove the tape on top of the dome.

Gently lift the dome cover STRAIGHT UP to ensure you clear the balance mechanism.

- 9. Remove the tape securing the balance and the zip tie securing the furnace.
- 10. If there is an autosampler attached, cut the strap holding it in place as shown to the right of the image below).



The procedure for unpacking the TGA is complete.

Set Up TGA 8000 System Components

Setting up the TGA 8000 system components consists of the following steps:

- Install the Purge Gases
- Level the TGA 8000

Install the Purge Gases

The TGA 8000 has gas inlets at the rear of the analyzer, (Sample Gas A and B, System (Balance) Gas, and Reactive Gas. Having two separate purges allows the flow rate of the purge gas in the balance area to be greater than that in the furnace tube area. This creates positive pressure that helps prevent reactive gases from entering the balance area. Using both a balance purge and a sample gas allows you to use reactive gases for various analyses. This System gas supply is used for the antistatic device. Finally, there is an inlet on the rear panel to connect Reactive gas that enters the sample area directly.

Overview of the Purge Flow through the TGA 8000

The balance purge gas enters the back of the analyzer and then the cavity under the balance cover. It then flows down the Pyrex ball joint into the furnace tube between the tube and the liner. It exits the side arm of the furnace tube into the exhaust tubing.



CAUTION: The system purge flow rate should always be greater than the sample gas flow rate to prevent decomposition products from entering the balance chamber. The recommended flow rate is 20 - 100 ml/min for the balance purge and 10 - 50 ml/min for the sample gas.

ATTENTION: Le débit de purge du système doit toujours être supérieur au débit de gaz échantillon afin d'éviter que les produits de décomposition ne pénètrent dans la chambre d'équilibrage. Le débit recommandé est de 20 - 100 ml / min pour la purge de l'équilibre et de 10 - 50 ml / min pour le gaz échantillon.



CAUTION: The total flow rate of the balance purge gas, sample gas, and reactive gas should be below 200 ml/min.

ATTENTION: Le débit total du gaz de purge d'équilibrage, du gaz échantillon et du gaz réactif doit être inférieur à 200 ml / min.

The Sample Gas enters through connectors (labeled Sample Gas A or Sample Gas B) on the back of the TGA 8000. It is applied as pressure 1 - 3 Bar (optimum 2 Bar). The software controls the gas flow controller to the sample area. Gas is directed to the furnace through Teflon tubing from the flow controller to an inlet port on the bottom of the Balance mounting plate. From here it flows through a chamber out of the plate and above the anti-connection tube into the furnace area..

You can use the same gas source (house line or cylinder) for both balance system purge gas and sample gas by using a serial pressure gauge setup (P/N N5190462).

Connecting the System Purge and Sample Gas Lines to the Gas Supply

To connect the system purge and sample gases, using separate sources, you will need parts included in the Spares kit (P/N N5320120).

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- 1. Connect suitable regulators to the gas supplies that will be used to purge the system (the customer is responsible for supplying the regulators). The regulators must be equipped with shutoff valves at the outlet. The shutoff valves must have a 1/4-in. NPT male thread on the outlet side.
- 2. Tape the threads of the outlet fitting on both shutoff valves with the thread sealant tape provided (P/N 09908134).
- 3. Connect the end of a female connector (P/N 09903196) to the taped male end of the shut off valve.
- 4. If you did not install a filter dryer into the purge gas or sample gas line, click here. Click on the **Back** button to return here.

If you wish to install the GMD 8000, click here.

- 5. Place a 1/8-in. nut and ferrules over one end of the 1/8-in. Teflon tubing (P/N 02506483).
- 6. Connect the 1/8-in. Teflon tubing and nut to the end of the NPT male connector on the gas supply.
- 7. Repeat steps 4 6 for the other gas line.

Connecting the System Purge Gas Line

- 1. Place a 1/8-in. nut and ferrules over the other end of the 1/8-in. Teflon tubing which is connected to the gas cylinder that is to be used for the System purge.
- 2. Connect the end of the Teflon tubing to the fitting labeled Balance Purge located on the back of the TGA 8000. Tighten the nut fingertight and then, using a wrench tighten a quarter turn past fingertight.
- **NOTE:** If you are splitting the System Purge to supply also Sample Gas A, cut a short length of 1/8in Teflon tube and connect as described above by using the T-splitter. Connect the line to System and the GSA to splitter.

Connect the Sample Gas Line

- 1. Place a 1/8-in. nut and ferrules over the other end of the 1/8-in. Teflon tubing which is connected to the gas cylinder that is to be used for the sample gas.
- 2. Connect the end of the Teflon tubing to the fitting labeled Sample Gas located on the back of the TGA 8000. Tighten the nut fingertight and then, a wrench, tighten a quarter turn past fingertight.

Connecting the System Purge and Sample Gases Using a Common Source

Instead of using two gas cylinders containing the same gas or two house lines of the same gas, you can use one source of gas for both the system purge gas and the sample gas, for example, compressed air or nitrogen. This can be done by installing gauges in series off of the regulator on the source. These gauges are P/ N N5190462. Detailed instructions on how to put two or more gauges together prior to installing them on the regulator are included with the gauges. You should also install the connectors for the tubing to the outlets of the gauges before attaching the gauges to the source. The cylinder or house line should be prepared with a regulator as described above. Attach the series of gauges to the outlet connector on the regulator with the arrows on the gauges, indicating the direction of the flow of gas, pointing away from the source.



Exhaust Gas Tubing

- 3. Connect the barbed fitting (P/N 09920026) to the exhaust port on the rear panel.
- 4. Insert the end of a piece of 0.25-inch Tygon tubing and to the barbed fitting connector on the back of the instrument labeled EXHAUST. The tube should be placed beneath a hood vent if you expect to have noxious exhaust fumes. Otherwise, it can just hang from the instrument.



WARNING: If the heating of materials could lead to the liberation of hazardous gases, the use of a fume extraction system will be required.

AVERTISSEMENT: Si le chauffage des matériaux peut entraîner la libération de gaz dangereux, il sera nécessaire d'utiliser un système d'extraction des fumées.

Connecting Reactive Gas

The TGA 8000 has a reactive gas assembly with which a reactive gas can enter the sample area. The reactive gas enter through the rear of the TGA

When using corrosive reactive gas, connect it to a connector on the rear of the balance dome. The assembly is underneath the balance dome and goes down into the upper furnace tube (ball joint).

Connect the TGA 8000 Components

The computer and optional printer should already be connected together and the Pyris software already installed. To complete the connection of the system components, the TGA 8000 has to be connected to the computer.



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

ATTENTION: Assurez-vous que tous les composants sont éteints et débranchés avant de commencer.

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The image below shows the rear panel connections on the TGA 8000.



- 1. Install the Pyris Software
- 2. Connect one end of the USB cable to the back of the TGA 8000.
- 3. Connect the other end of the USB cable to the computer.

IComputer will recognize new hardware, install the drivers from the Prgram Files\PerkinElmer\Pyris\Drivers folder.

- 4. From device manager determine which comport is assigned to the TGA8000
- 5. Connect the printer cable, if applicable, to an unused USB port on the computer.
- 6. Connect the analyzer, printer and computer power cords to AC outlets.
- 7. Once the system connections are made, switch on the system in the following order:
 - Computer
 - TGA 8000
 - Printer
- 8. Turn on the purge gas supply, and the water for the TGA 8000 cooling system, if applicable, or any other accessories for the cooling device you are using.

Configure the TGA 8000

Highlighting the TGA 8000 in Pyris Configuration dialog and clicking Edit displays the Instrument Configuration dialog. This contains fields for Name, Port, Serial Number, Accessories.

Before acquiring data, the software must be configured for the analyzer(s) connected to the computer. Follow the steps below to configure the TGA:

- 1. In the **Device Manager** determine the COM Port assigned to the TGA 8000.
- 2. Select **Pyris Configuration** from the Pyris group under **PerkinElmer Applications** on the **Programs** menu accessed from the **Start** button or select **Configure Analyzer** from the Pyris Manager Start button menu. The **Configuration** dialog box appears on the screen.

Analyzer Name: TGA 8000	Gas: Integrated Gas
Port COM3	Autosampler: Mounted
Serial Number: 52685020603	
Remote Control	
Enable Maxima	al Clients: (1-60)
Firmware Version	

The following is a description of the controls on the TGA 8000 Configuration dialog:

<u>Name:</u> If you are adding a TGA 8000, the system will display a default name in this field. Type the name you wish to assign to the analyzer, using a maximum of eight characters. The name identifies the analyzer in Pyris Software; it also appears in the Instrument button on the Pyris Manager panel and in the title bar of the Instrument Application.

Port: Displays the COM port to which the analyzer is attached and that you selected in the Add Analyzer dialog box. Best on Ports 1,2,3,4

Serial Number: Displays the serial number of the analyzer.

Accessories: Lists the available accessories for the analyzer. For the TGA 8000, the following accessories are available:

- Cooling Jacket Accessory
- Accupik
- GMD 8000.

Firmware Version: Click the Firmware Version button to display the version of firmware in the analyzer.

3. Click the Add Analyzer button. The Add Analyzer dialog box appears.

Add Analyzer	23
	Add
Port: COM1	Cancel
	Help

When a TGA 8000 is connected to the PC and has been added to the Pyris software using the **Add Analyzer** dialog, it should be identified in the Pyris **Configuration** dialog. If more than one TGA 8000 has been added, then the second one needs a unique name when being connected.

- 4. From the list of available ports, select the port to which you connected the analyzer. The TGA's Configuration dialog box is displayed.
- 5. The system detects the type of analyzer that is attached and displays the default name and other information for a TGA in this dialog box. Select the accessory, if any, that is connected to the TGA 8000 (for example, GMD 8000, Accupik).

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 - 6. Click **OK** to accept the entries.
 - 7. Click **OK** again 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 powered on, otherwise it will not be recognized by the Pyris software.

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

Starting the Pyris Manager

- 1. Select **Pyris Manager** from the Pyris group under **PerkinElmer Applications** in **All Programs** on the **Start** menu. The **Pyris Manager** is displayed on the screen.
- 2. Select TGA 8000 from the Pyris Manager. The TGA application loads and the Control Panel is displayed.

If you have an autosampler, you should now go to Autosampler Adjustment.

Configure the iOS App for the TGA 8000

This procedure will help guide you in configuring the TGA 8000 for use with an iPad/iPhone. The interface is designed so the Pyris Light app communicates with the TGA8000 via the PC which is connected to your company's network. It is important that you confirm with your network administrator that the PC has access to the network and that the iOS device can connect to the network via WiFi. There is no direct communication between the iPhone/iPad and the TGA 8000.

Requirements:

- iPhone (V6 and V6Plus) or iPad (V Air and V Air2). iOS version supported: iOS V8
- Lenovo M83 with Win7 (64bit) PKI supplied
- TGA 8000 with Pyris v12.1.1 connected to the PC via USB cable
- Pyris running and connected and communicating with the TGA 8000
- PC connection to the network
- Pyris Light App from iTunes

To set up an iOS connection follow this procedure:

1. Select **Configure Analyzer** from the **Start Pyris** button.



The TGA 8000 Configuration dialog appears.

2. In the **Remote Control** section click the Enable checkbox and select the Maximum number of devices the TGA 8000 can connect to. When complete click OK

TGA 8000 Configuration	X
Analyzer Name: TGA 1505 R Port: CDM3 Serial Number: 52685061505	Accessories Gas: Integrated Gas Autosampler: Mounted 🔽 Enable
Remote Control	30 🔵 (1-60)
Firmware Version	incel Help

- 3. Select **Remote Control** from the Pyris **Tools** menu.
- 4. In the **Remote Control** dialog the **Local IP** address and **Port** of the PC that the app will connect with is displayed. Ensure the **Start** button is selected. The green **Status** light confirms the port is open.

Status:	Cocal IP:	165.88.116.94 F	Port: 51201	Start	Stop
lients					
No	Connect Time	IP	Port		
		Refuse			
Please	click the right icons to do	wnload Pyris App.	iOS		

5. Open the **Pyris Light**

app on your iPad or iPhone.

The app opens.

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- 6. Click the New Analyzer button
- 7. Enter the **IP Address**, **Port Number**, and **Name of the Analyzer** (enter any name) then click **Next**.

iPad 12:27 PM	20% 🗁
Cancel New Analyzer	
ANALYZER	
165.88.116.94	
51201	
TGA8000	0
Nevt	
Place level the ID address and east of Duie scales which you want to seemed with, and then move to and	
Please input the P address and port of Pyris service which you want to connect with, and then move to next.	
Q W E R T Y U I O P	
ASDFGHJKL	Jone
★ Z X C V B N M ! ?	+
.?123 😄 .?123	Ű

This displays the **Settings** screen which displays the **General** settings, **Display** (graph) settings and **Network** connection settings.
iPad 🗢	12:28 PM	20% 🕞
< Back	Settings	Done
GENERAL		
Analyzer		TGA 8000
Display Name		TGA8000
Pyris Version		12.1.1.0101
Serial Number		526B5061505
DISPLAY		
X-axis		Time >
Curves		3 >
Auto Scale		
Time Range		Infinity >
NETWORK		
IP Address		165.88.116.94
Port		51201
	Accept	

8. Click **Accept** (these settings can be changed at any time). This displays the Main Screen displaying a list of all Analyzers.

iPad 훅	12:28 PM	20% 🗁
	Pyris	+
8	TGA8000 165.88.116.94:51201	>

9. Select your analyzer and the connection will be made. This is confirmed when the confirmation page appears.

iPad	3:45 P	M	Not Charging
🗙 Main	TGA80	000	
30.06 °C	-93.973 m	9	-9397.32% Ready
Furnace	AS	Method	View
•			At Temp
Furnace Status			At Lowered
AutoSampler Status			Returning
Up			Down
Press Up or Down button to me	ave furnace when the auto	eamplar is not husy	

- 10. Connection can also be confirmed by observing the **Remote Control** dialog in Pyris which displays the Local IP address and Port of the iPad or iPhone. Connection permissions can be controlled with all devices within this dialog.
 - **Start/Stop** button Starts and Stops connection with all devices.
 - **Refuse** button Blocks connection with this device.

erver Status:	🧿 Local IP: 📘	65.88.116.94 P	ort: 51201 Start	Stop
lients				
No	Connect Time	IP	Port	
1	12:28:21	165.88.182.137	21185	
		Refuse		
Please (click the right icons to dowr	nload Pyris App.	ios	

Initial Setup for the Balance and Hangdown Wires



CAUTION: Initial installation or movement of the instrument after installation calls for the below steps to be performed.

ATTENTION: L'installation initiale ou le déplacement de l'instrument après l'installation nécessite l'exécution des étapes ci-dessous.

- Leveling the TGA 8000
- Installing the hangdown wire and tare weight
- Adjusting the Convection Iris
- Adjusting the balance mechanism

Leveling the TGA 8000

Level the instrument using the spirit level provided.

- 1. Position the analyzer in the exact spot on the bench where you will be using it.
- 2. Remove the dome cover by gently lifting the cover straight up at least 8" after it is released from the balance plate. Failure to do this can result in damaging the balance mechanism!
- 3. Observe the level on the balance plate near the flange of the anti-convection tube.



- 4. Check to see if the analyzer rocks on the bench or if one foot is not touching the bench. Starting with that foot, turn the foot clockwise until it is touching the bench. Once all the feet are touching the bench, make sure that the analyzer is steady.
- 5. Look at the level to see if the bubble is in the center. If it is not, slowly turn the knobs of the adjustable feet until the bubble is centered in the level. The TGA 8000 is leveled when the bubble is centered inside the ring.
- 6. Once the analyzer appears to be level, make sure all of the feet are in contact with the bench and that the analyzer does not rock.

You have now finished leveling the TGA8000. Final leveling will be complete after the Autosampler calibration.

Install the Furnace Hangdown Wire

The TGA8000 comes with a kit that contains standard hangdown wires which is hung on the platinum hangdown ribbon on the furnace side of the balance.

To install a hangdown wire and tare weight, follow the procedure below:

- 1. Remove the dome cover by gently lifting the cover straight up at least 8" after it is released from the balance plate. Failure to do this can result in damaging the balance mechanism!
- 2. Expand the iris opening by pushing the iris arm in a clockwise direction.
- 3. Using tweezers, pick up the appropriate tare weight for the TGA 8000 and carefully hang it from the loop of the hangdown ribbon located on the right arm of the balance.
- 4. Place the hangdown wire alignment tool (P/N N5376622) on the anti-convection cap.



CAUTION: The platinum hangdown ribbons are extremely fragile. Be careful not to exert force on the loops of the hangdown ribbon or on the hangdown wire after it is attached. Handle tweezers carefully when placing them near the platinum hangdown ribbons, the hangdown wire, or stirrups as they may catch on one of the components.

ATTENTION: Les rubans de platine sont extrêmement fragiles. Veillez à ne pas exercer de force sur les boucles du ruban suspendu ou sur le fil pendulaire une fois qu'il est fixé. Manipulez délicatement les pinces en les plaçant près des rubans de pendage en platine, du fil pendulaire ou des étriers, car ils peuvent s'accrocher à l'un des composants.

- 5. Using tweezers, carefully pick up a hangdown wire and drop the wire through the hangdown wire tube. The wire will rest on the alignment tool.
- 6. Using tweezers, grasp the hangdown wire with the tip facing the ribbon loop and insert the tip through the loop; release the hangdown wire.
- 7. Remove the alignment tool and gently place a pan on the end of the hangdown wire.



Adjusting the Convection Iris

The iris is an anti-convection device. It prevents reaction gases from rising into the balance chamber from the furnace.



CAUTION: If the iris is not adjusted properly then soot from the sample will contaminate the balance mechanism. It is important that the hangdown wire does not touch the iris when it is closed.

ATTENTION: Si l'iris n'est pas réglé correctement, la suie de l'échantillon contaminera le mécanisme de l'équilibre. Il est important que le fil pendulaire ne touche pas l'iris lorsqu'il est fermé.

- 1. Close the iris until it is approx 1mm clearence between the edge of the iris and the hangdown wire all around. The wire should be centered in the iris.
- 2. If it is not centered then the balance mechanism must be adjusted.



Adjusting the Balance Mechanism

If the hangdown wire is not centered in the iris then you will have to adjust the balance mechanism position.



CAUTION: The balance mechanism is very fragile. Use extreme caution when handling.

ATTENTION: Le mécanisme de l'équilibre est très fragile. Soyez extrêmement prudent lors de la manipulation.

- 1. Ensure that the upper cooling fan is off; otherwise, it will cause the hangdown wire to swing.
- 2. The adjustment screws are easily accessible (one shown below red arrow). Loosen the screws and carefully move the balance to position the hangdown wire in the center of the iris.
- **NOTE:** A small amount of rotation of the hangdown wire (see Autosampler Calibration) is possible by rotating the balance about the hangdown wire centerline.
 - 3. Tighten the balance screws.

- 4. Adjust the iris again as per previous section.
- 5. Replace the balance cover



Autosampler Calibration



CAUTION: If the TGA 8000 is moved or installed for the first time, the Initial Setup for the balance and hangdown wires must be performed prior to Autosampler Calibration to ensure reliable operation of the autosampler.

ATTENTION: Si le TGA 8000 est déplacé ou installé pour la première fois, la configuration initiale des fils d'équilibrage et de déconnexion doit être effectuée avant l'étalonnage de l'échantillonneur automatique afin d'assurer un fonctionnement fiable de l'échantillonneur automatique.

1. Ensure the hangdown wire is facing in the direction of the sample tray. When a pan is hung on the hangdown wire the stirrups must be inline with the furnace center and the large black column that supports the balance mechanism.



2. In the Pyris Software TGA 8000 screen (located at the top of the screen) click Calibrate Autosampler from the **Calibrate** menu.



The Autosampler Calibration dialog box is displayed:

TGA8000 Autosampler	Calibration	
Eurnace Positio	n	Tray <u>R</u> otation
	Tray <u>V</u> ertical	
	Restore Default Va	lues
	<u>C</u> lose	

- 3. Calibrate the tray height by clicking on the **Tray Vertical** button and follow the onscreen prompt. Make sure an empty sample pan is present in position 1 in the sample tray. Ensure the hook of the hangdown wire is:
 - Aligned 2-3mm below the apex of sample pan handle (right image below)
 - The hangdown wire is centered within the furnace tube (right image below))
 - Aligned on the centerline of the apex. (left image below).
- **NOTE:** If the hangdown is not on the centerline of the apex, the unit will have to be adjusted using the leveling feet. Either raise or lower the foot nearest the furnace to ensure the hangdown wire is on center with the apex. If this procedure is done, re-check to ensure the iris is not contacting the hangdown wire (see Adjusting the Convection Iris section).



4. Calibrate the sample pickup and drop-off position by clicking on the **Tray Rotation** button and follow the on-screen prompt. For this calibration an empty sample pan must be present on the hangdown wire before starting. During the calibration the sample pan should hang on the centerline of the resting position on the tray to 1-2mm to the left the center as shown below.



5. To load an empty sample pan click on the Autosampler Control button.
The Autosampler Control dialog box is displayed:

ample Commands	
Select Carousel Location: 1	(1-48)
Load Sample Unload S	ample Change Sample
Piercing Mode: OFF	Change
Piercing Position	Piercing Depth
lovement	
Move Tray Upper	Move Tray Lower
Rotate Counterclockwise	Rotate Clockwise
Move All Defau	ult Position

6. Make sure an empty sample pan is present on position 1. Select carousel location as 1 and click **Load Sample** button.

Calibrate the TGA 8000

The TGA 8000 is calibrated at the factory and this calibration file is loaded from the CD upon installation. There is also a default calibration which removes all of the calibration adjustments and reverts to the hardware presets.

The TGA must be calibrated before obtaining accurate data. Calibration is performed using the Pyris Software calibration routine which is accessed from the **Calibrate** menu through the Method Editor window. There are four independent calibration routines accessed through the three tabs on the Calibration menu item that should be performed for a TGA with or without an autosampler:

- Weight Calibration
- Temperature Calibration
- Balance Drift Optimization
- Baseline Drift Optimization

In general, the calibrations should be performed in the above order; however, the weight calibration will be done first and will likely only need to be done again if the TGA is moved or re-leveled.

Before redoing the baseline calibration, restore the default baseline using the Restore function in the **Calibrate** menu. The 7-hour **Baseline Drift Optimization** needs only to be performed once, provided the full calibration was not set to defaults.

The **Temperature** and **Baseline** Calibration may be re-done to optimize the TGA for optimum temperature and weight loss accuracy. For many routine separations this level of calibration accuracy is unnecessary. It is wise to Save and Apply each calibration that is performed under a new and descriptive file name so it can be recalled in the future using the File Open menu item.

Performing a Weight Calibration

This calibration will be default depending on which Y Range you are using. Most people use the Low Range which gives a dynamic range of +/- 160 mg which is appropriate for almost all TGA analyses. This procedure will also work if you choose to calibrate the High Weight Y-Range which must be used if samples heavier than 160mg are to be analyzed. This procedure needs to be modified if you calibrate the Ultrasensitive Y-Range in that you will need to purchase and substitute an accurate 10mg reference standard. This may offer a slight advantage when analyzing very small samples and optimizing the sensitivity for small weight changes.

In order to perform the weight calibration, you will need the 100 mg calibration weight (P/N 09200501) in the Spares Kit (P/N N5320026).

1. Select **Preference**s from the **Tools** menu, then select the **TGA 8000** tab.

General Colors Real-Time Curves TGA 8000	Graph Autosampler	Save Purge Gas
Analyzer Constants Load Temperature: I C Go to Temp Bate: 200 ♀ °C/min Y Data C High Range (+/- 1300 mg) C Low Range (+/- 160 mg) C Ultra Sensitive Range (+/- 20 mg) V Drdinate Filter	─	control :: 30 🔮 (1-60)
	1	1

- 2. In the **Y Data** section select the range you plan to use and click **OK**.
- 3. Select Calibrate Instrument from the Calibrate menu.
- 4. Select the **Weight** tab in the **Calibration** menu.
- **NOTE:** Prior to calibrating the weight the display should look as below with the value in the Measured(mg) box reading 100.000. If it indicates another value you must restore the default weight calibration using the Calibration Restore Weight Calibration menu item

Uperator: Support	This calibration is applied.
5/ Low Bange	/21/2015 1:10:24 AM
Ref. Weight(mg) Measured(mg)	Denis Calibration I
100.000 100.000	

- 5. Type a name in the **Operator** ID field (optional).
- 6. Enter the weight of the calibration weight in the **Ref. Weight** field.
- **NOTE:** Remove any pan from the hangdown wire because once you click Begin Calibration you cannot remove it.

7. Click Begin Calibration.

A dialog box appears with instructions to remove anything in the sample pan. For the autosampler system, remove any sample from crucible "1". The autosampler should be in the Safe position.

8. Follow the instructions in the dialog box, then click **OK**. The analyzer is tared for a zero reading.

For the autosampler system, after accepting the reading, the crucible is unloaded and the furnace is lowered. After approximately 30 seconds, the autosampler rotates the tray so that position 1 is easily accessible.

9. When prompted, place and center the calibration weight in the sample pan and click the **OK** button. The value of the calibration weight is read and displayed in the dialog box. Click **OK** to accept the reading when the weight is stable.

For the autosampler, place the reference weight in crucible 1 and click **OK**. The value of the calibration weight is read and displayed. When the signal settles, click **OK** to accept the reading. The furnace goes to the Cool position and the crucible is returned to the tray.

- 10. The just measured value is automatically displayed in the **Measured** field. Click the **Save & Apply** button to send the new calibration value to the analyzer and save the calibration file.
- 11. Click the **Close** button to exit the Calibration window.

The weight calibration is complete.

Performing the Temperature Calibration

The temperature calibration is performed for a TGA 8000 with or without an autosampler. Perform the procedure below to calibrate the TGA for temperature.

NOTE: Prior to collecting the data to be used for temperature calibration as described below you should open a calibration file which has the temperature calibration parameters set to their default values, or you should go to the Restore menu item in Calibrate and restore the temperature calibration.

For a temperature calibration use the magnetic standards of test metals Alumel, Nickel, Perkalloy, and Iron supplied in the Spares kit (P/N N5320026). For calibrated analysis above 1000 °C substitute cobalt for one of the other standards; for example, check colbalt and uncheck iron. The calibration runs should use the same conditions under which you would run your samples and the scan rates for calibration should ideally bracket the rate(s) for sample analyses.. (However, selecting rates above 50 °C/min for calibration is usually not advisable.) Temperature calibration uses the Curie transition of the materials, that is, the point at which the magnetic properties disappear.

1. Select Preferences from the Tools menu, then select the TGA 8000 tab.

Real-Time Curves TGA 8000	Graph Autosampler	Save Purge Gas
Analyzer Constants Load Temperature: Go to Temp Bate: 200 • °C/min • Y Data • High Range (+/- 1300 mg) • Low Range (+/- 160 mg) • Ultra Sensitive Range (+/- 20 mg) ✓ Ordinate Filter	— 🥅 Remote C Maximal Clients	ontrol ; 30 🔮 (1-60)

- In the Y Data section set the range to Low Range +/-160mg, leave the Ordinate Filter ON, and click OK. Precondition the TGA by running a conditioning method as follows:
 - Heat from 30 C 10 1080 C at 200 c/min.
 - Cool from 1080 C to 30 C at 500 c/min.
- 3. While the conditioning method is running, cut 2-3mm lengths of test metals Alumel, Nickel, Perkalloy and Iron, place them into a ceramic pan, and then cover them with thin coating of alumina powder.
- 4. With a non-autosampler system, attach the pan onto the hangdown wire, raise the furnace, and observe the angle of the pan bale/stirrup.

With an autosampler system, place the ceramic pan with samples into position 1 on the autosampler tray. Click the **Autosampler Control** button in the Pyris software's control panel. Click **Load Sample "1"**. The tray will move and hook the pan onto the hangdown wire. Click the **OK** button to close the dialog box.

- 5. Raise the furnace.
- 6. Create a method for calibration, by selecting **New** from the **File** menu.



This method will collect data for all 4 calibration materials at 5 and 20 C/min, modify the scan rate and ranges as needed. For accurate temperature calibration above 1000C you also need to run the cobalt Curie standard (using a 2mm by 2mm piece of cobalt foil, run alone, not with other Curie standards) and using a similar temperature program as above but heating between 1050 and 1200C.

- 7. Select the **Sample Info** tab and fill in the **Sample ID**, **Operator ID**, **Comment**, and **File Name** as required.
- 8. Tare the weight by clicking the **Zero Weight** button in the **Control Panel**. The analyzer reads the weight of the sample which is entered into the Zero field of the **Enter Sample Weight** box in the Method Editor.
- 9. Open the furnace door and carefully place the magnet (P/N N5370466) around the furnace tube, then raise the furnace.
- 10. Rotate the magnet until the pan rotates from the magnetic field to its pre-magnet angle. Make sure the sample pan is not touching the sidewall of the furnace during this procedure. If it is, swivel the magnet until the pan is centered.

If this is not possible, use smaller pieces of Curie standards.

11. Click the Sample Weight icon on the Control Panel.

This enters the weight into the method. As the sample loses magnetism, the apparent weight reading decreases.



- 12. Click the **Start Method** button in the **Control Panel**.
- 13. After the run, move the sample pan without disturbing the contents to a spot on the autosampler carousel. It will be rerun later.

You are now ready to calculate the **Onset Value** for all four calibration standards.

Calculating the Onset Value

This is done in the **Data Analysis** window.

1. Click the Data Analysis icon on the toolbar, then open the data file in the Data Analysis window.



Make sure that the curve is displayed with the following axes:

X = Temperature (change by selecting Rescale X from the **Display** menu and then select **Temperature** in the dialog box).

- Y = Weight (change by selecting **Weight** from the **Curves** menu).
- 2. In the **Curves** menu select the **Weight** curve.
- 3. In the **Step Select** dialog select step 2 and step 5 through 12. (These two curves contain data for heating the Curie samples at 20 and 5 degrees per minute. (You can delete the original weight data curve.
- 4. For the active curve, select from the **Math** menu, derivative, this will display the derivative curve for the segment you are analyzing.
- 5. With the derivative curve active, **Select Peak Area** from the **Calc** menu.

Calulate the peak temperature at corresponds to each of the weight loses. This point will be used to determine one of the points for each weight loss step.

- 6. Select **Weight Loss curve** so the derivative curve goes to the background.
- 7. Select **Onset** from the **Calc** menu.
- 8. Move the two **red X marks** on the curve. Position the **left limit X** at the inflection point of the first derivative curve and place the **right limit X** past the inflection point on the level part of the curve. Alternatively type in the values using the peak value from the derivative calculation as the steepest slope and a temperature on the trailing baseline.
- 9. Click the **Calculate** button in the **Onset** dialog box. The **Adjust Tangents** box appears.
- 10. Adjust the tangents of each point until they cross and each is tangential at the point selected.

Perform this Onset calculation on each of the eight Curie temperatures (4 at 5 °C/min and 4 at 20 or 25 °C/min.

11. Click the **Calculate** button in the **Adjust Tangents** dialog box. The onset temperature is calculated for the sample and displayed on the screen (Onset X).



- 12. In the Method Editor, select **Calibrate Instrument** from the **Calibrate** menu.
- 13. Click the **Temperature** tab in the **Calibration** window. Enter the name of each reference material, the **Expected Onset** values, the **Measured Onset** values (four temperatures at two rates).
- 14. Check the standards you plan to use and uncheck the others. Always use a minimum of 3.

	Operator:		This calibra	tion is applied.	
			8/10/2015 9:57:	04 AM	
	Ref. Material	Exp. Onset(*C)	Meas. Onset(°C (at Rate 1)) Meas. Onset(°C) (at Rate 2)	Use
1	Alumel	154.2	166.07	169.52	•
2	Nickel	355.3	367.42	372.07	V
3	Perkalloy	596	600.44	603.6	1
1	Iron	770	791.38	794.44	•
5	cobalt	1121	1172.94	1179.4	Г
1	Rate Rate 1: 5 Rate 2: 20	°C/min °C/min			

- 15. Click **Save and Apply** to send the calibration values to the analyzer and save the current calibration file (SN_weightcal_st.tg8c).
- 16. Click **Close** to exit the Calibration window.

The temperature calibration is now complete. For the case where you are calibrating to 1200 C you will be performing the onset calculation as above on the Cobalt analysis, inputting the measured values into the table on the cobalt line, and selecting the cobalt checkbox instead of one of the other standards.

Performing a Balance Drift Optimization

The Balance Drift Optimization takes approximately 7 hours.

- 1. From the Pyris menu, select **Calibrate Instrument** from the **Calibrate** menu.
- 2. Select the TG Drift tab in the **Calibration** menu.

1	5/21/2015 1:10:24 AM
Baseline Optimization	Balance Drift Optimization
Minimum(*C): 30 Maximum(*C): 1000 Rate(*C/min): 20	Perform Balance Optimization(~7 hours
Perform Baseline Optimiza	ation <u>B</u> egin Calibration

- 3. On the **TG Drift** tab <u>uncheck</u> **Perform Baseline Optimization** and check **Perform Balance Optimization (~7 hours)** to perform the tests which compensate the balance for temperature rise in the balance chamber.
- 4. For a TGA 8000 without an autosampler, place a clean empty sample pan in the stirrup at the end of the hangdown wire. Place it there manually with tweezers while the autosampler is in the Safe position.

For an autosampler system, place a pan in Position "1" on the autosampler tray and use the **Autosampler Control** dialog box to load a sample.

5. Click Begin Calibration.

An estimate of the amount of time needed to perform the calibration is displayed and it begins to count down.

- 6. When the calibration is complete, click **Save & Apply** to send the new calibration value to the analyzer and save the file (i.e. SN_weightcal_st_balanceopt.tg8c where SN is the instrument serial number).
- 7. Click **Close** to exit the Calibration window.

Performing a Baseline Drift Optimization

The Baseline Drift optimization varies with scan rate and temperature range. Select a rate and range applicable to the application.

- 1. From the Pyris menu, select **Calibrate Instrument** from the **Calibrate** menu.
- 2. Select the **TG Drift** tab in the **Calibration** menu.

	5/21/2015 1:10:24 AM
Baseline Optimization	Balance Drift Optimization
Minimum(*C): 30 Maximum(*C): 1000 Data(*C): 20	Perform Balance Optimization(~7 hours)
Perform Baseline Optimization	Begin Calibration

- 3. On the **TG Drift tab** <u>uncheck</u> Perform Balance Optimization (~7 hours) and check Perform Baseline Optimization.
- 4. For a TGA 8000 without an autosampler, place a clean empty sample pan in the stirrup at the end of the hangdown wire. Place it there manually with tweezers while the autosampler is in the Safe position.

For an autosampler system, use the **Autosampler Control** dialog box to load a sample.

- 5. Enter the minimum and maximum temperatures between which the baseline will be determined (e.g., set minimum to 50 °C and maximum to 1150 °C).
- 6. Select a scanning **Rate** (°C/min) that you will be using for scanning analyses (e.g., 20 °C/min).
- 7. Click Begin Calibration.

An estimate of the amount of time needed to perform the calibration is displayed and it begins to count down.

- When the calibration is complete, click Save & Apply to send the new calibration value to the analyzer and save the file (SN_weightcal_st_balanceopt_baselineopt20C.tg8c).
- 9. Click **Close** to exit the Calibration window.

The TGA 8000 calibration is now complete. This ends the installation of the TGA 8000.

TGA 8000 Hardware <u>Maintenance</u>

Maintenance Overview

The TGA 8000 needs little routine maintenance other than proper treatment as a sensitive electronic device. Occasionally, the furnace or furnace tube may become coated with sample residue and require cleaning, or furnace components and other accessories, such as hangdown wires and thermocouples, may need to be replaced.

NOTE: The TGA 8000 case is painted metal. The exterior surfaces may be cleaned with a soft cloth, dampened with a mild detergent and water solution.

Maintenance of the TGA 8000 includes the following procedures:

- Cleaning the Furnace
- Cleaning the Furnace Tube
- Cleaning the Sample Pan
- Furnace Maintenance
- Furnace Tube Maintenance
- Furnace Hangdown Wire Maintenance
- Replacing the Thermocouple
- Hangdown Ribbon Replacement

Cleaning the Furnace

An automatic procedure for cleaning the furnace is included in the Pyris software. The procedure involves lowering the furnace to expose it to the air and then heating it to approximately 900 °C to burn off any materials coated onto the furnace surfaces.



WARNING: Since the furnace will be programmed to 900 °C, make certain that the protective furnace door is closed. DO NOT touch the furnace during this procedure.

AVERTISSEMENT: Comme le four sera programmé à 900 ° C, assurez-vous que la porte du four protecteur est fermée. NE PAS toucher le four pendant cette procédure.

1. Start the Pyris software and make sure the **Control Panel** is displayed.

â

2. Select the Clean Furnace icon

from the Control Panel.

3. The furnace will move to the lowered position and programmed to 900 °C. When the cleaning procedure is complete, the furnace will remain in the lowered position and cool down to 30 °C.

NOTE: To abort the furnace cleaning procedure at any time, select the Cool Furnace icon.

Cleaning the Furnace Tube

In order to clean the furnace tube, you must first remove the tube from the furnace. Clean the furnace tube using **one** of the following methods:

- 1. Soak and scrub the furnace tube in a strong detergent (e.g., alconox).
- 2. Soak the furnace tube in a strong solvent such as cleaning solution (e.g., concentrated H_2SO_4 and K_2CrO_7).



WARNING: Concentrated H_2SO_4 and K_2CrO_7 is a very strong, caustic acid solution. Wear protective gloves and safety glasses and perform this procedure in a fume hood.

AVERTISSEMENT: H₂SO₄ concentré et K₂CrO₇ est une solution d'acide caustique très forte. Porter des gants de protection et des lunettes de sécurité et effectuer cette opération dans une hotte aspirante.

3. Dip the furnace tube in a dilute hydrogen fluoride (HF) solution (10% solution, 5 min maximum).



WARNING: Hydrogen fluoride is a dangerous skin irritant. Wear protective gloves and safety glasses and perform this procedure in a fume hood.

AVERTISSEMENT: Le fluorure d'hydrogène est un irritant cutané dangereux. Porter des gants de protection et des lunettes de sécurité et effectuer cette opération dans une hotte aspirante.

- 4. Rotate the furnace tube over a Bunsen burner (heat up slowly to avoid cracking).
- 5. After cleaning, reinstall the furnace tube.

TGA 8000 Furnace Maintenance

Standard furnace maintenance consists of the following procedures:

- Removing the Furnace
- Replacing the Furnace
- Adjusting the Furnace

Removing the Furnace



WARNING: Before performing this procedure, shut down the system and remove the line power. Make sure the furnace has cooled sufficiently so that you do not burn yourself when removing the furnace components.

To perform this procedure the furnace must be in the lowered position.

AVERTISSEMENT: Avant d'effectuer cette procédure, arrêtez le système et retirez l'alimentation secteur. Assurez-vous que le four a suffisamment refroidi pour ne pas vous brûler lors de la dépose des composants de la fournaise.

Pour exécuter cette procédure, le four doit être en position abaissée.



1. Disconnect the furnace power cord from its connector located behind the left panel of the TGA. The furnace cable is screwed into the connector. To disconnect unscrew and gently pull forward on the connector.



- 2. Slide the instrument so the furnace is positioned off the edge of the bench
- 3. Loosen and remove the Clamp holding the Pyrex furnace tube to the base of the furnace assembly.
- 4. Remove the Pyrex furnace tube. You do not have to disconnect the Tygon exhaust tube from the furnace tube. Lay the furnace tube on the bench top.
- 5. Remove the inner Pyrex furnace lining.
- 6. Remove the platinum furnace shield by sliding it off the furnace. Do not deform the shield.
- 7. Remove the plate (slotted ring) sitting on the spring.
- 8. Remove the spring
- 9. Unscrew the large knurled furnace nut to completely remove the furnace from the mount
- 10. Remove the clip that is underneath the nut.



11. Lift the furnace assembly off of the mounting.



If you plan to use the thermocouple from the removed furnace on the new furnace, remove it as described in "Replacing a Thermocouple."

Replacing the Furnace

- 1. Insert thermocouple into furnace base following the thermocouple replacement procedure
- 2. Place the furnace on the furnace base. Note that there is a locating pin on the mounting assembly that fits into the hole on the bottom of the furnace to assist alignment.
- 3. Place the furnace assembly onto the mounting assembly.



- 4. Place the knurled furnace locking nut over the furnace.
- 5. Place the clip underneath the knurled furnace locking nut.



- 6. Straighten the furnace and tighten the knurled nut.
- 7. Slide the platinum furnace shield over the furnace.
- 8. Place the spring over the furnace and until it sits in the top of the metal furnace base. Make sure that the top of the spring is straight.

- 9. Place the slotted plate on top of the spring. Make sure the plate is straight and the slot aligns with the thermocouple.
- 10. Place the quartz insert over the furnace and seat it on the plate.
- 11. Place the Pyrex furnace tube over the furnace so that it rests on the O-ring on the top of the furnace tube base. Position the exhaust tube so that it is left of center when the furnace is out of the cooling position.
- 12. Place the clamp around the base of the furnace tube and top of the furnace tube base. The red handle should be placed so that it is facing the cooling housing. The exhaust tube should be to the right of the clamp. Note the position of the exhaust tube and clamp when the furnace is in the Cooling position in the right-hand photo below.
- 13. Plug in the furnace power cord to its connector on the left side of the TGA. There are male and female guides on the connector that allow the connector to go on in only one way.

After installing the standard furnace, you will have to adjust it with respect to the ball joint, hangdown wire, and sample pan.

Adjusting the Furnace

When your TGA 8000 with standard furnace and **no autosampler** is installed by a service engineer, he performs a series of adjustments in order to optimize its performance. If you need to remove the furnace and/or furnace tube, perhaps to clean it, you will have to perform these steps to ensure proper operation of the instrument. These include:

- Adjusting the Height of the Furnace
- Adjusting the Balance Mechanism
- Adjusting the Convection Iris

Adjusting the Height of the Furnace

The height of the furnace is pre-set at the factory therefore no adjustment should be required. However, if a thermocouple is replaced then the below procedure must be followed:



- 1. The sample pan should still be on the hangdown wire. If not, place a pan on the end of the hangdown wire.
- 2. Click Raise Furnace button on the Pyris Control Panel.
- 3. Check the position of the stirrup with respect to the tip of the thermocouple.
- 4. Adjust the height of the furnace using the furnace adjust height knob located below the furnace adjustment ring. Turn the knob counterclockwise to raise the furnace and clockwise the lower the furnace.
- 5. Raise the furnace using the knurl knob adjustment until the pan touches the tip of the thermocouple, this is done by observing the weight status in the status panel. When you see a weight change...
 - Turn the furnace knob adjustment 3 full turns CCW (this will lower the furnace ~3mm each turn is 1 mm).
 - Then turn the knob 2 full turns CW. The result will be the thermocouple will be about 1 mm below the sample pan.



CAUTION: Make sure that the sample pan hangs in the center of the furnace. It is important that the sample pan does not touch the side wall of the furnace or the tip of the thermocouple.

ATTENTION: Assurez-vous que la casserole d'échantillonnage est suspendue au centre du four. Il est important que le récipient d'échantillon ne touche pas la paroi latérale du four ou la pointe du thermocouple.

The TGA 8000 is now ready to be calibrated.

Adjusting the Convection Iris

- 1. Lift the top cover of the analyzer up to access the balance dome.
- 2. Remove the balance dome by turning the locking devices and pulling the arms out of the groove in the dome. Lift the dome straight up and away from the balance. The iris is now exposed.
- 3. Open the iris all the way by pushing the arm clockwise.
- 4. Loosen the screws on the two stands that hold the iris in place.
- 5. Move the iris around until the hangdown wire is centered in the iris.



- 6. When the hangdown wire is centered, tighten the screws on the stands. Do not overtighten; the iris arm will not be able to move.
- 7. Close the iris as far as possible without it touching the hangdown wire by turning the lever counterclockwise.
- 8. Replace the dome back onto the balance plate and lock in place.
- 9. Lower the access cover.

Furnace Tube Maintenance

Maintenance of the furnace tube consists of the following procedures:

- Removing the Furnace Tube
- Cleaning the Furnace Tube
- Replacing the Furnace Tube

Removing the Furnace Tube

- 1. Make sure that the furnace is in the lowered position.
- 2. Remove the exhaust line from the furnace tube side arm.
- 3. Remove the clamp around the base of the furnace tube.
- 4. Remove the glassware.



CAUTION: Do not pull the furnace tube very quickly or at an angle to avoid breaking the thin stem on the furnace.

ATTENTION: Ne pas tirer le tube du four très rapidement ou à angle pour éviter de briser la tige mince sur le four.

Replacing the Furnace Tube

This procedure is performed after removing the furnace tube.

- 1. Carefully place the tube over the furnace and place on the O-ring at the top of the metal furnace base.
- 2. Position the furnace tube so that the exhaust tube connector is left of center while the furnace is in the lowered position. The exhaust tube connector should be able to go into the cool position without hitting the side of the analyzer.
- 3. Place the clamp around the furnace tube bottom and the top of the furnace base and tighten. Make sure that the red piece is just to the left of the exhaust tube.



4. Replace the exhaust tube to the furnace side arm.

Note the positioning of the clamp and exhaust tube with respect to the autosampler when the furnace is in the lowered position, and with respect to the wall of the cooling area with the furnace in the cool position:

Cleaning the Furnace Tube

In order to clean the furnace tube, you must first remove the tube from the furnace. Clean the furnace tube using **one** of the following methods:

- 1. Soak and scrub the furnace tube in a strong detergent (e.g., alconox).
- 2. Soak the furnace tube in a strong solvent such as cleaning solution (e.g., concentrated H_2SO_4 and K_2CrO_7).



WARNING: Concentrated H_2SO_4 and K_2CrO_7 is a very strong, caustic acid solution. Wear protective gloves and safety glasses and perform this procedure in a fume hood.

AVERTISSEMENT: H_2SO_4 concentré et K_2CrO_7 est une solution d'acide caustique très forte. Porter des gants de protection et des lunettes de sécurité et effectuer cette opération dans une hotte aspirante.

3. Dip the furnace tube in a dilute hydrogen fluoride (HF) solution (10% solution, 5 min maximum).



WARNING: Hydrogen fluoride is a dangerous skin irritant. Wear protective gloves and safety glasses and perform this procedure in a fume hood.

AVERTISSEMENT: Le fluorure d'hydrogène est un irritant cutané dangereux. Porter des gants de protection et des lunettes de sécurité et effectuer cette opération dans une hotte aspirante.

- 4. Rotate the furnace tube over a Bunsen burner (heat up slowly to avoid cracking).
- 5. After cleaning, reinstall the furnace tube.

Replacing a Thermocouple

To replace a thermocouple, follow the procedure below:

1. Disconnect the plastic thermocouple connector plug from the base of the furnace by gently pulling up on the connector plug.





CAUTION: Do not bend the thermocouple severely as this may damage it. If the thermocouple appears old, worn, or broken, replace it (P/N N5321002).

ATTENTION: Ne pliez pas le thermocouple avec précaution car cela pourrait l'endommager. Si le thermocouple est vieux, usé ou cassé, remplacez-le (P/N N5321002).

- 2. Remove the tip of the thermocouple from inside the furnace by pulling the thermocouple straight down toward the bottom of the furnace.
- 3. Using the old thermocouple as a guide, carefully bend the new thermocouple in the way the old thermocouple was bent
- 4. Take the new thermocouple and, while looking inside the furnace, feed the tip of the new thermocouple through the thermocouple slot in the furnace stem until it is approximately 1 or 2 mm below the half-height of the furnace wall.



Helpful Hint: A new thermocouple is shipped in a plastic container with two caps, the cap is 10 mm in height, place this into the furnace and raise or lower the thermocouple such that it is just touching the bottom of the cap. Remove the cap.



5. Plug the plastic thermocouple connector plug on the base of the thermocouple into the base of the furnace. There are two small pins in the furnace base where the thermocouple connects. Be careful not to bend or break the thermocouple pins in the furnace base.



Furnace Hangdown Wire Maintenance

There are two hangdown wires associated with the furnace: a quartz (P/N N5370488) and a nichrome (P/N N5370490) wire. These wires come in kits.

Maintenance of the furnace hangdown wire consists of the following:

- Removing the Furnace Hangdown Wire
- Installing the Furnace Hangdown Wire



WARNING: Before performing the procedures below, make sure that the furnace has cooled sufficiently so that you do not accidentally burn yourself.

AVERTISSEMENT: Avant d'exécuter les procédures ci-dessous, assurez-vous que le four a suffisamment refroidi pour ne pas vous brûler accidentellement.

Removing the Hangdown Wire

1. Lift the round cover straight up to gain access to the balance.

A platinum balance ribbon is connected to the left and right arms of the balance. The ribbon has a loop at its end from which the hangdown wire is hung on the left side of the balance. A tare weight, specific for the particular hangdown wire used, is hung from the ribbon on the right side of the balance to offset the weight of the hangdown wire, stirrup, and sample pan or crucible.



CAUTION: The platinum ribbon is extremely fragile. Care should be taken not to exert force on the loop of the balance ribbon or on the hangdown wire after it is attached. Handle tweezers with extra care when placed in the vicinity of the platinum balance ribbon, the hangdown wire, or the stirrup. Tweezers can catch any of the components and cause damage.

ATTENTION: Le ruban de platine est extrêmement fragile. Il faut veiller à ne pas exercer de force sur la boucle du ruban de balance ou sur le fil de pendaison après qu'il est attaché. Manipulez les pinces avec plus de soin lorsqu'elles sont placées à proximité du ruban de balance en platine, du fil pendulaire ou de l'étrier. Les pinces peuvent attraper n'importe lequel des composants et causer des dommages.

- 2. To remove the hangdown wire from a non-autosampler TGA 8000:
 - i. Make sure that the anti-convection iris in the balance area is open all the way. Place the hangdown wire guide on the end of the convection flange at the end of the joint.
 - ii. Using tweezers, carefully remove the hangdown wire from the ribbon loop. Let go of the hangdown wire.

To remove the hangdown wire from an autosampler system:

- i. Make sure that the anticonvection iris is open all the way.
- ii. Use tweezers to carefully remove the hangdown wire from the platinum ribbon loop. Let the hangdown wire drop onto the wire guide.
- iii. Grasp the hangdown wire with the tweezers while removing the guide.
- iv. Carefully guide the hangdown wire down and out of the tube.

Installing the Hangdown Wire

- 1. Place the hangdown wire guide on the convection flange below the joint.
- 2. Make sure that the convection iris in the balance chamber is open.
- 3. Carefully remove a new hangdown wire from its container. Handle it with one hand.
- 4. From the balance chamber, drop the new hangdown wire through the hangdown wire tube. The end of the wire will rest on either the guide.
- 5. Check the orientation of the hook at the end of the hangdown wire. It should face towards the autosampler. Use tweezers to grasp the hangdown wire near the top. Insert the tip of the hangdown wire through the ribbon loop and release. Go to step 7.
- 6. For a nonautosampler system place an empty pan at the end of the hangdown wire.
- **NOTE:** Make sure that the proper tare weight is in place on the other side of the balance. If you need to change the weight, carefully remove the present one using tweezers. Pick up the new tare weight with tweezers and carefully hang it from the loop of the hangdown ribbon on the right side of the balance mechanism.



CAUTION: The platinum hangdown ribbons are extremely fragile. Be careful not to exert force on the loop of the hangdown ribbon or on the hangdown wire after it is attached. Handle tweezers carefully when placing them near the platinum hangdown ribbons, the hangdown wire, or stirrups as they may catch on one of the components.

ATTENTION: Les rubans de platine sont extrêmement fragiles. Veillez à ne pas exercer de force sur la boucle du ruban pendulaire ou sur le fil pendulaire une fois qu'il est fixé. Manipulez délicatement les pinces en les plaçant près des rubans de pendage en platine, du fil pendulaire ou des étriers, car ils peuvent s'accrocher à l'un des composants.

7. Replace the balance cover over the balance.

After installing a new hangdown wire on either system, you may have to adjust the height of the furnace with respect to the sample pan.

Hangdown Ribbon Replacement

The balance mechanism of the TGA 8000 is designed with replaceable platinum hangdown ribbons. If either the hangdown wire ribbon or the tare weight ribbon breaks, you can replace it using the TGA 8000 Hangdown Ribbon Replacement Kit (P/N N5370478).

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The platinum wire suspension consists of an ultrathin platinum ribbon with a loop on one end. The balance tare weight and the sample hangdown wire (quartz or nichrome) are supported from the loop on their respective ribbons. A small glass sphere is affixed to the other end of the ribbon. This sphere is used to suspend the ribbon from the glass capillary tube which is permanently mounted to the balance beam. The capillary tube is flared at the top to accept the ball on the ribbon. This design provides an inherently stable mount for the ball so that, in a TGA 8000 without an autosampler, the ball does not need to be permanently bonded to the capillary tube to achieve good balance performance. Should the platinum ribbon break, it can be removed easily; a pair of tweezers can easily grip the ball so that the ribbon can be removed.

For an autosampler system, the orientation of the platinum wire loop is important. If affects the position of the hook which is critical in sample pan loading and unloading. The flared tube design facilitates this alignment since the mount is stable. Once the loop is aligned, the ball is bonded to the capillary tube using an easily removable rubber cement.

Parts Included in the Hangdown Ribbon Replacement Kit (P/N N5370478)

Platinum Ribbon Insertion Tool	N5376741
Rubber Cement	09923480
Rubber Cement Application Tool	N5376742
Ribbons	N5370342

Replacing the Platinum Ribbon

- 1. Turn off the power to the analyzer.
- 2. Lift the balance dome straight up.



WARNING: Be careful not to touch components on the thermal controller PC board behind the balance. They may be hot to the touch.

AVERTISSEMENT: Veillez à ne pas toucher les composants de la carte de circuit imprimé du thermostat derrière la balance. Ils peuvent être chauds au toucher.



CAUTION: Be careful not to apply excessive force to any component of the balance assembly. Permanent damage may result.

ATTENTION: Veillez à ne pas exercer de force excessive sur un composant de l'ensemble de la balance. Un dommage permanent peut en résulter.

3. Locate the flared capillary tube mount and the ball support of the broken platinum ribbon.

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 - 4. Apply a few drops of acetone to soften the rubber cement holding the ball to the flared capillary tube.



WARNING: Acetone is extremely flammable. It has a flash point of 0°F. It can cause central nervous system depression. Harmful if swallowed. Causes irritation to eyes, skin, and respiratory tract. Avoid eye and skin contact. Avoid ingestion and inhalation. Refer to the Material Safely Data Sheet for further information.

AVERTISSEMENT: L'acétone est extrêmement inflammable. Il a un point d'éclair de 0°F. Il peut provoquer une dépression du système nerveux central. Nocif en cas d'ingestion. Provoque une irritation des yeux, de la peau et des voies respiratoires. Évitez le contact avec les yeux et la peau. Éviter l'ingestion et l'inhalation. Reportez-vous à la fiche de données de sécurité pour plus d'informations.

- 5. Using of tweezers, grasp the short piece of platinum wire extending above the ball.
- 6. Lift the ball off of the flared mount. If there is resistance, apply more acetone until the ball is free.



CAUTION: Do not apply excessive upward force! This may damage the balance.

ATTENTION: Ne pas appliquer de force excessive vers le haut! Cela peut endommager l'équilibre.

- **NOTE:** If no platinum wire extends above the ball, a small piece of stiff wire (in the kit) may be used to push up on the ball from the bottom end of the capillary tube.
 - 7. Once the ball is free from the capillary tube, clean off any residual rubber cement. Use as many drops of acetone as needed. Acetone that remains trapped in the tube may be "wicked" away with a tissue.
 - 8. Using tweezers, grasp the new replacement ribbon by the glass ball so that the loop hangs downward.
 - 9. Carefully feed the loop end of the ribbon into the flared end of the capillary. Some gentle flexing action on the ribbon may be necessary to get it to feed through the tube. Be careful not to kink the ribbon.
- **NOTE:** If the loop does not feed through the capillary tube properly, the platinum ribbon insertion tool (P/N N5376741) may be used to pull the loop through the tube. Feed the hooked end of the wire through the capillary tube from the bottom and capture the platinum ribbon loop. Carefully pull the platinum ribbon through the capillary tube.
- **NOTE:** This operation requires high dexterity with both hands. It is better accomplished with two people.
 - 10. Suspend the proper tare weight from the loop in the new ribbon; go to step 15.

The following alignment steps are required ONLY when replacing the sample hangdown wire side ribbon. (The tare weight ribbon may be fixed in any direction.)

- 11. Install a hangdown wire and pan.
- 12. Assess the position of the pan relative to the autosampler tray position. Decide in which direction and by how much the ribbon loop needs to be rotated to achieve the correct alignment.
- 13. If some correction needs to be made, use tweezers to grasp the short piece of
platinum ribbon above the glass ball resting in the flared capillary tube.

14. Slightly lift and rotate the ribbon to correct the alignment. This may take a few tries but it is extremely important to obtain the alignment shown below.



15. Carefully inspect (using a magnifier, if necessary) the position of the platinum ribbon within the capillary tube below the glass ball. It must hang straight down without touching the inner wall of the capillary tube. If it touches, the ball must be lifted slightly to allow the ribbon to center itself within the tube.

If you replaced the tare weight ribbon, go to step 17.

- 16. After centering the ribbon in the capillary tube on the sample hangdown wire side, check that the rotational alignment of the sample pan has not been disturbed. If so, repeat steps 14 and 15.
- 17. Locate and open the tube of rubber cement (P/N 09923480). Apply a tiny amount of cement to the end of the wire rubber cement applicator (P/N N5376742). Touch the cement to the area between the glass ball and the flared capillary tube.
- NOTE: Take great care not to disturb the position of the ball.

Repeat this step until you have placed cement around the circumference of the glass ball.

- **NOTE:** Be very careful not to apply too much rubber cement in one area such that it runs down the capillary tube.
 - 18. Allow 10 minutes for the glue to set.

Inspect the work to ensure that adequate cement was applied. You can test the cement by gently trying to remove the ball from the flared capillary tube by pulling on the ribbon above the ball. The ball should not move.

Re-inspect the section of the ribbon within the capillary tube. It should hang freely.

19. Replace the dome on the balance plate assembly, taking care not to touch any components.

Accessories

The following are accessories or optional items for use with the TGA 8000:

Quartz Hangdown Wire Kit	N5320140
NiCr Hangdown Wire Kit	N5320131
Cooling Jacket Accessory	N5370553
Accupik Puncture Device (for an autosampler system)	N5320016
Aluminum crucible liner	N5376732
Kit of 400 aluminum liners	N5370492
Stainless steel crucible liner	N5376749
Kit of 400 stainless steel crucible liners	N5370495
Platinum crucible liner	N5376702
Kit of 400 platinum crucible liners	N5370551
Tare Weights	
Tare weight TGA 8000 194 mg	N5320128
Tare weight TGA 8000 272 mg	N5320129
Tare weight TGA 8000 226 mg	N5320130
Tare weight TGA 8000 375 mg	N5370563

Cooling Jacket Accessory

The TGA Cooling Jacket Accessory allows cooling of the TGA 8000 and Pyris 1 TGA furnace chamber. The cooling jacket is double-walled glassware through which cooling water or antifreeze flows. For subambient operation, this accessory is to be used with a refrigerated coolant circulator. The recommended cooling device from PerkinElmer is the Polyscience Chiller (P/N N5370220/1). Note that a cooler that generates excess vibration or turbulence may result in the poor performance of the TGA.

Parts Included

Part Number	Description	Quantity
02506518	Tygon tubing 0.25-in. i.d.	10 feet
09903004	Hose clamps	4
09908739	Teflon tape	1 roll
09920073	Quick connect stem	2
09940294	Nylon tie wrap	6
N5370333	Glass tube	1

Parts included in the Cooling Jacket Accessory kit (P/N N5370553) are:

Installation

To install the cooling jacket accessory, follow the steps below:

- 1. Switch your TGA on. If it is already switched on, switch it off and then on again after a pause.
- 2. Press the Lower Furnace button on the Pyris software Control Panel to check that the furnace assembly is in the Lowered position.
- 3. If your TGA has an autosampler, make sure it is in the Safe position.
- 4. Remove the Tygon exhaust tube from the furnace tube.
- 5. Remove the quartz insert
- 6. Loosen and remove the clamp holding the Pyrex furnace tube onto the base of the furnace assembly.
- 7. Carefully lift the Pyrex furnace tube up and over the inner furnace sleeve and store the tube in a safe place.
- 8. Place the cooling jacket over the furnace.

The Liquid In and the Liquid Out connectors should be facing toward the left and the exhaust tube should be behind the furnace, as shown below.

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- 9. Replace the clamp around the base of the jacket to secure it to the furnace base.
- 10. Install the quartz insert
- 11. Swing the red handle around and tighten the clamp.

Make sure that the red handle does not touch the lower liquid connector (Cooling Liquid In).

- 12. Attach the exhaust tube to the connector in the back of the cooling jacket.
- 13. The TGA 8000 furnace plugs into the left side after you remove the left side panel.

Connecting to the Chiller



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 doit être efficace jusqu'à -20 ° C. Tout ce qui est inférieur à celui-ci compromettrait la puissance de transport de la pompe du refroidisseur. L'eau distillée est recommandée pour des températures comprises entre 15 ° C et 90 ° C. Pour un fonctionnement à -20 ° C à 100 ° C, 50% d'éthylène glycol dans l'eau est recommandé.



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 minimal de 15-20.

To connect to the chiller, follow the steps below:

- 1. Place the chiller on the floor to the left of the TGA.
- 2. Make sure that the chiller is switched off but that its power cord is plugged into a power supply.
- 3. 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.
- 4. Couplings suitable for several different hose sizes are provided with the chiller.
- 5. Push a hose (compatible with the desired coolant) onto the OUTLET coupling and secure with a hose clamp.
- 6. Connect the hose from the chiller OUTLET to the lower connector on the cooling jacket. Push the tube up onto the connector and over the two ribs.
- 7. Screw the other hose coupling into the INLET connection on the chiller and tighten with a suitable wrench.
- 8. Push a second piece of hose onto the INLET coupling and secure with a hose clamp.
- 9. Connect the hose from the chiller INLET to the upper connector on the cooling jacket. Push the tube up onto the connector and over the two ribs.
- 10. Place a hose clamp (P/N 09903004) around each hose to secure it to the jacket. Position the clamp so that it is between the two ribs and that they do not interfere with the door or the autosampler operation.
- 11. Place the tie wraps (P/N 09940294) around the two coolant hoses in strategic places to help keep them out of the way when the furnace changes position.

Suggested location is around the TGA 8000 foot below the furnace. Do Not tighten the tie-wrap – keep it very loose so as to not kink the tubing. Allow for excess tubing to allow furnace to be raised without stressing tubing. See the example below.

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Setting up the Chiller (refer to Circulator User Manual for more details)

- 1. Fill the reservoir with coolant.
- 2. Ensure that the cooling coils are completely covered. The maximum coolant level is 25 mm below the top of the reservoir.
- 3. 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).
- 4. 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.
- 5. The chiller controller will continue with its start-up sequence and then display "Standby" when ready.
- 6. 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.
- 7. Press the Power switch on the front panel.
- 8. The pump will begin operating.

- 9. Top up the coolant reservoir to compensate for the fluid in the external circuit.
- 10. Rotate the Select/Set control on the front panel until the Pump/AutoTune menu is displayed.
- 11. Press the Select/Set control until the pump speed bar is highlighted.
- 12. 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 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 n'accepte PAS de pression. Utilisez le débit le plus bas qui offre 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 de fluide de refroidissement très constant. Le liquide doit être exempt de bulles d'air. Des modifications du débit de liquide ou de 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.
- 2. The set point temperature value is highlighted.
- 3. Turn the control to set the temperature to the nearest whole degree Celsius.
- 4. Press the control again.
- 5. The degree fraction figures are highlighted.
- 6. Turn the control to select the desired fraction of a degree.
- 7. Press the control to accept the value.

You can now switch on the TGA and begin sample runs.

Accupik Accessory

The TGA 8000 Autosampler Accupik is an accessory designed to help protect the validity of your aqueous or volatile samples. Samples are sealed in volatile sample pans especially designed to fit the TGA 8000 crucibles. The crucibles then are positioned in the autosampler tray for the run. The Accupik is a mechanical device that attaches to the analyzer. From a command in the Pyris software, it pierces the sealed sample pan just before the crucible in which it resides is loaded from the autosampler to the hangdown wire and then enters the furnace. The puncturing of the sample pan provides an additional level of protection against volatilization of samples. Now you can program volatile sample analysis for unattended overnight runs. The Accupik attaches to the underside of the balance plate by inserting the standoff in one hole and using a screw in the other hole to secure it.

Mounting the Accupik

The Accupik attaches to the underside of the balance plate by inserting the standoff in one hole and using a screw in the other hole to secure it.

To attach the Accupik to the balance plate:

- 1. Locate the two holes on the underside of the balance plate.
- 2. Align the round standoff on the Accupik with the rear hole in the balance plate.
- 3. Using the screw provided, secure the Accupik in place with a screwdriver.



Alignment Procedure

NOTE: You must have a crimped sample pan in a crucible in the autosampler in order to align the Accupik properly. Use the Volatile Sample Pan Sealer Accessory (P/N N5370435) to crimp a sample pan (P/N N5370550).



1. When the Accupik is installed it should have at least 5.0mm space between the crucible handle and needle assembly.



2. From the Pyris Software TGA 8000 Control Panel, click on the Autosampler Control



The Autosampler Control dialog box is displayed.

Select Carousel Location: 1	(1-48)
Load Sample Unload S	ample Change Sample
Piercing Mode: OFF	Change
Piercing Position	Piercing Depth
ovement	
Move Tray Upper	Move Tray Lower
Move Tray Upper Rotate Counterclockwise	Move Tray Lower Rotate Clockwise
Move Tray Upper Rotate Counterclockwise Move All Defa	Move Tray Lower Rotate Clockwise

Note that the Piercing Position and Piercing Depth buttons are grayed out (inactive).

3. Click Change to set the Piercing Mode to ON.

The Piercing Position and Piercing Depth buttons become active.

ample Commands	
Select Carousel Location: 1	(1-48)
Load Sample Unload S	ample Change Sample
Piercing Mode: ON	Change
Piercing Position	Piercing Depth
ovement	
Move Tray <u>U</u> pper	Move Tray Lower
Rotate Counterclockwise	Rotate Clockwise
Move All Defau	ult Position
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4. Make sure that a crimped sample pan (use the Accupik volatile sample pan sealer to crimp a volatile sample pan) is in a crucible and that the crucible is in **position 1** in the autosampler tray.

- **NOTE:** The Accupik needs to be adjusted so that when the pan is punctured, the needle is close to the middle of the sample pan in the crucible but slightly to the right of the crucible's handle. There should be approximately 0.5 1 mm of space between the needle guide and the crucible's handle.
 - 5. Enter 1 in **Select Carousel Location**, then click the **Piercing Position** button.

The autosampler will rotate so that position 1 is underneath the Accupik needle.



6. Loosen the black thumbscrew adjustment knob to carefully move the stem so that the needle assembly (contains the needle tip) is slightly above the sample in the crucible.

The space between the needle tip and the top of the sample tray should be approximately 1 mm.



7. Click the **Piercing Depth** button.

This moves the autosampler tray up and down to pierce the sample.

8. Watch the position of the knob as the needle punctures the sample pan.

When the vertical direction or height of the needle is adjusted properly.

9. Check the sample, if it is pierced (there is a hole) and the sample pan is not deformed by the sleeve it is OK. If it is, the Accupik's height needs to be adjusted.

Needle Replacement/Adjustment Procedure

The needle should be replaced if it is old or contaminated. If a different size pan is used, you may need to adjust the needle in the stem in order to maintain the 5-mm space between the needle's tip and the top of the pan.

- 1. Remove the Accupik to replace the needle.
- 2. Using a screwdriver, loosen the screw that is attached to the metal shaft. Pull the old needle out and insert a replacement needle (P/N N5376668).



- 3. Replace and tighten the screw with the screwdriver.
- 4. Replace the Accupik onto the TGA 8000.
- 5. Check the alignment and adjust the Accupik as necessary.

TGA 8000 Autosampler

TGA Autosampler Safety Precautions



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 TGA.

AVERTISSEMENT: Assurez-vous que tous les opérateurs de l'instrument lisent et comprennent les précautions suivantes. Il est conseillé d'afficher une copie de ces précautions sur ou près de la TGA.

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



WARNING: Your TGA Autosampler is either installed at the factory or is designed to be installed by a PerkinElmer Service Engineer if purchased after the TGA. Do not try to install or uninstall the autosampler without the help of a Service Engineer.

AVERTISSEMENT: Votre TGA Autosampler est soit installé en usine, soit est conçu pour être installé par un ingénieur de service PerkinElmer si acheté après le TGA. N'essayez pas d'installer ou de désinstaller l'échantillonneur automatique sans l'aide d'un technicien de maintenance.



WARNING: Do not remove the gripper or covers. No user serviceable parts are inside the autosampler. Refer all servicing to a qualified service engineer.

AVERTISSEMENT: Ne retirez pas la pince ou les couvercles. Aucune pièce réparable par l'utilisateur ne se trouve à l'intérieur de l'échantillonneur automatique. Confiez toutes les réparations à un technicien qualifié.



WARNING: Never operate the TGA autosampler in an explosive atmosphere.

AVERTISSEMENT: Ne jamais utiliser l'auto-échantillonneur TGA dans une atmosphère explosiv.



CAUTION: Make sure that all cables and tubes are clear of the autosampler's path when going to the Load and the Safe positions.

ATTENTION: Assurez-vous que tous les câbles et tubes sont dégagés du trajet de l'échantillonneur automatique lorsque vous passez aux positions de chargement et de sécurité.



CAUTION: Never use the Autosampler Control dialog box to control the autosampler while it is loading or unloading a sample pan to the carousel.

ATTENTION: N'utilisez jamais la boîte de dialogue Contrôle automatique des échantillons pour contrôler l'échantillonneur automatique pendant le chargement ou le déchargement d'une casserole d'échantillons dans le carrousel.

How the TGA 8000 Autosampler Works

The TGA 8000 autosampler is computer controlled and fully automated. The sample tray can hold up to 48 crucibles. Carousel positions are labeled 1 through 48 for easy sample identification. New samples can be added to the carousel to replace samples that have been analyzed by simply pausing the play list and manually controlling the autosampler through the **Autosampler Control** dialog box. Once the autosampler is in the Safe position (all the way to the right), you can lift off the sample ring that holds the crucibles.

You can use the stainless steel sample pan liners (P/N N5376749) in the crucibles to keep them from getting dirty. Simply throw away the liner after use.

Autosampler Work Cycle

When you begin a sample run by starting a play list, the autosampler performs a typical work cycle, which includes the following steps:

- **NOTE**: When running samples with an autosampler, the status of the Antistatic Device should be Auto Enabled and the Upper Fan should be Off.
 - 1. Before the start of the run, the furnace is in the Cooling position or the Lowered position. The autosampler is in the Safe position.
 - 2. 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 autosampler tray turns to position the selected tray location properly.
 - 3. The autosampler swings to the Load position.
 - 4. The tray turns so that the crucible handle engages the hook in the hangdown wire.
 - 5. The antistatic device turns on.
 - 6. The furnace rises almost all the way up. It stays at that position for about 15 seconds while the antistatic device deionizes the joint area.
 - 7. The antistatic device turns off and the furnace finishes traveling up to engage the joint.
 - 8. The method runs.
 - 9. Your play list may contain an explicit Return Sample command. If using a Sample Group, the Sample List entry has an implied Return Sample command.
 - 10. At the end of the run, the antistatic device comes back on as the furnace goes to the Lowered position.
 - 11. The antistatic device turns off as the furnace goes to the Cooling position.
 - 12. The autosampler swings to the Load position with the selected location underneath the crucible.
 - 13. The tray rises so the crucible goes into the selected location.
 - 14. The tray turns so the crucible hook comes off of the hangdown wire.

Sample Handling - TGA 8000 Autosampler

The TGA 8000 measures the change in weight of a sample as a function of temperature or time. Proper sample preparation and handling are important for obtaining optimal results.

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. Proper sample preparation that maximizes the contact surface between the crucible and the sample will reduce any imbalance of the sample in the crucible and will result in maximum peak sharpness and resolution. 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 sample tray holds up to 48 crucibles. You can also place an aluminum liner (P/N N5370492) or a stainless steel liner (P/N N5370495) within the crucible to lengthen its usability. The liner can be used for runs under 600 $^{\circ}$ C.

Sample Atmosphere

Once the sample is loaded onto the hangdown wire and the autosampler returns to the Safe position, the sample is subject to the same atmosphere as a sample in a standard TGA 8000. The furnace rises up around the sample and the sheath gas, if used, baths the furnace liner; that is, the gas goes between the Pyrex furnace tube and the liner, not in the furnace.

Sample Loading

Before loading the samples into the crucibles, 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 ring. You can do this with the ring off of the autosampler assembly. After filling the locations, carefully place the ring over the gripper and settle the ring into position. There is a locating pin in the autosampler that goes into the hole in the tray. You could also keep the ring on the autosampler and use tweezers to load the crucibles into the ring. Make sure that the autosampler is in the Safe position. Be careful with the tweezers around the hangdown wire.

You can tare all of the crucibles in the tray using the Pyris Player Tare All feature. Your play list should 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 a new play list, with Sample Group highlighted, click the **Tare All** button to display the **Advanced Tare Options** dialog box. The tare weights are automatically put into the Sample List. If you chose **Populate from Tray** in the Advanced Tare Options dialog box, the Sample List is filled with a line for each position in the sample ring that contains a crucible and the tare weight of the crucible is included.

Setup Edit Play List View Play List View Sample List View History S	ample History
Player Steps	
 □- 1; Sample Group: □- 1.1: Sample List: □- 1.1: Sample: @ 1, 1.000 mg, -22.804 mg; □- 1.1.2: Sample: @ 2, 1.000 mg, -23.131 mg; □- 1.1.3: Sample: @ 3, 1.000 mg, -23.418 mg; □- 1.1.4: Sample: @ 4, 1.000 mg, -22.810 mg; □- 1.1.5: Sample: @ 5, 1.000 mg, -23.186 mg; □- 1.2: Data Analysis List; 	<u>A</u> dd a step Insert a step Delete this step
Edit Step) 1.1.5: Sample Enter Sample Info Save Da Directory Operator ID: File Name	ata As
	B <u>r</u> owse
Veight 1.000 mg Zero: 23.186 mg	5
Add a sample Insert a sample Delete sample Tare This	Weigh This

Once you have tared the empty crucibles, remove the sample ring, remove each crucible, load the sample into the crucible, and return the crucible to the same location in the sample ring. Return the sample ring to the autosampler. Now you can have the system 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 TGA 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 missing 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:

Setup	Edit Play List	View Play List	View Sample List	View History	Sample History
Player	Player Steps				
⊡ 1:	□ 1: Sample Group: Add a step □ 1.1: Sample List: C:\PE\Pyris352\Methods\Pyrs1std.tg1m Add a step □ 1.1: Sample: @ 1, 50.694 mg, -22.804 mg; Insert a step □ 1.1.2: Sample: @ 2, 14.847 mg, -23.131 mg; Insert a step □ 1.1.3: Sample: @ 3, 13.481 mg, -23.418 mg; Insert a step □ 1.1.4: Sample: @ 4, 31.850 mg, -22.810 mg; Insert a step □ 1.1.5: Sample: @ 5, -0.028 mg, -23.186 mg; Insert a step □ 1.2: Data Analysis List; Delete this step				
Edit Sto Meth Base	Edit Step) 1.1: Sample List Method Name: C:\PE\Pyris352\Methods\Pyrs1std.tg1 Browse Edit Method Baseline File Use Baseline Subtraction				
0	Select <u>P</u> lay List I	iem			~
© Din	Select Existing F ectory:	le	ije Name:		Browse
Add	a sample			Tare <u>L</u> ist	Weigh List

For efficient use of the autosampler and to increase sample throughput, you can purchase a second sample ring. This way you can prepare a second set of samples while the first set is running.

Running a TGA 8000 Autosampler Playlist

Pyris Player is the backbone of Pyris Software automation. It was created with autosamplers in mind. In addition to the standard play list items – Load Sample, Run Method, Return Sample – there is a Sample Group. 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. A Sample Group consists of a Sample List and Data Analysis List. A specific method is selected for the sample 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:

- Review the safety and warning notes for the TGA 8000 and the autosampler.
- Verify that the system purge and sample gas, tubing are properly connected.
- Turn on the gases; adjust the pressures. The recommended flow rate for the system purge is 20 100 ml/min. For the sample gas, it should be between 10 50 ml/min. For the furnace lift mechanism, use a pressure between 15 and 30 psi.
- Verify that the electrical and cable connections between the autosampler and the TGA 8000 and other components of the system are properly connected.

Turn on the power to the system components in the following order:

- Computer
- TGA 8000
- Printer
- 1. Prepare your samples.
- 2. Start Pyris Software and click the **TGA** button.
- 3. Click the **Pyris Player** button on the toolbar: 1. Either open an existing play list or create a new one.
- 4. There are many ways to use the play list with the autosampler. A quick way to create a play list 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.

Setup	Edit Play List	View Play List	View Sample List	View History	Sample History
Player	Steps				
⊡ 1:	Sample Group: 1.1: Sample Li 1.2: Data Ana 	st: lysis List:			Add a step
		Advanced Tare	Options		_ 🗆 ×
			Tare All o	options:	
Edit Ste	ep) 1: Sample Iment:	Tray Op Use This Group Only This Group Only Whole PlayList; S	otions Only One Tray Sample List items O Populate from Tray Sample List items Oni	Use More Than	n One Tray Tare Non-Stop DIAGNOSTIC ONLY
		Tare this Sample Add found items Duplicate locatio	e Group only; to the Sample List; ns get the same tare	value.	
			ОК	Cancel	_
Add	a sample			Tare All	Advanced

6. While taring, the **TGA Tare/Weigh System** screen is displayed.



7. The software will tare the crucibles it finds and populate the **Sample List** in the **Sample Group** for you.

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Setup Edit Play List View Play List View Sample List View History	Sample History
Player Steps	
 □ 1: Sample Group: □ 1.1: Sample List: □ 1.1.1: Sample: @ 1, 1.000 mg, -22,940 mg: □ 1.1.2: Sample: @ 2, 1.000 mg, -22,341 mg: □ 1.2: Data Analysis List: 	<u>Add a step</u> Insert a step Delete this step
Edit Step) 1.1.2: Sample Enter Sample Info Sample ID: Director	ata As
Operator ID: File Name Comment: Image: Comment in the image:	ne: Browse
Sample Details Weight: 1.000 mg Zero: 22.341 mg Weigh at Start of Run Location	: 2
Add a sample Insert a sample Delete sample Tare This	Weigh This

8. Remove the sample tray from the autosampler and place the samples in the crucibles. Carefully place the ring back on to the autosampler. You can also remove each crucible from the tray individually and load the sample into the crucible and return it to the tray.

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 TGA Tare/Weigh System screen appears and the system begins to weigh the samples listed in the Sample List. When complete, the weights are displayed, along with the tare weights, in each sample line.

Setup Edit Play List View Play List View Sample List View Histo	ry Sample History
Player Steps □ 1: Sample Group: □ 1: Sample List: C:\PE\Pyris\Methods\Condition Crucible.TG1M □ 1.1.1: Sample: @ 1, 14.890 mg, -22.925 mg: □ 1.1.2: Sample: @ 2, 50.479 mg, -22.331 mg: □ 1.2: Data Analysis List:	<u>A</u> dd a step Insert a step Delete this step
Edit Step) 1.1: Sample List Method <u>Name: viris\Methods\Condition Crucible.TG1M</u> <u>Browse</u> Baseline File <u>Use Baseline Subtraction</u>	Edit <u>M</u> ethod
© Select Elay List Item © Select Existing File Directory: File Name:	Biowse
Add a sample Tare List.	Weigh List

9. 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.

With **Data Analysis List** highlighted, click **Add a step**. Select **Display Curve**. Use the default **Use Current Run**. This means that the curve from the current run will be displayed after the run is over. Add a Pause line after this line if you want to be able to view the curve.

Setup	Edit Play List	View Play List View	Sample List View History	Sample History
– Player	r Steps			
⊡1: E	□ 1: Sample Group:			
Edit Sto	ep) 1.2.1: Displa Select <u>P</u> lay List	y Curve Item	Pum	V
Di	irectoru:	File Nam	nun	
Γ	nootory.			Browse
-Se	elect Curves to Di Veight	splay	Display Options ☐ Start at Time Zero	Weight %
			Steps: Start at: 1	End at: 2

10. Once you add **Display Curve** to the **Data Analysis List**, other items become available to add to the list.

Player Steps - 1: Sample Group: - 1.1: Sample List: C:\PE\Pyris\Methods\Con - 1.1.1: Sample: @ 1, 14.890 mg22.92 - 1.1.2: Sample: @ 2, 50.479 mg22.33 - 1.2: Data Analysis List:	dition Crucible. TG1M Add a step
L.2.1: Display Curve: Weight: Using Cu Edit Step) 1.2.1: Display Curve O Select Blay List.Item O Select Existing File Use Current Run Directory: File Name: O.YPE\Pyris\Data	to Add in the Player List: Display Curve Math Options Calculation Options Rescale Options Delete Curve Copy to Clipboard Save Data As Save All Print
Select Curves to Display	OK <u>C</u> ancel

- 11. Save the play list by selecting **Save Player** from the File menu.
- 12. Start playback of the play list by clicking on the **Start at Top** button or the **Start at Current Item** button on the Player toolbar.



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

TGA 8000 Autosampler Maintenance

Observe the following guidelines when cleaning the autosampler:

- 1. The black area of the autosampler is anodized aluminum. To clean it, you can use a cloth dampened with isopropyl alcohol.
- 2. The sample tray is clear anodized aluminum which can be cleaned with isopropyl alcohol.
- 3. To keep the ceramic crucibles clean, you can use the disposable aluminum liners (P/N N5370492) or stainless steel liners (P/N N5370495).