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1 About this manual

This manual describes the Low Temperature Evaporative Light Scattering Detector C-650 and provides all information required for its safe operation and to maintain it in good working order. It is addressed to laboratory personnel and operators in particular.

Read this manual carefully before installing and running your system and note the safety precautions in chapter 2 in particular. Store the manual in the immediate vicinity of the device, so that it can be consulted at any time.

No technical modifications may be made to the device without the prior written agreement of BUCHI. Unauthorized modifications may affect the system safety or result in accidents. Technical data are subject to change without notice.

NOTE
The symbols pertaining to safety are explained in chapter 2.

This manual is copyright. Information from it may not be reproduced, distributed or used for competitive purposes, nor made available to third parties. The manufacture of any component with the aid of this manual without prior written agreement is also prohibited.

The English manual is the original language Version B and serves as basis for all translations into other languages. If you need another language version of this manual, you can download available versions at www.buchi.com.

1.1 Abbreviations

ELSD: Evaporative Light Scattering Detector
Fig: Figure
TTL: Time to live (electrical signal)
2 Safety

This chapter points out the safety concept of the device and contains general rules of behavior and warnings from hazards concerning the use of the product.

The safety of users and personnel can only be ensured if these safety instructions and the safety related warnings in the individual chapters are strictly observed and followed. Therefore, the manual must always be available to all persons performing the tasks described herein.

2.1 User qualification

The device may only be used by laboratory personnel and other persons who on account of training or professional experience have an overview of the dangers which can develop when operating the device.

Personnel without this training or persons who are currently being trained require careful instruction. The present Operation Manual serves as the basis for this.

2.2 Proper use

The device has been designed and built for the use in laboratories. The ELSD C-650 is a stand-alone device. The C-650 is designed to detect compounds in the eluent from Preparative Chromatography.

2.3 Improper use

Applications not mentioned above are improper. Also applications which do not comply with the technical data are considered improper.

⚠️ DANGER

During any improper use, the effectiveness of the protection systems can be affected.

- Avoid any improper use of the devices!

The operator bears the sole risk for any damages caused by such improper use.

The following uses are expressly forbidden:

- Use of the device in rooms which require ex-protected devices.
- Use on samples which can explode or inflame (e.g.: explosives, etc.) due to shock, friction, heat or spark formation.
2.4 Safety warnings and safety signs

DANGER, WARNING, CAUTION and NOTICE are standardized signal words for identifying levels of hazard seriousness of risks related to personal injury and property damage. All signal words, which are related to personal injury are accompanied by the general safety sign.

For your safety it is important to read and fully understand the table below with the different signal words and their definitions!

<table>
<thead>
<tr>
<th>Sign</th>
<th>Signal word</th>
<th>Definition</th>
<th>Risk level</th>
</tr>
</thead>
<tbody>
<tr>
<td>⚠️</td>
<td>DANGER</td>
<td>Indicates a hazardous situation which, if not avoided, will result in death or serious injury.</td>
<td>★★★★★</td>
</tr>
<tr>
<td>⚠️</td>
<td>WARNING</td>
<td>Indicates a hazardous situation which, if not avoided, could result in death or serious injury.</td>
<td>★★★☆☆</td>
</tr>
<tr>
<td>⚠️</td>
<td>CAUTION</td>
<td>Indicates a hazardous situation which, if not avoided, may result in minor or moderate injury.</td>
<td>★★☆☆☆</td>
</tr>
<tr>
<td>no</td>
<td>NOTICE</td>
<td>Indicates possible property damage, but no practices related to personal injury.</td>
<td>★☆☆☆☆ (property damage only)</td>
</tr>
</tbody>
</table>

Supplementary safety information symbols may be placed in a rectangular panel on the left to the signal word and the supplementary text (see example below).

<table>
<thead>
<tr>
<th>Space for supplementary safety information symbols.</th>
<th>⚠️ SIGNAL WORD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source of hazard.</td>
<td></td>
</tr>
<tr>
<td>• List of measures to avoid the herein described, hazard or hazardous situation.</td>
<td></td>
</tr>
<tr>
<td>• …</td>
<td></td>
</tr>
<tr>
<td>• …</td>
<td></td>
</tr>
</tbody>
</table>

Table of supplementary safety information symbols

The reference list below incorporates all safety information symbols used in this manual and their meaning.

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Meaning</th>
</tr>
</thead>
<tbody>
<tr>
<td>⚠️</td>
<td>General warning</td>
</tr>
<tr>
<td>⚠️ ⚠️</td>
<td>Electrical hazard</td>
</tr>
<tr>
<td>⚠️ ⚠️</td>
<td>Heavy weight, avoid overexertion</td>
</tr>
<tr>
<td>Symbol</td>
<td>Meaning</td>
</tr>
<tr>
<td>--------</td>
<td>---------</td>
</tr>
<tr>
<td><img src="image" alt="Harmful to life-forms" /></td>
<td>Harmful to life-forms</td>
</tr>
<tr>
<td><img src="image" alt="Pinch point. Mechanical hazard." /></td>
<td>Pinch point. Mechanical hazard.</td>
</tr>
<tr>
<td><img src="image" alt="Fire hazard" /></td>
<td>Fire hazard</td>
</tr>
<tr>
<td><img src="image" alt="Hot item, hot surface" /></td>
<td>Hot item, hot surface</td>
</tr>
<tr>
<td><img src="image" alt="Device damage" /></td>
<td>Device damage</td>
</tr>
<tr>
<td><img src="image" alt="Inhalation of substances" /></td>
<td>Inhalation of substances</td>
</tr>
<tr>
<td><img src="image" alt="Chemical burns by corrosives" /></td>
<td>Chemical burns by corrosives</td>
</tr>
<tr>
<td><img src="image" alt="Wear laboratory coat" /></td>
<td>Wear laboratory coat</td>
</tr>
<tr>
<td><img src="image" alt="Wear protective goggles" /></td>
<td>Wear protective goggles</td>
</tr>
<tr>
<td><img src="image" alt="Wear protective gloves" /></td>
<td>Wear protective gloves</td>
</tr>
<tr>
<td><img src="image" alt="Heavy weight, lifting requires more than one person" /></td>
<td>Heavy weight, lifting requires more than one person</td>
</tr>
</tbody>
</table>
Additional user information

Paragraphs starting with NOTE transport helpful information for working with the device/software or its supplementaries. NOTES are not related to any kind of hazard or damage (see following example).

**NOTE**
*Useful tips for the easy operation of the device/software.*

## 2.5 Product safety

The ELSD is designed and built in accordance with state-of-the-art technology. Nevertheless, risks to users, property, and the environment can arise when the device is used carelessly or improperly.

The manufacturer has determined residual dangers emanating from the device

- if the device is operated by insufficiently trained personnel.
- if the device is not operated according to its proper use.

Appropriate warnings in this manual serve to make the user alert to these residual dangers.

### 2.5.1 General hazards

Pay attention to the following safety notices:

<table>
<thead>
<tr>
<th><strong>DANGER</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Death or serious injuries by use in explosive environments.</td>
</tr>
<tr>
<td>- Do not store or operate the device in explosive environments.</td>
</tr>
<tr>
<td>- Provide sufficient ventilation and make sure to directly withdraw fumes</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>WARNING</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Death or serious burns by flammable vapors, flames and sparks.</td>
</tr>
<tr>
<td>- Remove all sources of flammable vapors, flames and sparks</td>
</tr>
<tr>
<td>- Do not store flammable chemicals in the vicinity of the device</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>NOTICE</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Risk of device damage by liquids or mechanical shocks.</td>
</tr>
<tr>
<td>- Do not spill liquids over the device or its components</td>
</tr>
<tr>
<td>- Do not move the device when it is loaded with sample liquid</td>
</tr>
<tr>
<td>- Do not drop the device or its components</td>
</tr>
<tr>
<td>- Keep external vibrations away from the device</td>
</tr>
<tr>
<td>- Safely attach the device to the bench in earthquake prone regions</td>
</tr>
<tr>
<td>- Do not operate the device without the safety shield installed</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>NOTICE</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Risk of device damage by wrong mains supply.</td>
</tr>
<tr>
<td>- External mains supply must meet the voltage given on the type plate</td>
</tr>
<tr>
<td>- Check for sufficient grounding</td>
</tr>
</tbody>
</table>
2.5.2 Warning labels on housing and assemblies

The following warning sticker(s) can be found on the housing or assemblies of the C-650:

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Meaning</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image" alt="Hot item, hot surface" /></td>
<td>Hot item, hot surface</td>
<td>Sticker/label, located at the heating block</td>
</tr>
</tbody>
</table>

2.5.3 Personal protective equipment

Always wear personal protective equipment such as protective eye goggles, protective clothing and gloves. The personal protective equipment must meet all requirements of the supplementary data sheets for the chemicals used.

---

2.6 General safety rules

2.6.1 Responsibility of the operator

The head of laboratory is responsible for training his personnel.

The operator shall inform the manufacturer without delay of any safety-related incidents which might occur during operation of the device. Legal regulations, such as local, state and federal laws applying to the device must be strictly followed.

2.6.2 Duty of maintenance and care

The operator is responsible for ensuring that the device is operated in proper condition only, and that maintenance, service, and repair jobs are performed with care and on schedule, and by authorized personnel only.

2.6.3 Spare parts to be used

Use only genuine consumables and genuine spare parts for maintenance to assure good system performance and reliability. Any modifications to the spare parts used are only allowed with the prior written permission of the manufacturer.

2.6.4 Modifications

Modifications to the device are only permitted after prior consultation with and with the written approval of the manufacturer. Modifications and upgrades shall only be carried out by an authorized BUCHI technical engineer. The manufacturer will decline any claim resulting from unauthorized modifications.
3  Technical data

This chapter introduces the device specifications. It contains the scope of delivery, technical data, requirements and performance data.

3.1  Scope of delivery

Check the scope of delivery according to the order number.

NOTE
For detailed information on the listed products, see www.buchi.com or contact your local dealer.

3.1.1  Standard device

Table 3.1: Standard device

<table>
<thead>
<tr>
<th>Product</th>
<th>Order number</th>
</tr>
</thead>
<tbody>
<tr>
<td>ELS Detector C-650 (230 V/50 Hz)</td>
<td>11059105</td>
</tr>
<tr>
<td>ELS Detector C-650 (115 V/60 Hz)</td>
<td>11059106</td>
</tr>
</tbody>
</table>

3.1.2  Standard accessories

Table 3.2: Standard accessories

<table>
<thead>
<tr>
<th>Product</th>
<th>Order number</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-650 signal cable</td>
<td>11059100</td>
</tr>
<tr>
<td>C-650 autozero cable</td>
<td>11059165</td>
</tr>
<tr>
<td>C-650 external signal cable</td>
<td>11059229</td>
</tr>
<tr>
<td>C-650 RS232 cable</td>
<td>11059275</td>
</tr>
<tr>
<td>Gas tubing, 6 mm, 2 m + 1 m</td>
<td>11059276</td>
</tr>
<tr>
<td>Set of fuses (115 V)</td>
<td>11059172</td>
</tr>
<tr>
<td>Set of fuses (230 V)</td>
<td>11059173</td>
</tr>
</tbody>
</table>
### 3.1.3 Optional accessories

Table 3.3: Optional accessories

<table>
<thead>
<tr>
<th>Product</th>
<th>Order number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow splitter</td>
<td>11059007</td>
</tr>
</tbody>
</table>

### 3.2 Technical data overview

Table 3.4: Technical data

<table>
<thead>
<tr>
<th>Dimensions (L×H×D)</th>
<th>250×450×550 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Connection voltage</td>
<td>115 VAC (±10 %)/60 Hz/1.8 A</td>
</tr>
<tr>
<td></td>
<td>230 VAC (±10 %)/50 Hz/1.7 A</td>
</tr>
<tr>
<td>Weight</td>
<td>18.5 kg</td>
</tr>
<tr>
<td>Overvoltage category</td>
<td>II</td>
</tr>
<tr>
<td>Pollution degree</td>
<td>2</td>
</tr>
<tr>
<td>Environmental conditions</td>
<td>For indoor use only</td>
</tr>
<tr>
<td>Temperature</td>
<td>5–40 °C</td>
</tr>
<tr>
<td>Altitude</td>
<td>Up to 2000 m above sea level</td>
</tr>
<tr>
<td>Humidity</td>
<td>Maximum relative humidity 80 % for temperatures up to 31 °C, and then linearly decreasing to 50 % at 40 °C</td>
</tr>
<tr>
<td>Detection</td>
<td>High sensitivity photomultiplier</td>
</tr>
<tr>
<td>Light source</td>
<td>Selected high efficiency blue LED</td>
</tr>
<tr>
<td>Temperature range</td>
<td>Ambient to 100 °C</td>
</tr>
<tr>
<td>Gas flow control</td>
<td>Manual and computer controlled (power down) nebulization gas flow and patented auxiliary gas flow</td>
</tr>
<tr>
<td>Gas consumption</td>
<td>Less than 5 L/min</td>
</tr>
<tr>
<td>Eluent flow rate</td>
<td>0.1 mL/min to 5 mL/min</td>
</tr>
<tr>
<td>Device control</td>
<td>Microprocessor with stand alone manual keypad or PC control</td>
</tr>
<tr>
<td>Operating parameters control</td>
<td>Liquid crystal digital panel</td>
</tr>
<tr>
<td>Signal drift</td>
<td>Less than 2 mV/30 min</td>
</tr>
<tr>
<td>Signal output</td>
<td>0–1 V (analog), RS-232 (digital)</td>
</tr>
<tr>
<td>Inputs</td>
<td>Remote Autozero (contact closure and TTL)</td>
</tr>
<tr>
<td></td>
<td>Remote Power down Mode (contact closure and TTL)</td>
</tr>
<tr>
<td>Power Down Mode</td>
<td>General</td>
</tr>
<tr>
<td></td>
<td>Standby</td>
</tr>
<tr>
<td></td>
<td>Cleaning</td>
</tr>
<tr>
<td>Zero control</td>
<td>Manual Autozero and remote Autozero</td>
</tr>
</tbody>
</table>
4 Description of function

This chapter explains the basic principle of the device, shows how it is structured and gives a functional description of the assemblies.

4.1 Overview over the device

The C-650 is designed to detect compounds in the eluent from Preparative Chromatography. It is capable of monitoring eluent flow rates from 0.1 mL/min to 5 mL/min. Evaporative Light-Scattering Detection 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.

The detector is controlled via the keypad and digital LCD display on the front panel or by RS-232. The analog signal output can be sent to a recorder, an integrator or a data station such as BUCHI SepacoreRecord or SepacoreControl Software.

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

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.

Nebulization --> Evaporation --> Detection

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 device is presented below.

![Cross-sectional view of the detector](image)

4.2.1 **Nebulization**

The eluent from the chromatograph is nebulized by the inlet gas (typically air or nitrogen). At the outlet of the nebulizer, the aerosol travels through a chamber. Large droplets in the aerosol are drawn to a siphon while the fine mist travels to the evaporation tube. The overall design of the nebulizer is shown in Fig 4.3 and the nebulization chamber is shown in Fig 4.4.

The Flash Chromatography Nebulizer has a flow rate of 0.1 mL/min –5 mL/min and a back pressure of 4 bar (1 mL/min) by the use of water.
1. Liquid inlet
2. Coloured seal
3. Ring
4. Gas inlet

Fig 4.3: Design of the nebulizer

Fig 4.4: Glassware chamber
4.2.2 Evaporation of the solvent

A heated tube is used to evaporate the solvent. The exit of the heated tube leads directly into the detection chamber.

In liquid chromatography, water and organic solvents with low boiling points are typically employed (e.g. CH$_3$OH, CHCl$_3$, CH$_3$CN). A typical mobile phase for a reverse phase separation using Evaporative Light-Scattering Detection might be CH$_3$OH/H$_2$O (60/40) while a typical mobile phase for normal phase separation might be C$_6$H$_{14}$/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$_4$OH, (C$_2$H$_5$)$_3$N, NH$_4$ Acetate, NH$_4$ Formate, HCOOH, CH$_3$COOH and CF$_3$COOH.

4.2.3 Detection

The carrier gas transports the microparticles from the heating tube into the detection chamber (Fig 4.5).

The detector chamber contains a Light Emitting Diode (LED) and a photomultiplier that is positioned at an angle of 100° with respect to the light beam (Fig 4.5). When the carrier gas contains microparticles, the light is scattered and is detected by the off-axis photomultiplier.

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 the following equation.

\[ I = k m^b \]

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 parameters (e.g., the temperature and the nature of the mobile phase).

An inlet to provide additional gas is located immediately before the detector chamber to provide a concentric shield for the carrier gas. This serves to eliminate diffusion of the carrier gas and eliminate contamination of the detection chamber.

4.3 Eluent flow splitting

The C-650 detector is designed to work with flow rates from 0.1 to 5 mL/min. In preparative chromatography applications, the flow rate is mostly higher than 5 mL/min. The BUCHI preparative chromatography systems can deliver flow rates up to 250 mL/min. For preparative applications, the eluent flow rate coming out of the chromatography column must be split into 2 different flow rates:

- One part (usually about 1 mL/min) is directed to the C-650 and this part of the sample is destructed by the evaporation and diffraction process.
- The other part is directed to the fraction collector to collect the detected compounds.

The working principle is shown below:

![Fig 4.6: Working principle of the flow splitting](image-url)
5 Putting into operation

This chapter describes how the device is installed and gives instructions on initial startup.

NOTE
Inspect the device for damages during unpacking. If necessary, prepare a status report immediately to inform the postal company, railway company or transportation company. Keep the original packaging for future transportation.

5.1 Installation site

Put the device on a stable, horizontal surface. Consider the maximum product dimensions and weight. Obtain the environmental conditions as described in section 3.2 Technical data overview.

Installation prerequisites:
- Make sure, the device is not subject to drafts or significant temperature changes.
- Do not place it near air conditioning vents, windows, ovens, etc.
- Make sure that access to the power supply to disconnect the device must be kept at any time.
- Make sure to leave a distance of at least 20 cm between the device and another object or wall.
- Place the detector close to the outlet of the column to minimize extra column band broadening which would reduce the resolution of the chromatographic separation.

WARNING
Death or serious injuries by use in explosive environments.
- Do not operate the device in explosive environments
- Do not operate the device with explosive gas mixtures
- Before operation, check all gas connections for correct installation
- Directly withdraw released gases and gaseous substances by sufficient ventilation

CAUTION
Risk of minor or moderate injury by heavy weight of the device.
- Consult a second person to lift the device
- Do not drop the device
- Place the device on a stable, even and vibration-free surface
- Keep limbs out of crushing zone
- To transport the device, place your hands under the device

NOTICE
Risk of device damage by liquids or mechanical shocks.
- Do not spill liquids over the device or its components
- Do not move the device when it is loaded with sample liquid
- Keep external vibrations away from the device
- Safely attach the device to the bench in earthquake prone regions

NOTE
As a destructive detector, the C-650 should be the last one in the flow path or can be used with a splitter.
5.2 Electrical connections

NOTE
External connections and extension lines must be provided with a grounded conductor lead (3-pole couplings, cord or plug equipment). All used power cords must meet the input power requirements.

It is recommended that all components of the Flash 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.

<table>
<thead>
<tr>
<th>NOTICE</th>
</tr>
</thead>
</table>

Risk of device damage by wrong mains supply.
- External mains supply must meet the voltage given on the type plate
- Check for sufficient grounding

All electrical connections are made via the supply panel (Fig 5.7) on rear panel.

Connecting the Recorder/Integrator:
If a recorder or integrator is employed, connect the recorder input to the 1 V output terminal on the rear panel of the detector (Fig 5.7) and to the appropriate socket on the recorder/integrator.

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 (Fig 5.7) and to the appropriate socket on the controlling device (e.g. autosampler, pump, etc.).

Refer to section 6.2.2 to operate external autozero signal.

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 back panel of the detector (Fig 5.7) 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 lamp 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 2.0 bar.

NOTE
The electrical consumption of the controlled device must not exceed 20 mA under 12 VDC.
The blue cables are contact closure “input” cables that are used to power the unit down (see section 6.5) via a signal from an external device to the detector.

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.
Do not turn on the power at this time.
The power cord of this detector 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.

5.3 Gas supply
A supply of filtered, oil-free clean gas (e.g. \( \text{N}_2 \) or air if aqueous mobile phase) 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 unreactive with analysis).

The gas supply should include a pressure gauge. The pressure should be set at 2 bar. The unit is connected to the gas supply via the 6.0 mm plastic tubing (supplied) using the fitting on the upper left corner of the supply panel on the rear of the detector.

![Supply panel diagram](image)
After Parafilm is removed from the detector gas inlet, the tubing should be cut and firmly inserted into the fitting as shown below.

![Figure 5.8: Inserting the gas inlet tube](image1)

1. Cutting the tube square
2. Inserting the tube into the fitting until it bottoms
3. Pull the tube to check engagement of the grab

Two pieces of tubing are provided. If you are using the device 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 Fig 5.9.

![Figure 5.9: Removing the glass inlet tube](image2)

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.
## Exhaust venting and drain requirements

**WARNING**

Risk of toxic fume inhalation.
- Always route the black exhaust tube from the detector into a ventilated fume hood or exhaust vent.

The exhaust and drain 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 black exhaust tube from the detector can be cut and should be directed into a ventilated fume hood or exhaust vent.

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 cause condensation traps and could lead to poor results.

If gas from the 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 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 your local regulatory authorities for health and safety requirements.

The black exhaust line on the back of the unit 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 hood). Verify that no tube damage or inappropriate installation could allow a gas leak in laboratory.

Install the vent tube so that it cannot become blocked or bent, or restrict the gas flow from the detector to the 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 PVC tubing can be fitted over the exhaust tubing.
5.5 Installing the nebulizer/glass chamber assembly

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

NOTE
When installing the transparent black front cover, first fix its right side, and then push its left side. When removing the front cover, pull only its left side.
The installed nebulizer/glass chamber assembly is shown in Fig 5.10.

![Diagram of nebulizer/glass chamber assembly](image)

1. Siphon overflow
2. Gas one-way valve
3. Gas inlet fitting
4. Nebulizer
5. Nut
6. Glass chamber
7. Nut

Fig 5.10: Installing the nebulizer/glass chamber assembly

To install the glass chamber:
Remove the Parafilm™ from all detector openings and from the nebulization cell.
Position the glass chamber as shown in Fig 5.10 and tighten the black nut at the bottom. The glass chamber should be flush with the back wall as shown in Fig 5.11.

![Image of glass chamber](image)

Fig 5.11 Fixing the tip of the glass chamber
Use the large black nut to position the nebulizer on the glass chamber.

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 CH₂Cl₂), 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.

5.5.1 Connecting the siphon overflow

Attach a Tygon 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 is not immersed in the collected liquid. Make sure that liquid waste container complies with the solvent nature.

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

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

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

A drain tube with a bend or immersing the liquid will create pressure fluctuations in the detector and will lead to poor results.

If the solvent that you are using is not compatible with Tygon (e.g. THF), use a piece of PTFE 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.

5.5.2 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. Refer to Fig 5.10.

NOTE
Make sure you are using the correct black gas tubing orientation, where the white one-way valve is at the lower end (near the gas arrival).
5.6 Installing the flow splitter

5.6.1 Connecting the flow splitter

The flow splitter comes as complete kit (article number 11059007) and has to be mounted inside the detector as shown below:

![Flow splitter diagram]

1. Fasten the splitter with the fastening screw ① to the left side of the ELSD.
2. Connect the following tubings to the splitter kit:
   - Outlet ⑥ from the column
   - Connection tubing ④ to the fraction collector

**NOTE**
*Do not connect the splitter to the ELSD nebulizer before having adjusted the flow rate to 1 mL/min.*

Recommendation:
For preparative chromatography applications connect the column to the splitter after the conditioning step.

5.6.2 Adjusting the flow rate of the ELSD splitter

1. Remove the protecting sticker from the flow adjustment knob and keep the predefined adjustment.
2. Start the pumping system with the appropriate eluent for your application.
3. Check the flow rate. If the flow rate is not adjusted at 1 mL/min, adjust the flow rate with the flow adjustment knob until the delivered flow rate represents about 1 drop/second, which is about 1 mL/min (±10 %). Measure the flow rate using a graduated cylinder.
flask. This adjustment requires only a small rotation of the knob. Too big rotation could generate too high back pressure (about half clockwise rotation).

NOTE
The flow splitter is delivered equipped with a black restriction capillary having a 0.10 mm ID. This configuration is especially suited for normal phase separations using solvents with low viscosity. For reversed phase applications using solvents with viscosities higher than 0.5 cP, this capillary must be replaced with the violet restriction capillary having a 0.15 mm ID (Art No 11059075).

5.6.3 Connecting to the ELS nebulizer
1. Stop the pumping system.
2. Connect the splitter to the ELSD nebulizer using the provided union fitting (Art No 044302) (position ③ in Fig 5.12).

5.7 Powering up the device
Place the ON/OFF switch to the OFF position and plug the device 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 Software option information (activated or not) and then will present the signal (which should be 0 mV 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. Install the black front panel cover, first fix its right side, and then push its left side.

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.

5.8 Control panel
The Control Panel (Fig 5.13) includes a digital display and a number of buttons that are used to enter data.

5.8.1 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 switched on, the display will show a greeting message for a few seconds which includes the version number and date.
After the detector has completed the initialization procedures, the Status screen (Fig 5.14) will be presented. The signal should be close to zero.

![Status screen](image)

Fig 5.14: Status screen

The user interface is provided via a series of screens that are described in section 5.8.2. Some screens present information about the device status and cannot be edited by the user (e.g. the Status screen), while other screens (e.g. the Temperature/Gain screen, Fig 5.16) 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.**

- **Validates the value of the parameter that you have edited.**

- **ZERO**
  - Sets the present signal for the detector to zero.

- **LED**
  - Is used to power up the LED in the detector. When the LED is lit, the keyboard LED immediately above the button will be illuminated.

- **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.**

**5.8.2 User interface**

The Status screen (Fig 5.14) is the default screen and is presented after initialization of the detector. In addition, it will be automatically presented again if you have accessed another screen and have not made any keystroke within a few seconds.

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

**Status screen**

The Status screen (Fig 5.14) presents the present conditions of the detector. This screen cannot be edited, but the desired offset can be set via the Offset screen (Fig 5.15), the temperature and...
gain can be set via the Temp/Gain screen (Fig 5.16) and the pressure units can be selected via the Noise Filter/Pressure Unit screen (Fig 5.18).

Temperature value blinks if desired temperature is not reached and stable. The pressure value blinks if the gas pressure is lower than 2.0 bar.

When the button is pressed; the Offset screen (Fig 5.15), which is used to select the desired offset is displayed.

Offset screen

![Offset screen](image)

*Fig 5.15: Offset*

To increase the offset value, click on the key. 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 20 mV/sec.

Once you have set the desired offset, press the button to validate the new value.

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

Press the button to access the Temp/Gain screen (Fig 5.16).

Temperature/Gain screen

![Temperature/Gain screen](image)

*Fig 5.16: Temp/Gain screen*

The Temp/Gain screen is used to set the desired Temperature and Gain. When the screen is accessed, the cursor is on the Temp setting. This setting can be changed with the and buttons and validated by the button. The temperature range is 20 to 100 °C.
When the detector is 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 device and normal.

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 higher.

When you press the ▲ button, the Gain field can be edited. The gain range is from 1 to 12, each increment of one unit increases the gain by a factor of 2 (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 is 1 to 2048. After you have validated the desired gain setting, press the ▲ button again to display the Autozero Offset screen (Fig 5.17).

Autozero offset screen

![Autozero offset screen](image)

Fig 5.17: Autozero offset screen

This screen is used to allow the signal to reach the desired value when performing an autozero (by keyboard or external contact closure).

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 ▼ button.

After you have set the desired autozero offset, press the ▲ button to display the Noise Filter/Pressure Unit screen (Fig 5.18).
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 \texttt{+} or \texttt{-} keys, you change the filtering strength within the following range:

- “NO”: no filtering.
- 0.5 s: 0.5 second moving average filtering.
- 1…10 s: 1 to 10 seconds moving average filtering.

\textbf{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 1 sec., corresponding to a peak width of approximately 2 sec. at half-height. User manual section 6.4.5 details Filter optimization.

If you have changed the value, press \texttt{确认} to validate it before you press the \texttt{保存} 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 \texttt{+} or \texttt{-} key, and validated by the \texttt{确认} key. When you press the \texttt{保存} button, the LED screen (Fig 5.19) will be displayed.

LED screen

The LED screen is used to turn the light source On/Off and is equivalent to the Light Source button on the control panel. Use the \texttt{+} button followed by the \texttt{-} button to turn the LED on and the
button followed by the button to turn it off.

The # hours field indicates the number of hours that the LED has been in use. The lifetime of the LED is approximately 5000 h. 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 LED on the keyboard blinks.

NOTE
When you press the button, the Gas Valve screen (Fig 5.20) will be displayed.

Gas valve screen

[Image of Gas Valve screen]

Fig 5.20: 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 .

When you press the button, the power down screen (Fig 5.21) will be displayed.

Power Down screen

The Power Down screen (Fig 5.21) 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.

[Image of Power Down screen]

Fig 5.21: Power Down screen
The three options provided for external shutdown are summarized in the following table:

<table>
<thead>
<tr>
<th>Mode</th>
<th>Photomultiplier</th>
<th>Lamp</th>
<th>Heating</th>
<th>Gas flow</th>
</tr>
</thead>
<tbody>
<tr>
<td>General</td>
<td>Off</td>
<td>Off</td>
<td>Off</td>
<td>Off</td>
</tr>
<tr>
<td>Standby</td>
<td>Off</td>
<td>Off</td>
<td>On</td>
<td>Off</td>
</tr>
<tr>
<td>Cleaning</td>
<td>Off</td>
<td>Off</td>
<td>On</td>
<td>On</td>
</tr>
</tbody>
</table>

To select the desired Power Down mode, use the \[\text{or} \] key to access the desired mode and then press \(\text{to validate the selection.}\)

**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:

External event cable power down contact closure: 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.

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 \(\) to put the detector in power down mode.

**NOTE**
*To leave the power down mode, release the contact closure if power down has been activated by external event 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 (Fig 5.22).*

**Total Lifetime Elapsed screen**

![Image of Total Lifetime Elapsed screen]

*Fig 5.22: 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 (Fig 5.23) will be displayed.
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 (Fig 5.24) will be displayed.

Firmware Screen

This information screen presents the firmware Version B and date, 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 (Fig 5.25) will be presented.
Factory Menu Code Screen

Fig 5.25: Factory Menu Code screen

The Factory Menu Code screen is used by the service engineer to access a variety of service features and is not designed to be employed by the user.

5.9 Initial test procedures

5.9.1 Preliminary activities

This section presents a protocol that can be used to ensure that the detector is working in the proper way.

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

Before starting the tests for a new device or after storage, flush the detector with water at a flow of 1 mL/min for at least 15 minutes.

The following activities should be performed:

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

Access the Temperature/Gain screen, set the temperature to 50 °C and press . View the Status screen and verify that the temperature is rising to the setpoint on the Status screen. Temperature regulation is stable when the Temperature value stops blinking.
When the detector is 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 device and should not be a concern.

Provide gas to the detector and adjust the pressure to 2 bar (29 psi). If the pressure is less than 1.5 bar (21 psi), the pressure value blinks, indicating that the detector is not ready.

Make certain that the pressure of gas supplied to the detector is less that 4.5 bar (67 psi). If the pressure increases above 4.5 bar (67 psi), 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 the same.

Press the „ZERO“ button. The signal should be close to zero and remain constant.

Set the noise filtering to 1 second (refer to Fig 5.18).

### 5.9.2 Electronic noise test

To determine the electronic noise:

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

Make sure that the siphon is filled and the nebulizer inlet connector is blocked with Parafilm™ to avoid a Venturi effect.

Set gas pressure to 2 bar (29 psi) and temperature to 50 °C. Wait for stable temperature.

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

Record the level and autozero the detector again.

### 5.9.3 Background noise (stray light) test

To determine the background noise:

Do not turn on the pump (no solvent flow).

Make sure that the siphon is filled and the nebulizer inlet connector is blocked with Parafilm™ to avoid a Venturi effect.

Set gas pressure to 2 bar (29 psi) and temperature to 50 °C.

Switch on the light source.

Change the Gain to 1.

Set the offset to 0 mV.

Set the offset after Autozero to 0 mV (refer to Fig 5.17).

Autozero the detector.

Change the Gain to 12.

Wait 15 minutes for stabilization and record the signal level. The expected level is typically 100 mV to 150 mV. The exact value will vary slightly and small deviations should not be a cause for concern.
5.9.4 **Solvent noise test**

To determine the solvent noise:

Ensure that the gas is flowing at 2 bar (29 psi), the temperature is set to 50 °C and stable and
the pump is switched off.

Switch on the light source and set the gain to 12 and monitor the signal. Do not autozero the
detector. The signal may be negative.

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 1 mL/min.

Monitor the baseline for a few minutes.

If water is used as the solvent, the signal should be less than 10 mV. Higher values could be ob-
served if non-HPLC grade water (with a higher non-volatile residue) is used.

If an organic solvent is used, the signal should be less than 200 mV.

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 of approximately less than 100 mV).

**NOTE**

*The purity of the solvent is critical for a low background noise. The sensitivity is inversely propor-
tional to the solvent noise.*

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

*If the device fails the Solvent Noise test, it is most likely due to an impurity in the solvent rather
than a fault with the device. If changing the solvent source does not solve the problem, it may be
necessary to decontaminate the device as described in section 8.7.2 or clean the nebulizer as
described in section 8.5.*

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, CH₃COOH, CF₃COOH, NH₄ Formate, NH₄, Acetate, (C₂H₅)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).
5.9.5 Column noise test

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

To determine the column noise:

Turn off the pump and connect the column.

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.

Set the gain to 12 and monitor the baseline for a few minutes. A suitable column will provide a baseline that is 20–50 mV above the solvent baseline.

NOTE
If the mobile phase contains acidic modifiers (e.g. CF₃COOH), disconnect the detector and wash the Chromatography system for 12 h before starting to analyze unknown samples. This wash should be performed after the column noise test is completed, but need not to be performed after each analysis.
6  Operation

This chapter describes the operations that should be performed on a routine basis when you want to collect chromatographic data using the C-650 and gives examples of typical device applications and instructions on how to operate the device properly and safely.

6.1  Preparing the device for operation

<table>
<thead>
<tr>
<th>CAUTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>Risk of fire due to flammable liquids!</td>
</tr>
<tr>
<td>• Check all tubes for correct connection before starting the device.</td>
</tr>
<tr>
<td>• Make sure all tubes are in good condition (no cracks or cuts).</td>
</tr>
</tbody>
</table>

To prepare the detector for operation:

- Power up the detector by pressing the switch on the rear panel.
- Open the gas distribution valve and set the pressure to 2 bar (29 psi). The pressure is indicated on the Status screen.
- Ensure that the overflow siphon for the nebulization chamber contains sufficient liquid. If necessary, pump few mL of solvent through the device to fill the siphon.
- 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.
- Start the mobile phase flow through the device and allow the overall system to operate for at least 15 minutes to ensure that all components are equilibrated and a stable baseline is obtained.
- Switch on the LED light source of the detector by pressing the LED button on the control panel.

NOTE
The Solvent Noise test (section 5.9.4) and the Column Noise test (section 5.9.5) 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.

6.2  Auto-zeroing the detector

6.2.1  Manual

To auto-zero the detector:

- 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.
- Press the “ZERO” button. The detector will be automatically auto-zeroed at this point.
- 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.

NOTE
If you change the gain selection, make sure that the detector is auto-zeroed again before taking any measurement.
6.2.2 External

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 1 second long, with a maximum current of 20 mA at 5 V. If a TTL signal is used please make sure to use the correct polarity identified on the cable.

6.3 Routine operation of the detector

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

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

<table>
<thead>
<tr>
<th>CAUTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>Risk of minor or moderate injury by hot acid fumes.</td>
</tr>
<tr>
<td>• Do not operate the system with faulty parts</td>
</tr>
<tr>
<td>• Check device setup for proper sealing before use</td>
</tr>
<tr>
<td>• Do not inhale process fumes</td>
</tr>
<tr>
<td>• Operate the device inside an active fume hood</td>
</tr>
<tr>
<td>• Do not move the device or parts of it during operation</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>NOTICE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Risk of device damage by leaking liquids.</td>
</tr>
<tr>
<td>• Do not operate the system with faulty parts</td>
</tr>
<tr>
<td>• Check device setup for proper sealing before use</td>
</tr>
</tbody>
</table>

• 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 5.4).
• Ensure that the siphon is filled with liquid at all times. The overflow from the siphon should be collected in a suitable container.
• Make sure that all flow connections are properly tight. In case of any leak, switch off the pump immediately and remove the liquid.
• Never exceed a gas pressure of greater than 4.5 bar (67 psi).
• Avoid the use of solvent or compounds that could corrode the detector. The mobile phase is in contact with glass and PTFE tubing and the evaporation tube is made of stainless steel.

6.4 Optimizing the performance

6.4.1 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 1 mL/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 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 (Fig 6.26) [the conditions for the separation are – Column: ODS KromasilTM (5 µm, 30×2.1 mm), Sample: 4 µL (10 mg/L) Caffeine]. Eluent: Water, 0.2 mL/min, temperature as indicated). 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, ambient temperature and the nature of the mobile phase.

### 6.4.2 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 1 ppm 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).
The mobile phase should not contain non-volatile solvent modifiers. Volatile solvent modifiers (e.g., CHOOH, CH₃COOH, CF₃COOH, NH₂ Formate, NH₄ Acetate, (C₂H₅)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 PTFE, 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 3.5 bar (51 psi) may have to be adjusted in order to optimize the background noise and so Signal-to-Noise ratio.*

### 6.4.3 Sample pretreatment

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

### 6.4.4 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 in the detector, which may lead to an increase in the noise.

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

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 5.9.5) to obtain the baseline signal value corresponding to this column.
6.4.5 Optimizing the noise filter

The Digital Filter (Fig 5.18) 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:

<table>
<thead>
<tr>
<th>Peak width at 50% (second)</th>
<th>Proposed filter (second)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;1</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
</tr>
<tr>
<td>6</td>
<td>4</td>
</tr>
<tr>
<td>8</td>
<td>6</td>
</tr>
<tr>
<td>&gt;10</td>
<td>8 and higher</td>
</tr>
</tbody>
</table>

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 using the SOP test (injection of 5 ppm caffeine at gain 12). Peak width at half-height is 2.5 seconds.

<table>
<thead>
<tr>
<th></th>
<th>Filter 0 s</th>
<th>Filter 1 s</th>
<th>Filter 2 s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Signal height</td>
<td>124 mV</td>
<td>122 mV</td>
<td>110 mV</td>
</tr>
<tr>
<td>Noise (ASTM)</td>
<td>3.2 mV</td>
<td>1.1 mV</td>
<td>0.7 mV</td>
</tr>
<tr>
<td>Peak width (at 50 % height)</td>
<td>2.5 s</td>
<td>2.5 s</td>
<td>2.8 s</td>
</tr>
<tr>
<td>S/N</td>
<td>37</td>
<td>110</td>
<td>157</td>
</tr>
</tbody>
</table>

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

6.5 Powering down and shutting down the detector

If desired, some or all functions of the device can be powered down at the end of an automated series of analyses. These power down features are described in detail in Fig 5.21.

To shut down the device:

- Turn off the pump.
- Allow the nebulization gas to flow through the detector for few minutes (30 min is recommended) to drain the evaporation tube and detection chamber.
- Turn off the power to the detector (if desired).

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

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.

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

This chapter gives instructions on all maintenance work to be performed in order to keep the device in good working condition.

<table>
<thead>
<tr>
<th>WARNING</th>
</tr>
</thead>
<tbody>
<tr>
<td>Death or serious burns by electric current at cleaning.</td>
</tr>
<tr>
<td>• Switch off the device</td>
</tr>
<tr>
<td>• Disconnect the power cord and prevent unintentional restart</td>
</tr>
<tr>
<td>• Wait until the device is completely dry before reconnecting to mains</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>NOTICE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Risk of housing and device damage by liquids and detergents.</td>
</tr>
<tr>
<td>• Do not spill liquids over the device or parts of it</td>
</tr>
<tr>
<td>• Wipe off any liquids instantly</td>
</tr>
<tr>
<td>• Use ethanol or soapy water as detergent only</td>
</tr>
</tbody>
</table>

The ELSD C-650 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 intervention by the operator.

The following general recommendations are proposed:

• Maintain the detector in a clean laboratory environment.
• If the device 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 device.
• 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 chromatography 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:

• Let the mobile phase or solvent (recommendation: ethanol) flow to flush particles which could remain in the detector.
• Eventually increase temperature in order to dissolve possible deposit.
• Stop the mobile phase flowing but let the gas flow at least 30 min to dry to avoid particles deposit.
• Stop the gas flow.
• Shut down the detector.
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 device in routine operation. If the suggestions provided in this chapter do not remedy the problem, contact your local distributor.

The LED used as the Light Source has a long but finite lifetime (~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 LED on keyboard will blink.

7.1 Customer service

Only authorised service personnel are allowed to perform repair work on the device. These persons have a comprehensive technical training and knowledge of possible dangers which might arise from the device.

Addresses of official BUCHI customer service offices are given on the BUCHI website under www.buchi.com. If malfunctions occur on your device or you have technical questions or application problems, contact one of these offices.

The customer service offers the following:

• Spare part delivery
• Repairs
• Technical advice
8 Troubleshooting

8.1 General troubleshooting information

The ELSD C-650 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.

Do not disassemble the nebulizer. Disassembling the nebulizer will destroy it and this will void the warranty.

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

If the digital display does not illuminate when the detector is powered up, turn the unit off and inspect the main fuses. If necessary, replace the fuses with some of the same rating as the original ones for all 115 V and 230 V units. The fuses are located inside the main power module on the rear panel (Fig 5.7). A set of replacement fuses is delivered in the starting kit.

If the fuses are not blown or if the replacement fuses blow up again, contact your local distributor.

8.2 Initial troubleshooting activities

- Make sure that the device and all components of the detector are grounded to a true ground.
- Check to ensure that the liquid level in the siphon is appropriate, and there is no liquid pooling close to the evaporation tube inlet.
- Check that the gas pressure is sufficient and stable. The selected pressure for most applications is 2 bar (29 psi). Pressure above 4.5 bar (67 psi) can damage the pressure sensor. The gas filter should be clean and in place. Only use gas free of oil residue.
8.3 Perform the noise test

Repeat the tests described in section 5.9 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 5.9.2), Background Noise test (section 5.9.3) and Solvent Noise test (section 5.9.4) provide results that are similar to that obtained when the unit was initially installed, but the Column Noise test (section 5.9.5) 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).

8.4 Specific detector troubleshooting

- The mist from the nebulizer should be homogeneous. If it is not homogeneous, the nebulizer, the needle or the PTFE 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 8.5.

Do not disassemble the nebulizer. Disassembling the nebulizer will destroy it and this will void the warranty.

- 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 back pressure loop instead of a column. Alternatively, the LED 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 8.5. 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 might be too low or dead volumes in capillary connections may cause peak broadening.

- 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.

- 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 solvent at the rate of 2 mL/min, using a gas pressure of 2 bar (29 psi).
8.5 **Nebulizer cleaning and replacement procedures**

If the mist of the nebulizer is not homogeneous, the nebulizer, the needle or the PTFE tube may be obstructed. To remove the obstruction, pump a solvent (recommendation: ethanol) that can dissolve the foreign material. As an alternative, the nebulizer can be placed in an ultrasonic bath to dissolve the foreign material.

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

The nebulizer rear part is a strategic setting which mustn’t be dismounted for any reason. In case the user has removed it, the only solution is to perform a nebulizer replacement.

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

In case the nebulizer does not produce a spray and the liquid is drawn directly to the siphon even if the pressure display is 2 bar, make sure you are using the correct black gas tube orientation 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 Fig 5.10.

To remove the nebulizer from the device:

- Switch off the pump and the ELSD detector.
- Remove the black front panel cover. Pull its left side.
- Disconnect the nebulizer liquid inlet from the column.
- Disconnect the gas inlet from the nebulizer by pushing on the white inlet (refer to Fig 5.10 for details).
- 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. The black nut which maintains the nebulizer on the glass cell and its seal should be removed from the nebulizer.
- Remove the gas inlet quick fitting and the black plastic nut to avoid damaging the seals with the cleaning solvent.

![Fig 8.28: Removing parts from the nebulizer](image)

To clean the nebulizer:
- Fill an ultrasonic bath with water. Fill a beaker (50 or 100 mL) with approximately 2 cm 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.
- Place the nebulizer vertically in the beaker 2 cm 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.

![Fig 8.29: Cleaning the nebulizer](image)

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

To re-install the nebulizer or replace it by a new one or another nebulizer model:
- For re-installing the nebulizer after a nebulizer cleaning, re-install the gas inlet quick fitting and the black plastic nut with its seal.
- Reverse the order of previous steps (nebulizer removing). 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).
- 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.
- Install the black front panel cover, first fix its right side, and then push its left side.
- 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 orientation, 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.

Fig 8.30: Installing the nebulizer/glass chamber assembly

8.6 Gas flow problems

In case of gas flow problems, please contact the BUCHI Customer Service.

8.7 Cleaning and decontamination

8.7.1 Device cleaning

<table>
<thead>
<tr>
<th>CAUTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>Risk of minor burns by hot detector.</td>
</tr>
<tr>
<td>• Let the detector cool down safely for 15 minutes before cleaning.</td>
</tr>
</tbody>
</table>

- Switch the device off.
- Disconnect all connection cable (power cable, signal cable, autozero cable, RS-232 cable if any, device gas input and nebulizer tubing).
- 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.
8.7.2 **Device decontamination**

- Set the evaporation temperature to 100 °C and the gas pressure to 2 bar (29 psi).

Pump the appropriate solvent through the system at the rate of 1 mL/min. The solvent will be determined by the nature of the samples that were previously analyzed by 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 device. Maintain the flow and temperature during 3 hours at least.

- 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.

8.8 **Light source exchange**

In case the light source needs to be exchanged, please contact the BUCHI Customer Service.
9 Shutdown, storage, transport and disposal

This chapter instructs how to shut down the device, how to pack it for storage or transport, and specifies the storage and shipping conditions.

9.1 Preparing the device for storage and transport

To prepare the device for storage and transport, unplug the power cord and remove all glass parts from the device.

9.2 Storage and transport

Store the device at a dry place. Store and transport the device in its original packaging.

**WARNING**

Death or serious poisoning by contact or incorporation of harmful substances.
- Wear safety goggles
- Wear safety gloves
- Wear a laboratory coat
- Flush the device and clean all accessories thoroughly to remove possibly dangerous substances
- Do not clean dusty parts with compressed air
- Store the device and its accessories at a dry place in its original packaging

**CAUTION**

Risk of minor or moderate injury by heavy weight of the device.
- Consult a second person to lift the device
- Do not drop the device or its transport box
- Place the device on a stable, even and vibration-free surface
- Keep limbs out of crushing zone
Spare parts

This chapter lists spare parts, accessories, and options including their ordering information.

Order the spare parts from BUCHI. Always state the product designation and the part number when ordering spare parts.

Use only genuine BUCHI consumables and genuine spare parts for maintenance and repair to assure good system performance and reliability. Any modifications to the spare parts used are only allowed with the prior written permission of the manufacturer.

<table>
<thead>
<tr>
<th>Product</th>
<th>Order number</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-650 Flash nebulizer</td>
<td>11059162</td>
</tr>
<tr>
<td>C-650 Glass nebulization chamber</td>
<td>11059164</td>
</tr>
<tr>
<td>Black plastic nut for nebulization chamber diam. 13 mm</td>
<td>11059241</td>
</tr>
<tr>
<td>Black plastic nut for nebulization chamber diam. 30 mm</td>
<td>11059242</td>
</tr>
<tr>
<td>Black pneumatic tube with steel fitting and one way gas valve for nebulizer</td>
<td>11059227</td>
</tr>
<tr>
<td>Complete drain tube assembly</td>
<td>11059239</td>
</tr>
</tbody>
</table>
11 Declarations and requirements

11.1 FCC requirements (for USA and Canada)

English:

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to both Part 15 of the FCC Rules and the radio interference regulations of the Canadian Department of Communications. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment.

This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Français:

Cet appareil a été testé et s’est avéré conforme aux limites prévues pour les appareils numériques de classe A et à la partie 15 des réglementations FCC ainsi qu’à la réglementation des interférences radio du Canadian Department of Communications. Ces limites sont destinées à fournir une protection adéquate contre les interférences néfastes lorsque l’appareil est utilisé dans un environnement commercial.

Cet appareil génère, utilise et peut irradier une énergie à fréquence radioélectrique, il est en outre susceptible d’engendrer des interférences avec les communications radio, s’il n’est pas installé et utilisé conformément aux instructions du mode d’emploi. L'utilisation de cet appareil dans les zones résidentielles peut causer des interférences néfastes, auquel cas l'exploitant sera amené à prendre les dispositions utiles pour palier aux interférences à ses propres frais.
Declaration of conformity
Konformitätserklärung
Déclaration de conformité
Dichiarazione di conformità
Declaración de conformidad

BUCHI Labortechnik AG
Meierseggstrasse 40
9230 Flawil
SWITZERLAND

Declares, that the product / Erklärt, dass das Produkt / Déclare par la présente que le produit / Dichiara che il prodotto / Declara que el producto:

ELS Detector C-650

complies with the requirements of the European Directives / den Anforderungen der Richtlinien / est conforme aux exigences des directives européennes / soddisfa i requisiti delle norme europee / cumple los requerimientos de las Directivas Europeas:

2006/95/EEC  low voltage directive
2004/108/EEC  EMC directive

and is in accordance with the following standards / und den folgenden Normen entspricht / ainsi qu'aux normes suivantes / ed è conforme ai seguenti standard / y está conforme a losestandares siguientes:

EN 61010-1:2001
Safety requirements for electrical equipment for measurement, control and laboratory use - Part 1 : General requirements.

EN 61326-1:2013
(Electrical equipment for measurement, control and laboratory use. EMC Requirements: General requirements.)

Technical file responsible H. P. Gohn, Quality Manager and authorized EU representative
Meierseggstrasse 40, CH-9230 Flawil

Flawil, August 18th 2015

Christian Zwicky  Erich Koller
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