SEDEX MODEL 80LT

LOW TEMPERATURE EVAPORATIVE LIGHT SCATTERING DETECTOR

OPERATOR'S MANUAL

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Part Number 81000
Rev. 4.0 / Feb. 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)

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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.

1) 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.
2) Be sure not to obstruct the ventilation holes.
3) Ensure that no liquid leak occurs.
4) Verify that no gas tube damage or inappropriate installation could allow a gas leak. Make sure that all flow connections are properly tight.
5) The safety of any system integrating the instrument concerns the responsibility of the assembler of the system.
6) 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.
7) 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.
8) 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.
9) If the mobile phase includes an organic solvent, use an inert gas (i.e. nitrogen) to nebulize the mobile phase.
10) 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.
11) 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.

12) The gas pressure should not exceed 4.5bar (67psi). 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 photomultiplier

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

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

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

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

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

18) 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.

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

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

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

22) 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.

23) 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.

Table of Contents

1 Introduction

Thank you very much for purchasing a SEDERE LT-ELSD™ Model 80LT detector, which has been designed for analysis in a chromatography system.

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 Low Temperature Evaporative Light-Scattering Detector

The ELSD 80 Low Temperature Evaporative Light-Scattering Detector (Figure 1-1) is designed to detect compounds in the eluent from High Performance Liquid Chromatography (HPLC), preparative HPLC, Flash Chromatography or Counter Current Chromatography (CCC).

It is capable of monitoring eluent flow rates from 100µL/min to 5mL/min. Evaporative Light-Scattering Detector is a nearly universal technique which can detect any non-volatile analyte. 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.

LT-ELSD™ is a nearly-Universal and powerful detector. LT-ELSD is used as a valuable alternative to Refractive Index and UV/Vis detection (for the latter particularly when analytes do not possess any chromophore) and is also considered as relevant complementary equipment to LC/MS.

Figure 1-1: The SEDERE LT-ELSD™ 80 Detector

The detector can be controlled via the keypad and digital display on the front panel. The detector can be also controlled and monitored by an external computer using the RS-232 ports as an option. As an alternative, the analog signal output can be sent to a recorder, an integrator or a data station.

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 1-2).

NEBULIZATION → **EVAPORATION** → **DETECTION**

Figure 1-2: 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 photomultiplier; 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 1-3.

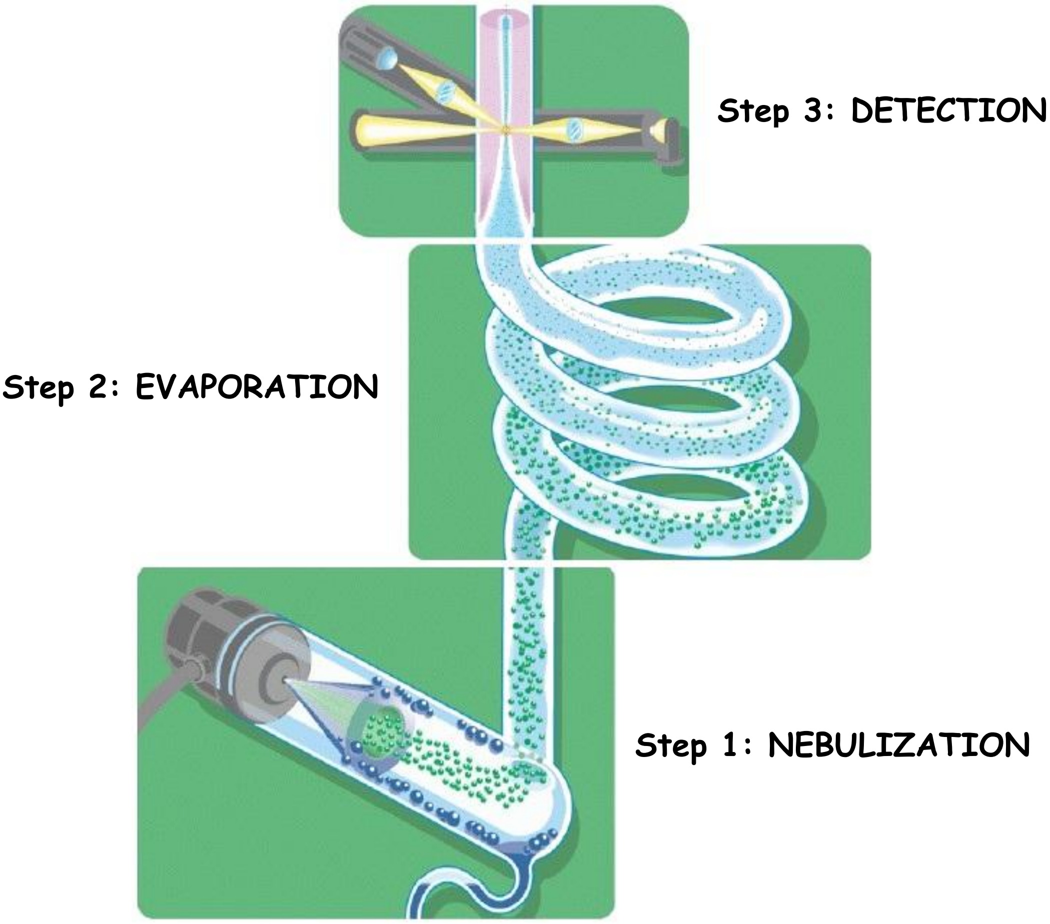


Figure 1-3: Cross-sectional View of the Detector

1.2.1 Nebulization

This first step transforms the eluent from the chromatography system into fine droplets. The smaller the droplet size, the lower the temperature needed to evaporate the liquid phase. With SEDEX detectors, the nebulizer (Figure 1-4) and the nebulization glassware chamber (Figure 1-5) 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.

Three different nebulizers are available to optimize performance of the detector at different Liquid Chromatography flow rates (refer to Table 1-1). The user should select the nebulizer to best match the flow rate that will be used with the separation when the detector is ordered (the optimal range for each nebulizer is indicated in Table 1-1). Nebulizer can be easily identified with the part number labelled on it. Replacement or additional nebulizers are available from your local distributor and can be easily installed as described in Section 5.3.5.

Nebulizer Model	Flow Rate Range	Back Pressure (with water)	Part Number
HPLC Nebulizer	100µL/min - 2.5mL/min	4bar (1mL/min)	80003
Combinatorial Chemistry Nebulizer	1.0mL/min - 4.0mL/min	4bar (1mL/min)	80008
Flash Nebulizer	100µL/min - 5mL/min	4bar (1mL/min)	80005

Table 1-1 Nebulizers for the SEDEX Model 80LT Low Temperature Evaporative Light-Scattering Detector

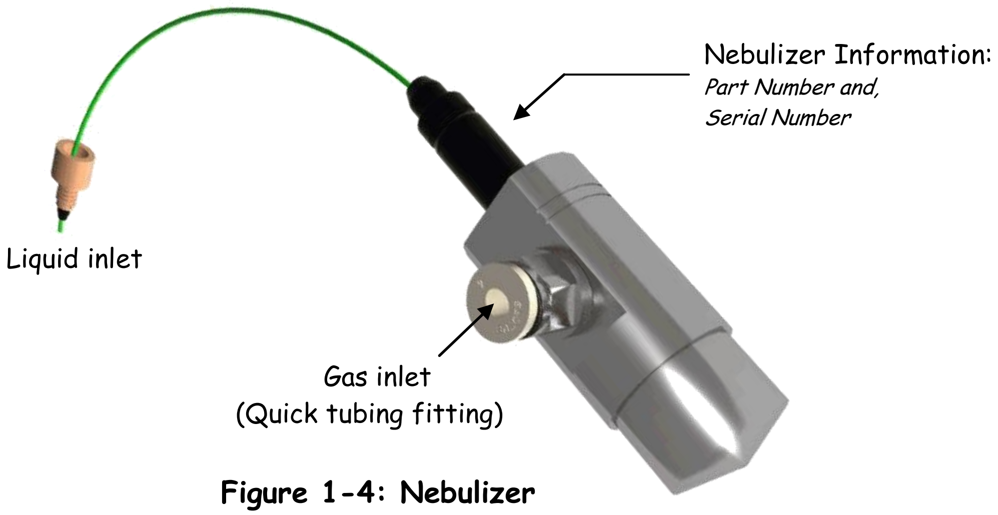


Figure 1-4: Nebulizer

At the outlet of the nebulizer, the aerosol travels through a glassware chamber (Figure 1-5). With SEDEX detectors, glassware chambers are 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 1-5: Glassware Chambers

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. SEDEX 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.

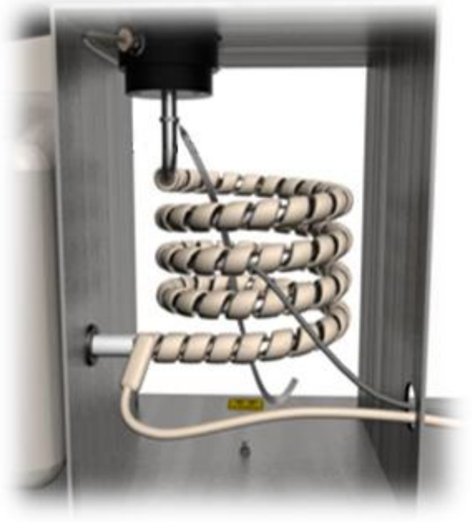


Figure 1-6: Evaporative tube

1.2.3 Detection

Analyte particles pass through a detection chamber (Figure 1-7) 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 Photomultiplier which converts the scattered light to signal and which is positioned at an angle of 120° with respect to the light beam. In SEDEX detectors a secondary gas inlet is used to concentrate the particles in the centre of the detection chamber to further increase sensitivity and to prevent it from contamination.

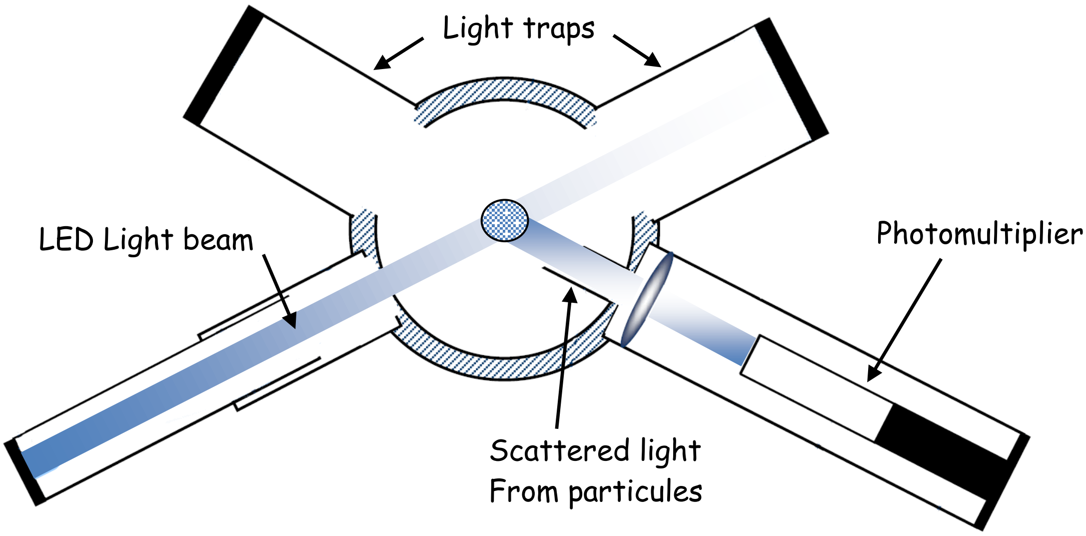
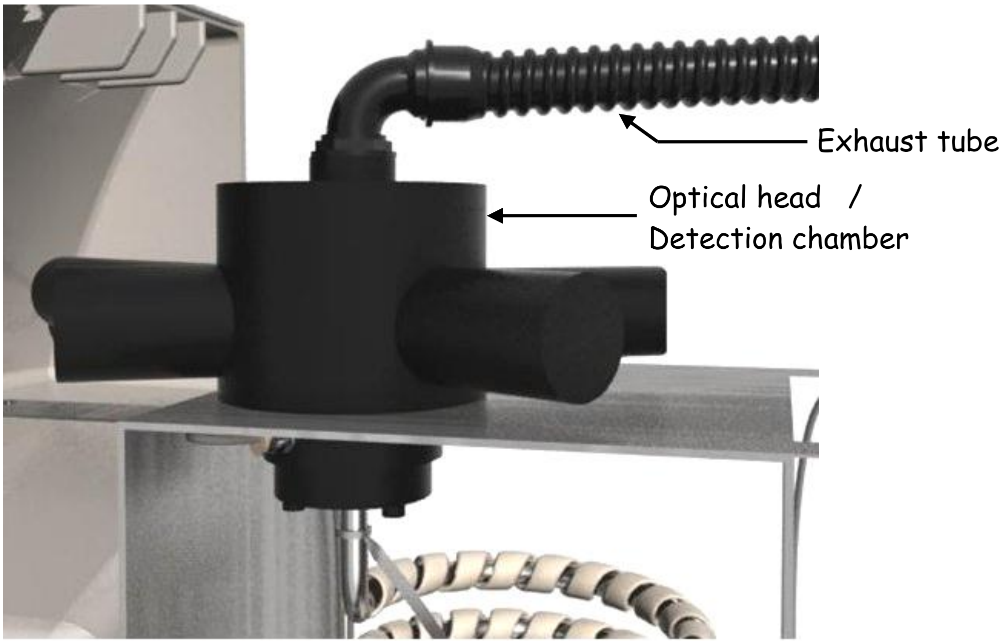


Figure 1-7: Detection Chamber



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 1-1.

$$\mathbf{I = k.m^{b}} \qquad \mathbf{(1\text{-}1)}$$

where: **I** is the intensity of light
m is the mass of the scattering particles
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 Content of this Manual

This manual is designed to describe the installation, operation, maintenance and troubleshooting of the SEDEX ELSD 80 Low Temperature Evaporative Light-Scattering Detector. It includes:

- *Chapter 2* - **Installation of the Detector**, describes suitable laboratory conditions for the detector and includes information about interfacing the detector to other devices.

- *Chapter 3* - **Start-up Procedure**, describes the role of the various controls and displays on the detector. In addition, this chapter discusses a number of activities to prepare the unit for routine data collection.

- *Chapter 4* - **Operating the Detector**, describes how to operate the Low Temperature Evaporative Light-Scattering Detector. It includes information about starting the unit on a routine basis, collecting data and shutting the unit down.

- *Chapter 5* - **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.

- A series of appendices are provided which include product specifications, a list of spare parts, Standard Operating Procedures and Software Chromatography Drivers.

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 your local distributor.

Please, visit our website for additional information or assistance:

www.sedere.com

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

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.

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2 Installation of the Detector

2.1 Overview

This chapter describes how the laboratory should be prepared to optimize the performance of the SEDEX ELSD 80 Low Temperature Evaporative Light-Scattering Detector and indicates how the unit is interfaced to other devices such as the column and the data recording device. When you have successfully installed the unit, refer to Chapter 3 for start-up procedures.

The detector is provided in three different configurations as described in Table 2-1. Each configuration includes the indicated nebulizer to allow the user to employ the nebulizer to best match the application.

The detector is provided in three different configurations as described in Table 2-1. The first configuration (with HPLC nebulizer) is dedicated to standard HPLC applications, the second one (with CC nebulizer) is dedicated to Combinatorial Chemistry using high eluent flow rates. The third one (with FLASH nebulizer) is a dedicated detector for Flash Chromatography.

All models can be provided with RS-232 activated (communication option) to be fully controlled by PC via drivers for Chromatography Software or not. The communication option can be activated afterwards. An accessory kit includes the components indicated in Table 2-2.

SEDEX ELSD Model 80LT	Flow Rate Range	Communication Option	115V	230V
Detector bundled with a *HPLC Nebulizer*	100µL/min - 2.5mL/min	Without With	80001 80001S	80000 80000S
Detector bundled with a *Combinatorial Chemistry Nebulizer*	1.0mL/min - 4.0mL/min	Without With	80401 80401S	80400 80400S
Dedicated Flash Detector bundled with a *Flash Nebulizer*	100µL/min - 5mL/min	Without With	80501 80501S	80500 80500S

Table 2-1: SEDEX Model 80LT Low Temperature Evaporative Light-Scattering Detector Versions

Some accessories included with the detector are described in Table 2-2.

Quantity	Description
1	Operator's Manual (this manual)
1	Nebulizer on a separate box (refer to Table 2-3 for exact model)
1	Glassware on a separate box
1	**Starting kit:** 1x Power cable, 1x RS232 cable (if software option activated) 1x Autozero cable, 1x Signal cable, 1x External Event cable 2 meters + 1 meter of 6mm O.D. gas tubing 1x set of replacement fuses

Table 2-2: Components Shipped with the SEDEX ELSD 80 Low Temperature Evaporative Light-Scattering Detector

The user can employ new nebulizers to best match his applications. The complete range of nebulizers for SEDEX ELSD 80 is indicated in Table 2-3.

Nebulizer Model	Flow Rate Range	Back Pressure (with water)	Part Number
HPLC Nebulizer	100µL/min - 2.5mL/min	4bar (1mL/min)	80003
Combinatorial Chemistry Nebulizer	1.0mL/min - 4.0mL/min	4bar (1mL/min)	80008
Flash Nebulizer	100µL/min - 5mL/min	4bar (1mL/min)	80005

Table 2-3: Nebulizers for the SEDEX ELSD 80 Low Temperature Evaporative Light-Scattering Detector

SEDERE provides a wide range of accessories (e.g. Gas Regulator with Filter and Manometer) 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). Two persons are needed to ensure an easy transport and avoid physical injury (Figure 2-1).

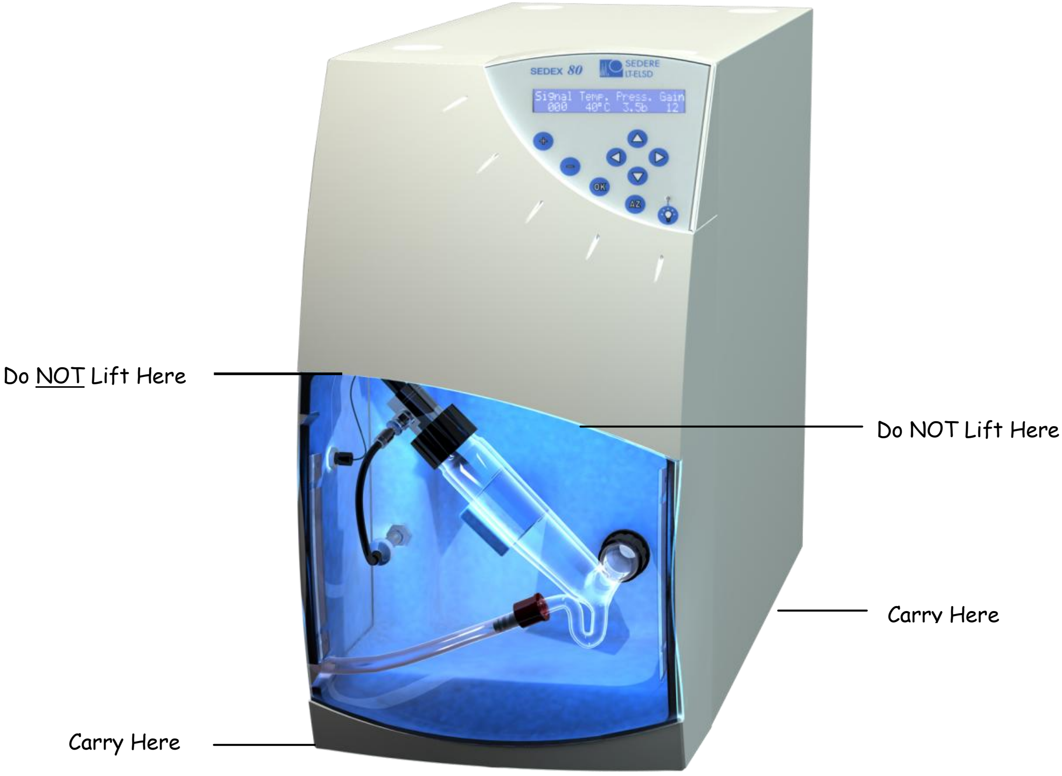


Figure 2-1: Carrying the Detector

2.3 Unpacking the Detector

Unpack components carefully, making sure all items in the Table 2-2 have been included. The glassware chamber and nebulizer are packed in separate boxes in the shipping box. 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 115VAC or 230VAC with 50/60Hz input power (depending on the country to which it is shipped). Ensure that the voltage value indicated on the power connector on the rear panel corresponds to the line voltage in your facility.

The detector requires 115V/1.8A or 230V/1.7A. Check that the power lines can provide sufficient current.

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. Any interruption of the protective earth grounding will cause a potential shock hazard that could result in serious personal injury. 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).

☞ Note: **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 exit the detector through the black exhaust tube located on the top 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 should be moderate so as to avoid turbulence in the glass cell siphon.

⚠ **The exhaust should not be allowed to enter the laboratory atmosphere and any appropriate accessory (e.g. solvent filter) should be disposed of in a manner that meets the local regulatory authorities for health and safety requirements.**

⚠ **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 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.

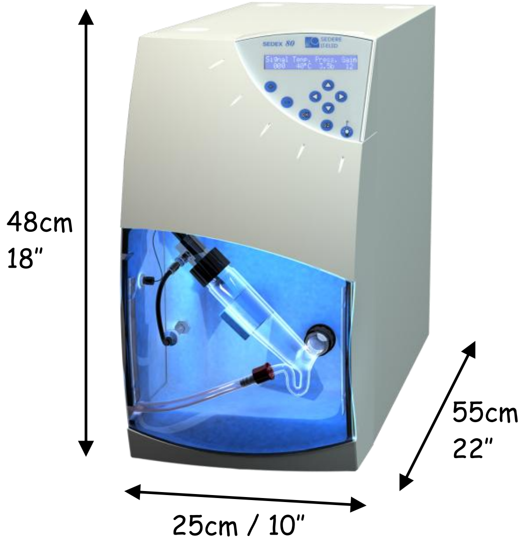
☞ Note: **Ensure that the Parafilm™ is removed from the exhaust tube before installing the unit.**

The drain tubing 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 Location of the Detector in the Laboratory

All components of the system (e.g. HPLC pumps, detector) should be located on a sturdy table. The detector should be placed in an area that is free from drafts or significant temperature changes. Do not place it near air conditioning vents, windows, ovens, etc.



When placing the detector in the laboratory, access to the power to disconnect the device (the appliance coupler or the mains plug) must be kept accessible at all time.

☞ Note: **The detector should be placed close to the outlet of the column to minimize extra-column band broadening which would reduce the resolution of the chromatographic separation.**

☞ Note: **As a destructive detector, the ELSD should be the last one in the flow path or alternatively be used with a splitter.**

2.4.5 Environmental Conditions

This instrument has been designed for the following conditions:

- Use inside buildings (IP20)
- 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 Category II
- Equipment Class I, Pollution degree: 2

2.5 Installation of the Unit

2.5.1 Detector rear panel

On the top of the detector rear panel are located the fan which provides cooling airflow through the detector and the black exhaust tube where gas and mobile phase vapour and solute particles exit the detector. The exhaust tube installation is described in Section 2.5.3.

On the back of the detector rear panel is located a supply panel (Figure 2-2) with the gas inlet connection (Section 2.5.2), the power module (with main power switch and lines fuses) and the electrical connection (Section 2.5.4).

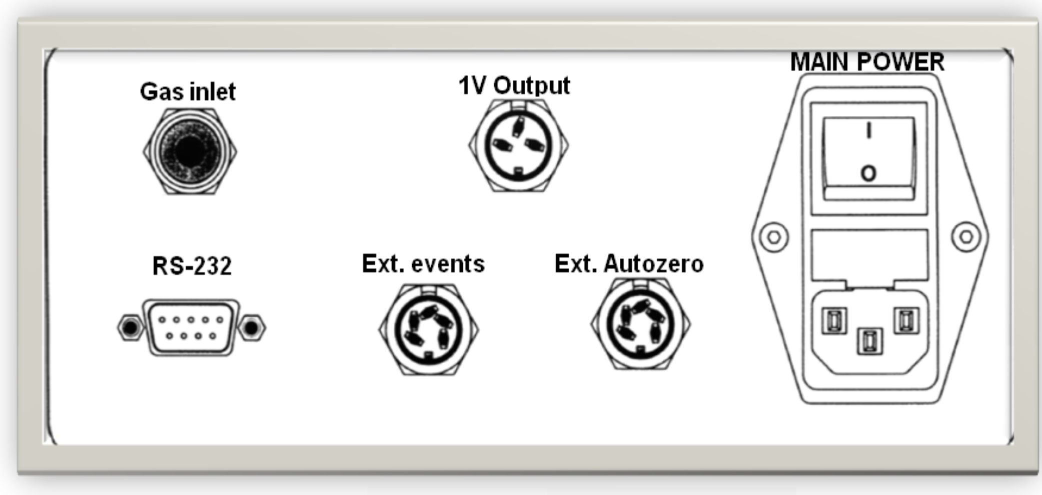


Figure 2-2: Supply Panel

2.5.2 Gas Supply

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 (Figure 2-2) which requires no additional fitting.

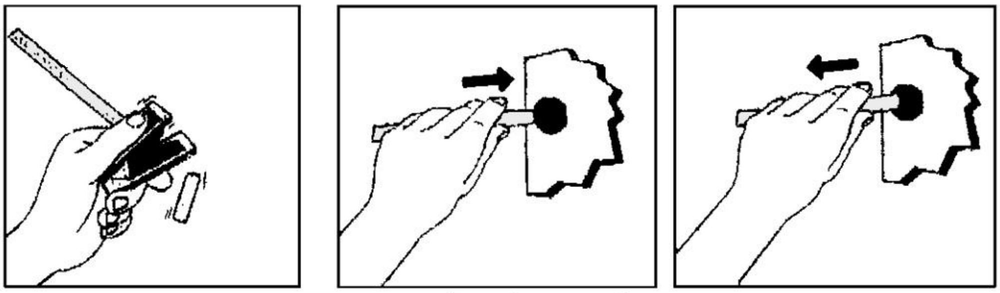
The gas supply must be stable and regulated by an external manometer. The gas must be oil-free, dry and filtered by a 0.01µm filter. The typical gas pressure to operate the detector is 3.5bars for both the HPLC and the CC models and 2.0bars for the Flash model.

⚠	**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.**

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

Note: **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.**

The tubing should be cut and firmly inserted into the fitting as shown in Figure 2-3, after removing the Parafilm™ from the detector gas inlet.



Cut the tube square.

Insert the tube into the fitting until it bottoms.

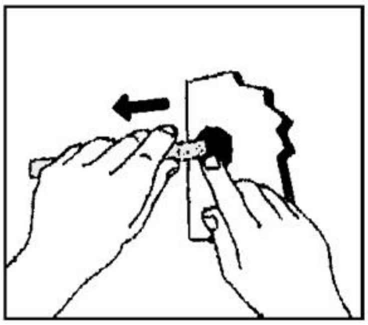
Pull the tube to check engagement of the grab.

Figure 2-3: 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 2-4.



To remove the tube, disengage the grab ring teeth by a simple manual pressure on the push sleeve and withdraw the tube from the fitting.

Figure 2-4: 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 particules 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 (excessive noise or loss of sample).

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 of tubing can be fitted over the exhaust tubing.

2.5.4 Electrical Connections

All electrical connections are made via the supply panel (Figure 2-2) on rear panel.

a) *Connecting the Analog Signal Output:*

If the Analog Signal output from the detector is to be employed, plug the Signal cable that is supplied into the *Signal* socket on the detector (Figure 2-2) and to the appropriate socket of the data collection device. Make sure to use the correct polarity identified on the cable.

This is not required if a Chromatography Software Driver is used to control the detector. In this case, there is no need to use an A/D convertor.

b) Connecting the External Autozero:

If the external autozero function is to be employed, plug the cable that is supplied into the *Ext Autozero* socket on the detector (Figure 2-2) and to the appropriate socket on the controlling device (e.g. autosampler, pump, etc.).

Refer to section 4.3.2 to operate external autozero signal.

If a TTL signal is used please make sure to use the correct polarity identified on the cable.

The external Autozero cable has to be connected to a “Start” information and not an “AutoZero” information on a controlling device (e.g. autosampler) <u>only if</u> a Chromatography Software Driver is used to control the detector. In this mode, the detector doesn’t proceed to an Autozero, it uses it as a signal synchronization for the driver.

Not using the Autozero connection for a “Start” information will impair the synchronization of the Signal and may not generate the final report and/or impair the retention time reproducibility.

c) Connecting the External Events Cable

If the external events functions are to be employed, plug the cable that is supplied into the appropriate socket on the rear panel of the detector (Figure 2-2) and to the appropriate socket on the controlling device (e.g. autosampler, pump, etc.).

The white cables are contact closure “output” cables that provide the ready/non-ready information to an external device. The detector will be in the “not-ready” mode (the contact will be in closed position) if any one of the following conditions is observed:

- The light source is off.
- The temperature is not at the indicated setpoint.
- The temperature is at the indicated setpoint but is not stable.
- The pressure is below 3.0bar (HPLC and CC models) or is below 2.0bar (FLASH model).

☞ Note: **The controlled device electrical consumption mustn't exceed 20mA under 12V DC.**

The blue cables are contact closure/TTL “input” cables that are used to power the unit down (see Section 3.2.2.h) via a signal from an external device to the detector. If the TTL signal is used please make sure to use the correct polarity identified on the cable.

d) Connecting the RS232 Port

If a computer is used to control the detector, the detector should be connected to the computer via a free RS232 port using supplied cable. In this mode, this avoids also to use an A/D convertor to convert the Analog Signal from the detector. Please note that the RS232 Communication is an option and requires to be enabled prior to use any drivers. This option can be activated at any time (contact your local distributor).

The software drivers for Chromatography System (i.e ChemStation, EZChrom Elite...) available are detailed in Appendix 4.

e) Connecting the Power Cord

Place the ON/OFF switch to the OFF position and plug the power cord into the socket on the rear panel of the detector.

The power cord of this instrument contains three wires which must be connected to a grounded line. All components of the chromatographic system should be connected to a common ground. If a two wire outlet is used, make certain that an adapter is used to connect the third wire to ground.

Do not turn on the power at this time.

2.5.5 Installing the Nebulizer/Glass Chamber Assembly

The transparent black front door can be removed to access the nebulizer and glassware chamber. When removing this transparent black front door, gently pull only its left side towards you. To install the door, first fix its right side, and then gently push its left side.

Parafilm™ is used to cover various openings inside the compartment, nebulizer and glassware to prevent dust particles from entering the detector during shipment.

The installed Nebulizer/Glassware Chamber assembly is shown in Figure 2-5.

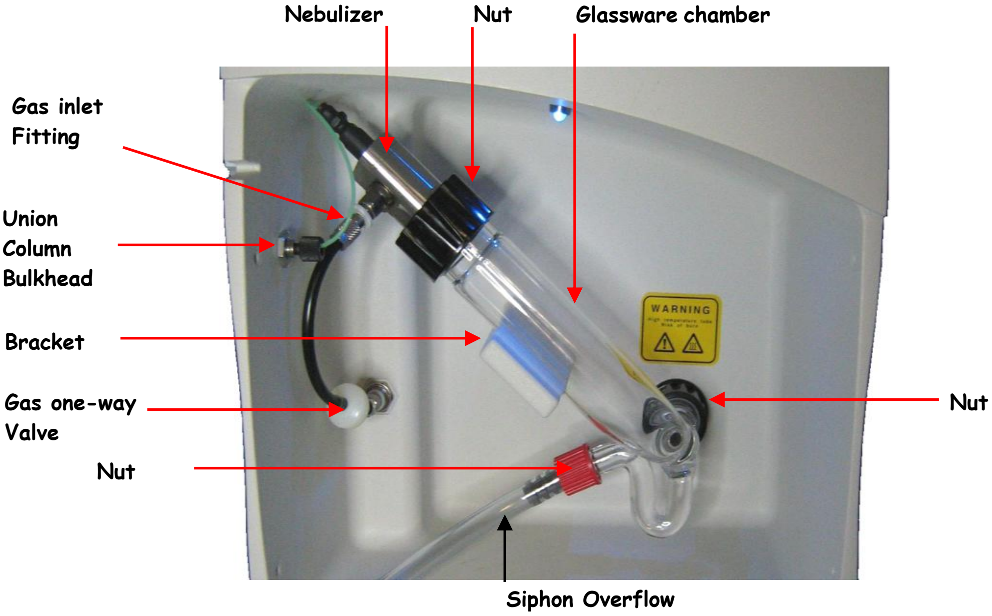


Figure 2-5: Installing the Nebulizer/Glassware Chamber Assembly

To install the assembly:

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

b) Position the glassware chamber as shown in Figure 2-5 and tighten the black nut at the bottom. The glassware chamber should be placed on the backwall with the backwall as shown in Figure 2-6.

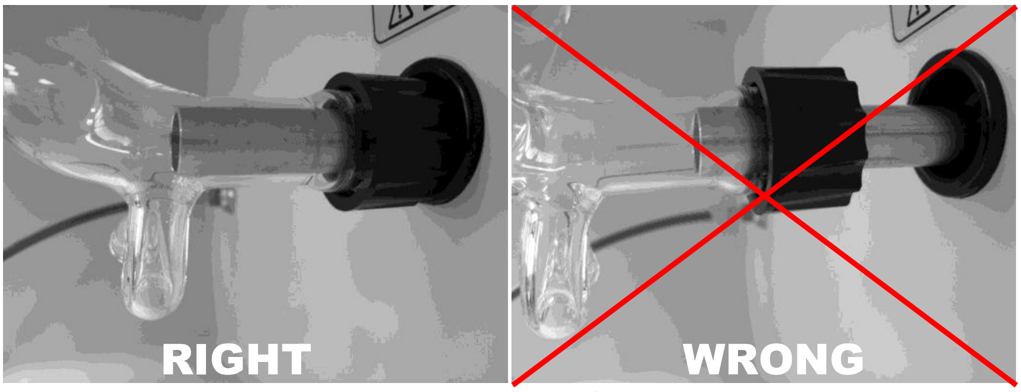


Figure 2-6: Fixing the Glass Chamber

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

d) Screw the inlet fitting in the bulkhead on the left side of the compartment. Special care must be taken when positioning this fitting. The nebulizer is terminated with a small piece of Teflon tubing with an outer green sleeve. For proper operation, the Teflon tubing must extend less than 2mm past the end of the green sleeve (Figure 2-7).

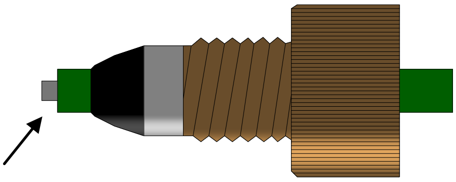


Figure 2-7: Nebulizer Inlet Fitting

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.

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

2.5.6 Connecting the Siphon Overflow

Attach the drain tube assembly to the end of the siphon tube using the tapered hose connector 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.

☞ Note: **A drain tube with a bend or diving in the liquid will create pressure fluctuations in the detector and will result in bad measurement results.**

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

If the solvent that you are using is not compatible with Tygon (e.g. THF), use a piece of Teflon tubing or any material you know compatible with your solvent in its place.

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

2.5.7 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 the nebulizer side. See Figure 2-5.

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

2.5.8 Connecting the Column

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

2.5.9 Powering Up the 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.

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 the version number and date it was created for a few seconds (the version number should be recorded as it may be required for service or troubleshooting) 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.

☞ Note: **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 3 to prepare the unit for routine operation.

3 Start-up Procedure

3.1 Overview

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

3.2 The Control Panel

The Control Panel (Figure 3-1) includes a digital display and a number of buttons that are used to enter data.

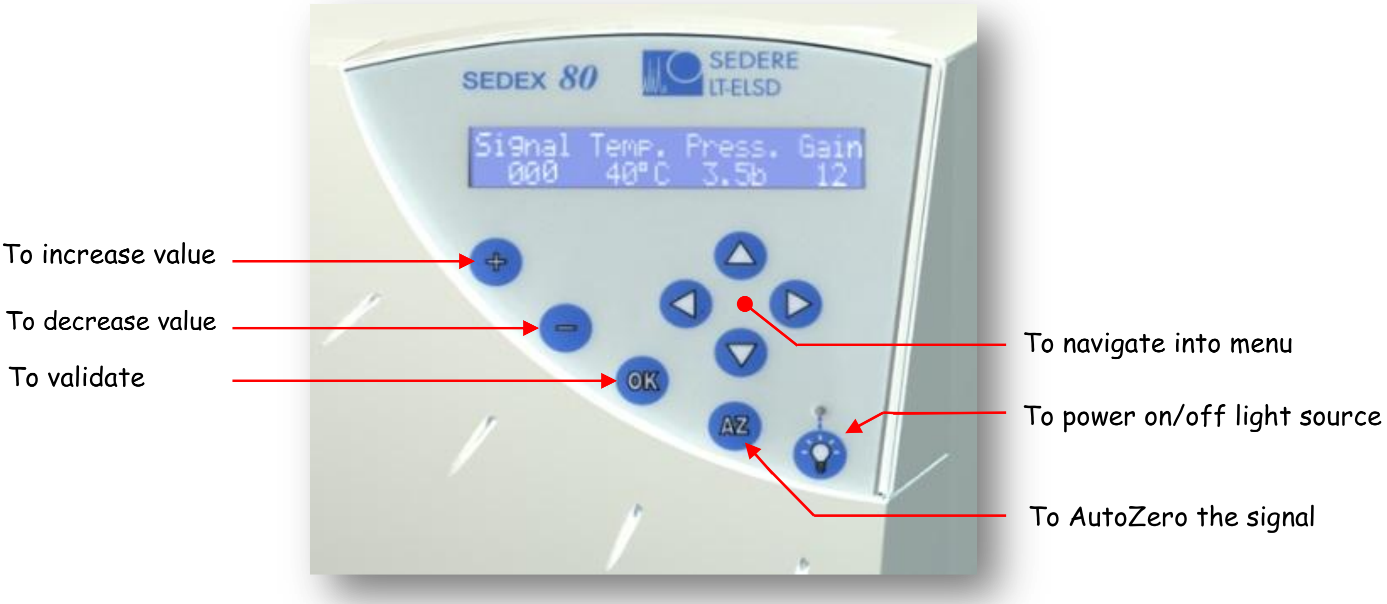


Figure 3-1: The Control Panel

3.2.1 The Digital Display

The digital display presents information about the present status of the detector and is used to enter a variety of parameters. When the detector is powered up, the display will present a greetings message that includes the version number and date that the version was created for a few seconds.

After the detector has completed the initialization procedures, it loads the last conditions used before the power off and the *Status* screen (Figure 3-2) will be presented.

Signal	Temp	Press	Gain
001	26°C	3.5b	1

Figure 3-2: The Status Screen

The user interface is provided via a series of screens that are described in Section 3.2.2. Some screens present information about the instrument status and cannot be edited by the user (e.g. the *Status* screen), while other screens (e.g. the *Temperature/Gain* screen, Figure 3-4) are used to enter the desired set points.

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

+	used to increase the present value of a user settable parameter (e.g. the offset) by 1 unit. If you keep the key pressed, the rate of change of the parameter will increase.
−	used to decrease the present value of a user settable parameter (e.g. the offset) by 1 unit. If you keep the key pressed, the rate of change of the parameter will increase.
OK	validates the value of the parameter that you have edited.
AZ	sets the present signal for the detector to zero by updated the offset value to set the present signal to 0mV.
💡	is used to power up the light source in the detector. When the light source is lit, the keyboard orange L.E.D. immediately above the button will be illuminated and blinks in case of light source time elapsed (warning).
△ ▽	changes the active line on the display to the next (previous) line or the next (previous) screen.
◁ ▷	moves the cursor on the display to the next (previous) field.

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

3.2.2 The User Interface

The *Status* screen (Figure 3-2) 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.

Each parameter change must be validated with OK button or the change will not be applied.

When the ▼ button is pressed, it displays the next line or the next screen.

3.2.2.a The *Status* Screen

The *Status* screen (Figure 3-2) shows the present conditions of the detector. This screen cannot be edited, but the desired offset can be set via the *Offset* screen (Figure 3-3), the temperature and gain can be set via the *Temp/Gain* screen (Figure 3-4) and the pressure units can be selected via the *Noise Filter/Pressure Unit* screen (Figure 3-5).

Temperature value blinks if desired temperature is not reached or not stable. Pressure value blinks if gas pressure is lower than 3.0bar (HPLC or CC detector models) or 2.0 for the FLASH detector model.

When the ▼ button is pressed; the *Signal Offset* screen (Figure 3-3), which is used to select the desired offset is displayed.

3.2.2.b The *Offset* Screen

Signal Offset (mV):
000

Figure 3-3: The Offset Screen

To increase the offset value, click on the + button. If you press the button quickly, the offset will increase by 1; if you press and hold the button, the value will increase at the rate of 20mV/sec.
Once you have set the desired offset, press the OK button to validate the new value.

When the instrument is Autozeroed, the Autozero operation updates the Offset value to set the Signal to 0mV.

Press the ▼ button to access the *Temp/Gain* screen (Figure 3-4).

3.2.2.c The *Temperature/Gain* Screen

Temp: 50°C
Gain: 1

Figure 3-4: The Temp/Gain Screen

The *Temp/Gain* screen is used to set the desired evaporation tube temperature and the photomultiplier Gain. When the screen is accessed, the cursor is on the *Temp* setting. This evaporation tube temperature can be updated with the + and − buttons and validated with the OK button.

The evaporation tube temperature range is from 20 to 100°C.

☞ Note: **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.**

☞ Note: **To maintain appropriate temperature control, when the lowest temperature is required, it should be set at least 5°C above ambient. Temperature stabilization typical time is 30 minutes. Please, note that the stabilization time for temperature close to the ambient temperature may be longer.**

When you press the ▼ button, the *Gain* setting field can be edited in the normal manner. The gain range is from 1 to 12 and each increase in gain setting produces a twofold signal amplification (e.g. if you change the gain from 1 to 4, the gain is increased by a factor of 8) and the full range of the gain from Gain 1 and Gain 12 is 1 to 2048. The Gain corresponds to the detector sensitivity, achieved by controlling the high voltage applied to the photomultiplier to amplify response.

After you have validated the desired gain setting, press the ▼ button again to display the *Autozero Offset* screen (Figure 3-5).

3.2.2.d The *Autozero Offset* Screen

Output Signal Value
After AZ : 000mV

Figure 3-5: The Autozero Offset Screen

This screen is used to allow the baseline level to reach the desired value after an AutoZero is performed (with the keyboard or with the external AutoZero signal input on detector rear panel).

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.

This setting can be changed with the + and − buttons and validated by the OK button.

After you have set the desired autozero offset, press the ▼ button to display the *Noise Filter/Pressure Unit* screen (Figure 3-6).

3.2.2.e The *Noise Filter/Pressure Unit* Screen

Filter: 1s
Press Unit: Bar

Figure 3-6: The Noise Filter/Pressure Unit Screen

The *Filter/Pressure Unit* screen is used to indicate if digital filtering is desired for the signal data (improves signal-to-noise ratio) and the desired units for the pressure display.

When the screen is presented, the cursor is on the *Filter* field. By pressing + or − keys, you change the filtering strength (time constant) within the following range:

- "NO": no filtering (row data).
- 0.5s: 0.5 second moving average filtering.
- 1s…10s: 1 to 10 seconds moving average filtering.

☞ Note: **For better results, the digital Filter should be used unless the peak(s) of interest are very poorly resolved (e.g. when Rs<1.5).
Default value is 1s, corresponding to a peak width of approximately 2 seconds at half-height. User manual section 4.5.5 details Filter optimization.**

If you have changed the value, press OK to validate it before you press the ▼ button to access the *Press Unit* line. The pressure unit line allows for the selection of KPa, bar or psi for pressure units, the desired selection is made via the + or − key, and validated by the OK key.

When you press the ▼ button, the *Light Source* screen (Figure 3-7) will be displayed.

3.2.2.f The *Light Source* Screen

LED: ON ####### H
Reset Time Elapsed

Figure 3-7: Light Source Screen

The *Light Source* screen is used to power the light source On/Off and is equivalent to the *Light Source* button on the control panel.

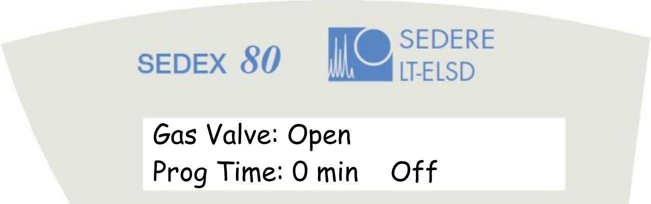
Use the + button followed by the OK button to power the light source on and the − button followed by OK the button to turn it off.

The *# hours* field indicates the number of hours that the Light Source has been in use and cannot be edited by the user. When the lifetime light source period has been reached (approximately 5000h), a message indicating that the maximum usage of the light source has been exceeded will be presented when the unit is powered up and the orange LED on the keyboard blinks when the light source is power on.

☞ Note: **The *Reset Time Elapsed* field should be validated with** OK **only when you change the lamp.**

When you press the ▼ button, the *Gas Valve* screen (Figure 3-8) will be presented.

3.2.2.g The *Gas Valve* Screen



Gas Valve: Open
Prog Time: 0 min   Off

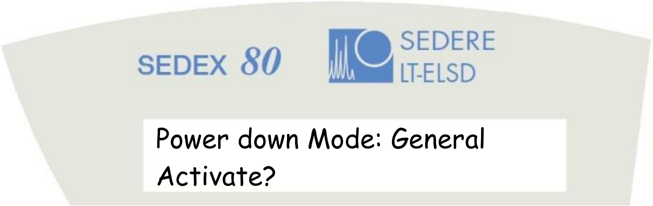
Figure 3-8: The Gas Valve Screen

The *Gas Valve* 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, indicate the appropriate time, then move the cursor to *Off* and use the + or − key to select *On* and press OK

When you press the ▼ button, the *External Shutdown* screen (Figure 3-9) will be displayed.

3.2.2.h The *Power Down* Screen

The *Power Down Mode* screen (Figure 3-9) is used to indicate which features should be shut down upon receipt of a power down signal from an external source or from the menu.



Power down Mode: General
Activate?

Figure 3-9: The Power Down Screen

The three options provided for external shutdown are summarized in Table 3-1.

Mode	Photomultiplier	Lamp	Heating	Gas flow
General	Off	Off	Off	Off
Standby	Off	Off	On	Off
Cleaning	Off	Off	On	On

Table 3-1: Power Down Options

To select the desired *Power Down* mode, use the + or − key to access the desired mode and then press OK to validate the selection.

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

Once the Power Down mode has been chosen and validated, the detector can be powered down in two ways:

- **With 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.
- **With Power down screen**: Press the ▼ button to access the power down screen, then press again the ▼ button to place the cursor on the *Power down activate* line.
  Validate with OK to put the detector in power down mode.

> ☞ Note: **To leave the power down mode, release the External 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.**

When the cursor is on the *Power down activate* line, pressing the ▼ button will present the *Total Lifetime Elapsed* screen (Figure 3-10).

3.2.2.i The *Total Lifetime* Elapsed Screen

Total Lifetime Elapsed
####### hrs

Figure 3-10: The *Total Lifetime Elapsed* Screen

The *Total Lifetime Elapsed* information screen indicates the usage of the detector and cannot be edited by the user. When you press the ▼ button, the *Serial Number* screen (Figure 3-11) will be displayed.

3.2.2.j The *Serial Number* Screen

Serial Number
##########

Figure 3-11: The *Serial Number* Screen

The *Serial Number* screen cannot be edited by the user. The last character indicates the detector hardware revision. When you press the ▼ button, the *Firmware* screen (Figure 3-12) will be displayed.

3.2.2.k The *Firmware* Screen

Firmware Version: #.#
Firmware Date: MM/YY

Figure 3-12: The *Firmware* Screen

This information screen presents the firmware version and date release, where MM is the month, and YY the year. The *Firmware* screen cannot be edited by the user.

When you press ▼ the button, the *Factory Menu Code* screen (Figure 3-13) will be displayed.

3.2.2.1 The *Factory Menu* Code Screen

Factory Menu Code: ____
Authorized Persons Only

Figure 3-13: The *Factory Menu* Code Screen

The *Factory Menu Code* screen is reserved for qualified personnel only to access a variety of service features and is not designed to be employed by the user.

3.3 Initial Test Procedure

3.3.1 Preliminary Activities

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.

☞ Note: **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.**

☞ Note: **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 (HPLC & CC models) or gas pressure to 2.0bar (FLASH model).**

The warm up period for the detector and light source is about 60 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 *Temperature/Gain* 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.

☞ Note: **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) or 2.0bar (29psi) for the Flash model only. If the pressure is less than 3.0bar (44psi) or 2.0bar (29psi) for the Flash detector model only, the pressure value blinks, indicating that the detector is not ready.

☞ Note: **Make certain that the pressure of gas supplied to the detector is less that 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) Press the AZ button. The signal should be close to zero and remain constant.

e) Set the noise filtering to **1s** (Refer to Section 3.2.2.e).

3.3.2 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 (2.0bar for the Flash model) and temperature to 50°C. Wait for stable temperature.

d) Set gain 12 and monitor the signal for a period of 5 min. The variation in the signal should be less than +/- 2mV (there may be some spiking of the signal).

e) Record the level and autozero the detector again.

3.3.3 Background Noise (Stray Light) Test

To determine the background noise:

a) 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.

f) Set gas pressure to 3.5bar (2.0bar for the Flash detector model) and temperature to 50°C. Wait for a stable temperature.

b) Switch on the light source.

c) Change the Gain to 1.

d) Set the offset after Autozero to 0mV (Refer to Section 3.2.2.d).

e) Autozero the detector.

f) Change the Gain to 12.

g) Wait 15 minutes for stabilization and record the signal level. The expected level is typically 100mV to 150mV for both the HPLC and CC detectors models and typically 2mV to 20mV for the Flash detector model. The exact value will vary slightly and small deviations should not be a cause for concern.

3.3.4 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 (51psi) or 2.0bar (29psi) for the Flash detector model, 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 12 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 200mV.
  - 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) mixture should provide a signal increase of approximately less than 100mV).

☞ Note: **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.**

☞ Note: **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 5.4.2 or clean the nebulizer as described in Section 5.3.5.

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).

3.3.5 Column Noise Test

☞ Note: **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	208
1.0	47
0.8	30
0.32	4.8

Table 3-3: Flow Rate versus Column Diameter Indication

c) Set the gain to 12 and monitor the baseline for a few minutes. A suitable column will provide a baseline that is no more than 20-50mV 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.**

4 Operating the Detector

4.1 Overview

This chapter describes the operations that should be performed on a routine basis when you want to collect chromatographic data using the SEDEX ELSD 80LT 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 3) and that the general chromatographic conditions for the separation have been determined.

4.2 Startup 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) or 2.0bar (29psi) for the Flash detector model. 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 *Temp/Gain* screen, which is accessed by pressing the ▼ button two times 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.

☞ Note: **The Solvent Noise test (Section 3.3.4) and the Column Noise test (Section 3.3.5) should be performed to verify that the detector is functioning in a proper manner.**

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

4.3 Auto-zeroing the Detector

4.3.1 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 *Temp/Gain* screen, which is accessed by pressing the ▼ button two times when the *Signal* screen is displayed.

b) Press the AZ 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* screen is accessed by pressing the ▼ button when the *Status* screen is displayed.

☞ Note: **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.**

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

4.3.2 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 Ext. Autozero socket on the rear panel (Section 2.5.3).

To auto-zero the detector, a contact closure signal or a TTL signal is used to short circuit the contacts. The signal should be at least 0.5sec 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.

4.4 Routine Operation of the Detector

In general, operation of an LC 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 2.5.2).

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

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.

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

d) Never exceed a gas pressure of 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.

After each session and before shutting down the system, the detector should be cleaned (flushed with a small amount of fresh solvent) in order to ensure good performances.

4.5 Optimizing Performance

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

4.5.1 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.

Note: **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.

As an example, consider the analysis of caffeine with evaporation temperatures of 30°C and 60°C (Figure 4-1). 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.

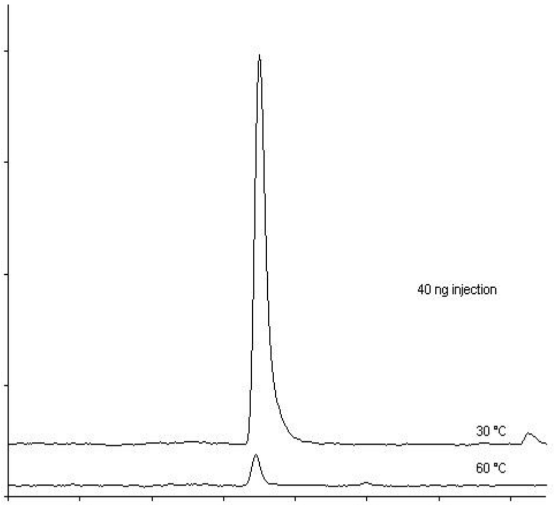


Figure 4-1: Chromatogram of Caffeine at Various Temperatures

4.5.2 Optimizing the Mobile Phase

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

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.

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.

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

Figure 4-2: Chromatogram with Various Solvent Quality

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.

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

4.5.3 Sample Pretreatment

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

4.5.4 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 3.3.5) to obtain the baseline signal value corresponding to this column.

4.5.5 Optimizing the Noise Filter

The Digital Filter (see section 3.2.2.e) 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% (Second)	Proposed Filter (Second)
<1	0
2	1
4	2
6	4
8	6
>10	8 and higher

Table 4-1 Digital Filter versus Peak Width Indication

These suggested values can be optimized depending on your specific chromatography, by decreasing Filter if peaks are poorly resolved (e.g. when Rs<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 2.5second.

	Filter 0s	Filter 1s	Filter 2s
Signal Height	124 mV	122mV	110mV
Noise (ASTM)	3.2mV	1.1mV	0.7mV
Peak Width (at 50% height)	2.5 second	2.5 second	2.8 second
S/N	**37**	**110**	**157**

Table 4-2 Sensitivity improvement depending on Filter

Signal-to-Noise ratio is multiplied by 3 when selecting Filter 1s without any peak broadening effect. If Signal-to-Noise ratio is more important than resolution, a Filter 2s or higher can be set to improve sensitivity even better.

4.6 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 3.2.2.h.

To shut down the instrument:

a) Before shutting down the system, the detector should be cleaned (flushed with a small amount of fresh solvent) for few minutes in order to ensure good performances. Then turn off the mobile phase flow.
b) Allow detector gas only to flow for few minutes (30minutes 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 3.2.2.g).
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.

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5 Maintenance and Troubleshooting

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

5.1 Overview

This chapter describes:

- The maintenance procedures that should be performed by the operator on a routine basis (Section 5.2).
- Troubleshooting activities that should be useful in determining the cause of erratic or erroneous results (Section 5.3).
- Cleaning and decontamination procedure that should be performed to maintain instrument performance (Section 5.4).
- The replacement of main fuses (Section 5.5)

5.2 Maintenance

The SEDEX ELSD 80 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.

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.

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 3.2.2.g

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.

☞ Note: **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.**

☞ Note: **The L.E.D. used as the Light Source has a long but finite lifetime (~ 5000 hours) and should be replaced periodically 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 up and the orange L.E.D. on keyboard blinks when light source is power on.**

After each session and before shutting down the system, the detector should be cleaned (flushed with a small amount of fresh solvent) in order to ensure good performances.

5.3 Troubleshooting

5.3.1 General Troubleshooting Information

The SEDEX ELSD 80LT 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).
- 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.

⚠

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.

☞ Note: **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.**

5.3.2 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) regarding HPLC and CC applications or 2.0bar (29psi) for FLASH applications. Gas consumption is about 4L/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 oil residue.

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

5.3.3 Perform the Noise Tests

Repeat the tests described in Section 3.3 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 3.3.2), Background Noise test (Section 3.3.3) and Solvent Noise test (Section 3.3.4) provide results that are similar to that obtained when the unit was initially installed, but the Column Noise test (Section 3.3.5) 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).

5.3.4 Specific Detector Troubleshooting

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 5.3.5.

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.

   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 5.3.5. 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.

c) 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:

   - 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.

   - 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.

d) 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 100°C and pump appropriate solvent at the rate of 2mL/min, using a gas pressure of 3.5bar (51psi) for both HPLC and CC detectors models; or 4mL/min to the detector, bypassing all splitting device and using a gas pressure of 2.0bar (29psi) for the Flash detector model. 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.

5.3.5 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.

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 over 2.0bars, 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 2-5.

To remove the nebulizer from the instrument:

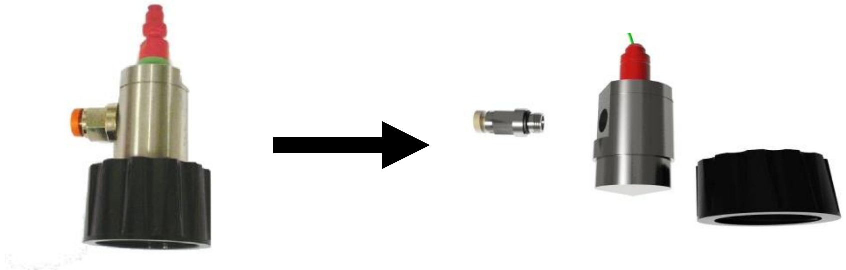
a) Switch off the pump and the ELSD detector.

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

c) Disconnect the nebulizer liquid inlet from the column and,
Disconnect the gas inlet from the nebulizer by pushing on the white inlet (refer to Figure 2-4 for detail).

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. Take care not to pull or twist the nebulizer capillary.

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



To clean the nebulizer:

a) 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.

b) 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.

c) 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.**

To re-install the nebulizer or replace it by a new one or another nebulizer model:

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.

☞ Note: **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.

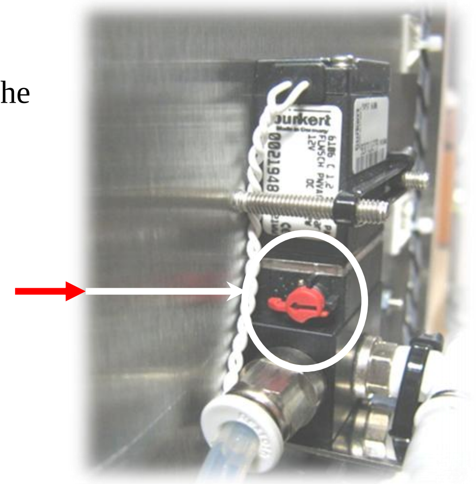
5.3.6 Gas Flow Problems

If the gas pressure is correct, but the nebulizer does not produce any spray (the liquid may be visible on the glassware), make sure that the black gas tubing for the nebulizer is in the correct direction, where the white one-way valve is at the lower end (near the gas arrival).

The gas pressure is indicated on the *Status* screen and is controlled by a valve inside the detector housing. If the gas menu command does not work, a manual bypass can be done on the gas valve to open it (to set gas flow).

To bypass the gas valve:

a) Switch off the detector and disconnect the detector from the main power supply,
b) Remove the detector cover,
c) The gas valve is located on the detector rear panel,
d) Turn the red switch on the gas valve
e) Replace the cover and power up the instrument.



5.4 Cleaning and Decontamination

5.4.1 Instrument Cleaning

1. Switch the instrument off.
2. Disconnect all connection cables (power cable, signal cable, autozero cable,..., instrument gas input and nebulizer tubing).
3. Allow the detector to cool down.
4. 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.

5.4.2 Instrument Decontamination

Set the evaporation temperature to 100°C and the gas pressure to 3.5bar (51psi) or 2.0bar (29psi) for the FLASH detector model.

Pump the appropriate solvent through the system at the rate of 2mL/min (4mL for the FLASH detector model) to the detector, bypassing all splitting device. 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.

5.5 Fuses replacement

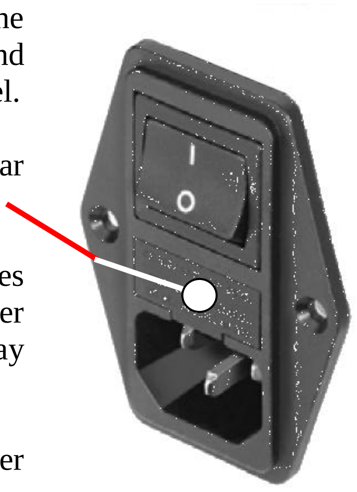
If the digital display does not switch on when the detector is powered up, power the unit off and inspect the main fuses. The fuses are located inside the main power module on the rear panel (Figure 2-2). A set of replacement fuses is delivered in the starting kit.

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

2. Locate the fuse compartment on detector rear panel and withdraw the fuses holder.

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

4. Reconnect the power cord to the power module and to main power lines.



If the fuses are not blown or if the replacement fuses 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.**

5.6 Light source

The L.E.D. used as Light Source has a long but finite lifetime (~ 5000 hours) and should be replaced periodically. A decreasing L.E.D. light intensity will cause decreasing signal heights over time. When this period of 5000 hours has been reached, a message indicating that the maximum usage of the lamp has been exceeded will be displayed when the unit is powered up and the orange L.E.D. on keyboard blinks when Light Source is power on.

The Light Source exchange part number is:

- 80007 for Sedex 80 Hardware from ‘A’ to ‘C’
- 80023 for Sedex 80 Hardware from ‘D’

Hardware revision can be checked as the last letter of the detector Serial Number (refer to 3.2.2.j).

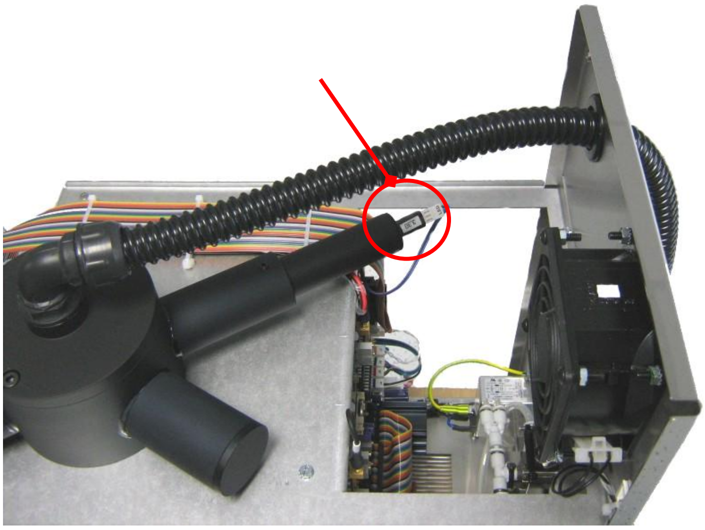
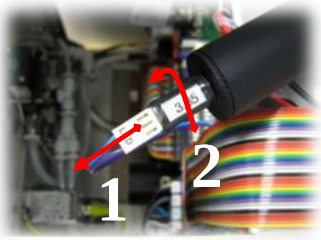


Figure 5-3: The Light Source

a) Switch the detector on and switch off the Light Source by pressing the 💡 button.

b) Switch the detector off and remove the detector cover.



c) Disconnect the light source connector (step 1) and unscrew the light source cap (step 2). Note: For reinstallation, the white connector cannot be placed in the wrong way (mechanical protection).

d) Screw the new light source cap. Do **not** plug in the light source connector now.

e) Switch on the detector and switch on the lamp by pressing the 💡 button. Do **not** plug in the light source connector now.

f) Check the voltage at VLED test point on ALIM004v2 Board (Figure 5-4). Do **not** plug in the light source connector now.

The ground should be one of the fixing screws that attach the board to the detector.

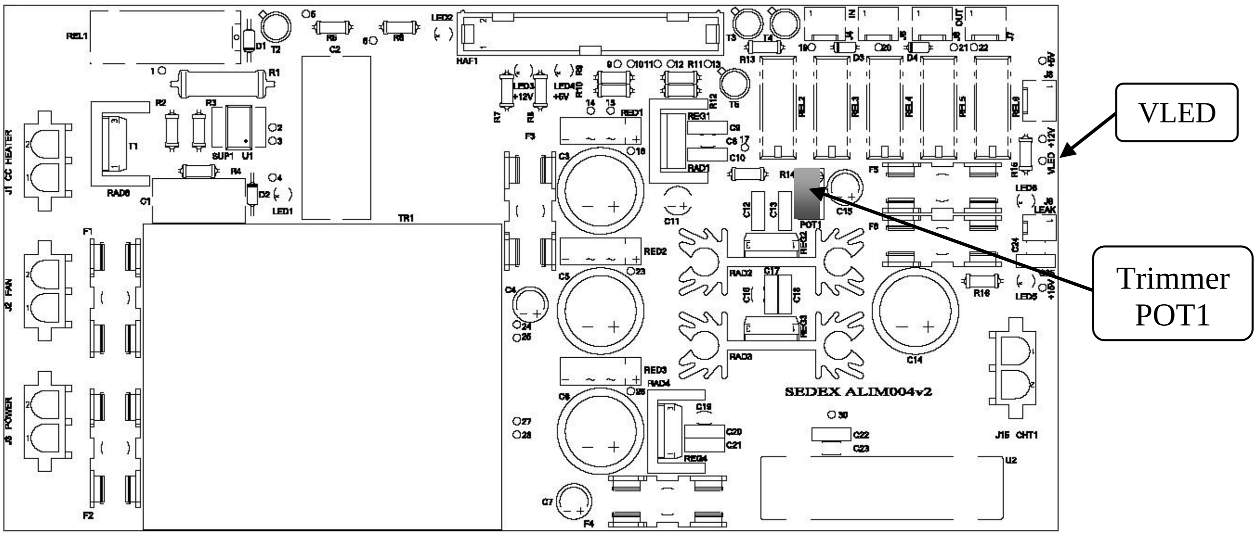


Figure 5-4: The ALIM004V2 Board Lamp Setting

g) The voltage at VLED must be set to the value that is provided with the new L.E.D. with a precision of 2 digits (example: + 3.40V). If the voltage is incorrect, adjust with trimmer POT1. Do **not** plug in the light source connector now.

h) Switch off the light source by pressing the 💡 button and plug in the white light source connector.

i) Switch on the light source by pressing the 💡 button.

j) Wait for 5 minutes and check the VLED voltage. If necessary, readjust the voltage with trimmer POT1.

☞ Note: **If the potential is not set accurately, the lifetime of the L.E.D. will be adversely affected.**

k) In the *LED menu* (Section 3.2.2.j), access the "Reset time elapsed" line, and press OK to reset the elapsed time counter for the L.E.D.

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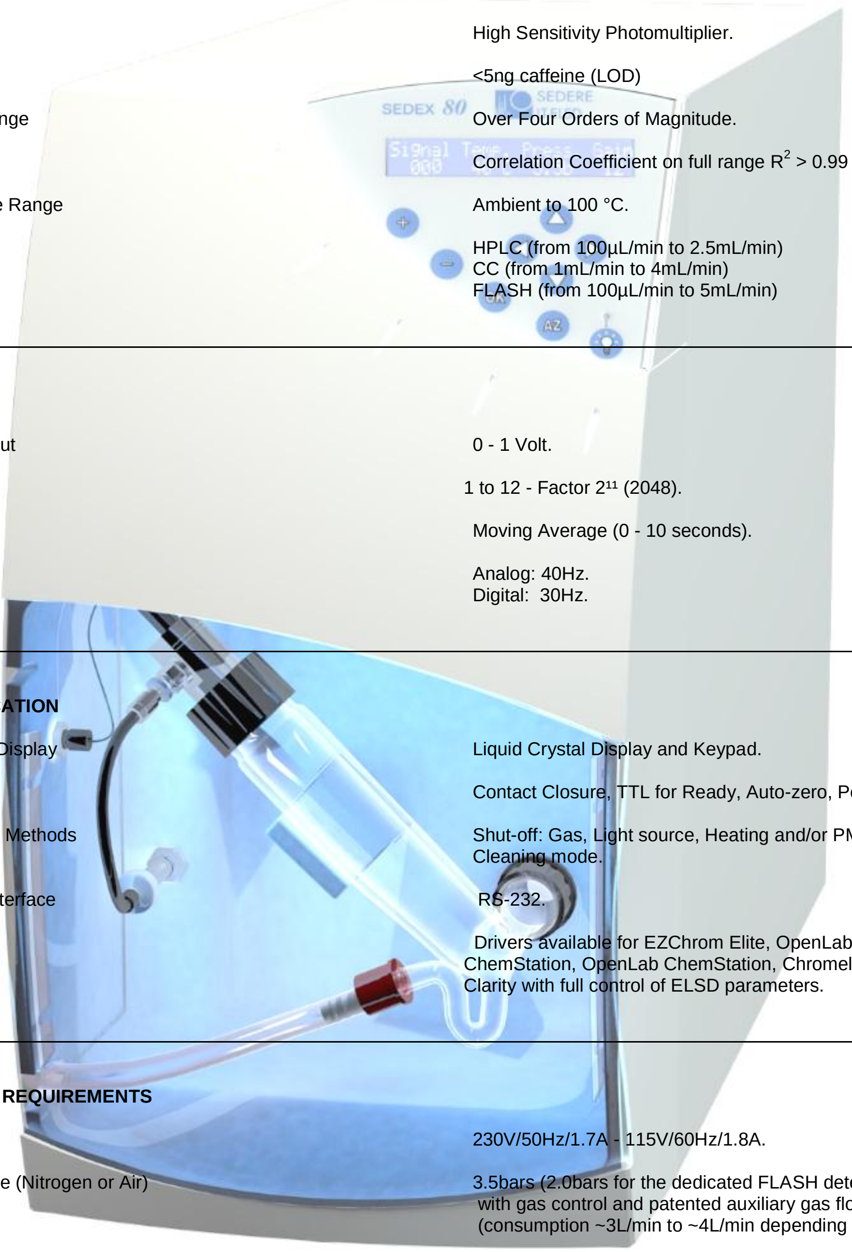
Appendix 1: Specifications

Technical Specifications



COMPONENTS

Light Source	Selected High Efficiency Blue LED Elapsed-time Counter.
Detection	High Sensitivity Photomultiplier.
Sensitivity	<5ng caffeine (LOD)
Dynamic Range	Over Four Orders of Magnitude.
Linearity	Correlation Coefficient on full range $R^2 > 0.99$
Temperature Range	Ambient to 100 °C.
Nebulizer	HPLC (from 100µL/min to 2.5mL/min) CC (from 1mL/min to 4mL/min) FLASH (from 100µL/min to 5mL/min)

DATA

Analog Output	0 - 1 Volt.
Gain Setting	1 to 12 - Factor 2^{11} (2048).
Filter	Moving Average (0 - 10 seconds).
Data Rate	Analog: 40Hz. Digital: 30Hz.

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 Cleaning mode.
Computer Interface	RS-232.
Software	Drivers available for EZChrom Elite, OpenLab EZChrom, ChemStation, OpenLab ChemStation, Chromeleon, Xcalibur and Clarity with full control of ELSD parameters.

EXTERNAL REQUIREMENTS

Power	230V/50Hz/1.7A - 115V/60Hz/1.8A.
Gas Pressure (Nitrogen or Air)	3.5bars (2.0bars for the dedicated FLASH detector model) with gas control and patented auxiliary gas flow (consumption ~3L/min to ~4L/min depending of the Nebulizer)
Dimensions	550mm (22”) D X 250mm (10”) W X 480mm (18”) H
Weight	16Kg (35lbs).

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Appendix 2: Spare Parts List

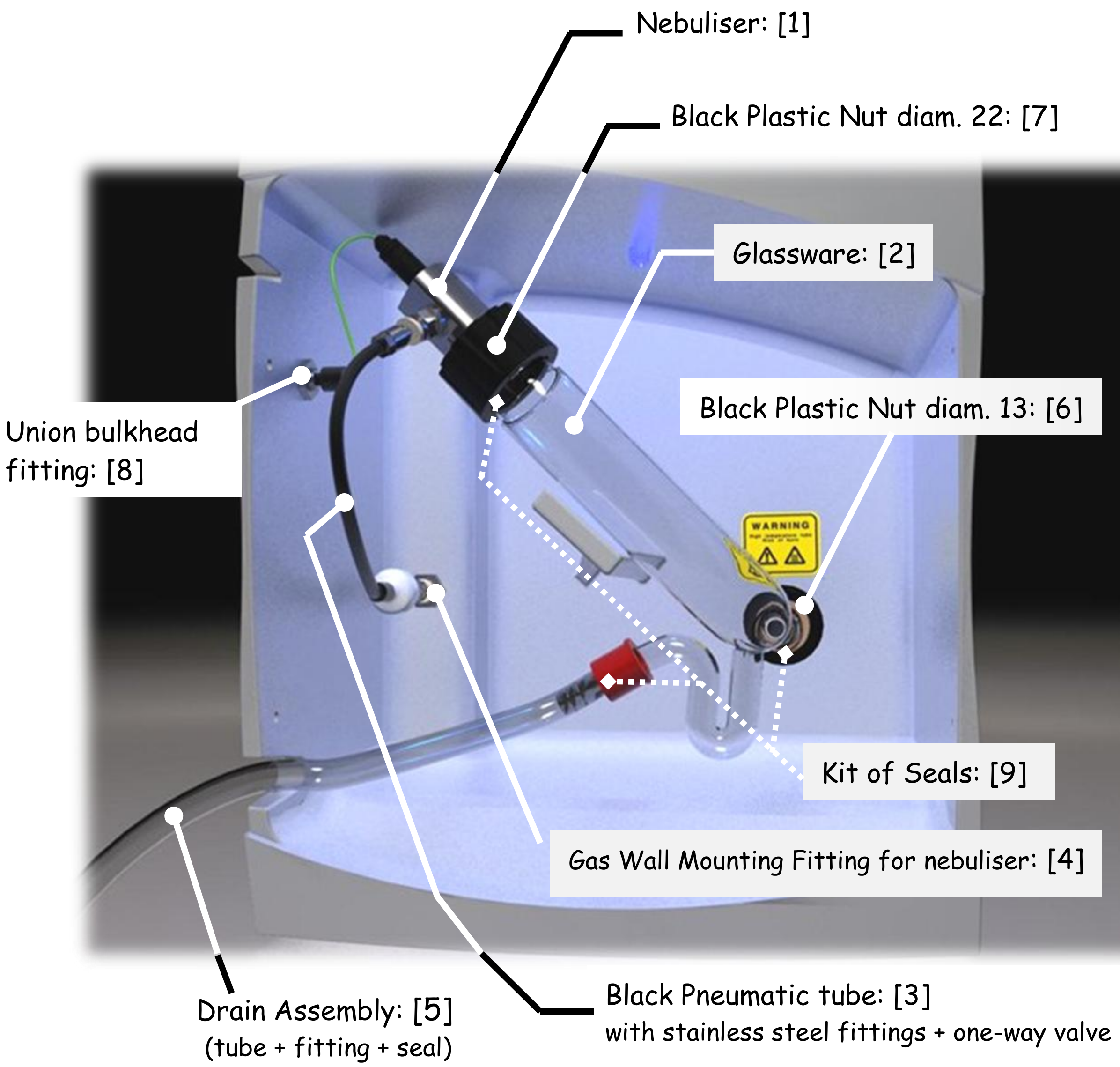

Figure A2-1: Identification of Spare Parts

Part	Item	Part Number
Nebulizer	[1]	
HPLC Nebulizer		80003
Combinatorial Chemistry Nebulizer		80008
FLASH Nebulizer		80005
Glassware Nebulization Chamber	[2]	
Glass Nebulization Chamber		85009-10
Gas		
Gas Regulator with Filter and Manometer		45100
Cartridge 0,01µm for Gas Regulator		45007
Black Pneumatic tube for Nebulizer *(with fitting + one-way valve)*	**[3]**	85016
Gas Wall Mounting Fitting		
4 mm Diameter (nebuliser)	**[4]**	55014
6 mm Diameter (rear panel)		45023
Gas Tube (6 mm diameter) /meter		45020
Drain / Exhaust		
Drain Tube Assembly *(includes stainless steel fitting and PTFE seal)*	**[5]**	45200
Black exhaust tube (2m50) (rear panel)		55016
Cables		
Signal Cable		55400
AutoZero Cable		55500
Ext. Events Cable		754006
RS232 Cable		75092
Fuses		
Main Power Fuses (set of 2 fuses)		45300-3.15T
Fuse kit (Main Supply + Internal Fuses		85072
Miscellaneous		
Black Plastic Nut for Nebulization Chamber		
13 mm Diameter	**[6]**	45700-13
30 mm Diameter	**[7]**	45700-22
Union Bulkhead Fitting	**[8]**	85002
Kit of seals for Glassware	**[9]**	85071
Starting kit (refer to Table 2-2 / Precise detector main voltage)		85097 -115 or -230
Transparent Black front panel shield window		85012

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

A3.1 Overview

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 A3-2 and the installation of the unit is described in Chapter 2.
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 A3.3.
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 A3.4.
P.Q. protocols demonstrate that the instrument performs according to sensitivity using Caffeine as a test compound.

The overall *Detector Performance report* is presented in Section A3.5.

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

☞ Note: **Before starting S.O.P for new instruments or after storage, the following operations must be performed:**
- Set gas pressure to 3.5bars (51psi); or 2.0bar (29psi) only for Flash model.
- 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.

S.E.D.E.R.E recommends that maintenance visits and SOP certification should be performed once a year by a S.E.D.E.R.E -authorized representative.

A3.2 Installation Qualification (I.Q.) Checklist

Model Number: SEDEX ELSD 80LT Instrument Serial Number: ___________

Location of the Detector: ____

a) Has the instrument been delivered as ordered (e.g. according to the U.R.S. or purchase order)? YES [ ] NO [ ]

b) Has the instrument been checked and verified as undamaged? YES [ ] NO [ ]

c) Has the required documentation been supplied? Is it of correct issue and appropriately identified by Model Number, Serial Number and Date? YES [ ] NO [ ]

d) Have details of all services and utilities required to operate the instrument been provided? YES [ ] NO [ ]

e) Have methods and instructions for user maintenance been provided along with contact points for service and spare parts? YES [ ] NO [ ]

f) 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 [ ]

g) 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 ______________________________________

A3.3 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 photomultiplier tube (PMT).

Model Number: SEDEX ELSD 80LT Instrument Serial Number: ____________

Location of the Detector: ____

a) Power up the detector

b) 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.

c) Apply air or nitrogen pressure of 3.5bar (51psi) for both the HPLC and CC detectors models; or at a pressure of 2.0bar (29psi) for the Flash detector model, monitored by a regulator and checked pressure gauge.

HPLC and CC Detectors Models:
The display should read 3.5bar +/- 0.1bar (51psi)

FLASH Detector Model:
The display should read 2.0bar +/- 0.1bar (29psi)

PRESSURE VALUE _______

PASS [   ] FAIL [   ]

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

The display should read 50°C +/- 1°C.

DISPLAY TEMPERATURE _______

PASS [   ] FAIL [   ]

e) Set the *Gain* to *1*, *Filter* to *1s* as described in section 3.2.2.e,
Temperature order to *50°* and allow *air or nitrogen gas* flow through the instrument at a pressure of *3.5bar (51psi)* for both the HPLC and CC detectors models; or at a pressure of 2.0bar (29psi) for the Flash detector model,
Power on the light source,
Set "*Offset After Autozero*" to *0mV* as described in section 3.2.2.d.,
Wait for temperature stabilisation.

Autozero the detector. Raise the Gain to 12, monitor the signal for 5 seconds and enter the value below. This value corresponds to the Stray Light value.

Observed Value ________mV

HPLC and CC Detectors Models:
The observed stray light readings should be in the range of 100 – 150mV

FLASH Detector Model:
The observed stray light readings should be in the range of 2 – 20mV

PASS [ ] FAIL [ ]

f) This test is performed in the same conditions as the Stray Light value determination (previous test).

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)	Segment	Noise (mV)	Segment	Noise (mV)
1		3		5	
2		4		6	

Minimum Value: _______ Maximum Value: ________ Mean Value: ________

The mean ASTM noise value should be less than 2mV.
PASS [ ] FAIL [ ].

g) 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 2.0mV.
PASS [ ] FAIL [ ]

> ☞ Note: **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 ____________________________________

A3.4 The Performance Qualification (P.Q) Protocol

This procedure checks the proper operation of the detector with respect to sensitivity and repetability using Caffeine as a test compound.

A3.4.1 is the Performance Qualification Protocol dedicated to HPLC or CC detector models whereas,
A3.4.2 is the Performance Qualification Protocol dedicated to FLASH detector model.

A3.4.1Performance Qualification (P.Q) Protocol for HPLC/CC Models

Model Number: SEDEX ELSD 80LT 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.

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

    Temperature : 40^{o}C
    Gain : 12
    Noise filtering : 1second
    Gas pressure : 3.5bar (51psi)
    Offset after AZ. : 0mv
    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 : Caffeine
    Flow rate : 1mL/min
    Mobile phase : 100% fresh HPLC water

2) Autozero the detector.

3) Make three (3) 20µL injections of 5ppm (5µg/mL) caffeine standard (for a full 20µL loop injection, deliver 100µL of the standard each time to ensure that the loop is completely filled with the standard). The average peak height (mV) should be:

At least 70mV under test conditions

Peak height 1 _______ mV
Peak height 2 _______ mV
Peak height 3 _______ mV
Average Peak height ______ mV

PASS [ ] FAIL [ ]

4) Set gain 6 and then autozero the detector.

5) Make six (6) 20µL injections of a 250ppm (250µg/mL) caffeine standard at gain 6. The repeatability should be:

3.0% RSD maximum.

Peak Area 1: ___________
Peak Area 2: ___________
Peak Area 3: ___________
Peak Area 4: ___________
Peak Area 5: ___________
Peak Area 6: ___________

Avg. Peak Area: __________

% Round Standard Deviation (RSD): ______%

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 ____________________________________

A3.4.2 Performance Qualification (P.Q) Protocol for FLASH Model

Model Number: SEDEX ELSD 80LT 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.

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

Temperature : 40°C
Gain : 12
Noise filtering : 1second
Gas pressure : 2.0bar (29psi)
Offset after AZ. : 0mv
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 : Caffeine
Flow rate : 1mL/min (into the detector)
Mobile phase : 100% fresh HPLC water

2) Autozero the detector.

3) Make six (6) 20µL injections of a 250ppm (250µg/mL) caffeine standard at gain 12.

4) The average peak height (mV) should be:

At least 450mV under test conditions

Peak height 1 _______ mV
Peak height 2 _______ mV
Peak height 3 _______ mV
Peak height 4 _______ mV
Peak height 5 _______ mV
Peak height 6 _______ mV

Average Peak height ______ mV

PASS [ ] FAIL [ ]

5) The repeatability should be:

3.0% RSD maximum.

Peak Area 1: ___________
Peak Area 2: ___________
Peak Area 3: ___________
Peak Area 4: ___________
Peak Area 5: ___________
Peak Area 6: ___________

Avg. Peak Area: __________

% Round Standard Deviation (RSD): ______%

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 _____________________________________

A3.5 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 ELSD 80LT 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 ____

Customer Signature ____

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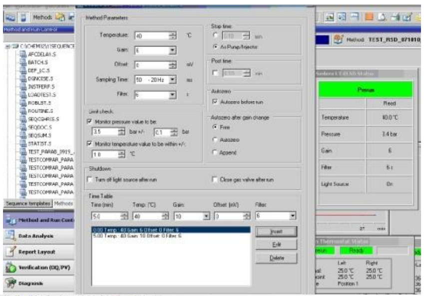
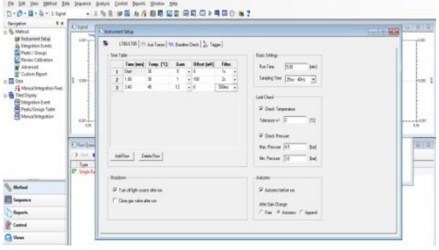
Appendix 4: Chromatography Software Drivers

SEDEX LT-ELSD™ detectors can be controlled by the keyboard however some Drivers can be proposed for a full control without need for an A/D convertor. Users can also program a time table for gain/filter/temperature changes as a function of time and make LT-ELSD™ detectors fully integrated in many HPLC equipments.

- **Driver for ChemStation™**
  Part number: 85089
- **Driver for OpenLab ChemStation™**
  Part number: 85094
- **Driver for EZChrom *Elite*™**
  Part number: 85090
- **Driver for OpenLab EZChrom™**
  Part number: 85096
- **Driver for Xcalibur™**
  Part number: 85093
- **Driver for Chromeleon™**
  Part number: 85095
  Only 6.8 SR10/SR11 with Timebase/Class 3
- **Driver for Clarity™**
  The driver is included in Clarity™ software



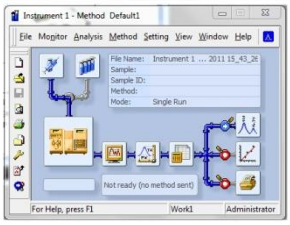



Instrument 1 - Device Monitor

Sedere 8x Detector 1 (SN 100280320G)
Not Ready (Method has not been sent)

Temperature 20 °C
Pressure 3,5 bar
Gain 6
Lamp ON
Gas OFF
Input
Lamp time 5018 h

For Help, press F1


Instrument 1 - Method Default1

File Name: Instrument 1 … 2011 15_43_26
Sample:
Sample ID:
Method:
Mode: Single Run

Not ready (no method sent)

For Help, press F1
Work1
Administrator

When using Drivers, the AutoZero cable may have to be connected to a "Start" information (Blue wire to a "Start" and white wire to a "GND/Ground" signal) on a controlling device (e.g. autosampler). In this mode, the detector doesn't proceed to an Autozero, it uses it as a signal synchronization for the driver.

Not using the Autozero connection for a "Start" information will impair the synchronization of the Signal and may not generate the final report and/or impair the retention time reproducibility.

For Minimum requirements, Installation and Operation with these drivers, you can directly refer to their respective Operator's Manuals. Please note that the Communication is an option and requires to be enabled prior to use any drivers. This option can be activated at any time (contact your local distributor).

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