

# **Agilent 1200 Infinity Series ELSD**

User Manual





## **Notices**

© Agilent Technologies, Inc. 2012, 2013

No part of this manual may be reproduced in any form or by any means (including electronic storage and retrieval or translation into a foreign language) without prior agreement and written consent from Agilent Technologies, Inc. as governed by United States and international copyright laws.

#### **Manual Part Number**

G4260-90001 Rev. C

#### Edition

08/2013

Printed in Germany

Agilent Technologies Hewlett-Packard-Strasse 8 76337 Waldbronn

This product may be used as a component of an in vitro diagnostic system if the system is registered with the appropriate authorities and complies with the relevant regulations. Otherwise, it is intended only for general laboratory use.

#### Warranty

The material contained in this document is provided "as is," and is subiect to being changed, without notice. in future editions. Further, to the maximum extent permitted by applicable law, Agilent disclaims all warranties, either express or implied, with regard to this manual and any information contained herein, including but not limited to the implied warranties of merchantability and fitness for a particular purpose. Agilent shall not be liable for errors or for incidental or consequential damages in connection with the furnishing, use, or performance of this document or of any information contained herein. Should Agilent and the user have a separate written agreement with warranty terms covering the material in this document that conflict with these terms, the warranty terms in the separate agreement shall control.

## **Technology Licenses**

The hardware and/or software described in this document are furnished under a license and may be used or copied only in accordance with the terms of such license.

## **Restricted Rights Legend**

If software is for use in the performance of a U.S. Government prime contract or subcontract, Software is delivered and licensed as "Commercial computer software" as defined in DFAR 252.227-7014 (June 1995), or as a "commercial item" as defined in FAR 2.101(a) or as "Restricted computer software" as defined in FAR 52.227-19 (June 1987) or any equivalent agency regulation or contract clause. Use, duplication or disclosure of Software is subject to Agilent Technologies' standard commercial license terms, and non-DOD Departments and Agencies of the U.S. Government will

receive no greater than Restricted Rights as defined in FAR 52.227-19(c)(1-2) (June 1987). U.S. Government users will receive no greater than Limited Rights as defined in FAR 52.227-14 (June 1987) or DFAR 252.227-7015 (b)(2) (November 1995), as applicable in any technical data.

#### **Safety Notices**

#### CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

#### WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

# In This Book

This manual contains information on:

- Agilent 1260 Infinity ELSD (G4260B)
- Agilent 1290 Infinity ELSD (G4261B)

#### 1 Introduction to the Detector

This chapter gives an introduction to the module, instrument overview and internal connectors

#### 2 Site Requirements and Specifications

This chapter provides information on environmental requirements, physical and performance specifications.

#### 3 Installing the Module

This chapter gives information about the installation of your ELSD.

## 4 LAN Configuration

This chapter provides information on connecting the module to the controller software.

#### 5 Using the Module

This chapter explains the operational parameters of the ELSD.

## 6 Optimizing Performance

This chapter gives hints on how to optimize the performance or use additional devices.

#### 7 Troubleshooting and Diagnostics

This chapter gives an overview about the troubleshooting and diagnostic features.

#### 8 Error Information

This chapter describes the meaning of error messages, and provides information on probable causes and suggested actions how to recover from error conditions.

#### 9 Maintenance

This chapter describes the maintenance of the ELSD.

#### 10 Parts and Materials for Maintenance

This chapter provides information on parts for maintenance.

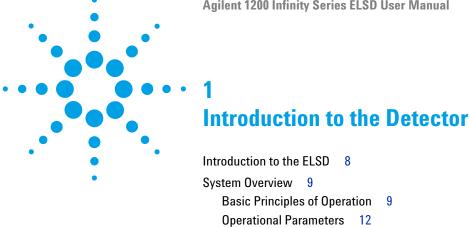
#### 11 Appendix

This chapter provides addition information on safety, legal and web.

# **Contents**

1	Introduction to the Detector 7	
	Introduction to the ELSD 8 System Overview 9	
2	Site Requirements and Specifications 17	
	Pre-installation Requirements 18 Site Requirements 19 Physical Specifications 22 Performance Specifications 23	
3	Installing the Module 27	
	Unpacking the Module 28 Installing the Module 30	
4	LAN Configuration 35	
	What You Have to Do First 36 TCP/IP Parameter Configuration 37 PC and Agilent ChemStation Setup 44	
5	Using the Module 49	
	Before Using the Detector 51 Instrument Controls 52 Operational Parameters 57 Controlling the ELSD During on Injection 62	
	Controlling the ELSD During an Injection 63 Controlling the ELSD within ChemStation or EZChrom General Considerations 67	66

6	Optimizing Performance 71  Do's and Don'ts of ELS Detection 72  Location of the Detector Module 73  Pumping systems 74  Mobile phase priming 75  Solvent recommendations 76
7	Troubleshooting and Diagnostics 77
	Troubleshooting 78 Troubleshooting an HPLC System 79 General Problems 80
8	Error Information 87
	What Are Error Messages 88 Module Specific Error Messages 89
9	Maintenance 99
	Introduction to Maintenance 100 Cautions and Warnings 101 Cleaning the Module 103 Inspection of Cables 104 Drying the Diffuser 105 Cleaning the Nebuliser 106 Cleaning Evaporator Tube 107 Putting the Instrument into Storage 108 Replacing Detector Firmware 109
10	Parts and Materials for Maintenance 115
	Identifying Parts and Materials 116
11	Appendix 117  General Safety Information 118  The Waste Electrical and Electronic Equipment Directive 121  Radio Interference 122  Agilent Technologies on Internet 123



This chapter gives an introduction to the module, instrument overview and internal connectors

Overview of ELS Detector 13

#### 1 Introduction to the Detector Introduction to the ELSD

# Introduction to the ELSD

The Evaporative Light Scattering 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 nebulized and carried by a gas flow through an evaporation chamber. The solvent is volatilized, leaving a mist of solute particles that scatter light to a photosensitive device. The signal is amplified and a voltage output provides the concentration of the solute particles passing through the light.

The Agilent 1260/1290 Infinity ELSD 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 Agilent 1260/1290 Infinity ELSD must be the last in series if used in conjunction with other detectors. If the Agilent 1260/1290 Infinity ELSD is being used as the last detector in a series, care must be taken not to exceed the recommended backpressure in detector cells in other units.

This manual instructs the user in the installation and operation of the Agilent 1260/1290 Infinity ELSD for stand-alone use and control using Agilent OpenLAB (ChemStation and EZChrom editions) and Agilent ChemStation.

# System Overview

# **Basic Principles of Operation**

#### **Nebulization**

The eluent inlet is connected to the nebuliser via a short length of stainless steel capillary tube. The incoming 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 flow into the evaporator section. Any larger droplets or the inefficiently nebulized droplets collect in the nebuliser chamber waste trap and then drain off via the waste outlet into a collection bottle.

#### **Evaporation**

After nebulization the atomized spray is propelled through the evaporation tube assisted by the carrier gas. In the evaporator section the solvent is removed leaving a stream of dry particles of the analyte. 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 randomizes the particle plume. The ELS Detectors use patented gas flow technology in the evaporation zone to aid evaporation at low temperatures. By adding a stream of dry nitrogen (evaporation gas) at the entrance of the evaporator tube less volatile solvents (for example, water) are easily evaporated. This evaporation gas is controlled by the user and facilitates sub-ambient operation.

#### Detection

Light in the optical chamber is passed through the instrument at right angles to the direction of particle 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 vapor 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.

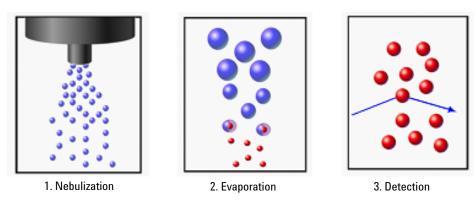


Figure 1 Principles of Operation

#### 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, see Figure 2 on page 11.

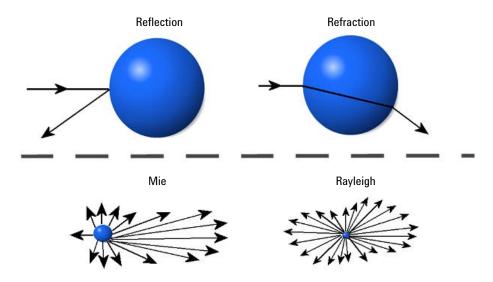


Figure 2 ELSD Scattering Mechanisms

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 < 5·10<sup>-2</sup>. 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.

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.

# **Operational Parameters**

The ELS Detector responds to all compounds that are less volatile than the mobile phase and is independent of a compound's optical properties. It therefore provides advantages over other spectroscopic detectors for detecting compounds that are deficient in a UV chromophore or fluorophore.

The removal of aqueous mobile phase within an ELSD is typically achieved by setting the evaporator temperature to the eluent's boiling point (e.g. 100 °C) in order to remove the solvent. For non-volatile compounds, operating at these high temperatures maximizes the signal response.

However, at these temperatures volatile and semi-volatile compounds are destroyed and are therefore not detected. This is problematic for small molecules, such as pharmaceuticals and drug candidates.

The ELS detectors are specifically designed to evaporate difficult solvents at ambient and sub-temperatures in order to maximize detection of semi-volatile compounds.

The ELSD has patented technology that reduces the evaporation time of highly aqueous solvents at low temperature, and also prevents the evaporation tube becoming saturated, which would otherwise prevent further evaporation occurring.

Using this patented evaporation gas technology, a 20  $\mu$ m droplet of water at 30 °C can be dried ca. 3x faster than just temperature alone. Using the Agilent 1290 Infinity ELSD (G4261B), water can be evaporated as low as 20 °C, providing maximum sensitivity to thermally sensitive compounds. For maximum sensitivity of non-volatile compounds the evaporation gas can be turned off at higher evaporation temperatures.

Therefore, unlike other ELS detectors, where the evaporator temperature is set according to the type of mobile phase, the ELS detector evaporator temperature is independent of the mobile phase. Consequently, the ELS detector can be set at 30 °C for all types of mobile phase provided the evaporator gas flow is adjusted accordingly. This method of operation ensures that the ELSD sensitivity is maximized even for low molecular weight compounds.

To prevent against unnecessary gas usage, a controlled gas shut off valve is integrated into the detector gas manifold. This will only allow gas to pass into the instrument when in RUN mode. Should the instrument default to STANDBY mode the gas will reduce to a default value of 1.2 SLM for 15 min before closing.

# **Overview of ELS Detector**



Figure 3 ELSD overview (front)

1	Front screen display
2	Keypad
3	Eluent inlet
4	Solvent waste outlet

## 1 Introduction to the Detector

System Overview



Figure 4 ELSD overview (rear)

1	Vapour Sensor vent
2	Serial RS232 port
3	LAN connector (only active on G4261B ELSD)
4	Firmware button
5	I/O connector (Remote Start input)
6	Mains Switch
7	Mains Input
8	Gas Inlet port
9	Exhaust port
10	Analogue Output

Table 1 ELS Detector I/O connections

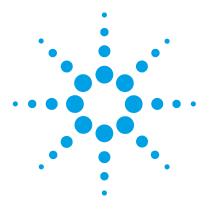
	I/O description	Pin number
Inputs	Timetable Start	14 & ground
	Injection Sync	13 & ground
	Remote A/Z	7 & ground
Output	Pump stop contact closure — normally open	3 & 10
	Ground (to case)	1, 5, 6, 11

# NOTE

In order to make appropriate remote start and A/Z connections from a third party LC, a Remote Start Cable (Remote start cable (third party LCs only) (PL0890-0350)) can be purchased from Agilent Technologies.

# 1 Introduction to the Detector

System Overview



# Site Requirements and Specifications

Pre-installation Requirements 18
Site Requirements 19
Physical Specifications 22
Performance Specifications 23
Specifications 23
Specification Conditions 24

This chapter provides information on environmental requirements, physical and performance specifications.

## 2 Site Requirements and Specifications

**Pre-installation Requirements** 

# **Pre-installation Requirements**

For a detailed description of the environmental and operating requirements of the ELSD, please refer to the Site Preparation Checklist.

This manual will give you an overview of the ELS detector, and describe its operation in more detail.

# **Site Requirements**

A suitable environment is important to ensure optimal performance of the instrument, please refer to the Site Preparation Checklist for more details.

# **Laser Safety**

The Agilent 1290 Infinity ELSD (G4261B) is classified as a "Laser Class 1" product (IEC825-1, CFR1040.10 & 1040.11). During normal operation of the G4261B ELSD no laser light is accessible to the user.

#### WARNING

#### Eye damage by laser light

Hazardous laser light can injure eyes.

- Do not remove covers and interlocks.
- Observe and note the laser warning signs carefully.

# **Power Considerations**

Check the operating voltage of your instrument on the IEC inlet fuse holder on rear of unit.

## WARNING

Hazard of electrical shock or damage of your instrumentation can result, if the devices are connected to a line voltage higher than specified.

→ Connect your instrument to the specified line voltage only.

#### 2 Site Requirements and Specifications

**Site Requirements** 

#### **CAUTION**

Inaccessible power plug.

In case of emergency it must be possible to disconnect the instrument from the power line at any time.

- → Make sure the power connector of the instrument can be easily reached and unplugged.
- Provide sufficient space behind the power socket of the instrument to unplug the cable.

## **Power Cords**

Your detector is delivered with a power cord which matches the wall socket of your particular country or region. The plug on the power cord which connects to the rear of the instrument is identical for all types of power cord.

#### WARNING

Absence of ground connection or use of unspecified power cord

The absence of ground connection or the use of unspecified power cord can lead to electric shock or short circuit.

- → Never operate your instrumentation from a power outlet that has no ground connection.
- → Never use a power cord other than the Agilent Technologies power cord designed for your region.

#### WARNING

#### Use of unsupplied cables

Using cables not supplied by Agilent Technologies can lead to damage of the electronic components or personal injury.

→ Never use cables other than the ones supplied by Agilent Technologies to ensure proper functionality and compliance with safety or EMC regulations.

**Site Requirements** 

# WARNING

# Unintended use of supplied power cords

Using power cords for unintended purposes can lead to personal injury or damage of electronic equipment.

→ Never use the power cords that Agilent Technologies supplies with this instrument for any other equipment.

# 2 Site Requirements and Specifications

**Physical Specifications** 

# **Physical Specifications**

 Table 2
 Physical Specifications

Туре	Specification	Comments
Weight	11 kg (non-cooled), 13 kg (cooled)	
Dimensions (height × width × depth)	415 x 200 x 450 mm (16.3 x 7.9 x 17.7 inches)	
Line voltage	100 – 240 VAC, ± 10 %	Wide-ranging capability
Line frequency	50 or 60 Hz, ± 5 %	
Power consumption	150 W (max)	Maximum
Ambient operating temperature	10-35 °C (50-95 °F)	
Ambient non-operating temperature	-40 – 70 °C (-40 – 158 °F)	
Humidity	< 80 % r.h. at 40 °C (104 °F)	Non-condensing
Operating altitude	Up to 2000 m (6562 ft)	
Non-operating altitude	Up to 4600 m (15091 ft)	For storing the module
Safety standards: IEC, CSA, UL	Installation category II, Pollution degree 2	For indoor use only.

# **Performance Specifications**

# **Specifications**

The instrument is suitable for indoor use only and is classified suitable under the following categories (EN 61010-1):2010

- · Installation category II
- Pollution degree 2
- · Safety class 1

 Table 3
 Performance Specification of the ELS Detector

Туре	Specification
Light Source	
G4260B	LED 480 nm (Class 1 LED product)
G4261B	LASER 405 nm, 10 mW (Class 3B)
Detector	PMT with digital signal processing
Nebuliser	OFF, 25 – 90 °C
Evaporator	
Non-cooled	OFF, 25 – 120 °C
Cooled	OFF, 10 – 80 °C
Gas Flow Range	0.9-3.25 SLM (controlled gas shut-off)
Short Term Noise	for G4260B <0.2 mV, for G4261B <0.1 mV under specified conditions. See "Specification Conditions" on page 24.
Drift	For both modules <1 mV/h under specified conditions. See "Specification Conditions" on page 24.
Operating Pressure	60 – 100 psi ( 4 – 6.7 bar)
Eluent Flow range	0.2 – 5.0 mL/min
Digital Output	10, 40 or 80 Hz ( 24 bit)
Analogue Output	0 – 1.25 V FSD

**Performance Specifications** 

 Table 3
 Performance Specification of the ELS Detector

Туре	Specification
Communication	Ethernet
	Serial (RS232)
	Remote Start Input
	Pump Stop: 1 Contact closure
PC control (software)	ELSD driver for Rev B ChemStation (RC.NET driver)
	ELSD driver for OpenLAB ChemStation edition
	ELSD driver for OpenLAB EZChrom edition
	ELSD Dimension Software
Remote operation	Remote Start Input
Safety features	Gas shut off Valve, Leak Detection, Laser Interlock

# **Specification Conditions**

ASTM: "Evaporative Light Scattering Detectors Used in Liquid Chromatography".

 Table 4
 Reference conditions

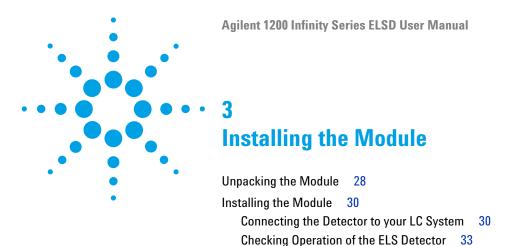
Gas flow	1.6 SLM
Neb temperature	40 °C
Evaporator temperature	40 °C
PMT Gain	1
Data Rate	40 Hz
Smoothing	G4260B: (30) G4261B: (1)
Light Source intensity	100 %

ASTM drift tests require a temperature change below 2 °C/h (3.6 °F/h) over one hour period. Our published drift specification is based on these conditions. Larger ambient temperature changes will result in larger drift. Better drift performance depends on better control of the temperature

fluctuations. To realize the highest performance, minimize the frequency and the amplitude of the temperature changes to below 1  $^{\circ}$ C/h (1.8  $^{\circ}$ F/h). Turbulences around one minute or less can be ignored. ASTM measurements require that the detector should be turned on enough time before start of testing.

2	Site	Regu	iremen	ts and	Sr	pecification	S
_	OILL	HUGUU		to unu	U	Jecineation	•

Performance Specifications



This chapter gives information about the installation of your ELSD.

# **Unpacking the Module**

# **Damaged Packaging**

If the delivery packaging shows signs of external damage, please call your Agilent Technologies sales and service office immediately. Inform your service representative that the instrument may have been damaged during shipment.

## **CAUTION**

"Defective on arrival" problems

If there are signs of damage, please do not attempt to install the module. Inspection by Agilent is required to evaluate if the instrument is in good condition or damaged.

- → Notify your Agilent sales and service office about the damage.
- → An Agilent service representative will inspect the instrument at your site and initiate appropriate actions.

# **Delivery Checklist**

Unpack the ELSD and accessories, and ensure that all parts and materials shown in the table below have been delivered with your module. Report any missing or damaged parts to your local Agilent Technologies sales and service office.

p/n	Description	
	G4260B	Agilent 1260 Infinity Evaporative Light Scattering Detector
OR	G4261B	Agilent 1290 Infinity Evaporative Light Scattering Detector
	PL0890-0355	Electronic Operation Manual, supplied on CD
	PL0890-0325	RS232 communication cable
	8121-0008	LAN shielded cable
	G4260-60005	1200 Series to ELSD Trigger Cable
	PL0890-0305	Gas inlet tube (2 m)
	PL0890-0310	Rear exhaust hose (PVC-2 m)
	G4261-63000	Solvent waste tube (1.9 m)
	N/A	One power cord (region dependant)
	N/A	ELSD Software Drivers

# **Installing the Module**

# **Connecting the Detector to your LC System**

The ELS detector must be installed by an Agilent service representative. Your Agilent service representative will:

- Unpack the ELSD.
- Remove all packing list items that are shipped with the instrument and present them to you for review.
- Unpack the accessories supplied with the instrument and check that you have received everything on the packing lists.
- · Install your ELS detector.
- 1 Place the module on the bench in a vertical position.
- **2** Connect the power cord to the IEC inlet at the rear of the unit.
- **3** Ensure the power switch of the module is OFF (switch stands out).
- **4** Connect the power cable to the power connector.
- **5** Attach the 4 mm OD gas tube into the gas inlet port at the rear of the instrument. The gas connection is a push-in fitting.
- **6** Connect the solvent waste tube to the waste outlet at the front of the detector and position the other end into a waste collection bottle.
- **7** Connect the front outlet tube to the waste bottle using the supplied tubing.
- **8** Ensure that the bottom of the waste tube is below the height of the waste outlet from the instrument.

NOTE

Though it will not cause any damage, if the solvent waste tube becomes submersed below the solvent level inside the waste container positive pressure will be exerted on the nebuliser chamber leading to excessive baseline noise.

#### WARNING

#### Risk of intoxication and contamination of detector

#### Toxic evaporation and accumulation of condensing solvent

- → The exhaust must be extracted to a suitable fume extraction system.
- → Attach one end of the exhaust hose to the rear of the unit.
- → Ensure the exhaust hose has an upward slope from the detector 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.
- → Make sure the other end of the exhaust hose is vented to a fume hood or other disposal unit.

#### **CAUTION**

Negative or positive backpressure at the exhaust

Baseline noise and reduced performance of the detector

- Do not connect the exhaust tubing directly to the extraction unit.
- **9** Connect the exhaust hose between the exhaust outlet and a fume hood.
- **10** For digital data collection and control connect the LAN cable to the LAN interface.

OR

Connect the RS232 serial cable.

- 11 Connect the analog cable (optional).
- 12 Connect the injection trigger cable to the Agilent autosampler.

#### NOTE

Analogue output cable (Analogue output cable (PL0890-0300))

#### NOTE

The ELS Detector is fitted with a LAN or standard RS232 3-wire serial interface.

Both connections provide a 24 bit80 Hz (alternatively 40 or 10 Hz) digital output for connection to a PC running a data acquisition package (e.g. ChemStation software).

For further information on how to configure the PC with OpenLAB or ChemStation software refer to the appropriate user guide supplied with the driver install.

## 3 Installing the Module

Installing the Module

#### NOTE

For further information on how to configure the TCP/IP settings on the detector, see "LAN Configuration" on page 35.

#### NOTE

For digital data collection using ChemStation or EZChrom, connect the ELS detector to the computer using the RS232 or LAN cable supplied with the detector. The 1200 Series to ELSD Trigger Cable (G4260-60005) is also required to start an injection, and should be used to connect to the 1200 instrument remote connector.

#### NOTE

The ELS Detector can be connected to auxiliary equipment, such as an autosampler, or pump via the 15pin remote start cable. The remote start cable can be configured in several ways to allow on-board timetable events to be triggered or to remotely auto-zero.

The ELS detector is equipped with 2 contact closures (normally-open) for stopping the operation of a pump if the unit reports an error condition. Pump stop facility must be employed if the instrument is to be left unattended, or if units are stacked.

The ELS Detector is also equipped with one contact closure, which is normally open, and two TTL logic inputs, both active-low (with internal pull-up resistors to 5 V).

For ELS-Detector I/O connections see Table 1 on page 15

- 13 Install ELSD drivers for full control via OpenLab CS or EZChrome.
- **14** Connect the LC column outlet to the eluent inlet at the front of the unit) using the shortest length of tubing possible (1/16 in OD, 0.010 in ID).

## NOTE

The eluent from the chromatography system is connected to the central front port of the ELS Detector via a low dead volume Valco® bulkhead connector.

The liquid inlet port is connected directly to the nebuliser by a short length (130 mm) of capillary tube giving a delay volume from port to nebuliser tip of  $\sim$ 4  $\mu$ L.

**15** Turn on the source gas to a pressure of about 4.1 – 6.9 bar.

# **Checking Operation of the ELS Detector**

- **1** Switch on the ELS Detector. If using OpenLAB CDS ChemStation or EZChrom, the front panel keypad will become locked when launching the software.
- **2** In ChemStation or EZChrom, set operational parameters (i.e. nebuliser and evaporator temperature), and download them to the instrument. Then turn the ELS detector status to **0n**.

#### NOTE

For control outside of ChemStation and EZChrom, use the front panel to change the ELS detector settings. Initiate heating by selecting **RUN** mode using the arrow keys on the front keypad.

#### NOTE

When the unit has reached temperature, the baseline noise should be checked to ensure that it is < 0.2 mV peak-peak. This verifies that the gas supply is clean and dry.

**3** If the baseline noise is within the limits, autozero the detector. However, if the baseline noise is outside the limits, refer to the Troubleshooting section for steps to rectify this.

#### NOTE

Baseline noise should not have increased significantly and should be  $\leq$ 0.5 mV. Typically pure water should give no more than 0.4 mV peak-peak, whilst pure organic solvents should be less than 0.3 mV.

- **4** Turn on the eluent flow and allow the system to stabilize.
- **5** Again check the baseline noise.

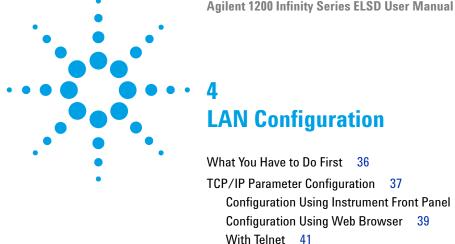
Where noise and all other conditions are acceptable, the instrument is ready to begin work.

#### NOTE

Some volatile buffers can generate considerable offsets and increased noise levels.

3 Installing the Module Installing the Module

PC and Agilent ChemStation Setup PC Setup for Local Configuration Agilent ChemStation Setup 47



software.

This chapter provides information on connecting the module to the controller

38

NOTE

This section only applies to the 1290 Infinity ELSD (G4261B). However, the 1260 Infinity ELSD (G4260B) will require the RS232 for communication as the LAN port is disabled and capped off to prevent use.



# 4 LAN Configuration What You Have to Do First

# What You Have to Do First

The Agilent 1290 Infinity ELSD (G4261B) has an on-board LAN communication interface.

- 1 Note the MAC (Media Access Control) address for further reference. The MAC or hardware address of the LAN interface is a worldwide unique identifier. No other network device will have the same hardware address. The MAC address can be found at detector start up (see "Configuration Using Instrument Front Panel" on page 38).
- 2 Connect the instrument's LAN interface to
  - the PC network card using a crossover network cable (point-to-point)
  - · a hub switch using a standard LAN cable.

# **TCP/IP Parameter Configuration**

To operate properly in a network environment, the LAN interface must be configured with valid TCP/IP network parameters. There parameters are:

- · IP address
- · Subnet Mask
- · Default Gateway

The TCP/IP parameters can be configured by the following methods:

- · by manually setting the parameters via the front panel of the detector,
- by manually setting the parameters through the G4261B ELSD web-interface,
- · by manually setting the parameters through a Telnet session.

The module can be configured, via the front panel, to obtain an IP address automatically or to use a static IP address (see Table 5 on page 37). When configured to obtain its IP address automatically, the module will request an IP address from the DHCP server after every power cycle.

NOTE

If the detector is assigned a temporary IP address (e.g DHCP server) then communication with the PC may be prevented following a power cycle of the instrument.

**Table 5** Default STATIC/IP parameters

IP address	192.168.254.27
Subnet Mask	255.255.255.0
Default Gateway	192.168.254.1

## **Configuration Using Instrument Front Panel**

Configuration of the TCP/IP parameters via the front panel is the recommended method for setting up the module's LAN interface. On-board configuration is only possible during the module's start-up procedure.

1 Power on the detector, and press any front key when the following message is displayed "Press a key to enter communication setup".

#### NOTE

If no key is pressed within 5 seconds, the ELSD will continue its start-up routine. The previously stored TCP/IP parameters will be used.

- 2 Select LAN using left/right arrow keys and press AZ/Stop to confirm.
- **3** Select either Static or DHCP (automatic) IP address assignment and press **AZ/Stop** to confirm.
- **4** If Dynamic IP address is selected, the instrument will try to obtain an IP address from the network.
  - When successful the detector will continue to the main menu screen.
- **5** If Static IP address is selected, the instrument will display the stored static TCP/IP settings.

#### NOTE

The detector will use the default TCP/IP settings if no IP address has been programmed previously (see Table 5 on page 37).

- 6 To enter a new IP address, use the up/down arrow keys.
- 7 To enter a new Subnet Mask use the up/down arrow keys.
- 8 To enter a new Gateway address use the up/down arrow keys.
- **9** When all values are populated press **AZ/Stop** to accept.

#### NOTE

The instrument will return an INVALID network setting message if the IP address and Gateway combination are incompatible.

The instrument will continue to the main menu screen to confirm the settings have been implemented.

Once the LAN settings are configured they are stored in memory even after a power cycle.

#### NOTE

The MAC address and Host Names of the instrument are displayed on the LAN configuration screens for reference.

## **Configuration Using Web Browser**

This procedure describes how to change the TCP/IP parameters and hostname using a PC, via a web browser.

1 Using a web browser such as Internet Explorer, type in the Host name or IP address of the ELSD into the address bar (for example http://192.168.254.27).

NOTE

The default host name is configured to the detector's serial number.

If communication is successful, the following login screen will appear:



- **2** To login, type the following information:
  - Username:

user

Password:

Agilent

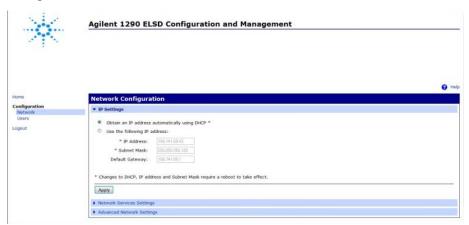
NOTE

The login details are case-sensitive.

#### 4 LAN Configuration

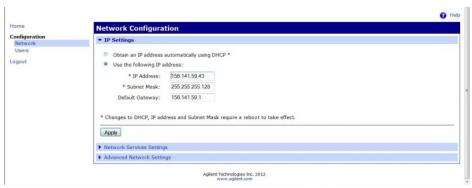
**TCP/IP Parameter Configuration** 

**3** From the left-hand menu, select **Network** to configure the TCP/IP settings.



The detector can be configured to

- · obtain an IP address automatically (DHCP),
- use a static IP address,
- · change Hostname (Advanced Network settings).
- 4 To use a static IP address, select Use the following IP address.
- 5 Enter the module's IP Address, Subnet Mask and Default Gateway address and click Apply.



- 6 Select Apply again to confirm the changes.
- 7 Select Logout to close the session.
- 8 Restart the module for the new TCP/IP settings to be implemented.

NOTE

Contact your system administrator for assistance on configuring the detector's network settings.

NOTE

Changes to the TCP/IP parameters, other than those described, could render the ELSD inoperable.

## With Telnet

Whenever a TCP/IP connection to the module is possible (TCP/IP parameters set by any method), the parameters may be altered by opening a Telnet session.

1 Open the system (DOS) prompt window by clicking on Windows **Start** button and select **Run...**.

OR

Press the Windows button and R simultaneously, and click OK.

- **2** Type the following into the **RUN** dialog:
  - Telnet <IP Address>
  - Telnet <host name>

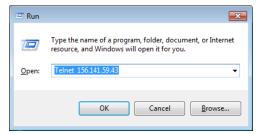


Figure 5 Starting a Telnet session

where <IP address> may be the assigned address via the instrument, a configuration session using the web interface of the default IP address (see Table 5 on page 37).

NOTE

If Telnet is not installed on your PC by default, you can turn on the feature via Windows Control Panel.

#### 4 LAN Configuration

**TCP/IP Parameter Configuration** 

When the connection is established successfully, the module responds with the following:

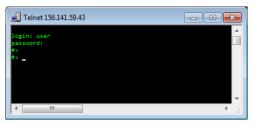


Figure 6 A connection to the module is made

- **3** Enter the following information when prompted:
  - · login:user
  - Password:Agilent

NOTE

The login details are case sensitive. The password will be not visible on screen.

4 At the #> prompt type set network? to view the available commands.

```
Telnet 156.141.59.43

Proset network

Syntax (syst network [options...]

outline:

ip-(coader)

summast = (school mast)

parterpy (gottera, ipande)

static [ontor)

static [ontor)
```

T	T 1 4
Table 6	Telnet commands

Value	Description
set network ?	displays syntax and descriptions of commands
set network	displays the current settings and list of commands
set network ip= <x.x.x.x></x.x.x.x>	sets new ip address
set network submask= <x.x.x.x></x.x.x.x>	sets new subnetmask
set network gateway= <x.x.x.x></x.x.x.x>	sets new default gateway
set network static=on/off	sets the ip to not change
set network dhcp=on/off	sets the device to or to not get its id from a dhcp server
set network autoip=on/off	sets the device to or tonot try to automatically get an ip
quit	closes telnet and disconnects you

**5** To change a parameter follow the style:

Parameter value, for example:

set network static=on
set network ip=156.141.59.43

Then press **Enter**.

Parameter refers to the configuration parameter you are defining, and value refers to the definitions you are assigning to the parameter. Each entry is followed by a carriage return.

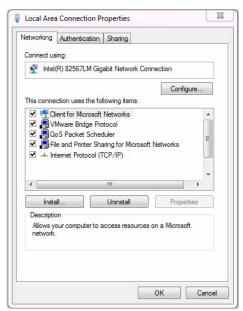
- **6** When you have finished typing the configuration parameters, type **quit** and press **Enter** to exit.
- 7 Restart the module for the new TCP/IP settings to be implemented.

# **PC and Agilent ChemStation Setup**

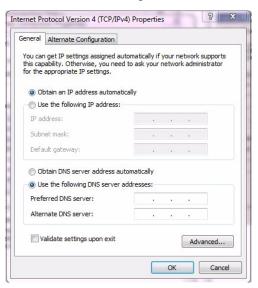
# **PC Setup for Local Configuration**

This procedure describes how to the change the TCP/IP settings on your PC to match the modules default parameters in a local configuration using a cross-over LAN cable (see Table 5 on page 37).

1 Open the Local Area Connection Properties and select Internet Protocol (TCP/IP). Then click on Properties.



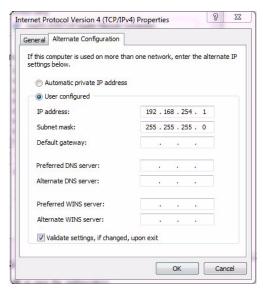
#### 2 Select Alternative Configuration.



## 4 LAN Configuration

**PC** and Agilent ChemStation Setup

- 3 Enter the following IP and subnet mask address for the PC:
  - IP address:
     192.168.254.1
  - Subnet mask:
     255.255.25.0



**4** Click **OK** to save the configuration.

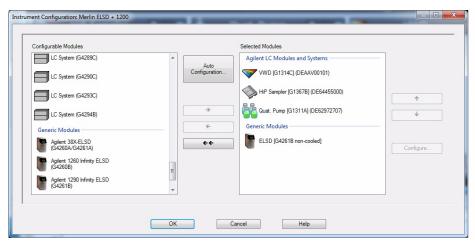
NOTE

It can take several seconds for the alternative network settings to take effect.

# **Agilent ChemStation Setup**

This procedure describes the Agilent OpenLAB ChemStation setup for the 1290 Infinity ELSD detector (G4261B) using LAN as the communication method.

1 Open the Instrument Configuration window.



**2** Select the required ELSD module and choose **Configure** to set the communication parameters.

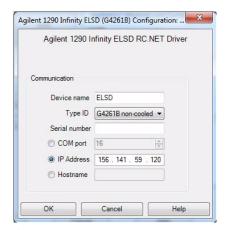


3 Select the instrument Type ID, according to the ELSD model connected.

#### 4 LAN Configuration

**PC and Agilent ChemStation Setup** 

4 Select IP Address and enter the module's IP address.



OR

Select **Hostname** and enter the instrument's serial number.



**5** Select **OK** to complete the configuration.

## NOTE

The detector's serial number is the default hostname.

#### NOTE

If the detector is assigned a temporary IP address (e.g DHCP server) then communication with ChemStation maybe be prevented following a power cycle of the instrument.

Recommend using a static IP address to ensure communication with ChemStation is preserved after power cycle.



# 5 Using the Module

before using the betector of
Instrument Controls 52
Display Screen 52
Keypad 52
Main menu bar 53
Sub-Menu Screen 53
Status Mode 55
Error conditions 56
Clearing an Error 56
Operational Parameters 57
Method 57
Evaporator Temperature 58
Nebuliser Temperature 58
Evaporator Gas Flow 59
Detector Gain (PMT) 59
Response Time (Smoothing) 60
Light Source Intensity (LED) 60
Power Mode 61
Data Output Rate (Hz) 61
LAN (TCP/IP Settings) 62
Controlling the ELSD During an Injection 63
Real-time Operation 63
Creating a Real-time Program 64
Starting and Stopping Real-time Control 64
Controlling the ELSD within ChemStation or EZChrom 66
General Considerations 67
Solvent Recommendations 68
Sample Preparation 68



## 5 Using the Module

**PC and Agilent ChemStation Setup** 

Column Considerations 68
Transferring ELSD Temperature Methods 70

This chapter explains the operational parameters of the ELSD.

# **Before Using the Detector**

On start-up of the Agilent 1290 Infinity ELSD (G4261B), either Serial or LAN must be selected as the method of communication.

If Serial communication is selected, then no further configuration is required. The Agilent 1260 Infinity ELSD (G4260B) can only be connected through Serial communication.

When LAN communication is selected, further configuration of the network settings is required (see "LAN Configuration" on page 35).

NOTE

The Agilent 1260 Infinity ELSD does not have LAN communication functionality.

## **Instrument Controls**

The ELS Detector can be used as a standalone detector via the front keypad and screen, as shown in Figure 7 on page 52 or via PC control using software (e.g ChemStation).

## **Display Screen**

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



Figure 7 ELSD Display Screen

## **Keypad**

The four arrows on the front of the instrument are used to navigate within the interactive menu bar. The AZ/Stop key has a dual function; it can be used to auto zero the ELSD at any time, unless a timetable is running. If the AZ/Stop key is pressed during an active timetable, the timetable will stop running and the ELSD will revert to STANDBY mode.

## Main menu bar

To change the current settings, use the arrow keys to navigate across the interactive menu bar until the desired option is flashing. Using the up/down arrow keys alter the parameter to the desired setting. In order to action any changes, the cursor must be returned to the "Home" position ( $^{\circ}$  or  $^{\circ}$ ).



When the cursor is located in the "Home" position, the actual detector values are displayed in the main screen. If the instrument is controlled via PC software, then the home key will display a locked icon § and the keypad will be disabled. To unlock the keypad, software control must be terminated.

## Sub-Menu Screen

The sub-menu screen is accessed from the front screen by selecting the  $\downarrow$  key: This screen allows changes to the following electronic parameters:

PMT	Set Signal Gain
SMTH	Set Time Constant
LED	Set Light Source Intensity (G4260B only)
PWR MODE	Set Mode of ELSD when powered up
HZ	Set Data Output Rate
LAN	Displays the Instruments TCP/IP settings (G4261B only)
-	

## 5 Using the Module

**Instrument Controls** 



Figure 8 G4261B sub menu screen

NOTE

The LED option is not displayed in the G4261B ELSD sub-menu.



Figure 9 G4260B sub menu screen

### **Status Mode**

The ELS Detector can be operated in two modes; STANDBY or RUN, both of which are described over page:

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 arrow 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 ELS detector, which is by default initiated automatically after power on (default can be changed using Power Mode, "Power Mode" on page 61). In *STANDBY* mode the heaters and light source are switched off, and the gas manifold valve is closed at power on. 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 flow set to a minimum flow of 1.2 SLM for 15 min 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.

#### **CAUTION**

Flooding the detector

If the instrument is left in Standby mode for longer than 15 minutes, gas flow to the unit is stopped to minimize gas usage.

The solvent pump must be turned off if the ELSD is going to be left in Standby mode longer than 15 minutes to prevent solvent flooding the detector.

#### **RUN**

The RUN mode is the detector's operational mode. In this mode the instrument is 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.

#### 5 Using the Module

**Instrument Controls** 

## **Error conditions**

The ELS Detector 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, light source and gas are turned off. A complete list of instrument errors and remedial actions are given in the troubleshooting section of this manual.

## **CAUTION**

Solvent flooding the detector

→ It is strongly recommended 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.

# **Clearing an Error**

Once the source of the problem has been corrected, select RUN mode to put the ELSD back into its operational state. If the problem has not been rectified the ELSD will repeatedly error when RUN mode is selected.

# **Operational Parameters**

## Method

The ELS Detector has 10 on board pre-set methods. These methods comprise, evaporator and nebuliser temperature and gas flow, which can be optimized for specific applications.

These 10 on-board methods are selected using the front keypad and screen, via the METHOD option.

In addition to the 10 on-board pre-set methods, the ELS Detector has a method XXX that allows modification of the ELSD parameters to be made without the need for software control. Method XXX allows the detector to be used in standalone mode via the front screen and keypad.

#### Loading a Method

To load one of the 10 on-board methods, highlight METHOD. Using the arrow 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. These on-board methods cannot be edited.

## **Evaporator Temperature**

The evaporator temperature is the most important setting on the ELS detector. This should be set according to the volatility of the compound(s) being analyzed.

If the compound is non-volatile, e.g. sugars, then the evaporator temperature should be set to 80 - 90 °C.

If the compound is semi-volatile, or has a low molecular weight, e.g. pharmaceutical drug, then the evaporator temperature should be set between 20 - 30 °C.

The evaporator temperature ranges for the ELS models are as follows:

G4260B/G4261B ELSD (non-cooled) OFF, 25 - 120 °C (1 °C increments) G4261B ELSD (cooled) OFF, 10 - 80 °C (1 °C increments)

The default evaporator temperature for both models is 30 °C.

## **Nebuliser Temperature**

The nebuliser temperature can be used to optimize signal response in addition to evaporator temperature. Higher nebuliser temperatures increase peak response, but the nebuliser temperature must not exceed the boiling point of the mobile phase.

The nebuliser temperature range for both models is: OFF, 25 - 90 °C (1 °C increments).

The default value is 30 °C.

## **Evaporator Gas Flow**

The evaporator gas flow is used to control the ELS detector's evaporation process. The evaporator gas value is set according to the mobile phase composition, with higher gas flows (e.g. 1.6 SLM) being used for aqueous eluent compared to those containing organic solvents.

The higher the evaporator temperature the lower the evaporation gas setting required (e.g. 1.0-0.9 SLM), regardless of mobile phase composition. Likewise, as the evaporator temperature is reduced to ambient and sub-ambient temperatures, the gas flow needs to be increased to compensate (e.g. 1.6-1.8 SLM).

The evaporation gas range for both models is: 0.9 - 3.25 SLM (0.05 increments).

The default value is 1.6 SLM.

# **Detector Gain (PMT)**

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

When setting the PMT (or Gain), both the signal and noise are simply amplified by the value set, so S/N values are unaffected. The raw signal output displayed on the parameter screen will reflect this increase or decrease in signal amplification.

Please note that the instrument output displayed on the main operating screen does not alter following a PMT change, thus the recorded baseline position will remain unchanged. Confirmation of a PMT change will be obvious by the change in baseline noise.

# **Response Time (Smoothing)**

The data outputted from the detector can be averaged to produce a smoother response. The smoothing width is set to the number of data points over which the data is averaged and can be regarded as a digital time constant. The smoothing range is settable from 1-50, (in increments of 1) which translates to 0.1-5.0 s.

For most HPLC applications the default value of 30 (3 s) is satisfactory. However for faster separations where peak widths <3 s, a setting of 1(0.1 s) is recommended.

For GPC applications where peak widths can be >30 s, a value of 50 (5 s) is recommended.

# **Light Source Intensity (LED)**

The G4260B ELS detector's LED intensity can be adjusted in order to bring the peak response back on-scale. The intensity range can be set between 1-100 %, with the default factory setting being 100 %, for maximum sensitivity. The LED setting is stored in memory and is retained even after a power on/off cycle.

This feature is extremely useful for preparative chromatography where samples of high concentration can be analyzed which would otherwise exceed the dynamic range of the detector.

NOTE

The Agilent 1260 Infinity ELSD (G4260B) performs an automatic auto-zero (i.e. 10 mV) following an LED change in order to keep the signal on-scale.

NOTE

The Agilent 1290 Infinity ELSD (G4261B) Laser light source does not have power adjustment.

## **Power Mode**

The instrument can be configured from the front panel sub-menu (see "Sub-Menu Screen" on page 53), to start in either RUN or STANDBY mode when the unit is switched on via the rear power button.

To configure the Power Mode, select the required Status Mode (i.e. STANDBY or RUN) you wish the unit to start-up in from the sub menu screen (see "Sub-Menu Screen" on page 53). The selected option will take effect the next time the module is powercycled. If RUN mode is selected as the desired Power mode, then the instrument will use the operating parameters stored in memory. In the unlikely event that the instrument encounters a fault during power-up the unit will automatically switch to STANDBY mode.

# **Data Output Rate (Hz)**

The rate at which the ELS Detector outputs data can be selected from the sub-menu screen. A 10 Hz output rate is selectable for standard LC applications, a 40 Hz output rate can be chosen for faster LC separations and 80 Hz is used for UHPLC type applications with very narrow peaks. The data rate is stored in memory and is retained even after a power on/off cycle. The default value is 10 Hz.

NOTE

All ELSD conditions are retained on power cycling of the ELSD detector.

# LAN (TCP/IP Settings)

This screen displays the 1290 Infinity ELSD (G4261B) current configured TCP/IP settings. See "LAN Configuration" on page 35 for details on how too set these values.

Figure 10 TCP/IP settings

# **Controlling the ELSD During an Injection**

The ELS Detector has the capability to change operational parameters in real-time, during a sample injection, using an on-board timetable.

## **Real-time Operation**

The ELS Detector can store in memory a series of time-based events, within a single timetable. This timetable allows the operational settings of the ELSD to be changed in real-time during a run.

The evaporator temperature (G4261B cooled), gas flow, PMT gain, and smoothing parameters can all be configured within this timetable to change during a sample injection.

The timetable can be used to program the gas flow, in order to compensate for the change in ELSD response across a solvent gradient. Alternatively, the timetable can be customized to create adjustable PMT gain across a sample injection for mixtures containing samples with wide concentration ranges.

The single timetable, stored on-board the ELSD, is only customizable using the ELSD DIMENSION software, which can be purchased from Agilent (ELSD Dimension Software (includes trigger cable) (PL0890-0375)), which also includes Trigger cable for Dimension Software (PL0890-0345)).

## **Creating a Real-time Program**

In order to create or modify the on-board timetable, DIMENSION software must be installed on your PC.

The DIMENSION software package allows you to create a timetable on a PC, which can subsequently be downloaded to the ELSD for later use.

The DIMENSION software also allows you to delete a time table from the ELSD detector memory.

The ELSD can only store a single timetable in memory, so the DIMENSION software can be used to create and save multiple timetables that can be downloaded individually at a later date.

For further information on how to use DIMENSION software, please refer to the DIMENSION software on-line help.

NOTE

Dimension software can only communicate with the ELSD using the Serial port. LAN communication is not supported in DIMENSION.

## **Starting and Stopping Real-time Control**

The ELSD contains an internal timer to initiate the time based events stored within the timetable. To start the internal timer and trigger the on-board timetable a contact closure input via the I/O connector on the rear of the instrument is required (see "Overview of ELS Detector" on page 13).

When the on-board timetable is triggered, the front panel of the ELSD will display TTRUN above the output, as shown in Figure 11 on page 65.

When the timetable is running, the current and the total run time are displayed, in minutes, at the top-centre of the ELSD display. When the timetable reaches the end of its run time, the ELSD will revert to RUN mode and be primed ready to start the timetable again.

During an active timetable, where the evaporator temperature is being controlled, the status of the instrument will change from READY to NOT READY. This is normal behavior and will not affect the running of the timetable.

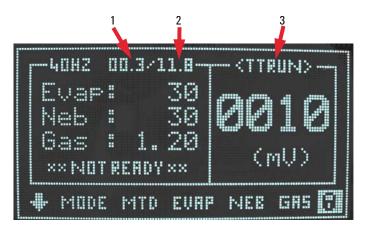


Figure 11 ELSD front panel display during timetable operation

1	Current runtime
2	Total runtime
3	On-board Timetable is active

The **\$TOP** button on the instrument's front keypad can be used to interrupt the active timetable whilst it is running. When the **\$TOP** button is pressed the instrument will revert to STANDBY mode.

# Controlling the ELSD within ChemStation or EZChrom

The ELS Detector can be controlled directly using OpenLAB ChemStation and EZChrom editions and ChemStation Rev B 04.03. Digital data acquisition is performed without the need for an A/D interface. Controlling the ELSD using OpenLAB provides full detector functionality with the added benefit of remote automation. The ELSD Driver is supplied on the CD supplied with the detector.

For further information on how to install and configure the ELSD driver refer to the appropriate user guide supplied with the driver install.

#### NOTE

The ChemStation and EZChrom drivers are only applicable with the following ELSD firmware revisions:

- EZChrom: Firmware v30.0.0 or higher
- ChemStation (with RC.Net driver): Firmware v30.0.0 or higher

## **General Considerations**

The ELS Detector 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 ELSD 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.  $\leq 0.254$  mm ( $\leq 0.010$  in).

The ELSD requires nitrogen of purity >98 %, at an inlet pressure of 4 – 6.7 bar. If in-house nitrogen is not available then we recommend the use of a nitrogen generator 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-volatilized eluent will increase baseline noise and reduce sensitivity.

The ELS Detector is a destructive technique and must be placed last when used in series with other detectors.

### Solvent Recommendations

Any solvent intended for use with the ELS Detector should be thoroughly degassed, filtered (0.45 µm) and fully compatible with the column(s). Solvents that are not properly degassed may cause problems at nebulization leading to a poor reproducibility.

Non-Volatile buffers are not compatible with the ELS Detector and should not be used. Only volatile mobile phase additives, such as those listed in Table 7 on page 69 should be used with the ELS detector.

Tetrahydrofuran (THF) stabilized with BHT, may increase the baseline noise level. Where possible unstabilized THF should be used with the ELS detector.

High boiling point solvents such as N-methylpyrrolidone (NMP), Dimethylsulphoxide (DMSO), m-Cresol and 1,2,4-Trichlorobenzene (TCB) are not recommended. Greater care and attention to instrument cleaning procedures should be exercised with these solvents.

## Sample Preparation

Samples containing particulate matter should be filtered through a 0.45 µm filter prior to injection.

## **Column Considerations**

The ELS detector will detect all non-volatile components in the mobile phase, which includes column-packing material. Column packing material will become chemically and mechanically broken down over the lifetime of the column, causing particles to enter the ELSD. This column "shedding" will lead to extremely high baseline.

Amino columns used with aqueous mobile phase are particularly prone to this type of shedding and should be checked regularly. To minimize column breakdown always follow the manufacturers instruction supplied with the column.

 Table 7
 Volatile mobile additives compatible with ELS detection

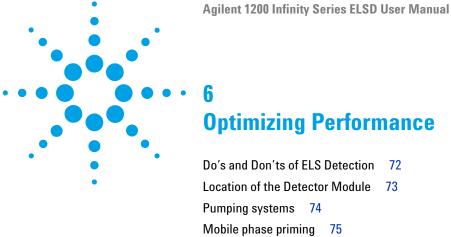
Mobile Phase Additive	рКа	pKb	pH Range	Bp (°C)	Mp (°C)
Acids					
Trifluoroacetic Acid (TFA)	0.3	13.70		72.4	-15.4
Formic Acid	3.75	10.25		100.7	8.3
Acetic Acid	4.75	9.25		116.0	16.6
Bases					
Ammonia	9.25	4.75		-33.4	-77.7
Methylamine	10.66	3.34		-6.6	-94.0
Ethylamine	10.81	3.19		16.6	-81.0
Triethylamine	11.01	2.99		89.3	-114.7
Buffers					
Ammonium Formate	3.8 9.2		3.0 - 5.0 $8.2 - 10.2$		120
Ammonium Acetate	4.8 9.2		3.8 - 5.8 $8.2 - 10.2$		111
Ammonium Bicarbonate	6.3 9.2 10.3		6.8 – 11.3		106
Ion-Pair Reagent					
Pentafluoropropionic acid (PFPA)	~0.6			97	
Heptafluorobutyric acid (HFBA)	~0.6			120	
Nonafluoropropionic acid (NFPA)	~0.6			140	
Tridecafluoroheptanoic acid	~0.6			175	

## **Transferring ELSD Temperature Methods**

The direct transfer of ELSD operating conditions from other manufacturers' ELS detectors, or other designs of ELSD (e.g. Agilent G4218A ELSD) to the ELSD will not provide equivalent performance.

As stated in "Basic Principles of Operation" on page 9, the operating temperatures of the ELS detector are set according to the type of analyte and not the mobile phase composition as with other ELS detectors. For example, when HPLC grade water is used as the mobile phase the Agilent G4218A ELSD requires an evaporation temperature between  $35-40\,^{\circ}\text{C}$ . Whereas the Agilent G4261B can be operated as low as  $20-30\,^{\circ}\text{C}$  for the equivalent solvent.

Therefore the transfer of operating conditions from other models of ELSD to the ELSD is not valid and the only way to ensure that the detector will provide the optimum analyte signal-to-noise is to follow the guidelines outlined in "Operational Parameters" on page 12.



This chapter gives hints on how to optimize the performance or use additional devices.

Solvent recommendations 76

## Do's and Don'ts of ELS Detection

## CAUTION

Decreased performance

High pressures on the internal chamber will lead to increased baseline noise and low sensitivity.

- → NEVER block the exhaust outlet.
- → NEVER allow the solvent waste outlet tube to become immersed in the waste solvent.
- → When placing more than one HPLC detector in series, always place the ELS detector last.
- → Only use volatile mobile phase additives.

#### **Location of the Detector Module**

Place the detector conveniently near your HPLC system. The modular design of the ELSD enables you to locate it anywhere within the limitations imposed by the length of the power cord, fluid lines and signal cables.

In order to keep liquid dead volume as low as possible and to minimize peak broadening in the lines, the distance between the column outlet and the flow cell inlet should be kept to a minimum.

Provide approximately four inches (10 cm) of space behind the unit so that the cooling fan intake is not impeded, and to allow easy access to the rear panel.

The ELS detector can be placed within 2 meters of an extraction unit, using the exhaust tube provided.

#### 6 Optimizing Performance

**Pumping systems** 

# **Pumping systems**

It is recommended to use a high-performance pumping system with no flow pulses to minimize nebulization problems. Inconsistent solvent flow will result in poor reproducibility.

A backpressure regulator maybe necessary on certain pumps in order to minimize pulsation. This can also be achieved by the column itself or a coil of  $0.127~\mathrm{mm}$  ( $0.005~\mathrm{in}$ ) ID tubing placed between the pump and the column.

# Mobile phase priming

The ELS detector does not require any mobile phase priming, other than that required to prime the solvent through the pump, damper, injector, column, etc. It is recommended that priming of the LC system be performed without the ELS detector attached, to prevent non-volatile impurities contaminating the ELS detector.

The mobile phase should be degassed and filtered, either by sparging with Helium or using an on-line degasser.

#### 6 Optimizing Performance

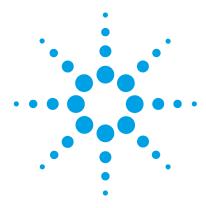
Solvent recommendations

#### **Solvent recommendations**

The maximum operating temperature of the detector and the boiling point of the solvent will determine the type of solvents compatible with the ELS detector. High boiling point solvents such as

- · N-methylpyrrolidone (NMP),
- · Dimethylsulphoxide (DMSO),
- · meta-Cresol (m-Methylphenol) and
- 1,2,4-Trichlorobenzene (TCB)

are not recommended. Greater care and attention to instrument cleaning procedures should be exercised with these solvents. It is recommended that any solvent be flushed from the detector for overnight and weekend storage.



# Troubleshooting and Diagnostics

```
Troubleshooting 78
Troubleshooting an HPLC System
General Problems
   Baseline noise 80
   Baseline spikes 81
   Low sensitivity 81
   Spiky peak tops but flat baseline
   Large Baseline offset 83
   Peak tailing 83
   Instrument Fails to zero 83
   No power 84
   No response (completely flat baseline) 84
   Temperature error as soon as instrument powered on 85
   Display not on, but power connected 85
   Evaporator Temperature reads zero at start-up and cannot be
   changed 85
   Vapor sensor error occurs, but there is no solvent or vapor leak inside
   unit 86
   Cooled Evaporator will not reach low temperature e.g. 10 °C 86
   High back-pressure from detector 86
```

This chapter gives an overview about the troubleshooting and diagnostic features.

#### 7 Troubleshooting and Diagnostics

**Troubleshooting** 

# **Troubleshooting**

If a problem is encountered Agilent Technologies advises that the troubleshooting section should be followed first to resolve the problem. If there is an error or fault and you follow the recommended course of action and the result is not satisfactory, then please direct the matter to Agilent Technologies or your local distributor.

Malfunctions within the ELS Detector can arise from three general sources:

- The ELS Detector itself can be dirty or operating outside specification.
- The HPLC system can have a broken, dirty, or non-optimally operating component, but the problem is manifesting itself in the ELS Detector.
- A mobile phase and/or column problem, which by its very nature is spread throughout the HPLC system but appears as a malfunction of the ELS Detector.

To troubleshoot the ELS Detector, you must be able to separate the performance of the ELS Detector within the HPLC system from its performance outside the HPLC system. This section begins with guidelines for testing the ELS Detector as a stand-alone. See "Module Specific Error Messages" on page 89 for possible cause and suggested solution.

# **Troubleshooting an HPLC System**

Standard practice is to add one component at a time back into the HPLC system so that the component causing the problem is easily identified if/when the condition reoccurs.

Begin troubleshooting by adding the pump to the ELS Detector first and finish by adding the column last. If another type of detector is available use it before the ELS Detector to aid in troubleshooting.

# **General Problems**

#### **Baseline noise**

Probable cause	Suggested actions
1 Poor nebulisation	Increase the temperature of the nebulizer by 10 °C until the baseline noise decreases
2 Insufficient evaporation	<ul> <li>Increase the temperature of the evaporator by 10 °C until the baseline noise decreases</li> <li>Increase the evaporation gas flow rate</li> <li>Decrease the nebulisation temperature</li> </ul>
3 Non-volatile additive in mobile phase	Use a volatile mobile phase as shown
4 Pressure difference created inside nebuliser chamber	<ul> <li>Ensure that the end of the liquid waste tube is not immersed in liquid</li> <li>Ensure that the exhaust tube at rear of unit</li> </ul>
	is not blocked, or extraction is too strong
<b>5</b> Pump pulsations, especially in microbore applications where low flow rates are used	<ul> <li>Use a pulse free pump</li> <li>Increase the back pressure on the pump by fitting a back pressure column between the pump and the injection valve</li> <li>Use a pulse dampener directly after the pump in the system</li> </ul>

# **Baseline spikes**

Probable cause		Suggested actions
1	Particulate matter in the gas supply	Filter the incoming gas, or change the supply
2	Column shedding	Replace column or fit an inline filter with a 0.2 µm membrane filter directly after the column
3	Poor nebulisation	Check solvent flow rate into ELSD is constant
		<ul> <li>Check inlet gas flow is &gt;60 psi and stable</li> </ul>
4	Insufficient evaporation	Increase the temperature of the evaporator by 10 °C until the baseline noise decreases
5	Non-volatile additive in mobile phase	Replace additive in mobile phase with volatile buffer

# Low sensitivity

Pr	obable cause	Suggested actions
1	Partial blockage in Nebuliser or nebuliser inlet tube	Pump a 50/50 water/acetone mixture into ELSD at highest flow rate possible (do not exceed 5 mL/min) for 16 h
2	Internal solvent trap is empty	Fill the front solvent trap with liquid until any excess flows out through front drain tube
3	Gas pressure too low	Ensure inlet gas pressure >60 psi
4	LED power too low	Ensure LED power is set to 100 $\%$
5	Optical chamber is contaminated	Clean contamination from widows in optical chamber
6	Light source power has decayed	Replace light source
7	Diffuser saturated with solvent	Stop the eluent flow and increase the evaporator temperature to maximum. Increase the flow rate to 2.8 SLM and wait 1 h

# Spiky peak tops but flat baseline

Pr	obable cause	Suggested actions
1	Inconsistent nebulization	Nitrogen is the recommended gas - others can be used but may not nebulize as efficiently
2	Incorrect gas being used	Change gas to nitrogen or evaluate different nitrogen sources
3	Poor regulation of inlet gases	If using bottled gas, check that gas regulator is functioning correctly and giving consistent flow
		Alternatively, use a pulse dampener
4	Insufficient smoothing	The broader the peaks, the higher the smoothing value is required. Increase smoothing to 50 for Flash separations
5	Sample precipitation during nebulization	Reduce sample concentration or inject same loading in larger volume
		<ul> <li>Check sample solubility in mobile phase eluents</li> </ul>
6	Inconsistent pump flow rates	See remedy for pump pulsation in the baseline noise section

# **Large Baseline offset**

Probable cause		Suggested actions
1	Inefficient evaporation	Increase the evaporator temperature and/or gas flow
2	High concentration of non-volatile buffer or stabiliser	Use a lower concentration of stabiliser, unstabilised solvent or a more volatile buffer (ammonium acetate or ammonium formate)
3	Contaminated diffuser	Perform cleaning procedure
4	Optics Heater failed	Refer to local distributor or Agilent Technologies

# **Peak tailing**

Probable cause		Suggested actions
1	Eluent particles lingering in the optical chamber	Increase evap gas flow rate
2	Poor chromatography	Optimize HPLC separation

#### **Instrument Fails to zero**

Pı	robable cause	Suggested actions
1	Offset too high or output unstable due to impurity in mobile phase	Stop pump flow and switch off unit. Restart unit and A/Z without liquid flowing
		<ul> <li>Refer to local distributor or Agilent Technologies</li> </ul>
		Optical section contaminated and requires cleaning

#### 7 Troubleshooting and Diagnostics

**General Problems** 

# No power

Probable cause		Suggested actions
1	Mains lead not connected	Attach mains lead to socket and inlet on rear of instrument
2	Fuse failure	Replace fuse
3	Power supply failure	Call Agilent Service representative

# No response (completely flat baseline)

Pr	obable cause	Suggested actions
1	Data acquisition leads not connected	Ensure connectors to computer or integrator are sound
2	Light source inactive	• Check LED intensity is 100 %
		Check LED or LASER is functioning correctly, by stopping solvent flow, cycling the power. Then reading the offset value in RUN mode
3	Output below 0 mV	Stop pump flow and A/Z without liquid flowing
4	Instrument in STANDBY mode	Select RUN mode
5	Nebuliser or nebuliser inlet tube blocked	Manually syringe water into ELSD front Inlet port to remove obstruction

# Temperature error as soon as instrument powered on

Probable cause	Suggested actions
1 Temperature probe fault or disconnected	Check RTD connections
	<ul> <li>Consult Agilent Technologies or your local agent for further advice</li> </ul>

## Display not on, but power connected

Probable cause	Suggested actions
1 Instrument Power Supply	Refer to local distributor or Agilent Technologies.
2 Faulty display	Refer to local distributor or Agilent Technologies.

# **Evaporator Temperature reads zero at start-up and cannot be changed**

Probable cause	Suggested actions
1 Peltier cooler has not initiated correctly	<ul> <li>Switch detector off then on at the power socket</li> </ul>
	<ul> <li>Consult Agilent Technologies or your local agent if problem persists</li> </ul>

#### 7 Troubleshooting and Diagnostics

**General Problems** 

# Vapor sensor error occurs, but there is no solvent or vapor leak inside unit

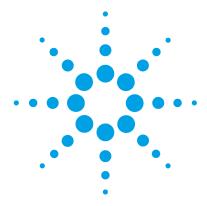
Probable cause		Suggested actions
1	Solvent vapor near the front of unit is being drawn into the unit	Remove any solvent bottle or solvent leak that is directly in front of the detector
2	Faulty Vapor sensor	Check the rear vapor sensor is not damaged/bent

# Cooled Evaporator will not reach low temperature e.g. 10 °C

Probable cause		Suggested actions	
1	Ambient lab temperature too high	Move detector to laboratory where ambient temperature is <25 $^{\circ}\text{C}$	
2	Faulty Peltier cooler unit	Consult Agilent Technologies or your local agent if problem persists	

# **High back-pressure from detector**

Probable cause		Suggested actions
1	Nebuliser or nebuliser inlet tube blocked	Manually syringe water into ELSD front Inlet port to remove obstruction



# Error Information

```
What Are Error Messages
Module Specific Error Messages
   Internal temperature exceeded lower limit
                                             89
   Internal temperature exceeded upper limit
   On-board Vapor sensor failed
   Rear Vapor sensor failed
   Vapor detected
                   90
   Leak detected 91
   Fan Failed 91
   Fan Stopped 92
   Nebulizer temperature limit exceeded
                                         92
   Evaporator temperature limit exceeded
                                          93
   LED light source error 93
   Evaporator gas flow rate limit exceeded
   Invalid Nebulizer temperature
   Invalid Evaporator temperature
                                   94
   Fan failed on cooled evaporator
                                   94
   Cooled evaporator current out of range or communication failed
                                                                  95
   Laser temperature out of range
                                   95
   Laser current out of range 95
   Laser interlock open or failed 96
   Communication to Laser failed
   Leak Sensor failed
                       97
```

This chapter describes the meaning of error messages, and provides information on probable causes and suggested actions how to recover from error conditions.



# 8 Error Information What Are Error Messages

# **What Are Error Messages**

Error messages are displayed in the user interface when an electronic, mechanical, or hydraulic (flow path) failure occurs which requires attention before the analysis can be continued (for example, repair, or exchange of consumables is necessary). In the event of such a failure, the red status indicator at the front of the module is switched on, and an entry is written into the module logbook.

# **Module Specific Error Messages**

These errors are detector specific.

#### Internal temperature exceeded lower limit

Error ID: 10

Air temperature inside the instrument is ≤10 °C

#### Probable cause

#### 1 The environmental temperature is outside the specified operating limits of the instrument

#### Suggested actions

- Increase ambient temperature where detector is located
- Move the detector to a warmer location

#### Internal temperature exceeded upper limit

Error ID: 11

Air temperature inside the instrument is >40 °C

#### Probable cause

#### 1 The environmental temperature is outside the specified operating limits of the instrument

#### Suggested actions

- Decrease ambient temperature where detector is located
- Move the detector to a cooler location

#### **On-board Vapor sensor failed**

Error ID: 12

The vapor sensor located on the main control board has failed.

# Probable cause Suggested actions 1 Vapor sensor not connected to the main board Please contact your Agilent service representative 2 Defective vapor sensor Please contact your Agilent service representative

# **Rear Vapor sensor failed**

Error ID: 13

The vapor sensor located on the rear panel of the module has failed.

Probable cause		Suggested actions
1	Vapor sensor not connected to the main board	Please contact your Agilent service representative
2	Defective vapor sensor	Please contact your Agilent service representative

#### Vapor detected

Error ID: 14

Solvent vapor threshold exceeded inside module.

Probable cause		Suggested actions
1	External vapor being drawn into unit	Remove any source of solvent vapours close to the module
2	Solvent leak inside unit	Please contact your Agilent service representative
3	Exhaust tube not fitted	Fit black exhaust tube

#### Leak detected

#### Error ID: 15

A leak was detected inside the module.

Probable cause	Suggested actions
1 Loose nebuliser fittings	Please contact your Agilent service representative
2 Blocked nebuliser causing leak at capillary fittings	Please contact your Agilent service representative

#### **Fan Failed**

#### Error ID: 16

Thermal shut-down of the main cooling fan

Probable cause		Suggested actions
1	Fan cable disconnected.	Please contact your Agilent service representative.
2	Defective fan.	Please contact your Agilent service representative.
3	Defective main board.	Please contact your Agilent service representative.

#### **8** Error Information

**Module Specific Error Messages** 

# **Fan Stopped**

Error ID: 17

A main cooling fan in the module has stopped

Probable cause	Suggested actions
1 Obstruction of fan blades	Please contact your Agilent service representative
2 Defective fan.	Please contact your Agilent service representative.
3 Defective main board.	Please contact your Agilent service representative.

# Nebulizer temperature limit exceeded

Error ID: 18

Nebuliser temperature exceeded threshold after stabilizing

Probable cause	Suggested actions
1 Defective thermocouple	Please contact your Agilent service representative
2 Defective nebuliser heater	Please contact your Agilent service representative
3 Defective main board.	Please contact your Agilent service representative.

## **Evaporator temperature limit exceeded**

Error ID: 19

Evaporator temperature exceeded threshold after stabilizing

Probable cause		Suggested actions
1	Defective thermocouple	Please contact your Agilent service representative
2	Defective evaporator heater assembly	Please contact your Agilent service representative
3	Defective main board.	Please contact your Agilent service representative.

# **LED** light source error

Error ID: 20

The LED light source has failed

Probable cause		Suggested actions
1	LED Light source not connected to the main board	Please contact your Agilent service representative
2	Defective LED Light source	Please contact your Agilent service representative

# **Evaporator gas flow rate limit exceeded**

Error ID: 21

Evaporator gas flow rate exceeded threshold after stabilizing

Probable cause		Suggested actions
1	Insufficient gas inlet pressure	Ensure the gas inlet pressure is above 60 psi
2	Defective mass flow controller	Please contact your Agilent service representative

#### **Invalid Nebulizer temperature**

Error ID: 22

Invalid nebulizer temperature reading

Probable cause		Suggested actions
1	Nebulizer heater not connected to the main board	Please contact your Agilent service representative
2	Defective nebuliser heater	Please contact your Agilent service representative

#### **Invalid Evaporator temperature**

Error ID: 23

Invalid evaporator temperature reading

Probable cause		Suggested actions
1	Evaporator heater not connected to the main board	Please contact your Agilent service representative
2	Defective evaporator heater	Please contact your Agilent service representative

# Fan failed on cooled evaporator

Error ID: 24

Fans on cooled evaporator module have failed

Probable cause		Suggested actions
1	Fan not connected to peltier assembly	Please contact your Agilent service representative
2	Defective peltier fan	Please contact your Agilent service representative

# Cooled evaporator current out of range or communication failed

Error ID: 25

Peltier module current outside of normal range or communication to peltier module has failed.

Probable cause		Suggested actions
1	Defective peltier assembly	Please contact your Agilent service representative
2	Peltier assembly not connected to main board	Please contact your Agilent service representative
3	Defective main board.	Please contact your Agilent service representative.

#### Laser temperature out of range

Error ID: 26

Temperature control on Laser assembly outside of normal operating range.

Probable cause	Suggested actions
1 Defective Laser assembly	Please contact your Agilent service
	representative

#### Laser current out of range

Error ID: 27

Laser current outside of normal operating range.

Probable cause		Suggested actions
1	Defective Laser assembly	Please contact your Agilent service representative

#### Laser interlock open or failed

Error ID: 28

The laser interlock on the detector enclosure is open or failed.

Probable cause		Suggested actions
1	Module enclosure is open	Please contact your Agilent service representative
2	Interlock not connected to main board	Please contact your Agilent service representative
3	Interlock wiring is faulty	Please contact your Agilent service representative
4	Defective main board.	Please contact your Agilent service representative.

#### **Communication to Laser failed**

Error ID: 29

Communication between main board and laser assembly has failed.

Probable cause		Suggested actions	
1	Laser assembly not connected to main board	Please contact your Agilent service representative	
2	Defective Laser assembly	Please contact your Agilent service representative	
3	Defective main board.	Please contact your Agilent service representative.	

#### **Leak Sensor failed**

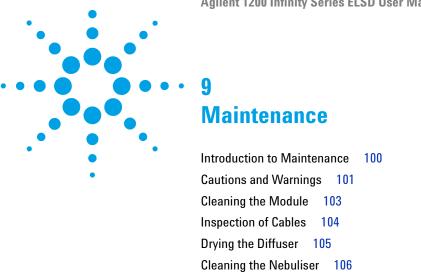
#### Error ID: 30

The leak sensor in the module has failed.

Probable cause	Suggested actions
1 Leak sensor not connected to the main board.	Please contact your Agilent service representative.
2 Defective leak sensor.	Please contact your Agilent service representative.
3 Defective main board.	Please contact your Agilent service representative.

#### **8** Error Information

**Module Specific Error Messages** 



Cleaning Evaporator Tube

Putting the Instrument into Storage 108

Replacing Detector Firmware 109

This chapter describes the maintenance of the ELSD.



#### 9 Maintenance Introduction to Maintenance

# **Introduction to Maintenance**

Trained personnel only should carry out maintenance inside the unit. There are no user serviceable parts inside the instrument. Unauthorized access to the instrument will invalidate the instrument warranty.

# **Cautions and Warnings**

#### WARNING

Toxic, flammable and hazardous solvents, samples and reagents

The handling of solvents, samples and reagents can hold health and safety risks.

- → When working with these substances observe appropriate safety procedures (for example by wearing goggles, safety gloves and protective clothing) as described in the material handling and safety data sheet supplied by the vendor, and follow good laboratory practice.
- → The volume of substances should be reduced to the minimum required for the analysis.
- Do not operate the instrument in an explosive atmosphere.

#### WARNING

#### **Electrical shock**

Repair work at the module can lead to personal injuries, e.g. shock hazard, when the cover is opened.

- → Do not remove the cover of the module.
- → Only certified persons are authorized to carry out repairs inside the module.

#### WARNING

#### Eye discomfort

The light source in the G4260B ELSD is a Class 1 LED product. Temporary discomfort may result from directly viewing the light produced by this source.

Do not look into the beam.

#### 9 Maintenance

**Cautions and Warnings** 

#### WARNING

#### Fire and damage to the module

#### Wrong fuses

- → Make sure that only fuses with the required rated current and of the specified type (super-fast, fast, time delay etc) are used for replacement.
- → The use of repaired fuses and the short-circuiting of fuse-holders must be avoided.

#### CAUTION

Electronic boards and components are sensitive to electrostatic discharge (ESD). ESD can damage electronic boards and components.

→ Be sure to hold the board by the edges, and do not touch the electrical components. Always use ESD protection (for example, an ESD wrist strap) when handling electronic boards and components.

# **Cleaning the Module**

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 deionized water. Ensure that no moisture enters the instrument.

#### WARNING

#### **Electrical shock and burns**

Liquid in the module electronics can cause shock hazard and damage the module.

- → Switch off and disconnect power cord from instrument before cleaning.
- → Do not use an excessively damp cloth during cleaning.
- → Drain all solvent lines before opening any fittings.
- → Allow the instrument to dry off completely before reconnecting power.

#### 9 Maintenance Inspection of Cables

# **Inspection of Cables**

Periodically inspect the connecting cables for signs of physical damage caused by abrasion, solvent spillage, impact etc.

Replace damaged cables, particularly the power cord, if any damage is observed.

# **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, proceed as follows:

- **1** Increase the gas flow to 2.8 SLM and increase the evaporator to maximum temperature.
  - The diffuser will be dried out and the instrument ready to use after approximately 1 h under these conditions.
- **2** Reset the instrument to the correct operating conditions and allow to stabilize before continuing.

# **Cleaning the Nebuliser**

A loss of sensitivity is a common indicator that the nebuliser requires cleaning. Flushing can remove blockages. Therefore it is recommended to initially flush the instrument with a suitable solvent (for example water).

The most common cause of nebuliser blockage is precipitation of mobile phase buffer. This blockage occurs either at the nebuliser tip or within the inlet tube leading to the nebuliser.

#### To clean the nebuliser, the following procedure is recommended.

- 1 Put the ELSD into RUN mode.
- **2** Set the evaporator and nebuliser temperatures to 40 °C and the gas flow to 1.6 SLM.
- **3** Set pump flow rate to 5 mL/min.
- **4** Remove the column, select a suitable solvent (for example water if using aqueous buffers) and pump for 3 h or set pump flow rate to 1 mL/min and run overnight.

NOTE

Pump the highest flow-rate possible if backpressure of ELSD is too high at 5 mL/min.

#### NOTE

It is not possible to pump solvent into the ELSD if the nebuliser becomes completely blocked.

It is strongly recommended to regularly flush the ELSD with water to keep the nebuliser clear of obstruction.

# **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, or a 1:1 mixture of acetone/water.

Depending on usage, it is recommended to clean the evaporator tube once a week or every 40 h of use as a preventative routine. It is also recommended to clean the unit following the use of buffers. If cleaning the unit does not cure the problems then consult Agilent Technologies for further assistance.

NOTE

Do not use solvents that contain additives when performing the cleaning procedure.

#### NOTE

Ensure that the instrument is at equilibrium under the above conditions before leaving the instrument unattended.

- 1 Set the evaporator temperature to 40  $^{\circ}$ C, the nebuliser temperatures at 40  $^{\circ}$ C and the gas flow to 2.8 SLM.
- 2 Pump the "cleaning" solvent into the instrument at 1 2 mL/min, (whilst in the RUN mode) overnight or for a minimum of 4 h if overnight operation is not possible.

#### 9 Maintenance

**Putting the Instrument into Storage** 

# **Putting the Instrument into Storage**

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

- 1 Flush the detector with a mixture of Acetone/Water (50/50) at 1 mL/min for 15 min.
- **2** Allow the instrument to cool to ambient temperature in STANDBY mode with the gas supply still connected.
- **3** Tip the instrument forwards to try and empty the solvent within the nebulization chamber through the front waste tube (i.e. into the bottle).
- 4 Pour 10 20 mL of acetone into the rear exhaust tube to flush out the internal solvent trap, collecting any overflow of acetone at the front solvent pipe.
- **5** Repeat step 3 to drain the acetone.
- **6** Disconnect the waste bottle.
- **7** Using the gas supply, blow nitrogen gas through the exhaust to evaporate any remaining acetone in the solvent trap. Cover the waste tube with tissue paper to collect any acetone residue.
- **8** Plug the exhaust, waste tubes and solvent inlet with the plastic caps provided.

## **Replacing Detector Firmware**

The ELSD contains control firmware that can be flash upgraded.

The Agilent 1290 Infinity ELSD (G4261B) also contains additional firmware on the peltier unit and the Laser assembly.

Firmware on all three assemblies can be upgraded using the same process.

Firmware upgrade is only possible via the serial port.

When For majority of internal repairs

Tools required Description

Allen keys

TERA TERM software Current firmware file

Parts required Description

Serial cable (supplied with instrument)

NOTE

Firmware versions v24.0.4 (for the 380-ELSD) and v24.0.2 (for the 385-ELSD), or later, are not compatible with 1260 and 1290 Infinity models.

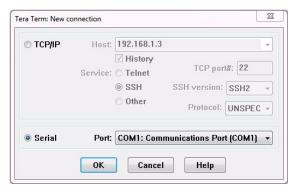
# To upgrade/downgrade the modules firmware carry out the following steps:

- 1 Connect the detector to a PC, via the serial port on the rear of the module, using a RS232 cable.
- 2 Open the Tera Term program, and select File > New Connection from the toolbar.

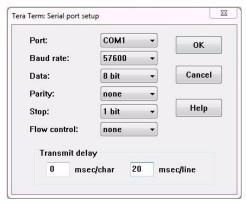
### 9 Maintenance

**Replacing Detector Firmware** 

3 Select Serial and choose the appropriate COM port, followed by OK.



- **4** From the toolbar menu , select Setup > Serial Port to configure the serial port connection.
- 5 Set the Serial port to the parameters shown below and click OK.



The **Transmit delay** values should be set according to the table below:

 Table 8
 Transmit delay values

Firmware type	msec/char	msec/line	
Main	0	20	<u>.</u>
Safety	0	35	
Laser	0	35	

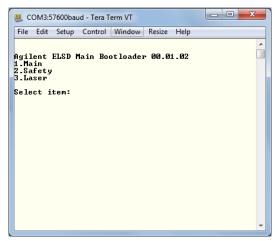
**6** Put the detector into Service mode by holding down the rear Flash button whilst switching on the module.

The front panel will display:

#### **SERVICE MODE**

#### **Reboot for normal operation**

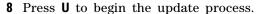
When in Service mode, the Tera Term displays the bootloader version and firmware menu.

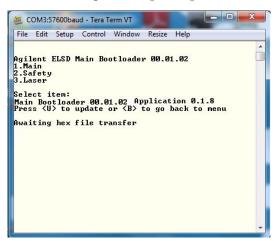


7 Select the firmware you wish to upgrade/downgrade by pressing the appropriate number (e.g. press 1 to upgrade the main control firmware).

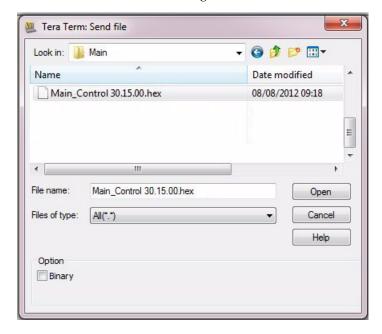
### 9 Maintenance

**Replacing Detector Firmware** 





**9** To choose the firmware file, select the **Send File** option from the **File** menu in the toolbar and navigate to the folder where the file is located.



10 Select the firmware hex file and click Open.

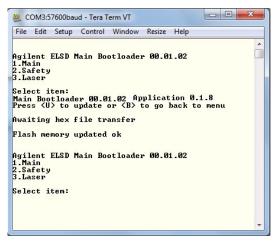
The download will begin straightaway and progress will be displayed, as shown.



NOTE

Do not disconnect or turn off the detector during the transfer process.

11 On successful completion of the file transfer, the software will display a Flash memory updated ok message.



**12** Power cycle the module and check that the firmware version displayed on the ELSD front panel at boot-up is correct.

## 9 Maintenance

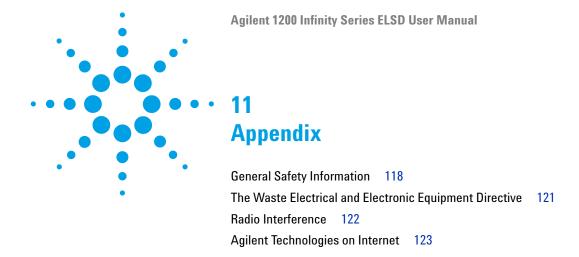
**Replacing Detector Firmware** 



This chapter provides information on parts for maintenance.

# **Identifying Parts and Materials**

p/n	Description
G4260B	Agilent 1260 Infinity Evaporative Light Scattering Detector
G4261B	Agilent 1290 Infinity Evaporative Light Scattering Detector
PL0880-0310	Analogue Cable
PL0890-0305	Gas inlet tube (2 m)
PL0890-0310	Rear exhaust hose (PVC-2 m)
PL0890-0315	Solvent waste tube (2 m)
PL0890-0325	RS232 communication cable
PL0890-0345	Trigger Cable for Dimension Software
PL0890-0350	Remote start cable (third party LCs only)
G4260-90000	ELSD Operation Manual



This chapter provides addition information on safety, legal and web.

# **General Safety Information**

# **Safety Symbols**

 Table 9
 Safety Symbols

Symbol	Description
$\triangle$	The apparatus is marked with this symbol when the user should refer to the instruction manual in order to protect risk of harm to the operator and to protect the apparatus against damage.
\$	Indicates dangerous voltages.
	Indicates a protected ground terminal.
	The apparatus is marked with this symbol when hot surfaces are available and the user should not touch it when heated up.
CLASS 1 LASER PRODUCT	External Laser warning label located on rear of detector
LASER LIGHT AVOID EXPOSURE TO BEAM CLASS 3B LASER PRODUCT 405cm 20mW CW EC 00825-1 Ed 2 0	Internal Laser warning label located on light source
	Internal Laser beam label located on light source

### WARNING

#### A WARNING

alerts you to situations that could cause physical injury or death.

Do not proceed beyond a warning until you have fully understood and met the indicated conditions.

### CAUTION

#### A CAUTION

alerts you to situations that could cause loss of data, or damage of equipment.

Do not proceed beyond a caution until you have fully understood and met the indicated conditions.

## **General Safety Information**

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

### WARNING

Ensure the proper usage of the equipment.

The protection provided by the equipment may be impaired.

→ The operator of this instrument is advised to use the equipment in a manner as specified in this manual.

## **Safety Standards**

This is a Safety Class I instrument (provided with terminal for protective earthing) and has been manufactured and tested according to international safety standards.

## **Operation**

Before applying power, comply with the installation section. Additionally the following must be observed.

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

Make sure that only fuses with the required rated current and of the specified type (normal blow, time delay, and so on) are used for replacement. The use of repaired fuses and the short-circuiting of fuse holders must be avoided.

Some adjustments described in the manual, are made with power supplied to the instrument, and protective covers removed. Energy available at many points may, if contacted, result in personal injury.

Any adjustment, maintenance, and repair of the opened instrument under voltage should be avoided whenever possible. When inevitable, this has to be carried out by a skilled person who is aware of the hazard involved. Do not attempt internal service or adjustment unless another person, capable of rendering first aid and resuscitation, is present. Do not replace components with power cable connected.

Do not operate the instrument in the presence of flammable gases or fumes. Operation of any electrical instrument in such an environment constitutes a definite safety hazard.

Do not install substitute parts or make any unauthorized modification to the instrument.

Capacitors inside the instrument may still be charged, even though the instrument has been disconnected from its source of supply. Dangerous voltages, capable of causing serious personal injury, are present in this instrument. Use extreme caution when handling, testing and adjusting.

When working with solvents, observe appropriate safety procedures (for example, goggles, safety gloves and protective clothing) as described in the material handling and safety data sheet by the solvent vendor, especially when toxic or hazardous solvents are used.

## The Waste Electrical and Electronic Equipment Directive

#### Abstract

The Waste Electrical and Electronic Equipment (WEEE) Directive (2002/96/EC), adopted by EU Commission on 13 February 2003, is introducing producer responsibility on all electric and electronic appliances starting with 13 August 2005.

### NOTE

This product complies with the WEEE Directive (2002/96/EC) marking requirements. The affixed label indicates that you must not discard this electrical/electronic product in domestic household waste.

**Product Category:** 

With reference to the equipment types in the WEEE Directive Annex I, this product is classed as a Monitoring and Control Instrumentation product.



### NOTE

Do not dispose off in domestic household waste

To return unwanted products, contact your local Agilent office, or see www.agilent.com for more information.

# 11 Appendix Radio Interference

## **Radio Interference**

Never use cables other than the ones supplied by Agilent Technologies to ensure proper functionality and compliance with safety or EMC regulations.

#### **Test and Measurement**

If test and measurement equipment is operated with equipment unscreened cables and/or used for measurements on open set-ups, the user has to assure that under operating conditions the radio interference limits are still met within the premises.

# **Agilent Technologies on Internet**

For the latest information on products and services visit our worldwide web site on the Internet at:

http://www.agilent.com

# Index

A	D	•
A Agilent on internet 123 ambient non-operating temperature 22 ambient operating temperature 22  B baseline noise 80 baseline spikes 81  C C ChemStation setup 47 cleaning evaporator tube 107 column consideration 68	data output rate 61 defect on arrival 28 delivery checklist 29 detector operation 33 detector sensitivity 12 detector front view 13 overview 13 rear view 14 diffuser drying 105 dimensions 22 display not on 85 display screen 52 drying	internal temperature exceeded lower limit 89 internal temperature exceeded upper limit 89 invalid evaporator temperature 94 invalid nebulizer temperature 94 laser current out of range 95 laser interlock open or failed 96 laser temperature out of range 95 leak detected 91 leak sensor failed 97 LED light source error 93 nebulizer temperature limit exceeded 92 on-board vapor sensor failed 90 rear vapor sensor failed 90 vapor detected 90 error
consideration 68  communication to laser failed 96  communication  LAN 51  serial 51  connecting  detector to system 30  considerations 67  controlling during injection 63  controlling  ChemStation 66  EZChrom 66  cooled evaporator current out of range or communication failed 95  cooled evaporator temperature problem 86	electronic waste 121 electrostatic discharge (ESD) 102 error conditions 56 error messages   communication to laser failed 96   cooled evaporator current out of range or communication failed 95   evaporator gas flow rate limit exceeded 93   evaporator temperature limit exceeded 93   fan failed on cooled evaporator 94   fan failed 91   fan stopped 92	error clearing 56 evaporator gas flow rate limit exceeded 93 evaporator temperature limit exceeded 93 evaporator temperature zero 85 evaporator temperature 12, 58 evaporator tube cleaning 107  F fan failed on cooled evaporator 94 fan failed 91 fan stopped 92 firmware replacing 109 flat baseline 82, 84

frequency range 22	laser interlock open or failed 96	nebulization 9
	laser safety 19	operational parameters 12
G	laser temperature out of range 95	operation
gain 59	leak detected 91	real time 63
gas flow 59	leak sensor failed 97	overview of detector 13
	LED light source error 93	_
H	LED 60	P
humidity 22	line frequency 22	packaging
	line voltage 22	damaged 28
T.	location	parts
inspecting cables 104	detector 73	damaged 29
inspecting cables 104 installation	low sensitivity 81	identifying 116
site requirements 17		missing 29
instrument controls 52	M	peak tailing 83
instrument errors 56	MAC address 36	physical specifications 22
instrument fails to zero 83	main menu bar 53	PMT 59
intensity	materials	power consideration 19
light source 60	identifying 116	power consumption 22
interactive menu bar 53	method 57	power cords 20
internal temperature exceeded lower	loading 57	power mode 61
limit 89		power switch 30
internal temperature exceeded upper	N	pre-installation
limit 89	nebuliser	requirements 18
internet 123	cleaning 106	preparation
introduction	nebulization 9	sample 68
ELSD 8	nebulizer temperature limit exceeded 92	priming 75
invalid evaporator temperature 94	no power 84	principle of operation 9
invalid nebulizer temperature 94	no response 84	pump 74
	non-operating altitude 22	R
K	non-operating temperature 22	
keypad 52		real-time control
	0	starting 64
L	on-board vapor sensor failed 90	stopping 64
LAN interface 36	operating Altitude 22	real-time program creating 64
LAN	operating temperature 22	
TCP/IP parameter configuration 37	operation principle	rear vapor sensor failed 90 replacing firmware 109
large baseline offset 83	detection 10	requirements
laser current out of range 95	evaporation 9	pre-installation 18
		p.o motanation 10

### Index

response time 60	U
S	unpacking 28
safety class I 119	V
safety	
general information 119	vapor detected 90
standards 22	vapor sensor error 86
symbols 118	voltage range 22
site requirements 17	10/
power cords 20	W
smoothing 60	waste
solvent	electrical and electronic
recommendation 68, 76	equipment 121
specification conditions 24	WEEE directive 121
specification	weight 22
physical 22	
specifications 17	
performance 23	
spiky peak tops 82	
status mode 55	
storage 108	
sub-menu screen 53	
T	
TCP/IP parameter configuration 37	
using front panel 38	
using web browser 39	
with telnet 41	
TCP/IP settings 62	
local configuration 44	
temperature error 85	
temperature methods	
transfer 70	
temperature 58	
troubleshooting	
error messages 88	
HPLC system 79	

### www.agilent.com

## In This Book

This manual contains information on the Agilent 1260 Infinity Evaporative Light Scattering Detector (G4260B) and the Agilent 1290 Infinity Evaporative Light Scattering Detector (G4261B).

The manual describes the following:

- Introduction
- · Site Requirements and Specifications
- Installation
- LAN Configuration
- Using
- · Optimizing
- · Troubleshooting and Diagnostics
- Error Information
- Maintenance
- Parts
- Safety

© Agilent Technologies 2012, 2013

Printed in Germany 08/2013



G4260-90001 Rev. C

