## OPERATOR'S MANUAL FOR PL-GPC 120

## Integrated GPC/SEC System

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# CE

### **DECLARATION OF CONFORMITY**

We,	Varian, Inc.		
	Essex Road		
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	UK		

declare that the product:

High Temperature Chromatography System				
PL-GPC 120 Part # PL0830-0000				

conforms with the requirements of EC Directives 89/392, 91/368 & 89/336 by complying with the following Harmonised European Standards:

Safety:	EN61010 - 1	Class I
		Installation category II
		Pollution degree 2
	EN61010 - 2 - 010	Class 2
EMC:	EN55022	Conducted emissions: Class B
		Radiated emissions: Class B*
	EN50082	ESD (C), Radiated immunity (A),
		Fast Transient Burst (B), Surge

immunity (B), Conducted immunity (A).

SOM.

Dr. S.O'Donohue Head Of Instrumentation, Church Stretton 9<sup>th</sup> June 2010

## **PL-GPC 120 WARRANTY**

(Extract from General Conditions of Sale)

Subject as hereinafter stated, if any goods supplied are proved to the reasonable satisfaction of the Seller to be defective in material or workmanship within a period of 12 months from the date of despatch and the Buyer notifies such defect to the Seller in writing within fourteen days of it becoming apparent the Seller shall repair or replace at its option the goods or any part thereof free of charge and any repaired (or replacement) goods will be guaranteed on these terms for the unexpired portion of the 12 month period **PROVIDED THAT** the Seller shall be under no liability in respect of any defect that has arisen because:-

- a. of fair wear and tear; or
- b. where the goods have not been used, maintained, stored or protected in the proper manner; or
- c. the goods have been altered in any way whatsoever or have been subject to unauthorised repair; or
- d. the goods have been improperly installed or connected (unless the Seller carried out such installation and connection); or
- e. in the case of Instrument Consumables (including wetted parts, lamps etc) they prove defective as aforesaid more than 30 days after delivery notwithstanding the foregoing provisions of this condition; or

f. the Buyer is in breach of any other contract made with the Seller such as the Company's General Conditions of Sale.

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## HEALTH AND SAFETY

#### **PL-GPC 120**

This instrument should be used only in accordance with the instructions stated within this manual. Users should observe the following general safety precautions:

- 1. Ensure that the instructions within this manual are understood and carried out in the operation of the detector. All persons utilizing the instrument should have adequate training in its proper set-up, operation, and particularly its safety features.
- 2. Voltages above 110V AC are present within the instrument; access covers should not be removed by anyone other than properly trained personnel. No attempt should be made to service the instrument without authorization from Varian's service department and contravention of this may result in personal hazard or damage to the instrument and will invalidate the manufacturer's warranty.
- 3. We stress the importance of standard laboratory safe practice (e.g. COSHH regulations) for dealing with electronic laboratory equipment, solvents, etc., in preventing accidents, fires, or potentially hazardous conditions.

If in any doubt about the use of the instrument contact your local Varian office or distributor.

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## SAFETY

The following general safety precautions must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the instrument. Varian, Inc. assumes no liability for the customer's failure to comply with these requirements.

#### Safety Symbols on the PL-GPC 120

The PL-GPC 120 contains the following safety symbols:



This label is affixed to the inner oven lid, which in normal operation may reach temperatures as high as 120 °C. When accessing the oven please ensure you are wearing appropriate gloves before touching this lid.

#### Signs and Pictograms Used in this Manual



#### WARNING:

The "WARNING sign" denotes a hazard. It calls attention to a procedure, practice which, if not correctly done or adhered to, could result in severe injury or damage or destruction of the instrument. Please do not proceed beyond a WARNING sign until the indicated conditions are fully understood and met.



#### **ATTENTION:**

The "ATTENTION sign" denotes relevant information. Read this information first before proceeding, it will be helpful or necessary to complete the task.



#### NOTE:

The "NOTE sign" denotes additional information.

It provides the user with advice and suggestions to facilitate the operation of the instrument.

#### Safety Practices

The following safety practices are intended to insure the safe operation of the equipment.

This is a safety class I instrument (requires protective earthing) and has been manufactured and tested according to international safety standards.



Electrical Hazards

Before applying power, comply <u>fully</u> with the installation instructions. Additionally the following must be observed:

Do not remove instrument covers when operating. Before the instrument is switched on, all protective earth terminals, extension cords, auto-transformers, and devices connected to it must be connected to a protective earth via a ground socket. Any interruption of the protective earth grounding will cause a potential shock hazard that could result in serious personal injury. Whenever it is likely that the protection has been impaired, the instrument must be made inoperative and be secured against any intended operation.

Removal of instrument panels may expose potentially dangerous voltages. Disconnect the instrument from all power sources before removing protective panels. Access to the interior of the instrument should be restricted to trained and qualified service personnel only.

Replace defective fuses only with size and rating stipulated on the rear panel next to the fuse holder and in the manual.

Replace faulty or frayed power cords.

Check the actual line voltage to confirm its value, before connecting this instrument to it.



The individual or group responsible for the use and maintenance of this equipment must ensure that appropriate decontamination is carried out if hazardous material is spilt on or inside the instrument. Before using any cleaning or decontamination method except those recommended by Varian, users should check with Varian that the proposed method will not damage the equipment.



Hot Surface Hazards

Hot surfaces may be exposed when the instrument is opened. These are indicated by a warning label. Allow these surfaces to cool before touching them.



**General Precautions** 

Perform periodic leak checks on supply lines.

Test the overtemperature protection systems for the oven on a regular basis (see the Cleaning & Maintenance section of this manual).

Do not allow flammable and/or toxic solvents to accumulate.

Follow recommended procedures and protocols for evacuation and disposal of flammable and/or toxic solvents. Never dispose of such products through municipal waste systems

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## **1** General Information

#### 1.1 Introduction

Gel Permeation Chromatography/Size Exclusion Chromatography (GPC/SEC) is the technique of choice for rapid and reliable characterization of polymer molecular weight and molecular weight distribution. Advances in polymer science and technology mean that a more diverse range of solvents and temperatures are required in the modern GPC laboratory, and with ever increasing workloads, the GPC/SEC system must deliver greater accuracy, reproducibility and reliability with automated high sample throughput.

Varian's PL-GPC 120 system specifically for GPC/SEC analyses between 30-120 °C, where elevated temperature is required to reduce the eluent viscosity rather than to dissolve the sample. Offering either manual injection as standard or an external autosampler option, the PL-GPC 120 gives improved resolution and reproducibility compared to conventional modular GPC systems.



Mark II- extended end panel for installation of a Viscometer or Light scattering system

#### 1.2 Specifications

Pump	Flow Range Flow rate reproducibility *	0.1-10.0 mL/min ≤0.07%
Oven	Temperature Range Temperature Stability Column Capacity	30-120 °C <0.05 °C/hour 6 x 30 cm columns
Detector	Differential refractometer Cell Volume Sensitivity * Noise * Drift * Wavelength	Deflection 8 μL 2 x 10 <sup>-7</sup> RIU/mV ≤80 μV (1.6X10 <sup>-8</sup> RIU) ≤10 mV/hour (2.0 x10 <sup>-</sup> <sup>6</sup> RIU/hour) 890 ± 50nm
Sample Injection	High temperature motorized valve with syringe loading	Flushed fixed loop; 5-5000 μL
Autosampler	Optional	
Instrument Control Instrument Interface		Microprocessor Windows® 95 or NT based Advanced PC control
General	Power requirements	210-250V AC 50/60 Hz, 10 A max
	Size (wxdxh) Packaged Size (wxdxh) Weight	810 x 540 x 580 mm 965 x 665 x 820 mm 130 kg; 286 lbs

\*These technical specifications have been determined by experimentation and reflect the total system performance. The values quoted have been calculated from analyses performed in THF at 40 °C.

**Safety:** The instrument is designed and built to comply with the current European electrical safety regulations (EN 61010, Parts I and II), and are certified to be EMC (EN 55022) compatible. The PL-GPC 120 is CE marked.

#### 1.3 Unpacking and Installation

Care has been taken to ensure that the instrument should be received in proper condition. The packing and protection are designed for normal hazards of road, rail or air transit. Any damage to the container or instrument should be reported immediately to your local distributor, or to Varian. It is recommended that the shipping container be kept, if possible, for re-shipment or return to a service centre.

Examine the shipping carton for visible signs of exterior damage. Unpack the instrument and examine for transit damage. **CAUTION – this instrument is very heavy. See Section 1.5.2.6 for safety instructions.** Check that all items on the packing list are included.

Notify your local distributor or Varian of any damage or missing items.

Item Number	Part Number	Description	Quantity
1		Instruction Manual	1
2		Control Software	1
Pump Accessories			
3		Pump syringe adapter (HP)	1
4		Syringe 10 mL (glass)	1
Cable Set			
5	4/097401 & 4/09740 EUR 90	Power Leads 230V 10A IEC	2
6		RI ouput cable	1
7		RI Polarity Invertor Cable	1
8		Serial I/O Cable	1
9		Remote DCU Start Lead	1
10		Pump 2 Null Plug	fitted
Chromatography			
11	PL0402-0100	Injection loop 100 µL	1
12	PL0402-0200	Injection loop 200 µL	fitted
13	PL0127-005	Intercolumn Connectors (10 cm)	3
General			
14	4/32620	Pot adjustment tool	1
15	4/32233/10A	Fuse 10A T - Main Unit	2
16		Fuses ( Degasser)	2
17	4/32640	Allen Wrench Set - Metric	1
18	4/32641	Allen Wrench Set - Imperial	1
19	4/32567	Exhaust Ducting (4")	3m
20	4/32686	Adapter & Clamping Ring (4")	1
21		1/16" x 1/16" Union	1
22		Parker Nuts & Ferrules	6
23		M4x12 Skt Head Cap Screws & Washers	8

#### Packing List

#### 1.4 Site Preparation Check List

Environmental Conditions



Temperature 15 to 35 °C (59 to 86 °F) At constant temperature

Avoid positioning in direct sunlight

Humidity 40-80%

Power



Bench Space



#### Extraction Requirements



230V (AC)  $\pm$ 10%, 15A, 50/60 Hz, Single phase with earth.

For countries where 230V will have to be installed the main power lead is provided with NEMA-15 type plug.

Ensure that the mains supply is fitted with a residual current circuit breaker.

The standard instrument will require the following bench space: Width 81 cm (55 in.), Depth 56 cm (22 in.), Height 60 cm (23 in.).

Note: <u>The oven lid open upwards to a full height of</u> <u>97 cm (38 in.). Please be aware of overhead</u> <u>obstructions.</u>

If a PL-LS 15/90, Viscotek 220R or PL-BV 400 is included in the instrument allow an additional 30 cm (12 in.) for each of these detector control systems on the width of the bench space.

The instrument is heavy weighing 130 kgs (286 lbs.) and thus it is advisable to locate the system on a sturdy bench, preferably fitted with wheels facilitating access to the rear of the unit should service be required.

The instrument is equipped with a fan-forced exhausts (4 in. dia) at the rear, which must be routed to a fume extraction system or vented into a hood via the supplied hoses. The vent hose for the instrument are 4 in. in diameter. The air flow of the extraction system should be <1SLM.

Situate the instrument in a well-ventilated area.

#### 1.5 Unpacking Instructions

#### **Tools required: -**

Phillips screwdriver (medium) Flat screwdriver (medium)

#### Unpacking

The instrument is shipped on a specialized pallet inside a wooden carton. The accessories are packed in and around the instrument. Unpack as follows: -

- 1. Remove the screws (Phillips screws, typically 3 per side) around the top edge of the carton, securing the lid in position. Lift off the lid.
- 2. Beneath the lid is a sheet of foam packaging protecting the top of the unit. Lift and remove this to reveal the top of the unit.
- 3. Five sections of foam packaging are found surrounding the unit; one at each side, one piece at the front and two small sections behind the unit. The wires are tucked down between the two foam sections at the back. Remove all of these. The packaging is design to be a tight fit around the unit, and it is sometimes easier to remove the screws described in section 4 before lifting this packaging clear.
- 4. Remove the screws (Phillips screws, typically 3 per side) from around the base of the carton to enable the collapsible wooden sleeve, forming the side walls of the carton, to be lifted over and clear of the unit.
- 5. The instrument will now be visible on its sub-pallet The unit is firmly held down to the pallet by plastic bands with tie the instrument down to the sub-platform above the main pallet. Cutting these bands will enable the unit to be lifted free from the pallet.
- 6. The unit can now be lifted clear of the pallet. **CAUTION:** It is recommended that no less than four persons lift the PL-GPC 120. Please refer to local statutory Manual Lifting Regulations & guidelines.

#### 1.6 Drying-out Period

After positioning the instrument on the laboratory bench it should be left to stand in a warm, dry, ventilated environment with the outer and inner oven lids and front flap opened until any condensation which may have formed during shipping has evaporated. A period of one hour should be sufficient. **The electrical safety of the** 

**instrument cannot be guaranteed until it has dried out**. Do not connect any electrical power until this period has expired.

#### 1.7 <u>Removal of the Transit Bolts</u>

The pump drive mechanism is bolted to the pump chassis with two transit bolts. The pump can be run with the transit bolts in place but the pump vibration will be translated into the system if they are not removed. To remove the transit bolts, remove the pump from the oven module and then remove the two socket head bolts from the base panel of the pump using a 4 mm Allen wrench. Store the transit bolts for future use and replace the pump into the oven module.

#### 1.8 Connections



Figure 1.1 Rear View of PL-GPC 120 Control Drawer- Mark I

- 1. Live and Neutral Fuses
- 2. Mains input
- 3. Power Link power to oven assembly
- 4. Digital Link microprocessor control of oven assembly
- 5. Inj Sync Injection synchronization to data acquisition system
- 6. RI Link RI feedback to control system
- 7. Serial RS232 connector 9 pin D type female
- 8. Alarm sounder
- 9. RI Detector outputs; 1V
- 10. Solvent module input
- 11. PUMP 1 pump control



Figure 1.2 Rear View of PL-GPC 120 Control Drawer- Mark II



- 1. Live and Neutral Fuses
- 2. POWER mains input
- 3. POWER LINK power to oven assembly
- 4. DIGITAL LINK control link to oven assembly
- 5. INJ SYNC injection synchronization to data acquisition system
- 6. RI LINK RI feedback to oven module
- 7. SERIAL I/O RS232 connector, 9 pin D type female
- 8. Alarm sounder
- 9. RI OUT RI detector analogue output, ±1V FS
- 10. SOLV CTRL solvent module control link
- 11. PUMP pump serial control
- 12. AUTOSAMPLER not used on PL-GPC 220
- 13. AUX INPUT  $1 \pm 1V$  additional analog input for graphical log
- 14. AUX INPUT 2 ±5V additional analog input for graphical log
- 15. Service Mode Switch service use only. Default to 'NORMAL' position
- 16. Debug Mode Switch service use only. Default to 'DISABLE' position
- 17. 'A' & 'B' service use only.

#### **Power Connections**



Ensure that the 'Power Link' (Item 3), the 'Digital Link' (Item 4) and the 'RI Link' (Item 6) are connected before plugging any power cables into the instrument.

- Before plugging in the power cable, ensure the power supply is matched to the instrument rating plate.
- Use only a supply with protective grounding.
- The correct fuses should be installed. Main module 230V (AC) ±10%, use two 1.25" 10A T 250V HBC fuses



RISK OF FIRE, REPLACE FUSES AS MARKED!

• If the voltage rating and fuses are correct plug in the power cable.

#### Extraction

Ensure the 4 in. diameter extraction hoses are securely fitted to the rear of the instrument and the outlets suitably extracted



The exhaust must be extracted to a suitable fume extraction system

#### **Communication Connections**

The PL-GPC 120 is controlled using a Windows® based computer interface, which must be installed on a suitable computer before the instrument can be operated.

#### **Computer Specification**



Pentium 4 operating at 2GHz 256MB of memory 30 MB of free hard disk space SVGA (800 x 600) resolution monitor and video adapter (Set to High Colour) Microsoft Windows 2000 or XP Professional operating system Microsoft compatible mouse One USB serial communication port per instrument.

#### 1.9 Installation Procedures- Software

#### Installation of the PL Instrument Control software from CD

Place the CD-ROM containing the PL Instrument Control software into the CD drive. In most cases the CD browser window will automatically open. However if the window does not appear then select the *Run* option in the *Start menu*, and type in **D:\launch.exe** (where D: denotes the CD drive).

From the CD browser window select the *Install the Software* option and follow the onscreen instructions, it is recommended that the default settings are selected.



Ensure that when you log on to the PC you have full administration rights.

You may need to restart your computer at the end of installation; if this is required you will be prompted to do so.

After successful installation of the PL Instrument Control software, the program is simply run by clicking on the application named PL Instrument Control (PLInstControl.exe) installed in the PL Instrument Control group of the Programs option in the Start menu.

The default location for the program files will be C:\Program Files\Polymer Laboratories\PL Instrument Control, which contains the following files: -

	🛅 Docs	📹 offline.ico	d online.ico	🔁 PLInstConfigEd.exe
	🔊 PLInstControl.dll	d PLInstControl.exe	🔊 PLLog.dll	
PL Instrument Control				
Select an item to view its description.	_			
See also:				
My Documents				
My Network Places				
My Computer				



Before running the program the software must be first configured, see Section 0.

#### Installation of the PL Instrument Control On-line Help from CD

From the CD browser window select the *Install Online Help* option and follow the onscreen instructions, it is recommended that the default settings are selected. The two help files (one for the PL Instrument Control software and the other for the PL Instrument Configuration Editor program) will be installed and the default location will be *C:\Program Files\Polymer Laboratories\PL Instrument Control\Docs*.

#### Installation of the USB 4-port Serial Adapter Software

It is often more convenient to control your PL-GPC 120, autosampler, DataStream and any other device via a USB to serial adapter, reducing the connection to the computer to one USB cable. The recommended USB to serial adapter is a Keyspan system and this section describes the procedure necessary to install the drives of this device on your PC.

Before connecting the PL-GPC 120 to the computer and starting the PL Instrument Control software the Keyspan USB 4-port Serial Adapter software needs to be installed. From the CD browser window select the *Install USB Drivers* option and follow the on-screen instructions, it is recommended that the default settings are selected.

Once the software installation has completed, connect the serial cables from the PL-GPC 120 system to the 4 port device and using the USB cable supplied connect the device to the computer. Once the device is plugged into the computer the hardware should be recognised and the USB automatically installed.



#### Configuring the PL Instrument Control Software



The Instrument Control Software needs to be closed down before starting this program.

The configuration of the instrument should normally be done by Varian-trained, Varian-qualified, or Varian-approved Customer Service Representatives.

During this process the serial ports of the USB to serial adapter are mapped to the controlled modules of the PL-GPC 120 and additional controlled devices.

To configure the PL Instrument Control software for the PL-GPC 120 system, you need to run the PL Instrument Configuration Editor to define the components that make up the instrument. The PL Instrument Configuration Editor is simply run by clicking on the application named PL Instrument Configuration Editor (PLInstConfigEd.exe) installed in the Service group of the PL Instrument Control group of the Programs option in the Start menu.





The first time the PL Configuration Editor program is run, the program displays **all** the components that are available for control.

For the initial configuration of the system select the required components from the list by left clicking on the name of the component so that the configuration matches the instrument.

If the configuration of the instrument is changed then add/delete the relevant components in the PL Configuration Editor so that the new configuration is matched.

To delete a component from the existing configuration then highlight the component in the list and select *Delete Component* from either the Configure menu or toolbar icon.

To add a component into the existing configuration then select *Add Component* from either the Configure menu or toolbar icon. The Add Component dialog will open, where the required component can be selected and added to the configuration.



Only one component can be deleted at a time. Multiple components can be added at a time.

Once the component configuration has been completed the Com ports need to be assigned correctly to each component. To set the Com ports double click on the

component name. The Configure Component dialog will open, where the correct Com port can be entered.

Configure Component	×
7 Com Port 🔽 Simulation Mode	Cancel
Chosen Name Pump	

The correct Com ports are according to the assigned addresses displayed in the Keyspan Serial Assistant software see section 0 for details.

**NOTE:** The *Simulation Mode* box needs to be unchecked in order to communicate to the component. Also can rename each component by typing in the desired name in the *Chosen Name* box.

Once all the Com ports are set correctly, close the configuration editor and wait while the new configuration is saved. While the new configuration is being saved the following message will be displayed: -





Updating the instrument configuration can take up to a minute so please be patient.

For further information on configuring a system then please see the on-line help within the PL Configuration Editor program.

#### Configuring the Control Software – PL-GPC120 + PL-AS RT

In the "Add Components" dialog click on the individual components that make up the system. The components that are required for the PL-GPC 120 with PL-AS RT are shown in the list below: -

🖷 PL Instrument Configuration Editor 📃 🗖 🔀				
<u>File Configure Tools View H</u> elp				
_ <b>1</b> : 중 1님 💡				
Component	Com Port	Simulate Mode		
PL-GPC Instrument     PL-ASRT [Autosampler]     PL-ASRT [Autosampler]     PH Injection Valve [Injection-Valve]     Pump     International Information Information Information     Pump     International Information     Pump     International Information     Pump     International Information     Pump     Internation     Pump     Pump     Internation     Pump     Internatin     Pump     Pump     Pump     Pump     Pu	4 5 5 5	Yes No No No		
•				
Ready				

Set the com ports in accordance to the com port mapping obtained from the Keyspan serial assistance.

#### Configuring the Connection Ports – PL-GPC 120 + PL-AS MT

E PL Instrument Configuration Editor				
Eile <u>C</u> onfigure <u>T</u> ools <u>V</u> iew <u>H</u> elp				
1 a a a 9				
Component	Com Port	Simulate Mode		
PL-GPC 120 + PL-AS MT     PL-AS MT [Autosampler]     PL-AS MT [Autosampler]     PL-AS MT Heated Block [Autosampler-Bloc     HT Injection Valve [Injection-Valve]     Pump     T100 Isocratic [Pump]     Oven     T20-0ven [Oven]     Detectors     HT-RI 120 [HT RI 120]	6 6 17 17 17 17	Yes Yes Yes Yes Yes Yes	1	
Ready				

To identify the Com ports that the computer has assigned to the 4 serial ports IDs, open the Keyspan Serial Assistant from the start menu;  $Programs \rightarrow$ , Keyspan USB 4-port Adapter  $\rightarrow$  Keyspan Serial Assistant. The dialogue as shown below will be displayed.

🗇 Keyspan USB 4-Port Serial Adapter Assistant	_ 🗆 X
Adapter Status Properties Port Mapping Diagnostics Help	
⊡ 🥔 USA49W-1	
COM3: Idle	
COM 4: Idle	

#### Serial RS232 Connector

The serial cable should be connected between the I/O socket at the rear of the PL-GPC 120 main module and COM 1 to 4 of the computer. A standard RS232 (DTE-DCE) 3-wire serial cable is provided in the accessory kit.

#### Fluid Connection

In general Valco fittings are used throughout the instrument, with the exceptions at the pump, the eluent in-line filter and the columns. During routine operation the fittings around the columns will be typically those that the user would come into contact with.



Valco fittings are mainly used throughout, but please consult your local Varian representative if unsure

#### 1.10 Precautions

#### **High Temperature**

The PL-GPC 120 is designed to control the temperature of the column oven and autosampler at temperatures up to 120 °C therefore the surface of the inner oven will get hot during normal operation. Please be careful and wear suitable protective gloves when accessing the column oven. Warning labels are affixed were appropriate for your protection



**BEWARE HOT SURFACES** 

#### **Solvent Vapors**

Vapor sensors are used in both modules of the PL-GPC 120 to alert the operator to solvent leaks. Liberal use of organic solvents in close proximity to the instrument may activate these vapor sensors, causing the instrument to shutdown.



*Please exercise care when using solvents close to the instrument: vapor sensors are present in the modules* 

#### Electrical

As part of the electrical safety regulations, forming part of the overall CE compliance, the unit is fitted with a surge safe device, located at the rear of the control drawer inside the instrument. This device protects the instrument from high voltage line surges by filtering the incoming supply. This device will cause the instrument to fail the insulation resistance test of the standard portable appliance test (P.A.T Test) and therefore should be unplugged before the P.A.T test is performed.



## 2 System Description and General Operation

#### 2.1 Instrument Overview



Figure 2.1 PL-GPC 120 Overview

- 1. Column Oven
- 2. Agilent 1100 series isocratic pump
- 3. Solvent Degasser
- 4. Control Drawer
- Optional Autosampler (PL-AS or Midas<sup>™</sup>)
- 6. Injection ports
- 7. Power Indicator
- 8. Temperature and RI Display and Status indicators
- 9. Waste ports
- 10. Control Software

#### 2.2 Injection System

The instrument is equipped with 3 injection lines; 2 are 0.02 in. ID tubing and 1 is 0.01 in. ID tubing. All three lines are temperature controlled to the oven temperature so that during sample injection the sample is pre-heated to the oven temperature.

Only one of the three injection lines can used at any one time, so please ensure the correct port to which the injection port is screwed into is connected to the valve. The tubes are labeled 1 through 3 in accordance with the injection port positions on the front of the instrument.

The 0.02 in. ID tubing is recommended for manual injections whereas the 0.01 in. ID tubing should be used if the autosampler feed is connected via the injection ports.

#### 2.3 <u>Column Oven</u>



Figure 2.3 Standard Oven Configuration

- 1. Differential refractive index detector
- 2. Injection Valve
- 3. Vapor Sensor
- 4. Column mounts
- 5. Temperature sensors
- 6. Oven fan with heaters

The low split line of the oven allows easy access to oven facilitating column exchange and routine maintenance. The temperature within the oven is controlled by eight active heaters; 3 are built into the base of the oven and 4 in the oven lid, the eighth heater is mounted directly in front of the fan and it is this heater that provides the fine control. Figure 2.4 below illustrates the location of the heaters (red lines) in the oven construction and the airflow around the heaters (black arrows).



Figure 2.4 PL-GPC 120 Oven Construction

#### 2.4 Optional Autosampler

The PL-GPC 120 is a manual injection system, however it is can be used with a PL-AS RT/MT or a Spark Holland Midas<sup>™</sup> autosampler. When an autosampler is used the injection needle of the autosampler is connected directly to the PL-GPC 120 injection valve via injection port A with the return connected to injection port B or C. For details of the plumbing connections see the autosampler manual and or Appendix 2 of this manual.

## **3 The Graphical User Interface**

A Windows based Graphical User Interface (GUI) providing total instrument control is used to operate the PL-GPC 120. This intuitive interface provides simplistic control as well as a comprehensive monitoring system. The instrument status can be rapidly assessed for each of the instruments modules.



For further information on the PL Instrument Control software please see the on-line help within the program.

To start the control software select the PL Instrument Control item <sup>IIII</sup> in the PL Instrument Control program group of the Programs option in the Start menu.

#### 3.1 <u>Overview</u>

PL-GPC Instrument		<u>_ 8 ×</u>
Instrument Settings Samples Automation Tools Help		
🖃 🛱 Sample Handling	OVEN : Ready	?
	Current Parameter Settings	iPC50 ColumnOven-MkII )
	Parameter Set Value	<u>_</u>
Current Vial Position	Temperature (C) 40	
(ml) (ml) (ml)		
_		
Injection-Valve		
Current Position Inject		
	Update	_
E O Pump		
Flow Rate (ml/min) 1.00 (1.00)	Select Action	
Pressure (MPa) 5.00	- No user actions selectable -	
Temperature ( *C ) <u>40.00</u> (40.00)		
F 🕅 🖨 🖪		
	Component View	
Summary		
View		
$    v \iota e W  $		

The Control screen is effectively divided into two main Views, these are:

- 1. The Summary View
- 2. The **Component View**

#### 3.2 <u>Summary View</u>

The summary view displays all the components of your PL-GPC 120 instrument as a "Tree" providing you with status for each of the modules and a quick way of access to the various components, parameters and options available within the PL Instrument Control software.



Clicking on the component names displayed in red in the *Summary View* of the screen changes the control display shown in the *Component View*.

A green LED next to the instrument component indicates the module is being controlled and running whereas a red LED would indicate the module is either not running or not controlled.

For further information on the *Summary View* please see the on-line help within the PL Instrument Control software.

#### 3.3 <u>Component View</u>

The component view displays the component view of the selected component providing you access to parameters and/or direct commands for that specific component. In general each component view contains a number of common items and options. The actual items available in a view are dependent on the component selected.

The general component view for all components except for autosamplers/carousels is shown below.

COMPONENT :	Status Bar	On-	line Help Button	<b>,</b> ?
Current Parameter Settings		θ		(COMPONENT)
Parameter 9 Parameter 1 (units) Parameter 2 (units) Parameter 3 (units) Parameter n (units) Parameter n (units)	Set Value Value 1 Value 2 Value 3 <i>Imeter Grid</i> Value n	Help	Output Window	
Select Action ACTION 1 ACTION 1 ACTION 1 ACTION 1 ACTION 2 ACTION	Action 3 Action 4 Action	on List		

Status Bar – This displays information about the selected component and it's current status.

On-line Help Button – This is a direct link to the on-line help for that component view.

Parameter Grid – This displays the current set parameter(s) for the selected component. To set and update the parameter(s) enter the required value(s) in the

Set Value column and press the Update button, Update. For parameters such as flow rate and oven temperature pressing the Update button will automatically set and run the component to the new value.

To undo any parameter(s) prior to pressing the Update button press the Undo button,

n

To reset the parameter(s) back to the default value(s) press the Factory Reset button,

*Help Output Window* – This displays a simple summary help for each parameter and action. To view this information, select the parameter or action. The information displayed in the *Help Window* will be a short description about the action or parameter, the Factory Default Value and the Minimum and Maximum Values for the parameter.

Action List – This is a drop down list of any actions that are available for the selected component. To run an action, select the required one from the Action List and press

the Submit Action button,

Action Button(s) – If there are any actions available for the selected component then they are displayed as *Action Buttons*, as well as being found in the *Action List*. To run an action, press the required Action button.

Press the Stop button, we to stop all the procedures currently running on the selected component.

The general component view for autosampler/carousel components is shown below.



The order in which the sample vial positions are selected defines the injection sequence. This can be viewed in the *Injection Sequence Lists*. Vial positions can also be selected using the *Sample Grid*.

Sample Injection Sequence Lists – The Vial Sequence List displays the order in which the sample vials will be injected. The sample name and number of injections can be defined for each sample. The order can be re-prioritized by simply highlighting the relevant vial(s) in the list and using standard Windows drag and drop actions. Sample vial(s) can also be removed from the injection sequence list by simply highlighting the relevant vial(s) and pressing the delete key on the keyboard.



The *Injection Sequence List* displays the actual injection sequence. This is calculated from the order the vials are to be injected and the number of injections per vial. This grid is read only.

Autosampler Block/Carousel Settings – This allows a new sequence of samples based on a set of default parameters (which will be applied to all the samples) to be set. The details for individual samples can then be changed as required. Also the current injection sequence can be reset within this option. To open the autosampler

block/carousel settings press



Autosampler Parameters – This allows the autosampler/carousel parameters for sample injection to be set. To open the autosampler/carousel parameter settings

press 篷 🖄.

Action List – This is a drop down list of the actions that are available for the autosampler/carousel component. To run an action, select the required one from the

Action List and press the Submit Action button,

Action Button(s) – The actions available for the autosampler/carousel component are displayed as *Action Buttons*, as well as being found in the *Action List*. To run an action, press the required Action button.



*Toggle Button* – This toggles between the *Action Buttons* and the *Sample Grid*.

Sample Grid – This displays the sample details and can be used to select the sample vial positions for the injection sequence. The grid also shows the vial positions that

have been injected (in black), the current vial position being injected (in red) and vial positions to be injected (in blue). To select/deselect a sample vial left click in the active box for the relevant vial position. Sample details can be added to the selected vial positions.



Sample vials can be added and pending vials can be removed after the injection sequence has started.

Sample Vial Data and Status									
#Vial	Active	Name	#Injections	Conc. (mg/ml)	Method	Sample Type	к	Alpha	<b>_</b>
1		Sample-1	1	0.000	None	Unknown	14.100	0.700	
2	•	Sample-2	1	0.000	None	Unknown	14.100	0.700	
3		Sample-3	1	0.000	None	Unknown	14.100	0.700	
4	☑	Sample-4	1	0.000	None	Unknown	14.100	0.700	
5	☑	Sample-5	1	0.000	None	Unknown	14.100	0.700	
6	☑	Sample-6	1	0.000	None	Unknown	14.100	0.700	
7	☑	Sample-7	1	0.000	None	Unknown	14.100	0.700	
8	☑	Sample-8	1	0.000	None	Unknown	14.100	0.700	
9	☑	Sample-9	1	0.000	None	Unknown	14.100	0.700	
10	☑	Sample-10	1	0.000	None	Unknown	14.100	0.700	
11	☑	Sample-11	1	0.000	None	Unknown	14.100	0.700	
12	☑	Sample-12	1	0.000	None	Unknown	14.100	0.700	
13		Sample-13	1	0.000	None	Unknown	14.100	0.700	
14		Sample-14	1	0.000	None	Unknown	14.100	0.700	▼

The order in which the sample vial positions are selected defines the injection sequence. This can be viewed in the *Injection Sequence Lists*. Vial positions can also be selected using the *Autosampler Block/Carousel Graphic*.

For further information on individual *Component Views* please see the on-line help within the PL Instrument Control software.

For information on the Toolbar menus please see the on-line help within the PL Instrument Control software.
## 3.4 Event Schedule Editor

The PL Instrument Control software provides the ability to automate the control of the PL-GPC 50 Plus using the *Events Schedule Editor*. To open the Events Schedule Editor and create a schedule, select the *Edit Schedule* option from the *Automation* menu on the toolbar. The editor will initially display a default *Events Schedule* consisting of a single *Process* and *Sequence* entry. This ensures you only have to add a *Procedure* entry for the schedule to be valid.

💕 Events Schedule Editor				
File Tree Schedule Help				
Schedule Details				
Author		Comments		
Creation Date / Time : 02/06/2006 11:19:5	6			
		1		
Name	Active	Procedure Action	Status	Wait Delay
Process	>			
Start : 02/06/2006 11:19:56	>			
ے Sequence	✓			O ( secs )
/ Beadu \				
Cricady /				

To add a *Procedure* right click on the *Sequence* entry in the schedule, the *Procedure Details* window will open where the required action can be selected.

🚅 Procedure Details			×
Procedure Details	File Details	Other	οκ
⊢ Procedure Details			
Component Select Componen	t		Cancel

Once the *Events Schedule* is completed press the Start button, **L** to start the schedule.

Most of the standard activities available manually within the software are available as entries for inclusion in an *Events Schedule*, for example: -

- Setting a component to a set of specific parameters e.g. oven temperature or pump flow rate.
- Performing an action on a component e.g. purging a detector.
- Loading a file e.g. an instrument method or injection sequence from disk.

The schedule can be used to allow as much or as little automation of the system as desired by the user. As a simple example, the *Events Schedule Editor* can be used to define a schedule that automates the initial setup of a system: -

- 1. Set the pump flow rate.
- 2. Set the oven temperature.
- 3. Automatically wait for the desired conditions to be reached, or even longer if desired.
- 4. Purge the detectors.
- 5. Autozero the detectors.

Message boxes can be displayed at key stages during an *Event Schedule*. These message boxes require user input before the rest of the schedule can be run. The text displayed in the message box is customisable allowing simple status information or more detailed instructions to be displayed to the operator. Two examples of this are: -

- Providing a simple guide path to an operator for the manual operation of the system.
- Providing feedback / key information to an operator at key stages of a more automated system.

The *Event Schedule Editor* also provides the ability to perform automatic repeating of single or multiple *Procedures* without any user input. This allows more complex automated schedules to be used. For example a schedule can be defined that: -

- 1. Automates the initial setup of the system.
- 2. Automatically loads a predefined injection sequence of samples.
- 3. Automatically injects and collects the data for the sample sequence.
- 4. Automatically shuts down the instrument or sets the instrument to a set of idle conditions on completion of the sample sequence.

For further information on the Event Schedule Editor please see the on-line help within the PL Instrument Control software.

## 4 Instrument Set-Up

## 4.1 Powering the Instrument ON/OFF

The PL-GPC 120 is powered on and off using the main power rocker switch at the front of the control drawer. Switching this on provides power to all the components integrated in the system. The rocker switch and the power indicator are illuminated when the power is on.

## 4.2 Controlling the Instrument

To start the control software select the PL Instrument Control item with in the PL Instrument Control program group of the Programs option in the Start menu.

On start-up the software will automatically connect to the instrument and report the status of the individual components.



IF AN AUTOSAMPLER/CAROUSEL IS CONNECTED THEN THIS WILL BE INITIALIZED ON START-UP OF THE SOFTWARE THUS MOVING THE ARM.

The initial view of the control software will be typically as shown below.

💣 PL-GPC Instrument		- 7 🛛
Instrument Settings Samples Automation Tools Help		
🖃 🚔 Sample Handling	-E PUMP : Ready	?
Current Vial Position (Unset) 0 (ml) <b>Autosampler.Block</b> Temperature (°C) 40.00	Current Parameter Settings Parameter Set Value Flow Rate (minnin) 1.0 Min Pressure (MPa) 0.0 Max Pressure (MPa) 10.0 Flow Ramp 1.2	( 1100 Isocratic )
Current Position Initialised	Select Action - No user actions selectable -	
Flow Rate 0.00 (Stopped) (ml/min) Pressure (MPa) 0.00		 
Oven     Oven     Temperature (°C)     40.00		
M     M		

A green LED next to an instrument component indicates the module is being controlled and running whereas a red LED would indicate the module is either not

running or not controlled. Initially the pump will not be running, i.e. no flow and therefore a red LED is shown next to the pump component.

To start the pump, go to the pump component view, set up the flow and pressure parameters as required and press the Update button, Update. After a few seconds the pump will start running at the set flow rate and the LED will change from red to green.

For further information on controlling the system please see the on-line help within the PL Instrument Control software.

## Purging the Pump

Before running the pump, the pump heads need to be completely filled with solvent. To purge the pump heads with solvent ensure the pump purge valve is closed and remove the waste PTFE tubing from the purge outlet and replace it with the supplied pump purge adapter. Connect the 10 mL syringe to the outlet of the pump purge adapter. Open the pump purge valve by turning the knob 180° counter clockwise and draw ~10 mL of solvent from the reservoir through the pump head into the syringe. When removing the syringe from the pump purge adapter, close the purge valve to prevent solvent siphoning through the system and onto the drip tray.

Complete the purging process by replacing the pump purge adapter back to the purge waste tube and run the pump at 1 or 2 mL/min with the purge valve open until the lines from the solvent reservoir to the pump are free of air and full of the solvent.

Once the lines to and from the pump are free of air, adjust the flow rate to 1.0 mL/min and close the purge valve so as to pump the solvent through the remainder of the system.

It is recommended that initially the columns are bypassed and the system thoroughly flushed with solvent.

#### Purging the RI Reference Cell

To purge the RI cell, ensure the solvent is flowing through the system. Then from the HT RI 120 component view of the PL Instrument Control software either press the

Start Purge Action button, surreuse or select Start Purge from the Action List and press the Submit Action button,

The solvent flow is now diverted through the reference side of the RI cell before going to waste. The reference cell will be purged for 30 seconds, unless the Purge Time parameter has been set differently. After a purge has been completed the system defaults back to normal mode, i.e. solvent flow only through the sample side of the RI cell.





If additional detectors are connected then please refer to the detector's Operator's Manual for guidance on flushing/purging the detector into solvent.

## Running the PL-GPC 120

Once the system has been flushed through with the required eluent the column(s) can be connected. If the column(s) are not already in the solvent that is to be used as the eluent they need to be transferred as indicated by the manufacturer. This can be performed in the PL-GPC 120, but do not flush the column(s) through the detector(s), simply flush the column(s) directly to waste.

After flushing the column(s) into the required solvent, stop the pump and connect the column(s) to the detector(s). Set the pump to the required flow rate, typically 1 mL/min and check all connections for leaks.

Once the checks have been completed place the oven lid back on to the oven compartment and close the PL-GPC 120 door. Allow the system to flush for at least 1 column set volume before making the first injection.

If an oven temperature is required then, from the oven component view, set the

temperature value and press the Update button, Update. Adjust the oven temperature cut-out on the front panel of the instrument to 5 °C above the set oven temperature. Once the temperature has been reached allow 2 hours for the system to stabilise before making an injection.



THE OVEN TEMPERATURE SET POINT MUST BE AT LEAST 10 °C LOWER THAN THE SOLVENT BOILING POINT.

#### Making a Manual Injection

To make a manual injection ensure the Injection Valve component view of the PL Instrument Control software is visible. Switch the injection valve into the Load position by either pressing the Load Action button, or select Load from the Action List and press the Submit Action button,

Fill the supplied 1 mL injection syringe with 3 to 5 times the volume of the injection loop and displace any air from the syringe. Carefully insert the needle into the injection port and dispense the contents of the syringe into the injection loop.

Ensure that the data collection software you are using is ready to collect and the sample details have been entered into the run list.

Switch the injection valve into the Inject position by either pressing the Inject Action

button, or select INJECT from the Action List and press the Submit Action button.

The data acquisition system will be automatically started when the injection is made, if the supplied injection trigger cable is being used.

INJECTION-VALVE : Ready	?
- Select Action	( HT Injection Valve )
Select Action	
Load	
Inject	

# 5 Maintenance



SWITCH INSTRUMENT OFF AND DISCONNECT BOTH POWER CORDS BEFORE UNDERTAKING ANY CLEANING OR MAINTENANCE PROCEDURES. ALLOW ANY HOT PARTS TO COOL BEFORE PROCEEDING.

## 5.1 <u>Cleaning</u>

The exterior of the instrument should be cleaned by wiping down with a soft cloth moistened with dilute detergent solution, followed by wiping down with a cloth moistened with deionised water.

- Ensure that no moisture enters the instrument.
- Do not attempt to clean any connectors. Ingress of moisture could cause a safety hazard and accessible pins may become bent.
- Allow exterior of instrument to dry off <u>completely</u> before reconnecting power.

## 5.2 Decontamination

## NOTE: TO BE CARRIED OUT ONLY BY TRAINED SERVICE PERSONNEL.

Wear appropriate personal protective equipment for this operation (gloves, safety glasses, lab coat and respirator if the level of hazard has been risk-assessed to be sufficiently high).

Remove instrument covers and panels as necessary to ascertain the extent of contamination. Excess quantities of solvent spilt on or inside the instrument should be mopped up using absorbent cloths, followed by repeated wiping down with soft cloths moistened with Acetone until the last traces of the hazardous liquid have been removed.

Identify and, if possible, correct the source of the leak. Inspect cabling, parts and surfaces to determine whether any damage has occurred. If in any doubt, contact your local Varian, Inc. Service Dept. for assistance before reassembling and reconnecting the instrument.

Allow the interior and exterior of the instrument to dry out completely before replacing panels and covers and reconnecting power.

Dispose of contaminated waste appropriately.

## 5.3 Testing Overtemperature Protection Systems

The oven is fitted with an independent over temperature protection system to eliminate potential hazards due to failure of the temperature controller. The trip point of the protection is simply adjusted at the front panel of the instrument with the tool provided. This system should be tested regularly:

## **Oven Overtemperature Protection Test**

If the instrument is at room temperature then it will be necessary to heat the oven, otherwise this test can be carried out at any operating temperature above 40°.

To heat the oven: set the oven temperature to 40° and the heat rate to 0.5°/min. Put the instrument into RUN mode or IDLE mode (using a suitable solvent e.g. acetone) and wait for the oven temperature to stabilize.

To test the oven protection system:

- Use the pot. adjust tool provided or a small screwdriver to reduce the setting on the oven over temperature display (on the right hand side of the front panel) until the value is 5° less than the actual oven temperature (displayed on the centre of the front panel).
- A loud audible alarm will sound and the decimal points on the over temperature display will flash. Do not press the 'reset' button until the test is completed.
- If the alarm does not sound: First check that the above procedure has been followed correctly. If the procedure has been carried out correctly but no alarm sounds, disconnect both power cords from the instrument and contact your local Varian, Inc. Service Dept.
- The power to the oven heaters and waste transfer line is now interrupted so it is necessary to wait until the displayed oven temperature falls in order to verify correct operation of the protection system.
- If the displayed oven temperature shows no decrease after 30 minutes while the alarm is sounding then switch the instrument off, disconnect both power cords and call your local Varian, Inc. Service Dept.
- Once oven cooling has been verified then adjust the over temperature display to 5° above the oven set point and press the 'reset' button to silence the alarm.
- This concludes the oven over temperature protection system test.

## 5.4 Testing Vapor Sensor

Simulating a solvent leak or spillage in the oven tests the vapor sensor. You will need a sheet of absorbent paper (crumpled into a loose ball), some Acetone and a syringe for these tests. Switch ON the PL-GPC 120 if necessary and set the oven temperature to 40 °C before carrying out the oven vapor sensor test.

#### Oven

- Heat the oven to 40° with a pump flow rate of 0.5 mL/min.
- Lift the end of the solvent return tube just out of the liquid so you can see the solvent dripping into the bottle.
- Check the vapor sensor threshold is set to 5.0 (in the oven configuration window)

- Open the outer and inner oven lids and place the paper on the floor of the oven near the RI, far away from the spiral heater and fan. See picture below.
- Squirt 5 mL of Acetone onto the paper and immediately close both lids.
- After a short period the oven module alarm will sound and a vapor error notification will appear in the control software.
- Wait for 2 minutes with the alarm sounding, and then check the solvent flow has stopped by watching for drips from the return tube.
- If the alarm condition does not occur or if the pump flow has not stopped after 3 minutes then switch off and disconnect the power cords from both units. Contact your local Varian, Inc. Service Dept. for assistance.
- Open the oven and remove the paper. Dispose of it appropriately. Leave the oven open for 1 minute to allow the Acetone vapor to disperse.
- Close both oven lids and clear the error on the control software.
- This concludes the oven vapor sensor test.





It is often necessary to leave the oven open for some time to clear the sensor from solvent contamination and once clean will be re-setting by rebooting the instrument.

## 5.5 <u>Degasser</u>

The on-line degassing unit is designed to remove dissolved gas from the solvent. The dissolved gas is removed from the solvent through a membrane (thin wall PTFE tubing) inside a vacuum chamber.

The degasser should normally operate with the MON A (green) light on, indicating that the system is operating efficiently. If the MON B (red) light is illuminated the indication is that the degasser is unable to maintain a vacuum and thus unable to degas the solvent. The possible reasons for this error condition are:

## • Solvent tubing contains too much air:

- 1. Turn off the degasser and fully flush the degasser with the operating eluent until the solvent flows without the presence of air bubbles.
- 2. Turn the degasser back on and ensure the MON A light remains on and the vacuum pump cuts in and out at regular intervals.

- Vacuum tube between the vacuum chamber and vacuum pump perished:
  - 1. Turn off the power at the front of the degasser unit.
  - 2. Remove the degasser from the solvent module by disconnecting the inlet tubing at the front of the degasser and connect it directly to the inlet of the pump.
  - 3. Pull the degasser forward from the solvent delivery module and disconnect the main cable from the rear of the degasser unit.
  - 4. Remove the four screws (two at each side of the unit) from the degasser and carefully lift up the cover.
  - 5. Disconnect the earth cable from the underside of the cover and remove the cover.
  - 6. Locate the tubing between the vacuum chamber and the vacuum pump and remove it. Replace with the new replaceable part obtainable from Varian.
  - 7. Replace the cover, reconnecting the earth cable and replacing the four screws.
  - 8. Return the unit to the solvent delivery module reconnect the mains cable and the plumbing.
  - 9. Turn the degasser back on and ensure the MON A light remains on and the vacuum pump cuts in and out at regular intervals.

## 5.6 <u>Pump</u>

Two types of pumping system have been used within the PL-GPC 120 systems, namely the Shimadzu LC-20 and the Agilent 1100. The maintenance for both systems will be described here.

## Shimadzu LC-20

The LC-20AD pump is a dual-plunger parallel-flow solvent delivery module, giving high performance accuracy and sensitivity and ideally suited to GPC applications.

Operation	6	1	2	3	6	Remarks
•	mths	yr	yr	yr	yr	
Plunger seal replacement		0				<ul> <li>Plunger sealing ability decreases when seals are worn</li> <li>Seals must always be replaced with seals</li> <li>Seals should be replaced after ~3 million pump cycles (~60 L)</li> </ul>
Plunger cleaning and inspection		0				
Diaphragm inspection and or replacement			0			Diaphragms should be replaced with plungers
Inspection and cleaning or replacement of OUT check valve		0				
Inspection and cleaning or replacement of IN check valve		0				

5.6.1.1 Maintenance Operation and Intervals

Inspection and cleaning or replacement of filter	0			Filter will eventually block
Inspection and cleaning or replacement of solvent filter		0		Filter will eventually block
O-ring (purge valve) replacement			0	
Fuse replacement			0	

Before attempting any maintenance on the pump, prevent solvent from flowing from the pump by removing the solvent inlet tube from the reservoir, or placing the reservoir at a position lower than the pump head.

## 5.6.1.2 Plunger Seal Replacement

Each of the two pump heads contains a plunger seal. The procedure outlined below refers to the replacement of the seal inside the right head; the procedure for the left head would be similar.



TO PREVENT BENDING OR BREAKING THE PLUNGER IT NEEDS TO BE MOVED INTO ITS SERVICE POSITION. THIS IS ACHIEVED USING A <u>SERVICE</u> <u>INTERFACE</u>.

#### 5.6.1.2.1 Removing the Pump Head

1. Remove the SUS tubing that connects the from the outlet check valve to the purge valve assembly.



2. Unscrew the PTFE tubing from the Inlet check valve.

3. Loosen the head bolts; release each one a little at a time, so as to keep the head square.

4. Remove the pump head.



#### 5.6.1.2.2 Replacing the Plunger Seal

The plunger must be fully inserted into the pump head using a seal removal/insertion tool.





#### 5.6.1.2.3 Installing the Pump Head



#### 5.6.1.2.4 Cleaning and Inspection of Plungers and Diaphragms



CAREFUL NOT TO BEND

THE PLUNGER.

WARNING



## 5.6.1.3 Inspecting and Replacing the Diaphragms

The diaphragms must be inspected whenever the plungers are replaced.

1. Remove the diaphragm. Inspect the diaphragm for damage or deformation. Replace the diaphragm if any damage is visible.



## 5.6.1.4 Reassembling the pump head

1. Place the diaphragm into the chamber.



Ensure the diaphragm is orientated correctly with reference to the diagram on the right.



2. Holding the plunger holder between the fingers and thumb, as shown, carefully insert it into the pump chamber and screw it hand tight.



Ensure that the diaphragm is not tilted.



- 3. Use the plunger tool to tighten the plunger holder.
- 4. Insert the head holder into the pump chamber.



Ensure you keep the head aligned with the plunger at all times, so you do not bend the plunger.

- 5. Insert the two socket head bolts into the correct holes in the head holder, and screw them in using a 4 mm Allen wrench.
- 6. Tighten each of the screws up a little at a time alternating between the screws so the head is securely and squarely fastened.
- 7. Install the pump head as described in section 5.7.1.2.3.





#### 5.6.1.4.1 Post Inspection Check

After cleaning and inspecting the diaphragms and plungers check that the diaphragms are correctly seated according to the following procedure.

Cleaning fluid tube S

Cleaning

fluid outlet

- 1. Attach a syringe to the cleaning ports outlet.
- 2. While plugging the inlet port with a finger push air into the outlet port from the syringe. Release the syringe plunger.
- 3. If there is no air leakage within the pump head, the syringe plunger will return to its original position. This indicates that the diaphragm was correctly installed.
- 4. If the air escapes in the pump head and the syringe plunger is easily pushed home then the head will again need to be stripped down to rectify the fault.

## 5.6.1.5 Cleaning the Check Valves

The check valves can be simply cleaned by pumping isopropyl alcohol (IPA) through the flow lines.

- Put the suction filter attached to the pump inlet tubing into a small reservoir (~250 mL) of IPA.
- 2. Remove the column(s) and replace with a resistor tube (0.1 mm I.D. x 2 M length).
- 3. Disconnect the detector and place the outlet from the resistor tube directly to waste.
- 4. Pump the IPA through the system for ~1 hour at 2 mL/min.



Column

Male nut

Injector end

Syringe needle

Syringe

Push in

Male nut

## 5.6.1.6 Inspection and Cleaning of Check Valves

Both heads (left and right) are fitted with an INLET and OULET check valve. The procedure described below is for the removal and cleaning of the inlet and outlet check valve for the right pump head only. The procedure is identical for the left hand side.

- 1. Disconnect the outlet tubing (SUS) from the pump head.
- 2. Disconnect the PTFE inlet tubing from the pump head.

- 3. Using a 10 mm wrench remove the INLET and OUTLET check valves from the head.
- Clean the check valves by immersing them in IPA in an ultrasonic bath for ~5 mins.
- 5. Replace the check valves. The check valves are distinguishable from each other because the threads are slightly different.
- 6. Reconnect the inlet and outlet tubing.





NEVER DISASSEMBLE THE CHECK VALVES. IF THEY ARE DISASSEMBLED THE PERFORMANCE MAY BE AFFECTED. BE CAREFUL NOT TO INVERT THE INLET CHECK VALVE, BECAUSE THE INTERNAL PARTS MAY FALL OUT.

## 5.6.1.7 Inspection and Cleaning of the Inline Filter

1. Disconnect the pump outlet tubing as shown.



2. Using a 10 mm wrench remove the inline filter assembly.



The pump outlet and filter is on the underside of the drain valve on the PL-GPC 50 Plus pumps.



Place the inline filter assembly into a bath of IPA and sonicate it for 5 mins.

## 5.6.1.8 Replacing the Frit

1. If the frit needs replacing then remove it with a pair of nippers or similar.





#### 5.6.1.9 Inspecting the Suction Filter

- 1. Remove the suction filter from the solvent reservoir with the pump inlet tubing.
- Place the filter in a tissue and disconnect the filter from the tubing. The filter may be screwed on to the end of the tubing or pushed into the tubing.
- 3. To clean the filter immerse it in a bath of IPA in an ultrasonic bath for 5 mins.



## 5.6.1.10 Replacement of Drain Valve O-ring

1. Unscrew and remove the drain valve knob.



2. Remove the O-ring from the valve shaft using a pair of long nose pliers.

3. Screw the drain valve knob back into the drain valve.



## Agilent 1100 Series

See Agilent 1100 Series Isocratic Pump Reference Manual at the back of this manual (Appendix 2)

## 5.7 Inline Filter

The inline filter, located in the column oven, is plumbed directly after the pump, and is the second filter in the solvent feed line. The first filter is a 10  $\mu$ m stainless steel filter connected to the end of the pump feed tubing. The inline filter is fitted directly after the pump to prevent particles (mainly carbon leached out of the pump seal during solvent transfer) entering the capillary tubing of the chromatography system. This SSI filter assembly contains a 2  $\mu$ m cartridge, which can be easily replaced. The high capacity, low volume filter cartridges are well swept and seldom have to be replaced. The high-pressure in-line filter has a maximum operating pressure of 15,000 psi. The filter element has a surface area of 2.5 cm<sup>2</sup>.

## Cartridge Replacement Procedure

- 1. Switch off the pump to stop the solvent flow.
- 2. Disconnect one or both of the 1/16 in. tubing connections and cut the cable tie to remove.
- 3. Using 5/8 in. and 1/2 in. wrenches, remove the closure from the filter housing.
- 4. Unscrew the filter element gently and remove gasket.
- **5.** Remove any debris adhering to the housing bore or to the surface of the closure.



#### DO NOT SCRATCH THE INTERNAL SURFACES.

- 6. Place a new filter gasket over the threaded portion of the replacement filter cup and screw the filter into the closure firmly by hand.
- 7. Place the filter seal on to the closure and screw into filter body. Using 5/8 in. and 1/2 in. wrenches tighten firmly.
- 8. Reinstall the filter in the pressure line, observing the proper flow direction.
- 9. Reconnect both of the 1/16 in. tubing connection.
- 10. Secure the in-line filter cartridge with the cable tie provided.



Figure 5.3.1 Exploded View of Inline Filter Assembly

## 5.8 Injection Valve

The injection valve used in the PL-GPC 120 system is a 6 port 2-position valve, manufactured by VICI Valco®, operating with a flat seal arrangement. The valve is electrically actuated and controlled by the on board microprocessor control system. Although the valve and seal material has been specifically chosen and pre-set for operation at high temperature with chlorinated solvents, following long term continuous use the valve seal will eventually wear and require replacement. A worn seal will often manifest itself by poor irreproducible injections.

## Injection Valve Seal Replacement

It is recommended that a qualified service engineer perform this task.

#### Tools required:

9/64 hex driver Valve rotor seal

#### Disassembly

- 1. Use a 9/64 hex driver to remove the socket head screws which secure the cap on the valve.
- 2. To insure that the sealing surface of the cap is not damaged, rest it on its outer face. Or, if the tubing is still connected, leave it suspended by tubing.
- 3. With your fingers, or a small tool, pry the valve rotor seal from the driver.
- 4. Examine the valve rotor seal-sealing surface for scratches. If scratches are visible to the naked eye, the valve rotor seal must be replaced. If no scratches are visible, clean all the parts thoroughly with an appropriate solvent (THF), taking care that no surfaces get scratched. It is not necessary to dry the valve rotor seal.





#### Reassemble

- 1. Replace the valve rotor seal in the driver, making sure that the valve rotor seal sealing surface with its engraved flow passages is facing out. The pattern is asymmetrical to prevent improper placement.
- 2. Replace the cap. Insert the two socket head screws and tighten them gently until both are snug. *Do not over-tighten them* the screws simply hold the assembly together and do not affect the sealing force, which is automatically set as the screws close the cap against the valve body.
- 3. Test the valve assembly by pressurising the system. If it doesn't hold pressure, report the fault to Varian, Inc.

## 5.9 Purge Valve Operation

The purge valve is located to right hand side of the column oven behind the end panel. Its purpose is to redirect the flow from the sample side of the RI cell to the reference side of the RI cell when a RI purge is requested. The valve can be opened either manually from the purge button within the control software or automatically when programmed to do so as part of the RI conditioning procedure.



The diagram below shows the flow path (green line) during PURGE mode.

Figure 5.5.1. Purge Valve Purge Mode and Solvent Flow



Figure 5.5.2. Purge Valve Normal Mode and Solvent Flow

## 5.10 Differential Refractive Index Detector

The differential refractive index detector (DRI) is designed specifically for high temperature chromatography giving an excellent signal to noise ratio even at temperatures up to 120 °C. The light source and detectors are both external to the oven and only the optical bench is heated and maintained at the oven temperature.

In normal operation very few problems are usual encountered with the DRI, since the tubing and cell are continually washed with solvent. However, to prevent contamination of the cell during the times when the system is not operated we recommend the system be flushed and stored in Acetone or Methanol. If the cell does become dirty, which will be manifested by a reduction in the chromatography performance and typically small peaks, we recommend the following sequence of maintenance to be carried out in order.

## Purging the DRI Cell

The following problems are usually indicative that the reference cell in the DRI detector requires purging with fresh solvent.

- drift on the baseline
- excessive noise on the baseline
- lack of sensitivity

## Flushing the DRI Cell with a Cleaning Agent

If purging the reference side of the DRI cell does not cure the baseline noise or lack of sensitivity then the next step would be to clean the cell by flushing both sides of the cell with solvent or an appropriate cleaning agent. Flushing the DRI cell is also advisable if the backpressure across the DRI is determined to be higher than normal. However, if no flow is achieved through the DRI system this exercise would prove ineffective and the problem should be reported to Varian, Inc.

# Note: the backpressure across the DRI system in THF at ambient temperature and at 1 mL/min is typically $\leq$ 3Bar.

For the majority of units, the wetted parts in the DRI system are stainless steel, Hastalloy and quartz and thus the most effective cleaning is achieved by pumping a dilute solution of nitric acid (20%) through the system. However, for some units (serial numbers on request), the wetted components are Nickel and quartz and therefore nitric acid **CANNOT** be used since it will dissolve the Nickel tubing.

#### Removal of the Cell and Replacement of seals

#### It is recommended that a qualified service engineer perform this task

Once the system has been operated at high temperature the DRI cell seals tend to bond to the DRI cell and thus if the cell is to be removed it is advised to also replace the cell seals. It is recommend that the cell is only removed as a last resort or if it is necessary i.e. the cell is broken or the seals are leaking. The following procedure describes both the removal of the cell and the replacement of the seals.

Before removing the RI cell and seals from the RI block, please ensure that the system is flushed with Acetone and there is no hazardous mobile phase in the RI cell and capillary tubing.

#### A. Tools required:

Allen wrench 3 mm RI flow cell tweezers Fine-tipped Tweezers

#### B. Procedure: -

- 1. Switch the instrument OFF and disconnect the power lead(s) from the back of the unit.
- 2. Remove the flexible stainless steel fibre optic cable and RI shroud from the RI assembly. The RI shroud is clamped in position with a M4 socket head bolt located at the fan end of the assembly.



On later units the shroud is cut clear of the fibre optic cable and thus it is not necessary to remove the fibre optic cable.

- 3. Remove the top stainless steel RI cell cap retainer and inspect the two top seals. If the seals are proud of the seating recess with respect to cell cap retainer surface they are OK.
- 4. Using RI flow cell tweezers remove the RI cell from the block. Try to get a firm grip on the cell before attempting to pull it out to prevent chipping the top of the cell.
- 5. If the cell comes out cleanly, inspect the bottom seals, by running a blunt object across the bottom surface. Again if the seals appear to be proud of the surface leave alone.
- 6. If the bottom seals need to be replaced it might be easier to remove the block from the oven to replace the seals. Please refer to the Service note "Removing the DRI Optical bench". Note: If a PTFE packing ring is removed with the seal then they need to be replaced with the new seal.
- 7. Once the cell is removed it should be cleaned in 20% nitric acid. Sonicating the cell in the acid ensures the acid enter and cleans the inside of the cell
- 8. If removing a broken cell from the RI assembly, ensure all the remains of the cell have been removed from the cell compartment before fitting a replacement cell.



Ensure that the arrow etched on the cell body is pointing up

- 9. Tighten the RI flow cell cap down as tight as possible by hand. DO NOT USE ANY TOOLS
- 10. Flow solvent through the RI at 1 mL/min and check for leaks.
- 11. Replace all retaining screws and cables removed in steps 1 to 4.
- 12. Reconnect the power lead(s) and switch the instrument on

# 6 Troubleshooting

## 6.1 <u>Errors</u>

Any error(s) that occur with communications or operations (running component actions, updating parameters etc.) with the instrument, the *Diagnostic Output Window* will automatically open with the error(s) displayed in the *Component Error Status Tab* of the *Diagnostic Output Window* as shown below. Each error is uniquely identified with a number that can be referenced back to the control code.

1	🗿 PL-GPC I	nstrument - Diagnosl	tic Window		X
	Display Helj	P			
ſ	Co	omponent Error Statu	s	Procedure List Servio	e Procedures
	Clear				Setup Email
L		Date / Time	Component	Description	Procedure ID
I	- Select -	02/06/2006 14:50:15	INJECTION-VA	Get Valve Position: Could not initialise specified comms p	ort. 4
	- Select -	02/06/2006 14:50:16	PUMP	Get Volume Flow: Could not initialise specified comms p	ort. 6
L	- Select -	02/06/2006 14:50:18	UVEN	Get PL Output: No response received from the device	2Π. 8 40
L	- Select -	02/00/2000 14.30.24	<b>N</b>	det la output. No response receiveu from the device.	
l					
l					
L					
1					

Figure 6.1.1 Diagnostic Output Window

**Note:** The information displayed in last two Tabs is primarily for service diagnostics and the Window does not need to be open for normal operation of the instrument.

If an error is displayed it needs to be cleared in order for the diagnostic window to be closed, allowing access back to the PL Instrument Control software. To clear an error, press the Select button to highlight the row and press the Clear button. Multiple errors can be selected at a time. Once all errors have been cleared the diagnostic window can be closed.

Clearing an error will stop all procedures that are running and attempting to communicate with the component. To re-establish communications with the component either select the *Reconnect option* from the *Instrument menu* or return to the component view and resend the parameters or repeat the required action.

The errors that can be displayed from the software are listed on the following pages.

## **General Component Errors**

Error	Cause(s)	Action
Could not initialize specified comms port	Incorrect Com port assigned to the component, the PL-GPC 120 is not powered on or the USB cable is not connected.	Ensure the correct Com port has been assigned within the PL Instrument Configuration Editor program. Ensure the PL-GPC 120 is powered on and the USB cable is connected.
No response received from the device	Incorrect Com port assigned to the component or communications lost with the component, e.g. power failure.	Ensure the correct Com port has been assigned within the PL Instrument Configuration Editor program. Ensure the PL-GPC 120 and/or component is powered on. If assigned Com port is correct and the system is powered on then contact Varian or your local agent.
Unrecognized response from the device		Turn the PL-GPC 120 off and then on again (ensure the control software has been closed before turning the instrument off). If this error persists then contact Varian or your local agent.
The device rejected the last command	Incorrect component assigned to the Com port.	Ensure the correct component has been assigned to the correct Com port within the PL Instrument Configuration Editor program.
The device returned an error	The component failed to complete an action.	Reinitialize the component from the PL Instrument Control software and ensure the initialization is completed successfully. Otherwise turn the PL-GPC 120 off and then on again (ensure the control software has been closed before turning the instrument off). If unsuccessful and no obvious cause for the error then contact Varian or your local agent.

## Specific Errors for the Standard Fitted Components

PL-GPC 120 Oven

Error	Cause(s)	Action
Temperature sensor is not responding correctly	The oven temperature sensor is no longer functioning.	Contact Varian or your local agent.

Oven temperature has become	The oven heater is unable	to	Ensure the instrument is located in a stable environment. If the
unstable	maintain a stable temperature.		agent.

Pump

Error	Cause(s)	Action
Pump discharge pressure has exceeded the set pressure upper limit	The system backpressure has exceeded the maximum pressure limit.	Inspect the flow line for any blockages. If no blockages are found the set limit is inappropriate and needs to be changed.
Pump discharge pressure has fallen below the set pressure lower limit	The system backpressure has fallen below the minimum pressure limit.	Inspect the flow line for any leaks. If no leaks are found the set limit is inappropriate and needs to be changed.
Leak sensor has detected a leak	A solvent leak has been detected.	Inspect the flow line and related plumbing for any leaks. Wipe away any solvent in the vicinity of the leak sensor.
Pump failed to home	The motor's home position has not been detected; the motor is not running or is slipping.	Turn the PL-GPC 120 off and then on again (ensure the control software has been closed before turning the instrument off). If this error persists then contact Varian or your local agent.
CMOS protection error	Contents of memory have been lost due to a power failure etc. (RAM file loss error).	Turn the PL-GPC 120 off and then on again (ensure the control software has been closed before turning the instrument off). If this message occurs every time the system is powered on then contact Varian or your local agent.
ROM failed self test		Contact Varian or your local agent.
RAM failed self test		Contact Varian or your local agent.

**NOTE:** The pump is automatically stopped when the pressure and leak errors have been detected. The minimum pressure function is deactivated for one minute after the pump has been started.

## **RI Detector**

Error	Cause(s)	Action
Autozero operation timed out	The RI failed to autozero correctly.	Purge the detector and ensure the measurement cell is flushed with solvent. If this error persists then contact Varian or your local agent.
The LED is not functioning correctly	The LED is no longer working correctly.	Contact Varian or your local agent.

## Specific Errors for the Additional Detector Components

## K2001 and K2501-VWD UV Detectors

Error	Cause(s)	Action
Wavelength out of range		Contact Varian or your local agent.
Wavelength not supported		Contact Varian or your local agent.

## PL-ELS 1000 Detector

Error	Cause(s)	Action
Evaporator temperature	The evaporator temperature has exceeded the control temperature by an unacceptable margin.	
Nebulizer Temperature	The nebulizer temperature has exceeded the control temperature by an unacceptable margin.	Turn off the PL-ELS 1000 and allow the detector to cool, if necessary, before rebooting. If this error persists contact Varian or your local agent.
Exhaust Temperature	The exhaust temperature has exceeded the control temperature by an unacceptable margin.	
Fan Fault	The cooling fan located at the front of the unit is not operating correctly.	Ensure the fan is rotating freely and is not obstructed. The fan can be checked from underneath the front panel without disassembly. If unsuccessful and no obvious cause for the fault then contact Varian or your local agent.
Gas Error	The mass flow controller within the PL-ELS 1000 is unable to sustain the required flow rate.	Ensure the gas is turned on at the source, that the gas inlet pressure is at least 60psi (higher inlet pressure may be required for higher temperatures and flow rates) and that there is not a gas leak between the source and the detector. If unsuccessful and no obvious cause for the error then contact Varian or your local agent.
Lamp Error	The lamp is not functioning within the correct specifications.	Ensure the lamp is illuminated during normal operation and off when the detector has been stopped. The lamp is visible through the ventilation slots in the rear panel. If unsuccessful then contact Varian or your local agent.
Over temperature Error	A controlled temperature within the instrument has exceeded the maximum allowable value.	Turn off the PL-ELS 1000 and allow the detector to cool, if necessary, before rebooting. If this error persists contact Varian or your local agent.
PIC Error	A communication error has occurred within the unit.	Turn off the detector and reboot. If this error persists contact Varian or your local agent.

Error	Cause(s)	Action
Thermocouple Error	The heated transfer control system has been turned on and the heated transfer line thermocouple has not been connected.	If there is no heated transfer line connected to the detector, ensure that the transfer line temperature within the PL-ELS 1000 component view is set to 0. If a heated transfer line is connected, ensure it has been connected correctly. If this error persists contact Varian or your local agent.
Vapor Error	A solvent leak has been detected either internally or externally to the detector. For operation of the detector in a solvent polluted environment the sensitivity of the vapour sensor can be adjusted via the PL-ELS 1000 component view.	Identify the location of the leak or the source of the high level of solvent vapor and remove before clearing the error. If the source of solvent vapor is found not to be associated with the detector but still prevents the PL-ELS 1000 operating correctly then reduce the vapor sensor sensitivity via the PL-ELS 1000 component view. Note: the vapor sensor sensitivity cannot be changed from the handset. If this error persists contact Varian or your local agent.
Vapor sensor device	The vapor sensor device has malfunctioned.	Contact Varian or your local agent.

## Errors for the PL-AS RT and PL-AS MT Autosamplers

<u>General</u>

Error	Cause(s)	Action
Could not initialize specified comms port	Incorrect Com port assigned to the Autosampler or the serial cable is not connected powered on.	Ensure the correct Com port has been assigned within the PL Instrument Configuration Editor program.
No response from device	Incorrect Com port assigned to the Autosampler or communications lost with the component, e.g. power failure.	Ensure the correct Com port has been assigned within the PL Instrument Configuration Editor program. Ensure the Autosampler is powered on. If assigned Com port is correct and the Autosampler is powered on then contact Varian or your local agent.
An incomplete response was received from the device		Turn the Autosampler off and then on again and then reinitialize the Autosampler. Ensure the initialization is completed successfully. If this error persists then contact Varian or your loca agent.
An unknown response was received from the device		
The device responded with an error	The Autosampler failed to complete an action.	Reinitialize the Autosampler from the PL Instrument Control software and ensure the initialization is completed successfully. If unsuccessful and no obvious cause for the error then contact Varian or your local agent.
No response from the auxiliary device	Oven Block component selected for system configuration but not present or loss of communications with the Syringe Pump and/or Oven Block.	Remove the Oven Block component from the system configuration within the PL Instrument Configuration Editor program. For the loss of communications then contact Varian or your local agent.
An unknown response was received from the auxiliary device		Turn the Autosampler off and then on again and then reinitialize the Autosampler. If this error persists then contact Varian or your local agent.

## Autosampler Arm

Error	Cause(s)	Action
X-Axis position error - On move but did not reach the desired number of steps	The Autosampler arm was unable to reach the selected position due to either the arm hitting an obstacle, the arm reaching a position outside the defined limits of the intended position or excessive friction in the mechanical movement.	Reinitialize the Autosampler from the PL Instrument Control software and ensure the initialization is completed successfully. If unsuccessful and no obvious cause for the jam then contact Varian or your local agent.
T-Axis position error - On move but did not reach the desired number of steps		
Z-Axis position error - On move but did not reach the desired number of steps		
X-axis movement error - Movement was detected on the X-axis without being commanded to move		
T-axis movement error - Movement was detected on the T-axis without being commanded to move	The Autosampler arm has been moved without being actively driven by the Autosampler.	Reinitialize the Autosampler from the PL Instrument Control software.
Z-axis movement error - Movement was detected on the Z-axis without being commanded to move		
Dallas ID Chip communications error		Contact Varian or your local agent.
Program memory checksum error		Contact Varian or your local agent.
Error	Cause(s)	Action
---	----------	-------------------------------------
RAM Test error		Contact Varian or your local agent.
Axis initialization error		Contact Varian or your local agent.
Invalid command		Contact Varian or your local agent.
Invalid number of parameters - the command had too many or too few parameters		Contact Varian or your local agent.
Parameter out of range - one of the parameters supplied by the command is out of range		Contact Varian or your local agent.
Invalid configuration variable		Contact Varian or your local agent.
Specified configuration value out of range		Contact Varian or your local agent.
Envelope error - the probe was commanded to move to a position outside of its operating envelope		Contact Varian or your local agent.
EEPROM read error		Contact Varian or your local agent.
EEPROM write error		Contact Varian or your local agent.
Timed move error - the probe did not perform the move in the required time		Contact Varian or your local agent.

profile that is too large to fit in the fixed buffer	Profile length error - the Vf, Vi, A and J parameters result in a profile that is too large to fit in the fixed buffer		Contact Varian or your local agent.
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Syringe Pump

Error	Cause(s)	Action
Initialization error	The syringe pump was unable to initialize.	Check for blockages and loose connections before attempting to reinitialize. The syringe pump will not accept commands until it has been successfully initialized. This error can only be cleared by successfully initializing the syringe pump. Use the Initialize Arm action for the Autosampler to initialise the syringe pump. If this error persists then contact Varian or your local agent.
Device not initialized	The syringe pump has not been initialized or has lost its positional information, i.e., a stall has occurred, but a move command has been issued.	Reinitialize the syringe pump. Use the Initialize Arm action for the Autosampler to initialize the syringe pump.
Plunger overload	The syringe plunger on movement is blocked by excessive backpressure.	Check the connecting tubing, syringe valve and syringe for blockages. The syringe pump must be reinitialized before normal operation can resume. This error can only be cleared by reinitializing the pump. Use the Initialize Arm action for the Autosampler to initialize the syringe pump. If this error persists then contact Varian or your local agent.
Valve overload	The syringe valve drive has lost steps.	Reinitialize the syringe pump by using the Initialize Arm action for the Autosampler. If this error persists then contact Varian or your local agent.
Plunger move not allowed	The syringe valve is in the bypass or throughput position.	Reinitialize the syringe pump by using the Initialize Arm action for the Autosampler. If this error persists then contact Varian or your local agent.

Error	Cause(s)	Action
Invalid command		Contact Varian or your local agent.
Invalid operand		Contact Varian or your local agent.
Invalid command sequence		Contact Varian or your local agent.
Command overflow		Contact Varian or your local agent.

## Oven Block (PL-AS MT Autosampler)

Error	Cause(s)	Action
Oven temperature has become unstable	The block heater is unable to maintain a stable temperature.	Ensure the Autosampler is located in a stable environment. If the environmental conditions are fine then contact Varian or your local agent.
Temperature sensor is not responding correctly	The temperature sensor is no longer functioning.	Contact Varian or your local agent.

#### Errors for the Midas Carousels

#### <u>General</u>

Error	Cause(s)	Action
Could not initialize specified comms port	Incorrect Com port assigned to the Carousel or the serial cable is not connected	Ensure the correct Com port has been assigned within the PL Instrument Configuration Editor program. Ensure the PL-GPC 50 Plus is powered on.
No response from device	Incorrect Com port assigned to the Carousel or communications lost with the component, e.g. power failure.	Ensure the correct Com port has been assigned within the PL Instrument Configuration Editor program. Ensure the Carousel is powered on. If assigned Com port is correct and the Carousel is powered on then contact Varian or your local agent.

Error occurred during initialization, the Midas cannot start.	The Midas Carousel failed to initialize correctly.	Reinitialize the Carousel from the PL Instrument Control software and ensure the initialization is completed successfully. If unsuccessful and no obvious cause for the error then contact Varian or your local agent.
An incomplete response was received from the device.		Turn the Carousel off and then on again and then reinitialize the
An unknown response was received from the device.		this error persists then contact Varian or your local agent.
The device responded with an error.	The Carousel failed to complete an action.	Reinitialize the Carousel from the PL Instrument Control software and ensure the initialization is completed successfully. If unsuccessful and no obvious cause for the error then contact Varian or your local agent.

## Syringe Dispenser

Error	Cause(s)	Action
The syringe valve did not switch		
The syringe did not reach the home position within time		
The syringe spindle did not make the correct number of rotations		and ensure the initialization is completed successfully. If unsuccessful and no obvious cause for the error then contact Varian or your local agent.
The spindle does not rotate		
The syringe valve did not find a valid position		

## Injection Needle

Error	Cause(s)	Action
The sample needle arm did not reach or leave the home position (vertical)		
The sample needle arm is in an invalid horizontal position while moving down		
The sample needle arm did not reach its destination within a certain time (horizontal)		Reinitialize the Autosampler from the PL Instrument Control
Too many or not enough steps needed to reach destination of horizontal needle movement		software and ensure the initialization is completed successfully. If unsuccessful and no obvious cause for the jam then contact Varian or your local agent.
Sample needle arm not in vertical home position while moving horizontally		
The sample needle arm is at an invalid horizontal position		
The sample tray is not at a valid position while moving the sample needle arm down		
Vial sensor sticks		Contact Varian or your local agent.

## Carousel Tray

Error	Cause(s)	Action
Incorrect tray rotation		Contact Varian or your local agent.
No sample tray positioned in the Midas		Ensure a tray is positioned in the Carousel. If there is no tray then contact Varian or your local agent.
The sample needle arm is not in the home position while rotating the tray		Contact Varian or your local agent.

## 6.2 <u>General Troubleshooting Guide</u>

### 1. BOOT UP PROBLEMS

- a) NO POWER
  - i) If power indicators, power switch or display panel not illuminated, check fuses at rear of control drawer.
- b) FAILS TO INITIALIZE PUMP
  - i) Check pump power switch is turned on.
  - ii) If using a Bischoff pump ensure the EXT mode is selected.
  - iii) Check power cable is securely fitted to the rear of the pump.
  - iv) Check pump serial link is connected correctly, to both the rear of the pump and the back of the PL-GPC 120 control drawer.
- c) INJECTION VALVE FAILURE
  - i) Check Link cables on the rear of control drawer are connected correctly.
  - ii) Try operating valve in the Service Engineer mode. Switching the valve back and forth several times should reinitialize the valve head.

### 2. <u>RI</u>

- a) FAILS TO AUTOZERO CORRECTLY
  - i) Purge RI thoroughly.
  - ii) Check purge column is not blocked (if fitted), by observing a significant decrease in system pressure when purge valve opens.
- b) LOW SENSITIVITY
  - i) Clean RI cell thoroughly (e.g. sonicate in 20% Nitric acid)

### 3. OVEN TEMPERATURE CONTROL

- a) FAILS TO HEAT
  - i) Check Overtemperature Cut-Out has not tripped. Should be set about 5 °C above operating temperature.
- b) TEMPERATURE UNSTABLE
  - i) Check oven fan is rotating at high speed. The speed of the fan is recorded in the Service engineer mode.
  - ii) Check oven lid is fully closed and seals properly.
  - iii) Check power line voltage not below 200V.
  - iv) Check ambient temperature above 10 °C.

#### 4. <u>PUMP</u>

- a) HIGH BACK PRESSURE GENERATED AT PUMP (AGILENT 1100)
  - i) Replace the purge frit.
- b) LOW FLOW RATE
  - i) Purge pump to remove air bubbles
  - ii) Service check valves

#### 5. CHROMATOGRAPHY

- a) RI BASELINE DRIFT
  - i) Allow sufficient time for system to equilibrate at temperature (8 hrs is usually enough).
  - ii) Ensure oven temperature remains stable.
  - iii) Check pump flow rate / backpressure is constant.
  - iv) Check all plumbing connections between Purge valve outlet and RI Ref inlet.
  - v) Check RI cell not cracked.
- b) RI BASELINE NOISE
  - i) Purge Pump head to clear air from Check Valves.
  - ii) Purge RI Reference thoroughly to remove any air bubbles.
  - iii) Check for presence of pump noise. Clean / replace check valves if necessary. The pump pressure is continually monitored when the Logging is turned on and the pressure history can be viewed in the log table or plotted graphically – See the LOG MENU in the graphical user interface.
  - iv) Clean / replace in-line filter element.

- v) Check degasser is operating correctly. The degasser pump motor should cut in and out to maintain low chamber pressure. If it runs continuously there could be an air leak.
- c) SMALL / NON-REPRODUCIBLE PEAKS
  - i) Ensure the injection loop is sufficiently flushed prior to injection. Need to inject ~3 times the loop volume +100  $\mu$ L to adequately fill the loop.
  - ii) If using an autosampler ensure the vials are adequately filled and enough sample is injected into the loop.
  - iii) Inspect Valco valve seal for scoring / wear.
  - iv) Check all autosampler tubing connections for leaks.
  - v) Check autosampler needle not blocked.
  - vi) Check autosampler syringe plunger seal is gas-tight.
- d) LONG RETENTION TIMES
  - i) Check pump flow rate.
  - ii) Check for leak on Purge valve.
- e) 'MIRRORED' PEAKS
  - i) Ensure purge valve is closed.
  - ii) Inspect RI cell for cracks, particularly in partition.

# Parts List

Product Description	Part No.
<u>PL-GPC 210/220/PL-GPC 110/120</u>	
Flow cell (RI)	PL0800-0004
Injection valve rotor seal	PL0800-0003
Gasket seals for RI cell (Pk of 4)	PL0800-0005
Piston seal setting tool	PL0800-0012
Vapor sensor assembly oven (complete) for PL-GPC 110/210 (sn 020-029+) and all PL-GPC 220's	PL0800-0056
Vapor sensor assembly oven (complete) for PL-GPC 110/210 (up to sn 020-028)	PL0800-0057
Vapor sensor assembly solvent module (complete) for PL-GPC 110/210 (up to sn 030-028)	PL0800-0058
Vapor sensor assembly solvent module (complete) for PL-GPC 110/120 (sn 030-029+) and all PL-GPC 220's	PL0800-0059
Injection valve cap	PL0810-0021
RI flow cell removal tool	PL0810-0026
Advanced PC control of PL-GPC 210	PL0810-0050
Hold-Up column for PL-GPC 210/PL-GPC 220	PL0810-1000
RI light source upgrade (PL-GPC 120/220)	PL0820-0030
PL-GPC 220 spares kit	PL0820-1120
RI Pre-amp PCB assy PL-GPC 210	PL091/23884
Vial locator solenoid and plunger	PL091/23926
Oven fan assembly	PL091/24260
RI block and fittings	PL091/24351
Autosampler syringe luer adapt	PL091/24618
RI rod & detector assy	PL091/24674
Interface PCB	PL091/24965
Autosampler syringe assembly (PL-GPC 220)	PL091/25088
RI light source (PL-GPC 120/220)	PL091/25296
Oven fan blade tool	PL092/24473
Surge safe	PL092/24893
Autosampler syringe	PL094/32261
In-line filter assembly	PL094/32334
Autosampler power supply	PL094/32408

Replacement In-line filter cartridges	PL094/32434
Fibre optic cable	PL094/32515
Bellows coupler for oven fan assembly	PL094/32522
Solvent (frit) filter	PL094/32561
Target 188EB PCB	PL094/32751
Autosampler syringe seal (PL-GPC 220)	PL094/32986
Control drawer power supply PFC (120/220/220+)	PL094/32995
Pump tubing cassette (PL-GPC 110/120)	PL094/33004
Purge valve/pressure transducer assembly (PL-GPC 110/210)	PL0800-0017
HP pump upgrade for PL-GPC 210+	PL0810-0060
HP pump PC control upgrade for PL-GPC 210+	PL0810-0070

Part No.

## Product Description

HP1100 Pump

HP1100 pump for use in PL-GPC 120 and PL-GPC 220	PL0820-1100
Plunger seal for HP1100 pump	PL0820-1101
Drive assy for HP1100 pump	PL0820-1102
Active inlet valve for HP1100 pump	PL0820-1103
PTFE Frits for HP1100 pump	PL0820-1104
Solvent filter for HP1100 pump	PL0820-1105
Outlet valve for HP1100 pump	PL0820-1107
Wash seal for HP1100 pump	PL0820-1108
Gold seal for HP1100 pump	PL0820-1109
Wash gaskets for HP1100 pump	PL0820-1110
AIV cartridge for HP1100 pump	PL0820-1111
Plunger for HP1100 pump	PL0820-1112
Plunger housing for HP1100 pump	PL0820-1113
Purge valve for HP1100 pump	PL0820-1114
Mainboard for HP1100 pump	PL0820-1115
Support ring for HP1100 pump	PL0820-1116
Lock screw for HP1100 pump	PL0820-1117
AIV adapter for HP1100 pump	PL0820-1118
HP1100 pump pulse dampener	PL0820-1121

Product Description	Part No.
Shimadzu Pump	
Solvent filter (body only)	PL0870-6010
Plunger seal	PL0870-6020
Solvent filter in line frit	PL0870-6030
Solvent filter maintenance kit	PL0870-6000
Plunger holder assy.	PL0870-6040
Inlet check valve assy.	PL0870-6050
Outlet check valve assy.	PL0870-6060
Pump head	PL0870-6070
Pump head holder	PL0870-6080
Photosensor assy.	PL0870-6090
Diaphragm (PCS/set)	PL0870-6100
Solvent Filter	PL0870-6110
Bushing (for suction filter)	PL0870-6120
Ferrule 3.0F-T (for suction filter)	PL0870-6130
FEP tube 3x1.5 (for suction filter)	PL0870-6140
Drain tube assy.	PL0870-6150
Washing tube assy.	PL0870-6160
ETFE tubing 1.6x0.8 (between block & check valve)	PL0870-6170
PEEK male nut 1.6MN	PL0870-6180
Pump Inlet block	PL0870-6190
Pressure sensor	PL0870-6200
Drain valve perflow assy.	PL0870-6210
O-ring	PL0870-6220
Damper assy.	PL0870-6230
Key top pump	PL0870-6240
Switch assy.	PL0870-6250
Transformer assy.	PL0870-6260
PCB main pump VP assy.	PL0870-6270
PCB sensor assy.	PL0870-6280
PCB PRSEN assy.	PL0870-6290
PCB panel assy.	PL0870-6300
PCB CPU assy.	PL0870-6310
Lithium battery CR2032	PL0870-6320
Voltage selector assy.	PL0870-6330
Leak sensor assy.	PL0870-6340
Fan for SPD60	PL0870-6350
Harness, front assy.	PL0870-6360
Cable, pressure sensor assy.	PL0870-6370

## **Connecting to a PL-AS MT Autosampler**

Before using the PL-AS MT autosampler, the needle and syringe pump of the autosampler must be plumbed directly to the injection valve within the column oven. Tubes are provided for this purpose with the automsampler; connection tubes or heated transfer line.

The narrow bore tube or one of the three narrow bore tubes within the heated transfer line should be connected to PORT 1 of the injection valve and directly to the needle. The transfer line should be routed via the cable clip on the arm, through the cable tie located beneath the needle assembly and then up to the front of the needle assembly to the top of the needle, see Figures 1 and 2. Gently connect the correct PTFE tube to the needle, a wrench can be fitted to the bottom nut if necessary to stop the barrel fitting turning while tightening the connection. The two tubes within the heated transfer line are 1.2 m long with a 0.010 in. ID thus having an internal volume of approximately 60 µL.



The heated transfer line is connected directly to the auxiliary ports on the LHS of the PL-GPC 120 and is controlled at the same temperature of the column oven with a upper maximum operating temperature of 120 °C.

- The wide bore PTFE tube should be connected to PORT 6 of the injection valve and directly to the right hand port of the autosampler syringe pump. This tube should be routed around the back of the autosampler to the valve passing through the cable clip on the rear of the autosampler, see Figures 3 and 4. This tube is 1 m long with a 0.040 in. ID thus having an internal volume of approximately 810 µL.
- The long (1.7 M) wide bore PTFE tube, typically located in the plastic bag at the rear of the autosampler, should be connected to the left hand port of the syringe pump and then routed to the solvent reservoir and submerged in the solvent.

The RS232 serial cable is connected between the 9 pin "Host" connector (the one not capped off) on the right hand side of the autosampler and the controlling Serial port



## Connecting the Midas<sup>™</sup> Carousel

### **Electrical Connections**

When operating with a Midas A/S using the Serial Cable supplied, attach one end of the cable to the S2 Port (7) on the carousel and the other to a free serial port on the USB hub or directly to the PC..



Before connecting the power cable, ensure the instrument voltage rating (on the rating plate label) matches your local power supply. Use only a supply with **protective grounding**. If the voltage rating and fuses are correct for your power source, plug in the power cable.

The Midas Carousel needs to be added to the instrument configuration in order to control it from the PL Instrument Control software. To add the carousel to the configuration open the PL Instrument Configuration Editor program and select *Add Component* from either the Configure menu or toolbar icon. From the Add Component dialog select either the Midas-24, Midas-87 or Midas-96 component depending on the carousel tray being used. Once the carousel component has been added assign the correct Com port.

For further information on configuring the system then please see the on-line help within the PL Configuration Editor program.

Set the MIDAS into SERIAL mode, for operation from the PL-GPC 120 control software.



Set the Midas Carousel to Serial mode to control the carousel from the PL Instrument Control software.

For information on controlling the Midas Carousel with the PL Instrument Control software then please see the on-line help within the control software.

### **Plumbing Connections**

For the Midas Carousel the needle and syringe valve must be plumbed to the injection valve onboard the PL-GPC 120.

The plumbing connections between the Midas autosampler and the PL-GPC 120 are as follows: -

- 1. The tube labelled with the red band should be connected to PORT A of the injection panel. This tube is 80 cm long with a 0.010 in. ID thus having an internal volume of  $40 \ \mu$ L.
- 2. The tube labelled with a yellow band should be connected to PORT B or C of the injection panel. This tube is 90 cm long with a 0.040 in. ID thus having an internal volume of 707  $\mu$ L.

The PTFE tubes should then be connected to the carousel as follows.

- 1. The tube labelled with the red band should be connected directly to the injection needle using a low dead volume Valco union.
- 2. The tube labelled with the yellow band should be connected to the centre port of the autosampler syringe pump.



Finally replace the tube for the wash solvent with a 1 M length of 0.04 in. ID tubing, so the tube comfortably reaches the solvent reservoir.

Also connect a drain tube to the outlet on the front of the carousel, venting it to a waste container below the carousel to collect the solvent used during the wash cycles.

In both cases the Injection synchronization pulse can be taken from the rear of the PL-GPC 120 control drawer to start the data acquisition module, since an injection pulse is obtained each time the injection valve is switched from Load to Inject.

For full details of the operation of the Midas A/S please consult the Operation Manual supplied with the A/S.

# Sections from Agilent 1200 Series Reference Manual

# PL-BV 400 HT Installation and Operator's Guide

## PL-LS 15/90

# Installation and General Operating Information