# OPERATOR'S MANUAL

# FOR

# PL-ELS 2100/2100Ice

# EVAPORATIVE LIGHT SCATTERING DETECTOR

Version 1.5

Polymer Laboratories

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# Documentation Revision History

6/26069

| Revision # | Date | Section Changed | Changes | Approval |
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| Draft | April 2003 | All | Originated | |
| Version A<br>(Version 1.0) | 16th May 2003 | All | Release | Reviewed by SJOD, NJW, PEC and EM |
| Version B<br>(version 1.01) | 26th August 2003 | | I/O Controls –remote A/Z Control software reference Storage/drying details QC Specifications | SJOD<br>Reviewed SB |
| Version C<br>(version 1.02) | 25th March 2004<br>19/4/04 | 1.2, 1.6, 2.4, 4.2<br>2.3 | Specifications AZ input & Gas /Gain<br>Gas management<br>Software include Gain /Smooth<br>Cleaning Evap tube change from S/BY to RUN<br>Addition of parameter menu info | SJOD/AW |
| Version D<br>(version 1.1) | 7th May 2004 | Table 2.1 & page 17 | Min gas flow 0.9SLM and S/By flow1.2SLM<br>Replaced new screen pics | |
| Version E<br>(version 1.1E) | 3/9/04 | Table 1.1<br>section 1.9 | Contact closure pins 4 & 10<br>Pump stop 3 & 10 | |
| Version F (1.2) | 25/07/05 | | French address | |
| Version G (1.3) | 31/10/05 | Safety Practices, 1.10, Appendix 6 | Cleaning & Decontamination Procedures, $N_2$ mandatory for flammable solvents & pump stop should be used | PEC |
| Version H (1.4) | March 2006 | Safety Practices, Health & Safety, 1.2, 1.4 & 1.5 | Digital output included. Protective earth connection requirement on mains supply. Power switch & coupler accessible at all times. IEC 60825-1 classification of LED source. | SB/PEC |
| Version I<br>(1.5) | May 2006 | All | PL-ELS 2100/2100 Ice included | SB<br>Reviewed SJOD |

Polymer Laboratories

# DECLARATION OF CONFORMITY

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

declare that the product:

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**Evaporative Light Scattering Detector**

***PL-ELS 2100 Part number 0860-0110/0860-0240***
***PL-ELS 2100 Ice Part number 0860-01110/0860-01240***

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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<br>Installation category II<br>Pollution degree 2 |
| | EN61010 –2 – 010 | Class 2<br>REINFORCED INSULATION |
| EMC: | EN 61326:1998 | |

S O'Donohue

Date: 17th May 2006

Dr. S.O'Donohue, Head or R&D –Instrumentation

# PL-ELS 2100/2100 Ice 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:-

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

WARNING

### ELECTRICAL HAZARDS

Opening of instrument panels may expose potentially dangerous voltages. Disconnect the instrument from all power sources before opening protective panels. Access to the interior of the instrument should be restricted to properly 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.
Ensure the power switch and appliance coupler at the rear of the instrument remain easily accessible at all times.

WARNING

### 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.

WARNING

### CLEANING

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 Polymer Laboratories (see Appendix), please check with Polymer Laboratories that the proposed method will not damage the equipment.

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

The unit should not be stacked more than two high without additional means of support and suitable precautions should be taken to prevent the units being knocked over

Eluents containing concentrations of >1% Acetic acid should be avoided

# HEALTH AND SAFETY

## PL-ELS 2100/2100 Ice

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

- 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.

- 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.

- 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.

- This instrument contains a light source, which has been classified according to the methods specified in IEC 60825-1 Ed 1.2 as a CLASS 1 LED PRODUCT.

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


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# Chapter 1
# General Information

## 1.1 Introduction

The PL-ELS 2100/2100 Ice evaporative light scattering detector or mass detector is a unique and highly sensitive detector for semi-volatile and non-volatile solutes in a liquid stream. It is mainly used as a concentration detector for High Performance Liquid Chromatography (HPLC). The solvent stream containing the solute material is nebulised and carried by a gas flow through an evaporation 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 "mass" or concentration of the solute particles passing through the light.

The PL-ELS 2100/2100 Ice 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 2100/2100 Ice must be the last in series if used in conjunction with other detectors. If the PL-ELS 2100/2100 Ice 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** | | Blue LED 480nm. Class 1 LED product. |
| **Detector** | | Photomultiplier tube with additional digital signal processing |
| **Temperature Range:** | | |
| Evaporator | | |
| PL-ELS 2100 | | Ambient-120°C (1°C increments) |
| PL-ELS 2100 Ice | *(Ambient temp <25°C)* | 10-80°C (1°C increments) |
| | *(Ambient temp >25°C)* | 15-80 (1°C increments) |
| Nebuliser | | Ambient-90°C (1°C increments) |
| **Gas requirements** | Flow rate | Up to 3.25 SLM @60 psi @25°C<br>With integrated automatic, controlled gas shut-off valve |
| | Pressure operating range | 60 – 100 psi (4-6.7 bar) |
| | Maximum Pressure | 100 psi (6.7 bar) |
| **Eluent Flow rate** | | 0-5 ml/min |
| **Analogue Output** | | 0-1V FSD |
| **Digital Output** | | 24bit digital data, 10Hz via serial port |
| **Communication** | | Serial I/O (RS232) |
| | Outputs | 1 User Contact closure<br>Pump stop: 1 contact closure<br>1 TTL +ve<br>1 TTL –ve |
| | Input | Auto zero |
| **Instrument Operation** | | Graphical Vacuum Fluorescent display<br>5 button keypad<br>10 predefined methods<br>PC based method utility program |
| **Power Requirements** | | 90/120V AC or 220/250V AC 50/60 Hz 2A max |
| **Detector Status** | | Standby, Run |
| **Size** | Unpackaged | 200x450x415 mm (wxdxh) |
| | Packaged | 360x700x600mm (wxdxh) |
| **Weight** | Unpackaged | 11kg (PL-ELS 2100)<br>13kg (PL-ELS 2100 Ice) |
| | Packaged | 16 kg (PL-ELS 2100)<br>18kg (PL-ELS 2100 Ice) |

## 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.

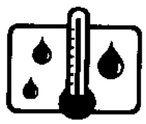
## 1.4 Packing list

*Standard Items*

- PL-ELS 2100 or PL-ELS 2100 Ice detector
- Manual for PL-ELS 2100/2100 Ice 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 (2m)
- Exhaust hose (PVC– 2.0m)
- Waste hose (Tygon SE200– 7cm)
- Solvent waste container (500ml)
- Valco Nut and ferrule (1/16”)

## 1.5 Site Preparation Check List

*Environmental Conditions*



Temperature 10 to 35°C  (50 to 95°F)
At constant temperature

Avoid positioning in direct sunlight

Humidity 10-80%

*Power*

USA and Japan 115V (AC) ±10%
50/60 Hz, 2A max, with a protective earth connection.

Europe 230V (AC) ±10%
50/60 Hz, 2A max, with a protective earth connection.

*Gas Supply*

Gas: Nitrogen (98% purity or better and filtered to 0.2µm)
*Notes:*

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

Gas flow up to 3.25 SLM @ 60 psi @25°C

Pressure operating range: 60 – 100 psi (4-6.7 bar)

Maximum Pressure: 100 psi (6.7 bar)

*Extraction Requirements*

During 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 (13mm ID PVC 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

## 1.6 Connections

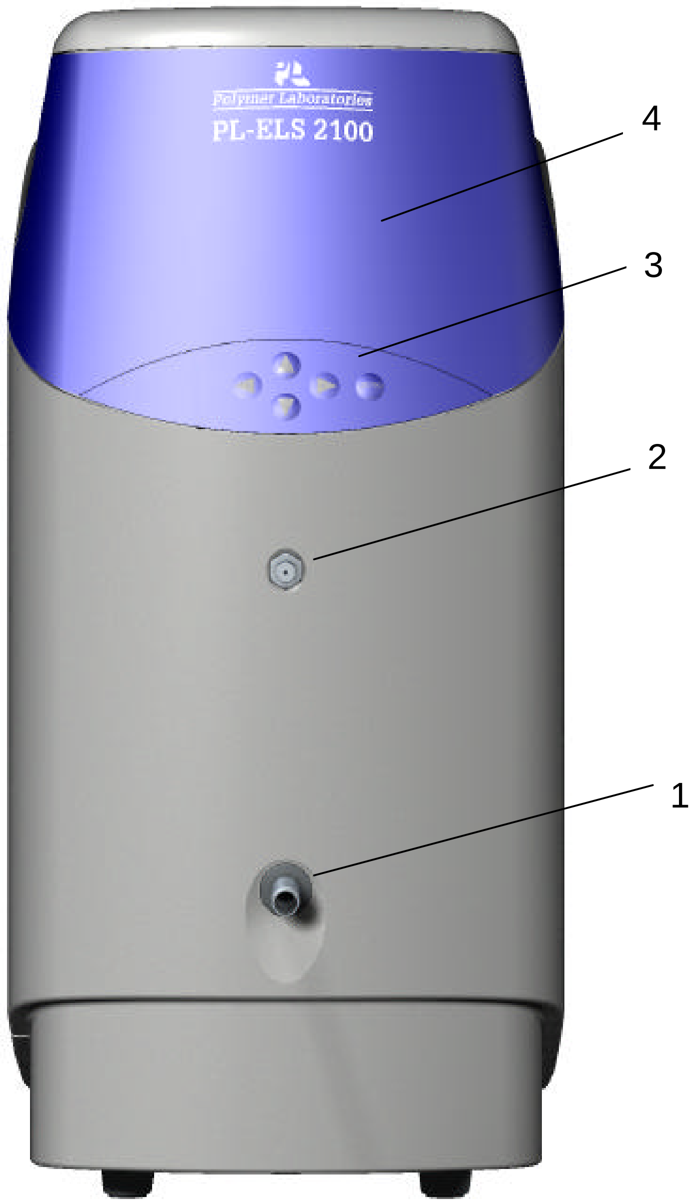



*Figure 1.1 Front View of PL-ELS 2100/2100 Ice*

1. Waste Outlet
2. Eluent Inlet
3. Keypad
4. Graphical Display

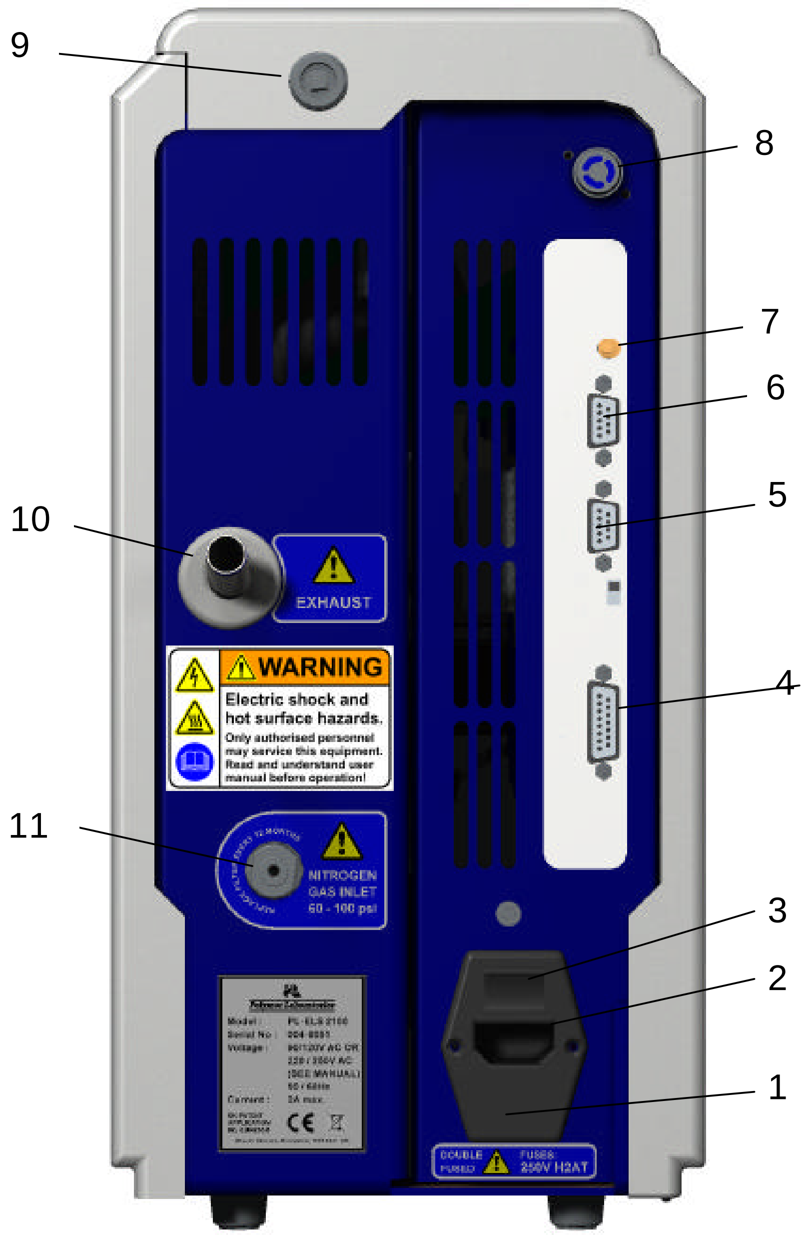



*Figure 1.2 Rear View of PL-ELS 2100/2100 Ice*

1. Fuses
2. Mains input
3. Mains switch
4. Connector control I/O –15 pin D type female
5. Control Firmware flash upgrade connector
6. Serial RS232 connector- 24bit digital output
7. Detector output-1V
8. External Vapour Sensor
9. Panel Lock
10. Exhaust port
11. Supply gas inlet

## 1.7 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) or 230V (AC) use two 250V H 2A T fuses

ATTENTION *This unit is double - fused.*

WARNING *RISK OF FIRE, REPLACE FUSES AS MARKED!*

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

## 1.8 Extraction

The PL-ELS 2100/2100 Ice is provided with tubing for venting the exhaust gases and vapours, and so does not need to be placed in a fume cupboard. Instead, the exhaust hose provided must be attached to the rear of the unit and vented to a fume hood or other disposal unit. Ensure the exhaust hose has an upward slope from the PL-ELS 2100/2100 Ice so that any condensed solvent is collected in the waste bottle at the front of the unit and to prevent it accumulating in the tubing.

ATTENTION *The exhaust must be extracted to a suitable fume extraction system*

## 1.9 Connections

### Control I/O connector

The PL-ELS 2100/2100 Ice can be connected to auxiliary equipment to pause or stop the operation of a pump or autosampler if the PL-ELS 2100/2100 Ice reports an error condition.

The PL-ELS 2100/2100 Ice is equipped with 2 normally-open contact closures and 2 TTL logic interfaces; one active low and one active high.

The PL-ELS 2100/2100 Ice can be auto-zeroed remotely.

NOTE

*Pump stop facility must be employed if instrument is to be left unattended, or if units are stacked*

*Table 1.1: Control I/O connector*

| | **I/O Description** | **Pin No.** |
|---|---|---|
| Outputs | User contact closure – normally open | 4 & 12 |
| | Pump stop contact closure – normally open | 3 & 10 |
| | Pump stop - TTL Active low | 2 & ground |
| | Pump stop - TTL Active high | 9 & ground |
| | Ground (to case) | 1, 5, 6 ,11 |
| Inputs | Remote A/Z | 7 & ground [firmware version 1.0.15. Units with serial # 004-161 and before will also require a wiring modification on the main PCB] |

The instrument is supplied with a connector for the I/O socket in order to make appropriate connections; however, a cable can be purchased for this from Polymer Laboratories (Part # 0860-0055).

### Serial RS232 Connector

The PL-ELS 2100/2100 Ice is fitted with a standard RS232 (DTE-DCE) 3-wire serial interface.

The serial RS232 connector provides a 24bit (10Hz) digital output for connection to a chromatographic acquisition device.

The PL-ELS 2100/2100 Ice can also be controlled from a PC using the RS232 interface and the PL-ELS 2100/2100 Ice graphical control software from Polymer Laboratories Ltd. Refer to the control software online manual for the operating instructions. If controlling the instrument from a PC with a serial port or USB port is required. If the USB port is used a serial to USB adapter is also required (0860-0620).

### Gas Connection

The instrument should be supplied with clean, dry nitrogen gas at a minimum head pressure of 60psi. A 4mm push-in connector is provided at the rear of the instrument for a convenient connection to the gas source.

To prevent against unnecessary gas usage, an automatic but controlled gas shut off valve is integrated into the gas inlet manifold. This will only allow gas to pass into the instrument when the instrument is operating. Should the instrument default to a standby mode the gas valve will close.

NOTE

*The gas inlet valve will be closed when the instrument is first powered on, and will only open once the instrument is set to RUN mode*

### Fluid Connection

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

ATTENTION

*Use only Valco fittings*

The liquid inlet port is connected directly to the nebuliser by a short length (190mm) of capillary tube giving a delay volume from port to nebuliser tip of ~5µl.

## *1.10 Precautions*

### Extraction

For correct operation and optimum performance of the PL-ELS 2100/2100 Ice, the unit must be vented to a fume hood or other means of vapour disposal using the exhaust hose provided. The exhaust hose should be routed upwards to allow condensed solvent to collect in the waste bottle at the front of the instrument.

ATTENTION

*Condensed solvent may be retained in the exhaust hose, 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.

## Flammable solvents

**WARNING**

*Since high temperatures may be employed in the instrument Nitrogen must be used as the nebulisation gas when flammable solvents are used.*

*The pump stop facility should be connected directly to your pump or chromatography system controller when flammable solvents are used.*

# Chapter 2
# System Description and General Operation

## 2.1 Basic Principles of Operation

### Nebulisation

The eluent stream enters the detector at the bottom of the evaporation chamber. The eluent inlet is connected to the nebuliser via a short length of stainless steel capillary tube. The eluent stream passes through the heated nebuliser and is mixed with the incoming nebuliser gas stream. The mixed gas and eluent stream form an aerosol plume containing a uniform dispersion of droplets that then passes as a continuous stream into the evaporator. The larger droplets or the inefficiently nebulised fraction collect around the entrance to the evaporator and then drain off via the waste outlet into a collection bottle. The wholly nebulised fraction continues down the bore of the evaporation chamber.

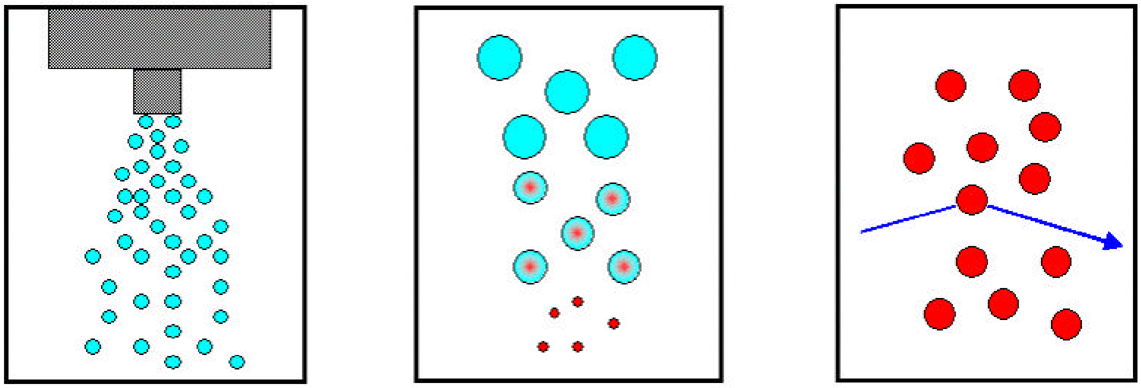
### Evaporation

After nebulisation 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, acting as an efficient heat exchanger, prevents ballistic particles reaching the scattering chamber and randomises the particle plume.

### Detection

Light from an LED is 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 photomultiplier is small and gives a constant baseline 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 gas flow, 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.



1. NEBULISATION 2. EVAPORATION 3. DETECTION

*Figure 2.1: Principles of Operation*

## 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 ($\lambda$) of the incident light. Rayleigh scattering is predominant when $r/\lambda$ is $< 5\text{x}10^{-2}$. 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 2100/2100 Ice, an estimate of the size of the particles involved compared to the wavelength of the incident light can 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 |
| | $\sigma$ | = | liquid surface tension |
| | $\rho$ | = | liquid density |
| | $\mu$ | = | 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 nebuliser and 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 ($\lambda$). 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 $r/\lambda$ 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.

## *2.2 Operational Parameters*

To achieve optimal performance from the instrument, certain operational conditions should be met. An understanding of the nebulisation/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 nebulised droplet size is performed by adjusting the eluent flow rate, evaporation temperature and 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 2100 & 2100 Ice are designed to receive eluent flow rates up to 5 ml/min, at ambient temperatures even for 100% water.

It is recommended that the instrument be operated at the lowest temperature required for complete evaporation of the eluent. Optimal temperature settings will be dependent on the volatility of the analyte and not 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 gas flow rate is the most varied parameter when optimising performance. The detector operates using clean dry nitrogen at flow rates up to 3.25 SLM. Adjustments to the gas flow rate can have marked effect on detector response. 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 1.1.

### Power On/Off

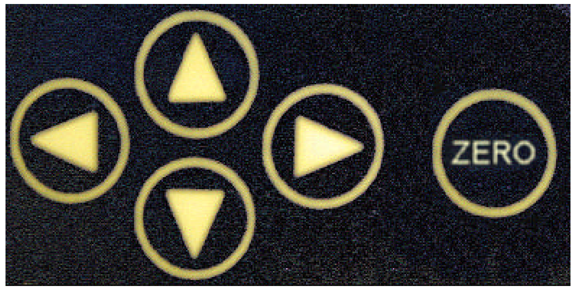
The mains power switch is located at the rear of the instrument just above the mains cable. Re-booting the PL-ELS 2100/2100 Ice can be accomplished by switching off the detector, waiting a moment (typically until the cooling fan stops rotating), and then switching back on. As with any electronic device, 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.

### Display

The graphical display on the front of the instrument indicates the current method, current status of the instrument, evaporator temperature, nebuliser temperature, gas flow and output. Operating parameters can be altered via the interactive menu bar at the bottom of the display.

### Keypad

The four arrows on the front of the instrument are used to navigate within the menu bar. The zero key is used to auto-zero the display.




### Interactive menu bar

To change the current settings, use the arrow keys to navigate across the menu bar until the desired option is flashing. Using the up/down arrow keys alter the parameter to the desired setting. When completed return to the lock position to action any changes made.


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

*Table 2.1 Menu bar functions*

| Function | Description | Options |
|---|---|---|
| ↓ | Access to the parameter settings menu | |
| MODE | Modes of instrument operation | STANDBY<br>RUN |
| METHOD | Displays and changes the instrument method | DEFAULT<br>1-10 programmable methods* |
| EVAP | Displays and changes the Evaporator temperature | Off, 25-120°C (PL-ELS 2100)<br>Off, 10-80°C (PL-ELS 2100 Ice) |
| NEB | Displays and changes the Nebuliser temperature | Off, 25-90°C<br>(in 1°C increments) |
| GAS | Displays and changes the gas flow | 0.90-3.25 SLM |
| 🔓 or 🔒 | Home (or lock) position displays actual values and actions any changes made in the menu options<br>The locked icon ( 🔒 ) will be displayed with the instrument is controlled by the PC based software | Real time display can be accessed by pressing the up and down arrow keys when in this position. |

* The method editor software supplied with the instrument enables the methods to be edited and stored on a PC and downloaded to the instrument (see section 2.4).

### Parameter Setting Menu

The parameter settings menu, which is accessed from the front screen by selecting the ↓ symbol and then pressing the ↓ arrow key, allows changes to the following three parameters:-


| Function | Description | Options |
|---|---|---|
| ↑ | Return to main menu | |
| Pmt | Gain adjust for the signal output | 1-10 (default 1) |
| Smth | Smoothing -Digital time constant | 1-50 (default 1) |
| LED | LED Intensity- Controls the output intensity (brightness) of the light source | 0-100% (default 100) |

**Pmt** (*Gain*)

This parameter sets the factor by which the out-put signal is amplified or divided once captured by the detector. The gain setting does not change the sensitivity of the detector, but merely amplifies or divides the captured signal by the inputted factor. The gain can be adjusted from 1 to 10 in increments of 0.1.

When setting a Pmt (or Gain) setting both the signal and noise are simply amplified by the value set. The raw signal output displayed on the parameter screen when the values are changed will reflect this increase or decrease in signal amplification. However, the instrument output and the output displayed on the main operating screen are automatically zeroed to 10mV, following a Pmt change and thus the recorded baseline position will remain unchanged. The magnitude of the baseline noise will be the obvious indication to the change in Pmt.

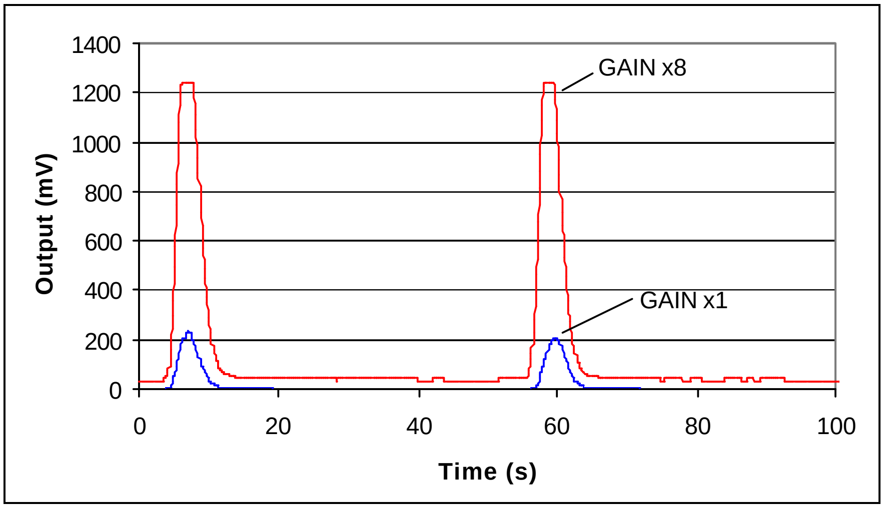



*Figure 2.3.1 Example of Changing the Pmt from 1 to 8*

**Smth (*Smoothing*)**

The data is continuously collected at a rate of 10 points per sec (10Hz) which can be averaged to produce a smoother out-put. The smoothing width set is the number of data points over which the data is averaged and can be regarded as a digital time constant. For most applications the default value of 1 is satisfactory, however for noisy or spikey data then it may be beneficial to average the raw data by increasing this smoothing parameter. The maximum smoothing width that can be applied is 50. The value is changed in increments of 1.

NOTE

*Due to the averaging of data narrow peaks may become broadened when smoothing is applied*

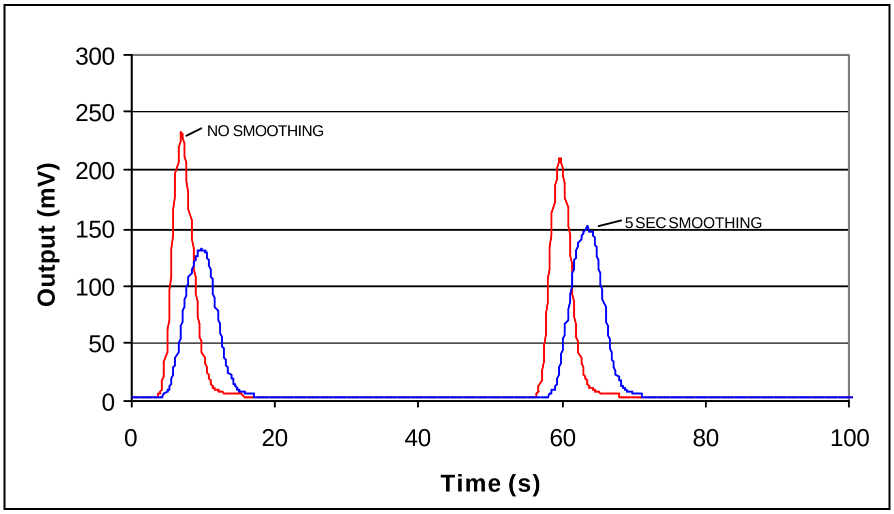
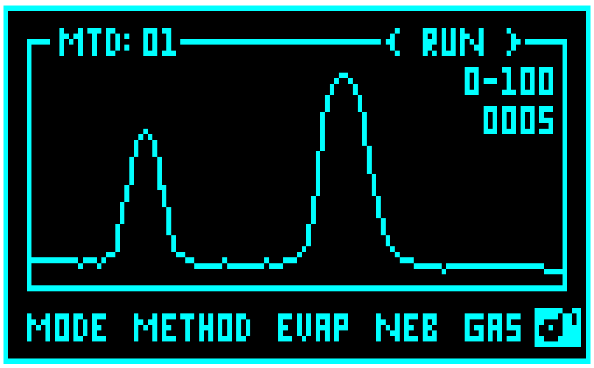



*Figure 2.3.2 Example of smoothing data*

**LED- (***LED Intensity)*
The LED intensity enables the brightness on the LED to be adjusted from 0(off) to 100%(default). This feature is extremely useful if the sensitivity of the detector needs to be reduced, bringing large peaks back on scale.

## Real Time Monitoring

The real-time output of the photomultiplier can be displayed graphically to provide the user with a means of monitoring detector output without data acquisition. Access to this display is achieved by pressing the up/down arrow keys whilst in home (lock) mode. Further ▲ or▼ key presses changes the display range.




## Modes of Operation

The PL-ELS 2100/2100 Ice can be operated in two modes; STANDBY and RUN, both of which are described below:

To display the current mode and/or select a new mode, highlight the ***MODE*** function on the instrument display. The current mode will now be displayed on the screen. Using the ▲▼ keys, scroll up or down until the desired option is displayed. The instrument acknowledges the command by displaying the mode of operation in the top right hand corner of the screen.

### *STANDBY*

The STANDBY mode is the "ground state" of the PL-ELS 2100/2100 Ice instrument, which is initiated automatically after power on. In this mode of operation the heaters are off, and the gas manifold valve is closed. The STANDBY mode gives the user a control platform in which to set-up the operational parameters (gas flow, nebuliser and evaporator temperatures) before switching the unit into RUN mode. The instrument will default to STANDBY mode should an error occur on the instrument

When the instrument is switched from RUN mode to STANDBY mode, following a command or error, then the gas management system is invoked and the gas set to minimum flow of 1.2SLM for 15minutes before the gas manifold valve is closed. This minimum "blanket" gas is enough to nebulise and evacuate solvent should the instrument default to STANDBY mode with solvent still flowing [firmware 1.1.8 and greater].

ATTENTION

*If the Instrument is left in Standby mode for longer than 15mintes, gas flow to the unit is stopped to minimise gas usage. It is strongly recommend that the pump stop from the I/O connector of the ELSD is connected to the HPLC pump to prevent solvent flooding the detector should an error occur*

### *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 heating or cooling the instrument will display 'NOT READY' to show the system has not reached the set conditions. When the instrument has equilibrated 'READY' will be displayed and the instrument is ready for use.

NOTE

*Using the instrument in 'NOT READY' status may not produce consistent results according to the set parameters, as the system is not equilibrated.*

## Using Methods

On power up, the PL-ELS 2100/2100 Ice control system defaults to the parameters set in the default ('XXX') method. Modification of the default method is permitted using the front keypad on the instrument. Ten custom methods can be stored onboard for specific applications, but cannot be edited via the keypad on the front of the instrument. Custom methods can be created using PL-ELS 2100/2100 Ice method editor software and subsequently downloaded onto the instrument.

### *Loading a Method*

To load one of the 10 set methods highlight ***METHOD***. Using the ▲▼ keys scroll up or down to the required method number. The instrument will acknowledge the change by displaying the method number in the top left hand corner. Editing of this method is prohibited via the front keypad; this can only be carried out using the method editor software (see section 2.4). Ten methods are stored onboard the instrument reflecting typical instrument conditions as displayed in the following table.

| **Method Number** | **Evaporator temp (°C)** | | **Nebuliser temp (°C)** | | **Gas Flow (SLM)** | |
|---|---|---|---|---|---|---|
| ELSD Model | PL-ELS 2100 | PL-ELS 2100 Ice | PL-ELS 2100 | PL-ELS 2100 Ice | PL-ELS 2100 | PL-ELS 2100 Ice |
| 1 | 0 | 30 | 0 | 30 | 1.6 | 1.6 |
| 2 | 30 | 25 | 30 | 30 | 1.6 | 1.6 |
| 3 | 50 | 20 | 30 | 30 | 1.6 | 1.8 |
| 4 | 50 | 15 | 50 | 30 | 1.6 | 2.2 |
| 5 | 70 | 20 | 50 | 80 | 1.6 | 1.2 |
| 6 | 90 | 40 | 50 | 40 | 1.6 | 1.4 |
| 7 | 90 | 50 | 50 | 50 | 1.2 | 1.0 |
| 8 | 120 | 80 | 50 | 60 | 1.2 | 1.0 |
| 9 | 120 | 80 | 90 | 80 | 0.9 | 0.9 |
| 10 | 120 | 80 | 90 | 90 | 2.8 | 3.0 |

*Table 2.2: Preset Methods*

### Error Conditions

The PL-ELS 2100/2100 Ice 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, LED & gas are turned off. A complete list of instrument errors and remedial actions are given in the troubleshooting section of this manual.

#### *Clearing an Error*

Once the source of the problem has been corrected, the error message will be cleared automatically on the instrument display.

NOTE

*Select RUN mode after clearing error*

## 2.4 PL-ELS 2100/2100 Ice PC Software

Two software utilities are provided with your PL-ELS 2100/2100 Ice; control and method editing software. Both these applications are installed from the CD provided when the setup.exe program is run. The software will automatically identify the model of ELSD attached to the PC and install the appropriate software version.

NOTE

*ELSD Software is only compatible with Windows 2000 & XP Pro*

## Control Software-Overview

This WindowsÔ-based graphical interface offers a second and complementary level to total detector control. An intuitive single control panel provides simplistic control as well as a comprehensive monitoring system. Operational parameters can be easily manipulated, saved or loaded by using the flexible Methods Editor enabling rapid set-up and custom method archiving.

## Configuring your PC and Detector

The PL-ELS 2100/2100 Ice requires a free Serial (RS-232) communications port (1 to 255) on your PC. Most computers are supplied with at least one serial port as standard. If your PC does not provide a serial port or you are already using the existing serial ports for other devices, then there are a couple of options available to you:

*1. Use a Universal Serial Bus Interface (USB)*

If your PC has one or more Universal Serial Bus (USB) connectors then you can use a "USB – Serial Port Adaptor" (part # 0860-0620), which provides a Serial Port connection to your PC. The Universal Serial Bus interface is supported on:

- Windows 98
- Windows ME
- Windows 2000
- Windows XP

NOTE

*USB is NOT Supported on NT 4.0. You may require extra software to use USB on Windows*

*2. Adding an Extra Serial Card to your PC - Using Multiple Serial Ports*

Multiple Port Serial cards are available, which allow 4, 8 and 16 extra serial ports to be added to your PC using a single PCI card.

## Connecting the Detector to your PC

- Ensure that the PL-ELS 2100/2100 Ice is switched on and operating normally.
- Make sure you have one free and valid RS-232 communications port (1 to 255).

- Connect a serial port on your PC to the port labelled "RS232" on the rear of the PL-ELS 2100/2100 Ice

## Configuring the ELS Control Software

On starting the PL-ELS2100/2100 Ice Control Software, the program will ask to specify the correct Serial port to be used for communications. The ELS 2100 control software needs to be configured for the Serial port you are using on your PC.

The first time the PL-ELS2100/2100 Ice Control Software is used, the detector will be allocated to Serial Port COM 1.

## Operating the ELS Control Software

Control of the PL-ELS 2100/2100 Ice is through the simple control window. Within the control window, the status of the detector is displayed, together with options to control the various instrument parameters. The control window is effectively divided into **three** main sections:

1.CONTROL

- View the current mode of operation
- Change the current mode of operation between Standby and Run
- Change the Current Method of operation between a stored custom method and the default method ‘XXX’
- Change Parameters in the default method ‘XXX’

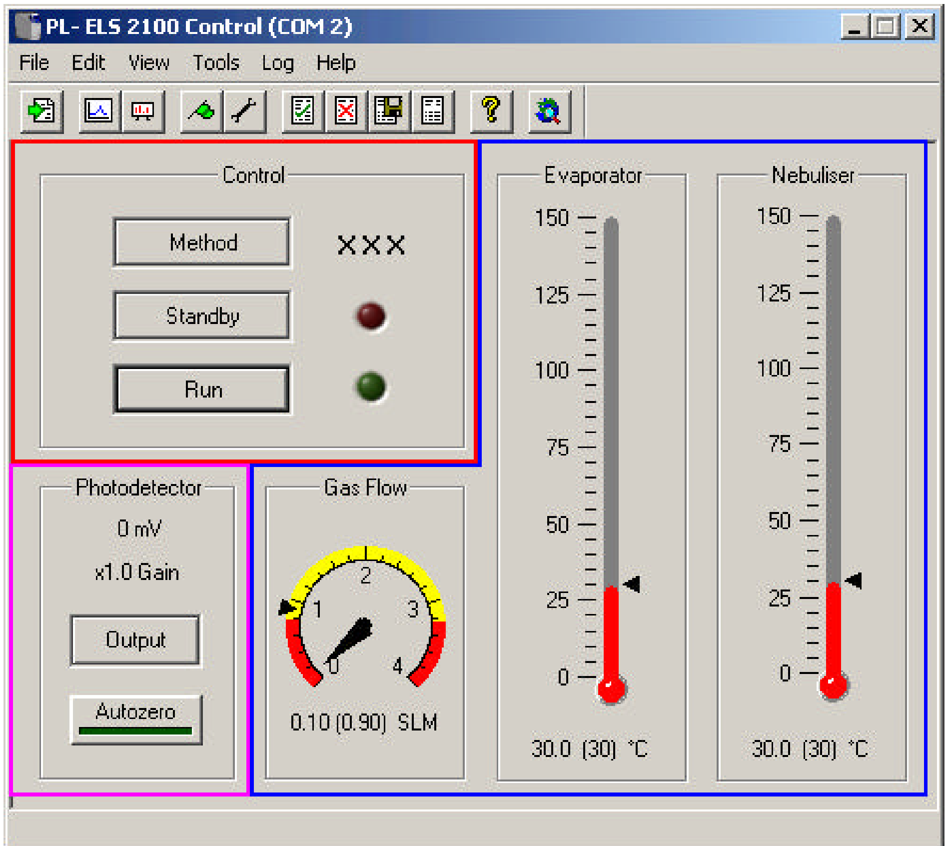
2. PARAMETERS STATUS

Displays the actual values against the set parameters for:

- The Evaporator Temperature
- The Nebuliser Temperature
- Gas Flow

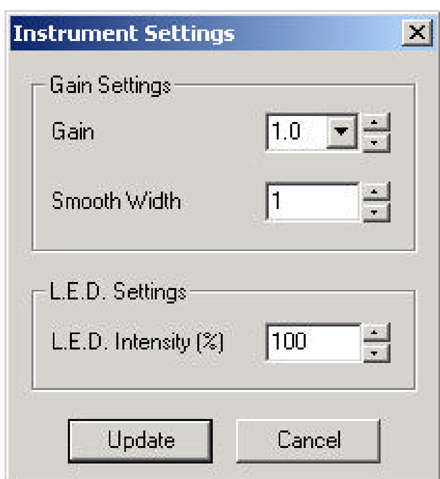
3. PHOTO DETECTOR

- Displays current detector output
- Displays current gain setting
- Remote Auto-zero




## Control Software Features

- The PL-ELS control software offers all the features offered on the instrument control panel and thus the instrument settings including gain adjust, smoothing and LED intensity can be adjusted via the Edit menu:- (**EDIT / Instrument settings** menu option). Changes made to these parameters need to be downloaded to the PL-ELS 2100/2100 Ice using the UPDATE button.



NOTE

*The L.E.D intensity is set for **ALL** methods used on the detector and is **NOT** Method specific.*

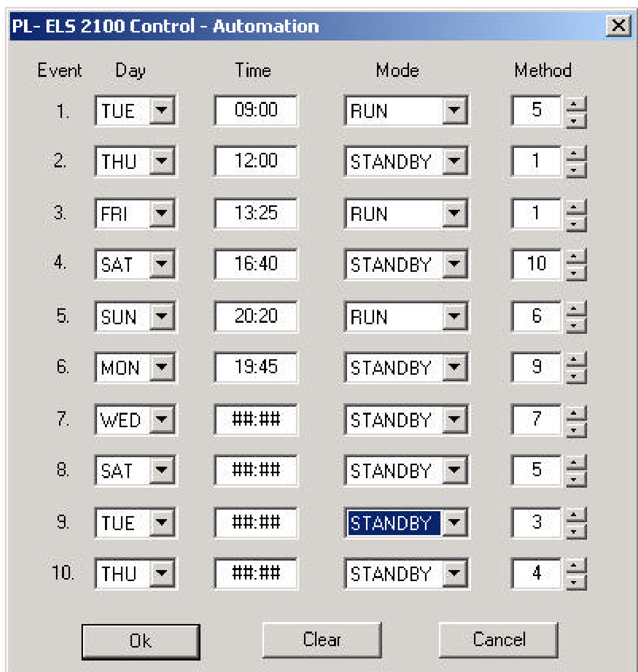
NOTE

*The L.E.D intensity default value of 100% is **NOT** restored following instrument shutdown and power-up. The user selected LED is the value that is retained.*

## Automation

The PL-ELS 2100/2100 Ice control software allows the user to set-up an automated sequence of events over a 24-hour period. Using a time triggered event approach (based on the PC clock), the user can select a method of choice to be loaded and/or the PL-ELS 2100/2100 Ice status changed.

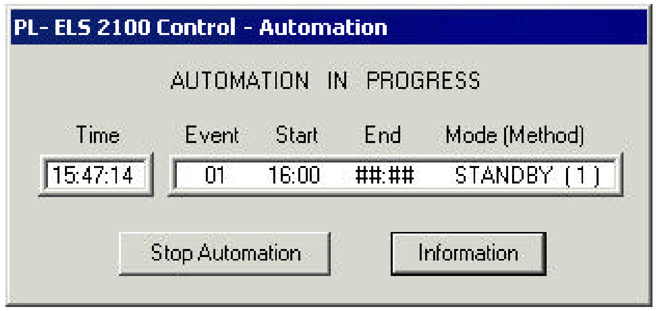
When automation is selected (**TOOLS** / **Automation** menu option), the following set-up dialog is displayed.




The sequence will start at event 1, at the correct time the mode will be selected and the relevant method will be loaded. Following this the sequence will then wait on event two's time so it is important to make sure the events time is correctly specified and in the correct order. The time **##:##** will terminate the automation sequence.

When **Ok** is selected, the Automation Sequence will be saved so that the next time you start the Automation Sequence you will always be presented with the last sequence of events.

When Automation is in progress, a small information box showing the automation progress will be displayed, along with current event information



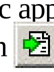

The automation procedure can be stopped at any point by selecting *Stop Automation*.

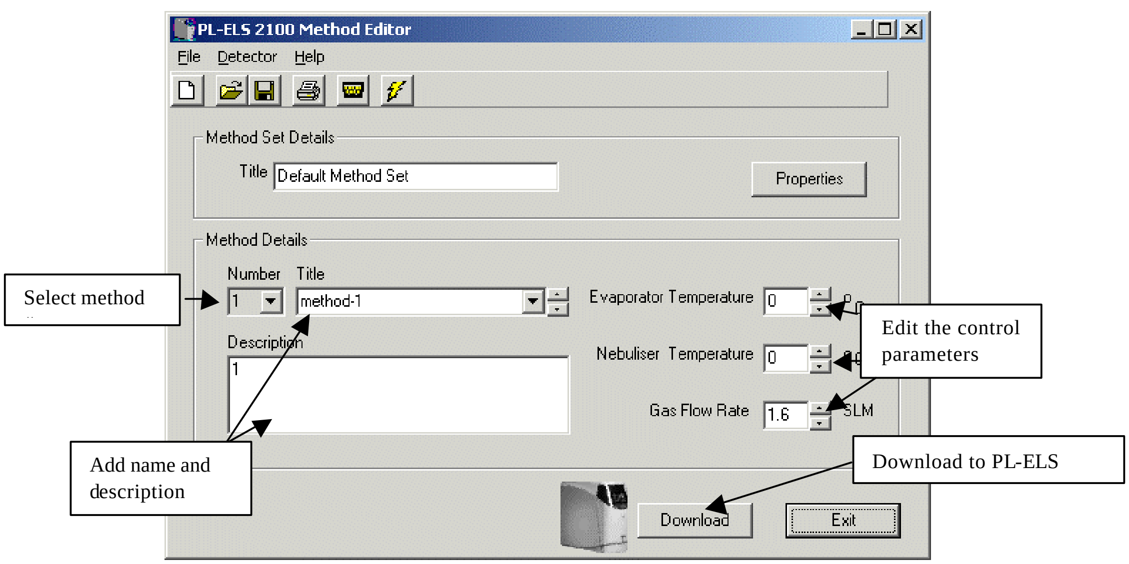
### System Test

This feature can be used to test that your detector is performing to the required operational standard set by Polymer Laboratories. When you select this feature (**TOOLS** / **Run system test** menu option), the software will run your detector through a number of tests and analyse the information it gains providing you with an analysis of the results.

Polymer Laboratories advise that this test is used occasionally to check the instruments performance, or when a problem is encountered.

### Method Editor -Overview

The PL-ELS 2100/2100 Ice can store 10 customs methods, which can be selected via the front of the instrument or via the control software. The method editor program allows the user to create, edit and download these custom methods for method development purposes or specific applications. The method editor program can be launched from the control software via this icon 



## Method Editor – Creating & Storing Methods

To change a custom method on the PL-ELS 2100/2100 Ice, you must first create a custom "Method Set" using The PL-ELS 2100/2100 Ice Method Editor program and subsequently download it to the detector. Individual custom methods cannot be downloaded to the detector; all 10 Custom Methods have to be downloaded simultaneously to the Detector as a "Method Set".

Each "Method Set" is stored as an individual binary format file with a File Extension (.ELS)

By default, these Method Set Files are stored in the PL-ELS 2100 Method folder allocated at the time the software was installed (C:\PL-ELS2100\default)

The loaded method set details can be printed out by selecting the print option from the FILE menu.

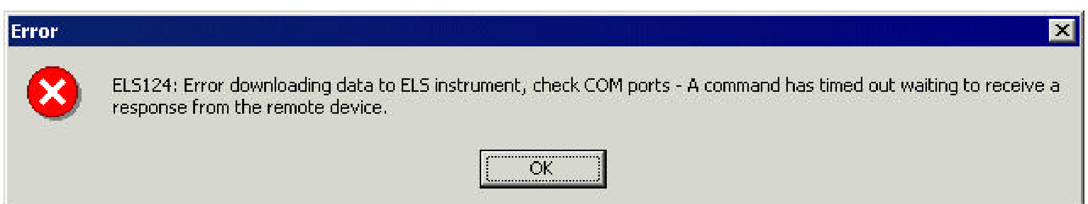
## Method Editor – Downloading Method Sets

To download a *Method Set* to the PL-ELS2100/2100 Ice,

1. Load the Method Set to be downloaded into the Method Editor
2. Ensure the Method Editor is configured for the correct communication port for the detector.
3. Ensure that any Control software session that is currently accessing the detector is closed down
4. Select the Download button  on the toolbar to download the methods on to the PL-ELS 2100/2100 Ice

If the download was successful, you will see a dialog saying: "**ELS Updated OK**"

If the download was unsuccessful, you will see the following dialog:




In this case, check that the Detector is connected to the PC and is switched on.

A comprehensive guide to both of these software control programmes are can be found in the On-line help supplied with the software

# Chapter 3
# Set-Up

## 3.1 General Considerations

The PL-ELS 2100/2100 Ice instrument should be thought of as a detector like any other designed for liquid chromatography. The main distinguishing feature is the ability to evaporate the solvent from the column eluent. Therefore, normal system set-up precautions should be remembered when starting to use the instrument. Any solvent intended for use with the PL-ELS 2100/2100 Ice 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. ≤ 0.010”.

The PL-ELS 2100/2100 Ice requires nitrogen (purity >98%), capable of generating 60-100psi inlet pressure. If in-house nitrogen is not available then we recommend the use of a nitrogen generator (Parker Balston Model N2-4000 – see Appendix 5), 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 under the chosen detector parameters – any non-volatilised eluent will increase baseline noise and reduce sensitivity.

NOTE

*Non-Volatile buffers are not compatible with the PL-ELS 2100/2100 Ice*

NOTE

*High concentrations of acetic acid (>1%) are detrimental to the operation of the PL-ELS 2100/2100 Ice*

## 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 2A or 230V 2A, on the IEC inlet fuse-holder on rear of unit.
- Attach the gas inlet tube to the 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 3.25 SLM throughout the temperature range.
- Connect the waste tube to the waste outlet at the front of the detector and position the other end in a waste collection bottle.
- The solvent waste container supplied with the detector can be sited on the bench in front of the instrument. Alternative waste collection vessels can be used if required but the bottom of the waste tube must be below the height of the waste outlet from the instrument. Waste tube must NOT be submerged in the liquid
- Connect the exhaust hose between the exhaust outlet and a fume hood.
- Connect the instrument to the data recorder (computer, chart recorder, etc.) using the cable provided. One output socket is provided giving a signal in the 0-1V range.
- Connect the column outlet to the eluent inlet at the front of the unit) using a short length (10cm) of tubing (1/16" OD, 0.010" ID) and a 1/16" Valco fitting.
- Turn on the source gas to a pressure of about 60-100 psi. The gas will not flow though the detector until the instrument in set to RUN mode.
- Switch on the PL-ELS 2100/2100 Ice, and select a suitable method.
- Initiate heating by selecting RUN mode using the arrow keys on the front keypad.
- 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 0.2mV 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 nebulised it will exit the instrument from the nebuliser drain tube.*

- Again check the baseline noise. It should not have increased significantly and should be ≤1mV. Typically pure water should give no more than 0.4mV peak-peak, whilst pure organic solvents should be less than 0.25mV.

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

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.

## *3.3 Optimisation Protocol*

### Gas Flow

An increase in gas flow rate causes a decrease in signal response, so lower gas flow rates are 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 evaporation of the eluent. In general, gas flow rates of 1.0-2.0SLM tend to be a reasonable compromise between baseline stability and high reproducible response for eluent flow rates >0.5ml/min. Reducing the eluent flow allows a reduction in the gas flow rate to maintain the optimum particle stream concentration, resulting in greater sensitivity.

When operating at sub-ambient temperatures in aqueous solvents, gas flow values of >2.0SLMs maybe necessary to control the baseline noise.

### Evaporator Temperature

The effects of altering the evaporator temperature tend to be less dramatic than changing the 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.

The PL-ELS 2100 Ice can be operated at sub-ambient temperatures to improve the detection of semi-volatile compounds. In aqueous solvents, baseline noise may become too excessive at temperatures <15°C. However, for organic solvents, the baseline can be controlled down to 10°C, provided the gas flow is increased to compensate.

### Nebuliser Temperature

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

### Procedure

Set the system at the minimum evaporator and nebuliser temperatures and a gas flow rate of 1.6 SLM. Make an initial injection of the test component and monitor the signal response. With each subsequent injection increase the nebuliser and evaporator temperatures by 10°C whilst reducing the gas flow rate, until optimum sensitivity is achieved. In some cases, especially for volatile samples, it has been found that maximising the nebuliser temperature permits a lower evaporator temperature and thus an increase in sensitivity.

# Chapter 4
# Routine Maintenance

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

**WARNING**

*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.*

**WARNING**

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

The PL-ELS 2100/2100 Ice 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.

## 4.1 Drying the Diffuser

If the instrument has been operated incorrectly 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 nebuliser drain tube. If this happens then simply turn off the liquid flow, increase the gas flow to 2.8SLM and increase the evaporator to maximum temperature. The diffuser will be dried out and the instrument ready to use after approximately 1hour 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 initially washed with a solvent suitable for the contamination. To perform this wash, pump the appropriate solvent into the instrument at 1 to 2ml/min whilst the instrument is in RUN mode. Following this procedure, dry out the instrument by running the system at maximum temperatures, without eluent flow. For removal of aqueous soluble contamination the instrument can be heated to high temperature and the impurity removed by ‘steam cleaning’ (PL-ELS 2100 only). To clean a unit, set the evaporator temperature to maximum, the nebuliser temperatures at 90°C and the gas flow to 2.8SLM with a 1ml/min flow of de-ionised water. Leave at temperature in the RUN mode for approximately 3-4 hours, or overnight. Depending on usage, it is recommended to clean the evaporator tube once a week or every 40 hours of use as a preventative routine. It is also recommended to clean the unit following the use of buffers.

ATTENTION *Do not pass solvent other than water through the unit during 'steam clean' procedure*

WARNING *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

WARNING *INSTRUMENT IS DOUBLED FUSED*

### Heater PCB Fuses

There are 3 fuses fitted to the main control PCB, one to protect each of the heater circuits

FH 1 & 3: H 1A FF 250V

FH 2: TR5 2A T 250V

### Light Source

The light source assembly is a Class 1 LED product as defined in IEC 60825-1 Ed 1.2. Whilst this is non-hazardous it is advisable to avoid staring into the beam.

## 4.4 Putting the Instrument into Storage

If the instrument is to be stored or not used for a period of time it is recommended to follow the procedure outlined below:-

1. Allow the instrument to cool to ambient temperature in STANDBY mode with the gas supply still connected.

2. Tip the instrument forwards to try and empty the 'P' trap contents through the front waste tube (i.e. into the bottle)

3. Pour 10 to 20mls of acetone into the exhaust tube to flush out the P trap, collecting the waste in the waste bottle at the front of the instrument.

4. Again tip the instrument forwards to drain the p-trap

5. Disconnect the waste bottle

6. Using the gas supply, blow nitrogen gas through the exhaust to evaporate any remaining acetone in the p-trap. Cover the waste tube with tissue paper to collect any acetone residue.

7. Plug the exhaust and waste tubes with the plastic caps provided.

8. Plug the solvent inlet.

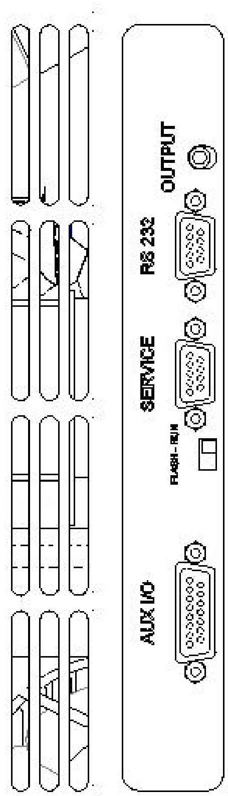
## 4.5 Upgrading the Firmware using Terminal

Tools required:

A copy of TERMINAL.exe or Hyperterminal.exe (supplied with Windows)

A copy of PL-ELS2100/2100 Ice firmware (e.g. 9041_202.HEX)

Serial cable (supplied with instrument)




1. Close down the control software and turn off the instrument.
2. At the rear of the unit move the serial cable from the connector labelled RS232 to the one labelled Service
3. Move the switch, located below the Service connector, to the down position (FLASH).
4. Run the TERMINAL.exe program (figure 4.4.1) and set the *Communications* in the ***Settings*** menu to those shown in figure 4.4.2.
5. Turn on the instrument and observe that the instrument successfully communicates with the terminal program (figure 4.4.3).
6. Press P on the keyboard (figure 4.4.4) and select the *Send Text File* option from the ***Transfers*** menu (figure 4.4.5).
7. Browse to select the new firmware (e.g. A:\9041_202.hex). Note: the default file extension is TXT and therefore the *All files* option will have to be selected to see the Hex file.
8. The program will now be downloaded (figure 4.4.6) and programmed (figure 4.4.7).
9. On completion of the programming close the Terminal program and turn off the instrument.
10. Move the serial cable back to the control connector and move the switch back to the up position (RUN).

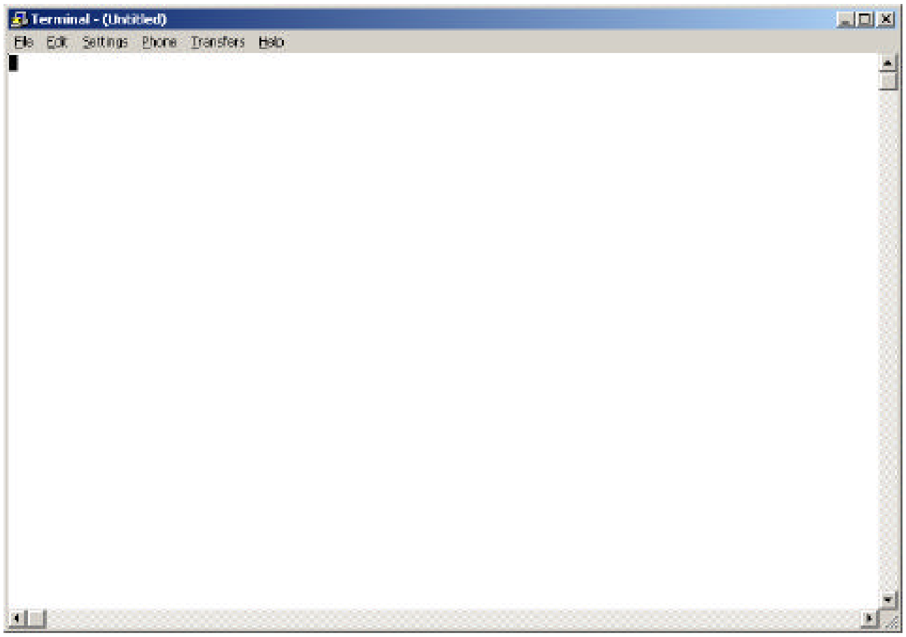



*Figure 4.4.1 Terminal program*

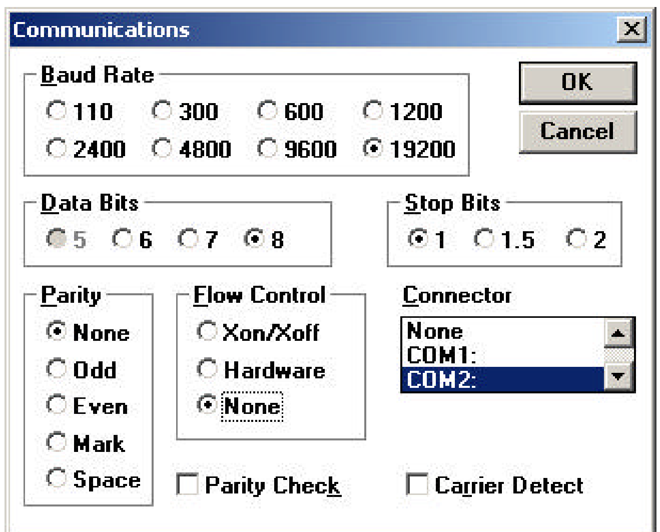



*Figure 4.4.2 Communications dialog*

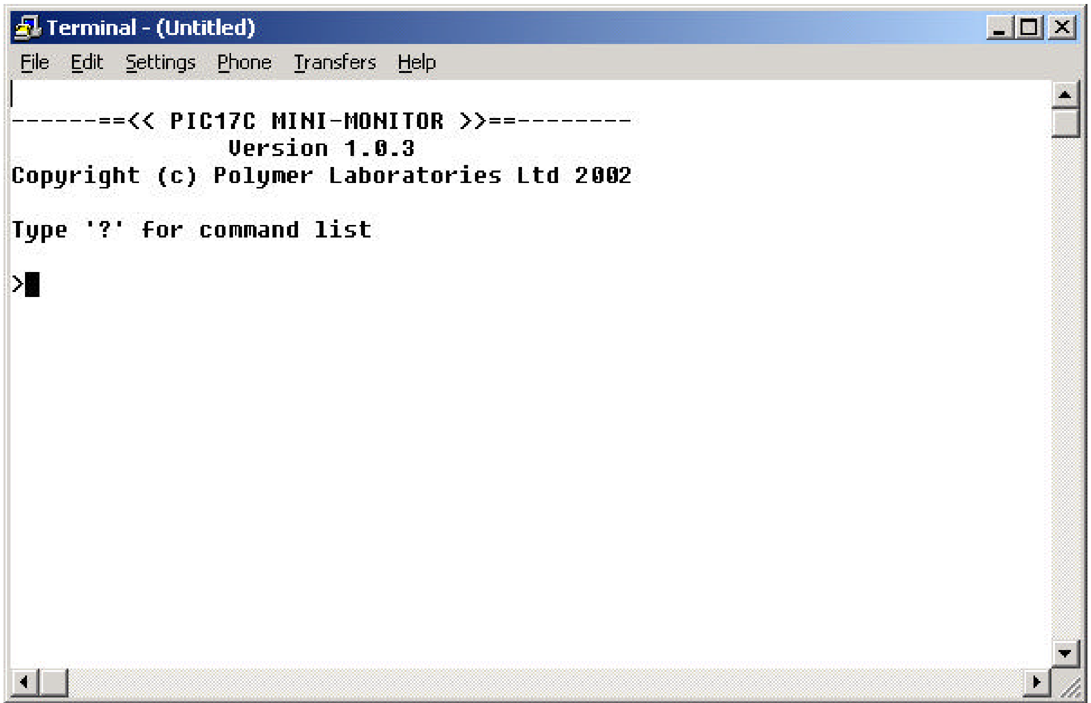



*Figure 4.4.3 Successful Communication*

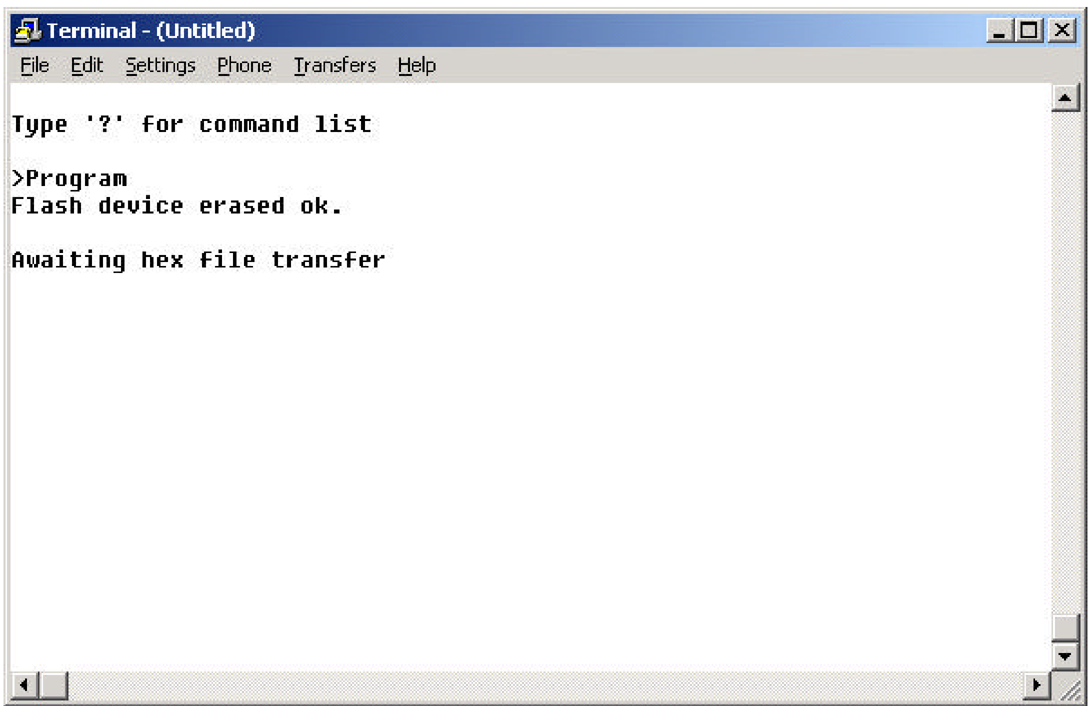



*Figure 4.4.4 Initialising the Programming*

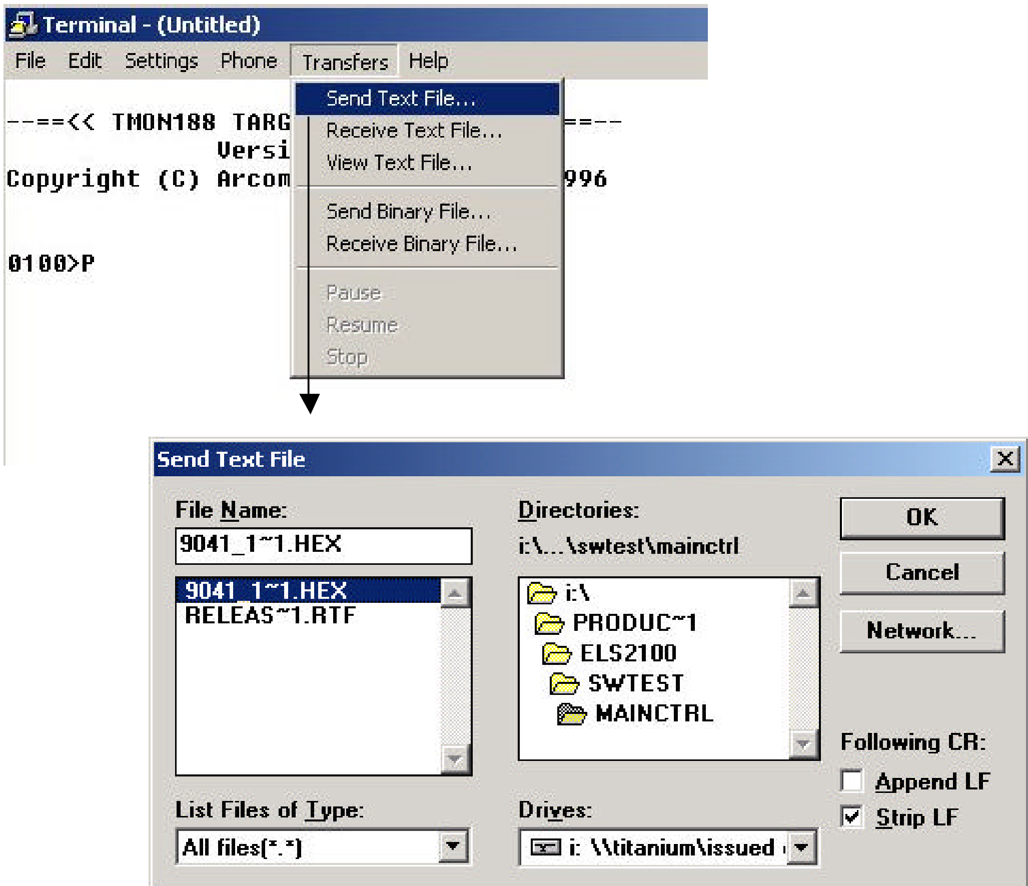



*Figure 4.4.5 Selecting the HEX file*

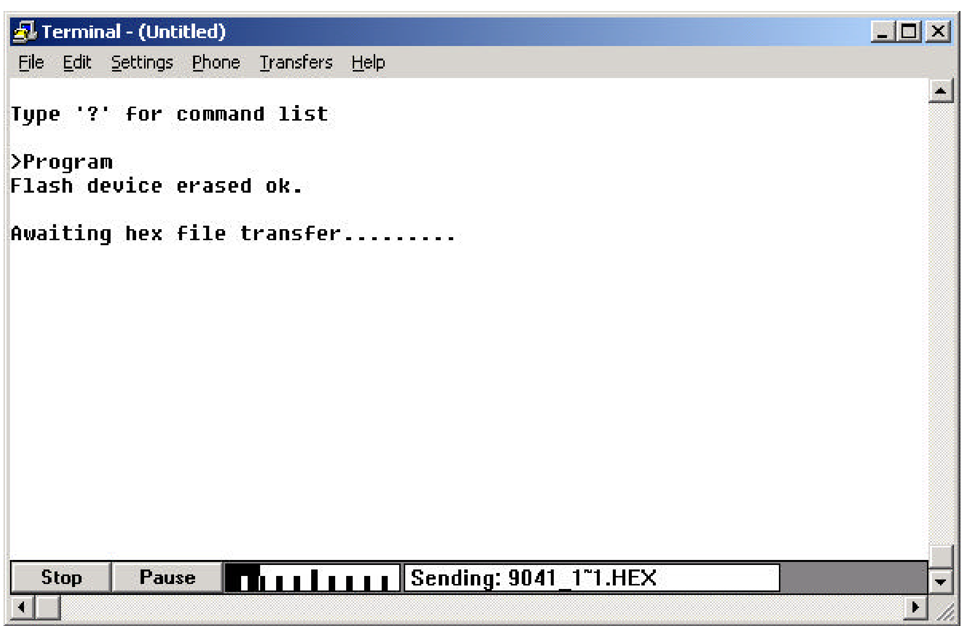



*Figure 4.4.6 Downloading the Program*

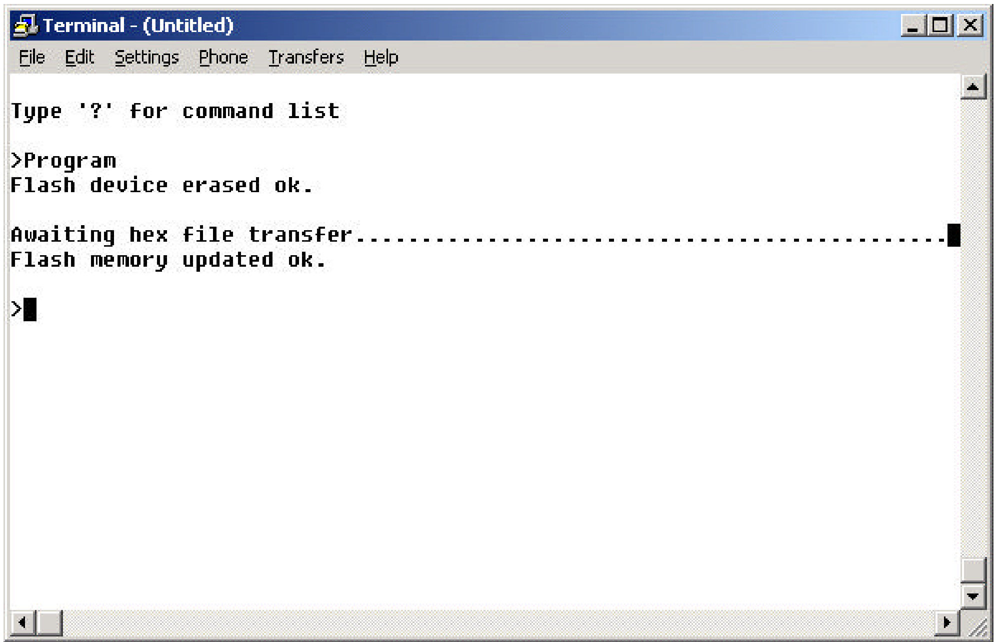



*Figure 4.4.7 Programming the EPROM*

# Chapter 5
# Troubleshooting

If a problem is encountered Polymer Laboratories advises that a system test be performed via the PL-ELS 2100/2100 Ice PC control software (section 2.4) to ensure that the detector is working correctly. 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 | Description | Suggestions |
|---|---|---|
| 10 | air temp <= 10°C inside enclosure | Environmental temp too cold |
| 11 | air temp > 40°C temp inside enclosure | Environmental temp too hot |
| 12 | on board sensor heater failed | Vapour sensor needs replacing |
| 13 | rear panel sensor heater failed | Check vapour sensor on rear of ELSD is not damaged/ Vapour sensor needs replacing |
| 14 | leak detected inside unit | Solvent vapour leak detected. Stop pump |
| 15 | Liquid leak sensor has detected liquid in drip tray | Liquid in the base of the unit- Stop pump and investigate |
| 16 | fan driver IC thermal shutdown | Fault with the circuitry / wires / fan Call service engineer if this happens regularly |
| 17 | Fan stopped | Fault with the circuitry / wires / fan Call service engineer if this happens regularly |
| 18 | Nebuliser temperature exceeded threshold after stabilising | Faulty thermocouple or heater control |
| 19 | Evaporator temperature exceeded threshold after stabilising | Faulty thermocouple or heater control |
| 20 | Light source error | Replace light source assembly |
| 21 | Evaporator gas flow rate exceeded threshold after stabilising | Check operating head pressure, otherwise faulty mass flow controller |
| 22 | Invalid nebuliser temp reading (FAULTY RTD) | replace nebuliser heater assembly |
| 23 | Invalid evaporator temp reading (FAULTY RTD) | replace evaporator assembly |
| 24 | Fan failure on cooled evaporator | Fault with the peltier unit. Call service engineer if this happens regularly |
| 25 | Evaporator tube current outside of normal range | Fault with the peltier unit. Call service engineer if this happens regularly |

## 5.2 General Problems

| Faults or Problems | Possible Cause(s) | Remedy |
|---|---|---|
| Baseline noise | The particle plume is not sufficiently dried in the evaporator tube. | • Increase the temperature of the evaporator by 10° intervals until the noise is acceptable |
| Baseline noise | 1. Poor nebulisation of solvent<br>2. Pressure difference created inside nebuliser chamber | • Increase the gas flow rate<br>• Decrease the nebulisation temperature<br>• Ensure that the end of the liquid waste tube is not immersed in liquid<br>• Ensure that the exhaust tube at rear of unit is not blocked, or extraction is too strong |
| Baseline noise | Pump pulsations, especially in microbore applications where low flow rates are used. | • Use a pulse free pump<br>• Increase the back pressure on the pump by fitting a back pressure column between the pump and the injection valve.<br>• Use a pulse dampener directly after the pump in the system. |
| Baseline spikes | 1. Particulate matter in the gas supply<br>2. Column shedding<br>3. Poor nebulisation | • Filter the incoming gas, or change the supply<br>• Replace column or fit an inline filter with a 0.2µm membrane filter directly after the column.<br>• Nebuliser temperature may be too high and solvent may be boiling; reduce nebuliser temperature |
| Low sensitivity and baseline noise | Diffuser saturated with solvent | • Stop the eluent flow and increase the evaporator temperature to 50°C above the current set temperature. Increase the flow rate to 2.8SLM and wait 15mins. |
| Large Baseline offset | 1. Inefficient evaporation<br>2. High concentration of non-volatile buffer or stabiliser<br>3. Contaminated diffuser | • Increase the evaporator temperature and/or gas flow.<br>• Use a lower concentration of stabiliser, unstabilised solvent or a more volatile buffer (ammonium acetate or ammonium formate)<br>• Perform 'steam clean' |
| Peak tailing | Eluent particles lingering in the optical chamber | • Increase gas flow rate |
| Instrument Fails to zero | Offset too high or output unstable | • Ensure the instrument is in RUN mode<br>• Refer to local distributor or Polymer Laboratories |
| No power | 1. Mains lead not connected<br>2. Fuse failure | • Attach mains lead to socket and inlet on rear of instrument<br>• Replace fuse |
| No response (completely flat baseline) | 1. Data acquisition leads not connected<br>2. Light source inactive<br>3. Instrument in STANDBY mode | • Ensure connectors to computer or integrator are sound<br>• Check LED<br>• Select RUN mode |

| | | |
|---|---|---|
| Temperature error as soon as instrument powered on | Temperature probe fault or disconnected | • Check RTD connections<br>• Consult Polymer Laboratories Ltd or your local agent for further advice |
| Display not on, but power connected, blue glow ON. | Instrument in service mode | • Rear panel switch-set to RUN<br>• Consult Polymer Laboratories Ltd or your local agent for further advice |
| Evaporator Temperature reads zero at start-up and cannot be changed. | Peltier cooler has not initiated correctly | • Switch detector off then on at the power socket<br>• Consult Polymer Laboratories Ltd or your local agent if problem persists |
| Vapour sensor error occurs, but there is no solvent or vapour leak inside unit. | Solvent vapour near the front of unit is being drawn into the unit<br><br>Faulty Vapour sensor | • Remove any solvent bottle or solvent leak that is directly in front of the detector<br>• Check the rear vapour sensor is not damaged/bent. |
| Evaporator will not reach low temperature e.g. 10°C | Ambient temperature too high<br><br>Faulty Peltier cooler unit | • Reduce ambient temperature to 25°C<br>• Consult Polymer Laboratories Ltd or your local agent if problem persists |

# APPENDIX 1

## *PL-ELS 2100/2100 Ice Quick User Guide*

The following is intended to be a quick guide for operation of your PL-ELS 2100/2100 Ice. However, the Operator's Manual must be read before attempting to operate the PL-ELS 2100/2100 Ice so that all the instrument controls are identified.

1. Before turning on your PL-ELS 2100/2100 Ice, ensure the following connections have been correctly and securely made to the instrument:-

| Item | Instrument connection | From/To | Comments |
|---|---|---|---|
| Power cord | Power in | Mains supply | The correct operating voltage for your PL-ELS 2100/2100 Ice is displayed on the rear panel |
| Gas inlet tube | Nebuliser gas inlet | Nitrogen generator or gas cylinder fitted with regulator | The gas must be clean and dry with a head pressure of at least 60 psi. Nitrogen is recommended for volatile solvents. |
| Exhaust hose | Rear Exhaust tube | Fume hood | Ensure extraction is not higher/stronger than the outflow of the ELSD |
| Analogue output cable | Output | Data collection system | Clear +ve, Black -ve |
| Serial cable | RS 232 | Computer com port | Optional instrument control by PL-ELS 2100/2100 Ice Control graphical interface |
| Waste Hose | Waste outlet | Place waste hose in the 500 ml bottle provided or alternative waste container | The end of the waste tube should not be immersed in the waste solvent |
| Column Connector | Eluent inlet | Chromatography column | The connecting tubing should be as short as possible and 0.01" ID use a 1/16'' Valco fitting to the detector |

2. Turn on the power.

3. Once booted, highlight **GAS**, **NEB** and **EVAP** to view set operating conditions on the instrument display using the left and right arrow keys. To alter the parameter values use the up and down arrow keys to select the desired value.

4. Alternatively select one of the 10 preset methods by pressing **METHOD** followed by ▲▼ to select the required method. The preset methods are listed in the following table. Methods can be edited and we recommend the updated methods be recorded on the Method record sheet enclosed in the manual.

| **Method Number** | **Evaporator temp (°C)** | | **Nebuliser temp (°C)** | | **Gas Flow (SLM)** | |
|---|---|---|---|---|---|---|
| ELSD Model | PL-ELS 2100 | PL-ELS 2100 Ice | PL-ELS 2100 | PL-ELS 2100 Ice | PL-ELS 2100 | PL-ELS 2100 Ice |
| 1 | 0 | 30 | 0 | 30 | 1.6 | 1.6 |
| 2 | 30 | 25 | 30 | 30 | 1.6 | 1.6 |
| 3 | 50 | 20 | 30 | 30 | 1.6 | 1.8 |
| 4 | 50 | 15 | 50 | 30 | 1.6 | 2.2 |
| 5 | 70 | 20 | 50 | 80 | 1.6 | 1.2 |
| 6 | 90 | 40 | 50 | 40 | 1.6 | 1.4 |
| 7 | 90 | 50 | 50 | 50 | 1.2 | 1.0 |
| 8 | 120 | 80 | 50 | 60 | 1.2 | 1.0 |
| 9 | 120 | 80 | 90 | 80 | 0.9 | 0.9 |
| 10 | 120 | 80 | 90 | 90 | 2.8 | 3.0 |

5. Select **MODE** to display the current instrument status and the up arrow key to choose the **RUN** mode.

The instrument will begin heating to the desired set point, and a '**NOT READY**' message is displayed on the instrument. Once at steady state conditions the instrument will display '**READY**' indicating the instrument is ready for use.

6. Turn on the eluent flow and wait ~15 mins for the instrument to become primed and conditioned for optimum performance.

7. On completion of your work, turn off the solvent pump and put the PL-ELS 2100/2100 Ice into **STANDBY** mode and turn off the power after 5 minutes.

# APPENDIX 2

## ***Method Record Sheet***

| Chromatography Conditions | Eluent | Eluent flow | Method # | Evap Temp | Neb. Temp | Gas Flow | Date Stored | Comments |
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# APPENDIX 3

## *PL-ELS 2100 Test procedure*

The PL-ELS 2100 is factory tested for sensitivity and baseline noise according to the following test procedure.

**Detector Settings**

*Nebuliser Temperature*: 50

*Gas flow rate*: 1.6 SLM

*Evaporator Temperature* 90

**Chromatography Conditions**

***Sample**:* Glucose

***Concentration**:* 0.125 mg/ml

***Eluent*** Water

***Injection Volume**:* 20µl

***Column**:* Direct injection*

***Eluent flow rate**:* 0.5 ml/min

***Note:** A back-pressure regulator (BPR) is required for direct injection into an ELSD to prevent baseline disturbance from injection pulse.

Pass Specifications for the Glucose/water chromatogram test

| | |
|---|---|
| **Peak height (mV)** | **300±30** |
| **Noise (mV)** | **0.20** |

# APPENDIX 4

## *PL-ELS 2100 Ice Test procedure*

The PL-ELS 2100 Ice is factory tested for sensitivity and baseline noise, at two temperature settings, according to the following test procedure.

**Chromatography Conditions**

| | | | |
|---|---|---|---|
| ***Sample***: | Glucose | ***Concentration***: | 0.125mg/ml |
| ***Eluent*** | Water | ***Injection Volume***: | 20µl |
| ***Column***: | Direct injection* | ***Eluent flow rate***: | 0.5ml/min |

***Inlet Tubing:*** 100m x 0.254mm (0.010”) PEEK

***Note:** A 500PSI back-pressure regulator (BPR) is required between the pump and injector to prevent baseline disturbance from injection pulse.

**Detector Settings: Step 1**

*Nebuliser Temperature*: 30 *Gas flow rate*: 1.6 SLM

*Evaporator Temperature: 80*

Step 1-Pass Specifications for the Glucose/water chromatogram test

| | |
|---|---|
| **Peak height (mV)** | **260±25*** |
| **Noise (mV)** | **0.20** |

**Detector Settings: Step 2**

*Nebuliser Temperature*: 30 *Gas flow rate*: 1.6 SLM

*Evaporator Temperature: 30*

Step 2-Pass Specifications for the Glucose/water chromatogram test

| | |
|---|---|
| **Peak height (mV)** | **150±15*** |

** These values were current at the time of the release of this manua, but are subject to review, so please check the test certificate provided with your instrument.*

# APPENDIX 5

## ***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 |
|---|---|
| Manual for PL-ELS 2100/2100 Ice detector | PL0860-0005 |
| Mains Lead (110-120V, 60Hz or 230V, 50Hz) | PL0860-0015 |
| 15 pin D-sub male connector for Aux I/O connection | PL0860-0020 |
| I/O cable | PL0860-0055 |
| Detector output cable | PL0860-0025 |
| Gas Inlet tube | PL0860-0030 |
| Exhaust hose (PVC– 2.0m) | PL0860-0035 |
| Waste hose (Tygon SE200– 7cm) | PL0860-0040 |
| Solvent waste container (500ml) | PL0860-0045 |
| Valco Nut (1/16”) pk 5 | PL0100-3601 |
| Valco ferrule (1/16”) pk 5 | PL0100-3602 |
| Vapour Sensor | PL0860-0415 |
| LED light source | PL0860-0410 |
| PL-ELS 2100 Display window and keypad | PL0860-0405 |
| PL-ELS 2100 Ice Display window and keypad | PL0860-1300 |
| Mains INLET fuses (pk 5) | PL0860-0420 |
| ELS Gas supply gauge and filter | PL0860-0401 |
| Serial to USB adapter | PL0860-0620 |

To order please contact your local office

# APPENDIX 6

## ***Nitrogen Generator Specifications***

# APPENDIX 7

## ***Cleaning & Decontamination Procedures***

**WARNING**

**Switch off and disconnect power cord from instrument before cleaning or decontamination. Allow any hot parts to cool before proceeding.**

### Cleaning

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. Allow instrument to dry off completely before reconnecting power.

### Decontamination

**THIS PROCEDURE TO BE CARRIED OUT BY TRAINED SERVICE PERSONNEL ONLY**

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

Disconnect the instrument completely and remove it to a fume cupboard if necessary. Empty the P-trap before moving the instrument (see 4.4 Section 2). Open the cover of the instrument to see if any solvent has contaminated the interior.

Excess quantities of liquid spilt on or inside the instrument should be mopped up using absorbent cloths, followed by repeated wiping down with soft cloths moistened with Acetone or deionised water (as appropriate to the spilt solvent) until the last traces of the hazardous liquid have been removed. If liquid has collected in the drip tray around the leak sensor then be very careful not to damage or displace the sensor bead whilst mopping up.

Identify and if possible correct source of the leak. Inspect cabling, parts and surfaces to determine whether any damage has occurred. If in doubt, contact Polymer Laboratories Service Dept for assistance.

Allow the interior and exterior of the instrument to dry out completely before closing the cover and reconnecting power.

Dispose of contaminated waste appropriately.