

OPERATOR'S MANUAL

FOR

PL-ELS 1000 and PL-ELS 1000 μ

**EVAPORATIVE
LIGHT SCATTERING
DETECTOR**

Version 4.0 September 2001



Polymer Laboratories

Polymer Laboratories Ltd, Essex Road, Church Stretton, Shropshire SY6 6AX, UK
Tel +44 (0)1694 723581, Fax +44 (0)1694 722171, Service Tel +44 (0)1694 724333

Polymer Laboratories Inc, 160 Old Farm Road, Amherst, MA 01002, USA
Tel: +1 413 253 9554, Fax +1 413 253 2476

Polymer Laboratories Ltd, Sourethweg 1, 6422 PC Heerlen, The Netherlands
Tel +31 (0)45 5414748, Fax +31 (0)45 5410005

Polymer Laboratories GmbH, PEKA Park T5 001 Otto-Hesse Straße 19 D 64293 Darmstadt,
Germany Tel +49 (0)6151 860690, Fax +49 (0)6151 860670

Polymer Laboratories SARL, Centre Silic Marseille Sud, Impasse du Paradou, Bâtiment A4, 13009
Marseille, France. Tel + 33 (0)491 176400, Fax + 33 (0)491 176401



Polymer Laboratories



DECLARATION OF CONFORMITY

We, Polymer Laboratories Ltd
Essex Road
Church Stretton
Shropshire SY6 6AX
U.K.

declare that the product:

Evaporative Light Scattering Detector

PL-ELS 1000

Part # 091/24797

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:	EN 61000-4-2; 4-3; 4-4; 4-5; 4-6; 4-11; 3-3	
		Electromagnetic compatibility and Mains voltage and flicker emissions
	ENV 50204	Electromagnetic compatibility
	EN 55022	Conducted emissions: Class B
		Radiated emissions: Class B*
	EN 60555-2	Harmonic emissions

1st April, 1998

Dr. S.O'Donohue, Technical Director -Instrumentation



Polymer Laboratories



DECLARATION OF CONFORMITY

We, Polymer Laboratories Ltd
Essex Road
Church Stretton
Shropshire SY6 6AX
U.K.

declare that the product:

Evaporative Light Scattering Detector

PL-ELS 1000 μ

Part # 091/25484

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:	EN 61000-4-2; 4-3; 4-4; 4-5; 4-6; 4-11; 3-3	
		Electromagnetic compatibility and Mains voltage and flicker emissions
	ENV 50204	Electromagnetic compatibility
	EN 55022	Conducted emissions: Class B
		Radiated emissions: Class B*
	EN 60555-2	Harmonic emissions

14th September 2001

Dr. S.O'Donohue
Technical Director - Instrumentation

PL-ELS 1000 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 (lamps and wetted parts which includes the nebulizer and evaporator assemblies,) 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.

SAVE AS PROVIDED ABOVE, THE SELLER WILL BE UNDER NO LIABILITY UNDER THE CONTRACT FOR ANY PERSONAL INJURY, DEATH, LOSS OR DAMAGE OF ANY KIND WHATSOEVER WHETHER CONSEQUENTIAL OR OTHERWISE INCLUDING BUT NOT LIMITED TO LOSS OF PROFITS AND THE SELLER HEREBY EXCLUDES ALL CONDITIONS, WARRANTIES AND STIPULATIONS EXPRESS OR IMPLIED, STATUTORY, CUSTOMARY OR OTHERWISE WHICH BUT FOR SUCH EXCLUSION WOULD OR MIGHT SUBSIST IN FAVOUR OF THE BUYER EXCEPT THAT SUCH EXCLUSION WILL NOT APPLY TO ANY IMPLIED CONDITION THAT THE SELLER HAS OR WILL HAVE THE RIGHT TO SELL THE GOODS WHEN THE PROPERTY IS TO PASS OR WHEN THE BUYER DEALS AS A CONSUMER (AS DEFINED IN SECTION 12 OF THE UNFAIR CONTRACT TERMS ACT 1977), ANY IMPLIED TERM RELATING TO THE CONFORMITY OF THE GOODS WITH THEIR DESCRIPTION OR SAMPLE OR AS TO THEIR QUALITY OR FITNESS FOR A PARTICULAR PURPOSE.

IN NO CIRCUMSTANCES WILL THE SELLER OR ITS EMPLOYEES, AGENTS, OR SUB-CONTRACTORS BE LIABLE FOR ANY LOSS OR DAMAGE OF ANY KIND WHATSOEVER WHETHER CONSEQUENTIAL OR OTHERWISE CAUSED DIRECTLY OR INDIRECTLY BY ANY NEGLIGENCE OR OTHER TORTIOUS ACT OR BREACH OF STATUTORY DUTY ON THE PART OF THE SELLER OR ON THE PART OF ANY OF ITS EMPLOYEES, AGENTS OR SUB-CONTRACTORS IN CONNECTION WITH OR ARISING OUT OF THE MANUFACTURE OR SUPPLY OF THE GOODS OR IN CONNECTION WITH ANY STATEMENT GIVEN OR MADE (OR ADVICE NOT GIVEN OR MADE) BY OR ON BEHALF OF THE SELLER.

Any statement made about the Seller's goods by the Seller or its servants or agents whether orally or in writing is intended for guidance only and the Buyer should not place any reliance thereon without specific enquiry and without ensuring that any matter of concern to him is specifically mentioned in the contract.

The Buyer is solely responsible for the suitability of the site for the installation of the goods, for obtaining all and any necessary consents and approvals under planning and building regulations and by-laws and for the preparation of the site, the constitution of foundations and the provision of services so that the site is suitable to receive the goods.

Unless expressly provided therein, the contract does not include the installation, erection or commissioning of any goods or equipment or the supervision thereof. Where installation is included in the contract it is the responsibility of the Buyer to advise the Seller of prevailing site conditions, and in particular but without prejudice to the foregoing, physical characteristics, availability of services, normal labour working hours and any local or national labour arrangement or practices which might affect the work, and any other relevant factors. Any additional costs caused by any interruption or delay not attributable to the Seller or to its employees or because the Seller cannot gain access to the site shall be charged to the Buyer together with an appropriate allowance for profit. The Buyer shall also be responsible for providing a safe and healthy working environment for the Seller's employees and the Seller's subcontractors and their employees and shall bear (or reimburse the Seller against) any loss, damage or compensation due to any person in relation to any death or personal injury or destruction or damage to property except to the extent that the same is due to the negligence of the Seller and the Buyer shall except as aforesaid keep the Seller fully indemnified in respect thereof.

SAFETY

Signs and Pictograms Used in this Manual



WARNING

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

ATTENTION:

The “ATTENTION sign” denotes relevant information.

Read this information first before proceeding, it will be helpful or necessary to complete the task.



NOTE

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.



WARNING

Electrical Hazards

Removal of instrument panels may expose potentially dangerous voltages. Disconnect the instrument from all power sources before removing protective panels.

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.



NOTE

General Precautions

Perform periodic leak checks on supply lines.

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

HEALTH AND SAFETY

PL-ELS 1000

WARNING: 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 utilising 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 authorisation from PL'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 (eg 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 PL office or local distributor.

In the UK and Europe

Polymer Laboratories Ltd

Technical Assistance

Tel +44 (0) 1694 723581

Fax +44 (0) 1694 722171

Service

Tel +44 (0) 1694 724333

Fax +44 (0) 1694 723994

In the US

Polymer Laboratories Inc

Technical Assistance & Service

Tel (413) 253 9554

Fax (413) 253 2476

In the Benelux countries

Polymer Laboratories Ltd

Technical Assistance & Service

Tel +31 (0) 45 5414748

Fax +31 (0) 45 5410005

In Germany

Polymer Laboratories GmbH

Technical Assistance & Service

Tel +49 (0)6151 860690

Fax +49 (0)6151 860670

In France

Polymer Laboratories SARL

Technical Assistance & Service

Tel 0033 491 176400

Fax 0033 491 176401

TABLE OF CONTENTS

DECLARATION OF CONFORMITY	I
DECLARATION OF CONFORMITY	II
PL-ELS 1000 WARRANTY	III
SAFETY	IV
SIGNS AND PICTOGRAMS USED IN THIS MANUAL	IV
SAFETY PRACTICES	V
HEALTH AND SAFETY	VI
CHAPTER 1 GENERAL INFORMATION	1
1.1 INTRODUCTION.....	1
1.2 SPECIFICATIONS	1
1.3 UNPACKING AND INSTALLATION	2
1.3.1 Packing list.....	2
1.4 SITE PREPARATION CHECK LIST.....	3
1.5 CONNECTIONS	4
1.5.1 Power Connections.....	5
1.5.2 Extraction.....	5
1.5.3 Communication Connections	6
1.5.4 Gas Connection.....	7
1.5.5 Fluid Connection.....	7
1.6 PRECAUTIONS	9
1.6.1 Extraction.....	9
1.6.2 Electrical.....	9
1.6.3 Flammable solvents.....	9
CHAPTER 2 SYSTEM DESCRIPTION AND GENERAL OPERATION	10
2.1 BASIC PRINCIPLES OF OPERATION	10
2.1.1 Nebulization	10
2.1.2 Evaporation.....	10
2.1.3 Detection	10
2.1.4 Theory	11
2.2 OPERATIONAL PARAMETERS.....	14
2.3 INSTRUMENT CONTROLS	14
2.3.1 Power On/Off.....	15
2.3.2 Display	15
2.3.3 LEDs.....	15
2.3.4 Handset	15
2.3.5 The Graphical User Interface	17
2.3.6 Modes of Operation.....	17
2.3.7 Using Methods.....	20
2.3.8 The Heated transfer Line.....	20
2.3.9 Setting the Instrument Time Constant	21
2.3.10 Error Conditions	21
CHAPTER 3 SET-UP	22
3.1 GENERAL CONSIDERATIONS	22
3.2 CONNECTING THE DETECTOR.....	22
3.3 OPTIMISATION PROTOCOL	26
3.3.1 Nebulizer Gas Flow.....	26

3.3.2	Evaporator Temperature	26
3.3.3	Nebulizer Temperature	26
3.3.4	Procedure	26
CHAPTER 4 ROUTINE MAINTENANCE		27
4.1	DRYING THE DIFFUSER	27
4.2	CLEANING EVAPORATOR TUBE	27
4.3	INFORMATION FOR SERVICE PERSONNEL	28
4.3.1	Heater PCB Fuses	28
4.3.2	Handset Batteries	28
4.3.3	Safety Earth Bond Screw	28
CHAPTER 5 TROUBLESHOOTING		29
5.1	INSTRUMENT ERRORS	29
5.2	GENERAL PROBLEMS	31
APPENDIX 1		33
	PL-ELS 1000 QUICK USER GUIDE	33
APPENDIX 2		35
	METHOD RECORD SHEET	35
APPENDIX 3		36
	PL-ELS 1000 TEST PROCEDURE	36
APPENDIX 4		37
	SPARE PART LISTING	37

Chapter 1

General Information

1.1 Introduction

The PL-ELS 1000 evaporative light scattering detector or mass detector is a unique and highly sensitive detector for non-volatile solutes in a volatile liquid stream. It is mainly used as a concentration detector in Gel Permeation Chromatography (GPC) and other forms of High Performance Liquid Chromatography (HPLC). The solvent stream containing the solute material is nebulized and carried by a gas flow through an evaporating chamber. The solvent is volatilised, leaving a mist of solute particles that scatter light to a photosensitive device. The signal is amplified and a voltage output results. The output is directly related to the “mass” of the solute particles passing through the light, hence the name “Mass Detector”.

The PL-ELS 1000 may be used alone, or as one of several detectors in a GPC or HPLC system. As the solvent or eluent is evaporated in the course of the analysis, the PL-ELS 1000 must be the last in series if used in conjunction with other detectors. If the PL-ELS 1000 is being used as the last detector in a series, care must be taken not to exceed the recommended back-pressure in detector cells in other units.

1.2 Specifications

Light Source		Tungsten/Halogen Lamp
Detector		Photodiode
Temperature Range*	Evaporator	30-300°C
	Nebulizer	30-220°C
	Heated Transfer Line	30-220°C
Gas Flow		0-2 SLM @60 psi @25°C (PL-ELS 1000)
		0-1 SLM @100 psi @25°C (PL-ELS 1000μ)
	Pressure operating range	60 – 100 psi (4-6.7 bar)
	Maximum Pressure	150 psi (10 bar)
Eluent Flow		0-5.0ml/min (PL-ELS 1000)
		0-0.5ml/min (PL-ELS 1000μ)
Analogue Output		0-10V
		0-1V
Communication		Serial I/O
	Outputs	2 Contact closures
		1 TTL +ve
		1 TTL -ve
		Heated Transfer line control
	Input	Remote auto-zero control
Instrument Control		Microprocessor
Instrument Interface		Detachable IR remote control
		Windows based PC control
Detector Status		Sleep, Gas Save, Standby, Heating, Ready and Error
Size	wxdxh	175x480x430mm
	Packaged wxdxh	670x340x580mm
Weight		15kg
	Packaged	22kg

* To achieve the maximum operating temperature, the unit must be supplied with either 110V or 240V as indicated. Voltage supplies below these values will limit the maximum operational temperature of the instrument.

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 Polymer Laboratories. 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. Check that all items on the packing list are included.

Notify your local distributor or Polymer Laboratories of any damage or missing items.

1.3.1 Packing list

Standard Items

- . PL-ELS 1000 detector
- . Manual for PL-ELS 1000 detector
- . Mains Lead (110-120V, 60Hz or 230V, 50Hz)
- . 15 pin D-sub male connector for Aux I/O connection
- . Detector output cable
- . Gas Inlet tube
- . Waste bottle (250ml) with side arm at 100ml
- . Nebuliser Drain tubing (Convolute PTFE – 230mm)
- . Extraction hose (Convolute PTFE - 2m)
- . Bottle drain tube (Convolute PTFE - 1.5m)
- . Exhaust cap to bottle waste tube (Convolute PTFE – 340mm)
- . Valco Nut and ferrule (1/16")
- . PL-ELS 1000 Control Software

Optional

- . Heated Transfer Line
- . Exhaust Hose (2" diameter)

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



USA and Japan 115V (AC) $\pm 10\%$
50/60 Hz, 6A max.

Europe 230V (AC) $\pm 10\%$
50/60 Hz, 3A max.

Gas Supply



Gas: Nitrogen (98% purity or better and filtered to 0.2 μ m)

Notes:

- \Rightarrow Air can be used for non flammable solvents
- \Rightarrow The mass flow controller is not calibrated for use with gases other than Air or Nitrogen
- \Rightarrow For operation with other inert gases contact Polymer Laboratories for advice.

Gas flow 0-2 SLM @ 60 psi @25°C (PL-ELS 1000)

Gas flow 0-1 SLM @ 100 psi @25°C (PL-ELS 1000 μ)

Pressure operating range: 0 – 100 psi (4-6.7 bar)

Maximum Pressure: 150 psi (10 bar)

Extraction Requirements



During the normal operation the carrier solvent is evaporated as it passes through the instrument and must be extracted safely at the rear of the unit.

The exhaust from the instrument (10mm ID convoluted PTFE tubing) must be extracted to a fume hood or similar solvent disposal unit.

If the extraction tube provided with the instrument is to be extended it is recommended that the diameter of the extension is increased to at least 50mm (2") diameter tubing so the extraction quality is not inhibited

1.5 Connections

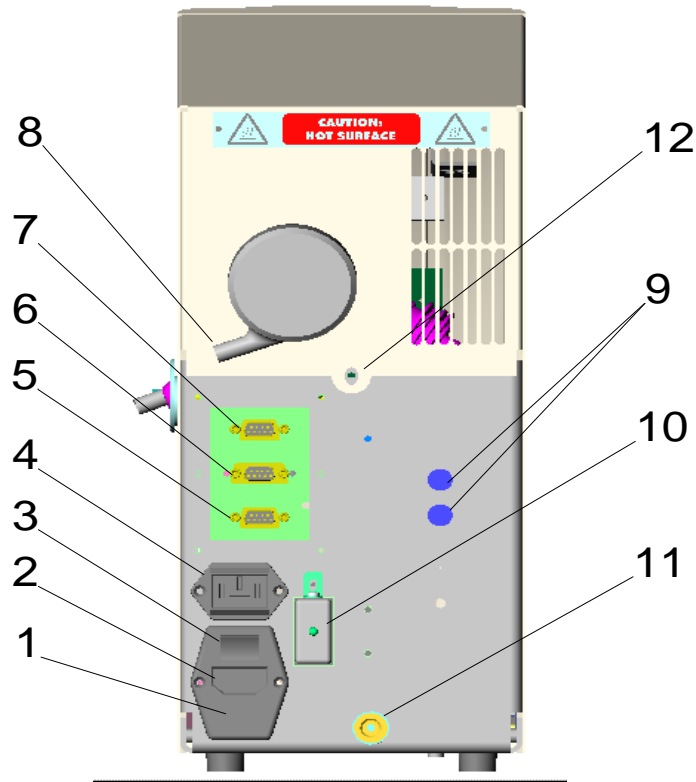


Figure 1.1 Rear View of PL-ELS 1000

- | | |
|---|--|
| 1. Fuses and voltage selector | 7. Serial RS232 connector – 9 pin D type female |
| 2. Mains input | 8. Exhaust |
| 3. Mains switch | 9. Detector outputs; 1V and 10V |
| 4. Heated transfer line mains supply | 10. Surge safe |
| 5. Heated transfer line thermocouple feedback – 9 pin D type female | 11. Supply gas inlet |
| 6. Connector control I/O –15 pin D type female | 12. Safety Earth Bond Screw. <u>Must be fitted.</u> |



NOTE

The heated transfer line terminal is for use only with equipment that has no live parts, which are ACCESSIBLE. The terminal must only be used to connect a heated transfer line manufactured by Polymer Laboratories Ltd, fitted with a Bulgin PX0770 appliance connector and rated to a working voltage of 300V which meets the requirements of EN61010-1:1998 for pollution degree 2, installation category II.

1.5.1 Power Connections

- ◆ Before connecting the power cable, ensure the instrument voltage rating matches your local power supply.
- ◆ Use only a supply with protective grounding.
- ◆ The correct fuses should be installed.

For 115V (AC) $\pm 10\%$, use two 6.3A F HBC 250V fuses

For 230V (AC) $\pm 10\%$, use two 5.0A F HBC 250V fuses



THIS UNIT IS DOUBLE - FUSED.



RISK OF FIRE, REPLACE FUSES AS MARKED!

- ◆ If the voltage rating and fuses are correct for your power source, connect the power cable.

1.5.2 Extraction

The PL-ELS 1000 is provided with tubing for venting the exhaust gases and vapours, and so does not need to be placed in a fume cupboard. Routing the exhaust gases through the nebulizer waste bottle allows a proportion of the eluent to be collected by condensation. The exhaust cap on the rear of the instrument is connected to the waste “bottle trap” by the 340mm length of convoluted PTFE hose. This hose is a push-fit onto the tubing stubs. The 2m length of hose then carries the gases from the bottle to a suitable fume extraction system. To optimise the efficiency of the solvent trap it is recommended that the exhaust tube is routed directly upwards from the bottle for the first metre before being routed to the extraction hood or other fume disposal unit. Illustration – see page 6.

In some cases, where a high level of extraction is required, it is possible to remove the exhaust cap and connect a 2” extraction hose directly to the rear of the instrument. In this situation, ensure the exhaust hose has a downward slope away from the PL-ELS 1000 so that any condensed solvent is carried away from the unit.



The exhaust must be extracted to a suitable fume extraction system

1.5.3 Communication Connections

1.5.3.1 Control I/O connector

The PL-ELS 1000 can be connected to auxiliary equipment to pause or stop the operation of a pump or autosampler if the PL-ELS 1000 reports an error condition.

The PL-ELS 1000 is equipped with 2 contact closures, which are normally open and 2 TTL logic interfaces; one active low and one active high.

The PL-ELS 1000 can also be autozeroed remotely.

Table 1.1: Control I/O connector

	I/O Description	Pin No.
Outputs	Contact closure – normally open	1 — 9
	Contact closure – normally open	2 — 10
	TTL Active low	11
	TTL Active high	12
	Ground (to case)	15
Inputs	Remote A/Z (Active low)	8
	Remotely switching between varies operation modes is available on request.	

1.5.3.2 Heated transfer line

The heated transfer line is connected to the rear of the instrument by two connectors; the heated transfer line power output and the heated transfer line thermocouple feedback. The thermocouple is a K-type where the brown (+ve) is connected to pin 5 and the blue (-ve) is connected to pin 1. If the thermocouple is disconnected and the heated transfer line is switched on, the instrument reports an error condition (**TFER**).



The heated transfer line must be connected prior to turning on the being switched on in the control software

1.5.3.3 Serial RS232 Connector

The PL-ELS 1000 is fitted with a standard RS232 (DTE-DCE) 3-wire serial interface.

The PL-ELS 1000 can be controlled from a PC using the PS232 interface and the PL-ELS 1000 graphical control software from Polymer Laboratories Ltd. Refer to the control software manual for the operating instructions.

1.5.4 Gas Connection

The instrument should be supplied with clean, dry nitrogen gas at a head pressure of 60psi (PL-ELS 1000) or 100psi for the PL-ELS 1000 μ . A 4mm push-in connector is provided at the rear of the instrument for a convenient connection to the gas source. The PL-ELS 1000 is fitted with a non-return valve in the gas line after the mass flow controller (MFC) to prevent solvent siphoning back into the gas line.

1.5.5 Fluid Connection

The eluent from the chromatography system is connected to the RHS of the instrument via the low dead volume Valco bulkhead connector provided.

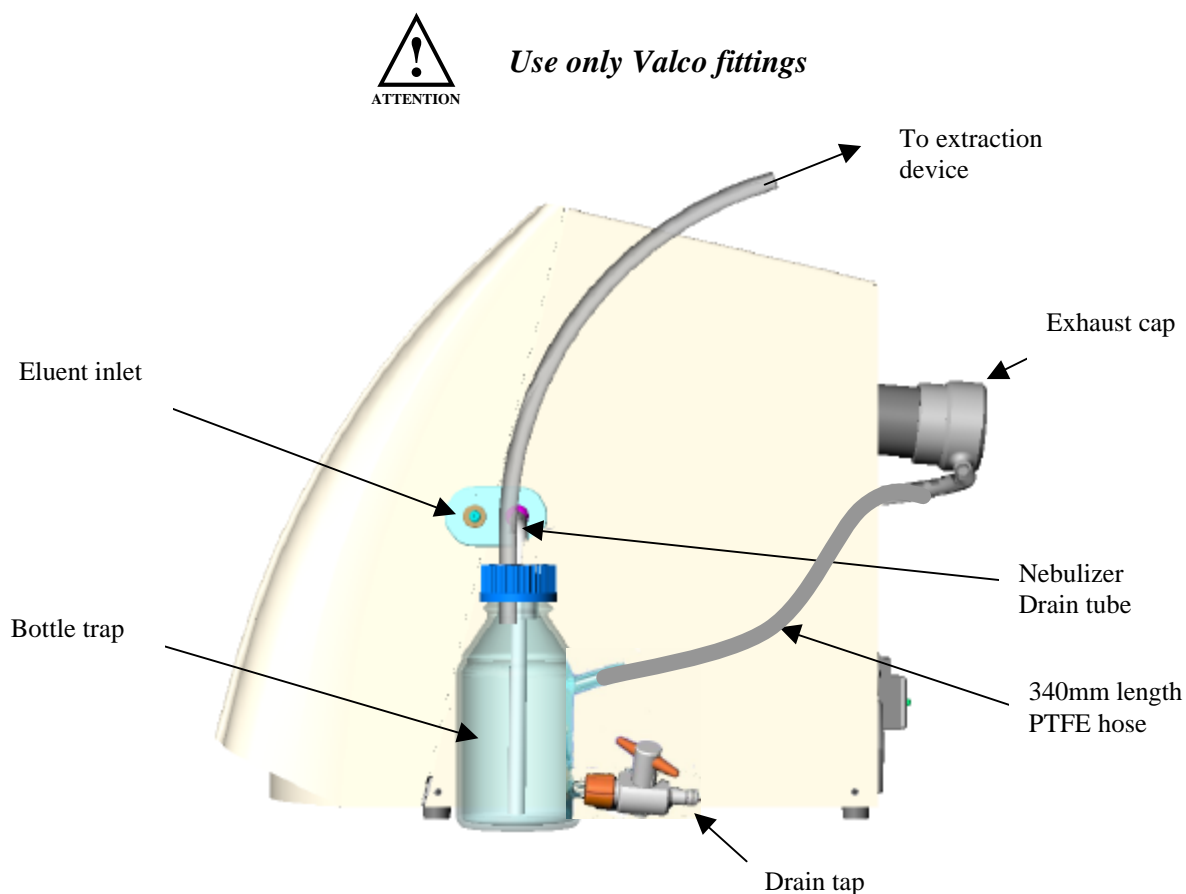


Figure 1.2 PL-ELS 1000 Fluid Connections

The 230mm length of convoluted PTFE hose (supplied) is pushed onto the nebulizer drain tube. The open end should be submerged in the waste bottle solvent to prevent air being sucked into the system.

When installing a PL-ELS 1000 with a bottle trap with drain port as pictured below, the PTFE hose is normally connected as indicated in Figure 1.3.

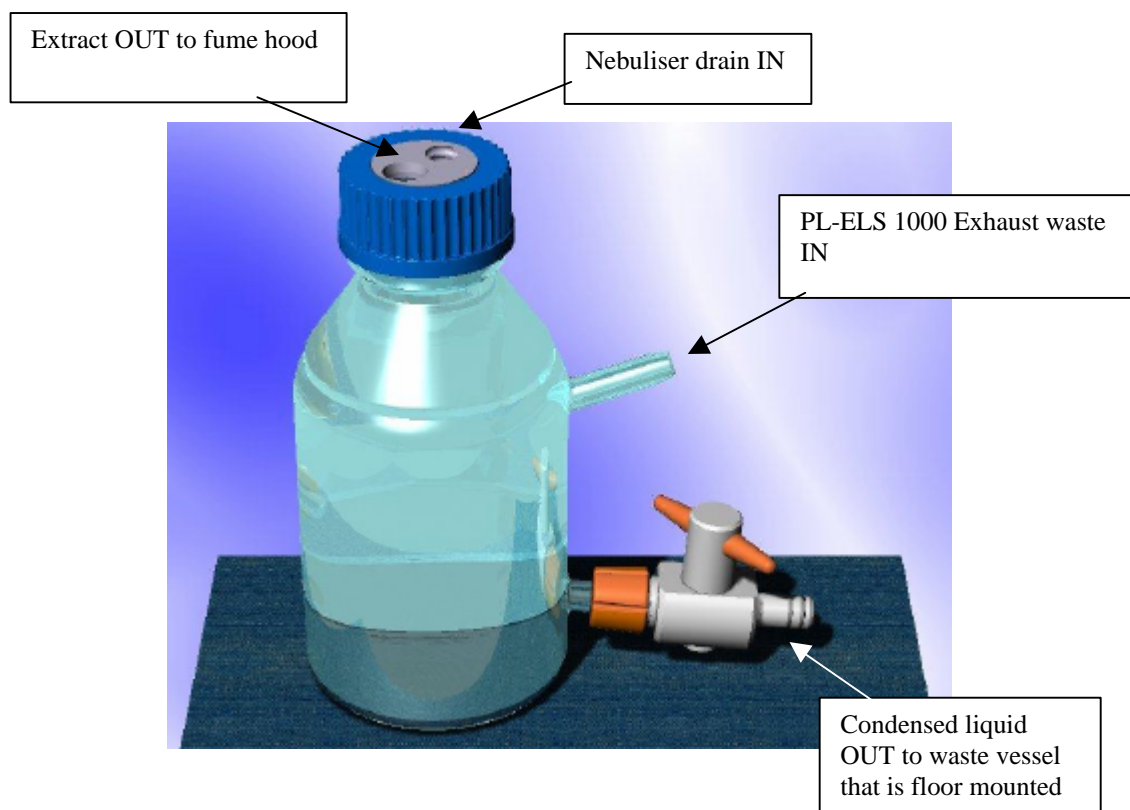


Figure 1.3 Self Draining bottle

Precautions

1.5.6 Extraction

For correct operation and optimum performance of the PL-ELS 1000, the unit must be vented to a fume hood or other means of vapour disposal using the exhaust hose provided. Due to the low gas flows and relatively low temperatures employed in the instrument, a majority of solvents readily condense in the exhaust hoses. It is therefore advisable to exercise caution when moving or disconnecting the exhaust hoses. Care must be taken to ensure that any condensation in the extraction hoses cannot re-enter the instrument.



Condensed solvent may be retained in the exhaust ducting; be careful not to spill this solvent when removing the ducting from the rear of the instrument

There is no fire risk within the instrument itself, as electrical components and supply are quite separate from the evaporation chamber – the risk arises on escape of fumes into the open laboratory. Also, if fumes are allowed to circulate within the working environment, then they could be harmful to operators and to sensitive equipment within the laboratory. Judicious use of the exhaust hose provided and careful disposal of fumes will prevent any problems.

1.5.7 Electrical

If your PL-ELS 1000 unit may be fitted with a surge safe device, please remove this device before attempting a standard portable appliance test (P.A.T Test) on the instrument. The surge safe device if fitted will be seen protruding through the rear panel of the instrument (item 10 in figure 1.1). This device is designed to protect the instrument from high voltage line surges by filtering the incoming supply. However, on later units this device has been removed since further susceptibility testing has proved it is not necessary. On these units a blanking plate will be found in place of the surge safe device.



READ ABOVE SECTION BEFORE ATTEMPTING A P.A.T TEST

1.5.8 Flammable solvents

Since high temperatures are employed in the instrument Nitrogen gas is recommended for use as the nebulization gas when flammable solvents are used.

Chapter 2

System Description and General Operation

2.1 Basic Principles of Operation

A schematic diagram of the PL-ELS 1000 is given in Figure 2.1 showing the main components as discussed below.

2.1.1 Nebulization

The eluent stream enters the detector at the bottom of the evaporation chamber. The eluent inlet is connected to the nebulizer via a short length of stainless steel capillary tube, 0.25mm i.d. for the PL-ELS 1000 and 0.125mm i.d. for the PL-ELS 1000 μ . The eluent stream passes through the heated nebulizer and is fed perpendicularly into the incoming nebulizer gas stream. The gas shears the droplets from the liquid needle as they begin to form atomising the solution into a uniform dispersion of droplets which then pass as a continuous stream into the evaporator. The larger droplets or the inefficiently nebulized fraction collect around the entrance to the evaporator and then drain off into the collection bottle at the side of the instrument. The wholly nebulized fraction continues down the bore of the evaporation chamber.

2.1.2 Evaporation

After nebulization the atomised spray is propelled through the evaporation chamber assisted by the carrier gas. In the evaporator the solvent is evaporated from the atomised spray leaving a dry particle plume. A diffuser[†] located in the evaporator assists in the drying of the particles, by acting as an efficient heat exchanger. Furthermore the diffuser prevents ballistic particles reaching the scattering chamber and randomises the particle plume.

2.1.3 Detection

Light from a lamp is collimated and passed through the instrument at right angles to the direction of gas flow. A light trap is located opposite the source of light to capture the transmitted incident beam eliminating internal reflections within the instrument body. When pure solvent is being evaporated, only its vapour passes through the light path and the amount of light scattered to the photodetector is small and gives a constant response. When a non-volatile solute is present, a particle cloud passes through the light path, causing light to be scattered. This scattered light enters the optical aperture of the detection system and generates a signal response from the photodiode in real time. The quantity of light detected is dependent on the solute concentration and solute particle size distribution.

As the detection process is affected by the size of the atomised droplets, the rate of evaporation and the nebulizer gas flow, then it is important to maintain steady conditions both internal and external to the instrument. A constant gas supply (volume and pressure), consistent eluent flow rate, and proper venting of the exhaust should be ensured.

[†] International Pat. A, W098/10279

2.1.4 Theory

There are four main processes by which the path of electromagnetic radiation or light can change direction when passing through a medium containing a suspended particulate phase. These are:

Rayleigh Scattering
Mie Scattering
Reflection
Refraction

The importance of each of these processes depends on the radius of the particle (r) compared to the wavelength (λ) of the incident light. Rayleigh scattering is predominant when r/λ is $< 5 \times 10^{-2}$. When particle dimensions are greater than $\lambda/20$ they no longer behave as point sources, and Mie scattering becomes predominant. Once particle size approaches the wavelength of incident light then reflection and refraction begin to prevail.

In order to decide which mechanism is predominantly responsible for the “scattering” observed in the PL-ELS 1000, an estimate of the size of the particles involved compared to the wavelength of the incident light must be made:

$$D_0 = \frac{585\sqrt{S}}{u\sqrt{r}} + 597\left(\frac{m}{\sqrt{S}}\right)^{0.45} \left(\frac{1000Q}{Q_a}\right)^{1.5} = n_a D^3 / n_a D^2$$

where D_0 = mean drop diameter
 n_a = number of drops in the size range, with diameter D
 σ = liquid surface tension
 ρ = liquid density
 μ = liquid viscosity
 u = relative velocity between the gas stream and the liquid stream
 Q = volumetric flow rate of liquid
 Q_a = volumetric flow rate of gas

The particulate size may be varied by altering the gas velocity, the eluent flow rate, the temperature of the nebulizer and also the initial solute concentration. It is evident from experiments and calculations that the particle radius (r) is approximately equal to or greater than the wavelength of light (λ). This suggests that the “scattering” is predominantly due to reflection and refraction.

Changes in the solute concentration and variations in the atomiser gas pressure influence the solute particle size. This relationship gives the instrument a maximum sensitivity around $r/\lambda = 4$. Detection declines rapidly when values for r/λ are above 5 or below 2.5. When $r/\lambda < 2.5$ the interference effects typical of Mie scattering cause the deflected light to be low in intensity at the measuring angles. As the particles increase in size, reflection and refraction become dominant and sensitivity increases. A further increase in the particle size causes the ratio of surface area to volume to decrease thus the sensitivity decreases.

The distribution tails as diameter increases, the largest particle in a distribution generally reaching twice that of the mean. Consequently, although there is undoubtedly some Mie and

Rayleigh scattering, the observed phenomena are predominantly due to reflection and refraction since the majority of the particles are larger than the incident wavelength.

The relative importance of refraction and reflection can be understood by examining the effects of the incident light on a single spherical particle whose equilateral axis lies in the same plane as the photodetector and light source. With this configuration, refraction is of greater significance than reflection. The majority of organic compounds have refractive indices between 1.3 and 1.5. Changes in the refractive index within this range will not greatly affect the quantity of light reaching the detector. This accounts for similarities in the sensitivity of the instrument to various compounds.

Therefore, this evaporative analyser is useful as a pure mass detector, providing that the material under investigation is non-volatile under the operating conditions of the instrument.

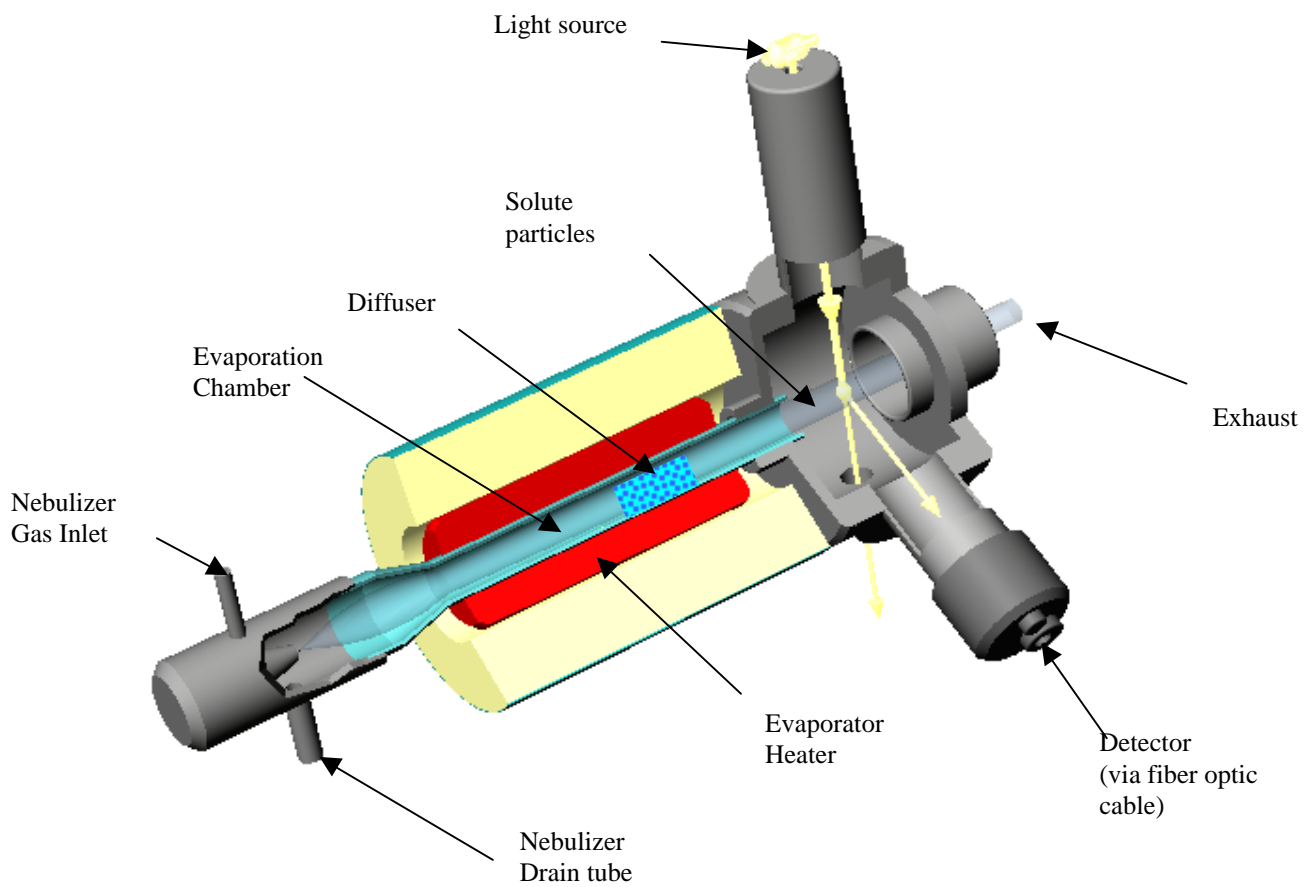


Figure 2.1 Principle of Operation

2.2 Operational Parameters

To achieve optimal performance from the instrument, certain operational conditions should be met. An understanding of the Nebulization/evaporation process will aid in selecting the best operational parameters for that application. In theory, optimal performance is achieved by producing a specific solute droplet size with a narrow size distribution. Manipulation of the nebulized droplet size is performed by adjusting the eluent flow rate, evaporation temperature, nebulizer temperature and nebulizing gas flow rate.

The eluent flow rate is generally determined by the chromatography, and is usually the least manipulated parameter. The effect of eluent flow rate on the instrument operation is dependent on the volume of eluent that must be volatilised and on the droplet particle size. A higher eluent volume may require a higher evaporative temperature, a higher gas flow rate, or both, to achieve optimal conditions. The PL-ELS 1000 is designed to receive eluent flow rates up to ~5 ml/min.

The operational temperature range for the PL-ELS 1000 is 30°C to 300°C. It is recommended that the instrument be operated at the lowest temperature required for complete evaporation of the eluent. Only volatile eluents should be used. Non-volatile eluent components (such as non-volatile salts) will not evaporate and may collect inside the instrument, requiring removal and cleaning of the evaporator tube. The optimal temperature setting is dependent on the eluent. The volume of eluent or eluent flow rate may also affect the temperature selection.

The nebulizing gas flow rate is the most varied parameter when optimising performance. The detector operates using clean dry nitrogen at flow rates from 0-2SLM. Adjustments to the nebulizer gas flow rate can have marked effect on detector response. Reducing the gas flow generally increases the particle diameter and so increases sensitivity. The maximum response is achieved when reflection and refraction increase as the particle size approaches the optimum diameter.

2.3 Instrument Controls

The front view of the instrument is shown in figure 2.2 below.

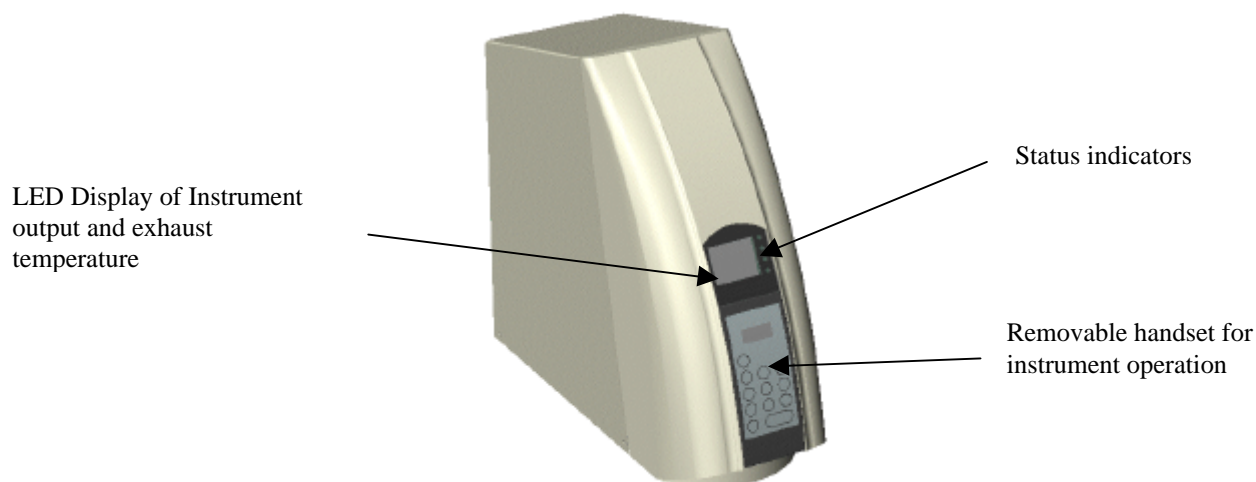


Figure 2.2 PL-ELS 1000 Front View

2.3.1 Power On/Off

The mains power switch is located at the rear of the instrument just above the mains cable. Re-booting the PL-ELS 1000 can be accomplished by switching off the detector, waiting a moment (typically until the cooling fan stops rotating), and then switching back on. As with any electronic device, if the unit is cold from shipment or storage in a non-heated area, it is recommended that the unit be allowed to come to room temperature before switching on.

2.3.2 Display

In normal operating conditions the upper value shown in red on the PL-ELS 1000 display is the detector output voltage in Volts. The lower display in green is the exhaust temperature in °C, measured in the exhaust tube. However, when the instrument is communicated with via the handset or the PL-ELS 1000 control software the top display momentarily shows the function being changed and the bottom display the value of that function.

2.3.3 LEDs

The four lights at the right hand side of the display show the operational status of the instrument. Each of the lights can be RED, ORANGE or GREEN. Generally RED indicates error or warning conditions, ORANGE changing conditions and GREEN ready. Full descriptions of the status conditions are given in “Modes of Operation” below.

2.3.4 Handset

The handset located on the front of the instrument is removable and operates the instrument via an Infra-red link. The handset can be used either docked on the front of the instrument or remotely up to 8 metres away from the instrument. To remove the handset from the instrument, lift the bottom of the handset out and away from the unit.

In general to operate the instrument press one of keys on the handset while pointing the handset towards the instrument (if using it remotely). The handset will then display the current setting of the parameter/function selected. If communication between the handset and the instrument was unsuccessful then three audible beeps rather than just one will be heard from the handset.



NOTE

‘Beep’ = Successful**‘Beep’ – Pause – ‘Beep’ = Unsuccessful**

To change the current setting, use the up and down arrows to change the parameter displayed on the handset. To action this change, again point the handset at the instrument and press **SEND**.

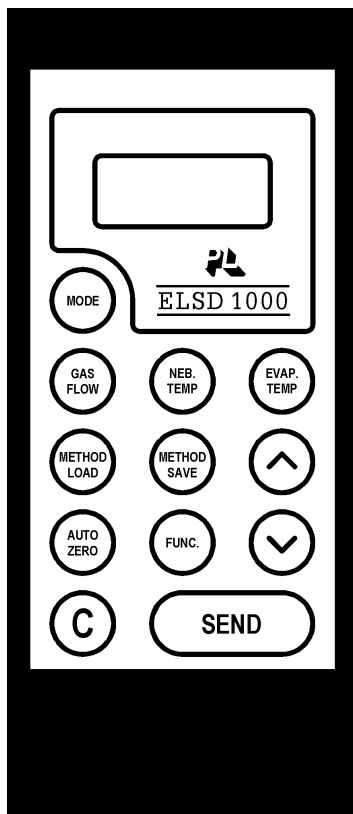
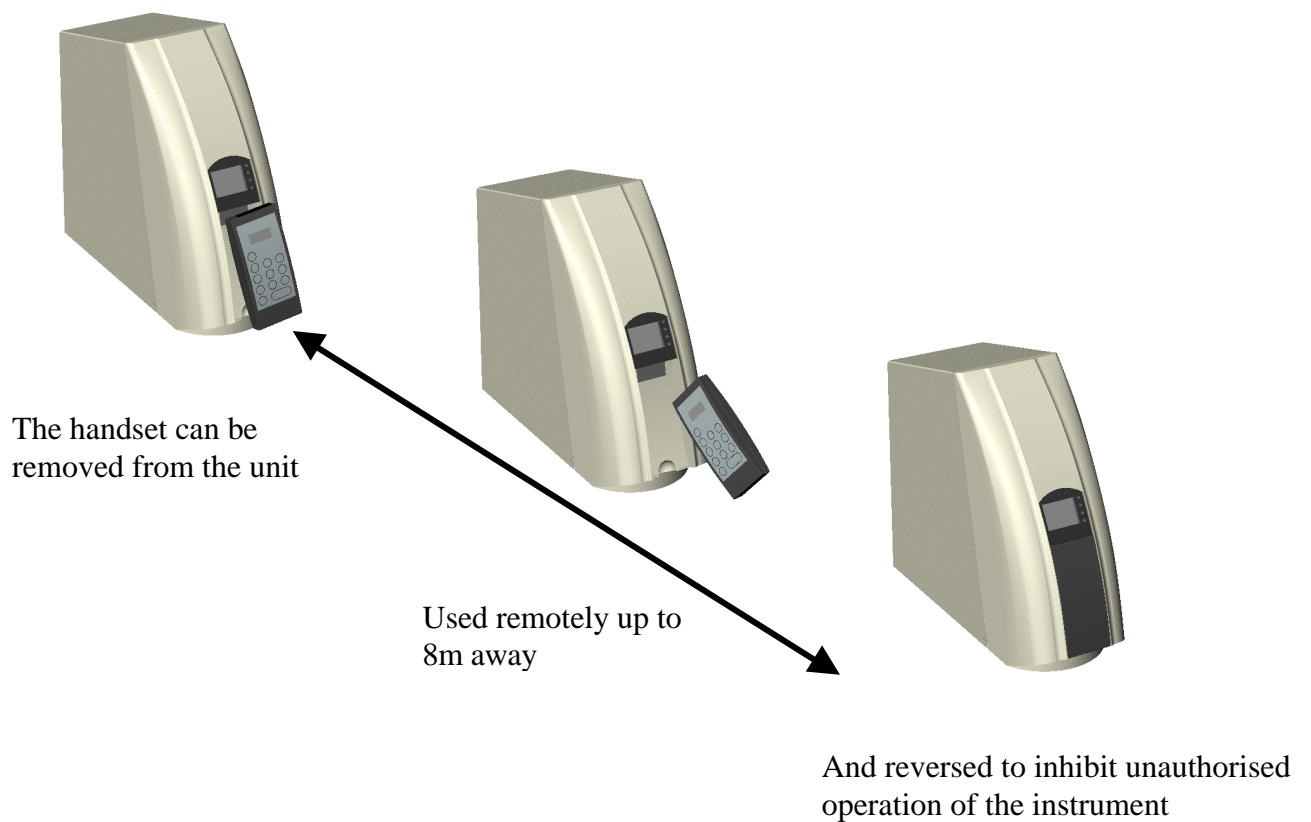


Figure 2.3 Dockable handset



The table below briefly describes the functions of each of the key commands.

Table 2.1 Function Keys for the Dockable Handset

Key	Description	Options
MODE	Modes of instrument operation	STANDBY RUN GAS SAVE SLEEP
GAS FLOW	Displays and changes the Nebulizer gas flow	0.0-2.0SLM (PL-ELS 1000) 0.0-1.0SLM (PL-ELS 1000 μ)
NEB. TEMP	Displays and changes the Nebulizer temperature	30-220°C
EVAP. TEMP	Displays and changes the Evaporator temperature	30-300°C
METHOD LOAD	To load a stored method	1-10
METHOD SAVE	To save a method following editing	1-10
AUTO ZERO	Auto zero the instrument	
FUNC.	Special functions	Heated Transfer Line ON/OFF Clear Error Time constant
C	Clears handset display	
SEND	Actions displayed parameter/function	
C & FUNC	Matches the instrument identification to the handset.	
C & EVAP. TEMP	Displays and changes the heated transfer line temperature	
^	Scrolls up through choices available	
∇	Scrolls down through choices available	

2.3.5 The Graphical User Interface

The PL-ELS 1000 can also be controlled from a PC using the control software provided. Details of the installation of this software, set-up and operation are provided with the software. Both control systems (handset and PC control) work in synergy, each communicating with the instrument independently.

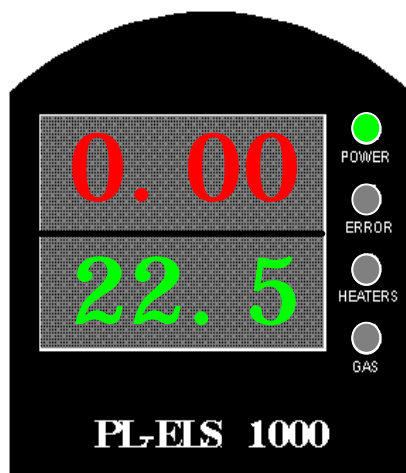
2.3.6 Modes of Operation

The PL-ELS 1000 can be operated in one of four operational modes; STANDBY, RUN, GAS SAVE and SLEEP, each of which is described below.

To display the current mode and/or select a new mode, press the **MODE** button on the handset. The current mode will now be displayed on the handset. By using the arrow keys, \uparrow and \downarrow , scroll up or down until the desired option is displayed. Press the **SEND** button to select this new mode of operation. The instrument acknowledges the command by momentarily displaying the mode of operation.

2.3.6.1 *STANDBY*

The STANDBY mode is the “ground state” of the PL-ELS 1000 instruments, which is initiated automatically after power on. In this mode of operation the heaters are off, the lamp is off and the gas flow is set to 0.1SLM. The STANDBY mode gives the user a control platform in which to setup the operational parameters; Gas flow, Neb. Temp and Evap. Temp before switching the unit into a RUN mode. In STANDBY mode only the power LED is illuminated

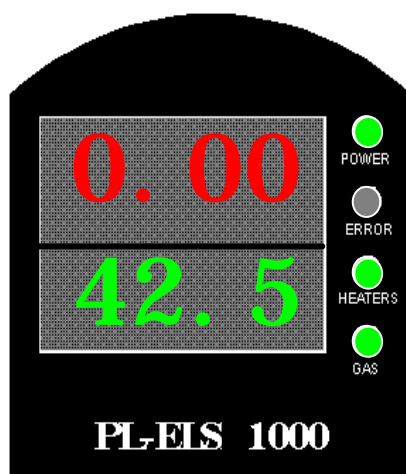


STANDBY MODE

- ◆ Heaters off
- ◆ Gas flow set to 0.1SLM
- ◆ Instrument can not be autozeroed

2.3.6.2 *RUN*

The RUN mode is the normal operational mode. The instrument is now being controlled at the set temperatures and gas flow, and the system is fully operational. During heat ramp, denoted as RUN, the heater LED flashes orange. At steady state, denoted as RUN (OK), the heater LED is illuminated green.

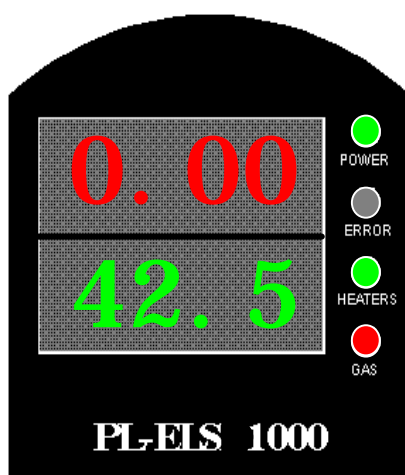


RUN (OK) Mode

- ◆ Heaters on and controlling set temperatures
- ◆ Gas on and operating at set flow
- ◆ Instrument is fully operational and at steady state conditions

2.3.6.3 GAS SAVE

The gas save mode controls the operational temperatures but sets the gas flow to a nominal low setting (0.1SLM) to conserve gas and recycle solvent when chromatography is non-essential. This mode can be used to idle the system when the liquid flow is turned off or to recycle the solvent into the nebulizer drain during the baseline sections of the chromatogram. The mode is denoted by an illuminated red GAS status LED.



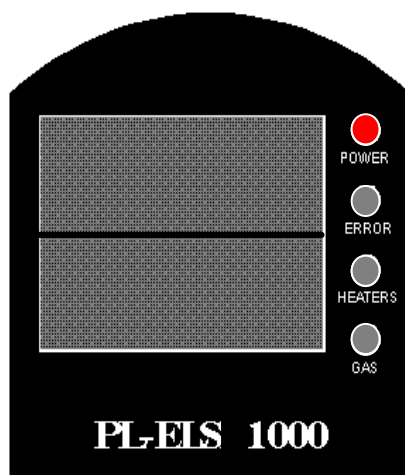
GAS SAVE Mode

- ◆ Heaters on and controlling set temperatures
- ◆ Gas flow controlled at 0.1SLM
- ◆ Instrument in fully operational and steady state conditions
- ◆ Eluent is not nebulized efficiently and therefore recycled into the nebulizer drain

2.3.6.4 SLEEP

The SLEEP mode is used for instrument shut down without the necessity to turn off the power and the gas supply. In SLEEP mode only the power LED is illuminated and coloured red.

SLEEP mode should only be selected for temporary shut down of the instrument e.g. overnight. If the instrument is not to be used for an extended period of time it is recommended that the unit is completely shut down.



SLEEP Mode

- ◆ Instrument shut down
- ◆ Mass flow controller closed

2.3.7 Using Methods

On power up, the PL-ELS 1000 control system defaults to the previous set of operating conditions. However, custom conditions for specific applications may be stored and loaded and subsequently used as the default conditions. Ten methods are stored onboard the instrument which have been developed for the applications displayed in the table below.

Table 2.2 Preset Methods

Solvent System	Method #	GAS FLOW	NEB. TEMP	EVAP. TEMP
THF, Chloroform, Acetone, Hexane	1	1.0	40	80
Water	2	1.5	90	120
Water/Acetonitrile (≥90% water)	3	1.5	80	90
Water/Acetonitrile (≤20% water)	4	1.0	40	70
Acetonitrile, Cyclohexane	5	1.0	40	90
DMF, DMAC	6	1.5	80	180
TCB, OCP, DMSO, NMP	7*	1.5	150	270
Unused methods	8-10	1.0	30	30

*The heated transfer line option will normally be required with these solvents, and should be set at the same temperature as the chromatography system. **Note:-** The conditions tabulated above are only one of many possible conditions that work for the given eluent. Eluent flow rate, gradient operation and the sample being analysed generally dictate the optimum conditions necessary for any application. Thus it is recommended that the optimisation guidelines are followed (Section 3.3 Optimisation Protocol) to achieve the best results.

2.3.7.1 Loading a Method

To load one of the 10 pre-set methods press **METHOD LOAD**. Using the $\uparrow\downarrow$ keys scroll up or down to the required method number and then press **SEND**. The instrument will acknowledge the change by momentarily displaying the method number. This method has now been down loaded as method 0 and may be edited without changing the stored method.

2.3.7.2 Saving a Method

Once the optimum conditions for a particular application have been established they can be stored as a method number for future use. To save a set of control conditions press **METHOD SAVE**, scroll to the required number using the $\uparrow\downarrow$ keys and press **SEND**. The instrument will acknowledge the change by momentarily displaying the method number. To keep track of your customised methods a “Customised Methods Table” is supplied in Appendix 1

2.3.8 The Heated Transfer Line

The heated transfer line is an optional item that is supplied by Polymer Laboratories Ltd. (Part # 091/24629/220 or 091/24629/110 – the last three digits denoting the operating voltage of your PL-ELS 1000). It is designed for operation with the PL-ELS 1000 for connection to a chromatograph running at elevated temperature. The line should be used for the analysis of semi-crystalline polymers (e.g. polyolefins) to prevent the polymer sample precipitating during the transfer from the column oven to the detector.

The 1/16” OD, 0.010” ID tube that runs through the center of the heated transfer line is connected between the chromatography system and the eluent inlet port on the right hand side of the PL-ELS 1000. The power and thermocouple cables coming from the heated transfer line are simply connected to the dedicated connectors at the rear of the unit. Once the heated transfer line

has been installed, it is controlled by the PL-ELS 1000. To activate the control press **FUNC**, scroll up or down using the **↑↓** keys until **TFER ON** is displayed. Now press **SEND** to activate the command. The set temperature of the heated transfer line can be displayed and changed by pressing the **C** and **EVAP. TEMP** keys simultaneously. The set **TFER** temperature will be displayed on the handset, which can be adjusted using the **↑↓** keys and activated by pressing the **SEND** key.

2.3.9 Setting the Instrument Time Constant

The time constant for the detector can be set to one of four settings: 0 (off), 1 (5secs), 2 (8secs) or 3 (15secs). The higher the number, the greater the effect of the electronic noise filter; however, too high a setting for a particular application may result in peak spreading and depression. It can be switched off by selecting 0. To set the time constant, press **FUNC**, **FUNC**, **FUNC**, until TIME C is displayed. Select the required value by scrolling, **↑↓** between 0-3 and then press **SEND**.

2.3.10 Error Conditions

The PL-ELS 1000 is equipped with a number of sensors and error checking facilities to ensure safe operation. If an error is detected the instrument gives an audible warning and a visible description of the error condition. In event of any error condition the unit defaults into the STANDBY mode in which the heaters, gas and lamp are turned off. A complete list of instrument errors and remedial actions are given in the troubleshooting section of this manual.

2.3.10.1 Clearing an Error

Once the source of the problem has been corrected the error can be cleared on the instrument by pressing **FUNC** twice followed by **SEND**.



Select **RUN** mode after clearing error

Chapter 3 Set-Up

3.1 General Considerations

The PL-ELS 1000 instruments should be thought of as detectors like any other designed for liquid chromatography. The main distinguishing feature is the ability to evaporate the solvent from the column effluent. Therefore, normal system set-up precautions should be remembered when starting to use the instruments. Any solvent intended for use with the PL-ELS 1000 should be fully miscible with any previously used in the liquid chromatograph; if there is any uncertainty, then a mutually miscible solvent should be run through the system as an intermediate liquid. The sample loop should also be flushed with miscible solvent where necessary. The intended eluent should be thoroughly degassed, contain no non-volatile salts or material and should be fully compatible with the column(s). All connections should be made with zero dead volume fittings and tubing with an I.D. $\leq 0.010''$. Micro bore fittings are available for the PL-ELS 1000 μ , to facilitate the connection to micro bore silica or PEEK tubing.

The PL-ELS 1000 itself requires nitrogen (purity >95%), or some other inert gas supply, capable of generating 60-100psi inlet pressure. If in-house nitrogen is not available then we recommend the use of a nitrogen generator (Model UHPPL4001), giving a constant uninterrupted supply of high purity gas. Air can be used with non-flammable solvent systems. The eluent of choice should be fully volatile at the chosen detector parameters – any non-volatilised eluent will increase baseline noise and reduce sensitivity.

3.2 Connecting the Detector

- Connect the power cord to the IEC inlet at the rear of the unit.

Check the operating voltage of your instrument, 110V 6A or 230V 3A, on the rating plate on rear of unit.

- Attach the gas inlet tube to the nebulizer gas inlet at the back of the instrument.

The gas connection is a 4mm OD push-in fitting. The gas should be dry, filtered and have a minimum inlet pressure of 60psi, 60-100psi is required to achieve the maximum operating instrument gas flow rate of 2 SLM throughout the temperature range.

- Connect the 340mm long exhaust hose between the exhaust cap at the rear of the unit, and the solvent waste collection bottle.

From the bottle trap the exhaust ducting should be taken vertically upwards for the first 1m of hose to assist the solvent recovery. Vent the exhaust hose into a well-extracted fume hood for optimum performance.

- Connect the instrument to the data recorder (computer, chart recorder, etc.) using the cable provided. Two output sockets are provided; the standard output gives a signal in the

range 0-10V, whereas the alternative output scales the signal to a 0-1V range. Both sockets are clearly labelled.

➤ Attach the nebulizer waste tube (part of the bottle trap) to the nebulizer outlet at the right hand side of the unit.

Add approximately 100mls of eluent to this bottle so that the end of the tube is submerged.

➤ Connect the column outlet to the eluent inlet at the side of the unit (1/16" Valco fitting) using a short length (10cm) of tubing (1/16" OD, 0.010" ID).

➤ Turn on the source gas to a pressure of about 60psi or greater.

➤ Switch on the PL-ELS 1000, and select a suitable evaporation temperature for the solvent being used; Table 3.1 suggests suitable settings for a wide range of commonly used solvents.

1. Press **EVAP. TEMP** to display the evaporation temperature set point.
2. Adjust the displayed temperature using the $\bar{U}\bar{U}$ keys.
3. Press **SEND** to action the change.

➤ Now set the gas flow to 1.5SLM and the nebulizer temperature to the temperature of the chromatography system or 30°C according to the three-step procedure above.

➤ Initiate heating by pressing the **MODE** button, select **RUN** by pressing the \bar{U} key and press **SEND**.

Whilst the unit is coming to temperature, the heating LED will flash orange. Once both the nebulizer and evaporator temperatures (and heated transfer line, if fitted and turned on) have equilibrated, the heating LED will be continuously illuminated green. The system is READY at this point.

➤ When the unit has equilibrated, the baseline should be checked to ensure that it is acceptable for the experiment. At this point, with no liquid flowing into the instrument, the noise should be no more than 200 μ V peak-peak. This verifies that the gas supply is clean and dry. Any spikes in the baseline are usually indicative of particulate matter or water in the gas supply.

➤ Turn on the eluent flow and allow the system to stabilise for approximately 15 minutes.



NOTE

The eluent flow can be turned on before the instrument has reached equilibrium. If the eluent is not efficiently nebulized it will exit the instrument from the nebulizer drain tube.

➤ Again check the baseline noise. It should not have increased significantly and should be ≤ 1 mV. Typically pure water should give no more than 300 μ V peak-peak, whilst pure organic solvents should be less than 200 μ V.



NOTE

Buffers and stabilisers can generate considerable offsets and increased noise levels.

If the baseline noise is excessive, then one of the following may be taking place:-

- poor evaporation due to evaporation temperature being too low

- poor nebulization due to temperature being too high and solvent boiling
- poor nebulization due to gas flow being too low

Any of these problems will cause the baseline to shift upwards, as the amount of scattered light within the instrument has increased (see Theory in above Section for fuller explanation). If the baseline continues to show unacceptable noise even when the evaporation conditions have been improved, and the solvent does not contain non-volatile species, then please refer to Chapter 5 Troubleshooting.

Where noise and all other conditions are acceptable, the instrument is ready to begin work.

Table 3.1 – Solvent Boiling Points and Suggested Evaporator Temperatures

SOLVENT	Boiling point(°C)	Evaporator Temp(°C)
1-Butanol	117.7	130
1-Chloronaphthalene*	260	300
2-Butanol	99.6	110
Acetic acid	117.9	130
Acetone	56.3	70
Acetonitrile	81.6	90
Benzene	80.1	90
Carbon tetrachloride	76.8	90
Chloroform	61.2	80
Cyclohexane	80.7	90
Dichloromethane	39.8	50
Dimethylacetamide	166.1	180
Dimethylformamide	153	170
Dimethylsulphoxide**	189	200
Ethanol	78.5	90
Formic acid	101	115
Heptane	98.4	110
Hexane	68.7	80
Hexanol	156.5	170
Methanol	64.7	70
Methyl ethyl ketone	79.6	90
N-methyl pyrrolidone	202	220
Ortho-dichlorobenzene	180.5	200
Pentanol	137.8	150
Tetrahydrofuran	66	80
Toluene	110.6	120
Trichlorobenzene*	214	250
Water	100	110

*heated transfer line normally required

** prolonged use with DMSO at elevated temperatures has been reported to cause extensive corrosion within the instrument and therefore its use is no longer recommended.



NOTE

The minimum heated transfer line temperature = temperature of chromatograph
The maximum heated transfer line temperature = boiling point of solvent – 10°C

The above table should only be used as a guide; the PL-ELS 1000 will require gas flow within the range 0.5-2SLM, and further optimisation of the method should be performed as described in the next section.

3.3 Optimisation Protocol

3.3.1 Nebulizer Gas Flow

An increase in nebulizer gas flow rate causes a decrease in signal response due to the resultant formation of smaller droplets that scatter less light. Therefore, lower gas flow rates tend to be more favourable since less gas is consumed and a better sensitivity is achieved. However, there comes a point at which this benefit is counterbalanced by the increase in baseline noise due to inefficient nebulization of the eluent resulting in large droplets. The particle size of these droplets will have become non-ideal, resulting in complex scattering mechanisms and poor detector performance. In general, gas flow rates of 0.5-1.5SLM tend to be a reasonable compromise between baseline stability and high reproducible response for eluent flow rates >0.5ml/min. Reducing the eluent flow demands a reduction in the nebulizer gas flow rate to maintain the optimum nebulized droplet size.

3.3.2 Evaporator Temperature

The effects of altering the evaporator temperature tend to be less dramatic than changing the nebulizer gas flow, although the temperature must be high enough to evaporate the solvent and to sufficiently dry the particle plume without having a detrimental effect on the sample being studied. If the temperature is too low then the solvent can saturate the diffuser resulting in high noise and spikes and if the temperature is too high the sample may be volatilised resulting in a small response.

3.3.3 Nebulizer Temperature

The nebulizer temperature is the least adjusted parameter. In the majority of cases the nebulizer temperature is set to the temperature of the chromatography system. However, increasing this temperature can improve instrument performance by increasing the efficiency of nebulization by reducing the viscosity and surface tension of incoming solvent. Setting the nebulizer temperature too high may result in a deterioration of detector performance due to solvent boiling in the nebulizer, giving rise to increased noise on the baseline due to spiking.

3.3.4 Procedure

Set the system at the minimum evaporator temperature (see Table 3.1 above for typical operating conditions) and the maximum gas flow rate. Introduce the solvent at the desired flow rate and while monitoring the baseline, incrementally reduce the gas flow rate until the baseline just starts to become noisy. If the baseline dramatically deteriorates when the gas flow is slightly reduced from the maximum setting, increase the temperature by 5°C until a noise free baseline is obtained. Once the evaporator temperature and gas flow have been optimised try increasing the nebulizer temperature to further improve the chromatography. In some cases, especially for volatile samples, it has been found that maximising the nebulizer temperature permits a lower evaporator temperature and thus an increase in sensitivity.

Chapter 4

Routine Maintenance

The PL-ELS 1000 is designed to be a low-maintenance instrument and does not generally require regular service. However, should the instrument performance deteriorate we recommend the following procedures to dry and clean the evaporator tube without dismantling the unit.

Trained personnel only should carry out maintenance inside the unit. Unauthorised access to the instrument will invalidate the instrument warranty.



WHEN DISCONNECTING TUBING FROM THE INSTRUMENT SOLVENT MAY LEAK OUT. PLEASE OBSERVE APPROPRIATE SAFETY PROCEDURES (E.G SAFETY GLASSES, GLOVES AND PROTECTIVE CLOTHING) ACCORDING TO THE SAFETY DATA SHEETS WHEN HANDLING SOLVENTS.



TO PREVENT PERSONAL INJURY, THE POWER CABLE MUST BE REMOVED FROM THE REAR OF THE INSTRUMENT BEFORE THE INSTRUMENT COVER IS REMOVED.

4.1 Drying the Diffuser

If the instrument has been operated at an incorrectly low evaporator temperature then the diffuser may become blocked with liquid. This is manifested by loss of signal, increased baseline noise and in the extreme case gas bubbling out of the nebulizer drain tube. If this happens then simply turn off the liquid flow, increase the gas flow to 2.0SLM and increase the evaporator temperature to approximately 50°C above the boiling point of the eluent used. The diffuser will be dried out and the instrument ready to use after approximately 15mins under these conditions. Reset the instrument to the correct operating conditions and allow to stabilise before continuing.

4.2 Cleaning Evaporator tube

If the evaporator tube becomes contaminated with non-volatile material resulting in poor chromatography it is recommended that the instrument is heated to high temperature and the impurity cleaned off by oxidation. To clean a unit set the evaporator temperature at 250°C, the nebulizer temperature at 100°C and the gas flow at 1.5SLM. Use air rather than nitrogen for the nebulizing gas and leave at temperature in the ***RUN*** mode for approximately 3-4 hours.



Do not pass solvent through the unit during burn procedure



ENSURE THAT THE INSTRUMENT IS AT EQUILIBRIUM UNDER THE ABOVE CONDITIONS BEFORE LEAVING THE INSTRUMENT UNATTENDED.

If cleaning the unit does not cure the problems then consult Polymer Laboratories Ltd for further assistance.

4.3 Information for Service Personnel

4.3.1 Heater PCB Fuses

There are 2 fuses fitted to the Heater PCB, one to protect the internal heaters and the other to protect the heated transfer line. Their ratings are:

FUSE 1: TR5 4A F 250V

FUSE 2: TR5 2A F 250V

4.3.2 Handset Batteries

The handset batteries are 3V Lithium AA size. Ensure that this type of battery is fitted at all times, and be careful to observe polarity as shown on the label in the handset battery compartment.



DISPOSE OF EXHAUSTED BATTERIES ACCORDING TO LOCAL WASTE DISPOSAL AND ENVIRONMENTAL REGULATIONS. DO NOT CRUSH, PUNCTURE OR INCINERATE.

4.3.3 Safety Earth Bond Screw

The screw located on the rear panel (#12, Rear View Diagram, Section 1.5) is for safety earth bonding.



THIS SCREW MUST BE FITTED TO ENSURE CONTINUED PROTECTION AGAINST ELECTRIC SHOCK

Chapter 5

Troubleshooting

If there is an error or fault, please refer to the following table. If you follow the recommended course of action and the result is not satisfactory, then please direct the matter to Polymer Laboratories or your local distributor.

5.1 Instrument Errors

Error Condition Displayed on LED	Possible Cause(s)	Remedy
EVAP Evaporator temperature	The evaporator temperature has exceeded the control temperature by an unacceptable margin	<ul style="list-style-type: none"> • Turn off the PL-ELS 1000 and allow the instrument to cool, if necessary, before rebooting. • If the problem persists consult Polymer Laboratories Ltd or your local agent
NEB Nebulizer Temperature	The nebulizer temperature has exceeded the control temperature by an unacceptable margin	<ul style="list-style-type: none"> • Turn off the PL-ELS 1000 and allow the instrument to cool, if necessary, before rebooting. • If the problem persists consult Polymer Laboratories Ltd or your local agent
EXH Exhaust Temperature	The exhaust temperature has exceeded the control temperature by an unacceptable margin	<ul style="list-style-type: none"> • Turn off the PL-ELS 1000 and allow the instrument to cool, if necessary, before rebooting. • If the problem persists consult Polymer Laboratories Ltd or your local agent
FAN Fan Fault	The cooling fan located at the front of the unit is not operating correctly	<ul style="list-style-type: none"> • Please ensure the fan is rotating freely and is not obstructed. The fan can be checked from underneath the front panel without disassembly. • Contact Polymer Laboratories Ltd or your local agent for further assistance
GAS Gas Error	The mass flow controller within the PL-ELS 1000 is unable to sustain the required flow rate	<ul style="list-style-type: none"> • Please ensure <ul style="list-style-type: none"> ➢ The gas is turned on at the source ➢ The inlet pressure is at least 60psi (higher inlet pressure may be required for higher temperatures and flow rates) ➢ There is not a gas leak between the source and the instrument ➢ If this problem persists consult Polymer Laboratories Ltd or your local agent for further advice.

LAMP Lamp Error	The lamp is not functioning within the correct specifications	<ul style="list-style-type: none"> • Please ensure the lamp is illuminated in both RUN and GAS SAVE modes and is off in STANDBY and SLEEP modes. The lamp is visible through the ventilation slots in the rear panel. • Contact Polymer Laboratories Ltd or your local agent for a replacement lamp (Part No. 094/30373)
OT Overtemperature Error	A controlled temperature within the instrument has exceeded the maximum allowable value	<ul style="list-style-type: none"> • Turn off the PL-ELS 1000 and allow the instrument to cool, if necessary, before rebooting. • If the problem persists consult Polymer Laboratories Ltd or your local agent
PIC PIC Error	A communication error has occurred within the unit	<ul style="list-style-type: none"> • Please switch off the instrument and reboot • If the problem persists consult Polymer Laboratories Ltd or your local agent for further advice
TFER Thermocouple Error	The heated transfer control system has been turned on and the heated transfer line thermocouple has not been connected	<ul style="list-style-type: none"> • Turn off the heated transfer line control system via the handset (Press FUNC , \bar{U} or \bar{U} followed by SEND) • If the problem persists consult Polymer Laboratories Ltd or your local agent for further advice
VAP Vapour Error	A solvent leak has been detected either internal or external to the instrument. For operation of the instrument in a solvent polluted environment the sensitivity of the vapour can be adjusted via the ELS PC control software	<ul style="list-style-type: none"> • Identify the location of the leak or the source of the high level of solvent vapour and remove before clearing the error. • If the source of solvent vapour is identified not to be associated with the instrument but still prevent the instrument operating correctly reduce the vapour sensor sensitivity via the PC control software. Note the vapour sensor sensitivity can not be changed from the handset. • If the problem persists consult Polymer Laboratories Ltd or your local agent for further advice
V.DEV Vapour sensor device	The vapour sensor device has malfunctioned and needs replacing	<ul style="list-style-type: none"> • Consult Polymer Laboratories Ltd or your local agent.

5.2 General Problems

Faults or Problems	Possible Cause(s)	Remedy
Baseline noise	The particle plume is not sufficiently dried in the evaporator tube.	<ul style="list-style-type: none"> • Increase the temperature of the evaporator by 10° intervals until the noise is acceptable
Baseline noise	Poor nebulization of solvent	<ul style="list-style-type: none"> • Increase the gas flow rate • Decrease the nebulization temperature
Baseline noise	Pump pulsations, especially in microbore applications where low flow rates are used.	<ul style="list-style-type: none"> • Use a pulse free pump • Increase the back pressure on the pump by fitting a back pressure column between the pump and the injection valve. • Use a pulse dampener directly after the pump in the system.
Baseline spikes	<ol style="list-style-type: none"> 1. Particulate matter in the gas supply 2. Column shedding 3. Poor nebulization 	<ol style="list-style-type: none"> 1. Filter the incoming gas, or change the supply 2. Replace column or fit an inline filter with a 0.2µm membrane filter directly after the column. 3. Nebulizer temperature may be too high and solvent may be boiling; reduce setting to recommended level
Low sensitivity and baseline noise	Diffuser saturated with solvent	<ul style="list-style-type: none"> • Stop the eluent flow and increase the evaporator temperature to 50°C above the current set temperature. Increase the flow rate to 2.0SLM and wait 15mins.
Large Baseline offset	<ol style="list-style-type: none"> 1. Light leakage through the exhaust tube 2. Inefficient nebulization 3. High concentration of non-volatile buffer or stabiliser 	<ol style="list-style-type: none"> 1. Ensure the exhaust cap/ducting is fitted and is light tight. 2. Increase the evaporator temperature and/or gas flow. 3. Use a lower concentration of stabiliser, unstabilised solvent or a more volatile buffer (ammonium acetate or ammonium formate)
Peak tailing	Eluent particles lingering in the optical chamber	<ul style="list-style-type: none"> • Increase gas flow rate • Increase extraction
Instrument Fails to zero	Offset too high or output unstable	<ul style="list-style-type: none"> • Ensure the exhaust cap/ducting is fitted and is light tight • Ensure the instrument is in RUN or GAS SAVE mode • Refer to local distributor or Polymer Laboratories
No communication with the handset	<ol style="list-style-type: none"> 1. Handset identification lost 2. Handset saturated with bright light. 	<ol style="list-style-type: none"> 1. Press C and FUNC simultaneously and press SEND 2. Reboot the PL-ELS 1000
No power	<ol style="list-style-type: none"> 1. Mains lead not connected 2. Fuse blown in mains lead plug 	<ol style="list-style-type: none"> 1. Attach mains lead to socket and inlet on rear of instrument 2. Replace fuse
No response (completely flat baseline)	<ol style="list-style-type: none"> 1. Data acquisition leads not connected 2. Light source inactive 	<ol style="list-style-type: none"> 1. Ensure connectors to computer or integrator are sound 2. Check lamp

Temperature error as soon as instrument powered on	Temperature probe fault or disconnected	<ul style="list-style-type: none">• Check RTD connections• Consult Polymer Laboratories Ltd or your local agent for further advice
--	---	---

APPENDIX 1

PL-ELS 1000 Quick User Guide

See next page

PL-ELS 1000 Quick Guide

The following is intended to be a quick guide for operation of your PL-ELS 1000. However, the Operator's Manual must be read before attempting to operate the PL-ELS 1000 so that all the instrument controls are identified.

1. Before turning on your PL-ELS 1000, ensure the following connections have been correctly and securely made to the instrument:-

Item	Instrument Connection	From/To	Comments
Power Cord	Power In	Mains Socket	The correct operating voltage for your PL-ELS 1000 is displayed on the rear panel
Gas Inlet Tube	Nebulizer Gas	Nitrogen generator or gas Cylinder fitted with regulator	The gas must be clean and dry with a head pressure of at least 60psi. Nitrogen is recommend for volatile solvents.
Exhaust Hose	Exhaust Tube	Fume Hood	
Analogue output cable	Output	Data collection system	Red +ve, Black -ve
Serial Cable	RS 232	Computer Com port	Optional instrument control by PL-ELS 1000 Control graphical interface
Nebulizer Drain Bottle	Nebulizer drain tube	Place drain tube in the 500ml bottle provided	Ensure the end of the tube is submerged in eluent.
Eluent Inlet	1/16 th Valco fitting	Chromatography column	The connecting tubing should be as short as possible and 0.01" ID.

2. Turn on the power.
3. Once booted press **GAS FLOW**, **NEB. TEMP** or **EVAP. TEMP** to view set operating conditions on the handset. When displayed change the value using the \bar{U} and \bar{U} keys and press **SEND** to update the set condition.
4. Alternatively select one of the 10 preset methods by pressing METHOD LOAD followed by \bar{U} or \bar{U} to select the required method number and press **SEND**. The solvent systems to which these methods are intended, are tabulated below. All methods can be edited and we recommend the updated methods are recorded on the Method record sheet enclosed in the manual.

Solvent System	Method #	GAS FLOW	NEB. TEMP	EVAP. TEMP
THF, Chloroform, Acetone, Hexane	1	1.0	40	80
Water	2	1.5	90	120
Water/Acetonitrile ($\geq 90\%$ water)	3	1.5	80	90
Water/Acetonitrile ($\leq 20\%$ water)	4	1.0	40	70
Acetonitrile, Cyclohexane	5	1.0	40	90
DMF, DMAC	6	1.5	80	180
TCB, OCP, DMSO, NMP	7*	1.5	150	270
Unused methods	8-10	1.0	30	30

It is recommended to operate the instrument at the lowest reasonable temperature to avoid evaporation of the sample and to increase sensitivity. Increasing the temperature will reduce baseline noise.

*The heated transfer line option is normally required with these solvents, and should be set at the same temperature as the chromatography system. **Note:-** All these methods can be edited, and the edited version can be saved by selecting **METHOD SAVE**.

5. Press **MODE** to display the current instrument status and the \bar{U} key to display **RUN**. Now press **SEND**. The instrument will start heating during which time the HEATER LED will flash orange. Once at steady state conditions the HEATER LED will be on continuously and GREEN indicating the instrument is ready to use.
6. Turn on the solvent pump and wait ~ 15mins following stabilisation for the instrument to become primed and conditioned for optimum performance.
7. On completion of your work, turn off your solvent pump, put the PL-ELS 1000 into STANDBY mode and turn off the power. If the instrument is switched into SLEEP mode, rather than turning off the power, then the instrument's mass flow controller will also turn off the gas flow.

APPENDIX 2

Method Record Sheet

Chromatography Conditions	Eluent	Eluent flow	Method #	Neb. Temp	Evap. Temp	Gas Flow	Date Stored	Comments
			1					
			2					
			3					
			4					
			5					
			6					
			7					
			8					
			9					
			10					

APPENDIX 3

PL-ELS 1000 Test procedure

The PL-ELS 1000 is factory tested for sensitivity and baseline noise according to the following test procedure.

Detector Settings

Nebulizer Temperature: 90°C *Gas flow rate:* 1.5 SLM

Evaporator Temperature 120°C *Time Constant:* Off [0]

Chromatography Conditions – direct injections

Sample: Glucose *Concentration:* 0.5 mg/ml

Eluent Water *Injection Volume:* 50µl

Column: 2m length of 0.010" ID tubing *Eluent flow rate:* 1.0 ml/min

Pass Specifications for the Glucose/water chromatogram test

Area (PL –Response.time)	>26000 (10V output)
Area (equiv.-mV.sec)	>1250
Noise (mV)	<0.3

APPENDIX 4

Spare Part Listing

The following list of spare parts was current at the time of issue of this manual. Please contact your local distributor or Polymer Laboratories for an up-to-date price list and availability.

Description	Part Number
Heated transfer line (110V)	091/24629/110
(230V)	091/24629/220
PL-ELS 1000 Control Software	091/24882
1/16 Bulkhead Assembly	094/32486
1/16 Lagging	092/24808
Handset Batteries	094/32858
Block Insulation	092/24807
Rear Panel Connector Assembly	091/24859
Control PCB Assembly	091/24928
Evaporator tube fitted with diffuser	091/24922
Cooling Fan	094/32747
Front Panel	091/25052
Fuses 110V (pk of 5)	094/30778
230V (pk of 5)	094/30777
Mass Flow Controller Assembly	091/24806
Handset	091/24778
Evaporator Heater Assembly 110V	091/24900/110
230V	091/24900/220
Heater Driver PCB	091/24925
Power Inlet Filter	094/32750
Gas Inlet Plug Assembly	091/24899
Interface PCB	091/24926
Light Trap Assembly	091/24902

Description	Part Number
Mirror Assembly	091/24878
Nebulizer Assembly (w/o heaters and RTD)	091/24706
Nebulizer Insulation	092/24920
Display PCB assembly	091/24923
Photodetector assembly	091/25064
Power supply	094/32845
Firmware upgrade kit	091/24906
Surge safe	094/32670
Tungsten Lamp	094/30373
Nebulizer heater assembly 110V	091/24941/110
230V	091/24941/220

Parts specific to the PL-ELS 1000 μ

Description	Part Number
Micro-bore 1/16 Bulkhead Assembly	094/33258
Micro-bore Front Panel	091/25485
Micro-bore Nebulizer Assembly (w/o heaters and RTD)	091/25487
Micro-bore mass flow controller assembly	091/25486

To order please contact your local office