

TL 9000 EGA SYSTEM



User's Guide

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Introduction

Introduction

TL 9000 is a Balanced Flow EGA (Evolved Gas Analysis) system capable of capturing an evolving gaseous sample from a thermal analyzer and analyzing it simultaneously using connected FT-IR and GC-MS instruments. It is intended for use with the following PerkinElmer instruments:

- A Frontier, Spectrum 100 series, Spectrum 400 series, or Spectrum One FT-IR spectrometer

WITH

- An STA 6000 (Simultaneous Thermal Analyzer) or TGA 4000 (Thermo Gravimetric Analyzer)

OR

- A Pyris 1 TGA

AND

- A Clarus 500 / 580 / 600 / 680 GC (Gas Chromatograph) connected to a Clarus 500 / 600 MS (Mass Spectrometer).

The TL 9000 system consists of the following:

- Gas flow cell – fitted to your spectrometer, this allows spectra of combustion or pyrolysis products to be obtained from your sample.
- Gas transfer line – fitted to your thermal analyzer, this allows the transfer of gaseous combustion or pyrolysis products from your thermal analyzer to the gas flow cell.
- Gas transfer line – fitted to the GC-MS system, this allows the transfer of gases from the gas flow cell to the GC-MS.
- Gas Sampling Valve (GSV) – installed in the GC detector compartment, this controls the sampling of the gases from the gas flow cell for analysis by the GC-MS system, either by injection on to the GC column for chromatographic separation or direct injection into the MS for scanning of known ions.
- Controller module and vacuum pump – fitted to the gas sampling valve assembly, this allows you to set and maintain the temperature of the gas flow cell and the gas transfer line. It also allows you to set and maintain the rate of gas flow through the entire system path.
- SeaLINK DIO-16 relay box – provides a link between the Pyris and Spectrum TimeBase software applications, allowing the start of acquisition of spectral data by Spectrum TimeBase to be triggered by Pyris. This relay box also synchronizes the start of analysis on the GC-MS using TurboMass software.

About this Guide

This User's Guide is divided into the following chapters:

Safety Information – Safety issues associated with use of the TL 9000. We recommend that all users read this chapter.

Software Installation – The procedures for installing the Spectrum, TimeBase, Pyris, TurboMass and Sealevel relay box software.

Hardware Installation – The procedures for installing the TL 9000 and associated hardware.

Preparing the System for Use – Configuring the hardware and software ready for data collection.

Setting up an Experiment and Collecting Data – Describes, in outline, how you use the system to run an experiment and collect data.

Routine Hardware Maintenance – The procedures for routine maintenance of the TL 9000 hardware.

Further information

For more detailed information on the software used by the TL 9000, access the on-screen help by selecting the **Contents and Index** option from the appropriate software Help menu.

For more information on your spectrometer, consult the user manuals associated with the instrument.

For more information on your thermal instrument, consult the Pyris on-screen help.

For more information on your GC-MS system, consult the user manuals associated with these instruments.

We recommend that you familiarize yourself with the individual instruments and software, before attempt to run complex experiments involving them in combination.

Conventions Used in this Manual

Normal text is used to provide information and instructions.

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

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

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

Notes, cautions and warnings

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

<p>NOTE: A note indicates additional, significant information that is provided with some procedures.</p>

CAUTION

We use the term **CAUTION** to inform you about situations that could result in **serious damage to the instrument** or other equipment. Details about these circumstances are in a box like this one.

D**Caution (Achtung)**

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

DK**Caution (Bemærk)**

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

E**Caution (Advertencia)**

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

F**Caution (Attention)**

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

I**Caution (Attenzione)**

Con il termine **CAUTION (ATTENZIONE)** vengono segnalate situazioni che potrebbero arrecare **gravi danni allo strumento** o ad altra apparecchiatura. Troverete informazioni su tali circostanze in un riquadro come questo.

NL**Caution (Opgelet)**

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

P**Caution (Atenção)**

Significa que a instrução referida tem de ser respeitada para evitar a **danificação do aparelho**.



WARNING

We use the term **WARNING** to inform you about situations that could result in **personal injury** to yourself or other persons. Details about these circumstances are in a box like this one.

D

Warning (Warnung)

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

DK

Warning (Advarsel)

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

E

Warning (Peligro)

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

F

Warning (Danger)

Nous utilisons la formule **WARNING (DANGER)** pour avertir des situations pouvant occasionner des **dommages corporels** à l'utilisateur ou à d'autres personnes. Les détails sur ces circonstances sont données dans un encadré semblable à celui-ci.

I

Warning (Pericolo)

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

NL

Warning (Waarschuwing)

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

P

Warning (Aviso)

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

Safety Information

Overview

This chapter contains details of the safety precautions. Everyone who is going to install and/or use the TL 9000 must read and follow the precautions before unpacking, installing or using the instrument.

Before performing any of the operations outlined in this manual, read all of the safety practices and instructions, and make sure that you understand the procedures explained.

If you do not understand any of the details regarding general safety precautions, please contact PerkinElmer before proceeding.

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<p>NOTE: In addition to the safety information given in this chapter, you should read the safety information for your spectrometer provided in the documentation supplied with the instrument, and for your thermal instrument in the Pyris on-screen help, plus GC- and MS-related safety information reported in their respective documentation or software.</p>

Warning Labels



Caution, hot surface.



Caution, risk of electric shock.



Warning, risk of personal injury.

Precautions



WARNING

Ensure that all instrument operators read and understand the precautions listed below. It is advisable to post a copy of the precautions near or on the instrument shelf.

The following precautions must be observed when using the TL 9000:

- Be sure that the voltage of the TL 9000 corresponds to the voltage used in your laboratory.
- Do not remove any of the panels from the controller module without first shutting down the instrument and disconnecting the power cord from the mains supply.
- Do not immerse the purge gas exit line in a liquid, as the liquid may be drawn back into the sample holder.
- Only high quality purge gases should be used with the TL 9000. Minimum purity of 99.995% is recommended. A high quality filter-dryer accessory is recommended for the removal of any moisture from the purge gases.
- It is very important that nothing falls into the gas flow cell or the controller module or the gas sampling valve (GSV) assembly. If this occurs, *IMMEDIATELY* switch off the power and contact your PerkinElmer Service Representative.
- The condition of all pipes, hoses and connectors should be checked regularly. Perform gas leak tests at all joints and seals of the gas system regularly, using an approved gas leak detection system.
- When the equipment is turned off, close all gas cylinder valves tightly at the cylinder. Bleed the remainder of the line before turning the exhaust vent off.



WARNING

To ensure safe operation, the exhaust tubing from the pump must be evacuated in a fume hood or disposed of properly outside the working environment.

Never cut, reduce or blank the exhaust line.



WARNING

NEVER USE FLAMMABLE GASES OR OXYGEN WITH THE TL 9000.

SERIOUS RISK OF PERSONAL INJURY OR DAMAGE CAN OCCUR.



WARNING

All heated parts, the gas flow cell housing, the gas transfer line and the GSV assembly, can become very hot, depending on the operating temperatures selected.

The outer surface of the gas transfer line, for example, can reach temperatures of approximately 100 °C when heated to 350 °C.

ALWAYS USE PROTECTIVE GLOVES WHEN HANDLING HEATED PARTS.

General Operating Conditions

The TL 9000 has been designed and tested in accordance with PerkinElmer specifications and in accordance with the safety requirements of the International Electro technical Commission (IEC). The TL 9000 conforms to IEC 61010-1 (Safety Requirements for electrical equipment for measurement, control and laboratory use) as it applies to IEC Class 1 (earthed) appliances.

The TL 9000 complies with the following EC directives:

- 2004/108/EC (electromagnetic compatibility)
- 2006/95/EC (electrical low voltage safety).

Only use the TL 9000 under the following environmental conditions:

Ambient working temperature 15 °C to 35 °C

Relative humidity 75% maximum (non-condensing)

Avoid any adjustment, maintenance or repair of the opened, operating instrument. If any adjustment, maintenance or repair of the opened instrument is necessary, this must be done by a skilled person who is aware of the hazard involved.

Whenever it is likely that the TL 9000 is unsafe, make it inoperative. The TL 9000 may be unsafe if it:

- Shows visible damage
- Fails to perform the intended measurement
- Has been subjected to prolonged storage in unfavorable conditions
- Has been subjected to severe transport stresses.

Environmental Conditions

The TL 9000 has been designed to be safe under the following conditions:

- Indoor use
- Altitude up to 2000 m
- Ambient temperatures of 5 °C to 40 °C
- A maximum ambient relative humidity of 80% for temperatures up to 31 °C, decreasing linearly to 50% relative humidity at 40 °C
- Mains fluctuations not exceeding $\pm 10\%$ of the nominal voltage.



WARNING

If the equipment is used in a manner not specified herein, the protection provided by the equipment may be impaired.

Electrical Safety

The TL 9000 controller module and pump come assembled within protective housings to prevent exposure to electrical hazards.



Removal of the protective housing or rear panel from the controller module is dangerous.

If the protective covers or service panels on any instrument appear to be damaged, turn the instrument off, secure it against any unauthorized operation and contact PerkinElmer for advice before using the instrument again.

Electrical protection

Installation Category: The instruments are able to withstand transient overvoltages typically present on the MAINS supply. The normal level of transient overvoltages is impulse withstand (overvoltage) category II of IEC 60364-4-443.

Pollution Degree 2: Normally only non-conductive POLLUTION occurs. Occasionally, however, a temporary conductivity caused by condensation must be expected.

Power supplies

The TL 9000 controller module and pump require an input voltage of *either 115 or 230 VAC*, at a frequency of 50–60 Hz. This power supply must not fluctuate by more than $\pm 10\%$ of the nominal voltage.

The power consumption is 2.5 A at 230 V (600 VA max) or 5.0 A at 115 V (600 VA max).



WARNING

Check the line voltage and frequency of the power point (socket) before the instrument is connected or the power is switched on. This should be carried out prior to the installation of the instrument and at any time the instrument is plugged into a different power point (socket).

The supply voltage is fixed at the factory, and cannot be changed.

Ensure that there is sufficient space between any cooling vents or fans and adjacent equipment or walls.

Power cords

The power cord supplied to connect the controller module to the power point (socket) should be appropriate for the country listed as the shipping destination.



WARNING

If the power cord is not appropriate for your country it must be removed from use immediately and replaced.

If the power cord has any physical damage it must be removed from use immediately and replaced.

To avoid electrical hazards, the ground pin on the power cord should not be interfered with in any way.

Ensure that there is a sufficient gap at the back of the controller module to allow the power cord to be removed safely, if required.

Fuses

**Electrical hazard**

To prevent potential injury to yourself and damage to the instrument, switch OFF all components in the system and disconnect them from the line power supply before you replace any of the fuses.

The rear of the controller module houses two removable fuses (5 × 20 mm) rated at 6.3 AT (slow-blow) for 230 V operation, and 10 AT (slow-blow) for a 115 V power supply.

The fuses can be checked or replaced with the use of a suitable flat-bladed screwdriver, by pushing and turning the fuse caps counter-clockwise.

Always replace fuses with the same type and rating.

CAUTION

The pump module operates at 24 V DC with a pump protection fuse inside the controller module that is rated as 1 AF (fast-blow fuse). If the pump is switched off during normal operation, then you must wait until the pressure or vacuum is released before restarting it again, otherwise the protection fuse will blow after a few seconds.

Cylinder Gas

You should provide adequate space for the supply of cylinder gas. The gas should be stored in a safe manner and be available for connection to the instrument when required.

Refer to safety instructions in the appropriate user's guide for your GC-MS system regarding gas cylinders, gas types and purity.

Specifications

Gas flow cell operating temperature	50–350 °C
Gas transfer lines operating temperature	50–350 °C
Gas sampling valves (GSV) assembly	50–350 °C
Minimum temperature steps	1 °C
Minimum settable temperature	50 °C
Mass flow controller range	From 10 to 200 ml/min
Pump type	Membrane and flap valves
Pump noise level	Maximum 55 dBA
Free pump capacity	Maximum 10 nl/min; vacuum 100 mbar absolute
Coarse dumper filter	Synthetic fiber
Cylinder fine filter	Synthetic fiber and optional carbon trap
Controller dimensions L × H × D	28 × 25.5 × 53 cm (D without rear plugs)
Controller weight	20.9 kg
Pump dimensions L × H × D	12.5 × 13.5 × 21 cm (D without front plug)
Pump weight	1.8 kg
Weight of cell assembly + transfer lines + GSV assembly and adapter	12.9 kg
Supply voltage	230 or 115 VAC
Supply voltage frequency	50/60 Hz
Maximum power consumption	2.5 A at 230 V, 600 VA max 5.0 A at 115 V, 600 VA max
Ambient working temperature	From +15 °C to +40 °C
Relative humidity	80% maximum without condensation
Protection degree IP (IEC 60529)	IP20



Software Installation

Software Applications

The TL 9000 requires the following PerkinElmer software applications:

Spectrum – Spectrum is the main PerkinElmer software package for collecting, viewing and processing IR spectra. It can be used to set up and collect data from a range of PerkinElmer spectrometers.

Spectrum TimeBase – Spectrum TimeBase enables you to collect many spectra of a sample, either continuously or at regular time intervals. This enables you to track the progress of a sample that is changing with time, for example, because of a chemical reaction that is occurring in the sample, or because the sample is being heated.

Pyris – Pyris software is designed to control PerkinElmer's Thermal Analysis instruments, and manage the data they produce.

TurboMass – TurboMass software is designed to control Perkin Elmer's GC and MS systems and manage the acquired data. This software must be installed in a separate computer to maximize acquisition performance and to avoid interactions with other software applications.

In addition to these software applications, software drivers for the SeaLINK relay box must be installed on the computer running Pyris; see *Installing the SeaLINK relay box driver software* on page 28 for details. The SeaLINK relay box is used to send start and stop software triggers from Pyris to Spectrum TimeBase and TurboMass.

Installing the Software

Before setting up the TL 9000 hardware, you should set up your computers and install the necessary software.

Minimum computer requirements

If you are supplying your own computer, make sure that it meets the minimum requirements for both Spectrum and Pyris, as set out in the *PC Requirements* section of the *Spectrum Administrator's Guide* (P/N L1050095 for software version 10.0 or later) and in the *Pyris Installation Guide* (P/N 09936940). The computer for the GC-MS instrument must also meet the minimum requirements of the TurboMass software as described in the *Clarus 560/600 GC-MS Tutorial* (P/N 09936769).

Installing Spectrum, Spectrum TimeBase, Pyris, TurboMass

Details of the installation procedures for Spectrum, Spectrum TimeBase, Pyris and TurboMass are given in the following guides:

- *Spectrum Administrator's Guide* (P/N L1050095 for software version 10.0 or later)
- *Spectrum ES Administrator's Guide* (P/N L1050100 for software version 10.3.3 or later)
- *Spectrum TimeBase Installation Leaflet* (P/N L1050040)
- *Pyris Installation Guide* (P/N 09936940)
- *Pyris Enhanced Security User's Guide* (P/N 09936942 for software version 10 or later)
- *TurboMass Software User's Guide* (P/N 09931016).

NOTE: Spectrum, or Spectrum ES, and Spectrum TimeBase (IR software) must be installed on the same computer, with the Spectrum software installed BEFORE Spectrum TimeBase.

If you are using standard Pyris or Pyris ES (thermal software), it can either be installed on the same computer as Spectrum and Spectrum TimeBase, or on a different computer. The computer must use a 32-bit operating system to enable the software drivers for the SeaLINK relay box to operate correctly.

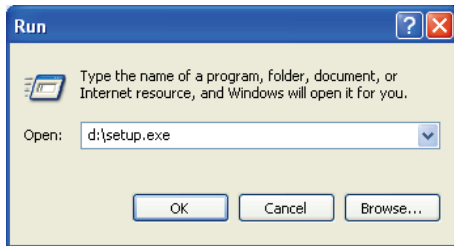
TurboMass (GC-MS software) must be installed on a separate approved computer.

Installing the SeaLINK relay box driver software

To install the relay box driver software, follow the steps described below.

NOTE: If you have installed your IR software and thermal software on different computers, the SeaLINK relay box driver software must be installed on the computer running Pyris.

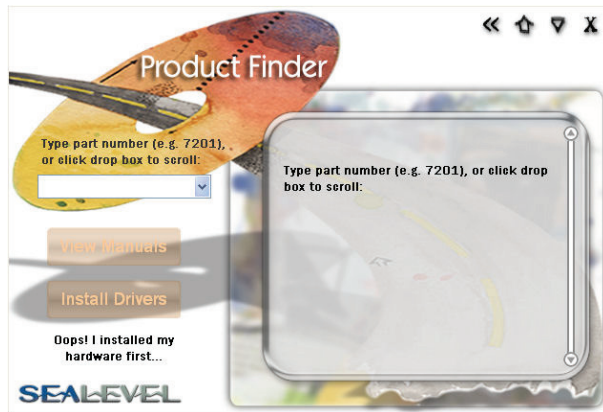
1. Place your *Sealevel Software CD* (P/N N5378150) into your CD drive.
2. If the installation program does not start automatically, from the Start Menu select **Run**.
The Run dialog is displayed.



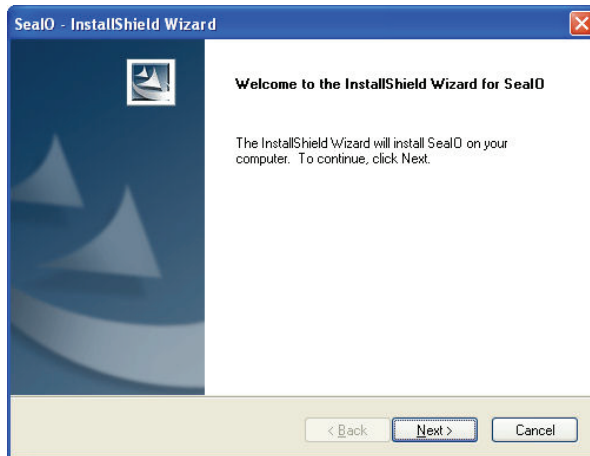
3. Enter **d:\Setup.exe** and then click **OK**.
Replace **d:** with the drive letter for your CD.
The following screen is displayed.



4. Select the **Install** option.
You are then prompted to enter a part number.

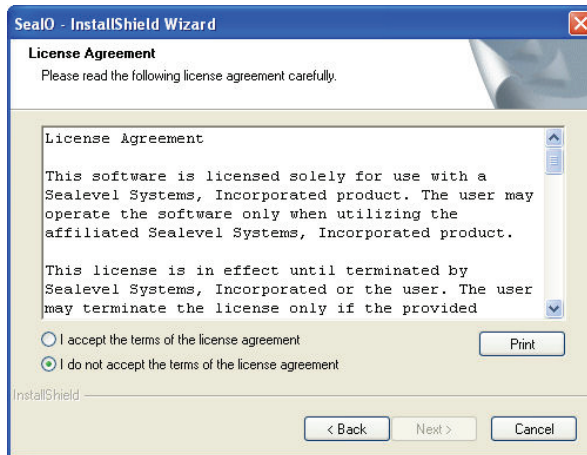


5. Type in the part number for your relay box.
You can find this on the base of the box itself.
6. Once you have entered the part number, click on **Install Drivers**.
The InstallShield Wizard dialog appears while the system is preparing to install the software. When the installer is ready, the Welcome dialog is displayed.



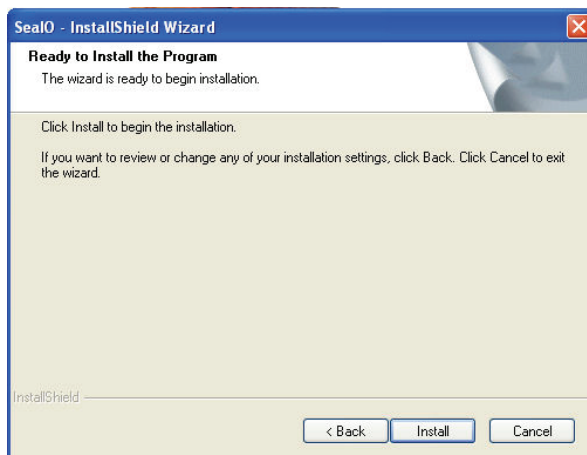
7. Click **Next**.

The License Agreement is displayed.

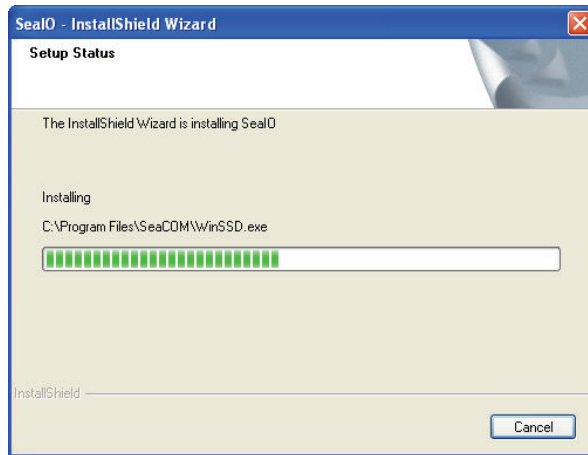


8. Read the license agreement and, if you accept the terms, select that option and then click **Next**.

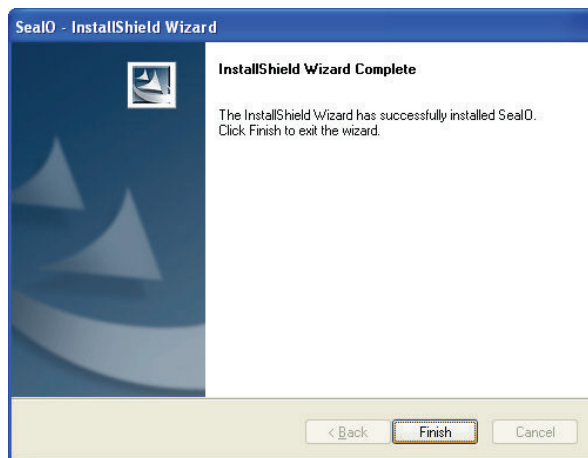
The InstallShield Wizard confirms that it is ready to start the installation.



9. Click **Install** to begin installing the relay box driver software.
The wizard keeps you informed of the progress of the installation.

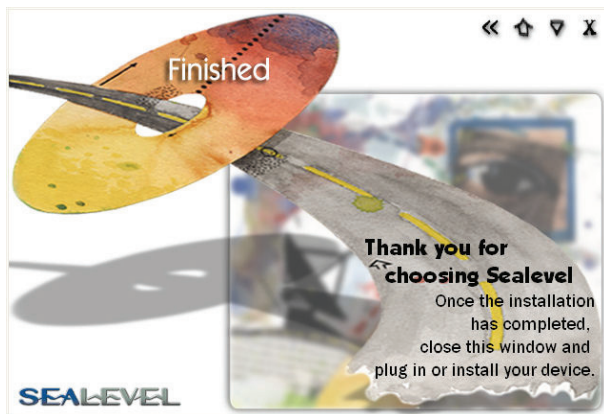


When the installation is complete, the InstallShield Wizard Complete page is displayed.



10. Click **Finish**.

A final message is displayed.



11. Close the Sealevel window.

NOTE: If you find that the SeaLINK relay box fails to accept trigger signals from Pyris, consult a PerkinElmer Service Representative as, depending upon the version of Pyris you are running, additional software drivers may be required.

Hardware Installation

Overview of TL 9000 and Associated Hardware

The TL 9000 hardware contains the following major components:

- Controller module (part number depends on mains power supply voltage).
- Gas flow cell to fit inside the FT-IR spectrometer sample compartment (P/N L8200221).
- Heated transfer line between the FT-IR spectrometer and the GC-MS (supplied with the gas flow cell).
- Heated transfer line between the FT-IR spectrometer and the thermal instrument, and configured for use with the STA 6000, TGA 4000 or Pyris 1 TGA as appropriate (supplied with the gas flow cell).
- Heated gas sampling valve assembly (P/N L8200222) with loop load and return PTFE tubes attached.
- SealINK DIO-16 relay box (P/N 09401085).

The following items are also included in the installation kit:

Part Number	Description
L8201801	Gas flow cell KBr windows (pack of 2)
L8201820	Gas flow cell window spanner
N9301355	Deactivated column, ID 0.1 mm
N9301356	Deactivated column, ID 0.25 mm
L8201941	Heated extension to valve assembly
L8201961	GC detector compartment adapter plate
L8200206	Vacuum pump
L8201809	Polystyrene test sample for gas flow cell
L8201807	Transfer line adapter kit for STA 6000 / TGA 4000
OR	OR
L8201882	Transfer line adapter kit for Pyris 1 TGA

Positioning of the instruments on the bench

The instruments should be positioned on a sturdy bench, at least 2.5–3 meters in length (with additional space for the two controlling computers as appropriate). Locate the GC-MS on the far left, and then position the FT-IR, the TL 9000 controller and finally the thermal analyzer in order to the right, as shown in Figure 1. Allow sufficient space between instruments to ensure adequate ventilation (at least 7–10 cm). Vacuum pumps must be placed on the floor. Maintain a space between the rear of the instruments and the wall to allow access for maintenance purposes.

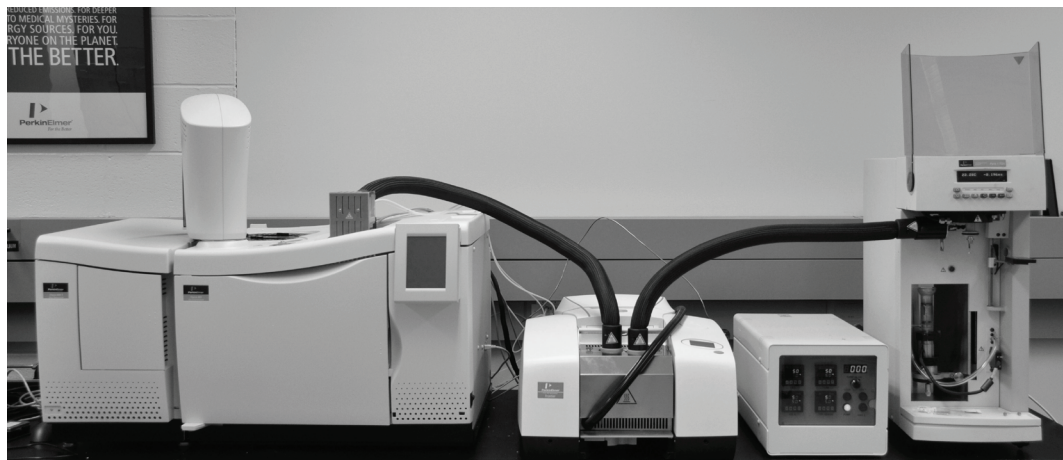


Figure 1 Instruments positioned on a bench

Installation of Analytical Instruments

Details of the installation procedures for your spectrometer are provided in the documentation supplied with the instrument.

Installation and setup instructions for your thermal instrument can be found in the Pyris on-screen help.

Details of the installation procedures for your mass spectrometer or gas chromatograph are provided in the documentation supplied with the instrument. Refer to the *Clarus 560/600 MS Hardware Guide* (P/N 09936768) and the *Clarus 600/680 GC Customer Hardware and Service Guide* (P/N 09936781), or the corresponding documents for an alternative GC-MS system.

TL 9000 Gas Flow Cell Installation

The gas flow cell (P/N L8200221) is designed as an accessory to fit in a PerkinElmer FT-IR spectrometer, as shown in Figure 2.



Figure 2 TL 9000 gas flow cell installed in a Frontier FT-IR spectrometer

To install the gas flow cell, follow the steps described below.

1. Remove the base plate from your spectrometer.

NOTE: The gas flow cell is supplied already fitted to a spectrometer base plate.

2. Carefully extract the cell assembly from the transport package and place it on a free work bench.
Pay attention to the longer transfer line connected to the heavy GSV box, which should be laid in a safe place.
3. Release the two connectors that secure the front cover of the gas flow cell, by giving them a quarter-turn in either direction.
One of the connectors is shown in Figure 3.

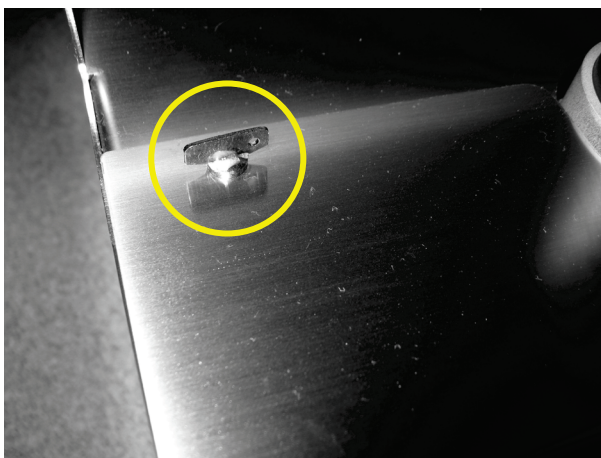


Figure 3 Gas flow cell cover connector (circled)

4. Carefully remove the front cell cover to reveal the internal cell assembly (Figure 4).

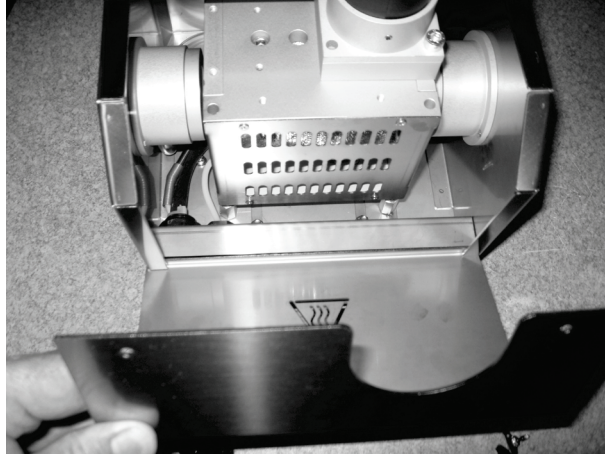


Figure 4 Removing the gas flow cell cover

5. Use a 5/16" (8 mm) socket wrench to loosen both of the nuts that secure the cell assembly to the chassis base (Figure 5) and then carefully remove both the nuts and the washers using a pair of tweezers.



Figure 5 Loosening one of the chassis nuts

6. Slide the cell holder towards you, and then carefully tilt and lift the cell assembly clear of its box (Figure 6).

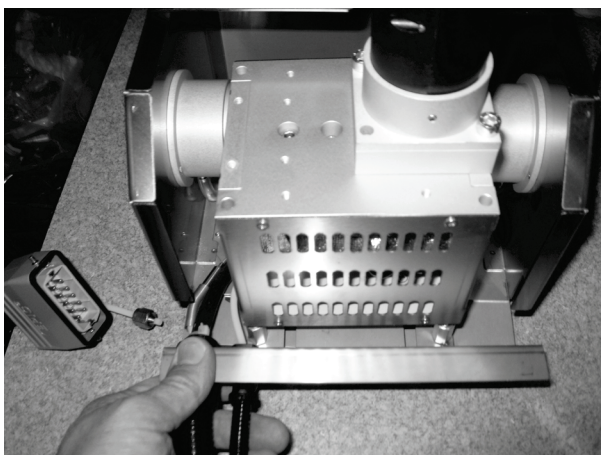


Figure 6 Removing the cell assembly from its box

7. Gently place the cell assembly on its side.
To avoid damage during transportation, including exposure to temperature variations and humidity that may affect their transparency to IR, the internal cell windows are not factory-installed and must be fitted as part of this hardware installation process before the flow cell is fitted to your spectrometer. KBr windows are supplied in packs of two (P/N L8201801) with the TL 9000. ZnSe windows (which provide other usable wavelength ranges) are also available but are provided separately (P/N L1270766).
8. Using the spanner provided (P/N L8201820), loosen and then remove the window cap (Figures 7–8).

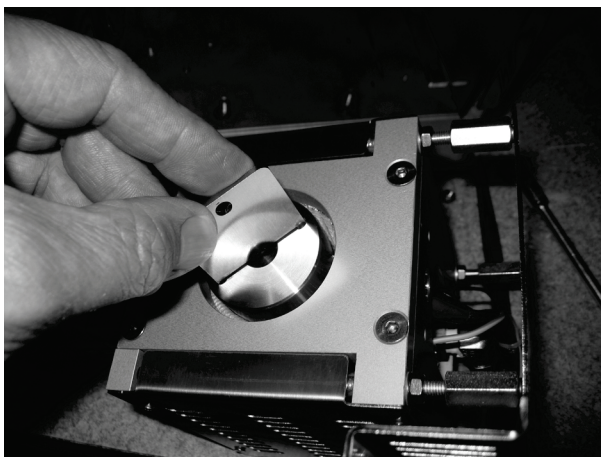


Figure 7 Loosening a window cap

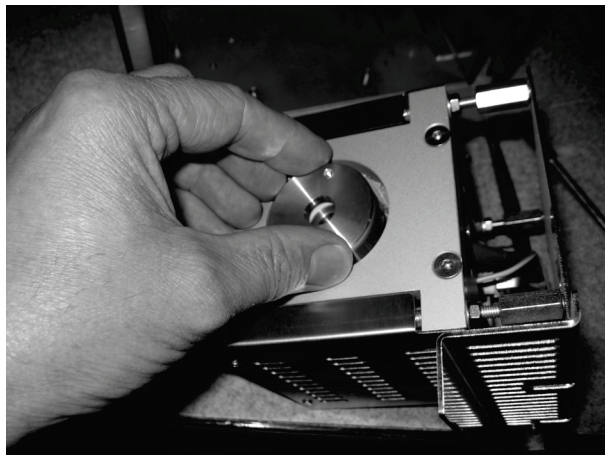


Figure 8 Removing a window cap

9. Insert the cell window (Figure 9).



Figure 9 Inserting a cell window with protective plastic cover

CAUTION

You are strongly advised to wear protective gloves when handling a KBr or ZnSe cell window, or leave the plastic protective cover on the window until you have fitted it into the cell (Figure 9).

ZnSe is toxic. KBr is hygroscopic; serious damage will be caused if you touch the surface of a window.

For long-term storage, we recommend that KBr windows are kept in a desiccator.

10. If appropriate, remove the plastic protective cover from the window (Figure 10). Ensure that the window is centered in the holder.

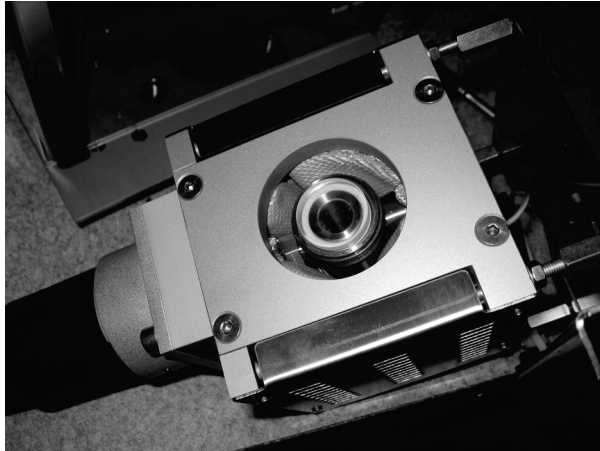


Figure 10 Fully installed cell window

11. Replace the window cap and secure it with the spanner provided.

CAUTION

Avoid over-tightening the window cap, as this can damage the cell window.

12. Turn the cell assembly over and then repeat steps 8 to 11 to insert the opposite cell window.
13. Once you have installed both cell windows, place the cell assembly back in its box.
14. Using a pair of tweezers, carefully replace the fixing washers and nuts (Figure 11).



Figure 11 Reassembling the gas flow cell

15. Replace the cell cover.

16. Finally, insert the cell assembly into the spectrometer IR sample compartment.

NOTE: You will need to manually align the cell to maximize the IR energy reaching the detector before you can use it. This will involve removing the cover and loosening the securing nuts. See *FT-IR spectrometer setup* on page 78 for details.

For many measurements it is useful to purge the sample area to avoid absorbance by atmospheric water vapor or carbon dioxide.

To aid this, the gas cell housing incorporates purged telescopic tubes enclosing the beam. These should be extended so that they touch the gas cell at both sides (Figure 12).



Figure 12 Gas flow cell detail

Gas Switching Valve Assembly Installation

The FT-IR spectrometer should be positioned on the right of the GC-MS so that the left transfer line (120 cm in length) from the gas flow cell will reach the GC and accommodate the gas switching valve (GSV) assembly in the detector compartment. The GC detector compartment should ideally be free from detectors; however, it is possible to install the GSV assembly in the rear detector position and install a detector in the front position with the use of an adapter plate (P/N L8201961) to gain space in the detector compartment. We recommend that you contact your PerkinElmer Service Representative for assistance if you want to install the adapter plate in your GC.

The figures in this manual show the installation of the GSV assembly at the front detector position.

NOTE: These instructions for the installation of the TL 9000 hardware refer to the Clarus 600 GC-MS, but the procedure is very similar for other Clarus GC-MS systems.

1. Switch off the main power supply to the GC and unplug the power cord.
2. Open the top cover of the detector compartment, push the two hinges outwards and remove the cover (Figure 13).



Figure 13 Removing the GC detector compartment cover

3. Using a flat screwdriver, remove the two screws securing the GC top cover. See Figures 14–15.



Figure 14 Removing the center top cover screw



Figure 15 Removing the right top cover screw

4. Lift up the top cover and support it using the arm provided.
5. Remove the outer cover fixing screws from the GSV assembly, and lift off the outer and inner covers to reveal the valve connections.

6. Pass the transfer line through the gap in the GC top cover created by removing the compartment cover, and locate the GSV assembly in the appropriate detector position (Figure 16).

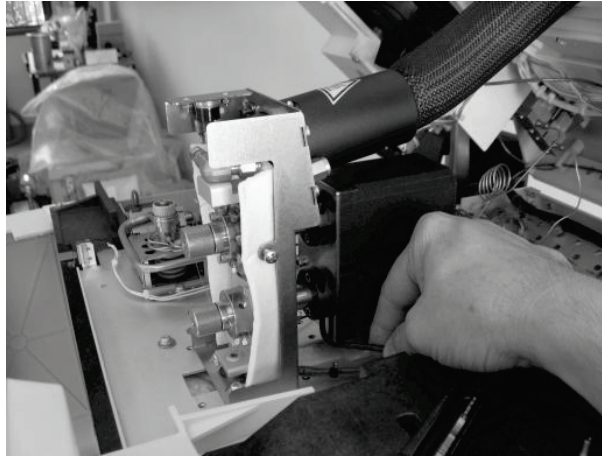


Figure 16 Positioning the GSV assembly on the GC oven

7. Using a screwdriver, pierce the insulation at the top of the oven to form a hole with an approximate diameter of 12 mm, aligned with the threaded fixing holes (Figure 17).

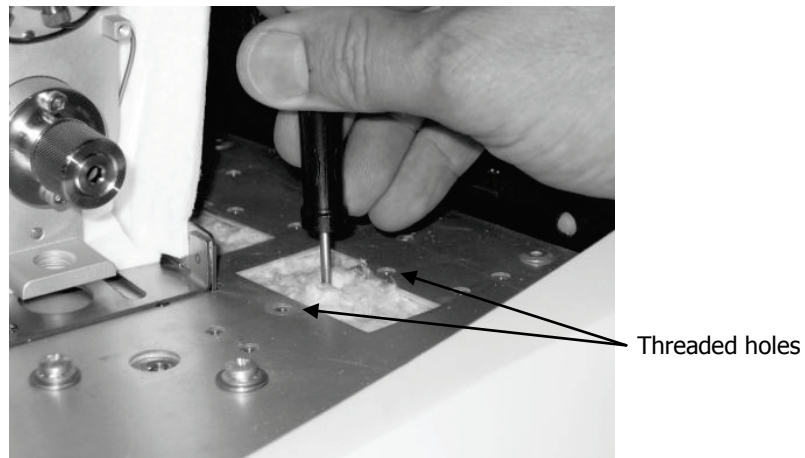


Figure 17 Piercing a hole in the oven insulation

8. Screw the heated extension tube (P/N L8201941) into the bottom of the GSV heated block and tighten it by hand (Figure 18).

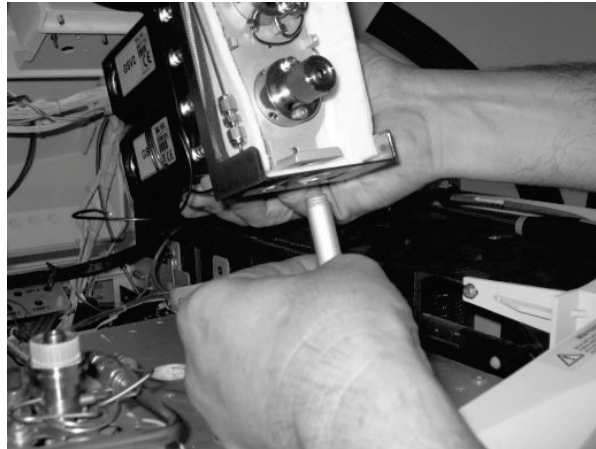


Figure 18 Installing the heated extension tube

9. Insert the tube into the hole in the oven insulation, align the GSV assembly using the fixing screws, and tighten using a screwdriver (Figures 19–20).

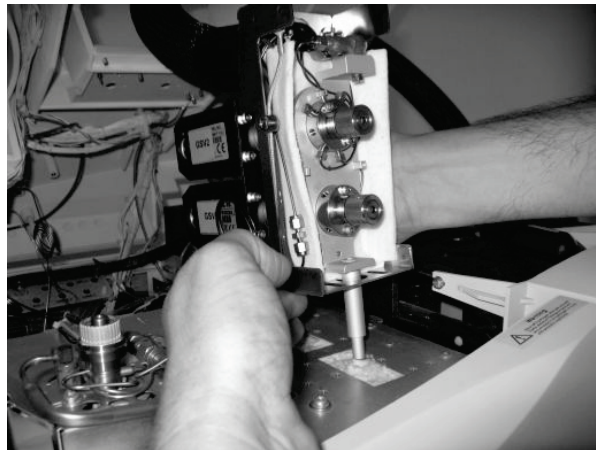


Figure 19 Inserting the GSV assembly

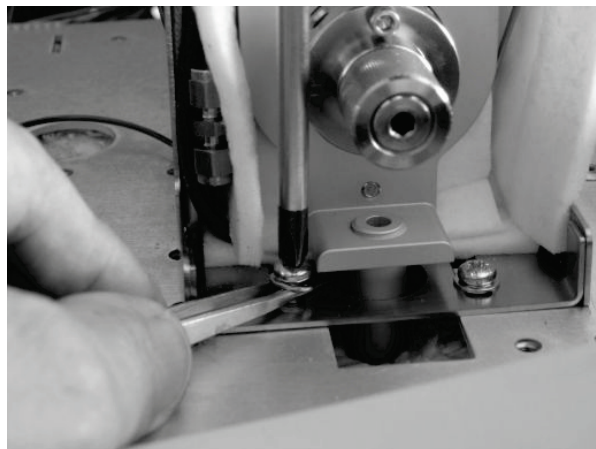


Figure 20 Tightening the GSV base fixing screws

10. Unroll the 1/16" carrier gas tubing on the left-hand side of the GSV assembly and use it to pierce the oven insulation through the nearest access hole for the injector port (Figure 21).

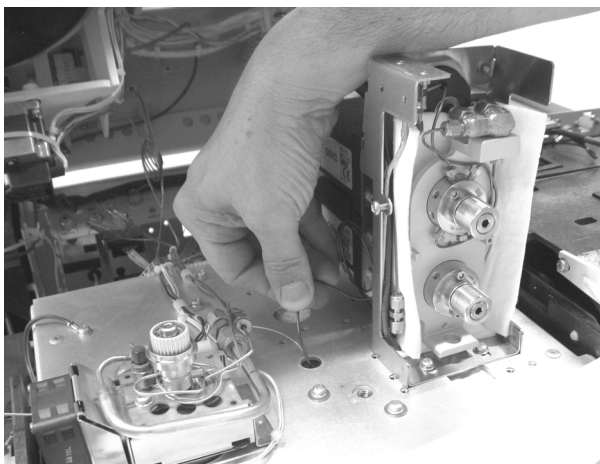


Figure 21 Piercing the oven insulation using the carrier gas tubing

11. Feed the carrier gas tubing into the GC oven.
12. Connect the carrier gas tubing to the outlet of the injector port inside the oven using the nut (P/N 09903392) and ferrule (P/N 09920127) provided with the GSV assembly (Figure 22).

OR

Connect the carrier gas tubing directly to an auxiliary Programmable Pneumatic Controller module or, less desirably, a manual pressure regulator.

NOTE: Connecting directly to the injector will only allow analysis of the gaseous samples from the thermal instrument. Using a Programmable Pneumatic Controller allows you to configure the system to continue injecting liquid samples. Refer to the *Clarus 600/680 GC Customer Hardware and Service Guide* (P/N 09936781) or your PerkinElmer Service Representative for additional information.

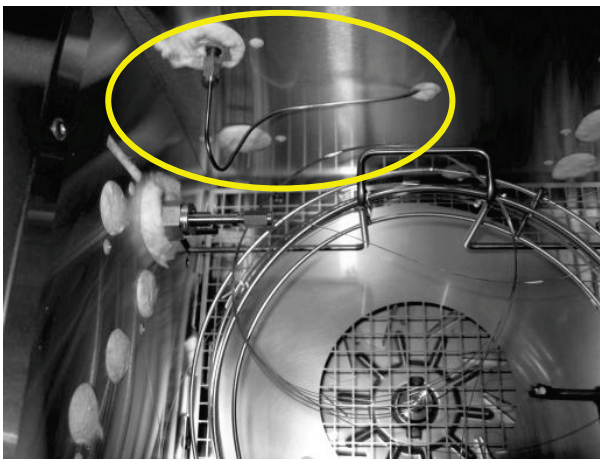


Figure 22 Connecting the carrier gas tubing to the injector outlet (circled)

13. Use a ceramic cutter to remove a short length from the end of each capillary transfer line.
To obtain a clean cut, use the cutter to score the tubing and then snap off the cut length by hand. Refer to the *Clarus 600/680 GC Customer Hardware and Service Guide* (P/N 09936781) for additional information on how to cut capillary columns.
14. Thread a stainless steel nut (P/N L8201956) and 0.25 graphite-polymer ferrule (P/N L8201957) onto the end of each transfer line, as shown in Figure 23.
These parts are provided with the GSV assembly.

NOTE: The use of other nuts or ferrules may result in a greater amount of air penetrating the GC-MS, leading to column degradation and ion source contamination.

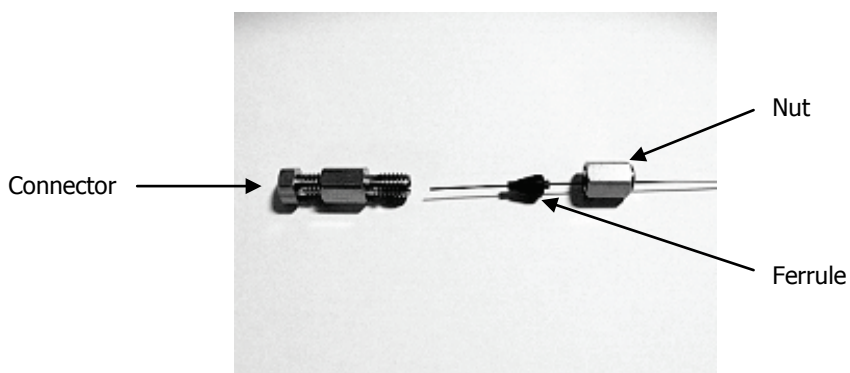


Figure 23 Correct arrangement of the nut and ferrule for connecting the transfer lines

The next stage of the installation is to connect the capillary lines to the correct ports on the GSV assembly. All the lines are held inside the oven alongside the GC column. Figure 24 shows the reference numbers for the ports on both of the gas switching valves; these are described in Tables 1–2.

NOTE: The stainless steel tubing, loop and connectors are factory fitted.

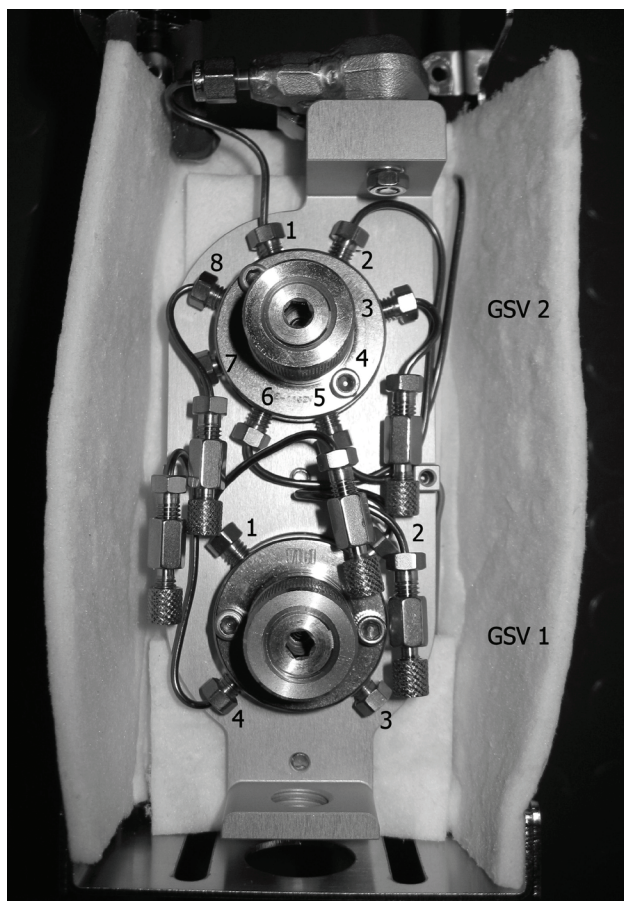


Figure 24 GSV assembly with related port numbers

Table 1 Description table for the GSV2 ports

Port	Description	Note
1	Sample inlet	Silicone-coated stainless steel tubing (OD 1/16")
2	Sample loop	Silicone-coated stainless steel tubing (OD 1/16")
3	Capillary column	Column installed in GC
4	Pre-heated carrier gas	Silicone-coated stainless steel tubing (OD 1/16")
5	Sample loop	Silicone-coated stainless steel tubing (OD 1/16")
6	Loop load tubing	Silicone-coated stainless steel tubing (OD 1/16")
7	Exhaust outlet	Empty
8	On-line restrictor	Deactivated column, ID 0.1mm, length approx 3 m

Table 2 Description table for the GSV1 ports

Port	Description	Note
1	Restrictor to MS gas transfer line	Deactivated column, ID 0.25 mm, length approx 1 m
2	Capillary column	Column installed in GC
3	Exhaust outlet	Empty
4	On-line restrictor	Deactivated column, ID 0.1 mm, length approx 3 m

15. Take the ends of the capillary lines from inside the GC oven and push them through the heated tube connected to the GSV assembly (Figure 25).

**Figure 25 GC oven with installed capillary column and restrictors**

16. Install each of the capillary lines (including the GC column) into the correct port of the GSV assembly and secure them using the stainless steel nut and graphite-polymer ferrule.

Push each line through the connection until you feel it stop, before tightening the nut with a 1/4" spanner until the capillary line does not move when pulled.

17. Check the PTFE tubes for the loop load ($\frac{1}{8}$ ") and the return (4 mm), which are supplied preinstalled, and tighten if necessary (Figures 26–28).

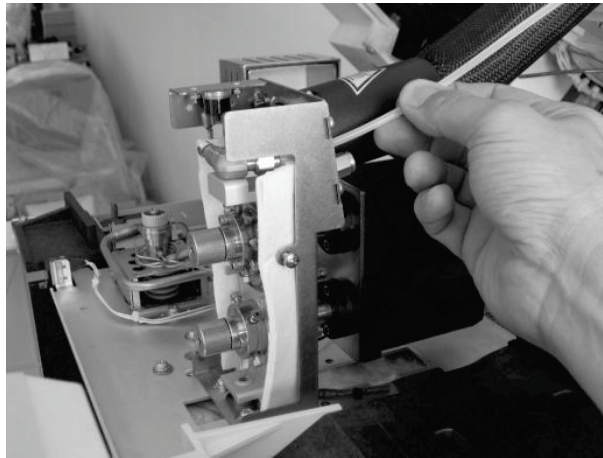


Figure 26 Locating the return tube



Figure 27 Checking for tightness of the loop load tube fittings

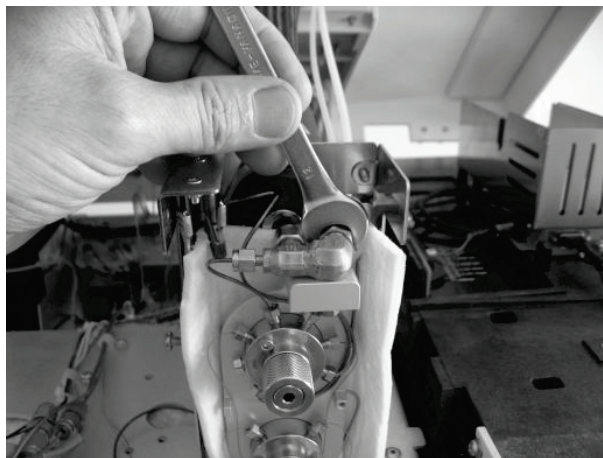


Figure 28 Checking for tightness of the return tube fittings

18. Fit the insulated cover over the GSV assembly, taking care not to trap any capillary lines (Figure 29).



Figure 29 Fitting the insulated cover over the GSV assembly

19. Install the outer cover over the GSV assembly (Figure 30).



Figure 30 Installing the GSV assembly outer cover

20. Tighten the left and right fixing screws on the outer cover (Figure 31).



Figure 31 Tightening the cover fixing screws

21. Pass the GSV heater power supply and thermocouple cables underneath the GC top cover from the rear of the GC and into the detector compartment (Figure 32).

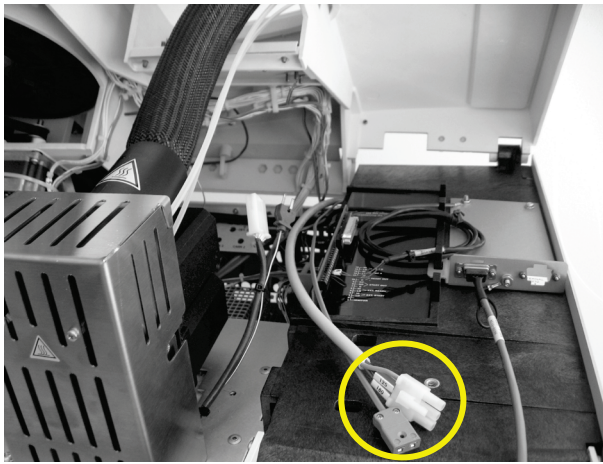


Figure 32 Inserting the GSV heater cables (circled) under the GC cover

22. Securely connect the cables to the heater and thermocouple connectors on the GSV assembly.

NOTE: The power supply voltage to the GSV assembly is 24 VAC.

The GSV assembly is now installed in the GC (Figure 33).

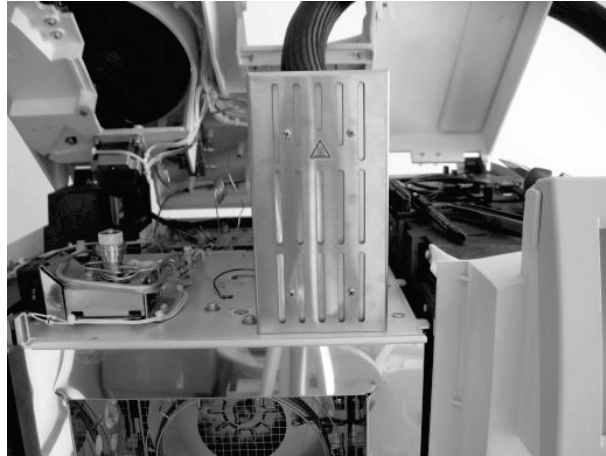


Figure 33 GSV assembly correctly installed in the front detector position

Before replacing the GC top cover, the remote I/O cable must be installed to allow an external trigger to start data acquisition by the GC-MS.

23. Insert the I/O cable under the top cover and connect wires 105 and 106 to GC connector positions 3 and 4, respectively (Figure 34).

This connection synchronizes the TL 9000 controller module with the start of the GC method. These events can be triggered either manually by activating the GSV2 valve on the controller module, or electronically by the Pyris software through the SeaLINK relay box (see *SeaLINK Relay Box Installation* on page 71).

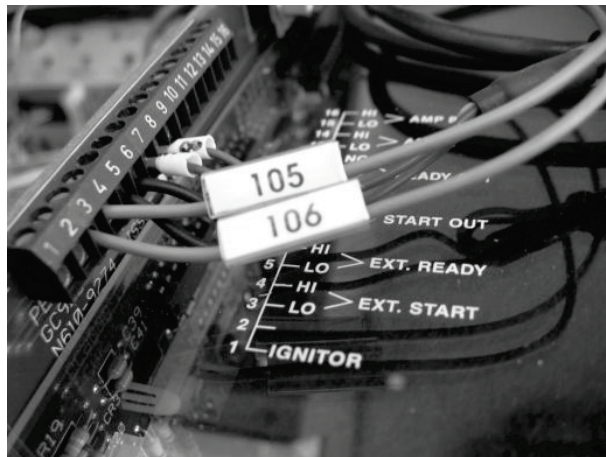


Figure 34 Connecting the remote I/O cable to GC external start connections

24. Remove the three screws securing the right-hand panel on the GC to access the electronic compartment.

25. Feed the two remaining connectors of the remote I/O cable into the compartment, as shown in Figure 35.

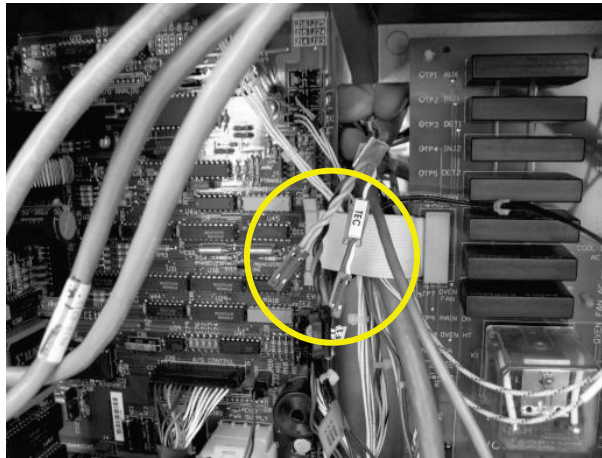


Figure 35 Passing the connectors of the remote cable (circled) into the GC electronic compartment

26. Locate the time event sockets TE1 to TE6 at the bottom right of the GC main board and connect the remote cable connectors TEA and TEB to any two of these sockets (Figure 36).

These connections will be used to program the time event table in the GC method, which will control the GSV1, GSV2 and LOOP load phases at the controller module. For more information, refer to *TurboMass Setup* on page 91.

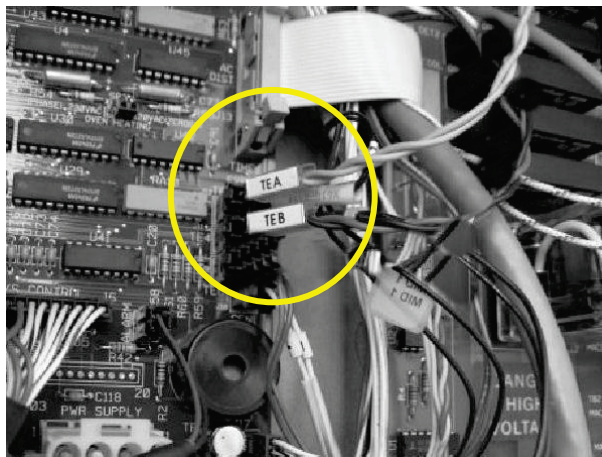


Figure 36 Connecting the remote cable to the available time event sockets on the GC main board (circled)

27. Unhook the GC top cover and close it down, fixing the cover screws in place (Figure 37).



Figure 37 Closing the GC top cover

28. Connect the motor cables to the GSV assembly motors, ensuring that the cables are connected to the correct motor as indicated by the labels (Figure 38).



Figure 38 Connecting the GSV cable motors to the controller

29. Use the spiral braid provided to arrange the cables and pipes into a single cord, as shown in Figure 39.



Figure 39 Arranging the cables and pipes using the spiral braid

Controller Module and Vacuum Pump Installation

The controller module should be positioned on a bench, adjacent to either the FT-IR spectrometer or the thermal instrument, so that there is at least 10 cm free space between it and the wall, and a similar gap at both sides. The vacuum pump should be positioned on the floor adjacent to the controller module, to avoid any vibration being transmitted to the instruments.

CAUTION

The controller module should never be covered.

The controller vacuum pump must be placed on the floor in a safe and dust-free area.

1. Turn the controller module so that the rear panel is facing you.
2. Connect all the male connectors with the appropriate female sockets, paying attention to the labeling to ensure that the connections are correct (Figure 40).
The pump cable is unlabeled but fits into the pump socket only.

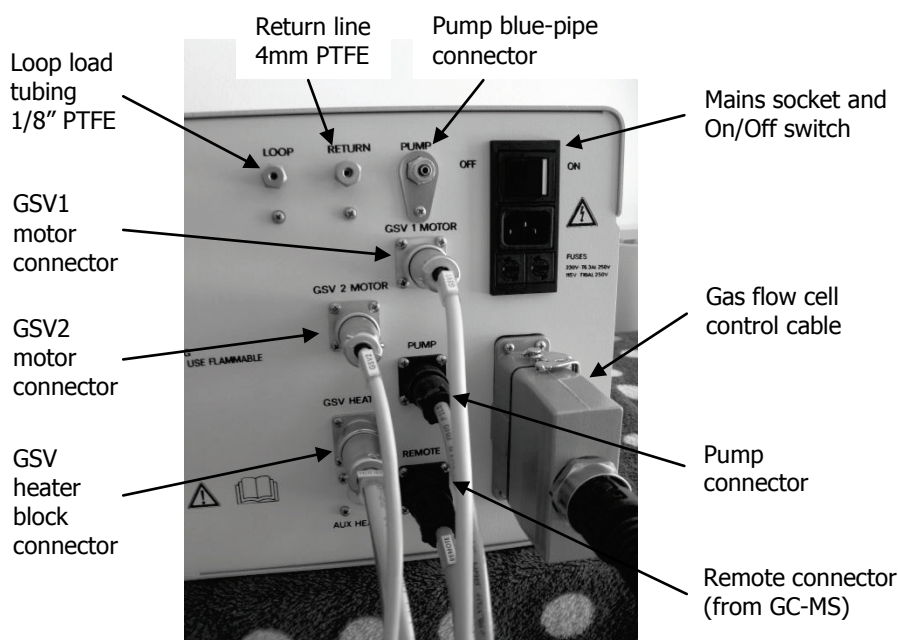


Figure 40 TL 9000 controller module (rear view) with electrical connectors in place

3. Fit the control cable from the gas flow cell into the 16-pin connection point and then clip it into place, by pulling the clip-lever towards you.
4. Hand tighten all the ring nuts to secure the connections.

5. Insert the blue pipe from the vacuum pump into the PUMP port, and hand tighten the lock ring (Figure 41).
6. Fit the 1/8" PTFE tube from the GSV assembly to the LOOP port, hand tighten the nut, and then secure it with a quarter turn using a 7/16" spanner (Figure 41).
7. Fit the 4 mm PTFE tube from the GSV assembly to the RETURN port, hand tighten the nut, and then secure it with a quarter turn using a 1/2" (12 mm) spanner (Figure 41).

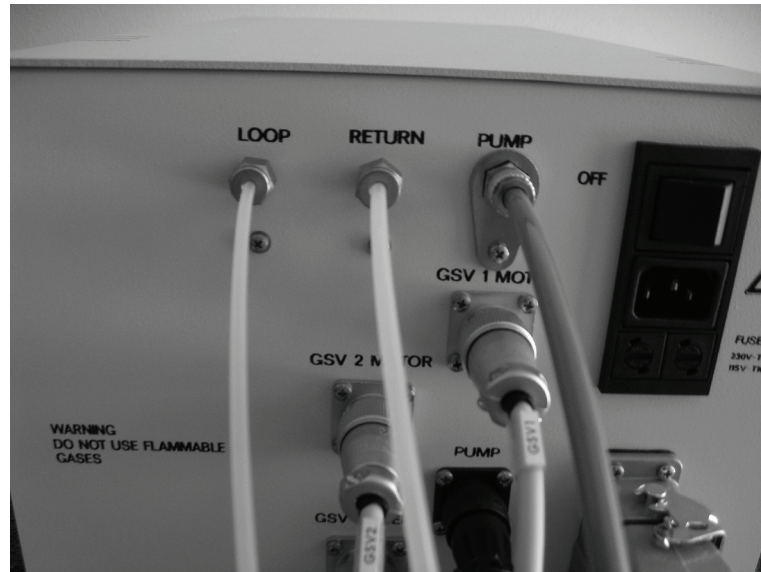


Figure 41 TL 9000 controller module (rear view) with pipelines fitted

8. Turn the controller module so that the front panel faces the front of the bench.

Gas Transfer Line Installation (STA 6000 / TGA 4000)

To connect the gas transfer line from the gas flow cell to your thermal instrument, follow the steps described below.

CAUTION

During the fitting, take care to ensure that nothing falls accidentally into the furnace of your instrument, as this may cause irreparable damage to its measurement systems.

1. Remove the lid from the furnace compartment.
2. Below the lid, loosen and remove the three fixing screws (Figure 42).

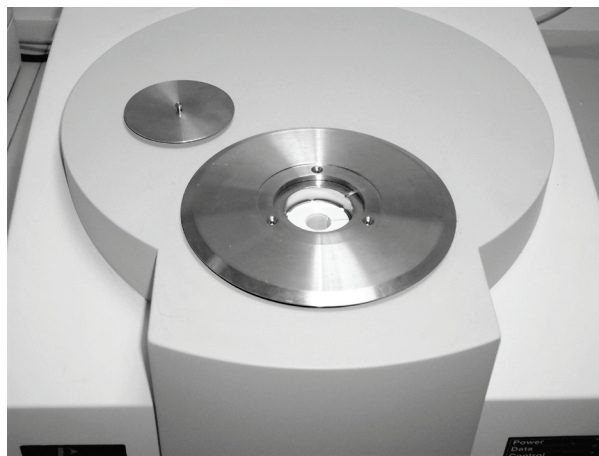


Figure 42 STA 6000 with furnace lid and securing screws removed

3. Position the transfer line support unit on top of the furnace compartment, and then secure it with the three M3 × 30 cross-headed screws provided (Figure 43).



Figure 43 Fitting the transfer line support unit

4. Slide the transfer line bracket as far back as it will go.
This is the bracket's open position (Figure 44).

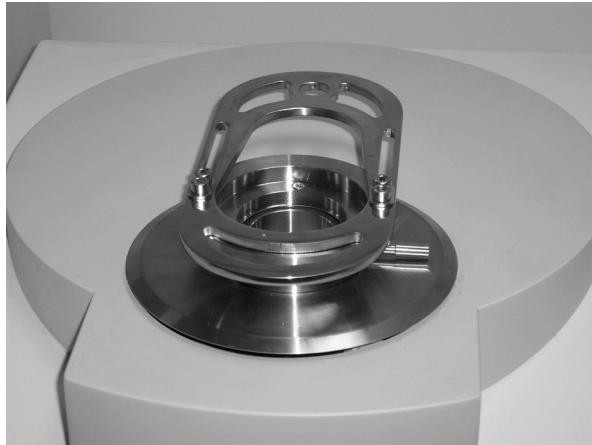


Figure 44 Bracket in open position

5. Replace the furnace lid.

NOTE: Use the furnace lid provided with the TL 9000 adapter kit once the transfer line support unit is fitted.

6. Place the end of the gas transfer line into the slot at the rear of the bracket.
See Figure 45, which shows the "not in use" configuration for the thermal instrument / gas transfer line assembly.

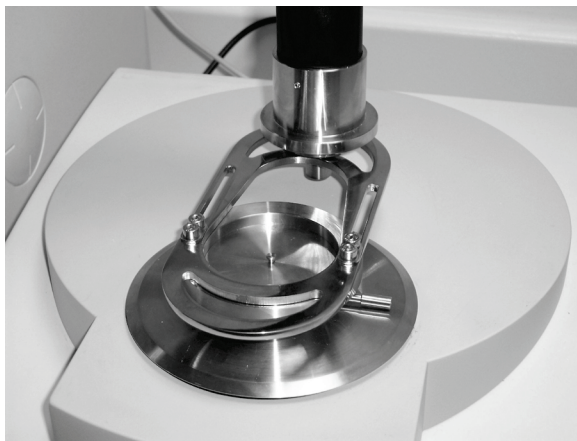


Figure 45 Gas transfer line assembly in "not in use" configuration

Gas transfer line installation (Pyris 1 TGA)

To install the Pyris 1 TGA transfer line adapter you must first remove the standard upper furnace ball joint and fit the upper furnace replacement supplied with the gas transfer line. Follow the steps described below.

1. Remove the platinum sample pan and hangdown wire, and store them in a safe place.



Figure 46 Pyris 1 TGA, with sample pan and hangdown wire removed

2. Remove the flow deflector (anti-diffusion cap) from the furnace upper ball joint, by unscrewing the two grub screws (hex-head screws) using the Allen key provided in the Pyris 1 TGA spares kit (Figure 47).

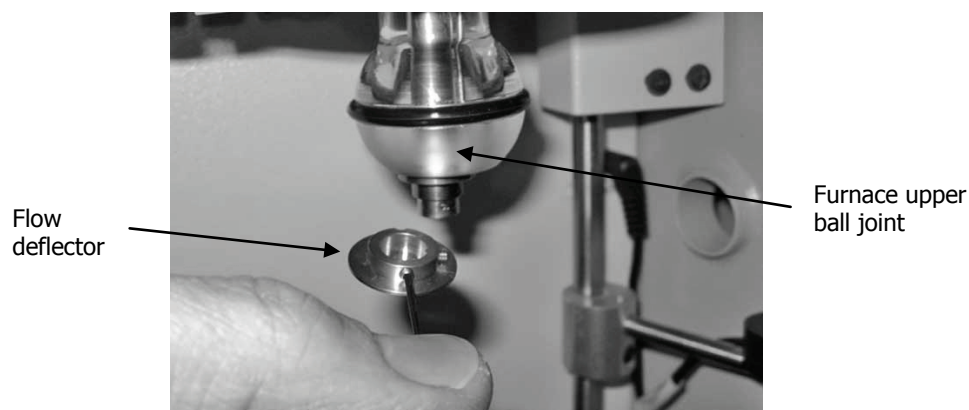


Figure 47 Removing the flow deflector

Store the flow deflector and grub screws in a safe place, for future use.

3. Loosen and remove the purge gas flow connector at the left of the Pyris 1 TGA (Figure 48).

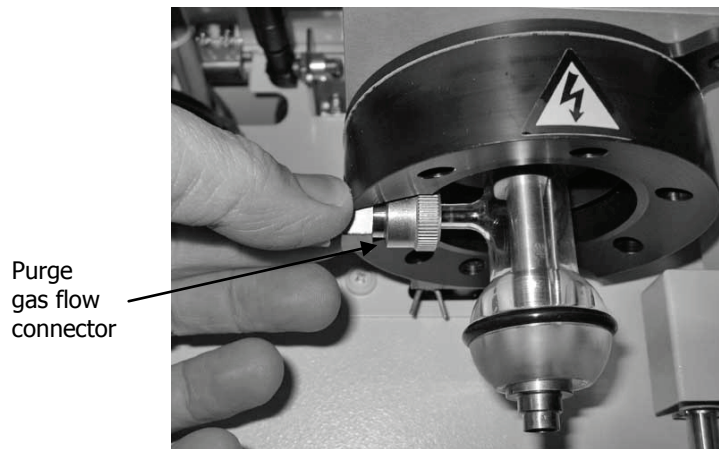


Figure 48 Removing the purge gas connector

4. Use a long-handled Allen key to loosen the four screws securing the furnace upper ball joint and then carefully remove it (Figure 49).

CAUTION

Take care to catch the furnace upper ball joint as the last of the screws is removed.

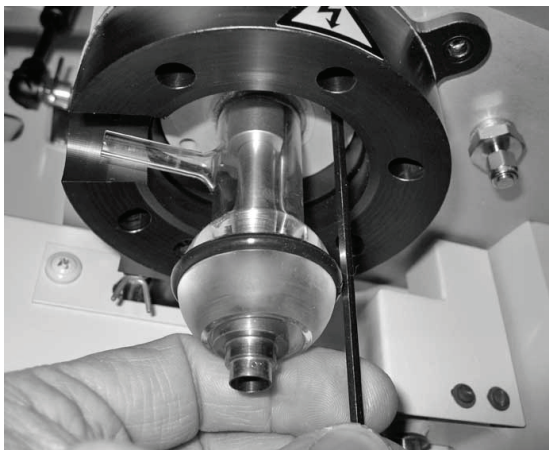


Figure 49 Removing the furnace upper ball joint

Keep the screws and washers in a safe place for future use.

5. Using a screwdriver, remove the two Phillips screws securing the plastic ionizer (Figure 50).

This loosens but does not detach the ioniser.

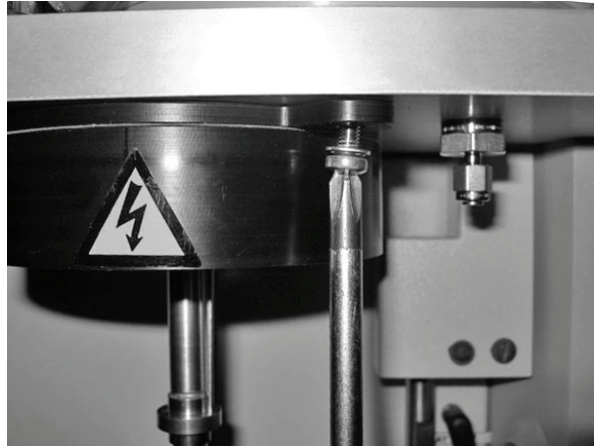


Figure 50 Loosening the plastic ionizer

6. Locate your upper furnace replacement (Figure 51).

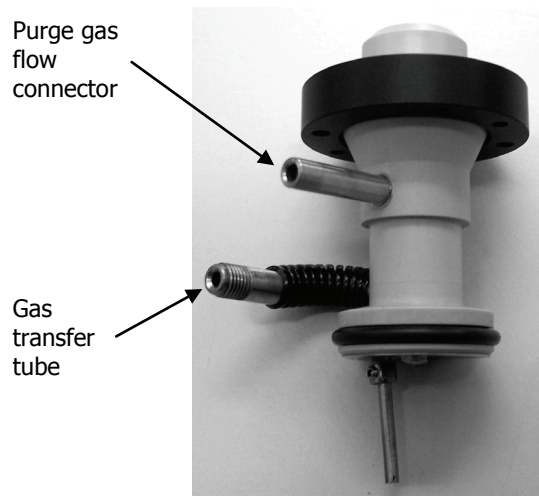


Figure 51 Upper furnace replacement

7. Slide the upper furnace replacement up over the central tube of the Pyris 1 TGA and attach it to the underside of the ionizer using the four screws provided (Figure 52).

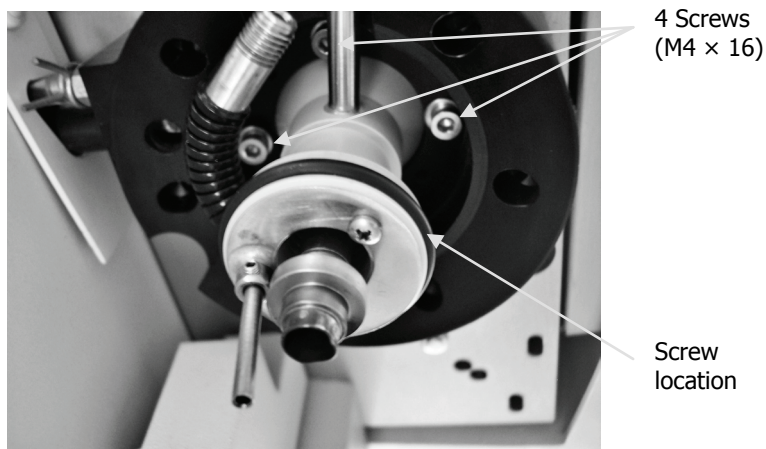


Figure 52 Upper furnace replacement fixed to ionizer

8. Locate your transfer line sleeve support and remove the metal ring that protrudes from it by loosening the two screws that hold it in place (Figure 53).

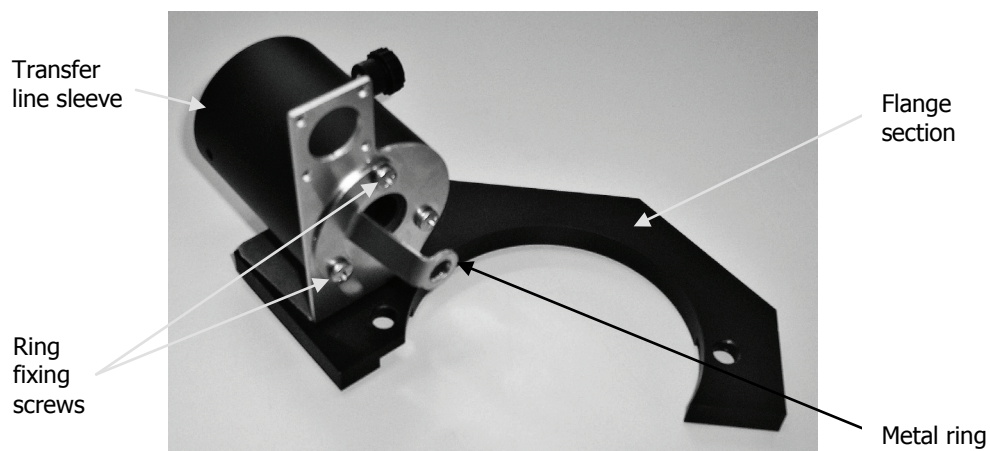


Figure 53 Gas transfer line sleeve support

Keep the metal ring and fixing screws in a safe place for future use.

9. To fix the gas transfer line sleeve support to your Pyris 1 TGA, begin by placing the flange section around the ionizer (Figure 54).



Figure 54 Positioning the gas transfer line sleeve support around the ionizer

10. Using an appropriate Allen key, secure the flange to the underside of the ionizer using the two M4 × 25 screws provided, each with three elastic washers (Figure 55).

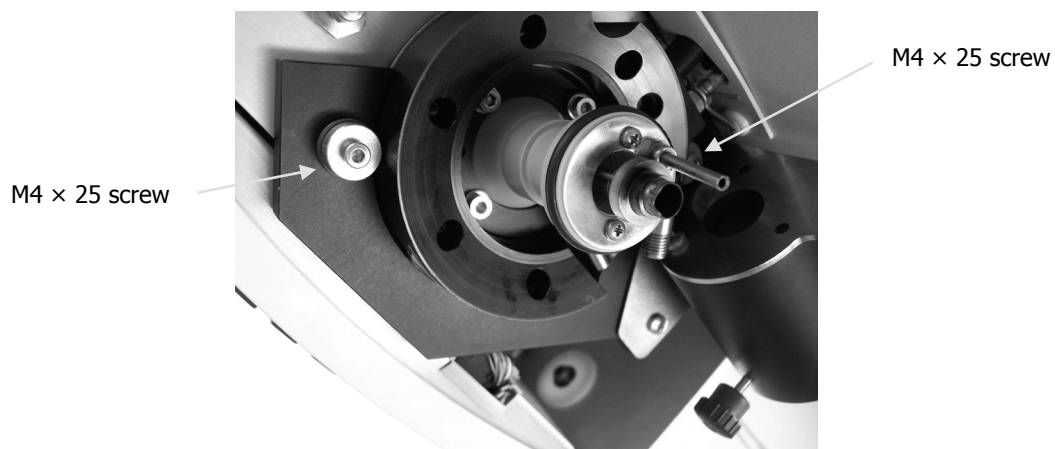


Figure 55 Fitting the gas transfer line sleeve support to the ionizer

11. Fit the metal ring previously removed from the gas transfer line sleeve support (Figure 53) over the end of the gas transfer tube (Figure 51) and then secure it back onto the gas transfer line sleeve support (Figure 56).

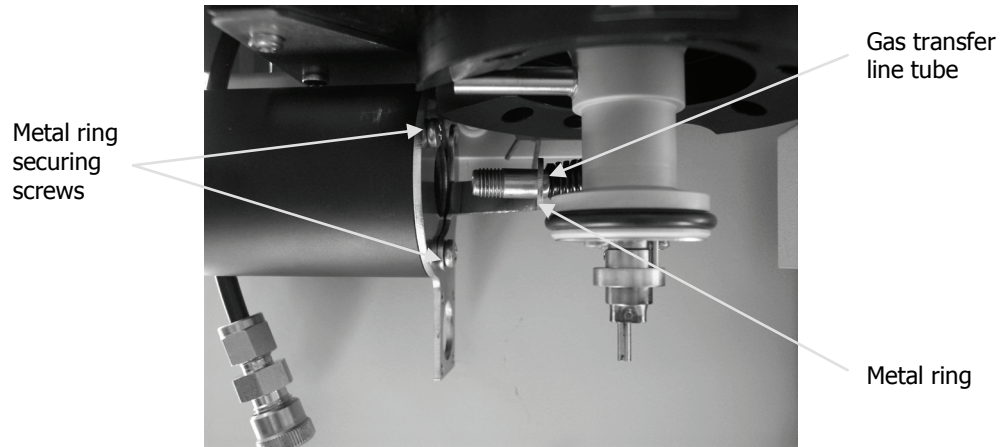


Figure 56 Fitting the metal ring to the gas transfer line

12. Take your gas transfer line and place the securing nut and O-ring onto the end of it (Figure 57).

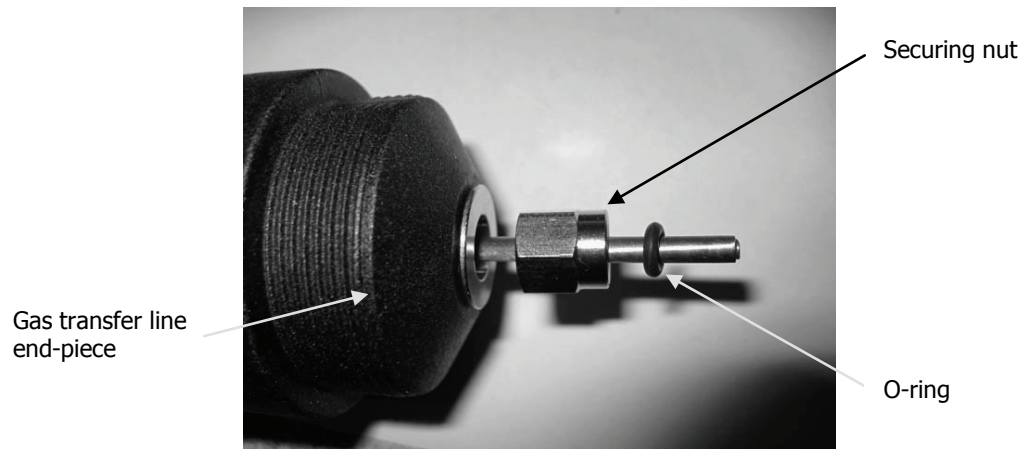


Figure 57 Gas transfer line ready for connection

13. Slide the gas transfer line through the sleeve.

14. Carefully align the end of the gas transfer line with the gas transfer tube, then fit them together and tighten the securing nut (Figure 58).
Finger-tighten the black plastic knob on the transfer-line sleeve to clamp the transfer line in place (shown in Figure 60).

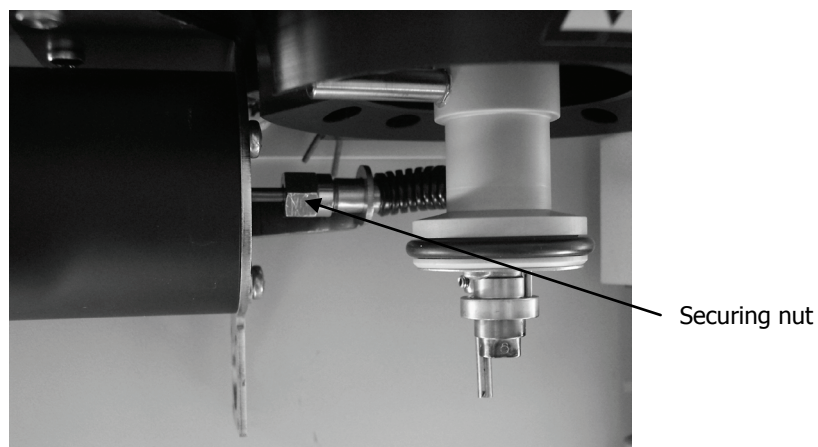


Figure 58 Gas transfer line fully connected

15. Use the two grub screws to attach the new flow deflector (Figure 59).

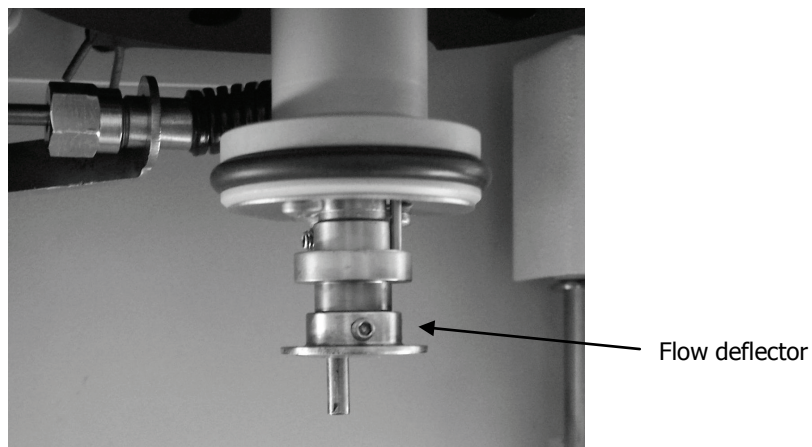


Figure 59 Flow deflector attached

16. Fit the Pyris 1 TGA purge gas flow to the new connector (Figure 60).

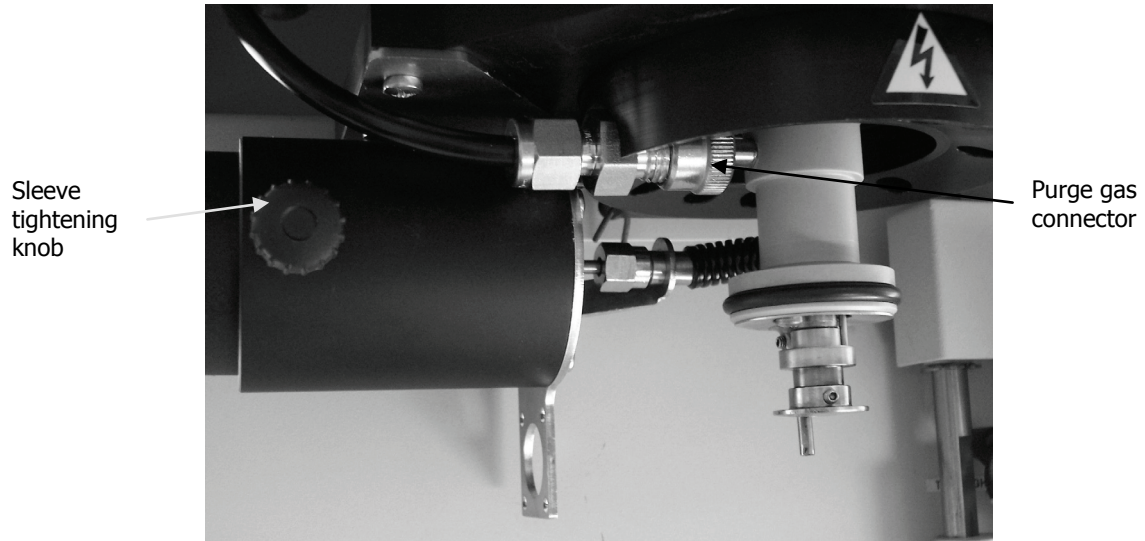


Figure 60 Purge gas flow connected

17. Refit the sample pan and hangdown wire (Figure 61).

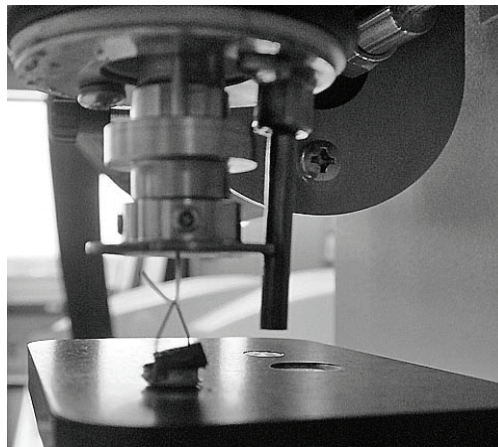


Figure 61 Sample pan and hangdown wire refitted

18. Check that the fume pipe protruding from the gas deflector is not touching the furnace; it should end a few millimeters above it (Figure 62).

The fume pipe is supplied pre-aligned. However, in the event that the fume pipe is too long, the adapter fixing it can be unscrewed and the pipe pushed upward a little.



Figure 62 Fume pipe position

Removing the gas transfer line

In the event that you want to use your Pyris 1 TGA without having it connected to the gas transfer cell, you should follow the steps described below to remove the gas transfer line.

1. If necessary, remove the platinum sample pan and hangdown wire, and store them in a safe place.
2. Loosen both the securing nut that connects the gas transfer line to the gas transfer tube and the black plastic knob on the transfer line sleeve (Figure 63).

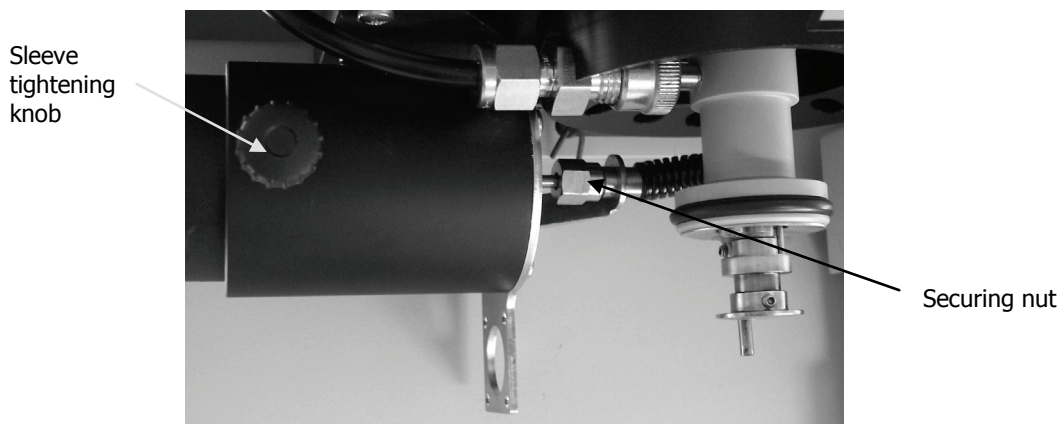


Figure 63 Removing the gas transfer line

3. Carefully slide the transfer line out, through the sleeve, and clear of your Pyris 1 TGA.
4. Remove the O-ring and the securing nut from the end of the gas transfer line and fit them back onto the gas transfer tube, without the gas transfer line.
5. Fit the blanking plug provided (P/N L8201898) into the securing nut.

SeaLINK Relay Box Installation

To connect the SeaLINK relay box (P/N 09401085) to your computer, follow the steps described below.

NOTE: Ensure that you have installed the software drivers before you install the relay box. See page 28 for details.

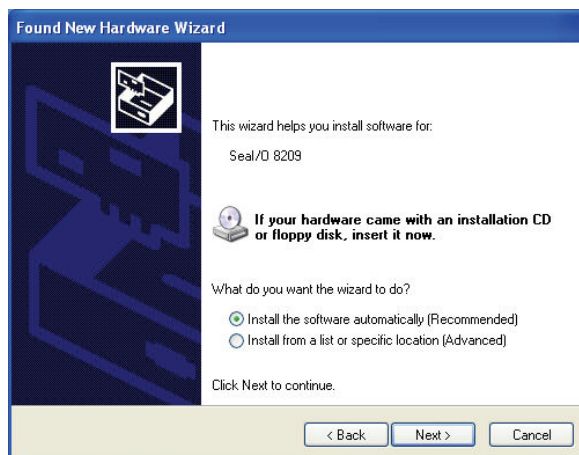
1. Connect the USB cable to the Port:A side of the relay box and then plug the other end into a free USB port on the computer that runs Pyris.

NOTE: You should ensure that you choose a USB port that is unlikely to be used for any item other than the relay box. If you move the relay box to another USB port, you will have to go through this installation procedure again for the new port.

Your computer will automatically recognize the relay box as a new item of hardware and will start up the Found New Hardware Wizard.



2. Select the **No, not this time** option and then click **Next**. The wizard starts.



3. At this point, contrary to the instruction given on screen, do NOT insert the Sealevel software disk. Simply click **Next**.

The wizard keeps you informed of the progress of the installation.



When the installation is complete, the Completing page is displayed.



4. Click **Finish**.

NOTE: The Found New Hardware Wizard may repeat the hardware installation process a number of times, as it loads each channel separately.

5. Connect the remote start RS232 cable (P/N L1365756) to a free COM port on the computer that runs Spectrum and Spectrum TimeBase.

NOTE: This may be the same computer as that running Pyris or a different one.

6. Insert the bifurcated wires of the RS232 cable into the two cable connectors labeled "0" on the Port:B side of the relay box and secure them in place with a screwdriver (Figure 64).
7. Connect the wires from the external start connections on the GC (shown in Figure 34) to the terminals labeled "1" on the Port:B side of the SeaLink relay box (Figure 64).

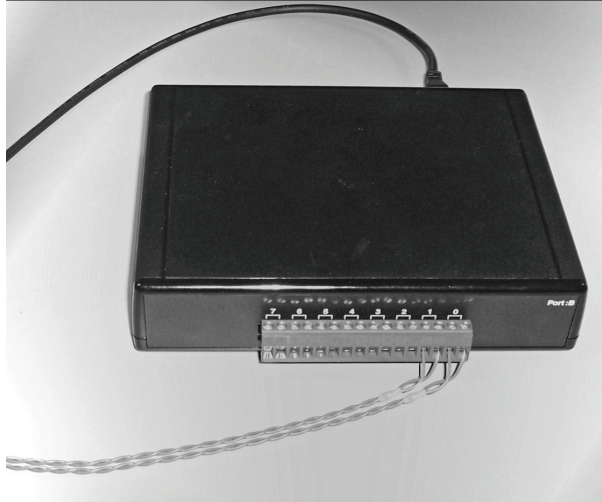


Figure 64 SeaLINK relay box showing connections to the FT-IR and GC-MS computers



Preparing the System for Use

Configuring the Software

The following pages describe how to configure the Spectrum, Spectrum TimeBase and Pyris software for use with the TL 9000 hardware. Details of the configuration of the TurboMass software can be found in the *Clarus 560/600 GC-MS Tutorial* (P/N 09936769).

For information on how to configure the software components to suit your particular experimental requirements, see the appropriate on-screen help which you can access by selecting the **Contents and Index** option from the software Help menu.

Spectrum setup

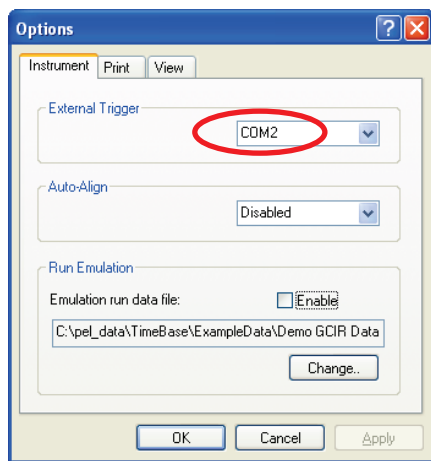
If you have not previously been using Spectrum in conjunction with your spectrometer, you should follow the steps below to add the instrument to Spectrum and to establish it as the default instrument for both Spectrum and Spectrum TimeBase.

NOTE: If you have already added your spectrometer to the Spectrum software, simply carry out step 5 of the procedure described below to ensure that Spectrum TimeBase recognizes your spectrometer as the default instrument.

1. Launch the Spectrum software, log on and select the **Work Offline** option.
2. Select **Add Instrument** from the Instruments submenu of the Setup menu.
The Instrument Install wizard is started.
3. Follow the on-screen instructions provided by the wizard to add your spectrometer to the Spectrum software.
4. When the wizard completes, log off from Spectrum.
5. Log on again to Spectrum, this time connecting to your instrument.
This sets your spectrometer as the default instrument. Spectrum TimeBase always operates with the default instrument.

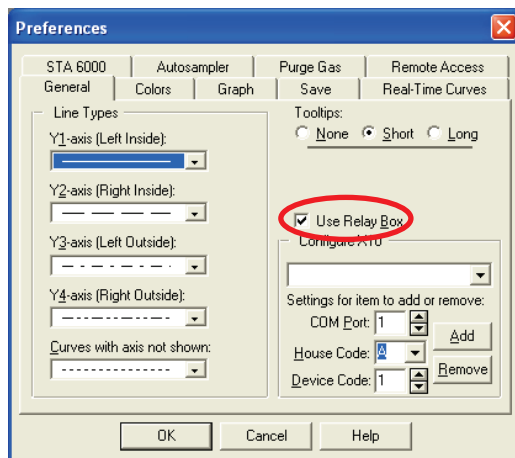
Spectrum TimeBase setup

Within Spectrum TimeBase, use the **Options** menu item to set the External Trigger option to the COM port used by the SeaLINK relay box. This ensures that Spectrum TimeBase knows where the external trigger is coming from.



Pyris setup

Within Pyris, select **Preferences** from the Tools menu. On the General tab, ensure that the **Use Relay Box** checkbox is checked. This ensures that any external trigger generated by Pyris will be sent to the SeaLINK relay box.



Setting up the Hardware

FT-IR spectrometer setup

NOTE: Full details of how to prepare your spectrometer for use are given in the documentation supplied with the instrument.

When the gas flow cell is installed in the instrument it will automatically be included in the beampath, as shown in the Spectrum TimeBase software. The beampath is shown on the Beam tab in the Setup Instrument dialog available from the Instrument menu.

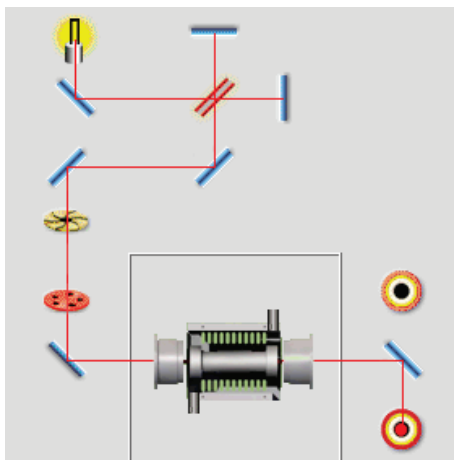


Figure 65 Beampath in Spectrum software showing the gas flow cell

The default instrument setting will change to values typically used for evolved gas analysis.

Setting	Value
Ordinate Scale	%T
Resolution	8 cm ⁻¹
Number of Scans	2
CO ₂ / H ₂ O suppression	Off

Once you have fitted the gas flow cell into the IR sample compartment of your spectrometer, you will need to make minor adjustments to its position, within the cell assembly, to maximize the level of infrared energy reaching the detector in your instrument.

1. Switch on your spectrometer and then start up the Spectrum software.
2. Log on, and select your instrument.
3. Remove the gas flow cell, on its baseplate, from the IR sample compartment.
4. Select the **Monitor** option from the Measurement menu.
The software displays the energy of the beam.
5. Record the maximum energy value reached.
6. Replace the gas flow cell, then remove the stainless steel cover and loosen the securing nuts.
See *TL 9000 Gas Flow Cell Installation* on page 37 for details.
7. Carefully push and pull the cell backwards and forwards until the highest energy readings are displayed.

NOTE: The maximum energy reading with the cell in place should be approximately 25–30% of that shown when the cell and its baseplate are totally removed from the IR sample compartment.

Energy readings significantly below this level may indicate a poorly aligned gas flow cell, or dirty or damaged cell windows. See *Cleaning and replacing gas flow cell windows and gaskets* on page 107 for details of how to clean the cell windows.

8. Tighten the securing nuts and replace the cover.

Thermal instrument setup

Full configuration instructions for your thermal instrument can be found in the Pyris on-screen help.

GC-MS instrument setup

Full configuration instructions for your GC-MS instrument can be found in the TurboMass on-screen help and the *Clarus 560/600 GC-MS Tutorial* (P/N 09936769).

Controller module setup

Preparing the controller module for use involves setting the operating temperatures for both the gas flow cell and the gas transfer line, and setting the gas flow rate.

1. Switch on the controller module, using the main power switch on the rear of the unit.
2. Open the door on the front of the module to reveal the front panel (Figure 66).

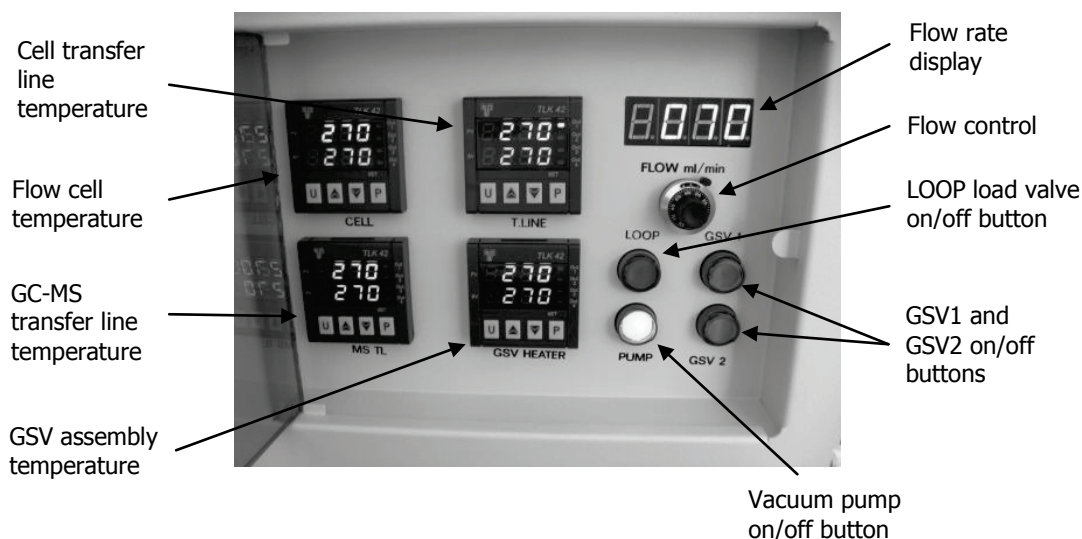


Figure 66 Front panel of TL 9000 controller module

The front of the controller features:

- An on/off button for the vacuum pump flow.
- A flow rate adjustment control, and a digital flow rate display.
- A temperature controller for the gas flow cell (labeled CELL).
- A temperature controller for the transfer line from the thermal instrument to the gas flow cell (labeled T LINE).
- A temperature controller for the transfer line from from the gas flow cell to the GSV assembly (labeled MS TL).
- A temperature controller for the GSV assembly (labeled GSV HEATER).

The readouts on the temperature controllers show the actual device temperature in red, and the operating temperature setpoint for the device in green.

3. To change the operating temperature of a device, press the button labeled P on the appropriate temperature controller.

The display changes to show **SP1** (set point 1).

4. Use the up-arrow and down-arrow buttons to adjust the operating temperature to the required value (standard values are 270–280 °C).

The operating temperature required will vary, depending upon the material you are working with. You should set the temperatures to values which will prevent condensation of likely combustion or pyrolysis products.

CAUTION

The maximum temperature value is 350 °C, which should not be used continuously. The recommended operating temperature range for the rotors in the GSV assembly is 200–350 °C.



WARNING

If you have changed the windows in the gas flow cell from the standard KBr type (P/N L8201801) to ZnSe (P/N L1270766), the temperature setting should not exceed 250 °C. ZnSe evolves toxic fumes at high temperatures.

See Controller module setup on page 80 for details of how to set the temperature.

5. When you have set the temperature to the required value, press P again to save the value.
6. Repeat steps 3 to 5 to set the operating temperature for all the temperature controllers.
7. When the readouts on the front of the module show that the temperatures of all the components have reached at least 50 °C, press the button labeled PUMP on the front panel of the module to switch on the vacuum pump.
8. Push the locking device on the right of the gas flow control upwards, to release it.
9. Adjust the flow control until the flow rate display on the front panel shows the rate required in ml/min.
The maximum flow rate is 200 ml/min.

NOTE: The flow rate set on the controller module must be less than that entering your thermal instrument, so as to leave a residual flow. The residual flow ensures that air is not drawn into the furnace, burning the sample.

If you are using a Pyris 1 TGA, a Thermal Analysis Gas Station (TAGS) should be used to set the flow rate for your thermal instrument.

10. Lock the flow control.

Gas flow cell setup

The FT-IR sample compartment area may be purged with dry air or nitrogen. A flow rate of 300–500 ml/min is suitable. To initially remove any unwanted vapor, a higher flow rate, for example 2–3 l/min, may be used for a few minutes.

Vacuum pump, gas transfer line and SeaLINK relay box setup

No setup procedures are required to configure these hardware components.



Setting up an Experiment **and Collecting Data**

Readying the Hardware

1. Switch on the GC-MS and allow a suitable operating vacuum to stabilize.
2. Switch on the spectrometer and the thermal instrument.

NOTE: You should also ensure that the gas flow and cooling for the thermal analyzer are switched on.

3. Switch on the controller module.
4. When the read-out on the front of the controller module shows that the temperatures of all the components have reached at least 50 °C, press the green button on the front panel of the module to switch on the vacuum pump.
5. Allow the gas flow cell and the gas transfer lines and GSV to reach their specified operating temperatures.

NOTE: In the event that any component fails to reach its operating temperature, or the temperature fluctuates, see *Re-tuning the temperature controls* on page 101.



WARNING

All heated parts, the gas flow cell housing, the gas transfer lines and the GSV assembly, can become very hot, depending on the operating temperatures selected.

The outer surface of the gas transfer line, for example, can reach temperatures of approximately 100 °C when heated to 350 °C.

ALWAYS USE PROTECTIVE GLOVES WHEN HANDLING HEATED PARTS.

Loading your Sample

STA 6000 / TGA 4000

1. Place an empty crucible on the sensor, replace the furnace lid and tare the empty weight.
2. Remove the crucible and place your sample in it.
3. Carefully, with a pair of tweezers, lower the crucible into the furnace compartment.
4. Position the gas transfer line on top of the furnace compartment, so that it is flush with the transfer line support unit (Figure 67).



Figure 67 Inserting the gas transfer line

5. Slide the transfer line bracket as far forward as it will go, into its closed position (Figure 68).



Figure 68 Transfer line bracket in closed position

Pyris 1 TGA

Follow the instructions in your thermal instrument hardware guide.

Pyris – Creating a Method

Within Pyris, use the Method Editor to create a method to control your thermal instrument.

The method should include actions to switch the external channel used by TimeBase ON when spectrum data are to be acquired and OFF at the end of the experiment. These can be set up using the **Trigger an External Event** option in the Pyris software.

The Program tab of the Pyris Method Editor is shown in Figure 69.

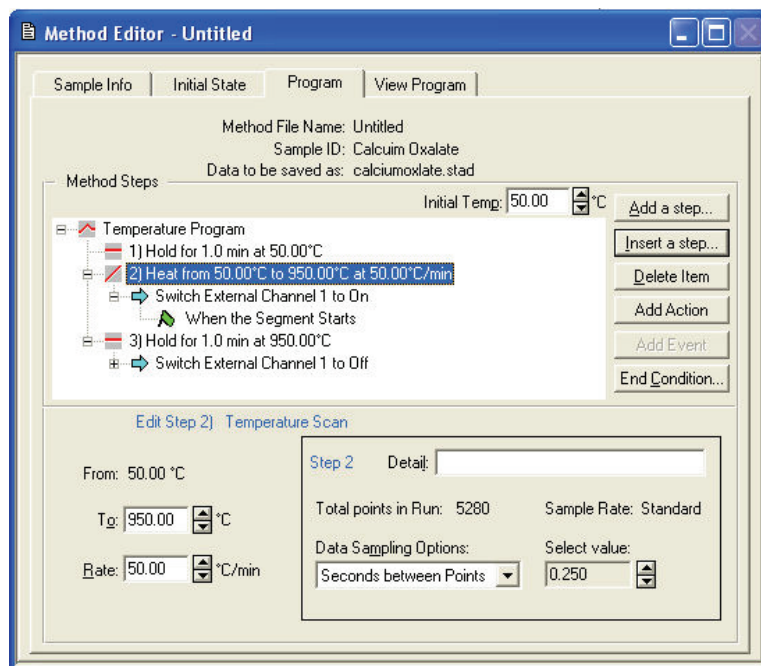


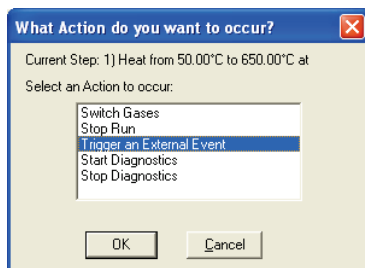
Figure 69 Program tab of Pyris Method Editor

The above experiment starts when Pyris reaches a temperature of 50 °C. The sample is held at that temperature for 1 minute and then heated up to 950 °C at a rate of 50 °C per minute. It is then held at 950 °C for a further minute.

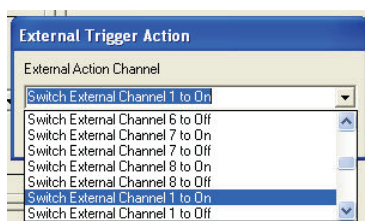
The trigger to start the acquisition of data by Spectrum TimeBase happens after the sample has been held at 50 °C for 1 minute. The trigger to stop data acquisition happens when the sample has been held at 950 °C for 1 minute.

To set up an external event trigger from the Program tab:

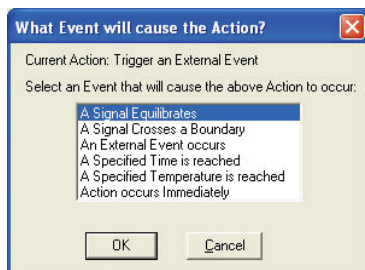
1. Click the **Add Action** button on the Method Editor screen (Figure 69).
You are asked what type of action you want to occur.
2. Select the **Trigger an External Event** option, and click **OK**.



3. Select which external channel is to be triggered and whether it is to be switched On or Off.



4. Once you have selected a channel and trigger type, specify the type of event that will cause the trigger to occur.



NOTE: For the TL 9000, an additional command line is needed to switch on external channel 2, which starts the GC-MS acquisition. The GC method then runs the program described in the TurboMass software and controls the relevant valves in the GSV assembly. The Pyris software can then be commanded to switch off channel 2 a few seconds after switching it on. This will not affect the GC method, which will continue for the specified run time.

Spectrum TimeBase Setup

Specifying CO₂ and H₂O suppression

If the combustion of your sample is likely to generate H₂O and/or CO₂, you should ensure that the CO₂/H₂O suppression feature is switched on.

1. Select the **Setup Instrument** option from the Instrument menu.
2. Select the **Instrument** tab.
3. Ensure that the **CO₂/H₂O Suppression** checkbox is selected.

If you want the spectral data to include H₂O and CO₂, ensure that the checkbox is not selected.

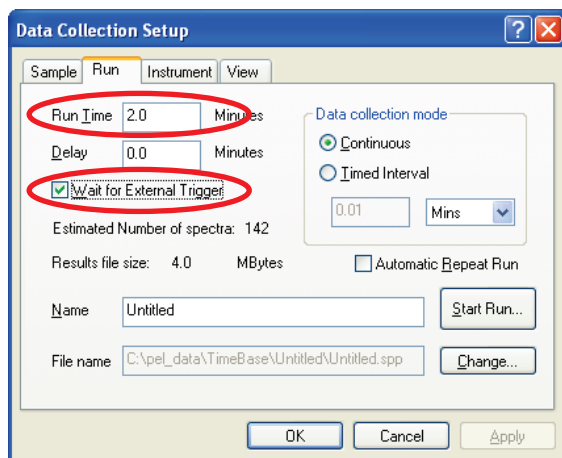
Running a background scan

Before you acquire spectral data, you should carry out a background scan.

- Select **Scan Background** from the Instrument menu.

Data collection setup

1. Select **Setup Data Collection** from the Instrument menu.
2. On the Run tab of the Data Collection Setup dialog, specify how and where Spectrum TimeBase is to capture spectral data.



In particular, you should ensure that:

- The **Wait for External Trigger** box is checked, so that the start of data acquisition is controlled by Pyris.
- The **Run Time** is set to a length of time sufficient to allow for data to be acquired for the full length of the experiment, based on the temperature range and the rate of heating or cooling you entered in your Pyris method. See *Pyris – Creating a Method* on page 87 for details.

NOTE: If required, you can also use a Delay value in conjunction with the **Wait for External Trigger** command. This would allow you, for example, to create a Pyris method in which the trigger to start data collection is sent at the start of an isotherm, but the first spectrum is not actually acquired until heating has started.

If you use a Delay value, you should be aware that the delay period is included as part of the Run Time specified. So a run time of 3 minutes, with a delay of 1 minute, would result in 2 minutes' of data being collected.

TurboMass Setup

There are two modes in which to operate the GC-MS system in combination with the TL 9000 hardware. A brief description of each will be given here before the setup of the TurboMass software is described.

Chromatographic separation mode

In this mode, the GSV assembly operates an internal three-way valve to fill the loop tubing with a "snapshot" of the gaseous sample passing through the transfer line from the gas flow cell. This gas sample is collected for a default time of 20 seconds, after which the GSV2 valve moves into the injection position and the sample is introduced on to the GC column. The column separates the sample into its components and produces a chromatogram. The components then pass into the MS where a mass spectrum for each is obtained, which can be compared to a library of spectra to identify the compounds in the mixture.

The operation of the GSV assembly in this mode is controlled using the TEA connection attached to any available timed event output on the GC main board (Figure 36).

On-line mode

In this mode, the GSV assembly connects the gaseous sample being transferred from the gas flow cell directly to the ionizer of the MS (through a capillary restrictor to prevent overload of the ionizer). The high vacuum of the MS continuously withdraws a portion of the gas sample through the restrictor and produces a mass spectrum. Since no separation is taking place, the system is configured to analyze for the presence of selected ions throughout the experiment (Single Ion Recording).

The on-line mode is controlled using the TEB connection attached to any available timed event output on the GC main board (Figure 36).

Creating methods to control the GC-MS

The steps in setting up the TurboMass software for an experiment are given below. For full details of how to operate the software, refer to the *TurboMass Software Guide* (P/N 09931016) supplied with your software.

On starting TurboMass you need to create a new project for your experiments and then set up your GC and MS method collection parameters. The exact parameters required will depend on your sample, and the range of time over which you need to sample from the thermal instrument. This is why it is important to determine the characteristic thermal behavior of the sample before setting up your GC-MS method.

The TurboMass top-level screen contains the GC and MS status, sample list and access to all other functions (Figure 70). The pane on the left side of the screen shows the status of the GC and the MS. You need to set up the MS tune parameters, the GC method and the MS method for your experiment.

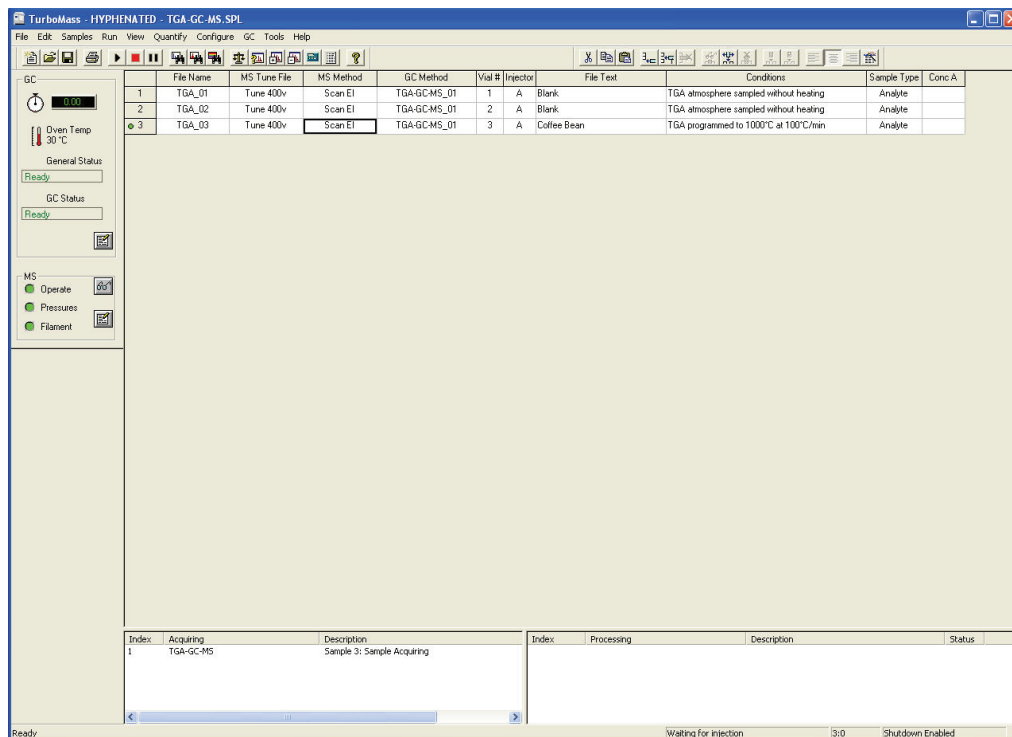



Figure 70 TurboMass top-level screen

1. Click on the icon  to display the Tune page (Figure 71).
This page allows you to tune the mass spectrometer, set the GC interface temperature, and monitor the instrument vacuum. The parameters in the MS Tune File are used when running the MS method.

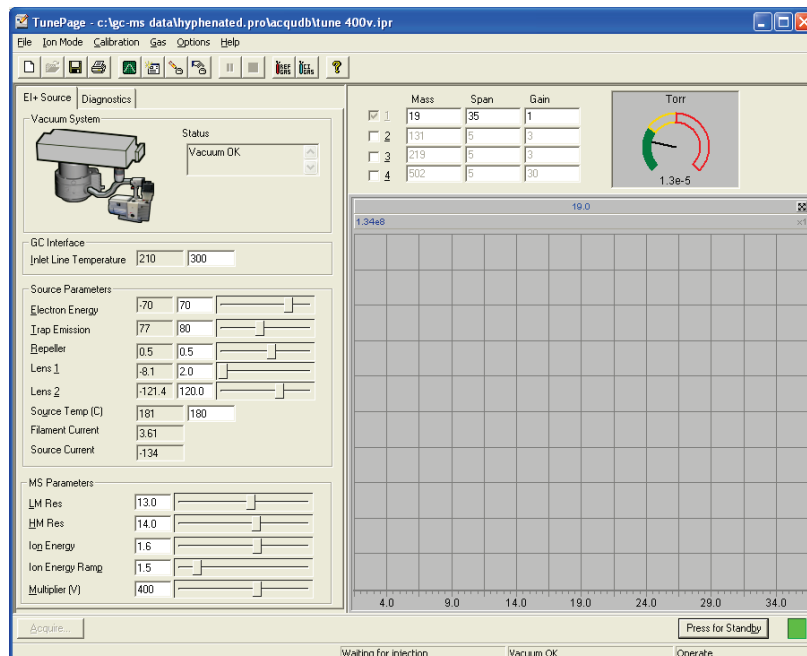


Figure 71 Tune page in TurboMass

2. Perform an AutoTune on the MS.
Refer to *Running UltraTune Custom (AutoTune)* in the *TurboMass Software Guide* (P/N 09931016) for further information.
3. Set up a GC method using the Instrument Control dialog (Figure 72).

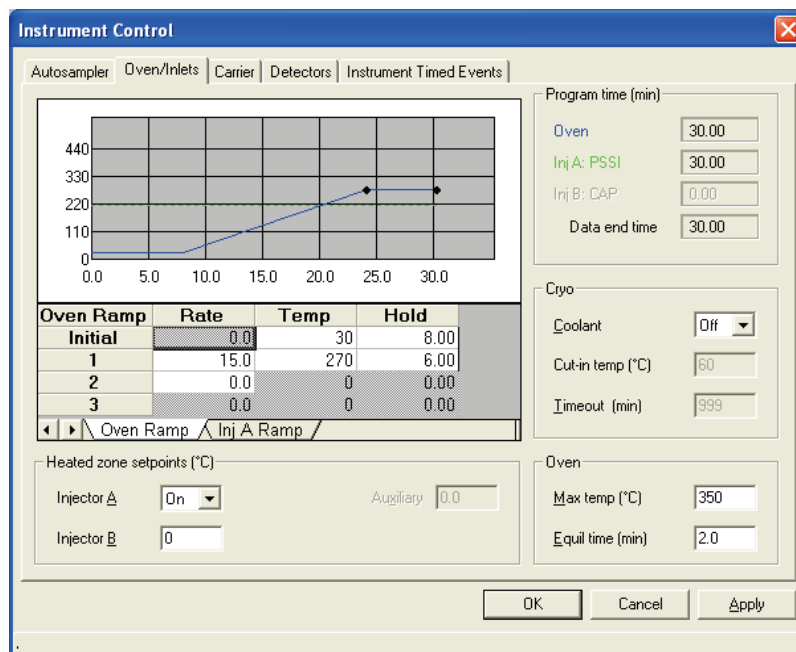


Figure 72 GC method oven temperature program in the Instrument Control dialog

In this example, the oven temperature is isothermal at 30 °C for the first 8 minutes, while the sample is collected on the GC column, and then ramps up to 270 °C. The GC Injection Source should be set to **Manual** on the Autosampler tab.

A thermal ramp program is usually suitable when using chromatographic separation mode, depending on the characteristics of your sample. When operating in on-line mode, the oven should be held at the same temperature as the transfer line to prevent any of the components from condensing in the restrictor.

4. Use the Instrument Timed Events tab to activate the correct timed event output for your experiment, which will operate the GSV assembly (Figure 73).

The timed event outputs to be activated are those to which the TEA and TEB connections were made during the installation of the GSV assembly (Figure 36). The output must first be configured to operate the corresponding valve (V1–6), and then the TurboMass software must be setup to display the correct valve settings. Refer to your GC-MS system documentation and the *TurboMass Software Guide* (P/N 09931016) for further information.

Once the software is correctly configured, the valves will be available for controlling from the Instrument Timed Events tab. The valve assigned to TEA should be activated if using chromatographic separation mode, and the valve assigned to TEB should be activated if using on-line mode. The initial settings in the lower right area of the tab should be **Off**. The appropriate valve is selected in **Event**, and the value set to **On**. A time of 0.01 min is recommended to ensure that the switching of the valve occurs almost immediately after the GC-MS analysis is triggered by the SeaLINK relay box.

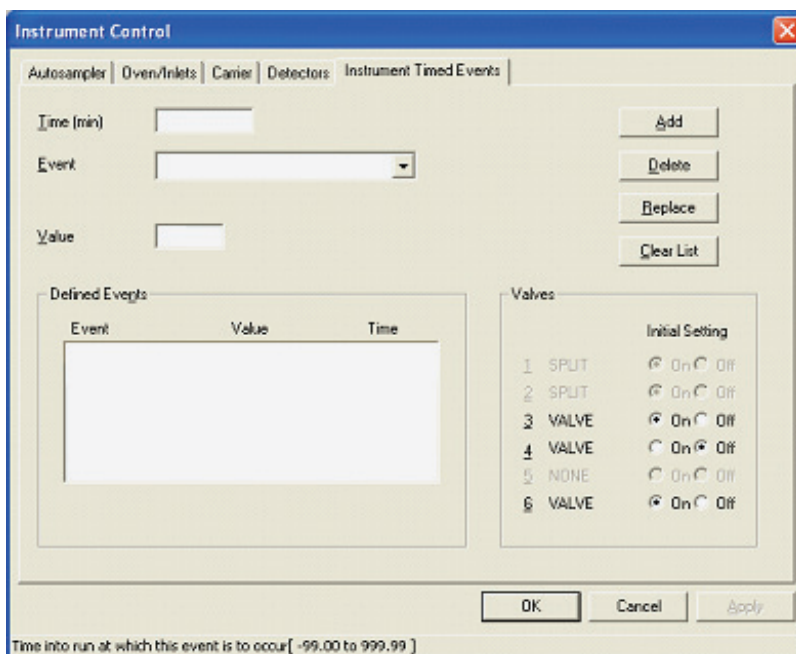


Figure 73 Instrument Timed Events tab

- Using the Function List Editor in TurboMass, set up an appropriate MS method. Select either **MS Scan** for chromatographic separation mode or **SIR** (Single Ion Recording) for on-line mode (Figure 74).

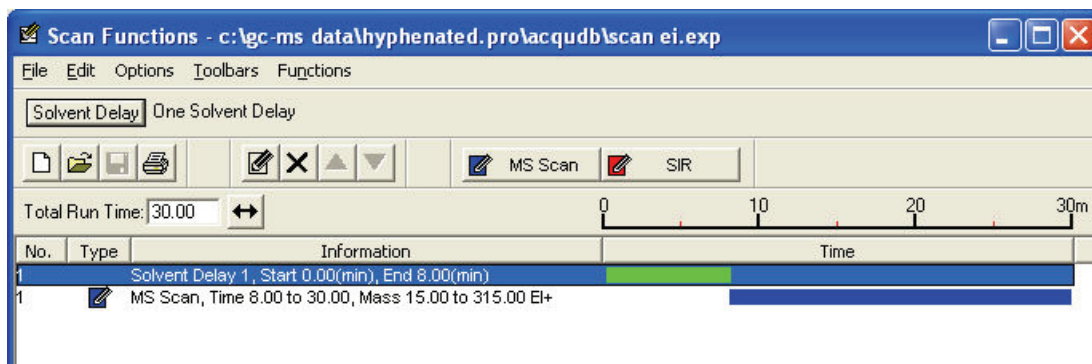


Figure 74 Function List Editor for setting up an MS method

The Function List Editor allows you to set the start and end times, mass range and ionization mode of the method, and how the data are stored and processed. A solvent delay can be included, which delays activating the MS filament until oxygen introduced during switching of the GSV assembly has passed through the system. This is particularly important if operating in on-line mode, when a delay of 1 or 2 minutes is recommended.


Refer to *Function List Editor* in the *TurboMass Software Guide* (P/N 09931016) for further information.

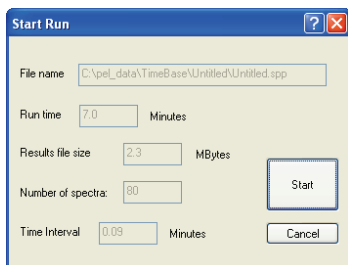
- Set up the sample list for the experiment, with the GC and MS methods entered into the appropriate fields.
- Select **Run** from the TurboMass top level Run menu to open the Start Sample List Run dialog.
- Check that the correct samples are selected and click on **OK**.
The GC-MS will wait for a trigger to start the analysis. In the TL 9000, the trigger to start the GC method comes from the input to the external start connections (shown in Figure 34). The GC itself then triggers the start of the MS method.

You are now ready to start the experiment using Pyris.

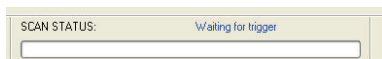
Collecting Data

When you are ready to start your experiment and collect data, follow the steps described below.

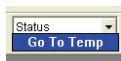
1. Start the TurboMass software.
When set up, TurboMass will display the GC and MS status as **Ready** and **Not Acquiring**.
2. Start the Pyris software.
3. Start Spectrum TimeBase and select your IR instrument.
4. Either click on the  icon in the toolbar or click the **Start Run** button on the Run tab of the Data Collection Setup dialog.
The Start Run dialog is displayed.



5. Click **Start**.
6. Until data acquisition is triggered by the Pyris method, the Spectrum TimeBase toolbar will show a SCAN STATUS of **Waiting for trigger**.

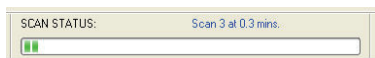


7. Select the Pyris method previously created and enter the sample details.
8. Press the **Start** button on the Pyris control panel.
While Pyris heats the sample to the experiment's starting temperature, it shows a status of **Go To Temp**.



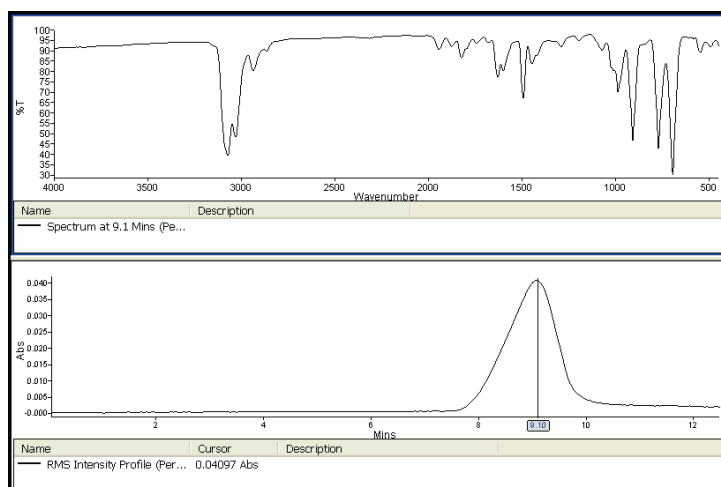
When the experiment starting temperature is reached, the Pyris status changes to show the first step in the method.

Pyris triggers data acquisition in Spectrum TimeBase, and the SCAN STATUS changes to show scans taking place.

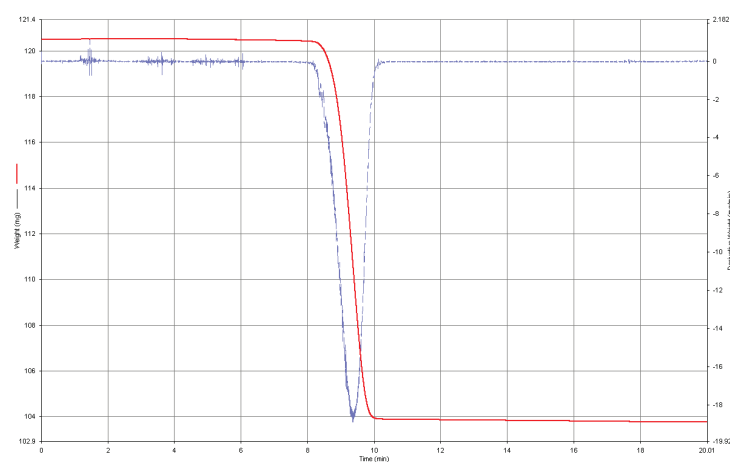


9. The data acquisition in TurboMass is also triggered by the Pyris software. The software displays **Waiting for injection** and then **Acquiring**.

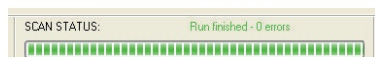
Experimental data can be viewed in Spectrum TimeBase.



And also in Pyris.



10. Collection of data in Spectrum TimeBase continues until the end of its run time, and similarly in TurboMass.





Routine Maintenance

Thermal Instrument, FT-IR Spectrometer, GC-MS

For details of maintenance procedures for your thermal instrument, see the Pyris on-screen help.

For details of maintenance procedures for your FT-IR spectrometer, see the documentation supplied with the instrument.

For details of maintenance procedures for your GC-MS system, see the documentation supplied with the instruments.

Controller Module and Vacuum Pump

Cleaning

Clean the outside of the instruments using a damp cloth. If necessary, a mild detergent may be used. Before you clean an entire instrument, always perform a patch test on an inconspicuous area.

Avoid spilling liquid into the instruments. Clean all external spills immediately. If anything is spilled enters the main body of the instruments, switch off the power and contact a PerkinElmer Service Engineer.

Re-tuning the temperature controls

In the event that the temperature controllers fail to reach their operating temperature, or if the temperature fluctuates, carry out the following steps:

1. Switch off the controller module and allow the system to return to room temperature.
2. Switch the controller module back on.
3. Press and hold the button labeled U on the appropriate temperature control panel for at least one second.

The panel will briefly display the message **Tune**, and a red LED at the top right of the panel flashes until the retuning process has been completed.

<p>NOTE: Re-tuning of temperature controls must be carried out before the temperature of the unit reaches 80% of its set value. For example, if the gas flow cell is set to 200 °C, re-tuning must be carried out before the temperature reaches 160 °C.</p>

Filter maintenance

The flow gas pumped through the system is treated using a double-filtering system within the controller module (Figure 75).

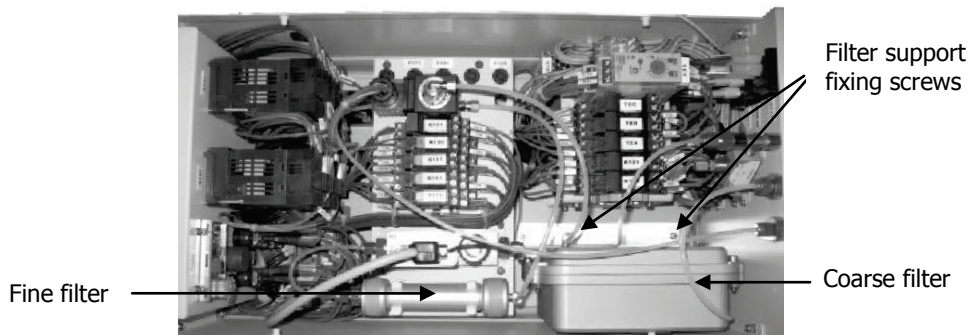


Figure 75 Controller module showing the double-filter system

The filters should be inspected once a year (or more frequently, depending on the number and type of samples analyzed) and replaced if necessary. Follow the steps below.

NOTE: We recommend that filter maintenance be carried out by trained PerkinElmer service engineers only, using parts supplied by PerkinElmer.

1. Ensure that the controller module is switched off and unplugged from the mains supply.
2. Loosen the four screws securing the top cover of the controller module and remove it. Locate the coarse filter (Figure 75).
To gain working space, you might find it useful to remove the 1/8" PTFE tubing on the upper side of the filter box and the 4 mm PTFE tubing at the filter inlet.
3. Remove the two screws on the bottom of the coarse filter support and extract the filter box.
4. Remove the four screws securing the top cover of the filter box and inspect or replace the internal filter.

5. If replacing the coarse filter element (P/N L8201804), fit the new filter with the side windows turned towards the gas inlet at the back of the instrument (Figure 76).

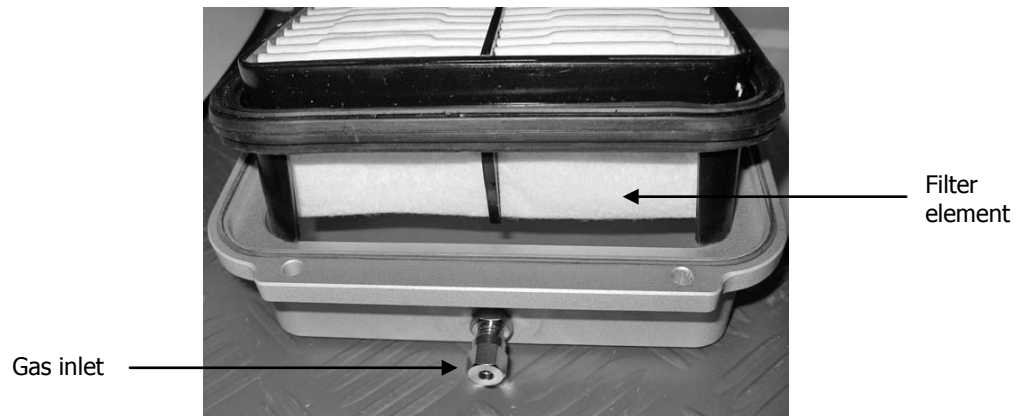


Figure 76 Coarse filter

NOTE: The fine filter is a disposable item and should be replaced annually, or more frequently if appropriate (P/N L8201810).

6. Replace the top cover and the 4 securing screws on the filter box, and secure within the controller module using the two screws.
Take care when replacing the cover to ensure that you do not twist or distort the O-rings on the lid of the filter box.
7. Replace the top cover of the controller module and secure the screws.

Gas Transfer Line

Volatile components evolved from a sample during an experiment may condense on the end of the gas transfer line that is connected to your thermal instrument.

It is good practice to clean the gas transfer line end-piece daily by immersing it in a beaker containing a suitable solvent, such as acetone or isopropyl alcohol. An ultrasonic bath can be used if necessary.



WARNING

Ensure that the gas transfer line has cooled to room temperature before carrying out any maintenance activities.

Preparing to clean the STA 6000 / TGA 4000 gas transfer line

- Before cleaning, unscrew the end-piece as shown in Figure 77.



Figure 77 Gas transfer line – unscrewing the end-piece

CAUTION

When refitting the end-piece, take care to screw it onto the end of the gas transfer line until it touches the flat base.

Failure to tighten the end-piece sufficiently will result in severe damage to the furnace of your thermal instrument when the gas transfer line is fixed to the sample well.

Preparing to clean the Pyris 1 TGA gas transfer line

- Disconnect the gas transfer line from your instrument, as described in *Removing the gas transfer line* on page 70.

Checking the end-piece O-ring

- After cleaning the gas transfer line, check the condition of the O-ring (Figure 78) as this may need to be replaced periodically (P/N L8201881).

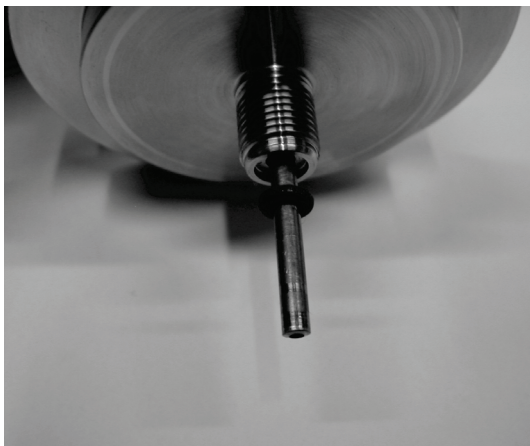


Figure 78 Gas transfer line – end-piece O-ring

Gas Flow Cell



WARNING

Ensure that the gas flow cell has cooled to room temperature before carrying out any maintenance activities.

Replacing the cell discharge tube

Periodically you will need to replace the tube that handles material discharged from the gas flow cell (P/N L8201825). The frequency will depend upon the samples you work with and the nature of the experiments you carry out.

1. Remove the gas flow cell from the spectrometer.
See *TL 9000 Gas Flow Cell Installation* on page 37 for details.
2. Locate the gas discharge tube at the bottom of the left hand side of the cell.
3. Loosen and remove the nut securing the discharge tube, using a 1/2" (12 mm) spanner.
4. Remove the tube and clean inside it.
5. Replace and secure the discharge tube (Figure 79).

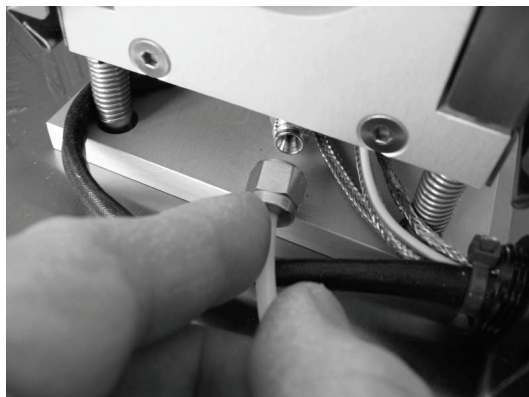


Figure 79 Replacing the gas cell discharge tube

6. Return the gas flow cell to the spectrometer.
See *TL 9000 Gas Flow Cell Installation* on page 37 for details.

NOTE: Once you have returned the gas flow cell, you should re-align the spectrometer beam to maximize the energy passing through the sample. See *FT-IR spectrometer setup* on page 78 for details.

Cleaning and replacing gas flow cell windows and gaskets

If the IR energy passing through to the detectors decreases significantly, it may be necessary to clean the gas flow cell windows. If they become fogged or are cracked, they may need to be replaced.

In addition, the KBr windows supplied with the cell can be changed. Other window types, for example ZnSe, provide different usable wavelength ranges.

CAUTION

If you change the windows in the gas flow cell from the standard KBr type (P/N L8201801) to ZnSe (P/N L1270766), the temperature setting in the cell should not exceed 250 °C. See Controller module setup on page 80 for details of how to set the temperature.

1. Remove the gas flow cell from the spectrometer and remove the window cap.
See *TL 9000 Gas Flow Cell Installation* on page 37 for details.
2. Remove the window and clean it with an organic solvent or replace it with a new one.
3. Check the condition of the O-ring gasket.
High temperature gaskets can be cleaned with an organic solvent or replaced, if required (P/N L8201818).
4. Return the window to the cell and secure the window cap.
5. Repeat steps 2 to 4 for the other window.
6. Return the gas flow cell to the spectrometer.
See *TL 9000 Gas Flow Cell Installation* on page 37 for details.

NOTE: Once you have returned the gas flow cell, you should re-align the spectrometer beam to maximize the energy passing through the sample. See *FT-IR spectrometer setup* on page 78 for details.

SeaLINK Relay Box

There are no specific routine maintenance procedures associated with the SeaLINK relay box.

Replacement Parts List

Gas cell

Part Number	Description
L8201801	KBr cell window (2 pack)
L1270766	ZnSe cell window (2 pack)
L8201825	Gas cell discharge tube
L8201818	Gas cell gasket (4 pack)

Controller module

Part Number	Description
L8201804	Controller module coarse filter element
L8201810	Controller module fine filter
L8200801	Vacuum pump (24 VDC)

Gas transfer line

STA 6000 / TGA 4000

Part Number	Description
L8201881	Gas transfer line end-piece O-ring (4 pack)
L8201878	Clamp lower O-ring
L8201877	Clamp upper O-ring

Pyris 1 TGA

Part Number	Description
L8201881	Gas transfer line end-piece O-ring (4 pack)
L8202022	Gas transfer line securing nut
09902131	Furnace replacement O-ring

GSV Assembly

Part Number	Description
09903392	1/16" column nut (injector connection)
09920127	1/16" ferrule (injector connection)
L8201956	1/16" short nut (connection to valves)
L8201957	0.25 GSV ferrule (connection to valves)
L8201953	1/16" low volume connector (connection to valves)
L8201950	1/8" PTFE tubing (loop load tube)
L8201825	4 mm PTFE tubing (exhaust tube)
L8201954	GSV replacement tubing kit (1/16" silicone-coated stainless steel)
N9301355	Deactivated column, ID 0.1 mm (restrictor for on-line mode)
N9301356	Deactivated column, ID 0.25 mm (restrictor to MS gas transfer line)

Decontamination

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

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

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

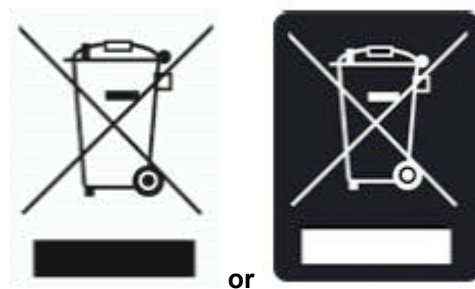
<http://las.perkinelmer.com/OneSource/decontamination.htm>

Alternatively, if you do not have access to the internet contact Customer Care:

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

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

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, and in accordance with the regulatory guidelines in your area.

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

The requirements for waste collection, reuse, recycling, and recovery programs are set by the regulatory authority in your location. Contact your local responsible person (such as your laboratory manager) or authorized representative for information regarding applicable disposal regulations.

See the PerkinElmer web address below for information specific to PerkinElmer products, and contact details for the Customer Care department in your region.

<http://las.perkinelmer.com/OneSource/Environmental-directives.htm>

Products from other manufacturers may also form a part of your PerkinElmer system. These other manufacturers are directly responsible for the collection and processing of their own waste products under the terms of the WEEE Directive. Please contact these manufacturers directly before discarding any of their products. Consult the PerkinElmer web address (above) for manufacturer's names and web sites.

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