SEDEX LT-ELSD LC™

Low Temperature Evaporation Light-scattering Detector

Operator's Manual

SEDERE SAS
PARC VOLTA-BP 27
9, Rue Parmentier
94141 Alfortville Cedex
FRANCE
Phone +33 1 45 18 05 18
Fax + 33 1 45 18 05 25

Revision 1.1 (April 2014) **Part number : 55900**

SEDEX LT-ELSD LC™

Operator's Manual

Revision 1.1

April 2014

This product is covered by the following:

Patent Application Number: 97 16240 (date Dec. 22, 1997)
Patent Application Number: FR 98 15518 (date Dec. 9, 1998)
Patent Application Number: US 6, 148,661 (date Nov. 21, 2000)
Patent Application Number: FR 01 09 202 (date Jul. 11, 2001)
Patent Application Number: EP 02 290 580.6 (date Mar. 8, 2002)
Patent Application Number: FR 03 01652 (date Feb. 12, 2003)
Patent Application Number: US 2003/0086092 A1 (date May 8, 2003)
Patent Application Number: FR 03 13706 (date Nov. 24, 2003)
Patent Application Number: EP 1 447 663 A1 (date Aug. 18, 2004)
Patent Application Number: US 6, 936, 174 B2 (date Aug. 30, 2005)
Patent Application Number: FR 06 50372 (date Feb. 2, 2006)
Patent Application Number: FR 07 52752 (date Jan. 19, 2007)
Patent Application Number: FR 2007/050669 (date Jan. 22, 2007)
Patent Application Number: FR 13 56476 (date Jui. 03,2013)

All information in this manual is subject to change without notice and does not represent a commitment on the part of SEDERE SAS, please consult SEDERE SAS if you suspect any error or omission.

SEDERE SAS believes that the information in its user manuals is accurate at the date of publication.

Printed in France on recycled paper

Warnings and Safety Precautions

The following general safety precautions must be observed during all phases of operation, service, and repair of this instrument.

Before switching on the instrument, check that all protective earth terminals and power cords must be connected to earth. Any interruption of the protective earth grounding will cause a potential shock hazard that could result in serious personal injury.

Read the Operator's Manual (this manual) carefully and thoroughly before you use the detector, comply with the operating and safety precautions and keep this manual for future reference. Only personnel, who are trained in the detector's use, should use the detector.

Maintain a well-ventilated laboratory. If the mobile phase contains a volatile organic solvent, ensure that the laboratory is well ventilated so that a build-up of vaporized solvent cannot occur.

1) Be sure not to obstruct the ventilation holes.

2) Ensure that no liquid leak occurs.

3) Verify that no gas tube damage or inappropriate installation could allow a gas leak. Make sure that all flow connections are properly tight.

4) The safety of any system integrating the instrument concerns the responsibility of the assembler of the system.

5) Avoid open flames and sparks. Do not use an open flame and do not use any equipment that can cause sparks in the same room as the instrument.

6) Make sure the power connector of the instrument can be easily reached and unplugged and provide sufficient space behind the power socket of the instrument to unplug the power cord.

7) The detector must be plugged into a grounded power line. Make certain that all parts of the instrument are properly connected to a common ground.

8) If the mobile phase includes an organic solvent, use an inert gas (i.e. nitrogen) to nebulize the mobile phase.

9) The exhaust from the detector should be vented into a fume hood or similar system. Make certain that the output gas does not escape into the laboratory. Take into consideration any solvent filter that could be required by your local environmental laws.

10) When working with the instrument please observe appropriate safety procedures site and Personal Protection Equipment (PPE) (e.g. googles, safety gloves and protective clothing) due to solvents manipulation.

11) The gas pressure should not exceed 4.5bar (67 psi). Make certain that the gas flow is maintained while the mobile phase flows through the detector. If the gas flow is interrupted for extended periods of time, organic solvents could possibly damage the pressure sensor and/or the photodetector.

12) Do not use corrosive materials that could damage the inner metal surfaces (stainless steel) of the detector.

13) Do not use any liquid or gas that support combustion under temperatures reached by the detector.

14) Access inside the instrument is restricted to a suitably skilled technician.

15) Do not dismount the optical head or electronic boards while the instrument is powered up. This can destroy the detector.

16) The siphon overflow tube must contain liquid at all times.

17) Do not disassemble the nebulizer or touch any component inside the nebulization chamber. This can lead to the deposition of contaminants that could affect the signal.

18) Do not adjust any component inside the detector unless specifically authorized to do so by your dealer.

19) If the instrument is used in a manner not specified by the manufacturer, the protection ensured by the instrument can be ineffective.

20) The user is responsible for decontamination if hazardous material is spilled on or inside the instrument.

21) The user is responsible for detector end of life recycling. You must not discard this electrical/electronic product in domestic household waste. This product is classed as a *Monitoring and Control instrumentation* product. Detector internal parts present no danger for recycling. Make certain than detector has been cleaned to ensure no solvent or solute can remain in detector drift tube.

22) The warning symbols on the instrument indicate the following:

Risk of burn

Electric shock risk

Warning: The information in a warning statement relates to a condition or action that could lead to personal injury.

All information in this manual is subject to change without notice and does not represent a commitment on the part of SEDERE. Please consult SEDERE if you suspect any error or omission.

SEDERE believes that the information in its user manuals is accurate as the date of publication.

Content of this Manual

This manual is designed to describe the installation, operation, maintenance and troubleshooting of the SEDEX LC Low Temperature Evaporative Light-Scattering Detector. It includes:

- *Chapter 1* - **Introduction**, describes the basics of Evaporative Light-Scattering detection.
- *Chapter 2* - **Installation of the Detector**, describes suitable laboratory conditions for the detector.
- *Chapter 3* - **Start-up Procedure / User Interface**, describes the role of the parameters of the detector, the user interface options and menus. In addition, this chapter discusses a number of activities to prepare the unit for routine data collection.
- *Chapter 4* – **Initial Test Procedure** describes protocols that can be used to ensure that the instrument is working in the proper way.
- *Chapter 5* – **Operating the detector**, describes the operations that should be performed on a routine basis when the user wants to operate the detector.
- *Chapter 6* - **Maintenance and Troubleshooting**, describes a series of activities that should be performed on a periodic basis to ensure maximum performance. In addition, this chapter includes a protocol that can be used to determine the cause of problems that are observed with the instrument.
- Three appendices are provided which include product specifications, a list of spare parts and SOP (IQ/OQ/PQ).

For Further Information

For additional information about Evaporative Light-Scattering Detectors, Applications, Bibliography, Sales or Maintenance, Questions or Suggestions, don't hesitate to contact your local distributor.

Please, visit our website for additional information or assistance:

www.sedere.com

Table of Contents

1 Introduction

Thank you very much for choosing SEDEX LC™, which Low Temperature Evaporative Light-scattering Detector (LT-ELSD™) has been designed as a detector for liquid chromatography and parented techniques.

As one of the originators of this detection mode, SEDERE remains exclusively focused on this technology as a core competency and provides a complete and versatile product line dedicated to Low Temperature Evaporative Light-Scattering Detector (LT-ELSD™).

As the industry leader, SEDERE leverages decades of experience and customer knowledge to continually raise the bar for High Sensitivity, High Flexibility and High Fidelity detector performance for chromatography laboratories.

1.1 The Evaporative Light-Scattering Detector

The SEDEX LC™ ELSD is a Low Temperature Evaporative Light-Scattering Detector designed to detect compounds in the eluent from Liquid Chromatography System. It is capable of monitoring eluent flow rates from 200µL/min to 2mL/min. Evaporative Light-Scattering Detection is a nearly universal technique which can detect any analyte which is less volatile than the mobile phase. Unlike other types of detection mode such as UV Detection, it is not dependent on the absorption of radiation and is not affected by the absorption characteristics of the solvent. Thus, solvents which absorb UV radiation can be used. As the solvent is completely evaporated, a gradient can be performed to optimize the separation.

The detector is controlled either via the device front panel or a computer using chromatography software and the dedicated SEDEX Driver for SEDEX LC™.

The detector includes a nebulization cell, an evaporation tube and a detection chamber. The evaporation tube is heated in order to evaporate the solvent.

1.2 Principle of Operation

There are three discrete steps in the operation of the detector; nebulization of the eluent, evaporation of the solvent and detection of the compound(s) of interest (Figure 1).

Figure 1. Schematic Diagram of an Evaporative Light-Scattering Detector

Nebulization involves the conversion of the eluent into a fine aerosol. This aerosol is directed to an evaporator to vaporize the solvent, then the mist is irradiated by a light source and the scattered light is measured by a photodiode; which is related to the concentration of the compound of interest in the sample.

A cross sectional view of the instrument is presented in Figure 2.

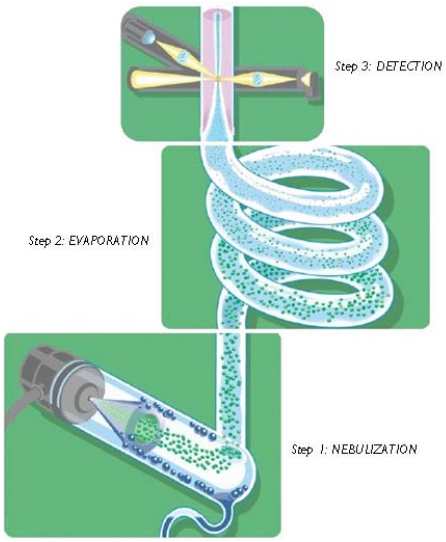

Figure 2. Cross-sectional View of the Detector

1.2.1 Nebulization

This first step transforms the eluent from the liquid chromatography system into fine droplets. The smaller the droplet size, the lower the temperature needed to evaporate the liquid phase. The nebulizer (Figure 3) and the nebulization glassware chamber (Figure 4) are designed to eliminate the biggest droplets of the solvent. The transformation of the eluent into fine droplets is made by a nebulizer which uses
typically nitrogen.

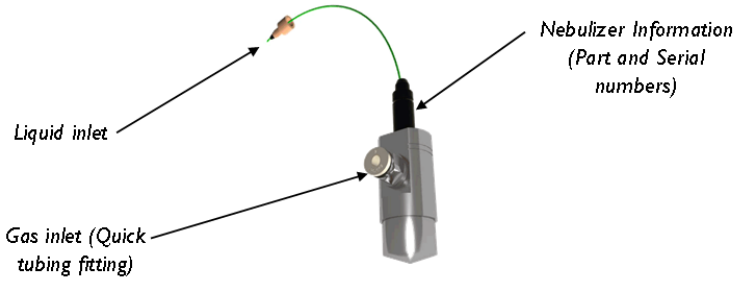

Figure 3. Nebulizer (illustration only)

Replacement nebulizers are available from your local distributor and can be easily installed as described in Section 2.5.4.

At the outlet of the nebulizer, the aerosol travels through a glassware chamber (Figure 4). The glassware chamber is designed to eliminate the biggest droplets of the solvent. Large droplets in the aerosol are drawn to a siphon while the fine mist goes to the evaporation tube.

Figure 4. Glassware Chamber

1.2.2 Evaporation of the Solvent

This second step begins when the droplets are carried by the gas flow into the evaporation tube to evaporate the mobile phase. The detector drift tube has been designed to optimize the efficiency of the required evaporation with the lowest temperatures.

In liquid chromatography, water and organic solvents with low boiling points are typically employed (e.g. CH_3OH, $CHCl_3$, CH_3CN). A typical mobile phase for a reverse phase separation using Evaporative Light-Scattering Detection might be CH_3OH/H_2O (60/40) while a typical mobile phase for normal phase separation might be $C_6H_{14}/CHCl_3$ (60/40).

If acids, bases and salts are used to modify mobile phase to provide the desired separation, they should be able to be readily evaporated, sublimed or decomposed into gases in the evaporation tube. Mobile phase modifiers that are commonly used when an Evaporative Light-Scattering Detector is employed include NH_4OH, $(C_2H_5)_3N$, NH_4 acetate, NH_4 formate, HCOOH, CH_3COOH and CF_3COOH.

The exit of the heated tube leads directly into the detection chamber.

1.2.3 Detection

Analyte particles pass through a detection chamber where they are hit by an incident light beam produced by a Blue Light Emitting Diode (L.E.D.).

The amount of light scattered by the particles is measured using a photosensitive device which converts the scattered light to signal and which is positioned at an angle of 120° with respect to the light beam. Furthermore, a secondary gas inlet is used to concentrate the particles in the center of the detection chamber to further increase sensitivity and to prevent it from contamination.

The intensity of the scattered light is a function of the mass of the scattering particles and generally follows an exponential relationship, which is shown in equation:

$$I = k.m^b$$

where: I is the intensity of light, m is the mass of the scattering particles, and k and b are constants.

A plot of log I versus log m provides a linear response. The values of the constants (k and b) are dependent on a variety of experimental conditions (e.g. the temperature and the nature of the mobile phase).

1.3 SEDERE's patented SAGA

In addition to standard light scattering detection after evaporation, SEDERE provides an advanced feature: the SEDEX Automated Gain Adjustment "SAGA" (Patented).

SAGA is a built-in algorithm that simplifies the use of ELSD and enhances the dynamic range of the detector. Using this feature, the gain value is dynamically set to avoid saturation of the detector:

- when signal value is low, the gain is automatically set to the highest value, providing the user the highest sensitivity of the detector,
- when the signal value is high, the detector monitors instantaneously the signal and switches between lower gain values in order to avoid signal saturation. The gain adjustment is done quantitatively so that instant signal reconstruction is done to provide the user a single gain-like acquisition.

Whereas standard ELSD signal saturates to 1 volt or so, SAGA extends the higher signal range to several tens of volts. Since most of analog to digital (A/D) converter devices cannot handle levels higher than 1volt, a dedicated software driver is needed to control SAGA-enhanced ELSDs and acquire full range chromatograms without limitation.

☞ **When using SAGA feature, the SEDEX LC™ souhld be controlled by a chromatographic software with the SEDEX dedicated driver. When using the SAGA feature with an A/D converter board on the analogue output of the detector, the signal saturates at 1100mV.**

1.4 For Further Information

For additional information about Evaporative Light-Scattering Detectors, Applications, Bibliography, Sales or Maintenance, Questions or Suggestions, don't hesitate to contact SEDERE and its worldwide network of authorized representatives to provide customers assistance.

Our Web site includes e-mail addresses and phone numbers for representatives' locations worldwide.

Please, visit our website for additional information or assistance:

www.sedere.com

1.5 SEDERE Location Information

SEDERE has two locations in France:

- Administration and head office are located in Alfortville (near Paris).
- Production is located in Orléans.

SEDERE SAS

9 Rue Parmentier
Parc Volta - BP 27
F-94141 Alfortville, France
Phone: 33 (0)1 45 18 05 18
Fax: 33 (0)1 45 18 05 25
e-mail: manager@sedere.com

2 Installation of the Detector

2.1 Overview

This chapter describes how the laboratory should be prepared to optimize the performance of the SEDEX LC™ ELSD, refer to Chapter 1 for start-up procedures.

Some accessories included with the detector are described in Figure 5.

Quantity	*Part Number*	*Description*
1	50900	Operator's Manual (this manual)
1	50050	SEDEX LC™ Nebulizer
1	45005	Glassware
1	51350	Starting Kit (gas and electrical connectics cables)

Figure 5. Components shipped with the SEDEX LC™

SEDERE provides a wide range of accessories available (e.g. Gas Regulator with Filter and Manometer [part number 45100]) to support the operation of the detector. A complete listing of all spare parts and accessories is included in Appendix 2.

2.2 Lifting and Carrying the Detector

Once the instrument is unpacked, ensure that no cables or tubing are connected when you carry the instrument. The detector should be lifted by the bottom (e.g. place your hands under the instrument, on left and right side of the device). Two persons are recommended to ensure an easy transport and avoid physical injury.

2.3 Unpacking the Detector

Unpack components carefully, making sure all items in the Figure 5 have been included. The glassware chamber and nebulizer are packed in protective foam inside the nebulization chamber compartment in the front panel. If there is any damage to a carton or its contents or if any component appears to be missing, report to your local distributor immediately.

If there is any evidence that the main unit has been damaged, do not plug the unit into the power line. Contact your local distributor immediately.

It is strongly recommended that the shipping box be retained for future use (to transport the detector or return to repair).

2.4 Laboratory Requirements

2.4.1 Power Requirements

The detector is configured for input voltage from 100 to 240 V with 50/60Hz. The detector requires up to 150Watts. Check that the power lines can provide sufficient current and/or voltage.

The detector must be connected to a properly grounded three prong plug to ensure proper operation of the instrument. If a two prong outlet is used, make certain that the ground wire is used to ground the instrument. It is recommended that all components of the HPLC system are connected to a common ground.

The detector should not be connected to an electrical line which also serves units with a large power drain or which may be subject to power surges. Such units include refrigerators, ovens, centrifuges and fume hoods.

2.4.2 Gas Requirements

A supply of filtered, oil-free clean gas (e.g. N_2 or air) is required to operate the detector. Pure gas is not required as gas is only a carrier vector for the solid particles (e.g. air from an air compressor is acceptable if un-reactive with analysis conditions).

☞ **Gas quality is mandatory for high performance detection with ELSD: The gas should be free from particles (dust) and from oil. The gas purity has negligible impact of the ELSD performance.**

Fire and explosion hazard.

Do not use gas that support combustion with combustible solvents. Do not use air as a carrier gas when the mobile phase contains flammable components.

The gas supply should include a pressure gauge. A manometer with a (0.01µm) filter [part number 45100] is available as an option. Replacement filter cartridges are available as part number 45007.

2.4.3 Exhaust Venting and Drain Requirements

The carrier gas containing volatilized mobile phase and sample components exits the detector through the black exhaust tube located on the detector rear panel.

The black exhaust tube from the detector can be cut and should be directed into a fume hood or exhaust vent. If a vacuum is used, it must be moderate so as to avoid turbulence in the glass cell siphon.

The vacuum must be moderate to avoid turbulence in the glass cell siphon or liquid spilled into the evaporation tube.

Avoid loops or bends in the black exhaust tubing which could create condensation traps resulting in bad measurement results.

If gas from the fume hood enters the detector (i.e. a negative pressure exists between the detector and the fume hood), it is possible that foreign material from the fume hood could contaminate the detector.

☞ **Ensure that the all shipping protections are removed from the exhaust tube or other parts before installing the unit.**

The drain tubing (connected to the glassware) must be directed to an appropriate container regarding to the solvent nature. The user is responsible for decontamination or recycling of any residue, regarding to the local authorities environmental requirements.

Please check with your local regulatory authorities for health and safety requirements.

2.4.4 Environmental Conditions

This instrument has been designed for the following conditions:

- Use inside buildings
- Altitude below 2000 meters
- Ambient temperature from 5°C to 40°C
- Maximum humidity of 80% for temperatures under 31°C, with linear decrease down to 50% at 40°C
- Maximum variations for main power voltage: ±10% from nominal voltage.
- Transitory overvoltage of class II
- Pollution degree: 2

2.5 Installation

2.5.1 Detector rear panel connections

On the top of the detector rear panel is located the fan, which provides cooling airflow through the detector. The black exhaust tube is on the left of the rear panel (not shown in Figure 6) where gas, mobile phase vapor and solute particles are go out from the detector. The exhaust tube installation is described in Section 2.5.3).

On the back of the detector rear panel (Figure 6) is located a supply panel with the gas inlet connection (Section 2.5.2), the power module (with main power switch and lines fuses) and the electrical communications connections (Section 2.4.1).

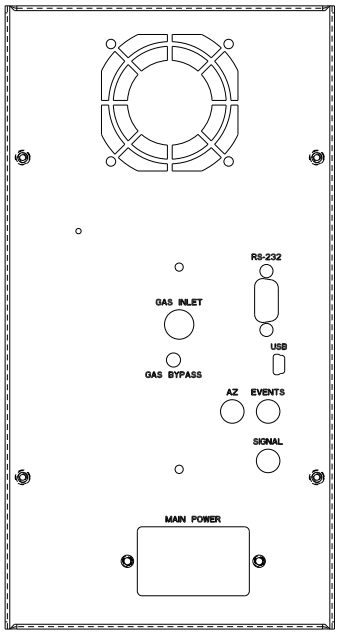

Figure 6. Rear Panel of the detector

2.5.2 Gas supply connection

The unit is connected to the gas supply via the 6.0mm plastic tubing supplied with the detector on the gas quick-fitting inlet on the detector rear panel which requires no additional fitting.

The gas supply must be stable and regulated by an external manometer. The gas must be oil-free, dry and filtered by a 0.01µm filter. The typical gas pressure to operate the detector is 3.5bar.

Fire and explosion hazard.

Do not use gas that support combustion with combustible solvents. Do not use air as a carrier gas when the mobile phase contains flammable components.

☞ **Make certain that the pressure of gas supplied to the detector is less than 4.5bar (67psi). If the pressure increases above 4.5bar, the pressure sensor may be damaged. This damage is not covered by the warranty.**

The tubing should be cut and firmly inserted into the fitting as shown in Figure 7, after removing shipping protections from the detector gas inlet.

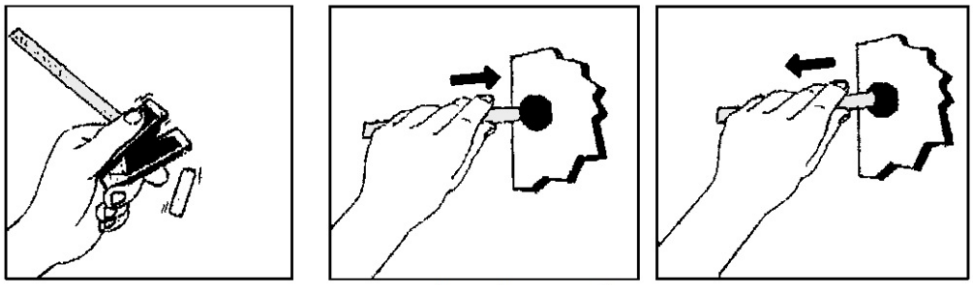

Figure 7. Inserting the Gas inlet tube

Two pieces of tubing are provided. If you are using the instrument with an external filter, connect the gas source to the filter and then connect the filter to the back of the unit.

Make certain that no tube damage or inappropriate installation could allow a gas leak in laboratory.

To remove the gas inlet tube (if necessary); refer to Figure 8.

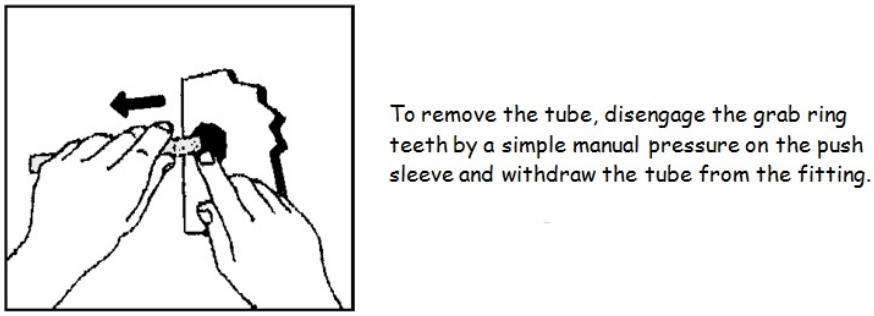

Figure 8. Removal of the Gas inlet tube

2.5.3 Vent the exhaust line to a fume hood

The black exhaust line on the back of the unit containing the volatilized mobile phase and sample particles and should be vented to a fume hood. Make certain that the fume hood withdraws gas from the detector (i.e. there should be a positive pressure between the detector and the fume hood). Verify that no tube damage or inappropriate installation could allow a gas leak in laboratory.

The vacuum must be moderate to avoid turbulence in the glass cell siphon or liquid spilled into the evaporation tube.

Avoid loops or bends in the black exhaust tubing which could create condensation traps resulting in bad measurement results.

If gas from the fume hood enters the detector (i.e. a negative pressure exists between the detector and the fume hood), it is possible that foreign material from the fume hood could contaminate the detector.

Install the vent tube so that it cannot become blocked or bent, or restrict the gas flow from the detector to the fume hood in any way. Avoid long tube installations in upward direction creating condensation dropping back into the detector.

If an extension tube is required (i.e. the supplied tube is not long enough), a suitable length of ¾"ID tubing can be fitted over the exhaust tubing.

2.5.4 Installing the nebulizer/glass chamber assembly

The installed nebulizer/glassware chamber assembly is shown in Figure 9.

To install the assembly:

a) Remove shipping protections from all detector openings and from the nebulization cell (these coatings are used to prevent dust particles from entering the detector during shipment).

Figure 9. Glassware & Nebulizer setting - for reference

b) Position the glassware chamber as shown in Figure 9 and tighten the black nut at the bottom. The glassware chamber should be pushed as far as possible on the back wall.

c) Use the large black nut to position the nebulizer on the glass chamber.

d) Screw the nebulizer inlet fitting to the dedicated fitting on the system.

e) Fill the siphon overflow on the nebulizer/glass chamber assembly with the mobile phase that will be used for the separation. If you are using a very volatile solvent (e.g. hexane or CH_2Cl_2), then use water to fill the overflow. The liquid should fill the bent part of the siphon, but should not pool in the bottom of the evaporation tube.

f) Make sure that no liquid leak could affect the detector performance or create laboratory pollution.

2.5.5 Connecting the siphon overflow

Attach the drain tube assembly to the end of the siphon tube using the red tapered hose connector (with the Teflon ring in place) and lead the tube to waste and drain. Locate the tube in such a way that the discarded part of the solvent can flow freely from the siphon and ensure that the end of the tube never dives in the collected liquid. Make sure that the liquid waste container complies with the solvent nature.

Ensure that no siphon liquid leak could affect detector performances or create laboratory pollution.

Install the drain tubing (it can be cut) in a way to the siphon outlet aligns straight to the waste container –without loops or bends-, so that the waste liquid flows smoothly through the drain tubing.

Fix the drain tubing at the inlet of the waste container so that the end of the drain tubing never dives into the liquid in the container.

A drain tube with a bend or immersing the liquid will create pressure fluctuations in the detector and will result in bad measurement results.

If the solvent that you are using is not compatible with the drain tube (e.g. THF), use a piece of Teflon tubing or any material you know compatible with your solvent in its place. When using this type of tubing which is generally more rigid, make sure that it is safely installed so that it will not damage the nebulization cell (Glassware).

Please check your local regulatory authorities for recycling solvents and health and safety requirements.

2.5.6 Connecting the nebulization gas to the nebulizer

Attach the nebulization gas tube coming out from the front panel to the nebulizer gas inlet fitting located on then Nebulizer side. Make sure you are using the correct orientation, where the white one-way valve is at the lower end (near the gas arrival).

2.5.7 Connecting the LT-ELSD to the chromatographic system

LT-ELSD is typically connected at the end of the separating system: after column, or in the case of a multi-detector system, after either spectrophotometric detectors or splits (when used in parallel with other destructive detectors, such as mass spectrometry).

Attach the fitting from the union bulkhead to the outlet of the column.

2.5.8 Connecting back panel cables

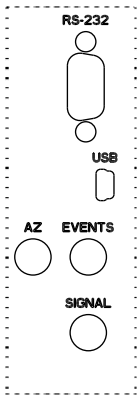

Figure 10. Back panel cables connectors

Depending on your setup, connect on the back panel the cables for

- Signal (analog 0-1 V output from the detector): this cable outputs the signal from the detector, for use with A/D Converter boards or paper integrators
- Events (External Events): double pair cable
  - The first pair (Green / Black) is dedicated to powerdown activation when the contact is closed, the ELSD goes into powerdown mode until the opening of the contact
  - The second pair (Orange / Yellow) is dedicated to output a ready/non ready status from the ELSD to the chromatographic system, this contact is typically used to delay an injection when the detector is away from settings (pressure or temperature)
- AZ (Autozero) : this cable is used to apply an autozero to the ELSD : when the contact is closed for a short time, an autozero is done on the ELSD.
- RS-232 or USB cables have to been connected to the computer of chromatographic controller software, in the case of use of a SEDEX LC™ compatible driver.

Please note that "AZ" , and "Events" cable are contact closures output or input, but are also compatible with TTL signals. When using cables on contact closure compatible devices, there is no polarity to respect, when using the TTL mode, please refer Figure 11 to get the color correspondence of the two wires of each function. Contact a SEDERE representative if you need assistance in setting up the cables, in addition, refer your HPLC system manual to get information on the contact closure / TTL options and polarity provided by your HPLC manufacturer.

Cable connector color	*Back Panel Labelling*	*Function*	*Wire color*	*Polarity (TTL connection)*
Green	*AZ*	Autozero	Red	+
			Black	-
Purple	*Events*	Powerdown	Green	+
			Black	-
		Ready / Non Ready	Orange	+
			Yellow	-
Black	*Signal*	Signal analog output	Red	+
			Black	-

Figure 11. AZ, Event and Signal cable wire color code and polarity

2.5.9 Powering up the instrument

Place the ON/OFF switch to the OFF position and plug the instrument into the wall socket. Turn on the unit via the ON/OFF switch. The display will present initialization screens and will then present the signal (which should be 0mV or very close to it), the temperature (which should be the ambient temperature), the pressure (which should be zero or very close to it) and the gain. Avoid leaks at all connections and check for leakages when you switch the pump on.

The liquid level in the siphon must be stable and should be equal at both sides. If the vacuum is too strong, liquid is drawn into the evaporation tube or generate air bubbles from the drain tube and both resulting in bad measurement results.

Refer to Chapter 1 to prepare the unit for routine operation.

3 Start-up Procedure / User Interface

This chapter describes:

- the role of the controls and the digital display on the control panel,
- the start-up test procedure,
- how to prepare the instrument for operation.

3.1 The Control Panel

The *Control Panel* (Figure 12) includes a digital display and a keypad that is used to enter data. Both the role of each button and user interface menus is described in the following sections.

Figure 12. The Control Panel

3.2 The Digital Display

The digital display gives information about the present status of the detector and is used to set a variety of parameters. When the detector is powered up, the display will present a greetings message (Figure 13) that includes the firmware version number for a few seconds.

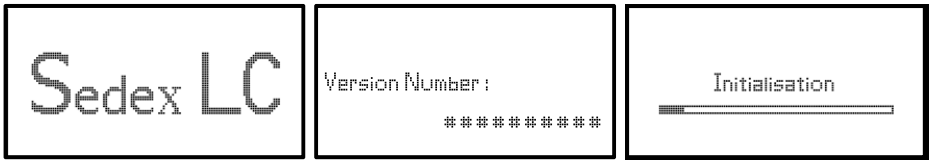

Figure 13. Initialization screens

After the detector has completed the initialization procedures, the *Status* screen (Figure 14) will be presented: operational conditions on start-up are the last conditions used before the power off.

The user interface is provided via a series of screens that are described in Section 3.4. Some screens give information about the instrument status and cannot be edited by the user (e.g. the *Status* screen), while other screens (for example the *Configuration* screen, Figure 17) are used to enter the desired parameters.

3.3 Keypad

The keys on the control panel are used to provide the following functions:

activates or deactivates powerdown functions (blinks when power down is activated),

is used to increase the present value of a user settable parameter (e.g. the temperature) by 1 unit. If you keep the key pressed, the rate of change of the parameter will increase,

is used to decrease the present value of a user settable parameter (e.g. the temperature) by 1 unit. If you keep the key pressed, the rate of change of the parameter will increase,

validates the value of the parameter that you have edited,

sets the present signal for the detector to 0mV by updating the offset value,

is used to power up the light source in the detector. When the light source is switched on, the nebulization chamber compartment is lit,

navigation pad through the menus.

The keys on the control panel are deactivated to prevent unwanted changes during operation when the detector is controlled by a computer.

Each parameter change must be validated with 

button or the change will not be applied.

When navigating through menus using the navigation pad  , line to edit is under brackets, use up and down key to change the line to edit, left and right keys to select the character to change, and modify the value using 

, 

and 

for validation.

Please refer Figure 16 for a map of menus and functions available from the menu screen (Section 3.4.2).

3.4 The User Interface

3.4.1 The *Status* Screen

The *Status* screen (Figure 14) is the default screen and is presented after initialization of the detector. In addition, it will be automatically presented again if you have accessed another screen and have not made any keystroke within a few seconds.

The *Status* screen is the default screen that summarizes important operational parameters:

- Gain,
- Temperature of the evaporation tube ("T°"),
- Noise filtering ("Filter"),
- Pression ("P°").

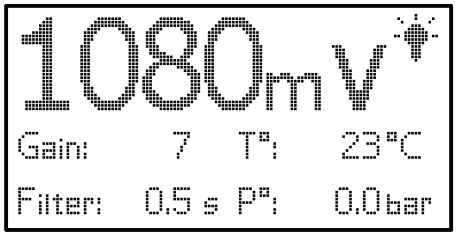

Figure 14. Status Screen

In addition, the status screen gives the user an instant view of signal value. The symbol allows the user to verify that the light source of the detector is on or off (in addition to visual information of lightning of the nebulizer chamber compartment).

☞ **Note that in the case of a remote control from a chromatographic software (using the suitable dedicated driver), the control of the SEDEX LC™ LT-ELSD through the keypad is deactivated. All parameters have to be set in the software: the *Status* screen remains the default menu and provide instant values of the detector.**

The *Status* screen (Figure 14) shows the present conditions of the detector. This screen cannot be edited, but the desired gain, temperature, and noise filtering can be set via the *Setup menu* screen (Figure 25), and both the pressure and temperature units can be selected via the C*onfiguration* screen (Figure 17).

Temperature value blinks if desired temperature is not reached or not stable. Pressure value blinks if gas pressure is lower than 3.0bar or over 4.0bar.

Light source symbol will blink if the life time of the LED is over 5000hours.

If a programmed gas shutoff is programmed, an I*nformation* screen (time remaining before gas shutoff) will be alternatively shown with the *Status* screen.

3.4.2 The *Menu* Screen

The *Menu* screen can be accessed from the *Status* screen at anytime (when the ELSD is not controlled by a software) by pressing OK or any button of navigation pad .

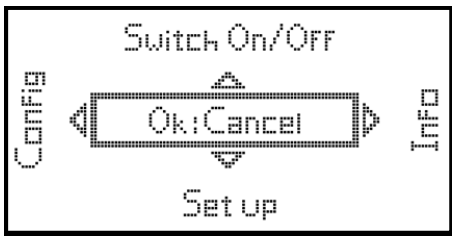

Figure 15. Menu screen

After 10 seconds of inactivity (no key pressed), the *Status* screen will be displayed back. An alternative is to press the OK key to go back instantaneously to the *Status* screen.

The menus accessible from the Menu screen are:

- *Config* screen, is a configuration menu where ELSD interface and shutdown parameters are set:
  - Pressure and temperature unit selection,
  - Power down mode selection and control,
  - Gas shut-off control (automation),
  - Screen contrast control,
  - Factory menu access (limited access for qualified technicians).
- *Switch On/Off* screen, provides the user the control over different features of the detector:
  - Light source control,
  - Gas shut-off valve programing and control.
- *Info* screen, is the general information menu :
  - Serial Number information,
  - Gas Shut off delayed activation information,
  - Firmware version,
  - Boot loader version,
  - Total lifetime counter,
  - Light Source lifetime counter,
  - Signal Offset value,
  - Access to Error menu.
- *Set-up* screen, is a configuration menu where ELSD operational parameters are set:
  - Gain control,
  - Temperature control,
  - Filter control,
  - Signal Offset control.

Each menu is described in the following sections (sections 3.4.3 to 3.4.6). A general view of screens and functions available from the *Menu Screen* and other screens is given in Figure 16.

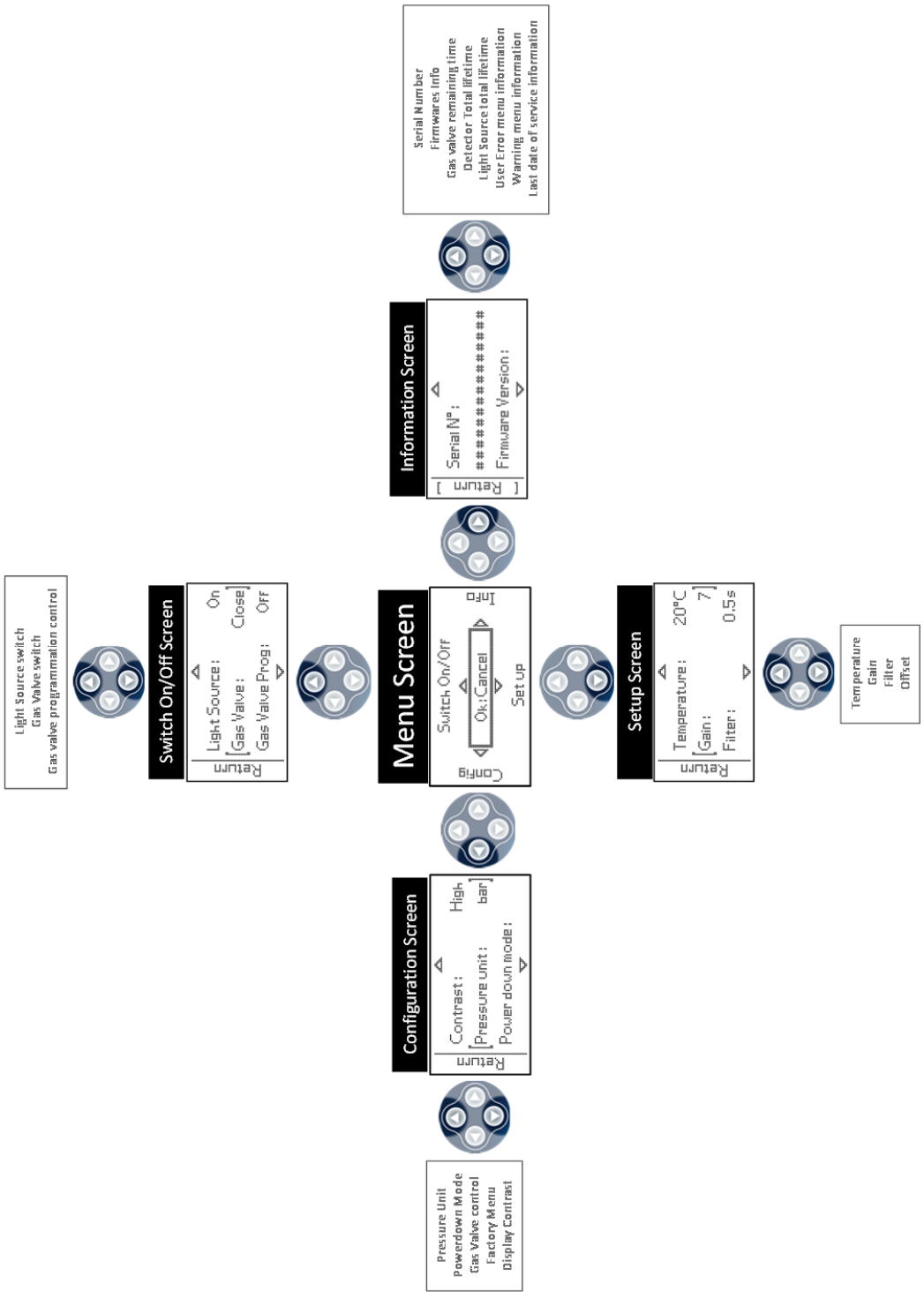

Figure 16. General View of User Interface Screens

3.4.3 *Config* Screen

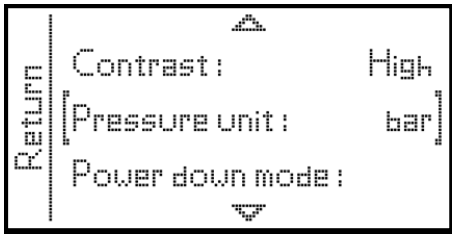

Figure 17. The Configuration Screen

The *Config* Screen is displayed when the left arrow on the navigation pad is pressed through the *Menu* screen. It offers general configuration settings for both the user interface and automation features of SEDEX LC™.

Editable function is indicated between brackets and is centered on the screen: The Config Screen items are displayed as a vertical loop, so that both up and down arrow of the navigation pad can be used to access functions. Use left and right keys to select the character to change, and modify the value using , and .

3.4.3.1 P° Unit

This function allows the user to change the pressure unit used in the SEDEX LC™ user interface. Possible units are bar, psi & kPa.

3.4.3.2 P.D.M: Power down Mode

This function allows the user to select and apply a power down on the SEDEX LC™. Three power down modes are available to the user and are described in Figure 18

Mode	Photodetector	Light source	Heating	Gas Flow
General	Off	Off	Off	Off
Standby	Off	Off	On	Off
Cleaning	Off	Off	On	On

Figure 18. Power down modes

To select the desired *Power down mode*, use the or key to access the desired mode and then press to validate the selection. Power down activation menu appears (Figure 19) on validation and give the user the option to apply instantaneously the selected power down mode. The detector exits from the power down mode when the key is pressed.

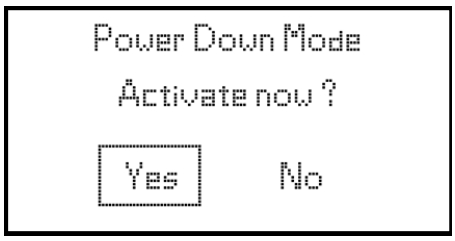

Figure 19. Power down activation menu

☞ **Before applying General or Standby powerdown modes, mobile phase flow must be stopped. Flowing liquid through the detector when the gas is shutoff can seriously damage the detector and will void the warranty.**

☞ **It will take a few minutes to attain operating status from *General* power down mode, as the temperature must stabilize.**

The selected powerdown mode can also be activated:

- with an External Event Cable / power down signal (contact closure or TTL Signal at low level): the detector will stay in the selected power down mode while the contact remains closed. It comes back in normal mode when the contact closure is released,

- by pressing the key (the power down activation menu will be displayed).

☞ **To leave the power down mode, release the External Event Cable contact closure or TTL signal if power down has been activated by the external event (cable) or press any key if power down has been activated from the *Power down* screen.**

☞ **In the case of Cleaning and Standy power down modes, the heating temperature is the active setting in the *Set-up* screen.**

☞ **When the detector is initially powered up or if you change the temperature or after a General powerdown, the temperature may first overshoot the setpoint slightly and then stabilize at the desired point. This initial overshoot is due to the regulation of the instrument and should not be a concern.**

3.4.3.3 Gas Valve Prog

The *Gas Valve Prog* screen is used to open/close the gas valve and to setup a program to close the gas valve after a user selected time period. To use this feature, move the cursor to the time field using the navigation pad , indicate the appropriate time (between 0 and 999 min) using the or key and then press to validate the selection.

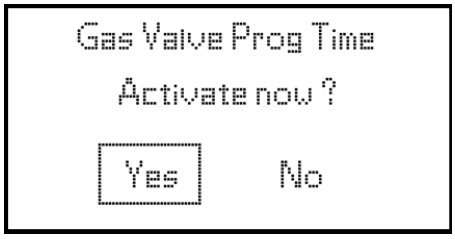

Figure 20. Gas Valve Activation Screen

The *Gas Valve Activation* screen will appear (Figure 20): select "Yes" to shut-off gas after the programmed time.

Note that an information screen providing the time remaining before gas shut off will be displayed alternatively with the *Status* screen.

☞ **Before shutting off the gas, mobile phase flow must be stopped. Flowing liquid through the detector when the gas is shutoff can seriously damage the detector and will void the warranty.**

3.4.3.4 Contrast

This function allows the user to modify the display contrast within three preselected settings.

3.4.3.5 Factory Code Menu

This function is for exclusive use of qualified SEDERE personals or partners, it allows advanced low level diagnostics and after sale service functions.

3.4.4 Switch On / Off Screen

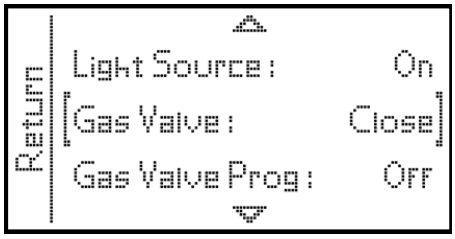

Figure 21. Switch On/Off screen

The *Switch On/Off* Screen is displayed (Figure 22) when the up arrow on the navigation pad  is pressed through the *Menu* screen

The *Switch On/Off* screen provides the user switching possibilities of the SEDEX LC™ detector.

3.4.4.1 Gas Valve Prog

The *Gas Valve Prog* function is used to open/close the gas valve and to setup a program to close the gas valve after a user selected time period. To use this feature, move the cursor to the time field

using the navigation pad , indicate the appropriate time (between 0 and 999 min) using the or key and then press to validate the selection.

The *Gas Valve Activation* screen will appear (Figure 20): select "Yes" to shut-off gas after the programmed time.

☞ **Before shutting off the gas, mobile phase flow must be stopped. Flowing liquid through the detector when the gas is shutoff can seriously damage the detector and will void the warranty.**

3.4.4.2 Gas Valve

Gas Valve function provides an instantaneous activation or deactivation of Gas Shut off ; "Open" deactivate the gas Shut off , "Close" will activate the shut off (pressure on the Status Screen becomes zero).

☞ **Before shutting off the gas, mobile phase flow must be stopped. Flowing liquid through the detector when the gas is shutoff can seriously damage the detector and will void the warranty.**

3.4.4.3 Light Source

The *Light Source* function allows the user to switch on or switch off the light source. This function is also available by using the key .

When switched off, the light source symbol will disappear from the status screen, and the nebulization chamber enlightenment will also been shut off.

3.4.5 Information Screen

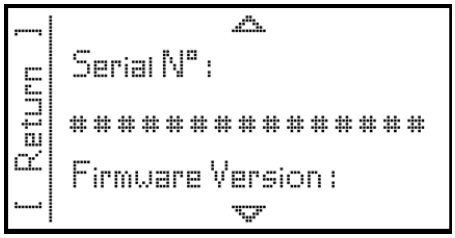

Figure 22. The Information Screen

The *Information* Screen is displayed (Figure 22) when the right arrow on the navigation pad is pressed through the *Menu* screen.

The *Information* Screen offers general monitoring and information features of the SEDEX LC™.

The *Information* lines are displayed as a vertical loop, so that both up and down arrow of the navigation pad can be used to access the information.

3.4.5.1 Serial Number Information

Displays the serial number of your SEDEX LC™ Unit.

3.4.5.2 Firmware version Information

Firmware information for your SEDEX LC™ Units: this firmware is the internal program of your ELSD.

3.4.5.3 Bootloader Version

The Bootloader is an internal software dedicated to update features. Firmware can be updated by qualified technician during service actions.

3.4.5.4 Total Life

General counter of your SEDEX LC™ Unit lifetime. This lifetime cannot be reset.

3.4.5.5 LED Life

Specific light source counter of your SEDEX LC™ Unit lifetime. This lifetime is reset when changing the Light Source module.

When the expected life time is exceeded (5000 hours), a message on system startup will advise the user to change the light source module (Figure 23).

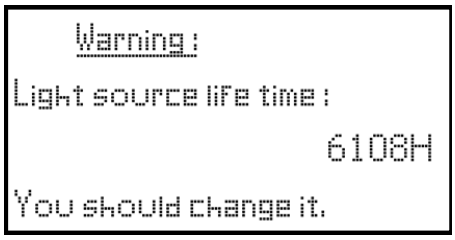

Figure 23. LED Lifetime exceeded warning screen

When running exceeded lifetime light source module, the Light source symbol on the status screen will blink.

The unit remains operational but losses of performances or light source failure have to been expected.

3.4.5.6 Gas Valve Prog

Counter for gas valve shut off. In the case of gas valve programming, the value of this information item is the time left before effective gas shut off. (*Note* Gas shut off can be cancelled anytime by accessing the dedicated function in the Switch On / Off screen, cf. section 3.4.4.1)

3.4.5.7 User Error Menu

This item is a shortcut to access the *Error Code Information* screen. When this item is visible, a hit on the right arrow of the navigation pad will enter this screen (Figure 24).

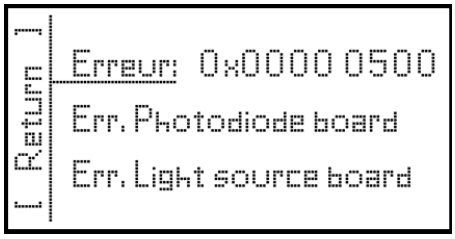

Figure 24. User Error Screen

Information available in this screen is forwarded to the user on system startup. This information provides necessary information for troubleshooting and servicing your detector.

3.4.6 Setup screen

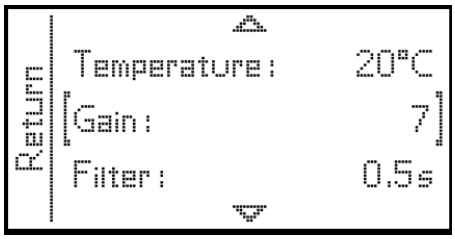

Figure 25. The Setup Menu Screen

The *Setup* screen provides to the user a fast and convenient interface to change routine parameters.

The *Setup* Screen is displayed (Figure 22) when the down arrow on the navigation pad  is pressed through the *Menu* screen

The *Setup* screen provides the user the control over operational parameters of the ELSD.

Editable function is indicated between brackets and is centered on the screen: The Configuration Screen items are displayed as a vertical loop, so that both up and down arrow of the navigation pad  can be used to access functions. Use left and right keys to select the character to change, and modify the value using , and .

3.4.6.1 Gain

The gain of the photodetector can be changed anytime through this menu. The value is from 0 to 7. Between each gain value a factor of 2 is applied is applied.

When selecting the "Dyn" position, the SAGA feature is activated : Gain value is automatically managed by the detector, please refer Section 1.3 for more details about this feature

☞ **When activating the SAGA feature, a SAGA compatible driver should be used to interface the ELSD with chromatographic data software and getting the highest benefit of SAGA feature. The analog output of the detector cannot handle the full response range of SAGA enhanced ELSD and will saturate at 1100 mV**

3.4.6.2 Filter

This function is the noise filtering algorithm setup. The values provided are Off / 0.5 / 1 to 10 seconds (by 1s steps). This noise filtering is applied on the signal and act as a time constant by smothering the baseline noise.

When set to the off position, the signal is a raw signal without filtering.

The user should adapt the filter value to average peak width expected on the chromatogram, note that high speed chromatography require low filter values (0.5 or 1 s), while wide chromatographic peaks can be detected with higher filter settings. The recommended value is 1 second for most of HPLC analysis.

3.4.6.3 Offset

This function can be helpful when the user wishes to have a positive signal value instead of zero, especially with some acquisition systems which have only positive signal acquisition capability. The offset is a shift of the baseline level.

3.4.6.4 Temperature

This function sets the evaporating temperature value (between 20 °C and 100 °C).

4 Initial test procedure

This section presents a protocol that can be used to ensure that the instrument is working in the proper way. A detailed standard operating procedure (I.Q./O.Q./P.Q.) is presented in Appendix 3.

Temperature and pressure units are here expressed in default units of the SEDEX LC™ LT-ELSD: pressure is given as bar, and the temperature as °C. The units can be changed to your preference in the *Configuration* screen (section 3.4.3).

☞ **When the instrument is set-up, the procedures indicated below should be performed to determine the specific characteristics of your unit. We suggest that you save the results in a permanent location, as they can be very useful when you are performing troubleshooting activities.**

☞ **Before starting the tests for a new instrument or after storage, flush the detector with water at a flow of 1mL/min at least 30 minutes with a temperature set to 50°C and gas pressure to 3.5bar.**

The warm up period for the detector and light source is about 30 minutes.

The following activities should be performed:

a) Power up the instrument. When the detector is shipped from factory, the gain is set to 1 and the offset to 0mV. The *Signal* screen should indicate 000 (or a very small signal).

b) Access the *Setup* screen, set the temperature to 50°C and press OK. View the *Status* screen and verify that the temperature is rising to the setpoint on the *Status* screen. Temperature regulation is stable when the Temperature value stops blinking.

☞ **When the detector is initially powered up or if you change the temperature, the temperature may first overshoot the setpoint slightly and then stabilize at the desired point. This initial overshoot is due to the regulation of the instrument and should not be a concern.**

c) Provide gas to the detector and adjust the pressure to 3.5bar (51psi). If the pressure is less than 3.0bar (44psi), the pressure value blinks, indicating that the detector is not ready.

☞ **Make certain that the pressure of gas supplied to the detector is less than 4.5bar (67psi). If the pressure increases above 4.5bar, the pressure sensor may be damaged. This damage is not covered by the warranty.**

☞ **If you have an external gas gauge, make sure that the external reading and the reading on the *Status* screen are in good agreement.**

d) Set the noise filtering to 1s (Refer to Section 3.4.6.2).

e) Press the AZ button. The signal should be close to zero and remain constant.

4.1 Electronic Noise Test

To determine the electronic noise:

a) Do not switch the light source on. Do not switch the HPLC pump on (no solvent flow).

b) Make sure that the siphon is filled and the bulkhead is blocked with Parafilm™ to avoid a Venturi effect.

c) Set gas pressure to 3.5bar and temperature to 50°C. Wait for stable temperature.

d) Set gain 7 and monitor the signal for a period of 5 min. The variation in the signal should be less than +/- 1mV (there may be some spiking of the signal).

e) Record the level and autozero the detector again.

4.2 Background Noise (Stray Light) Test

To determine the background noise:

f) Do not turn on the HPLC pump (no solvent flow) and make sure that the siphon is filled and the bulkhead is blocked with Parafilm™ to avoid a Venturi effect.

g) Set gas pressure to 3.5bar and temperature to 50°C. Wait for a stable temperature.

h) Switch on the light source.

i) Change the Gain to 1.

j) Autozero the detector.

k) Change the Gain to 7.

l) Wait 15 minutes for stabilization and record the signal level. The expected level is typically 50mV to 100mV. The exact value will vary slightly and small deviations should not be a cause for concern.

4.3 Solvent Noise Test

To determine the solvent noise:

a) Make sure the siphon is filled and ensure that the gas is flowing at 3.5bar, the temperature is set to 50°C and stable and the pump is switched off.

b) Switch on the light source, wait 15 minutes and set the gain to 7 and monitor the signal. Do not autozero the detector. The signal may be negative.

c) Bypass the column and connect the detector to the mobile phase delivery system and pump the solvent that you expect to use for your analyses through it at a flow rate of 1mL/min.

d) Monitor the baseline for a few minutes.

- If water is used as the solvent, the signal increase should be less than 10mV. Higher values could be observed if non-HPLC grade water (with a higher non-volatile residue) is used.
- If an organic solvent is used, the signal increase should be less than 20mV.
- For mixed aqueous/organic solvents, the expected signal is approximately linear with respect to the concentration of organic phase in the solvent (e.g. a water/organic solvent 50:50 v/v mixture should provide a signal increase of approximately less than 20mV).

☞ **The purity of the solvent is critical for a low background noise. The sensitivity (signal to noise ratio) is inversely proportional to the solvent noise.**

☞ **In most cases, distilled water and HPLC grade solvents are satisfactory. When you are comparing solvents from different sources, the most critical parameter is the *Residue After Evaporation*; this parameter should be less than 1ppm to maximize the sensitivity of the detector.**

If the instrument fails the Solvent Noise test, it is most likely due to an impurity in the solvent rather than a fault with the instrument. If changing the solvent source does not solve the problem, it may be necessary to decontaminate the instrument as described in Section 6.5 or clean the nebulizer as described in Section 6.4.5.

When filtering the solvent, verify that it does not extract any contaminant from the filter.

The mobile phase should not contain non-volatile solvent modifiers. Volatile solvent modifiers (e.g. CHOOH, CH_3COOH, CF_3COOH, NH_4 Formate, NH_4. Acetate, $(C_2H_5)_3N$)…) can be used, but they may increase the noise level at high gain settings. In addition, the solvent should not contain preservatives, (e.g. Tetrahydrofuran may contain BHT as a stabilizer).

4.4 Column Noise Test

☞ **When strongly retained compounds are slowly eluted from the column, excessive noise will be observed.**

To determine the column noise:

a) Turn off the pump and connect the column.

b) Restart the pump and allow the mobile phase to flow through the system. It is suggested that you flush the column with a strong solvent for a few minutes before attaching it to the detector. The flow rate to be used is dependent on the column ID. and is indicated in the following table.

Column ID (mm)	Flow Rate (µL/min)
4.6	1000
2.1	200
1.0	50
0.8	30
0.32	5

Figure 26. Flowrate versus column internal diameter recommendations

c) Set the gain to 7 and monitor the baseline for a few minutes. A suitable column will provide a baseline that is no more than 5-10mV above the solvent baseline.

Note: **If the mobile phase contains acidic modifiers (e.g. CF_3COOH), disconnect the detector and wash the HPLC system for 12h before starting to analyze unknown samples. This wash should be performed after the column noise test is completed, but need not be performed after each analysis.**

5 Operating the Detector

5.1 Overview

This chapter describes the operations that should be performed on a routine basis when you want to collect chromatographic data using the SEDEX LC™ Low Temperature Evaporative Light-Scattering Detector. In this discussion, we assume that you have demonstrated that the instrument is operating in an acceptable manner (see Chapter 1) and that the general chromatographic conditions for the separation have been determined.

5.2 Start-up sequence

To prepare the detector for operation:

a) Power up the detector by pressing the switch on the rear panel.

b) Open the gas distribution valve and set the pressure to 3.5bar (51psi). The pressure is indicated on the *Status* screen.

c) Ensure that the overflow siphon for the nebulization chamber contains sufficient liquid. If necessary, pump few mL of solvent through the instrument to fill the siphon.

d) Select the desired temperature. The temperature is set on the *Setup* screen, which is accessed by pressing the button two times the down key on the navigation pad  when the *Status* screen is displayed.

e) Start the mobile phase flow through the instrument and allow the overall system to operate for at least 30 minutes to ensure that all components are equilibrated and a stable baseline is obtained.

☞ **The Solvent Noise test (Section 4.3) and the Column Noise test (Section 4.4) should be performed to verify that the detector is functioning in a proper manner.**

☞ **The liquid level in the siphon must be stable and should be equal at both sides.**

5.3 Auto-zeroing the Detector

5.3.1 Manual Auto-zeroing of the Detector

To auto-zero the detector:

a) Set the Gain to the desired value. The gain is set on the *Setup* screen, which is accessed by pressing the button two times the down key on the navigation pad  when the *Status* screen is displayed.

b) Press the  button. The detector will be automatically auto-zeroed at this point.

c) If the signal is to be offset, set the offset at this time. The offset value is edited in the *Setup* Screen which is accessed by pressing the button two times the down key on the navigation pad  when the *Status* screen is displayed.

☞ **The offset must be selected after the detector is auto-zeroed, as the Auto-zero operation sets the signal to 0 by modifying the offset.**

☞ **If you change the gain selection, you may need to make an auto-zero again before taking any measurement.**

5.3.2 External Auto-zeroing of the Detector

If desired, the auto-zero command can be initiated by an external device such as the HPLC system controller. To employ this feature, a cable from the external device is plugged into the AZ socket (Auto-zero) on the rear panel (Section 2.5.1).

To auto-zero the detector, a contact closure signal or a TTL 5V signal (active low) is used. The signal (TTL active low) or shorten of the circuit (contact closure) should be at least 0.1sec long, with a maximum current of 20mA at 5V.

If a TTL signal is used please make sure to use the correct polarity identified on the cable (refer Section 2.5.1).

5.4 Routine Operation of the Detector

In general, operation of an HPLC system with Evaporative Light-Scattering Detection is similar to operation of the system with other detectors.

During operation of the detector, the following points should be considered:

a) Make certain that the exhaust from the detector is led into a fume hood or other device and make sure that there is a continuous flow of gas through the detector (i.e. no constriction). If a vacuum is used, ensure that the vacuum effect will not disturb the detector (Section 2.4.3).

b) Ensure that the siphon is filled with liquid at all times. The overflow from the siphon should be collected in a suitable container.

c) Make sure that all flow connections are properly tight. In case of any leak, switch off the pump immediately and remove the liquid.

d) Never exceed a gas pressure greater than 4.5bar (67psi).

e) Avoid the use of solvent or compounds that could corrode the detector. The mobile phase is in contact with Glass and Teflon tubing and the evaporation tube is made of Stainless Steel.

The exhaust gas should not be allowed to enter the laboratory to avoid any injury or laboratory pollution.

Leakage of hazardous solvents may cause personal injury or laboratory pollution.

5.5 Optimizing Performance

If you transfer a method from another manufacturer's ELS detector, operating conditions will need to be optimized for maximum performance.

5.5.1 Selecting the Optimum Temperature

There are two factors that should be taken into account when selecting the optimum temperature for the detector:

- Increasing temperature will optimize the evaporation of the mobile phase.
- Decreasing temperature will minimize the decomposition of thermally labile compounds and the volatilization of semi-volatile compounds.

A very reasonable start is to set the temperature to 60°C if an aqueous mobile phase is used and 40°C if an organic mobile phase is used (these temperatures are suggested for a flow rate of 1mL/min). At higher flow rates, more elevated temperatures may be required to minimize the noise.

☞ **If the mobile phase used is not easily volatile, such as DMSO or DMF, temperature should be increased to allow correct evaporation process.**

The temperature can be readily adjusted during the method optimization process.

If you suspect that the compound of interest is thermally labile or semi-volatile, a lower temperature could be used to improve the sensitivity by reducing the thermal decomposition or evaporation. For a given flow rate and solvent, there is, however, a point at which the noise in the chromatogram is dramatically increased because not all of the mobile phase is vaporized.

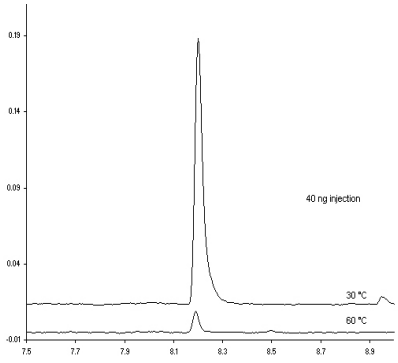

Figure 27. Chromatograms of Caffeine with ELS detection at various evaporation temperatures

As an example, consider the analysis of caffeine with evaporation temperatures of 30°C and 60°C (Figure 27). It is clear that the use of a low temperature provides significantly better sensitivity for volatile and thermally sensitive compounds.

The minimum temperature that can be used is dependent on the flow rate and the nature of the mobile phase.

5.5.2 Optimizing the Mobile Phase

Particulate matter in the mobile phase will increase the background noise.

The purity of the solvent is critical for a low background noise. The sensitivity is inversely proportional to the solvent noise.

In most cases, distilled water and HPLC grade solvents are satisfactory. When you are comparing solvents, the most critical parameter is the *Residue After Evaporation*; this parameter should be less than 1ppm to maximize the sensitivity of the detector.

The purity of the solvent is a critical issue in the noise. When filtering the solvent, verify that it does not extract any contaminant from the filter.

As an example, consider the analysis of a sample in a pure water mobile phase and a polluted water mobile phase (Figure 28). It is clear that the use of an insufficient quality solvent can dramatically decrease your S/N ratio (Sensitivity).

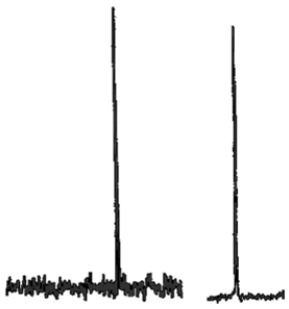

Figure 28. Chromatograms obtained with various mobile phase qualities

The mobile phase should not contain non-volatile solvent modifiers. Volatile solvent modifiers (e.g. CHOOH, CH_3COOH, CF_3COOH, NH_4 Formate, NH_4 Acetate, $(C_2H_5)_3N$)…) can be used, but they may increase the noise level at high gain settings. In addition, the solvent should not contain preservatives, (e.g. Tetrahydrofuran may contain BHT as a stabilizer).

The wetted parts of the detector are made from Teflon, Stainless Steel, and Glass. Make sure that the solvents do not react with these materials.

☞ **Depending on the mobile phase nature and flow rate, the suggested gas pressure 3.5bar (51psi) may have to be adjusted in order to optimize the background noise and so Signal-to-Noise ratio.**

5.5.3 Sample Pretreatment

If the sample contains any particulate matter, it should be filtered through a 0.2µm or 0.45µm filter before injection.

5.5.4 Column Treatment

The chromatographic column typically contains microparticles which are used to separate the compounds of interest. Under certain conditions, the column packing will undergo chemical and/or mechanical breakdown, this may lead to the introduction of particulate matter into the detector, which may lead to an increase in the noise.

The breakdown of the column packing is dependent on a variety of factors including the particle size, type of column packing, the manufacturer of the column and the nature of the mobile phase (high pH may degrade silica based columns).

When you install a new column, we suggest that you pump the mobile phase through it for few minutes before connecting it to the detector. This will flush out the microparticles that remained in the column after its manufacture. After installing a new column, we also suggest that you perform the Column Noise test (Section 4.4) to obtain the baseline signal value corresponding to this column.

5.5.5 Optimizing the Noise Filter

The Digital Filter (see section 3.4.6.2) allows maximizing Signal-to-Noise ratio by filtering the noise. The filter strength should be optimized according to the peak shape, and more specifically to the peak width.

The following table proposes some Filter settings depending on peak width:

Peak Width at 50% height (seconds)	Proposed filter (seconds)
< 1	0.5
2	1
4	2
6	4
8	6
>10	8 and higher

Figure 29. Recommended Filter value as a function of peak width

These suggested values can be optimized depending on your specific chromatography, by decreasing Filter if peaks are poorly resolved (e.g. when Resolution < 1.5), or increasing Filter when optimizing Signal-to-Noise ratio.

Example: Comparison of digital filters with a signal with Peak width at half-height of 2.5 second.

	Filter Off	Filter 1s	Filter 2 s
Signal Height	124 mV	122 mV	110 mV
Noise (ASTM)	1.5 mV	0.7 mV	0.4 mV
Peak Width (50%)	2.5 second	2.5 second	2.8 second
S/N	83	174	220

Figure 30. Sensitivity improvement and effect on peak shape of varous filter settings

Signal-to-Noise ratio is multiplied by 2 when selecting Filter 1s without any peak broadening effect. If for your analysis, Signal-to-Noise ratio is more important than resolution, a Filter of 2s or higher can be set to improve sensitivity.

5.6 Shutdown Sequence

If desired, some or all functions of the instrument can be powered down at the end of an automated series of analyses. These power down features are described in detail in Section 3.4.3.2.

To shut down the instrument:

a) Turn off the mobile phase flow.

b) Allow detector gas only to flow for few minutes (15 minutes is recommended) to drain the evaporation tube and detection chamber.

c) Turn off the gas supply if desired at the source or close the detector gas valve (see section 3.4.3.3 or 3.4.4.2).

d) Power off the detector (if desired).

If you are using a mobile phase which contains salts, acids or bases, pump few mL of water or ethanol through the system before switching off the detector to prevent any deposition of substances and possible corrosion of the instrument.

If the detector will not be used for some time, it is recommended to remove it from the liquid chromatography flow path in order to avoid any clogging of the nebulizer or deposition of substances inside the detector.

Closing gas valve while the pump is still running may result in serious nebulizer damage.

6 Maintenance and Troubleshooting

It is recommended that the detector is maintained and calibrated once a year by a SEDERE-authorized representative. There are no components inside the detector that need to be serviced by the user.

6.1 Overview

This chapter describes:

- The maintenance procedures that should be performed by the operator on a routine basis (Section 6.3).
- Troubleshooting activities that should be useful in determining the cause of erratic or erroneous results (Section 6.4).
- Cleaning and decontamination procedure that should be performed to maintain instrument performances (Section 6.5).
- The replacement of main fuses (Section 6.6)

6.2 Maintenance

The SEDEX LC™ Low-Temperature Evaporative Light-Scattering Detector is designed to require a minimum of maintenance activities. If preventive maintenance activities are followed, the detector should provide high sensitivity measurements without any further intervention by the operator.

The following general recommendations are proposed:

- Maintain the detector in a clean laboratory environment.
- If the instrument is not going to be used for a period of time, flush out any mobile phase that contains acids, bases or salts to prevent the deposition of foreign matter on components or corrosion of the instrument.
- Only use clean gas (particle-free and oil residue-free).

Closing gas valve while the pump is still running may result in serious nebulizer damage.

If ELSD is used as a second detector and is not being used for some time, it is recommended to remove it from the liquid chromatography flow path in order to avoid any clogging of the nebulizer or deposition of substances inside the detector.

6.3 For an efficient Preventive Maintenance:

After each session and before shutting down the HPLC system, the ELSD should be cleaned in order to ensure good performances.

Preventive maintenance consists in cleaning the detector before shutting down after the last analyses:

a) Let the mobile phase or solvent flow to flush particles which could remain in the detector. The solvent used for this cleaning should not contain any additive nor buffer.

b) Eventually increase temperature in order to dissolve possible deposit.

c) Stop the mobile phase flowing but let the gas flow at least 30min to avoid particles deposit.

d) Stop the gas flow if desired at the source or close the detector gas valve (see section 3.4.4.2.

e) Shut down the detector (if desired).

☞ **The time required for each step depends on the application, solvents, type and concentration of the samples and should be determined accordingly.**

☞ **It is not necessary to access inside the instrument in routine operation. If the suggestions provided in this chapter do not remedy the problem, contact your local distributor.**

☞ **The LED used as the light source has a long but finite lifetime and should be replaced periodically only by a skilled technician. When this period has been reached, a message indicating that the maximum usage of the lamp has been exceeded will be presented when the unit is powered, and the symbol on the *Status* Screen will blink when the light source is power on.**

6.4 Troubleshooting

6.4.1 General Troubleshooting Information

The SEDEX LC™ Low Temperature Evaporative Light-Scattering Detector is designed to be incorporated into a Liquid Chromatography system. It is important to note that the detector response reflects the overall performance of the system, and a "problem" that is seen on the detector output may not necessarily be a "detector problem". In almost all cases, there is one and only one cause for a problem. As an example of this point, if the user observes a noisy baseline, it is possible that the problem is due to:

- The pump (e.g. a defective check valve).
- The mobile phase (e.g. improper degassing or high residue after evaporation).
- The column (e.g. elution of strongly retained components, or "bleeding" column).
- The nebulizer (e.g. lack of maintenance).
- The detector (e.g. an electronic problem).

It is very unlikely that two problems occur at the same time. The role of the troubleshooting activities is to determine the cause of the problem. In the following, we assume that the operator has already determined that other components of the system are operating in an appropriate way.

Do not disassemble the nebulizer. Disassembling the rear part of the nebulizer (engraved white shrink seal) will destroy it.

☞ **The control panel and instrument electronics do not contain any replaceable components. If the suggestions provided in this chapter do not remedy the problem, contact your local distributor.**

6.4.2 Initial Troubleshooting Activities

a) Make sure that the instrument and all components of the detector are grounded to a true ground.

b) Check to ensure that the liquid level in the siphon is appropriate, and there is no liquid pooling close to the evaporation tube inlet.

c) Check that the gas pressure is sufficient and stable. The selected pressure for most applications is 3.5bar (51psi) and gas consumption is 3L/min. Pressure above 4.5bar (67psi) can damage the pressure sensor. The gas filter should be clean and in place. Only use gas free of particle and oil residue.

d) Ensure that the flow rate of the pump is constant and check that there are no leaks in the chromatography system.

6.4.3 Perform the Noise Tests

Repeat the tests described in Section 1 and compare the observed data to the results that were obtained when the unit was initially installed. These tests can be very valuable to isolate the problem.

As an example of this point, if the Electronic Noise test (Section 4.1), Background Noise test (Section 4.2) and Solvent Noise test (Section 4.3) provide results that are similar to that obtained when the unit was initially installed, but the Column Noise test (Section 4.4) provides a significantly different value than what was obtained at installation, it is likely that the problem is in the column (e.g. highly retained compounds are being eluted).

6.4.4 Specific Detector Troubleshooting

Do not disassemble the nebulizer. Disassembling the nebulizer will destroy it.

a) The mist from the nebulizer should be homogeneous. If it is not homogeneous, the nebulizer, the needle or the Teflon tube may be partially obstructed. To remove the obstruction, pump a solvent that can dissolve the foreign material. As an alternative, the

nebulizer can be placed in an ultrasonic bath to dissolve the foreign material. Instructions about cleaning of the nebulizer are presented in Section 6.4.5.

b) If the sensitivity of the detector is low, ensure that there are no leaks in the system. Make sure you are using a fresh sample and consider running the test using a backpressure loop instead of a column. Alternatively, the light source may need to be replaced or the nebulizer could be obstructed.

c) If the noise test did not show that the problem could be caused by the application or the system, a decrease in the sensitivity is often caused by the nebulizer (main cause). Clean the nebulizer as described in Section 6.4.5. If the sensitivity does not return to normal, the nebulizer might need to be replaced. Please note that the root cause might also be in different module, i.e. volumes injected by the autosampler might be too low or dead volumes in capillary connections may cause peak broadening.

d) If the detector signal is saturated or if there is a decrease in the dynamic range of the system, it is possible that a residue is passing through the detector cell: this will lead to an intense signal due to a significant amount of light-scattering. This residue may be a result of the elution of strongly retained materials from the column, or may come from the solvent. To determine the cause of the problem, bypass the column and observe the signal intensity:

e) If the signal returns to normal, strongly retained materials are eluting from the column. Flush the column with a strong solvent to elute all material.

f) If the signal does not return to normal, the solvent contains a too high residue material, after evaporation and is not suitable for use with the detector.

g) If the noise of the detector without solvent is high or if ghost peaks occur, it is possible that foreign material is present in the drift tube. In this situation, increase the temperature to 95°C and pump appropriate solvent at the rate of 2mL/min, using a gas pressure of 3.5bar (51psi). The solvent will be determined by the nature of the samples that were previously analyzed with the detector. If you do not know the nature of the sample, ethanol is a good choice. Do not use solvents that can potentially corrode the instrument. Maintain the flow and temperature during 3 hours at least.

6.4.5 Nebulizer Cleaning and Replacement Procedures

With time, the nebulizer can get clogged by sample and mobile phase materials. A dirty or clogged nebulizer can cause increased baseline noise and decreased sensitivity. The following procedure can be used to clean the nebulizer.

The nebulizer is a consumable; the nebulizer lifetime depends directly on the conditions of use and care. The present section gives general directions for nebulizer maintenance: if cleaning procedures are ineffective for initial performances rehearsal, the user should consider a nebulizer replacement.

If the mist of the nebulizer is not homogeneous, the nebulizer, the needle or the Teflon tube may be obstructed. To remove the obstruction, pump a solvent that can dissolve the foreign material. As an alternative, instructions about nebulizer cleaning are presented in this section.

Handle the nebulizer carefully and do not disassemble the rear part of the nebulizer, which is protected by the white thermal seal. Improper handling of the nebulizer will destroy it.

The nebulizer rear part results from a very tricky setting which mustn't be dismounted for any reason. In case it has been removed or unscrewed, the only solution is to proceed to a nebulizer replacement.

In case of the nebulizer doesn't produce a spray and the liquid drawn directly to the siphon even if the pressure display is 3.5bar, make sure you are using the correct black gas tube direction fitting for the nebulizer, where the white one-way valve is at the lower end (near the gas arrival) on the front panel. The installed Nebulizer/Glass Chamber assembly is shown in Figure 9.

6.4.5.1 To remove the nebulizer from the instrument:

a) Switch off the pump and the ELSD detector.

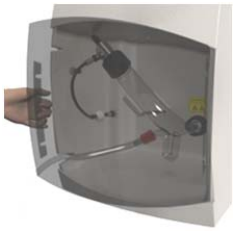

Figure 31. Front window removal

b) Remove the black front panel cover (Figure 31). Pull its left side.

Figure 32. Disconnecting fluids inlets

c) Disconnect the nebulizer liquid inlet from the column and disconnect the gas inlet from the nebulizer by pushing on the white inlet (Figure 32).

Figure 33. Removing the nebulizer

d) Remove the nebulizer from the glass cell by unscrewing the black plastic nut with the right hand whilst maintaining the nebulizer with the left hand (Figure 33). Take care not to pull or twist the nebulizer capillary.

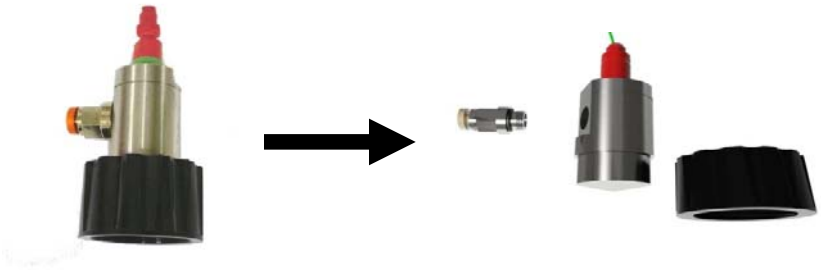

Figure 34. Nebulizer preparation for cleaning

e) Remove the gas inlet quick fitting and the black plastic nut to avoid damaging the seals with the cleaning solvent (Figure 34).

6.4.5.2 To clean the nebulizer:

f) Fill an ultrasonic bath with water. Fill a beaker (50 or 100mL) with approximately 2cm of an appropriate solvent. The solvent is dependent on the nature of the material that is present in the nebulizer. In most cases, ethanol is a satisfactory solvent.

Figure 35. Nebulizer Cleaning (not more than 2 cm of cleaning liquid)

g) Place the nebulizer vertically in the beaker 2cm solvent bath. The nebulizer outlet should be placed at the bottom of the bath and the nebulizer inlet liquid tubing should

be pointing up. Take care to ensure that the rear part of the nebulizer is not in contact with the solvent.

h) Clean the nebulizer for approximately 30 minutes with the solvent, and then replace the solvent with water and clean for an additional 30 minutes.

If the nebulizer cannot be repaired by cleaning by pumping solvent through it or with an ultrasonic bath, it requires a replacement.

6.4.6 To re-install the nebulizer or replace it by a new one

a) After a nebulizer cleaning and for re-installing the nebulizer, re-install the gas inlet quick fitting and the black plastic nut with its seal.

b) Reverse the order of previous steps (nebulizer removing). In case the black gas tubing has been removed, make sure you are using the correct direction, where the white one-way valve is at the lower end (near the gas arrival).

c) Make sure there is no liquid or gas leak at all connections and check for possible leakage that could affect the detector performance or create laboratory pollution when you turn on the pump.

d) Install the black front panel cover, first fix its right side, and then push its left side.

e) Test the nebulizer to ensure that it is working properly.

☞ **If the Nebulizer cleaning procedure does not solve the problem, contact your local distributor for a nebulizer replacement.**

In case the black gas tubing has been removed, make sure you are using the correct direction, where the white one-way valve is at the lower end (near the gas arrival).

Avoid leaks at all connections and check for possible leakage when you turn the pump on again.

6.5 Cleaning and Decontamination

6.5.1 Instrument Cleaning

a) Switch the instrument off.

b) Disconnect all connection cables (power cable, signal cable, auto-zero cable, USB cable..., instrument gas input and nebulizer tubings).

c) Allow the detector to cool down.

d) Clean the outside of the detector with a non-abrasive cloth. If necessary, a liquid such as soapy water or ethanol can be used to remove stains or foreign material.

6.5.2 Instrument Decontamination

Set the evaporation temperature to 95°C and the gas pressure to 3.5bar (51psi).

Pump the appropriate solvent through the system at the rate of 2mL/min. The solvent will be determined by the nature of the samples that were previously analyzed with the detector. If you do not know the nature of the sample, ethanol is a good choice. Do not use solvents that can potentially corrode the instrument. Maintain the flow and temperature during 3 hours at least.

6.6 Fuses replacement

If the digital display does not switch on when the detector is powered up, power the unit off and inspect the main fuses. The fuses are located inside the main power module on the rear panel (Figure 6). A replacement fuse is delivered in the power socket fuse chamber.

Figure 36. Power socket fuse chamber with spare fuse shown (on the left)

a) Power off the instrument and unplug the power cord from the main power lines and from the power module on detector rear panel.

b) Locate the fuse compartment on detector rear panel and withdraw the fuses holder (using for example a screwdriver n°1 or smaller).

c) Remove and discard fuses. Insert new fuse into the holder and the holder into the power module. The fuse holder only fits in one way into the power module.

d) Reconnect the power cord to the power module and to main power lines.

If the fuses are not blown or if the replacement fuse blow up again, power off the detector and unplug the power cord from main power and contact your local distributor.

For safety protection, replace fuses with only the same type of fuses 5x20mm and rated T3.15AL 250V fuses.

To avoid electric shock, power off and unplug the power cord from the main power before examining fuses.

6.7 Light source

The LED used as light source has a long but finite lifetime (5000 hours) and should be replaced periodically. A decreasing light intensity will cause decreasing signal heights over time. When this period has been reached, a message indicating that the maximum usage of the lamp has been exceeded will be presented when the unit is powered, and the symbol on the *Status* Screen will

blink when the light source is power on. For replacement, contact your SEDERE-authorized representative.

Appendix 1. Technical Specifications

SEDEX LC™

Low Temperature Evaporative Light-scattering Detector

Components

Detection	High sensitivity Photodiode
Light source	Selected High Efficiency Blue LED (470nm) with Elapsed-time Counter
Temperature Range	Ambient to 100°C
Nebulizer	LC (200µL/min to 2mL/min)
Sensitivity	< 5 ng sulfanilamide (LOD)

Data

Gain Setting	1 to 7 or Dynamic gain management: SAGA
Filter	Moving Average
Data Rate	up to 30 Hz

Communication

Selection & Display	Liquid Crystal Display and Keypad.
Event	Contact Closure, TTL for Ready, Auto-zero, Power down.
Power-down Methods	Shut-off: Gas, Light source, Heating and/or PMT
Computer Interface	USB, RS-232.

External Requirements

Power	100-240V/50-60Hz
Gas Pressure (Nitrogen or Air)	3.5bar with gas control and patented auxiliary gas flow
Dimensions	530mm (21") D x 250mm (10") W x 330mm (13") H
Weight	15Kg (33lbs).

Appendix 2. Spare Parts List

Part Description	Part Number
Light Source	50020
Nebulizer	50050
Glassware Nebulization Chamber	45005
Gas Regulator with Filter and Manometer	45100
Cartridge 0,01µm for Gas Regulator	45007
Drain Tube Assembly (includes stainless steel fitting and PTFE seal)	45200
Black exhaust tube (2m50)	55016
Black Plastic Nut for Nebulization Chamber (13 mm)	45700-13
Black Plastic Nut for Nebulization Chamber (30 mm)	45700-22
Starting Kit (Gas and electrical connectics cables)	51350

Appendix 3. Standard Operating Procedure and I.Q./O.Q./P.Q.

The Standard Operating Procedure **(S.O.P.)** is provided to perform the Installation Qualification **(I.Q.)**, Operational Qualification **(O.Q.)** and Performance Qualification **(P.Q.)** to confirm that the instrument is functioning in compliance with manufacturer specifications and therefore validate the instrument at your site. The original Declaration of Conformity to manufacturing specifications is shipped with each instrument to certify that the instrument passed the final test.

An *Installation Qualification* checklist is presented in Section 0 and the installation of the unit is described in Chapter 1.

I.Q. should be performed when the instrument is newly installed and each time it is moved to a different location.

The procedure for the *Operational Qualification* is described on the worksheet presented in Section A.3.2.

O.Q. protocols demonstrate that the instrument performs according to its functional and operational specifications.

The procedure for the *Performance Qualification* is described on the worksheet presented in Section 0.

P.Q. protocols demonstrate that the instrument performs according to sensitivity using Sulfanilamide as a test compound.

The *Overall Detector Performance report* is presented in Section A.3.4.

☞ **The present document should be filled with blue ink, except customer authorization for black ink.**

Before starting S.O.P for new instruments or after storage, the following operations must be performed:

- **Set gas pressure to 3.5bars (51psi).**
- **Set the temperature to 50°C and wait for stabilization.**
- **Flush detector with 1mL/min of water at least 30 minutes.**

The warm up period for the detector and light source is about 30 minutes.

SEDERE recommends that maintenance visits and SOP certification should be performed once a year by a SEDERE-authorized representative.

A.3.1 Installation Qualification (I.Q.) Checklist

Model Number: SEDEX LC™ LT-ELSD Instrument Serial Number: _____

Location of the Detector: _____

e) Has the instrument been delivered as ordered (e.g. according to the U.R.S. or purchase order)?

YES ☐ NO ☐

f) Has the instrument been checked and verified as undamaged?

YES ☐ NO ☐

g) Has the required documentation been supplied? Is it of correct issue and appropriately identified by Model Number, Serial Number and Date?

YES ☐ NO ☐

h) Have details of all services and utilities required to operate the instrument been provided?

YES ☐ NO ☐

i) Have methods and instructions for user maintenance been provided along with contact points for service and spare parts?

YES ☐ NO ☐

j) Is the selected environment suitable for the instrument (i.e. is adequate room provided for installation, operation and servicing)? Have appropriate solvent services and utilities (electricity, nitrogen gas, ventilation, solvent waste recovery, etc.) been provided?

YES ☐ NO ☐

k) Has health, safety and environmental information relating to the operation of this instrument been provided?

YES ☐ NO ☐

The manufacturer's procedure for the proper **Installation Qualification** of this instrument was completed by the following certified person:

Name _____

Title/Affiliation _____

Date _____ Signature _____

A.3.2 Operational Qualification (O.Q.) Protocol

This procedure checks the proper operation of the detector with respect to stability of the electronic boards, the energy of the light source and the sensitivity of the photo detection system.

Model Number: SEDEX LC™ LT-ELSD Instrument Serial Number: _____

Location of the Detector:_____

l) Power up the detector

m) Seal the solvent inlet connection (1/16" male fitting) with a plug connection or a piece of Parafilm™ and fill the glass siphon with water.

n) Apply air or nitrogen pressure of 3.5bar (51psi), monitored by a regulator and checked with a pressure gauge.

The display should read 3.5bar +/- 0.1bar (51psi)

PRESSURE VALUE:_____

PASS ☐ FAIL ☐

o) Set the temperature to 50°C and wait until the temperature stabilizes (typically 30 minutes).

The display should read 50°C +/- 1°C.

DISPLAY TEMPERATURE: _____

PASS ☐ FAIL ☐

p) Set the *Gain* to *1*, *Filter* to *1s* as described in sections 0 and 3.4.6.2.

Temperature order to 50°C and allow air or nitrogen gas flow through the instrument at a pressure of 3.5bar (51psi),

Power on the light source,

q) Wait for temperature stabilization.

r) Auto-zero the detector. Raise the Gain to 7, monitor the signal for 5 seconds and enter the value below.

OBSERVED VALUE:_____ mV

The observed stray light readings should be in the range of 50 – 100mV

PASS ☐ FAIL ☐

s) This test is performed in the same conditions as the Stray Light value determination.

Measure the noise over six 1 min segments with the ASTM method.

For some Chromatography Software the minimum method acquisition time must be at least 1.10min for the ASTM noise determination.

Segment	Noise (mV)
1	
2	
3	
4	
5	
6	

Minimum Value: _____ Maximum Value: _____ Mean Value:

The mean ASTM noise value should be less than 1mV.

PASS ☐ FAIL ☐

t) This test is performed in the same conditions as the Stray Light value determination. Collect data for 15 minutes.

Initial Signal Level ____mV Final Signal Level ____mV

15Min Drift ___mV

The baseline drift should be less than 1.0mV.

PASS ☐ FAIL ☐

☞ **If any part of the diagnostics fails, the detector may need more time to equilibrate. In any doubt, please refer to your local distributor for service.**

The manufacturer's procedure for the proper **Operational Qualification** of this instrument was completed by the following certified person:

Name _____

Title/Affiliation _____

Date _____ Signature _____

A.3.3 The Performance Qualification (P.Q) Protocol

Model Number: SEDEX LC™ LT-ELSD Instrument Serial Number: _____

Location of the Detector _____

☞ Note: **Before this procedure is performed, it is necessary to complete the Installation Qualification (Part I) and the Operational Qualification (Part II). The test report presented below or your own appropriate reference test should be used.**

Remove the plug connector (or Parafilm™) from the solvent inlet connector before connecting the capillary to the nebulizer liquid inlet union.

u) Set the conditions as follows and wait for equilibration time (30min):

- Temperature : 50°C
- Gain : 7
- Noise filtering : 1s
- Gas pressure : 3.5bar (51psi) air or nitrogen
- Siphon : Filled with water
- Inlet tube : 1/16" tubing loop, 0.005" I.D. x 200cm between the pump and the autosampler to create backpressure
- Solute : Sulfanilamide
- Flow rate : 1mL/min
- Mobile phase : 100% fresh HPLC water

v) Set gain 7 and then autozero the detector.

w) Make six (6) 20µL injections of a 250ppm (250µg/mL) sulfanilamide standard at gain 7.

x) Report peak areas and heights in the following table, calculate round standard deviation (RSD) on peak areas, and the average peak height.

- The repeatability should be 3.0% RSD maximum.
- The average peak height should be over 150 mV

Segment	Peak Height (mV)	Peak Area
1		
2		
3		
4		
5		
6		
Average		
RSD (%)		

% Round Standard Deviation (RSD): _____ %

PASS ☐ FAIL ☐

% Average peak Area:_____ mV

PASS ☐ FAIL ☐

The manufacturer's procedure for the proper **Performance Qualification** of this instrument was completed by the following certified person:

Name _____

Title/Affiliation _____

Date _____ Signature _____

A.3.4 Overall Detector Performance

After the Installation Qualification (I.Q.), Operational Qualification (O.Q.) and Performance Qualification (P.Q.) procedures have been completed, the **Overall Detector Performance** document should be completed to verify the completion of all tests.

Model Number: SEDEX LC™ LT-ELSD Instrument Serial Number: _____

Location of the Detector _____

Part I - Installation Qualification (I.Q.) Date :

PASS ☐ FAIL ☐

Part II - Operational Qualification (O.Q.) Date :

PASS ☐ FAIL ☐

Part III - Performance Qualification (P.Q.) Date :

PASS ☐ FAIL ☐

The above instrument was certified by the following certified manufacturer's representative:

Name _____

Title/Affiliation _____

Date _____ Signature _____

The above instrument was certified with the presence of the following customer's representative:

Customer name _____

Customer title/Affiliation _____

Date _____ Signature _____

INDEX