Agilent 1260 Infinity II
Multi-Angle Light Scattering Detector

User Manual

Notices

Manual Part Number

Document No: D0029607 Rev. B

Edition 07/2023

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Safety Notices

CAUTION

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

WARNING

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

In this Guide….

This manual covers the Agilent 1260 Infinity II Multi-Angle Light Scattering Detector (G7885A).

Please read this manual thoroughly before using the instrument!

Thank you for purchasing this detector from Agilent Technologies. You have acquired a state-of-the-art instrument which enables you to perform unique experiments and the characterization of the most complex particle, polymer and protein samples.

This manual has been written for laboratory technicians who are responsible for operation and maintenance of laboratory equipment. It is assumed that the user of this manual is familiar with standard laboratory terminology. To ensure optimum operation conditions, please read the manual completely and carefully before using the detector. Use this product only in accordance with the guidelines and instructions listed.

This manual describes in detail:

- Characteristics
- Operation and Maintenance

Important:

- Do not use this instrument before completely understanding the full content of this manual.
- In the event that the instrument is loaned or transferred to a new owner please ensure this manual is passed on with the device.
- If this manual or any warning labels attached to the device become lost, get damaged or are unreadable, promptly obtain replacement documents/ labels from your nearest Agilent representative / distributor.
- To ensure safe operation at any time, please read the safety instructions before using the instrument.
- In case of any uncertainty how to operate this detector, consult your Agilent representative before starting to use this product.

NOTE

Agilent offers basic, advanced and repeated trainings to support the user.

Contents

1 Introduction

This chapter provides an overview of the principles and theory of Light Scattering detection. Furthermore, the most important functions as well as specifications of the detector are discussed.

After-Sales Service

If any problem occurs with this instrument, inspect it immediately and take appropriate corrective action as described in chapter "Troubleshooting" on page 53. If the problem persists or symptoms occur that are not described in this document, disconnect the mains voltage and contact your Agilent representative.

Maintenance

Each component and the entire system should be checked periodically to ensure that they function normally, otherwise the analysis data may not be precise and reliable anymore. Typically, a yearly service is recommended.

Agilent offers special maintenance and service contracts to ensure maximum lifetime and proper operation of the instrument during the entire life cycle. Before shipment from the factory, this device was thoroughly inspected and tested.

The result of the inspection is documented in the attached protocol. Referring to this protocol, please check the results of the hardware validation on a regular basis.

Safety Instructions

The user manual addresses persons who are qualified as chemical laboratory technicians or have completed a comparable vocational training.

The following knowledge is required:

- Fundamental understanding of liquid chromatography.
- Familiarity with substances that are used for laboratory experiments.
- Awareness regarding the health risks of chemicals.
- Participation during a training by Agilent or an authorized person.

If you do not belong to this or a comparable professional group, you may not perform the work described in this user manual under any circumstances. In this case, please contact your superior.

The manufacturer does not warrant for any damage or injury resulting from incorrect operation and maintenance, non-observance of the instructions in this manual. Prior to delivery, this instrument has been tested for safe usage.

- To ensure safe operation of the instrument, please read these safety instructions carefully before use and follow them always while operating the instrument.
- Observe all of the Warning and Caution sections listed below. They are extremely important for safety and to maintain a proper working device.

In this manual, Warning / Caution messages are always indicated using the following conventions:

WARNING **A warning alerts you to situations that could cause physical injury or death.**

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

CAUTION **A caution alerts you to situations that could cause loss of data, or damage of equipment.**

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

Site Requirements

WARNING

- Pay attention to the weight of the detectors. The detector weighs 16.5 kg (36.4 lbs). Use proper lifting techniques to avoid potential injuries. Ensure that you can handle the weight of the load.
- Consider the entire weight of the combined system including all the components. The lab table on which this detector is installed should be sufficiently strong to support the total weight of the whole LC system. It should be level, stable and have a depth of at least 600 mm. Otherwise, the instrument could tip over or fall off the table.
- Take measures to prevent the instrument from falling in the event of an earthquake or other disaster. Strong vibrations could cause the detector to fall over, resulting in injury and damage.
- All other equipment connected to this instrument must be approved to an appropriate safety standard and have reinforced insulation from the mains.

CAUTION

- Avoid installation sites that are exposed to corrosive gases, strong vibrations, or excessive dust. Otherwise, the performance and operability of the detector could be impaired, and its lifetime may be shortened.
- Never place any liquid on top of the detector.

Bench Space

The module dimensions and weight (see Table "Physical Specifications" on page 23) allow you to place the module on almost any desk or laboratory bench. It needs an additional 2.5 cm (1.0 inch) of space on either side and approximately 8 cm (3.1 inches) in the rear for air circulation and electric connections.

If the bench shall carry a complete LC system, make sure that the bench is designed to bear the weight of all modules.

NOTE The instrument is heavy and thus it is advisable to locate the system on a sturdy bench.

NOTE Allow additional bench space for a PC if the instrument is to be computer-controlled.

Power Considerations

WARNING

- Ground the instrument.
  To prevent electric shock in the event of an accident or electrical discharge, the detector must be connected to protective earth. This is also important to ensure stable operation and good data signals.
  Ensure that all connected devices are grounded via the mains connector as well.
- Do not place heavy objects onto the power cord and keep any hot objects away. This can damage the cord, resulting in fire, electrical shock, or malfunction. If the cord is damaged, immediately unplug the mains power cord and contact your Agilent representative.
- Never modify the supplied power cord. Do not bend it excessively or pull on it. Failure to do so could damage the cord, resulting in fire, electrical shock, or malfunction.
- Never replace the detachable power cord by an inadequately rated cord. This can result in fire, electrical shock, or malfunction.
- The power supply voltages and power consumption of this instrument are listed in table "Physical Specifications" on page 23. Connect the detector only to a power supply of the voltage indicated. Otherwise, fire or electric shock may occur. Ensure that the power supply voltage is stable and has sufficiently constant current capacity to operate all the components of the system. If not, the detector will not operate at its rated performance levels.
- If the power cord or its insulation gets damaged, unplug the cord and contact your Agilent representative for a replacement cable immediately.
- Only use mains connection cables admitted for use in your country. We strongly recommend to only use cables supplied by Agilent.
- In case of danger, immediately unplug the mains power cord either at the wall power outlet, the power strip or on the rear of the devices.

Solvent Information

WARNING

- Solvents used for Liquid Chromatography (LC) may be flammable and toxic. Therefore, the room where the detector is installed, should be well ventilated. Otherwise, solvent vapors could cause poisoning or may form an explosive atmosphere, ignite, and cause fire.
- LC can use large amounts of flammable organic solvents. Any open flame in the surrounding of this instrument is strictly prohibited. Do not use the detector in the same room with any other equipment that emits or could potentially emit sparks. Sparks could cause fire. Provide fire extinguishers for use in case of fire.
- Provide protective equipment near the instrument. If solvent gets into the eyes or on the skin, it must be flushed away immediately. Provide suitable equipment such as eye wash stations / safety showers as close as possible to the system. To avoid injuries due to spraying chemicals (e. g. by an insufficiently connected tubing) it is advisable to always wear eye protection when using the instrument.

Using the Instrument

WARNING

- Use this instrument ONLY in the manner specified in this manual and for the intended purpose. Using this detector in another manner or for any other purpose may impair the protection provided by the instrument and could cause accidents or unsafe conditions. The detector must not be used for clinical or medical purposes.
- When opening or closing the front cover, be careful not to pinch your fingers to avoid any injuries.
- Take thorough measures to prevent build-up of static electricity. Static electricity could result in fire or explosions.
- Always wear protective glasses and laboratory gloves when handling solvents or samples.
  Solvent getting into the eyes could cause blindness.
  If solvent gets into the eyes, flush immediately with large amounts of water, use eye wash stations, and seek medical treatment.
- Always wear laboratory gloves when handling any toxic, biologically infective samples or aggressive solvents.
- Do not use flammable sprays (hair sprays, insecticide sprays, etc.) near the instrument. They could ignite and cause fire.

CAUTION

- Before the instrument is being powered off for a longer period, its flow lines must be thoroughly flushed with filtered pure water to remove all remaining salt from the fluid path. Failure to do this and damage caused by improper use and precipitating buffer salts, or follow-up damages resulting from these actions, are specifically excluded from the warranty.
- Never let dry out the measuring cell. If the detector is disconnected from the flow path, then close the “flow inlet” and “flow outlet” with the delivered plugs.
- Never block the flow outlet of the detector when the inlet is connected to the flow path of the LC system. An overpressure will cause internal leaks and can damage the device.

NOTE

Before flushing the detector with water, make sure that the eluent (organic eluent with salt additives) used is miscible with water.

If necessary, first flush the detector with pure organic eluent and then switch to water or use a transition solvent!

For more information concerning immiscible solvents, refer to section "Miscibility Chart of Solvents" on page 61.

NOTE

For long time storage, flush the detector with a degassed mixture of pure water and 20 % methanol. Afterward, disconnect the detector from the flow path and close the inlet and outlet ports with the supplied plugs.

Maintaining the Instrument

WARNING

- Disconnect the solvent flow and unplug the instrument before inspection, maintenance, or replacement of any part. Otherwise, electrical shock, short-circuit accidents, or other damages could occur.
- Never remove the top cover of the detector while the unit is connected to the mains power supply. This could cause injury or malfunction of the instrument.
- Do not remove screws and open the main cover of the instrument. This can result in electrical shock, or malfunction of the instrument. The main cover does not need to be removed for routine maintenance, inspection and / or adjustment. Let your Agilent representative perform any repairs requiring removal of the instrument's cover.
- If the power cord plug gets dusty, remove the plug from the power outlet and wipe the dust away with a dry cloth. Dust could cause fire.
- Replace fuses only with fuses of proper type and capacity. Otherwise, the safety regulations are not fulfilled.
  If a fuse blows again after replacement, immediately unplug the mains power cord and consult your local Agilent representative.

NOTE

Replace parts only with original parts from Agilent. Any other parts may cause damage and malfunction.

Cleaning the Instrument

WARNING

- Never use pure alcohol or thinner solvent for cleaning the housing surface. They could cause fire and discoloration.
  To clean the surfaces of the closed housing, use a dry cloth and – if necessary – a bit of glass cleaner.
- Do not allow flammable and / or toxic solvents to accumulate.

Handling Leak and Waste

WARNING

- Liquids associated with this instrument may be classified as carcinogenic, biohazardous, flammable, or radioactive. Should these liquids be used, it is strongly recommended that this application be accomplished in an isolated environment designed for these types of materials in accordance with federal, state, and local regulatory laws, and in compliance with your company's chemical / hygiene plan in the event of a spill.
- Dispose the waste liquid properly in accordance with the instruction by your administrative department.
- Never let hazardous liquids reach the public sewage system.
- Before disposing of the equipment or sending it away for repair, you are required to decontaminate the device in a technically correct manner.
  All materials or fluids used for decontamination must be collected separately and disposed of properly.

Laser Warning

WARNING

The laser inside the instrument is a class 3B laser device based on the norm IEC 60825-1:2007. Do not open the detector. Hazardous laser radiation is emitted when open. Avoid direct exposure to the beam. Never bypass the safety guards to activate an opened device.

Static Electricity Warning

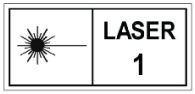
WARNING

- Liquid Chromatography (LC) and other laboratory applications can use flammable organic solvents as the mobile phase. Operators must be constantly on guard against accidents involving fire or explosion. The major cause of these accidents is static electricity. Devising preventive measures for accidents involving static electricity is difficult as the causes can be many, varied, individual, coincidental and / or simultaneous.
- LC systems are often used in association with large quantities of flammable solvents, thus the risk of large-scale damage is high, should an accident occur.
- The best way to prevent static electricity accidents is simply to prevent the occurrence and accumulation of electrostatic charges. Therefore, the use of antistatic shoes, tables, mats and floor covering is recommended.

Safety Symbols

The following safety symbols are attached to the cover on the back of the instrument. They are intended to alert you to the following safety information.

Table 1 Safety Symbols

Symbol	Description
	The apparatus is marked with this symbol when the user should refer to the instruction manual in order to protect risk of harm to the operator and to protect the apparatus against damage.
	Label indicates a CLASS 1 LASER PRODUCT.
	Confirms that a manufactured product complies with all applicable European Community directives. The European Declaration of Conformity is available at: http://regulations.corporate.agilent.com/DoC/search.htm

Device Characteristics

Delivery Content

The delivery of the detector includes the following components:

- Agilent 1260 Infinity II Multi-Angle Light Scattering Detector:
  - with connection kit, Accessory Kit (tubing, cables, connectors, fittings, and plugs)
  - User Manual (this document as PDF file on USB)
- Declaration of Conformity to manufacturing specifications

The device is electronically and mechanically tested. The fluid system of the detector is usually pre-filled with solvent (standard solvent is an 80 % water / 20 % methanol mixture) as documented on a clearly visible adhesive label on the device, if delivered with a system.

General Purpose

The Multi-Angle Light Scattering detector measures scattered laser radiation reflected from sample material (nanoparticles / polymers) separated by an LC system. This allows to determine molar mass, particle size and even branching of particles or macromolecules (for more information, refer to section "Working Principle" on page 18).

The detector uses high sensitive photon detectors with high-speed 24 bit A/D converters as well as a thermally controlled and stabilized measurement bridge to achieve best possible accuracy and performance. The integrated Ethernet LAN interface allows to control the instrument and to transfer all measured data to a PC running the Agilent WinGPC Software which can integrate further detectors like multi-angle light scattering (one MALS detector per WinGPC Software license), refractive index, and UV detectors.

Working Principle

For the determination of the mass and the size of particles, static light scattering can be used. A laser beam illuminates the sample. Due to the electrical field of the incident light, the electron shell of a molecule oscillates with the same frequency as the incident light. Every charge that moves emits radiation, and so every molecule emits light. Assuming that the sample has no conductivity, one can, to a first approximation, treat this radiation as a dipole scatterer that emits radiation isotropically.

Each atom in the sample thus acts as a light source. The sample consists of a large amount of molecules that can have more than one scattering center if a molar mass limit is reached (anisotropic scatterer). The more scattering centers there are, the more light is emitted. This means that the total intensity of the scattered light depends on the molar mass and sample concentration. The light from all these scattering centers interferes, and this interference pattern can be used to obtain information about the size of the sample.

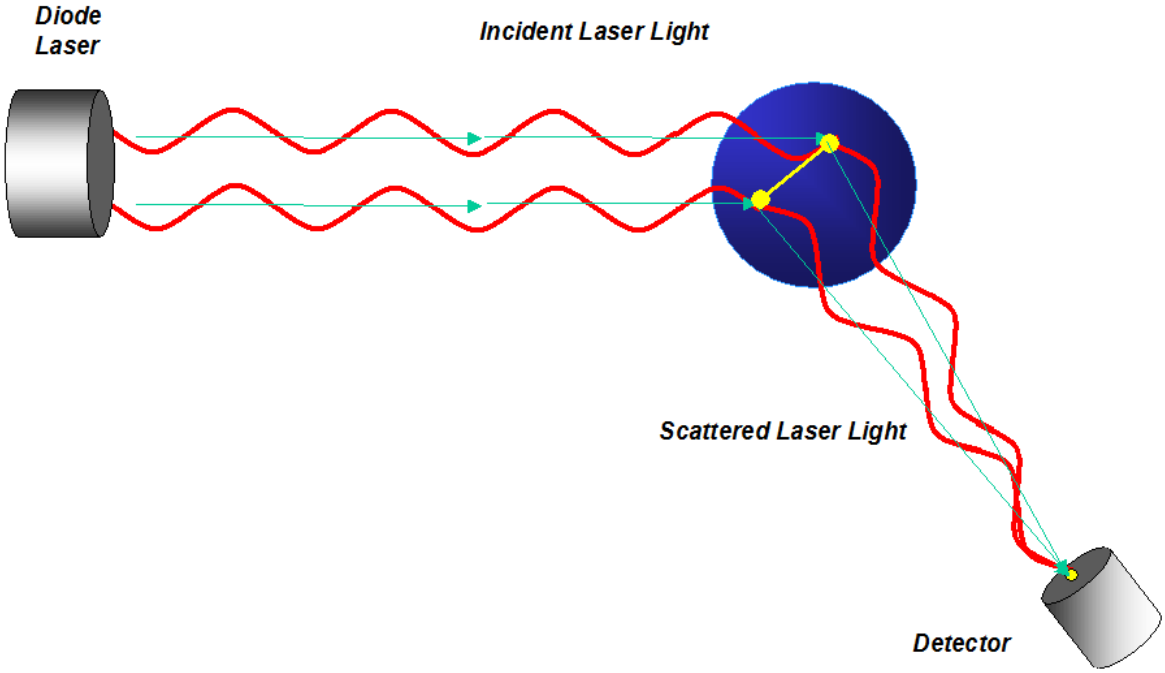


Figure 1 Schematic view of the light scattering setup

The scattering angle is defined as the angle between the passing laser beam and the scattered ray of light, captured by a photon detector at the respective angular position.

All incident light rays are in the same phase of maximum intensity. If they are scattered at an angle of 0°, they still have the same phase. All partial rays have their maximum and minimum at the same position. Due to the constructive interference, this is where the highest signal intensity occurs.

If we go to larger scattering angles and consider two partial rays scattered at two different centers, the partial rays have a phase shift due to the different positions of the centers. The interference of the two rays is no longer purely constructive and the intensity decreases. The greater the distance between the scattering centers, the lower the intensity. This means that with increasing scattering angles an intensity decrease is measured, which depends on the size of the particles. To calculate the complete interference pattern, all scattering centers of the molecule must be considered.

Thus, static light scattering takes advantage of two fundamental physical principles when light interacts with matter:

- The intensity of the scattered light is proportional to the product of sample concentration times molar mass of the sample.
- Small particles scatter light in all directions with nearly the same intensity (isotropic scatterer) while large particles scatter light mainly in the forward direction (anisotropic scatterer).

To utilize these facts for a determination of mass and size, the following experimental setup is used:

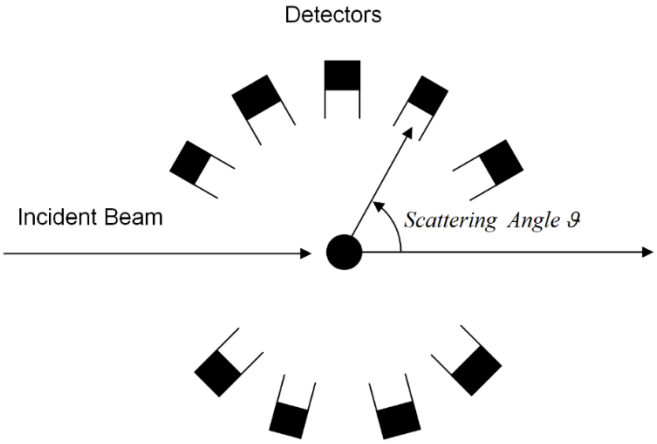


Figure 2 Scattering Geometry

Light is scattered by a particle and detected at different scattering angles. The intensity of the scattered light is determined by several detectors mounted at different angles ϑ.

Several factors contribute to the measured intensity. The measuring cell contains a collection of sample particles and solvent molecules. All these scatterer move and produce a time-dependent interference pattern.

The time-dependent interference pattern due to particle motion can be neglected in static light scattering, because the measuring time (about 0.1 sec) is much larger than the typical time constants for particle motion (in the microsecond range, depending on particle size). In dynamic light scattering, on the other hand, this time dependence is measured, and from this the size of the particles is calculated.

The contribution of the solvent can be eliminated by subtraction. For batch experiments, this means that the intensity distribution of the pure solvent is measured first and subtracted from the result of the sample in the solvent. For chromatographic runs, this means that the baseline is set to the solvent scattering level and this baseline is subtracted from the peak. The remaining scattering intensity is called the “Excess Rayleigh Ratio” in honor of Lord Rayleigh, who first described the scattering of light mathematically in 1871.

In general, there are two contributions from the scattering of particles. The angular dependence of the particles and how they sum up due to density fluctuations.

The scattering equation describes the intensities as a function of the angle ϑ for small concentrations:

$$\frac{R(\vartheta)}{cK} = MP(\vartheta)$$

With

$R(\vartheta)$ Excess Rayleigh Ratio = $\frac{I(\vartheta)}{I_0}\frac{r^2}{V}$

where

- $I(\vartheta)$ is the intensity measured at the angle ϑ,
- I_0 the incident intensity,
- r the distance from the scattering center, and
- V the scattering volume;

c is the concentration of the sample in solvent,

K the contrast factor = $\frac{\left(2\pi n_0 \frac{\partial n}{\partial c}\right)^2}{\lambda^4 N_a}$

where

- n_0 the refractive index of solvent,
- $\frac{\partial n}{\partial c}$ the refractive index increment of the sample (consistent with literature),
- λ the wavelength in vacuum, and
- N_a Avogadro's number;

M is the molar mass of the sample,

$P(\vartheta)$ the form factor of the sample, describing the angular dependence.

The first principle described above is directly visible. The intensity of the scattering is proportional to the concentration times the molar mass of the sample. The second principle is hidden in the form factor $P(\vartheta)$. The form factor describes the angular dependence of the scattering. It is defined as the intensity scattered at angle ϑ divided by the intensity scattered at angle 0. For zero scattering angle, the form factor is equal to 1.

The form factor depends on the size and shape of the particles.

Safety Functions

Temperature Limitation

A safety switch deactivates the cell heating when the maximum temperature of 70°C is exceeded.

Leak Sensor

To prevent the detector from internal leakages, which can be caused by wrong maintenance or a back pressure higher than 5 bar, the detector has an internal leak sensor. It is an optical sensor for aqueous components which does not come into contact with the solvent.

Vapor Sensor

To prevent the detector from internal leakages of organic substances, which can be caused, for example, by a back pressure higher than 5 bar, the device has an integrated vapor sensor. It is a sensor for organic components inside the instrument.

Physical Specifications

Table 2 Physical Specifications

Type	Description	Comments
Size [mm]	460 mm x 260 mm x 160 mm (L x W x H)	
Weight [kg]	12.6 kg (27.8 lbs)	
Ambient operating temperature	10 - 30°C (for indoor use only)	
Humidity	20 - 80 % RH (non-condensing)	
Power supply	100-240 V~ ± 10 % at 50-60 Hz, 155 W	
Fuses	2x T3.15A H 250V~	IEC60127-2, UL recognized
Operating altitude	≤ 2000 meters	
Safety standards: IEC, EN, CSA, UL	Overvoltage Category II	For indoor use only.
ISM Classification	ISM Group 1 Class B	According to CISPR 11

Performance Specifications

Table 3 Detector Specifications

Type	Description
Measurement range size	8 nm – 500 nm (R_g), depending on sample
Measurement range mass	10^3 Da – 10^9 Da (without Mie correction, depending on $\partial n/\partial c$)
Maximum pressure load	5 bar (500 kPa; 72.5 psi)
Solvent compatibility	Aqueous and organic solvents compatible with the wetted material, pH range: 2 – 11 (pH value higher 11 is considered damaging) no strong oxidizing acids
Cell material	Glass, titanium, and PTFE + 25% carbon
Cell volume	63 µL
Cell heating	10 °C above room temperature and up to 50°C 0 = off Stability: ± 0.01°C at 35°C cell temperature Safety switch at 70 °C No active cell cooling
Data rates	Maximum sampling rate: 6 Hz
Detection Range	12° - 164° at 20 angles
Laser	Wavelength: 660 nm at 10 – 120 mW Lifetime: 10 000 hours
Safety Functions	• Leak sensor • Vapor sensor
Liquid connections	Coned 10-32 UNF ports for tubing with 1/16 inch outer diameter
Status Indicators	LASER LED: (green) ON when the laser is active ERROR LED: (red) ON if a vapor, leakage, or an external error occurred
Software	Agilent WinGPC Software

Table 3 Detector Specifications

Interfaces	
Communication	Ethernet (RJ45) LAN interface; (standard IP address: 192.168.254.140)
Service Interface (RS232)	D-sub type 9-pin (DE-9) serial interface, female ± 16 Volt, max. cable length: ≤ 3 m (for setting of IP and MAC address)
4 x analog In	± 5 V at 24 bits (0.6 µV resolution)
2 x analog Out	0 - 10V at 16 bits (for chosen angles, with gaining)
Error In	TTL Pull Up 10 kΩ, 0 - 3.3 V
Error Out	Relays, contact closure, max 24 V, 0.1 A
Injection Ready In	TTL Pull Up 10 kΩ, 0 - 3.3 A
Injection Ready Out	Relays, contact closure, max 24 V, 0.1 A

For details on cables and connectors, refer to section "Cable and Connector Specifications" on page 57.

Detector Setup

Front Side

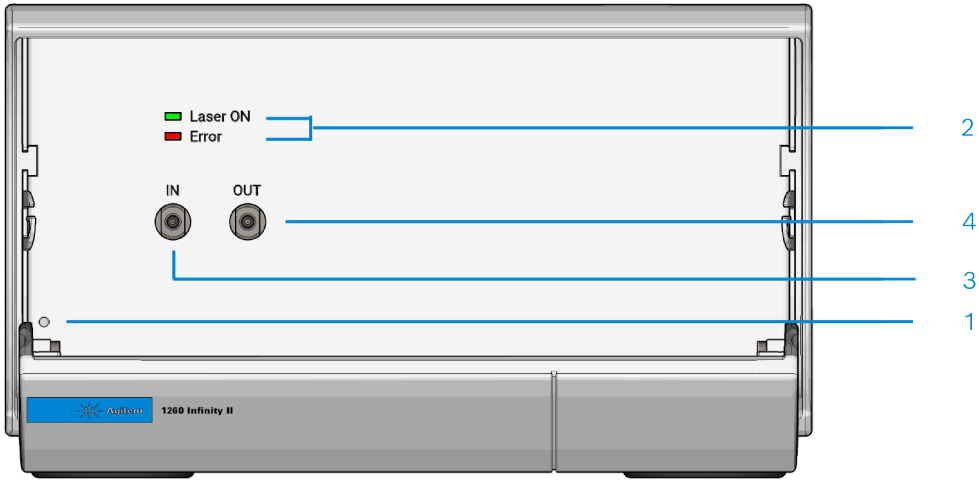


Figure 3 Front view with open front cover

The following connections and indication elements are accessible on the front panel:

1	Power On LED	If the system is powered on and running, the small LED is illuminated.
2	Status LED	The two LEDs show the actual status of the device: • LASER ON: green illuminated if the laser inside the detector is active. • ERROR: red illuminated if an internal vapor or leakage error occurred, or an external error is notified. The error state and the status of the laser will also be indicated via the software.
3	Inlet Port	This port is the flow inlet of the detector.
4	Outlet Port	This port is the flow outlet of the device which can be connected to the next detector or to the waste bottle.

CAUTION

Never block the outlet port if any flow is attached to the inlet port. This can lead to a high pressure inside the detector which causes internal leakages.

NOTE

If the detector is not in use and is disconnected from the flow, flush it first with pure water and then with an 80 % water / 20 % methanol mixture. Then plug the inlet and outlet ports. Please NEVER let dry out the measuring cell.

Before flushing the detector with water, make sure that the eluent (organic eluent with salt additives) used is miscible with water.

If necessary, first flush the detector with pure organic eluent and then switch to water or use a transition solvent!

For more information concerning immiscible solvents, refer to section "Miscibility Chart of Solvents" on page 61.

NOTE

To ensure best practice and avoid potential damage, we recommend the use of tubing, fittings, ferrules, and plugs supplied only by Agilent.

Rear Side

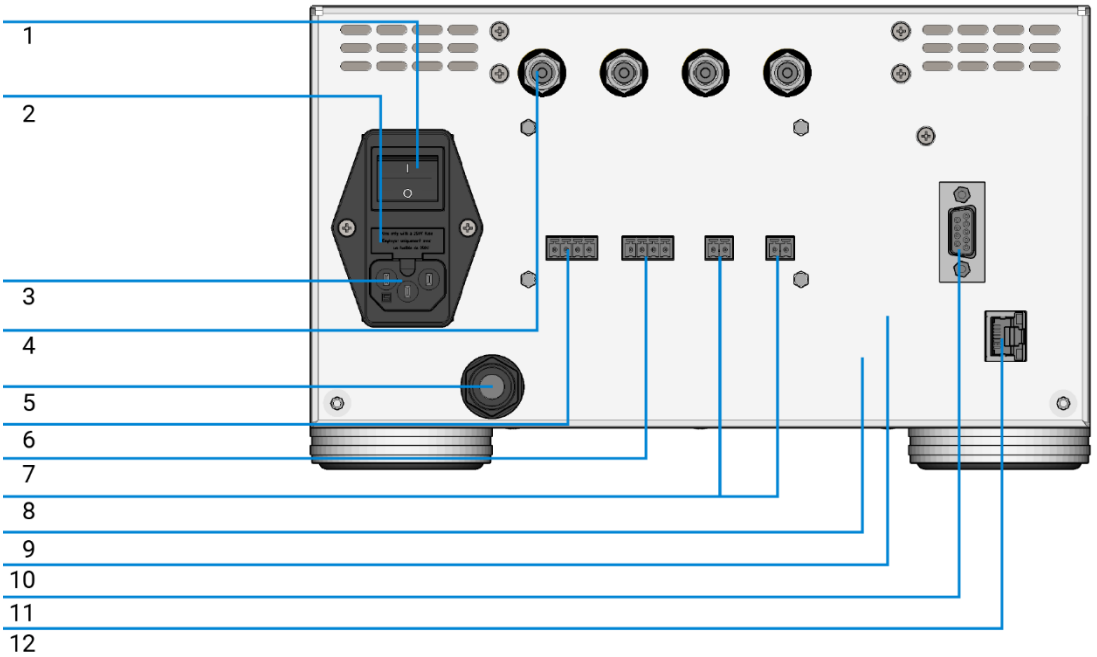


Figure 4 Detector rear panel

1	Mains Power Switch	With this toggle switch the detector can be powered on.
2	Fuse Holder	The fuse holder is placed between mains power switch and power connection. It can be opened by using a small screwdriver.
3	Mains Power Connection	Connect the supplied mains power cable to this connection.
4	Analog In	Use these connections to read in up to four analog detector signals from other detectors like RI, UV, and so forth. The ports are able to read in ± 5 volt signals. The setup of the detector connections can be configured in the corresponding software. NOTE Please use only detector cables supplied by Agilent.

5	Drain Outlet Port	Connect the drain tube to this connection. Internal leakages will be guided out of the equipment via this port. CAUTION Do not block the port!
6	Digital In and Out	External injectors or start signals can be connected to this connector strip. Incoming start signals will also be transferred to the Out connection to share it with other devices.
7	Error In and Out	Connect external error signals to this connection. Incoming error triggers can be transferred to the Out connection to share the signal with other devices.
8	Analog Out	Via these connections, the detector can emit the signals of two free selectable LS angles to supply them to external devices.
9	Type Identification Plate	Label with model and serial number.
10	IP Address Label	Label with configured network IP address.
11	RS-232-C Interface	This D-sub 9-pin female serial interface connector is used to set up the IP address of the detector.
12	RJ 45 (LAN) Connection	For control of the detector, please connect this port to the Ethernet adapter on the PC or – if more than one device with Ethernet connection is used – to the corresponding hub. Please use the cable supplied by Agilent.

WARNING Any other instruments connected to this instrument must be approved to a suitable safety standard and must include reinforced insulation from the mains.

CAUTION If incorrect power is connected to the digital I/O's, it could damage the interface or main board. Please refer for details to the corresponding section in this manual. For more help, please contact your local Agilent representative.

NOTE Only use power cables admitted for use in your country. To ensure best practice and avoid potential damage, we recommend the use of original cables and connectors supplied only by Agilent.

NOTE Make sure that the power plug on the rear of the detector is always accessible, so that the detector can be disconnected instantly from the power supply.

Detection Angles

The instrument works with individual photon sensors at different positions. All these detectors are arranged in the horizontal scattering plane around the cell.

The detection angles are independent of the solvent used, due to the cell design.

The following detection angles are used:

12°, 20°, 28°, 36°, 44°, 52°, 60°, 68°, 76°, 84°, 90°, 100°, 108°, 116°, 124°, 132°, 140°, 148°, 156°, 164°

NOTE Which of the angles can be used depends on the application.

2 Operation

This chapter describes the tasks concerning maintenance and care of the detector.

General Handling

Please obey the following guidelines and recommendations to avoid the risk of injuries and to prevent damage to the instrument:

WARNING

- When using organic solvent or aggressive aqueous solutions, please wear protective clothing and equipment such as safety goggles. It is recommended that a sink or suitable equipment such as eye wash stations / safety showers are provided as close as possible to the detector, in case the solvent in use comes into contact with the eyes or skin. To avoid injuries due to spraying chemicals (e.g., by an insufficiently connected tubing) it is advisable to permanently wear eye protection.
- Never put the detector into operation if the detector housing is damaged.

CAUTION

- Do not operate the detector with a dry measurement flow cell. Before starting the control software and the laser, make sure the fluid lines of the detector are filled and flushed thoroughly with liquid.
- When any abnormality such as liquid leakage is observed, turn off the power immediately and unplug the instrument from the mains power source.
- Never let dry out the measurement cell. If you need to store or ship the detector, close the flow inlet and outlet with plugs. Please use the supplied plugs with 10-32 UNF thread.
  Please contact Agilent if you need correct plugs.
  Don't plug the ports with any homemade tools or with a simple tape!
- For longer storage please flush the fluid lines of the instrument with a mixture of 80 % filtered and degassed water and 20 % methanol, ethanol or THF (if using organic solvent), to make sure, that there will be no bacterial growth inside the fluid path.
- When the eluent is changed, make sure that the liquids are miscible, and that no crystallization or precipitation of salt additives or remaining sample occurs inside the fluid path.
- When switching between two immiscible solvents (e.g. methanol – hexane), a neutral intermediate solvent (such as THF) miscible with both, the initial and the final solvent, must be used to flush the whole system thoroughly in between (see section "Miscibility Chart of Solvents" on page 61).
- Before changing the solvent, make sure that no sample remains in the detector flow path. First flush the flow path with the present pure solvent before you change the solvent.
- Use the instrument only in a clean environment.
- Don't use the detector in rooms with high humidity (≤ 80 %, non-condensing).
- Do not use the detector at room temperature higher than 30° Celsius. This will disturb the temperature stabilization of the measurement cell and the laser. This could result in baseline instabilities.
- Do not use the detector in rooms with high vapor concentration. This may cause instabilities in the baselines and will activate the internal vapor senor of the detector. Measurements are not possible!

The following guidelines and recommendations are to increase the lifetime of the laser source:

- If the laser is not used during the weekend, switch off the laser source in the Agilent WinGPC software or switch off the whole instrument.
- If the detector is not in use for a longer period, switch off the whole instrument. Please close the control software first. For details refer to the software manual.

The following guidelines and recommendations will help you to simplify the handling of the device:

- Switch on the hardware before starting the software.
- After switching on the hardware and laser, the detector needs 1 hour to warm up and reach a stable baseline. Please wait for a stable baseline before starting measurements.
- If the detector is not in use overnight, do not stop the flow through the cell if you use an organic eluent with salt additives. This can lead to the formation of corrosive substances. Keep the system running with a low flow rate (0.1 mL/min).

Commissioning

After connecting all fluid lines, power on the detector with the mains power switch on the rear of the device. Then start the Agilent WinGPC Software (see section "User Interface" on page 37) on the connected PC. On the **Login** screen, select **Agilent 1260 MALS** to activate the data acquisition of the MALS detector. Enter the standard IP address 192.168.254.140.

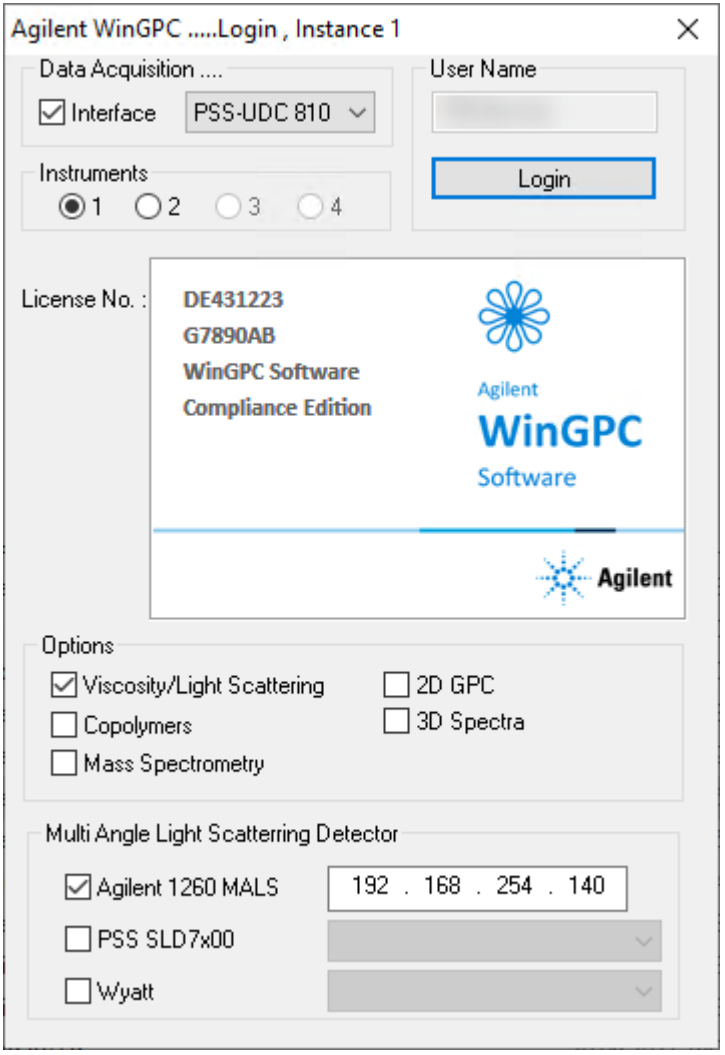


Figure 5 Login screen

Flush the measuring cell and the entire fluid path with the solvent to be used for the measurements. The detector is usually shipped pre-filled with a solvent (standard solvent is an 80 % water / 20 % methanol mixture), which is indicated by a sticker on the outside of the housing. When the solvent is to be changed, please pay attention to the notes in section "Changing the Solvent" on page 39.

NOTE

Before starting measurements, the detector signals must be calibrated in the software.

For more details, refer to the Agilent WinGPC User Guide.

User Interface

The Multi-Angle Light Scattering Detector is fully controlled via a user interface which is embedded into the Agilent WinGPC Software.

The user interface allows configuration of the detector settings as well as manual control of functions (Laser, Cell Heater On / Off, Error Signal), data acquisition and data evaluation.

To add the detector to the method, in the **Method** window of the WinGPC Software, navigate to the detector **Agilent 1260 MALS** in the resource tree under **Lightscattering**. Drag and drop the detector in the **Detector** section of the method window. The MALS detector must always be the last detector added to the method.

To edit the detector properties (for example, which angles are automatically displayed), right-click on the detector in the resource tree and select **Edit** from the context menu.

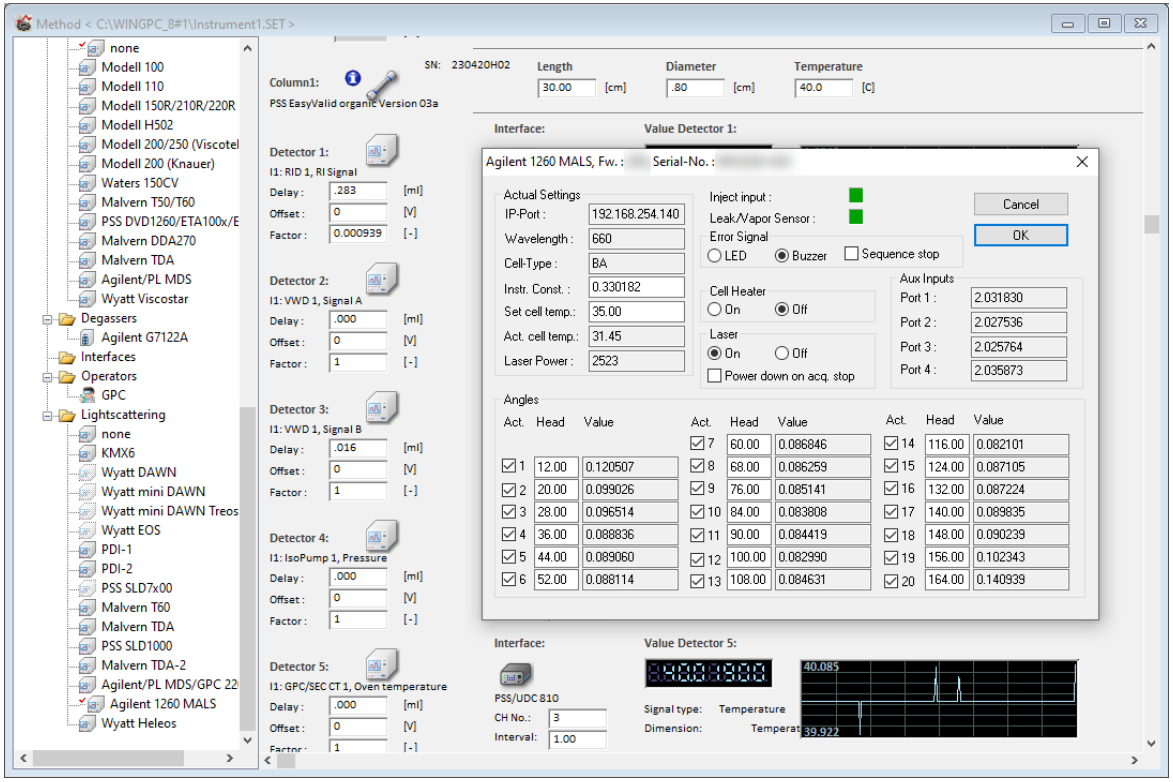


Figure 6 Properties window

To include the external ports (1-4) in data acquisition, select the channel **Aux** (01-04) from the list of the corresponding field **CH No.**:

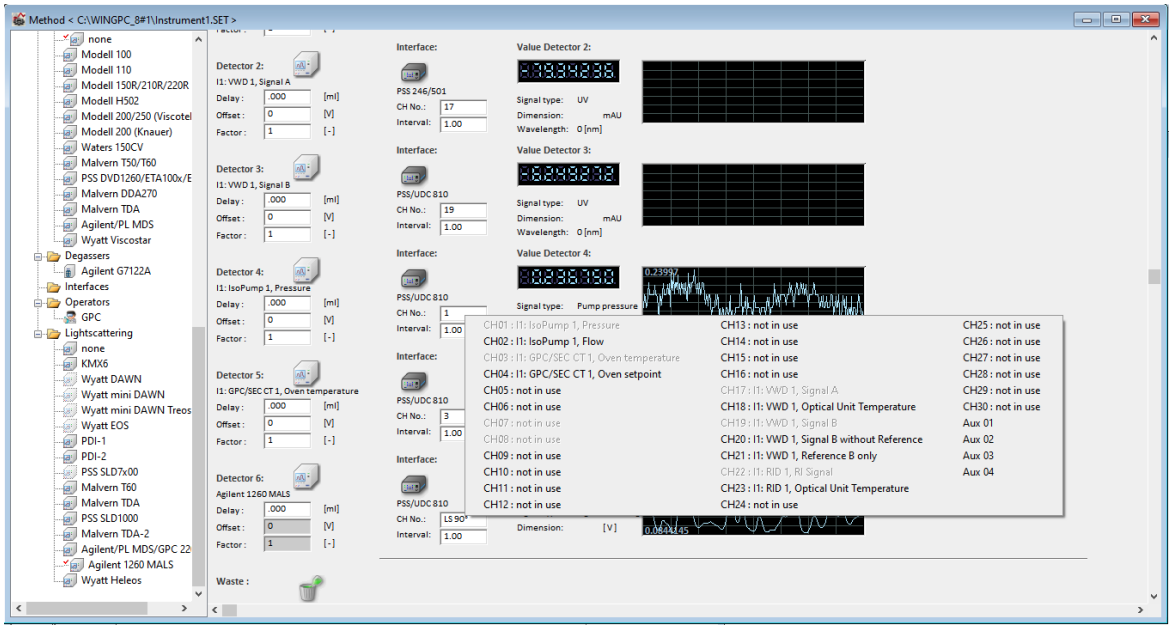


Figure 7 Channel selection

For detailed information how to operate correctly the detector using the Agilent WinGPC Software, please refer to the Agilent WinGPC User Guide.

Changing the Solvent

WARNING

- Wear protective clothing and equipment appropriate for the chemicals in use.
- Before changing the solvent, make sure that no sample remains in the detector flow path. First flush the flow path with the present pure solvent before you change the solvent.
- When the solvent is to be changed, the measurement cell and the whole fluid path must be flushed thoroughly with the new solvent.

NOTE

When the solvent is changed, make sure that the liquids are miscible and that no precipitation occurs inside the fluid path (please refer to section "Miscibility Chart of Solvents" on page 61"). When switching between two immiscible solvents, first use an intermediate solvent miscible in between, or if necessary, use co-miscible solvents stepwise to prepare the whole system for the use of the new solvent.

Storage

Short-Term Storage

If the detector is not in use overnight, do not stop the flow through the cell if you use an organic eluent with salt additives. This can lead to the formation of corrosive substances. Keep the system running with a low flow rate (0.1 mL/min).

You do not need to turn off the detector during short-term storage.

If the laser is not used, turn off the laser source in the WinGPC Software. This allows you to control (turn on and off) the laser remotely without turning off the detector.

Long-Term Storage

If the system is not in use for a longer period of time, proceed as follows:

NOTE

Before flushing the detector with water, make sure that the eluent (organic eluent with salt additives) used is miscible with water.

If necessary, first flush the detector with pure organic eluent and then switch to water or use a transition solvent!

For more information concerning immiscible solvents, refer to section "Miscibility Chart of Solvents" on page 61.

1. Flush the fluid lines of the detector first with pure, filtered water and then with an 80 % water / 20 % methanol mixture.
2. Disconnect the fluid lines and close the flow inlet and outlet with the supplied plugs. Do not leave the inlet or outlet port fittings open to the air for longer than necessary.
3. Then turn the instrument off.

Store the instrument in a clean, dry indoor place under stable ambient conditions.

Before the detector is used again, carefully flush the fluid path of the instrument once more with degassed pure water before connecting it to the system.

NOTE

To ensure best practice and avoid potential damage, we recommend the use of tubing, fittings, ferrules, and plugs supplied only by Agilent.

Leak Detection

To protect the detector from internal leakage, which can be caused, for example, by back pressures of more than 5 bar, the detector has an internal leak sensor. It is an optical sensor for aqueous components which does not come into contact with the solvent.

The alarm threshold level for the leak sensor can be adjusted in the control software depending on the location and environment of the device.

When an internal leak is detected, a warning sound or the ERROR LED on the front panel will be activated when enabled (**LED** or **Buzzer**) in Agilent WinGPC Software. For more details about the leak sensor and error signaling, refer to the Agilent WinGPC User Guide.

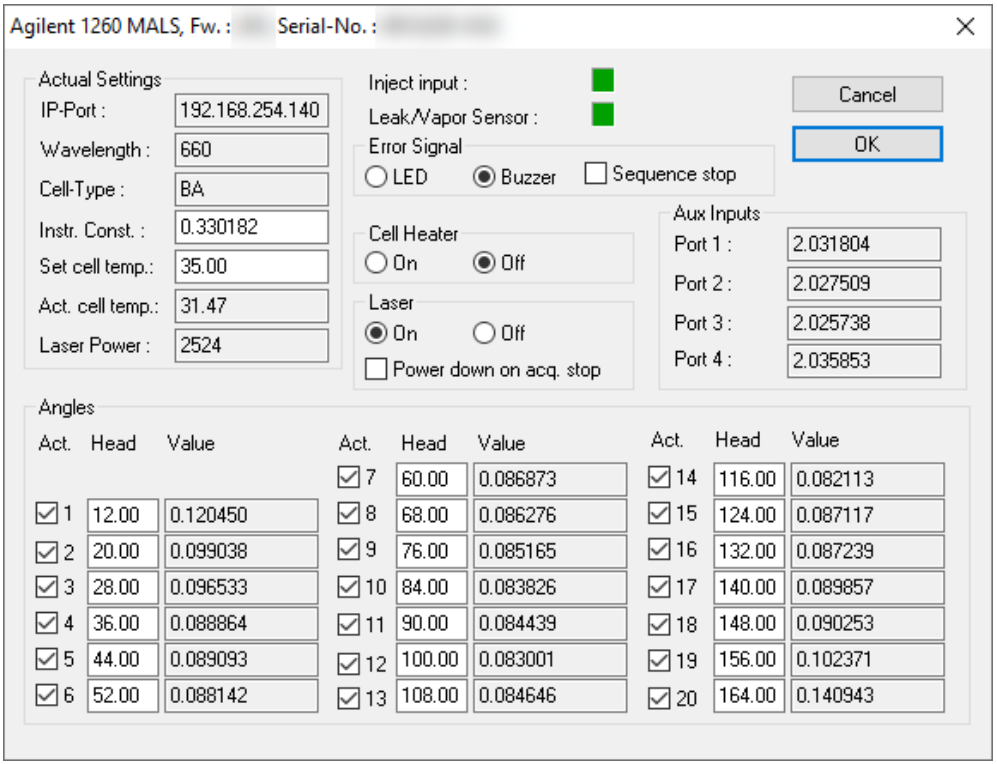


Figure 8 Agilent 1260 MALS detector properties window

If the detector is used embedded in a fully controlled system, you can configure to stop the pump while running a sequence in case of a leakage to protect the detector from major damage. This requires that the option **Sequence stop** is enabled in the properties window.

If the detector is used as a single detector, for example with the Agilent WinGPC Software, the external error indicator can be used to stop the upstream LC system.

Please check the following points if a leak is detected:

- Check the drain connection on the rear of the detector or – if no tube is connected – the area below the connection.
- If no liquid can be determined, try to run the system again.
- If leakage fluid is discovered, please stop the flow, close the software, disconnect the inlet and outlet tubing, and turn off the device.
  Please contact your local Agilent representative! The detector needs to be repaired.

NOTE

For further search for the source of the leakage, it is necessary to open the cover of the instrument. This repair work may be carried out only by Agilent service technicians. For support requiring to open the instrument's cover, please contact your Agilent representative.

After inspection of the detector and removing of all drain liquid:

1. Make sure that the device is powered on.
2. Reconnect the inlet and outlet tubing.
3. Restart the Agilent WinGPC Software and allow some solvent to flow through the detector.
4. If the leak error message appears again, disconnect the inlet and outlet tubing, turn off the device and contact your Agilent representative.

Vapor Detection

To protect the detector from internal damages, which can be caused, for example, by back pressures of more than 5 bar, the device has an integrated vapor sensor. It is a sensor for organic components inside the instrument.

The alarm threshold level for the vapor sensor has to be adjusted in the control software depending on the location and environment of the device. The factory preset value of the threshold level is 3500. Only in case of a critical laboratory environment it is necessary to change the value.

When a higher vapor level is detected, a warning sound or the ERROR LED on the front will be activated when enabled (**LED** or **Buzzer**) in Agilent WinGPC Software. For more details about the leak sensor and error signaling, refer to the Agilent WinGPC User Guide.

If the detector is used embedded in a fully controlled system, you can configure to stop the pump while running a sequence in case of a vapor detection to protect the detector from major damage. This requires that the option **Sequence stop** is enabled in the properties window (see Figure 8).

If the instrument is used as a single detector, for example with the Agilent WinGPC Software, the external error indicator can be used to stop the upstream LC system.

A vapor alarm can also be caused by the external environment. Therefore, please check first:

- Is any leakage close to the detector?
- Is the leak caused by a leaking connection at the inlet or outlet?
- Has the detector been cleaned with ethanol or methanol?
- Has anyone cleaned the tables nearby the detector with an alcoholic cleaning agent?
- Is the higher vapor content due to other causes in the lab environment (for example, eluent storage bottle, waste bottle)?

NOTE

If an external vapor source cannot be identified, it is necessary to open the case of the instrument and check the fluid line connections. This repair work must be carried out only by your Agilent representative. Please contact your Agilent representative for service.

After checking the connections, turn the instrument on, start the software and check if the error message is indicated again.

If the error message appears again, turn off the device immediately, disconnect the inlet and outlet tubing, and contact your Agilent representative.

3 Maintenance

This chapter describes the tasks concerning maintenance and care of the detector.

Preventive Maintenance

Take preventive measures to ensure that the detector performs consistently at an optimal level. To maintain the instrument in the best possible condition, the following precautionary measures are recommended.

In general, we recommend to use good laboratory practices (GLP):

- Use only high-purity solvent (HPLC grade) for mobile phases. Water should be bottled HPLC grade or filtered and deionized tap water.
- Filter all solvents to prevent particulate contamination and tubing blockages.
- Use only high-purity gases when drying contact areas.
- Ensure that all new tubing is thoroughly flushed before making fluid connections.
- Follow the short and long-term storage procedures that are described below.

Routine cleaning of the external surfaces of the instrument can be carried out using a clean, damp cloth.

NOTE Immediately clean any spills that occur on or near the device using methods appropriate for the type of spill. Some solvents can damage the appearance and function of the instrument.

Please periodically check the fluid connections at the front panel from time to time for any leakages.

Routine Maintenance

A general routine maintenance is not necessary. If there is a problem, please refer to the troubleshooting guide below. If you need more help, please feel free to contact your Agilent representative.

Detector Cleaning

During the use of the detector, after some time the measurement cell may become contaminated by sample material and buffer substances which can stick on the cell surface or remain inside the cell.

An indication for a contaminated cell can be:

- high baseline levels at low and high scattering angles in general,
- especially raised baselines for the low angles compared to the high angles,
- increased noise levels even for blank runs (without sample), or
- elevated baseline levels after repeated measurements.

To clean the detector cell, we recommend rinsing the cell with various cleaning solutions according to the procedure described in the following section.

To Flush the Detector Cell

WARNING Do not remove or disassemble the cell for cleaning. Let your Agilent representative perform any cleaning requiring removal of the cell. It is essential to ensure a correct operation of the detector after cleaning, and this activity must be performed only by your Agilent representative.

WARNING Wear protective clothing and equipment appropriate for the chemicals in use.

NOTE Use different cleaning solutions for cell rinsing, as the success depends on the sample material causing the contamination. Special cleaning solutions should be tested for unknown substances. In this case, please contact your Agilent representative.

For more information concerning immiscible solvents, refer to section "Miscibility Chart of Solvents" on page 61.

Please prepare the following, different cleaning solutions:

- 50 mL of ultra-pure water (Milli-Q grade),
- 5 mL of acetone (p. a.), and
- 5 mL of 40 – 60 % nitric acid (HNO3, 6 M) in water (p. a.).
- Hellmanex III 2 % solution (optional, instead of nitric acid)

NOTE

Instead of nitric acid, Hellmanex III 2% solution can be used first if it is available. Exposure to the cell for 30 minutes is sufficient. If this does not help, proceed with nitric acid as described in the cleaning procedure on page 51.

CAUTION

- Make sure, that all solutions have HPLC grade purity level and contain no particles.
- Additionally, we recommend the use of syringe prefilter (pore size 0.1 µm) supplied by Agilent. It is very important to avoid that large particles are introduced into the cell.

Use the following accessories from the maintenance kit to flush the detector:

- 1× syringe with Luer lock connection,
- 1× Luer lock connector with a piece of tubing and fitting, and
- 1× drain tubing and fitting.

To connect the syringe to the detector, use the delivered Luer lock adapter. If you change the cleaning solution, always remove the old solution completely from the syringe.

Please follow the next steps to flush the detector cell:

1 Stop the pump and the flow through the detector.

2 Disconnect the tubing at the inlet and outlet port.

3 Connect a large inner diameter capillary to the outlet port and direct it into a waste bottle.

4 Place a piece of tissue paper below the tube connection to collect escaping liquid.

5 Connect the Luer lock adapter to the tubing at the inlet of the detector.

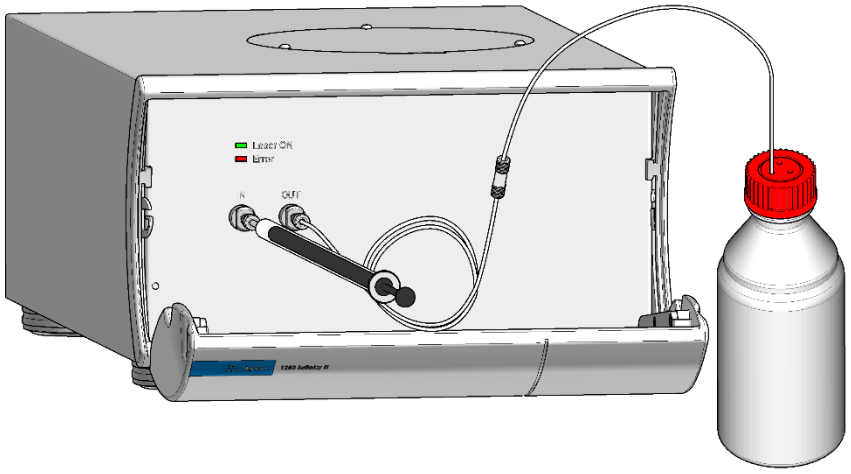


Figure 9 Detector cell cleaning with syringe

6 Connect the drain tubing to the outlet port to drain all outrunning cleaning solution to a waste bottle.

CAUTION

Do not collect acid liquids with a cloth or tissue paper. Acid vapors could get into the device and damage it by corrosion.

Proceed as follow:

7 a First, rinse the cell with the salt-free solvent previously used for measurement.
b Fill the syringe with ultra-pure water.
c Connect the filled syringe to the adapter.
d Flush the cell slowly step by step with 10 mL of pure water.

NOTE Make sure that the previously used eluent is miscible with water.

8 a Fill the syringe with Acetone.
b Connect the syringe to the Luer lock adapter.
c Flush the detector slowly with 2.5 mL.
d Wait 10 minutes and flush slowly again 2.5 mL through the detector.

9 a Fill the syringe with ultra-pure water.
b Flush the detector with 10 mL of water.
c Wait 10 minutes and flush the detector again with 5 mL water.

10 a Fill the syringe with nitric acid solution mixture (instead of nitric acid, you can also use Hellmanex III 2% solution if it is available).
b Flush the detector with 2.5 mL of nitric acid.
c Wait 30 – 60 minutes and flush the detector again with 2.5 mL nitric acid.
d Wait again 10 minutes.

11 a Fill the syringe with ultra-pure water.
b Flush the detector with 10 mL of water.
c Wait 15 minutes and flush the detector again with 10 mL water.

12 a Fill the syringe with the salt-free eluent previously used for measurement.
b Flush the detector with 10 mL of the eluent.

NOTE Make sure that the previously used eluent is miscible with water.

13 a Reconnect the tubing to the inlet and outlet.
b Start a low flow (0.5 – 1 mL) and flush the detector for 30 minutes.
c Start a measurement to verify the result of the cleaning procedure.

NOTE Pay attention not to overtighten the fittings.

4 Troubleshooting

This chapter describes the meaning of system problems and provides information on probable causes and suggested actions how to recover from these problems.

System Problems

Table 4 System problems

Problem	Probable Cause	Suggested actions
Mains power switch is on but **Power On** LED is off	Bad mains connection	✓ Inspect the power cable.
	Blown fuses	✓ Check the fuses on the rear panel. ✓ Please consult your Agilent service representative.
Red **Error** LED is on	Internal Leak	✓ Check external fluid connections.
	High vapor level detected	✓ Check if vapor is coming from the exterior of the detector (open solvent bottle, fumes close to detector). ✓ Please contact your Agilent service representative.
Bubbles appear through the output tubing	Loose fittings at the fluid connections	✓ Check external fluid connections. ✓ Tighten the inlet and outlet fittings. ✓ The use of an in-line degasser is recommended.
No solvent flow or high back pressure caused by the detector	Incorrect pump flow rate	✓ Check pump flow rate.
	Salt precipitation blocks the cell and/or tubing	✓ Try to flush the fluid path in the reverse direction.
	Blocked tubing connection	✓ Open the connections and check the fittings and ferrules for damages and salts. Note: If a fitting, a ferrule or tubing is damaged, then only replace it by a new one and never by a previously used part. ✓ Please contact Agilent for replacement parts.
Signal is noisy / signal jumps	Detector does not get stabilized	✓ Check the cell temperature setup.
	Cell temperature is not stable	✓ Check the function of the fan.
	Air gets into the flow path	✓ Check the fluid connections.
	No flow on the detector	✓ Check the fluid connections.
	Cell is contaminated	✓ Clean the measurement cell (for details, see section "Detector Cleaning" on page 49).
	Poor solvent quality	✓ Prepare new solvent
	Release of particles from the column	✓ Replace column with connector

Table 4 System problems

Baseline drifts at low and high angles	Solvent composition changes	✓	Connect new solvent.
	System gets dusty	✓	Filtrate the solvent.
		✓	Clean the system (for details, see section "Detector Cleaning" on page 49).
	LC system inline filter is damaged	✓	Change the inline filter.

Should the problem not be corrected even after taking the corrective actions above, consult your Agilent representative.

5 Identified Cables

This chapter provides information on cables and connectors used with the detector.

Cable and Connector Specifications

NOTE The use of cables supplied only by Agilent is recommended, to avoid any damage to the system.

Ethernet LAN cable

p/n	Description
8121-3135	LAN-Cable 7ft, Cat. 6 with 2 x Cat. 6 RJ45 connector, shielded, 3 m; IEC 60603-7-5, flame-retardant according to IEC 60332-1, halogen-free according to IEC 60754-2, low-smoke in compliance with IEC 61034, WEEE / RoHS compliant.

Detector Cable

p/n	Description
	Unitronic LiYCY – data cable with copper screening, 2x 0.14 mm² twisted cores, outer ø 3.9 mm, PVC-based core insulation colored in accordance to DIN 47100 Core-Ident-Code, outer sheath of special PVC-based compound, flame-retardant according to VDE 0482, part 265-2-1 / IEC 60332-1, WEEE/RoHS compliant with one or two RCA/Cinch male connectors (see next item).

RCA (“Cinch”) Connectors

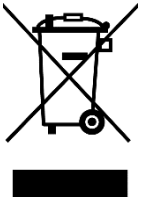
p/n	Description
	RCA/Cinch male connector, housing made of blue anodized aluminum alloy, strain relief spring, solder connection for central conductor, WEEE/RoHS compliant.

6 Appendix

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

Waste Electrical and Electronic Equipment Directive

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



NOTE

Do not dispose of in domestic household waste
To return unwanted products, contact your local Agilent office, or see http://www.agilent.com for more information.

Agilent Technologies on Internet

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

https://www.agilent.com

Miscibility Chart of Solvents

	Acetic Acid	Acetone	Acetonitrile	Benzene	Butanol	Carbon Tetrachloride	Chloroform	Cyclohexane	Dichloromethane	Dimethyl Formamide	Dimethyl Sulfoxide	Dioxane	Ethanol	Hexane	Methanol	Methyl t-Butyl Ether	Pentane	Propan-2-ol	Tetrahydrofuran	Toluene
Acetic Acid																				
Acetone																				
Acetonitrile																				
Benzene																				
Butanol																				
Carbon Tetrachloride																				
Chloroform																				
Cyclohexane			x																	
Dichloromethane																				
Dimethylformamide								x												
Dimethyl Sulfoxide								x												
Dioxane																				
Ethanol																				
Hexane	x		x							x	x									
Methanol								x						x						
Methyl t-Butyl Ether																				
Pentane	x		x							x	x				x					
Propan-2-ol (Isopropyl alcohol)																				
Tetrahydrofuran (THF)																				
Toluene																				
Water				x	x	x	x	x	x					x		x	x			x

x = immiscible

CAUTION

- Do not mix organic solvents (see table above) with aqueous solvents (acids, alkaline solutions, or buffer salts).
- Make sure that all components of the LC system are compatible with the solvents in use.

In This Book

The manual describes the following:

- Safety and Related Information
- Product Description
- Specifications
- Maintenance Procedures
- Troubleshooting
- Spare Parts

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Edition 07/2023

D0029607 Rev. B

Agilent