



# **Agilent 1260 Infinity Multi-Detector Suite**

## **User Manual**



**Agilent Technologies**

# Notices

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### WARNING

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

## In this book...

This manual contains information on:

- Agilent 1260 Infinity GPC/SEC MDS (G7800A)
- Agilent 1260 Infinity Bio-SEC MDS (G7805A)

### **1 Introduction to the 1260 Infinity MDS**

This chapter gives an instrument overview and an introduction to the function of the MDS detectors.

### **2 Site Requirements and Specifications**

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

### **3 Installation**

This chapter gives information about the installation of your MDS instrument.

### **4 Controlling the 1260 Infinity MDS**

This chapter describes how to control the instrument.

### **5 Operating the 1260 Infinity Multi-Detector Suite (MDS)**

This chapter describes the operational parameters of the instrument.

### **6 Optimizing Performance**

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

### **7 Troubleshooting and Diagnostics**

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

## In this book...

### **8 Error Information**

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

### **9 Maintenance**

This chapter describes the maintenance of the 1260 Infinity Multit-Detector Suite.

### **10 Parts and Materials for Maintenance**

This chapter provides information on parts for maintenance.

### **11 Appendix 1– Theory of Operation**

This chapter provides an overview of the principles and theory of RI, Viscometry and Light Scattering detection.

### **12 Appendix 2 – General Safety Information**

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

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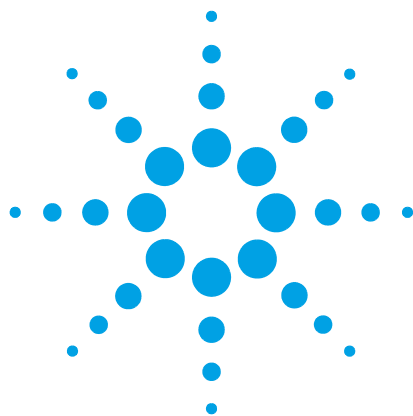
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This chapter gives an instrument overview and an introduction to the function of the MDS detectors.



## Introduction

The Agilent 1260 Infinity Multi-Detector Suite (MDS) is a high-end multiple detector platform, which provides not only accurate molecular weight data independent of the chemistry of the standards, but also gives insight into the behavior of the polymer in solution.

The system is available as two configurations:

**1 Agilent 1260 Infinity GPC/SEC MDS (G7800A):**

A system optimized for organic use with options of refractive index detector, dual angle light scattering detector, and viscometer.

**2 Agilent 1260 Infinity Bio-SEC MDS (G7805A):**

A system optimized for aqueous use with options of dual angle light scattering detector, viscometer, and dual angle light scattering with dynamic light scattering detector.

The key features include:

- *Ultra low dispersion* - Building on the Agilent 1200 Infinity Series UHPLC capabilities, the 1260 Infinity MDS is minimizing dispersion in GPC/SEC. The light scattering flow cell has a market-leading cell volume of just 10  $\mu$ L. The high backpressure RI cell is just 6  $\mu$ L. The viscometer is based on the latest advances in silica technology to create the ultimate balanced bridge.
- *Excellent baseline stability* - Each detector can be individually heated from 30 – 60 °C to ensure stable baselines and excellent signal-to noise performance. Advanced optical design drastically reduces baseline spiking in LS traces commonly seen at low angles.
- *Adaptable and upgradable* – any combination of detector may be fitted within the system, and the instrument may be expanded at any time allowing system capabilities to expand as budget allows.

The instrument offers the ability to collect digital detector outputs directly into Agilent GPC/SEC software via a USB connection. Output signals from external detectors can also be collected via the two independent analog input connections.

With state of the art detector design and control interfaces built around ease of use, the 1260 Infinity MDS is the solution to expanding GPC/SEC beyond conventional limits.

“[Performance Specifications](#)” on page 34 gives an overview of the system’s performance specifications.

## Instrument Overview

### Basic Principles of Operation

The 1260 Infinity MDS is designed to complement existing liquid chromatography systems to allow them to be used for GPC/SEC, configured with dual or quad detector cabinets depending on the detector choice, see [Figure 1](#) on page 12. Storage areas in the cabinet doors hold documentation, essential instrument accessories, and tools. If a detector cabinet has an empty detector bay, a tidy drawer system is supplied, providing a convenient location for storing columns and larger accessories.



**Figure 1** 1260 Infinity MDS Quad and Dual Enclosure Configurations

## MDS Control Unit

The MDS is controlled using the MDS Control Unit, which is fixed on top of the detector enclosure(s). The control unit remains identical regardless of the configuration of detectors in the system, allowing easy upgrading of additional detectors. The control unit features an LCD display with built-in keypad. The keypad is used to control all aspects of the MDS, and the LCD displays the status of the detectors within the instrument.

The MDS Control Unit provides the following:

- Full control of each connected MDS detector using the built-in keypad and display,
- status monitoring of each connected MDS detector,
- PC control digital data acquisition into Agilent GPC/SEC Software via the USB port,
- receive and pass through a sample injection trigger for synchronized data acquisition,
- two independent analog inputs for connecting signal outputs from external detectors,
- eight analog outputs  $\pm 1$  V,
- safety system,
- service utility for debugging and firmware upgrading of the instrument.



**Figure 2** MDS Control Unit

## MDS Detector Enclosures

The 1260 Infinity MDS features two complementary detector enclosures, which are designed to fix one on top of the other. Each detector enclosure has two detector bays; therefore, for a single or dual detector system, only the top enclosure is employed. For a one or three-detector system the remaining detector bay will be fitted with “tidy” draws for storing columns and other hardware. Each enclosure has a door on the front that can be configured to open from the left or right. Each door provides a storage area: the top door for manuals, and the bottom door for holding spanners.

Upgrading the 1260 Infinity MDS with additional detectors is simple. Tidy draws are swapped out for detectors and if a triple system is required a bottom enclosure is added.

The inlets and outlets of each detector that is fitted are sited at the enclosure(s) front. Connection between the detectors feature low dispersion tubing and minimized tubing distances to maintain low dispersion levels in the system resulting in excellent peak shapes with minimal band broadening.



**Figure 3** Front view of the 2- and 4-Bay MDS systems

## Safety and GLP Features

The 1260 Infinity MDS comes complete with several safety features and good laboratory practice (GLP) features to aid user confidence in the laboratory. The instrument is equipped with a leak sensor – should a spillage occur within the instrument, a signal is sent via the Auxiliary I/O Port. The port may be connected to the LC system operating with the 1260 Infinity MDS to shut down the pump and minimize solvent spillage.

The keypad is locked on receiving a sample injection trigger via the Auxiliary I/O port or from a **Start Collection** command from the Agilent GPC/SEC Software, if used. Locking the keypad ensures that the parameters of the detector(s) cannot be changed while the sample injection run is in progress.

## Application Areas

The versatile setup of the 1260 Infinity MDS allows different types of analysis to be performed, from the traditional ‘conventional’ GPC/SEC using a column calibration to the information-rich multi-detector GPC/SEC techniques that allow both accurate and reliable molecular weight information to be determined as well as giving key information on the behavior of the polymer molecules in solution. Any combination of the MDS detectors may be employed, with the following combinations being the most common.

## **G7800A – Agilent 1260 Infinity GPC/SEC MDS**

### **Conventional GPC**

The simplest offering is the MDS fitted with a refractive index (RI) detector. This system can be used to collect ‘conventional’ GPC data to allow molecular weight analysis via the use of a column calibration in the traditional GPC experiment.

### **GPC/Viscometry**

Combining the MDS RI detector with the MDS Viscometer allows the GPC experiment to be expanded; giving molecular weight information independent of the chemistry of the standards that are used to perform the calibration. This approach increases the accuracy of molecular weights that are generated and allows the structure of the polymer molecules to be probed in detail.

### **GPC/Light Scattering**

Combining the MDS RI detector with the MDS Light Scattering (LS) detector allows the determination of accurate molecular weights without the need for a column calibration. Furthermore, the size of the polymer molecules in solution can be determined by this method for high molecular weight materials.

### **Triple Detection**

The MDS may be configured for triple detection analysis employing the MDS RI, Viscometer, and LS detectors. The most powerful form of the GPC experiment, triple detection GPC allows accurate molecular weights to be obtained without the need for a column calibration, and gives detailed information about the structure and solution behavior of the polymer molecules.



## **G7805A– Agilent 1260 Infinity Bio-SEC MDS**

For the 1260 Infinity Bio-SEC MDS, Agilent Technologies uses highest-quality materials in the flow path (also referred to as wetted parts), which are widely accepted by life scientists, as they are known for optimum inertness to biological samples. Explicitly, the complete flow path is free of stainless steel and other alloys containing metals such as iron, nickel, cobalt, chromium, molybdenum, or copper, which can interfere with biological samples. The G7805A detector options require a concentration detector, such as the 1260 Infinity Bio-inert DAD.

### **SEC/Dual Angle Light Scattering**

Combining the MDS Light Scattering (LS) detector with a concentration detector allows the determination of accurate molecular weights without the need for a column calibration. Furthermore, this technique is excellent for the detection of aggregates.

### **SEC/Dynamic Light Scattering**

Complementing the MDS Light Scattering (LS) detector, DLS provides hydrodynamic size information for biomolecules.

## Data Collection and Analysis

The 1260 Infinity MDS provides on-board digital data acquisition, allowing data to be collected from the built-in detectors directly into Agilent Software via the USB port at the rear of the control unit. The data is collected at 24 bit resolution with collecting rates of 1, 2, 5 and 10 Hz selectable within the software.

The software that is used to control and collect data from the 1260 Infinity MDS is dependent on the model:

- Agilent GPC/SEC Software is designed and optimized for use with the 1260 Infinity GPC/SEC MDS system.
- Agilent Bio-SEC Software is designed & optimized for use with the Agilent 1260 Infinity Bio-SEC MDS system.

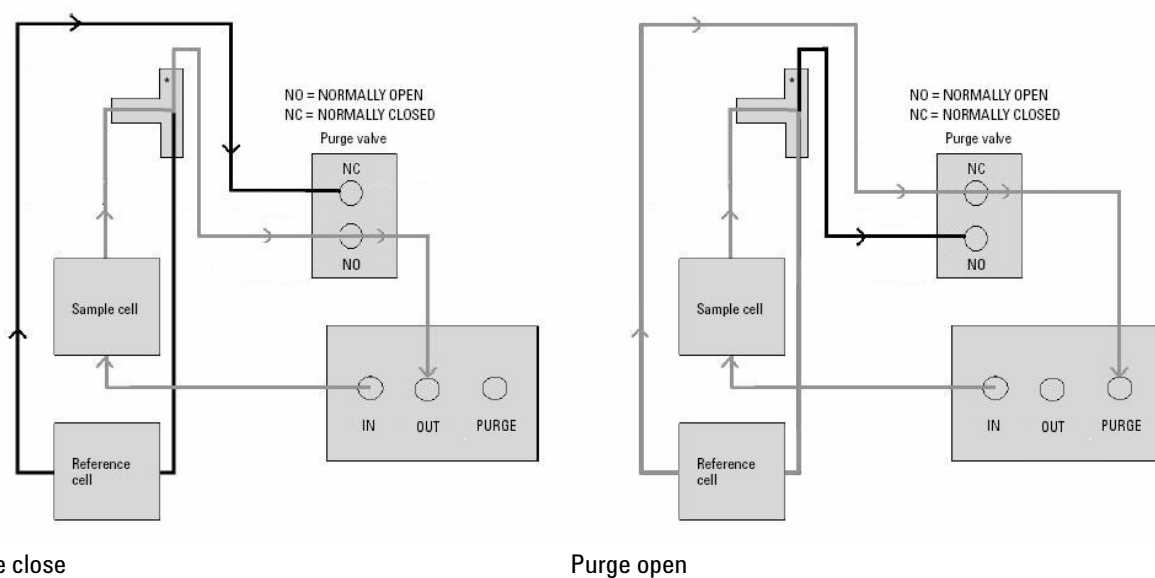
The 1260 Infinity MDS detector outputs are also available as analog signals (8 x  $\pm 1$  V A/D outputs accessed from the rear of the control unit), allowing the MDS to be used with any existing liquid chromatography system that supports Analog to Digital data collection.

It should be noted that a significant number of external HPLC software packages are not designed to perform multi-detector GPC calculations.

## The 1260 Infinity MDS Refractive Index Detector

The MDS differential refractive index detector is a high performance device that is specifically designed for GPC. The RI detector features high signal-to-noise and low levels of drift and is fitted with a low dispersion cell to maintain peak shapes throughout the sample injection. The heated cell environment means that excellent temperature stability is maintained, giving ultra-stable baselines.

**Table 1** Flow paths of MDS RI Detector with purge close/open



Grey lines – flowing path

Black lines – immobilized mobile path

\*The T-connection results in both sides of the flow cell always being exposed to the same pressure

The design of the cell allows the detector to be used in series, with a maximum of one detector placed behind. The maximum recommended flow rate that is permitted through the RI Detector is 5 mL/min. For more viscous solvents (for example 2-propanol), it is not recommended to work at the maximum flow rates.

## 1 Introduction to the 1260 Infinity MDS

### The 1260 Infinity MDS Refractive Index Detector

Before running solvent through the device, make sure that the solvent within the detector is completely miscible with the intended solvent to be used. In case of immiscible solvents, the cell must be rinsed through with an intermediate solvent (for example acetone), which is miscible with both solvents.

#### NOTE

The MDS RI detector is shipped in acetone.

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## The 1260 Infinity MDS Viscometer

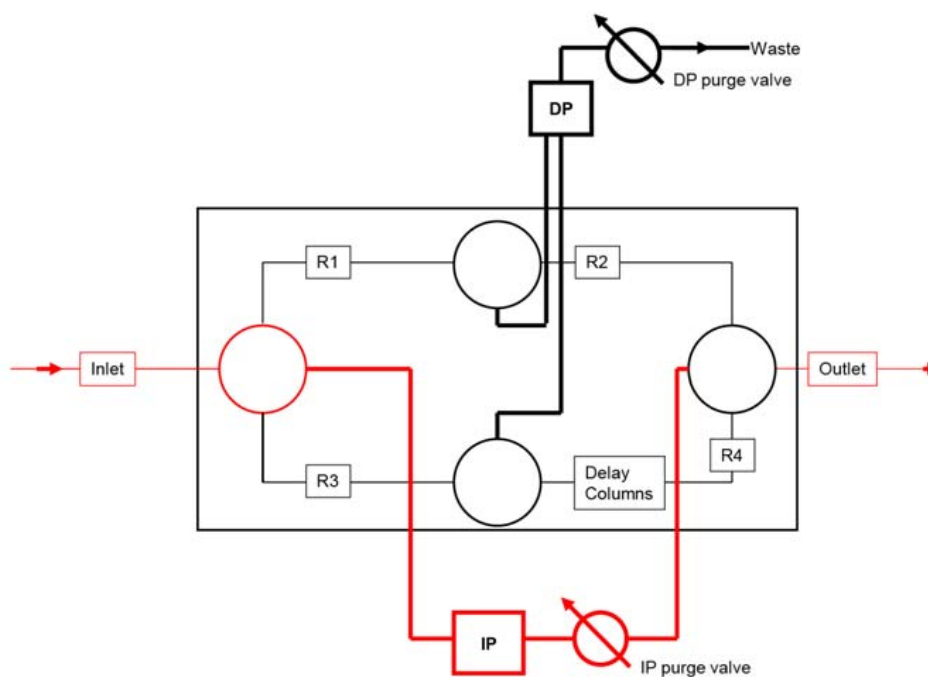
The MDS Viscometer is a temperature-controlled four-capillary fused silica bridge viscometer that is capable of measuring solution-based viscosities for a wide range of polymer samples and molecular weights. The balanced bridge design ensures that high sensitivity and a low shearing rate are maintained throughout the molecular weight range of analysis, giving excellent baseline stability. The pressure transducers that are used in the system give a high sensitivity and fast response time ensuring that sharp, clean peaks are obtained. The viscometer outputs of differential and inlet pressure values allow a dynamic measurement of inlet pressure to be used in calculations for greater accuracy in results.

The MDS Viscometer has a pressure rating of 50 kPa (7 psi) so it is recommended that no additional detectors be connected after the detector. Exceeding this backpressure on the viscometer will lead to internal leaks.

The maximum recommended flow rate that is permitted through the viscometer is 2 mL/min. For more viscous solvents (for example 2-propanol), it is not recommended to work at the maximum flow rates. Do not exceed 5000 Pa for the differential pressure (DP) and 100 kPa for the inlet pressure (IP).

## 1 Introduction to the 1260 Infinity MDS

### The 1260 Infinity MDS Viscometer



**Figure 4** Flow path of MDS Viscometer during purge

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Red lines – flowing path

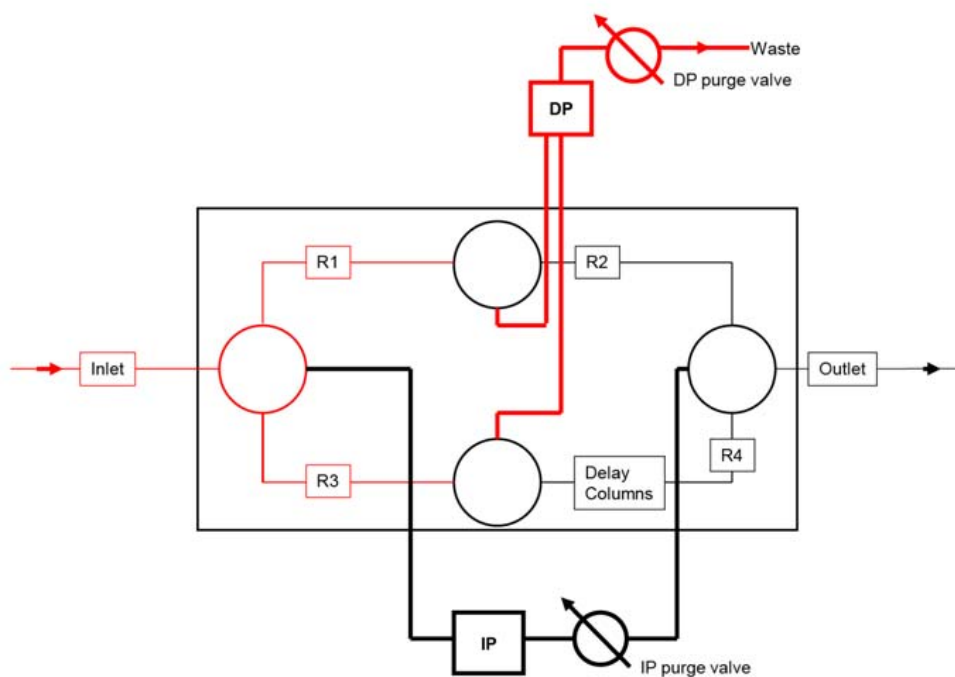
---

Black lines – immobilized mobile path

---

The T-connection results in both sides of the flow cell always being exposed to the same pressure

---



**Figure 5** Flow path of MDS Viscometer during DP purge

---

Red lines – flowing path

---

Black lines – immobilized mobile path

---

The T-connection results in both sides of the flow cell always being exposed to the same pressure

---

Before running solvent through the device, make sure that the solvent within the detector is completely miscible with the intended solvent to be used. In case of immiscible solvents, the bridge and transducers must be rinsed through with an intermediate solvent (for example acetone), which is miscible with both solvents.

**NOTE**

The GPC/SEC MDS viscometer is shipped in tetrahydrofuran.

## **1 Introduction to the 1260 Infinity MDS**

### The 1260 Infinity MDS Dual Angle Light Scattering Detector

## **The 1260 Infinity MDS Dual Angle Light Scattering Detector**

The MDS LS detector is a Dual Angle (15 ° / 90 °) Light Scattering Detector, which is capable of measuring the molecular weight and molecular dimensions of polymer molecules in solution without the need for a column calibration. The light scattering detectors maintain excellent signal-to-noise and the small cell volume size and heated cell environment ensure that excellent peak shapes are maintained.



## The 1260 Infinity MDS Dynamic Light Scattering Detector

The MDS DLS detector is a 90 ° Light Scattering Detector, with a further Dynamic Light Scattering detector on the same sample cell. This allows all the same functionality as the Dual Angle Light Scattering Detector but with hydrodynamic size measurements, without increasing dead-volume.

Before pumping solvent through any of the detector modules, make sure that the solvent within the detector is completely miscible with the intended solvent to be used. In case of immiscible solvents, the detector must be purged through with an intermediate solvent (for example acetone), which is miscible with both solvents.

**NOTE**

The MDS LS detector and Bio-SEC DLS detector are shipped in 2-propanol (IPA).

---

**1 Introduction to the 1260 Infinity MDS**  
The 1260 Infinity MDS Dynamic Light Scattering Detector



## 2 Site Requirements and Specifications

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



## 2 Site Requirements and Specifications

### Pre-installation Requirements

## Pre-installation Requirements

The MDS operation manual describes the environmental and operating requirements of the instrument. You must prepare your laboratory according to these instructions before the system can be installed. If you have misplaced your copy, you can obtain a replacement from your local Agilent office. Alternatively, download a PDF from the Agilent web site, [www.Agilent.com/chem](http://www.Agilent.com/chem).

## Site Requirements

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

### Laser Safety

When the Agilent LSD or LS/DLS detector is installed, the 1260 Infinity MDS is classified as a "Laser Class 1" product (IEC825-1, CFR1040.10 & 1040.11). During normal operation of the Agilent 1260 Infinity MDS no laser light is accessible to the user.

#### WARNING

##### Eye damage by laser light

**Hazardous laser light can injure eyes.**

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

### Power Considerations

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

#### WARNING

##### Hazard of electrical shock or damage of your instrumentation

**can result, if the devices are connected to a line voltage higher than specified.**

- Connect your instrument to the specified line voltage only.
- 

#### CAUTION

Inaccessible power plug.

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

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

## Power Cords

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

### WARNING

#### **Absence of ground connection or use of unspecified power cord**

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

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

### WARNING

#### **Use of unsupplied cables**

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

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

### WARNING

#### **Unintended use of supplied power cords**

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

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

## Bench Space

The module dimensions and weight (see [Table 2](#) on page 33) allow you to place the module on almost any desk or laboratory bench. It needs an additional 2.5 cm (1.0 inches) 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 HPLC system, make sure that the bench is designed to bear the weight of all modules.

### NOTE

The instrument is heavy; 20 kg (44 lb) for an MDS dual compartment and 40 kg (88 lb) for quad compartment and thus it is advisable to locate the system on a sturdy bench.

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### NOTE

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

---

## Location of the Instrument

Place the instrument in an area that does not experience large temperature fluctuations.

Avoid positioning the MDS:

- in direct sunlight,
- near heating equipment,
- near ventilation fans,
- near sources of vibration and electrical noise,
- near open windows and doors,
- in areas where corrosive gases exist,
- in dusty areas.

### NOTE

The instrument should not be stacked more than two enclosures high and suitable precautions should be taken to prevent the unit being knocked over.

---

### NOTE

Do not place solvent bottles or additional 1200 Infinity LC modules on the instrument.

---

## 2 Site Requirements and Specifications

### Site Requirements

#### Extraction Requirement

The MDS is not fitted with extraction, but depending on the solvents used it might be necessary to site the unit under an extraction hood or installed in a fume cupboard.

#### CAUTION

Temperature stability impaired

Locating the unit in a fume cupboard may compromise the temperature stability of the detector(s).

→ Place instrument outside of fume cupboard and use an extraction hose to vent fumes to fume enclosure.

---



## Physical Specifications

**Table 2** Physical Specifications

Type	Specification	Comments
Weight	10 kg (Dual compartment), 20 kg (Quad Compartment)	
Dimensions (height × width × depth)	400 × 490 × 390 mm (15.8 × 19.2 × 15.4 inches),	Dual compartment
	400 × 490 × 650 mm (15.8 × 19.2 × 25.6 inches)	Quad compartment
Line voltage	100 – 240 VAC, ± 10 %	Wide-ranging capability
Line frequency	50 or 60 Hz, ± 5 %	
Power consumption	150 W	Maximum
Ambient operating temperature	15-30°C (59-86°F)	
Ambient non-operating temperature	-40 – 70 °C (-40 – 158 °F)	
Humidity	<95 %, at 30 °C (86 °F)	Non-condensing
Operating altitude	Up to 2000 m (6562 ft)	
Non-operating altitude	Up to 4600 m (15091 ft)	For storing the module
Safety standards: IEC, CSA, FDA	Installation category II, Pollution degree 2, 21CFR 1040.10& 1040.11	For indoor use only.

## 2 Site Requirements and Specifications

### Performance Specifications

# Performance Specifications

**Table 3** Optional Refractive Index Detector Performance Specifications

Type	Specification
Detection type	Deflection
Wavelength	658 nm
Short-term noise	<80 $\mu$ V
Cell volume	6 $\mu$ L
Pressure rating	350 kPa (50 psi)
Wetted materials	316 SST, Quartz
Temperature range	30 – 60 °C
Temperature stability	$\pm$ 0.2 °C
pH range	2 – 10

**Table 4** Optional Viscometer Detector Performance Specifications

Type	Specification
Shear rate (typical)	3000s <sup>-1</sup>
Sensitivity $\eta_{sp}$	1·10 <sup>-5</sup> Pa/s
Pressure rating	50 kPa (7 psi)
Temperature range	30 – 60 °C
Temperature stability	$\pm$ 0.2 °C
pH range	2 – 10

**Table 5** Optional Dual Angle LS Detectors Performance Specifications

Type	Specification
Sample cell volume	10 µL
Scattering volume	0.01 µL
Laser wavelength	658 nm
Scattering angles	15 ° and 90 °
Temperature range	30 – 60 °C
Temperature stability	± 0.2 °C
pH range	2 – 10

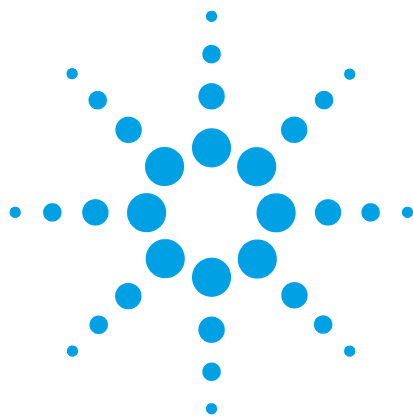
**Table 6** Optional DLS Detector Performance Specifications

Type	Specification
Cell volume	10 µL
Scattering Volume	0.01 µL
Measurement range (Rh)	1 – 1000 nm
Laser wavelength	658 nm
Measuring angle	90 °
Minimum sample loading	40 µg <sup>1</sup>
Temperature range	30 – 60 °C
Temperature stability	± 0.2 °C
Correlator	512 channels, multi-tau
Data acquisition time	0.1 – 3600 s
pH range	2 – 10

<sup>1</sup> 150 KDa Monoclonial Antibody – Conditions: Agilent Bio SEC-3, 300 Å, 7.8 x 300 mm, 3 µm (p/n 5190-2511), Mobile phase: PBS pH 7.4, Flow rate 0.75 mL/min

## **2 Site Requirements and Specifications**

### **Performance Specifications**



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This chapter gives information about the installation of your MDS instrument.



## Unpacking the Module

### Damaged Packaging

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

#### CAUTION

"Defective on arrival" problems

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

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

### Delivery Checklist

Unpack the 1260 Infinity MDS and accessories, and ensure that all parts and materials shown in the table below have been delivered with your module. Report any missing or damaged parts to your local Agilent Technologies sales and service office.

#### NOTE

The system and detector(s) are supplied separately in their own packaging.

---

## Delivery Checklist 1260 Infinity GPC/SEC MDS

<b>p/n</b>	<b>Description</b>
G7800A	1260 Infinity MDS Dual OR Quad Compartment Power cord
G7800-68000	Agilent 1260 Infinity MDS Installation Kit
G7800-63000	Drawer Unit (OPTIONAL)
G7800-68001	1260 Infinity GPC/SEC MDS 1200 Accessory Kit (OPTIONAL)
G7800-68002	Universal Accessory Kit (OPTIONAL)

### Optional Detectors:

<b>p/n</b>	<b>Description</b>
G7801A	Agilent 1260 Infinity GPC/SEC MDS RI Detector
G7802A	Agilent 1260 Infinity GPC/SEC MDS Viscometer
G7803A	Agilent 1260 Infinity GPC/SEC GPC MDS Dual Angle LSD

### 3 Installation

#### Unpacking the Module

## Delivery Checklist 1260 Infinity Bio-SEC MDS

<b>p/n</b>	<b>Description</b>
G7805A	1260 Infinity Bio-SEC MDS Dual Compartment Power cord
G7800-68000	Agilent 1260 Infinity MDS Installation Kit
G7800-63000	Drawer Unit (OPTIONAL)
G7800-68001	1260 Infinity GPC/SEC MDS 1200 Accessory Kit (OPTIONAL)
G7800-68002	Universal Accessory Kit (OPTIONAL)

#### Optional Detectors:

<b>p/n</b>	<b>Description</b>
G7808A	Agilent 1260 Infinity Bio-SEC LSD
G7809A	Agilent 1260 Infinity Bio-SEC LS / DLS



## Installation Kit

Agilent 1260 Infinity MDS Installation Kit (G7800-68000) contains the following accessories required to install the instrument:

<b>p/n</b>	<b>Description</b>
5062-2463	Corrugated tubing, PP, 6.5 mm id, 5 m
G7800-63001	Agilent 1260 Infinity MDS USB Cable
PL0100-3601	Valco Nut, 1/16 inch, 5 EA
G7800-87000	PTFE Tubing 0.25 mm ID x 1.6 mm OD (2 m)
G7800-87001	PTFE Tubing 1.0 mm ID x 1.6 mm OD (2 m)
PL0100-3602	Valco Ferrule, 1/16 inch, 5 EA
R005400012	Double-ended wrench, 1/4 in. and 5/16 in.
G7800-90000	Agilent 1260 Infinity MDS Operators Manual CD
G7800-10000	Agilent 1260 Infinity MDS USB driver

## Installing the Instrument

If your 1260 Infinity MDS is part of a complete Agilent 1200 Infinity Series liquid chromatograph, you can ensure optimum performance by installing the following configurations. These configurations optimize the system flow path, ensuring minimum delay volume.

### The MDS with the Agilent 1200 Infinity Series

The 1260 Infinity MDS is designed to operate with Agilent 1200 Infinity LC modules. The instruments are connected by feeding the outlet from the column or UV detector to the MDS and then back into either the waste bottle or into the HPLC detector compartment, depending on whether you wish to employ the detector that is already housed with the 1200 Infinity LC. Control of the MDS is not integrated into OpenLAB chromatography software or the Instant Pilot, so use of the keypad or the Agilent GPC/SEC software is required.

**NOTE**

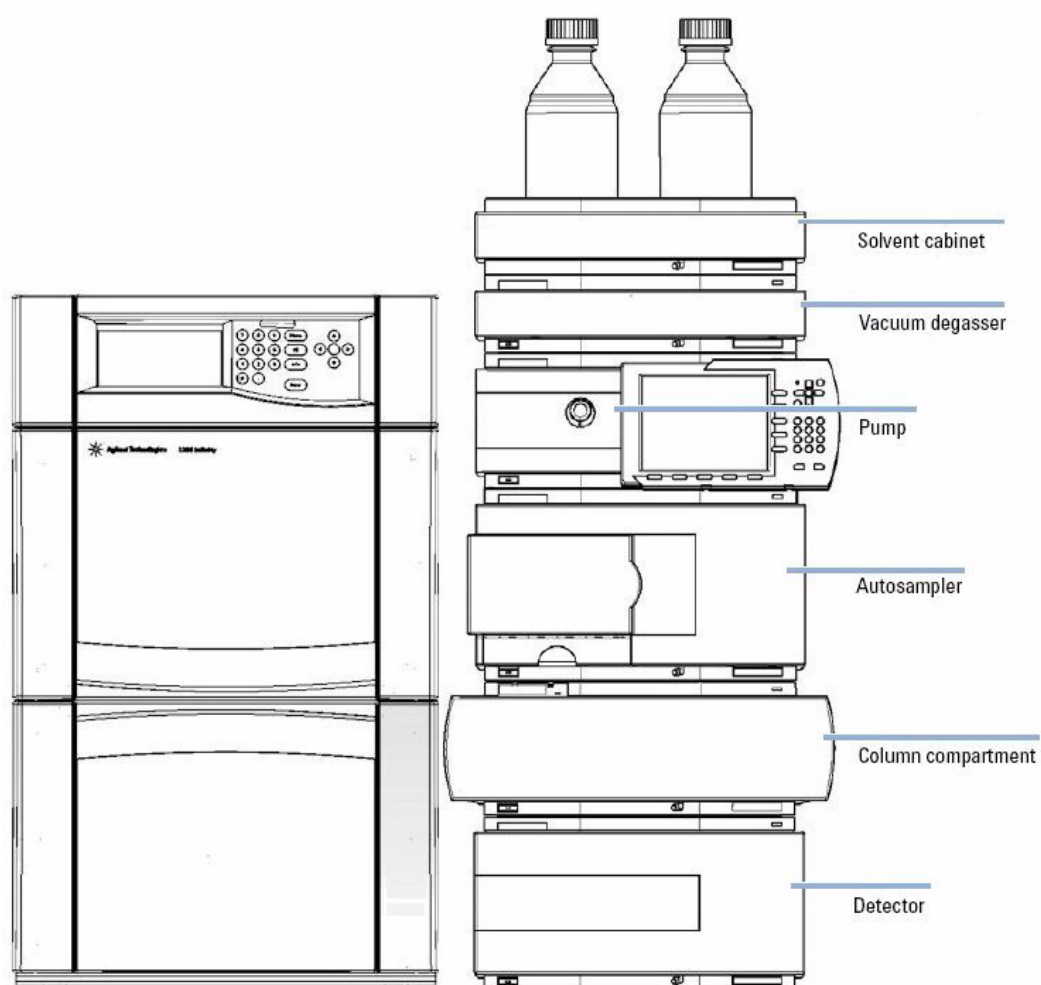
To perform GPC calculations on the collected data, the Agilent GPC/SEC Software is required, especially in case of multi-detector calculations. For this reason, it is highly recommended to always use Agilent GPC/SEC Software for control and collection.

---

## Dual Stack Configuration

Ensure optimum performance by installing the modules of the Agilent 1200 Infinity LC System in the following configuration (see [Figure 6](#) on page 43). This configuration optimizes the flow path for minimum delay volume and minimizes the bench space required.

It is recommended to place the Agilent 1260 Infinity MDS on the left-hand side of the LC system for easier access to the detector modules.



**Figure 6** Recommended Configuration

## **Connection to a 3rd-Party LC System**

The MDS system can be connected to any LC system (fully integrated or modular) that is capable of providing an isocratic flow of eluent. The MDS can be used with such systems by connecting the column or UV detector outlet to the MDS and the MDS waste line either back to the LC waste bottle or into the internal detector within the LC, assuming that you wish to use the existing detector system.

Data collection and analysis can be carried out using acquisition software capable of reading external analog signals and performing GPC type calculations.

Should you wish to use one or more existing detectors positioned within your LC system in conjunction with the MDS detector(s) and collect into Agilent GPC/SEC Software, the existing detectors will require analog output(s).

The analog outputs from the detectors can be connected to the analog inputs on the MDS.

## Installing the G7800 MDS

### WARNING

#### Heavy weight

**When detectors are installed the MDS dual & quad compartments weigh approx. 20 kg (44 lbs) and 40 kg (88 lbs), respectively.**

- Carry the instrument with two people.
  - Bend your legs rather than your spine and care that your back is straight.
  - Ensure that the load is as close to your body as possible.
  - Ensure that you can handle the weight of the load.
- 

### CAUTION

#### MDS drivers not installed

The connection between the MDS and the PC can't be established if the MDS drivers are not installed.

- Install the MDS driver before you connect the USB cable to the PC.
- 

- 1 Place the module on the bench in a vertical position.
- 2 Install the detector(s) into the appropriate bay.
- 3 Install and configure the Agilent GPC/SEC or Bio-SEC Software on the PC, if required.
- 4 Install the MDS drivers on the PC, if required.
- 5 Ensure the power switch at the rear of the instrument is OFF.
- 6 Connect the power cable to the power connector at the rear of the instrument.
- 7 Connect the USB cable to the PC.
- 8 Connect the remote start cable to the remote port on any 1260 Infinity series LC module.
- 9 Connect the fluid tubings, see “[Fluid Connections](#)” on page 49.
- 10 Turn on the power by switching the main power rocker located at the rear of the control unit to ON.

### 3 Installation

#### Installing the Instrument

## Powering the Instrument On or Off

- 1 Turn on the power by switching the main power rocker located at the rear of the control unit to ON.

The front panel display screen initially shows the Start-up screen.

#### NOTE

Switching the MDS on provides power to all the components integrated in the system.

---

#### NOTE

The control unit also sets up the Home screen (see [“The Home Screen”](#) on page 65).

The display should resemble the physical location of the detector(s) in the MDS and the Menu system (see [“The Menu System”](#) on page 69) so that only the installed detector options are available.

---

## Detector Output Channel Assignment

When powered on, the MDS Control Unit performs the following initializing procedure:

- Instrument components are checked and set.
- The detector bays are probed to determine the detector(s) installed with the detector(s) set to the last set of parameters used.
- The detector output(s) are assigned according to the location of the detector(s) installed.
- The **Home** screen and the menu system are setup.

The MDS Control Unit automatically probes the detector bays to discover which detectors are present. The detector bays are probed in the following order:



### 3 Installation

#### Installing the Instrument

As the system finds each detector, it assigns the detector's data outputs onto the output channels (digital and analog), so the lowest numbered channel will be the output from the first detector. For the viscometer and light scattering detector, the channels are ordered as follows:

**Table 7** Detector channel assignment

Detector	Channel 1	Channel 2
Viscometer	Differential Pressure (DP)	Inlet Pressure (IP)
Light Scattering Detector	15 ° (low Angle)	90 ° (high Angle)

The two analog inputs are always added as the last two channels of the system. An example is shown in [Table 8](#) on page 48, where RI, Viscometer and Light-scattering detectors are installed.

**Table 8** Analog input channel assignment

Detector Position	Detector	Detector Output	Assigned Output Channel
Top Left	RI	RI	1
Top Right	LS	Low Angle (15 °)	2
		High Angle (90 °)	3
Bottom Left	Viscometer	Differential Pressure (DP)	4
		Inlet Pressure (IP)	5
Analog Inputs	Ch1	Analog input channel 1	6
	Ch2	Analog input channel 1	7
		Spare	8

#### NOTE

Only digital output channels 1 – 4 can be collected when using Cirrus software.



## Fluid Connections

The fluid connections are located on the front panel of each detector module; the inlet port is on the left while the right port is the outlet. Ensure the capillaries that are connected to the inlet and outlet ports are not reversed since this could cause damage to the detector.

The MDS RI detector has a pressure rating of 350 kPa (50 psi) so additional detectors can be connected after the outlet, e.g. the MDS Viscometer. However exceeding this backpressure on the RI detector will lead to internal leaks and damage to the flow cell.

The MDS Viscometer has a pressure rating of 50 kPa (7 psi) so it is recommended that no additional detectors be connected after the detector.

The maximum recommended flow rate that is permitted through the viscometer is 2.0 mL/min. For more viscous solvents (for example 2-propanol), it is not recommended to work at the maximum flow rates. Do not exceed 5000 Pa differential pressure (DP) and 100 kPa for the inlet pressure (IP).

The MDS LS and LS/DLS detectors have pressure ratings of 350 kPa (50 psi) so additional detectors can be connected after the outlet.

### CAUTION

High backpressure

Exceeding backpressure limits will lead to internal leaks and damage to the detector.

→ Do not exceed the backpressure limit of the detectors.

---

The tubing required for all detector configurations is shown in the following section.

### 3 Installation

#### Fluid Connections

## Single Detector System

Parts required	p/n	Description
	G7800-87000	PTFE Tubing 0.25 mm ID x 1.6 mm OD (2 m) Connection from column to detector
	G7800-87001	PTFE Tubing 1.0 mm ID x 1.6 mm OD (2 m) Connection from detector to waste and/or purge waste
	PL0100-3601	Valco Nut, 1/16 inch, 5 EA
	PL0100-3602	Valco Ferrule, 1/16 inch, 5 EA
	IN4-34055	5PSI Back-Pressure Regulator (BPR) Connection from RI detector to waste

#### Fluid connections - LS Detector or Viscometer only

- 1 Establish the fluid connections as shown below.



#### Fluid Connections- RI Detector only

- 1 Establish the fluid connections as shown below.

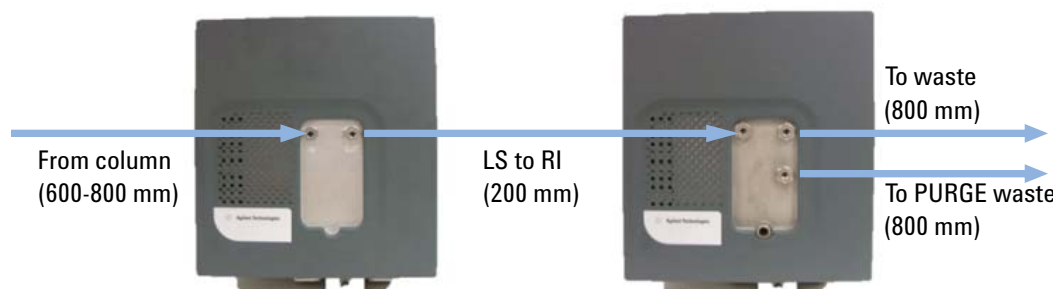


## Dual Detector System

Parts required	p/n	Description
	G7800-87000	PTFE Tubing 0.25 mm ID x 1.6 mm OD (2 m) Connection from column to detector and between detectors
	G7800-87001	PTFE Tubing 1.0 mm ID x 1.6 mm OD (2 m) Connection from detector to waste and/or purge waste
	PL0100-3601	Valco Nut, 1/16 inch, 5 EA
	PL0100-3602	Valco Ferrule, 1/16 inch, 5 EA

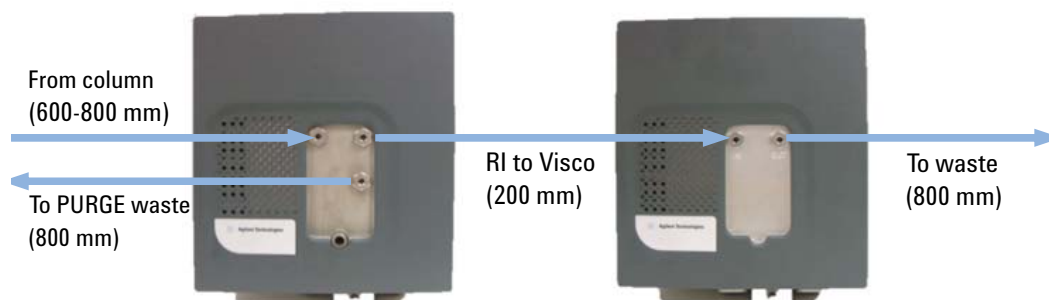
### Fluid Connections - LS AND RI Detectors

1 Establish the fluid connections as shown below.



### Fluid Connections - RI AND Viscometer Detectors

1 Establish the fluid connections as shown below.

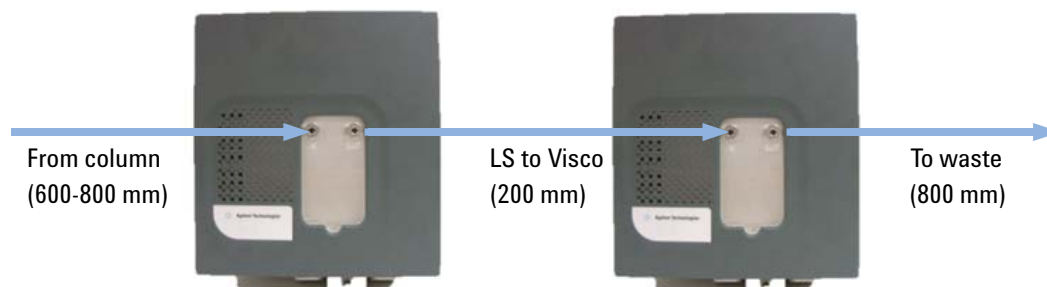


### 3 Installation

#### Fluid Connections

#### Fluid Connections - LS AND Viscometer Detectors

- 1 Establish the fluid connections as shown below.

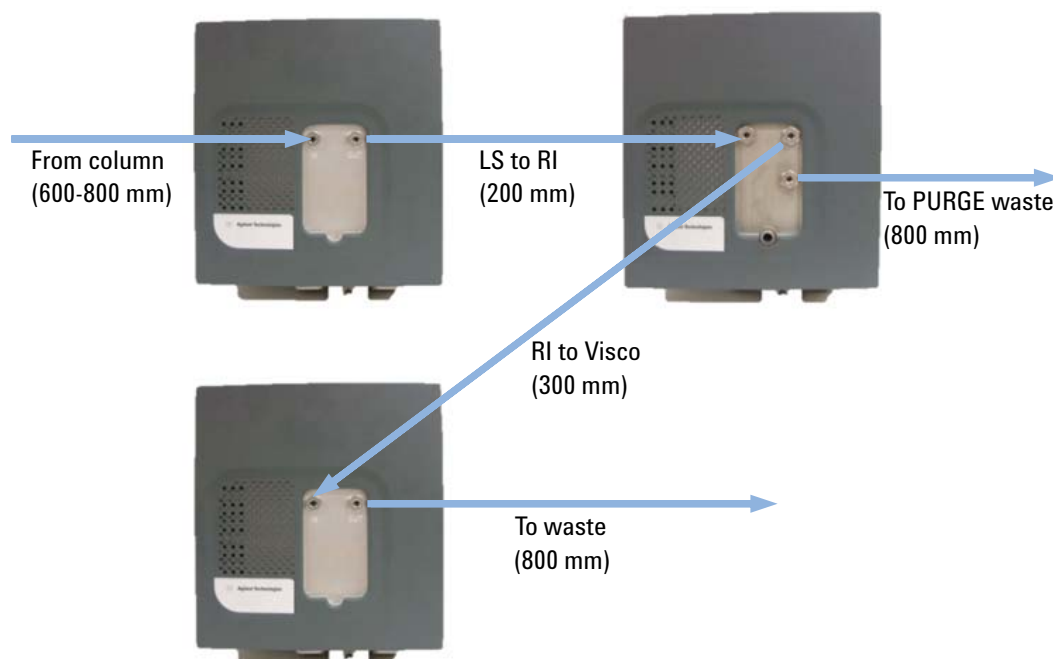


## Triple Detector System

Parts required	p/n	Description
	G7800-87000	PTFE Tubing 0.25 mm ID x 1.6 mm OD (2 m) Connection from column to detector and between detectors
	G7800-87001	PTFE Tubing 1.0 mm ID x 1.6 mm OD (2 m) Connection from detector to waste and/or purge waste
	PL0100-3601	Valco Nut, 1/16 inch, 5 EA
	PL0100-3602	Valco Ferrule, 1/16 inch, 5 EA

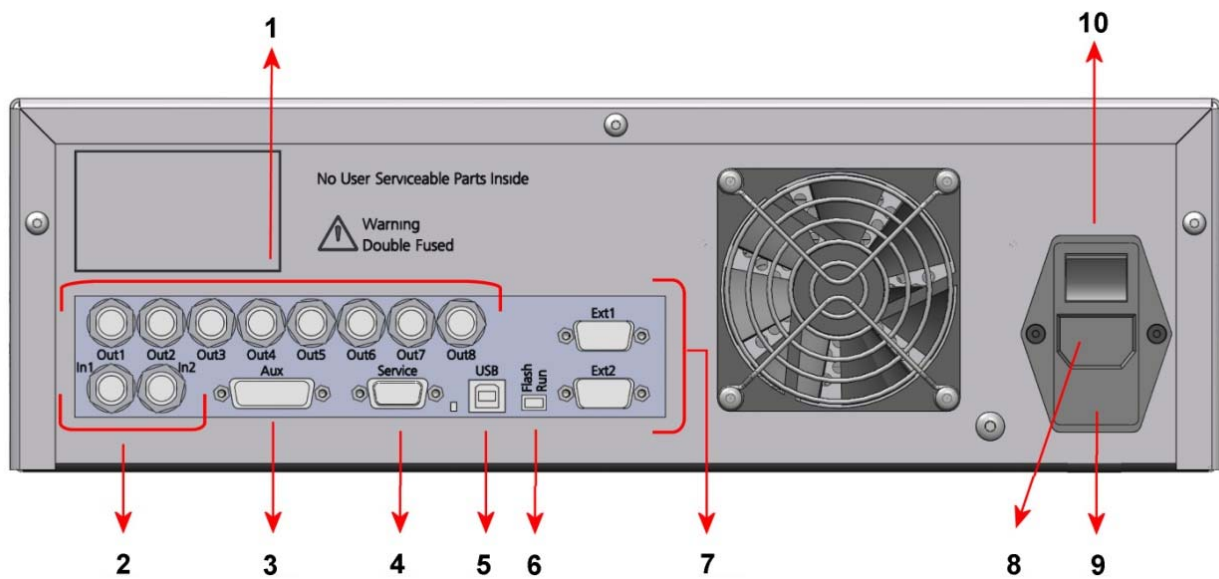
### Fluid Connections - LS, RI and Viscometer Detectors

1 Establish the fluid connections as shown below.



## Other Connections

All power, signal and communication connections are made on the rear panel of the MDS Control Unit, see [Figure 7](#) on page 54.



**Figure 7** Rear View of the MDS Control Unit

1	Analog outputs
2	Analog inputs
3	Auxiliary I/O port
4	Service port
5	USB port
6	Service switch (Run/Flash)
7	Serial RS232 ports (reserved)
8	Mains input
9	Fuses
10	Mains switch

## USB Port

### CAUTION

USB drivers not installed

→ Install the USB drivers before you connect the USB cable to the PC.

---

The Agilent 1260 Infinity MDS is fitted with a USB 2.0 full-speed connection for digital data acquisition into Agilent GPC/SEC or Bio-SEC software.

## Analog Outputs

The MDS is supplied with  $8\pm 1$  V analog outputs. The analog outputs are not fixed to specific detectors but are assigned according to the detector(s) present in the instrument.

## Analog Inputs

The MDS has two independent analog input channels located on the rear panel of the Control Unit. These are used for connecting signal outputs from external detectors to be collected digitally into Agilent GPC/SEC software via the USB cable.

Each input can have a zero offset applied.

## Auxiliary I/O Port

### CAUTION

The Pump stop facility must be employed if the instrument is to be left unattended.

- Connect the auxiliary cable ( Agilent 1260 Infinity MDS Auxiliary I/O cable (G7800-63003)) between MDS auxiliary port and remote port on 1200 Series LC System.

The MDS is fitted with an auxiliary 15 pin I/O connection for connecting the supplied auxiliary I/O cable. The auxiliary I/O cable provides the following:

- A 'Stop Pump Request' allowing a pump to be stopped if any error occurs on the MDS.
- Receive a sample injection trigger for synchronized data acquisition.

**Table 9** MDS I/O Connections

	I/O Description	Pin Number	Wire ID
Inputs	Sample Injection Trigger Input	5 & Ground	7 & GND
	Spare TTL Input	6	8
Outputs	Pump Stop Contact Closure - Normally Open	3 & 10	1 & 2
	Sample Injection Trigger Output - Normally Open	4 & 11	5 & 6
	Pump Stop - TTL Active Low	2 & Ground	4 & GND
	Pump Stop - TTL Active High	9 & Ground	3 & GND
	Ground (to case)	1, 7, 13 & 14	GND

## Serial RS232 Port

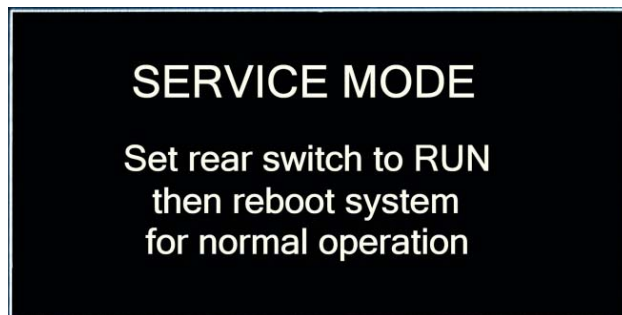
The MDS is fitted with two serial RS232 ports. These two ports are reserved for future use.



## Service Port and Switch

The 1260 Infinity MDS is fitted with a serial RS232 service port and service switch for debugging and firmware upgrading of the instrument.

If the instrument is inadvertently powered on with the service switch set to Flash then the front panel screen will display.

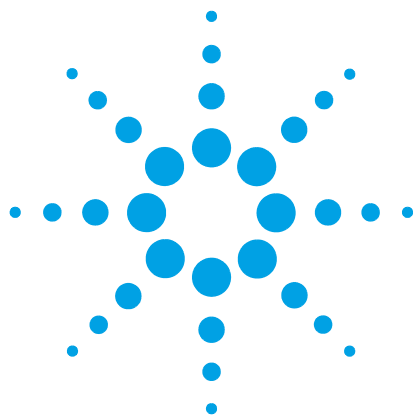


**NOTE**

For normal operation ensure the Service Switch is in the Run position prior to normal operation.

### **3 Installation**

#### **Other Connections**



## 4 Controlling the 1260 Infinity MDS

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The System Options Menu Screen	94
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This chapter describes how to control the instrument.



## 4 Controlling the 1260 Infinity MDS

### Controlling the Instrument

# Controlling the Instrument

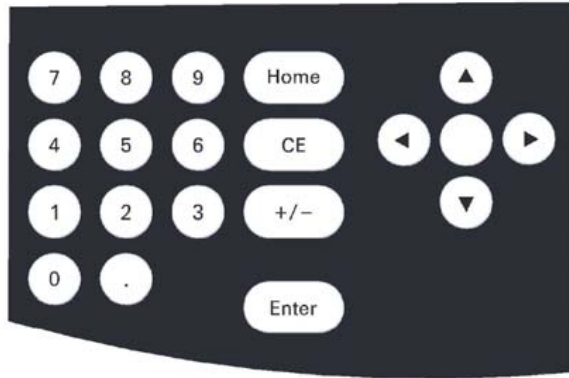
The MDS can be controlled via PC, or using the built-in keypad and display on the MDS Control Unit. The control unit provides the following:

- Monitoring detector temperature and signal outputs.
- Setting parameters on each detector for example temperature or purge time.
- Performing actions on a detector for example autozero or purge.
- Access to service parameters for each detector.
- Monitoring the status of the system for example a detector heating, the LS laser off or any errors.
- Access to system information for example firmware version of a detector.

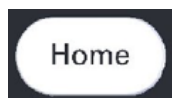


**Figure 8** MDS Control Unit

## The Keypad



**Figure 9** The MDS Keypad

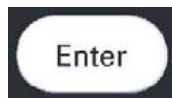


- Displays the Home screen.
- Abandons any non-committed changes.



**Select**

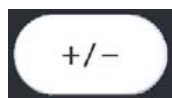
- From the Home screen
- Selects the **Main Menu** screen.
- From menu screens
- Selects the highlighted menu item.
  - Actions the highlighted menu action.
  - Commits the new parameter value typed in.



- From the Home screen
- Selects the **Main Menu** screen.
- From other menu screens
- Selects the highlighted menu item.
  - Actions the highlighted menu action.
  - Commits the new parameter value typed in.



- Cancels a not-committed parameter entry (the original value is redisplayed).
- Stops the purge detector action.
- Goes back one menu screen.



- Changes the sign of the parameter value being entered.

## 4 Controlling the 1260 Infinity MDS

### Controlling the Instrument



During Menu navigation

- Moves the cursor up or down a menu list.

While Editing a Parameter


- Increments/decrements numerical parameters by step-value.
- Cycles through parameter states (e.g. Normal/Invert).




- The Right arrow acts as Select.
- The Left arrow acts as Cancel.
- Pressing both keys simultaneously locks / unlocks the keypad.

### Locking and Unlocking the Keypad

The MDS Keypad automatically locks when:

- The MDS Control Unit receives a sample injection trigger via the Auxiliary I/O port or from a Start Collection command from the Agilent GPC/SEC software, if used. The injection icon  is displayed.
- The software is used to control the MDS. If the keypad becomes unlocked, the control software automatically locks it again within a couple of seconds.
- An error occurs

The display shows the Home screen with the lock icon , when the keypad is automatically locked.

The lock and injection icons disappear from the Home screen when the keypad is unlocked.

The MDS Keypad remains locked when:

- The MDS Control Unit receives a sample injection trigger via the Auxiliary I/O port without Agilent GPC/SEC software used for data collection. The injection icon also remains displayed.

#### CAUTION

Unlocked keypad


Data can be lost if the keypad is unlocked and used while data collection is in progress or if the system is being controlled from a PC.

→ Lock the keypad during data collection or while controlling the system from the PC.


#### NOTE

Navigation through the menu system is not possible while the keypad is locked.

- 1 To manually lock the keypad, press the left and right arrow keys simultaneously.

When locking the keypad the display will show the Home screen with the lock icon  displayed.

- 2 To manually unlock the keypad, press the left and right arrow keys simultaneously.

The lock icon  will be removed from the Home screen when the keypad is unlocked.

**NOTE**

Unlock the keypad manually in order to use the keypad and display to control the instrument.

---

## The Instrument Display

### The Start-up Screen

The Start-up screen is displayed when the MDS is powered on. The screen displays the product name and copyright and that the instrument is initializing, checking onboard components and probing the detector bays for the MDS detector(s) connected. Once the instrument has been initialized the Home screen is displayed.

**NOTE**

The keypad is not functional during the Start-up screen.

---

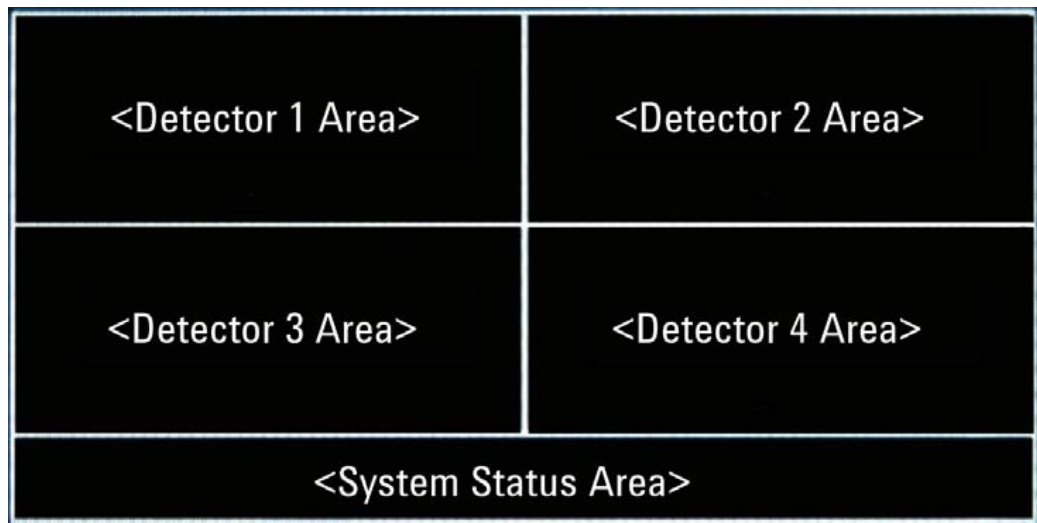


## The Home Screen

The Home screen is first displayed on the completion of the instrument initialization, during which the Start-up screen was displayed. The Home screen is dynamic and only shows information for the MDS detectors that are present in the detector enclosures. This screen can be regarded as a summary page, which displays the following:

- The status of the system
- The type of detector connected
- The status of each detector connected
- The current outputs of each detector connected
- Any errors that occur

The Home screen consists of four Detector Areas, even if the instrument is a single or dual detector system, and a System Status Area.



**Figure 10** Layout of the MDS Home Screen

Each Detector Area on the screen displays specific information for a detector and corresponds to the actual physical location of the detectors in the MDS Detector Enclosures. For example, if the MDS RI detector is located in the left hand detector bay of the top detector enclosure then the RI detector outputs and status appears in the top left Detector Area of the Home screen. If a detector bay has no detector present then the Detector Area on the Home screen will be left blank.

## 4 Controlling the 1260 Infinity MDS The Instrument Display

This mapping of detectors is done during the initialization of the instrument where each detector bay is probed to determine if any MDS detectors are present. See “Powering the Instrument On or Off” on page 46 for further information on the instrument initialization.

### Example Configuration



**Figure 11** MDS with RI, LS and Viscometer (VS) Detectors

The information displayed in the Detector Area is specific to the MDS detector connected but in general the following information is displayed:

- The detector type, that is RI, LS and VS (Viscometer)
- The detector signal output(s)
- The current temperature of the detector
- The detector status icons

For the LS Detector Area the L and H refer to the Low and High angles of the dual angle MDS LS detector, the 90 ° is the High angle and the 15 ° is the Low angle.

For the VS Detector Area the DP and IP refer to the Differential and Inlet Pressures of the MDS Viscometer.


The detector status icons displayed are:

**Table 10** Status icon information

Status Icon	Detector Area	Description
T	All	Temperature Status: <ul style="list-style-type: none"> <li>• Static – Detector at set temperature</li> <li>• Flashing – Detector going to set temperature</li> <li>• Flashing with a cross – Heater is off</li> </ul>
P	RI & VS	Purge Valve Status: <ul style="list-style-type: none"> <li>• Flashing – Purge Valve Open</li> </ul>
A	RI Only	Autozero Status: <ul style="list-style-type: none"> <li>• Flashing – Autozero in progress</li> </ul>
L	LS Only	Laser Status: <ul style="list-style-type: none"> <li>• Static – Laser On</li> <li>• Flashing with a cross – Laser off</li> </ul>

The System Status Area of the Home screen displays information for the whole MDS system. The following information is displayed in the System Status Area:

- The system status state, i.e. Ready or Not Ready (see [Table 10](#) on page 67)
- The keypad status
- The data collection injection status
- Any errors that occur

For the keypad status there are two states: locked and unlocked. For the locked state the Lock icon  is displayed. The Lock icon will be displayed when:

- The keypad is manually locked.
- The Agilent Software GPC/SEC or Bio-SEC Software connects to the MDS.
- The Control Unit receives a sample injection trigger via the Auxiliary I/O port or from a **Start Collection** command from the Agilent GPC/SEC software, if used.
- An error occurs.

## 4 Controlling the 1260 Infinity MDS

### The Instrument Display

When the keypad is unlocked the Lock icon is removed from the System Status Area. For further details on locking and unlocking the keypad see [“Locking and Unlocking the Keypad”](#) on page 62.

The data collection injection status indicates that an injection has been made and data collection is in progress. This is indicated with the

Injection icon  being displayed and will occur when:

- The Control Unit receives a sample injection trigger via the Auxiliary I/O port or from a **Start Collection** command from the Agilent GPC/SEC software, if used.

This will also automatically lock the keypad with the Lock icon displayed in the System Status Area. If Agilent GPC/SEC software is used for data collection then on completion of the sample injection run or on an Abort Run command sent then the Injection icon will be removed from the System Status Area. Otherwise manually unlocking the keypad will remove the Injection icon.

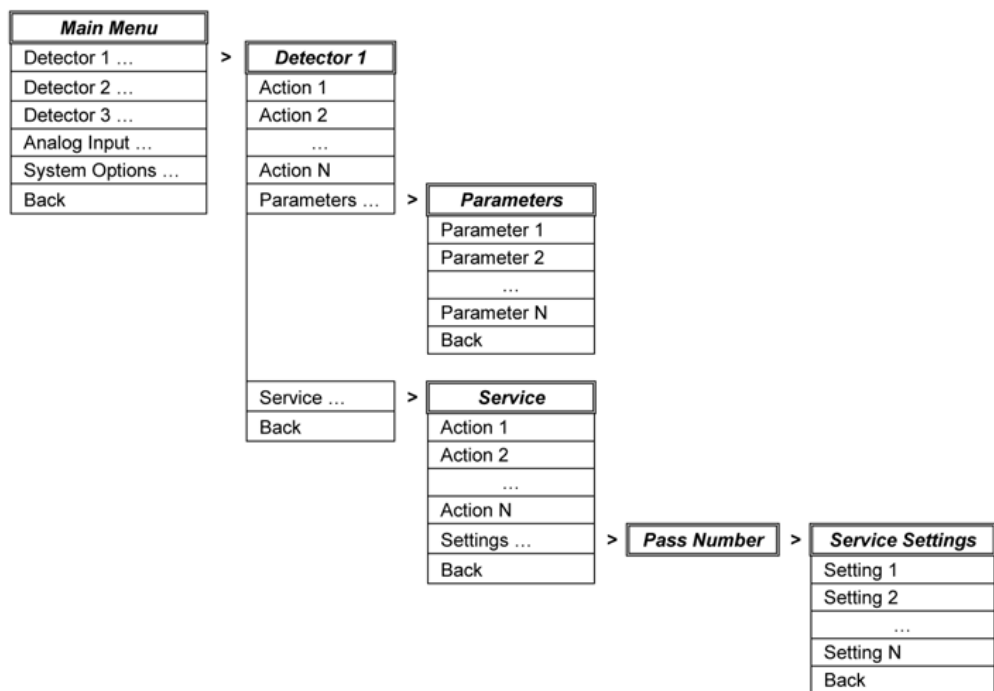
The System Status Area will also display any error that occurs, with error code and error message.

From the Home screen pressing either the **Select** or **Enter** keys on the keypad accesses the MDS Menu System.

The Home screen can be displayed at anytime by pressing the **Home** key on the keypad.

## The Menu System

The MDS Menu System is accessed from the Home screen of the display by pressing either the **Select** or **Enter** keys on the keypad. The Menu System comprises of a series of screens, which allows access to system and detector information and specific actions and parameters for the detector(s) connected. The general structure of the Menu System is as follows:



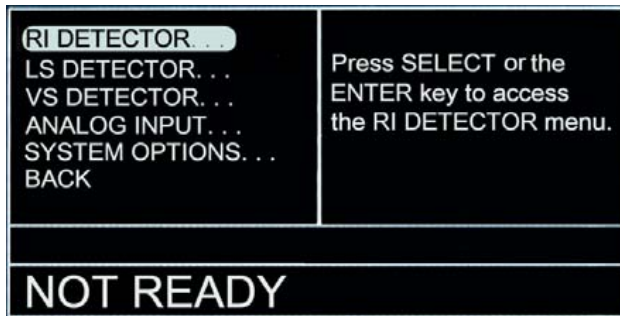
**Figure 12** MDS Menu System Structure

## 4 Controlling the 1260 Infinity MDS

### The Instrument Display

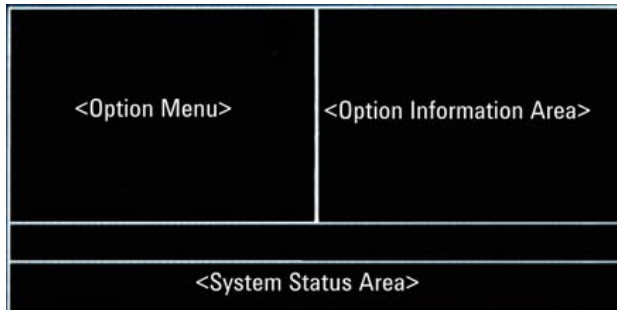
#### The Main Menu screen

The **Main Menu** screen is the top level menu of the MDS Menu System from this menu options for each detector and system can be accessed. This screen is dynamic and only shows options for the MDS detectors that are present in the detector compartments.



**Figure 13** The MDS Menu System

The **Main Menu** screen consists of an Options menu, an Options Information Area and a System Status Area.



**Figure 14** Layout of the MDS Main Menu Screen

The Options menu lists the detector and system menus available for your MDS. The full list of menu options is shown below.

- **RI Detector...**
- **LS Detector...**
- **VS Detector...**
- **Analog Input...**
- **System Options...**
- **Back**

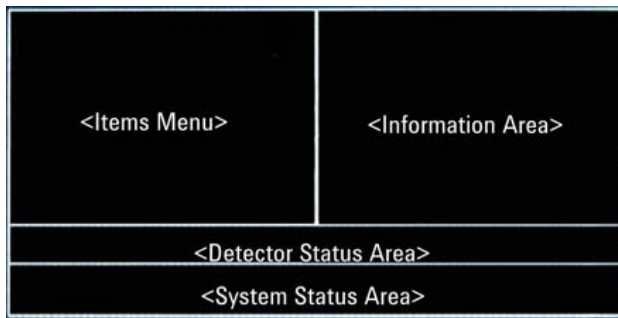
To select the required menu option, use the up and down arrow keys on the keypad to highlight the menu and then press either the **Select** or **Enter** keys. Selecting the **Back** option returns the display to the Home screen. The Options Information Area displays information about the highlighted menu option. The System Status Area within the **Main Menu** screen only displays the system status states either Ready or Not Ready. See [Table 10](#) on page 67 for details.

**4 Controlling the 1260 Infinity MDS**  
The Instrument Display

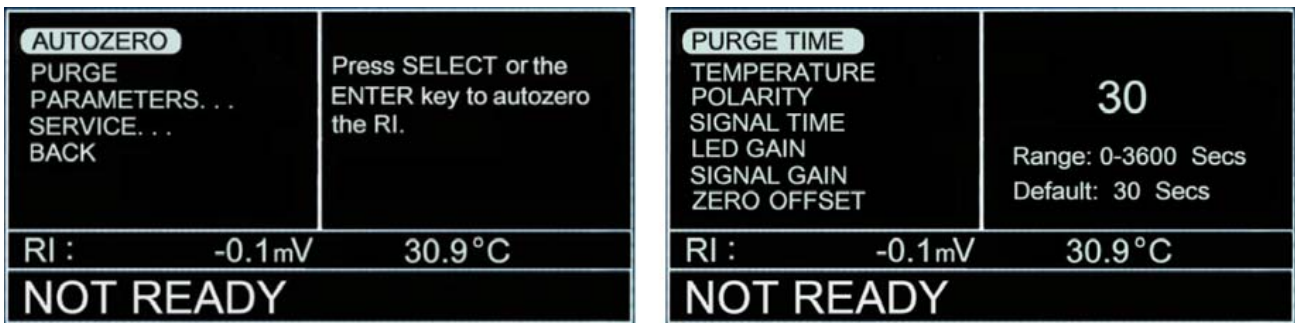
**The Detector and System Menu screens**

The Detector and System Menu screens are used to access the options (e.g. parameters and detector actions) available for the detector(s) connected and the actual instrument. These are all the menu screens accessed from the **Main Menu** screen. The content of the individual screen will depend on the option selected, however the screens will follow a similar format and consist of the following:

- Items List
- Information Area
- Detector Status Area
- System Status Area



**Figure 15** Layout of the MDS Detector and System Menu Screens



**Figure 16** RI Detector Menu Screens



The Items List consists of a scrollable list of items that are available for the detector or system menu option selected. The items available in the menu option will be one or more of the following:

- **Sub Menu...** This provides access to further options for the detector(s) connected and the actual instrument. Sub Menus are identified by the " ..." appended to the end of the item text.

---

- **Action** This is an action that can be performed on the selected detector e.g. RI Autozero or Purge DP.

---

- **Parameter** This is a specific parameter for the selected detector or system. It is either editable or read only. All editable values are stored when the instrument is powered off.

**NOTE**

Except Temperature and Laser Power.

- **Component** This is a component of the selected detector that can be controlled and it's state changed for example LS Laser On / Off or RI Purge Valve Open / Closed.

---

- **Back** This returns the menu screen up one level in the menu system.

In the Item List a ▼ and / or ▲ character can be displayed. This indicates that more items are available either down or up the list.

To navigate through the Item List use the up and down arrow keys on the keypad.

**NOTE**

During some actions the keypad will be locked with the Lock icon displayed in the System Status Area.

Some actions can be cancelled by pressing the **CE** key on the keypad. See "The Keypad" on page 61.

The Information Area for Sub Menu, Action, Component and Back items displays information about the currently highlighted item and how to select it. For the Component item the information displayed is dynamic and depends on the current state of the component e.g. LS Laser On or Off.

## 4 Controlling the 1260 Infinity MDS The Instrument Display

The Information Area for a highlighted Parameter item displays the current set value, the settable range for the parameter and the default value.

To edit a highlighted parameter press either the **Select**, **Enter** or right arrow keys on the keypad. The current set value of the parameter will now be highlighted in the Information Area. Either type in the required value from the keypad or use the up and down arrow keys to scroll through the available values. Press either the Enter or Select keys on the keypad to set the new parameter value. The set value will no longer be highlighted and the cursor returns to the Items List. If an invalid value is entered, the entry will be ignored and the parameter value reverts to the last valid value set.

To cancel an entry and return the cursor to the Items List press either the **CE** or left arrow keys on the keypad.

For read only parameter values Read Only and the value units will be displayed in the range and default value respectively.

The Detector Status Area displays the current detector selected and the detector signal and temperature outputs. For the two analog inputs only the outputs are displayed. The information displayed for the individual detectors is shown overleaf.

**Table 11** Status icon information

Detector	Displayed Detector Information		
RI	<Output> mV	<Temperature> °C	
VS	<DP Output> Pa	<IP Output> kPa	<Temperature> °C
LS	<15 ° Output> mV	<90 °Output> mV	<Temperature> °C
Analog In	Ch1 <Output> mV	Ch2 <Output> mV	

The System Status Area within the **Detector** and **System Menu** screens displays the system status states either Ready or Not Ready (see [Table 10](#) on page 67) or information about an action performed or a change in state of a component. The action or component information is displayed throughout the selected **Detector Menu** screens unless another action or component state change is selected. On returning to the **Main Menu** or **Home** screens the information is cleared.

The System Status Area also displays any detector errors that occur. The system status, action or component information will be replaced with the error. An error code and message will be displayed. For further details on system and detector errors see [“What Are Error Messages”](#) on page 133.

For detailed information on the individual **Detector** and **System Menu** screens see [“The RI Detector Menus”](#) on page 76 and following chapters.

## The RI Detector Menus

The 1260 Infinity MDS RI detector menus are used to access the specific options, actions, components and parameters for the RI detector. To access these menus select the **RI Detector** option from the **Main Menu** screen. The RI Detector menu screens that are available are as follows:

- The **RI Detector** Screen
- The **RI Parameters** Screen
- The **RI Service** Screen
- The **RI Service Settings** Screen

User required actions and parameters are located in the **RI Detector** and **RI Parameters** screens. Components and parameters for service and checking the RI detector are located in the **RI Service** and **RI Service Settings** screens. The complete structure of the MDS RI Detector Menu screens is as follows:

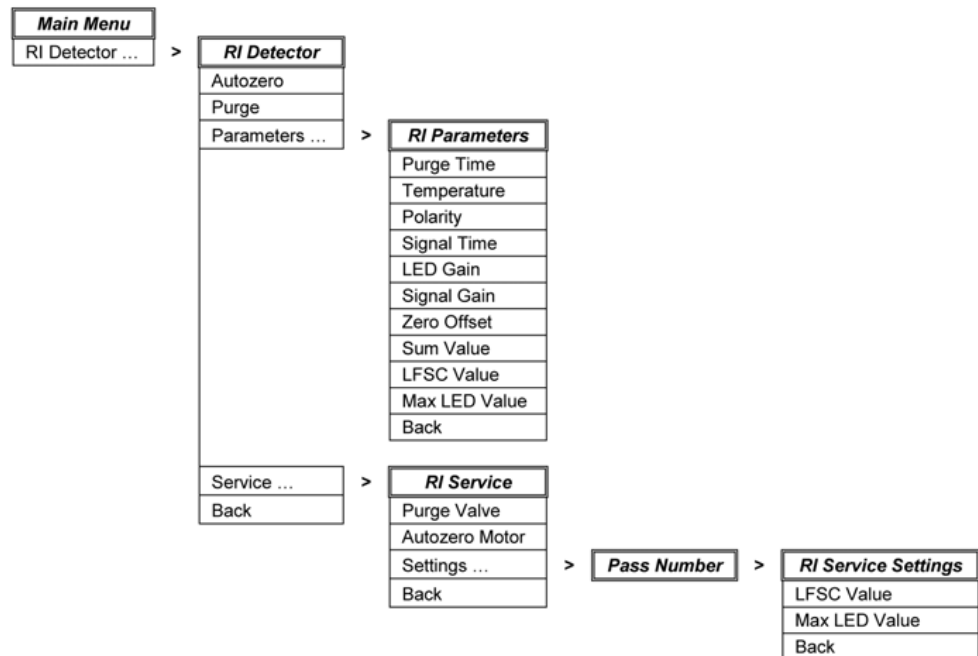


Figure 17 The MDS RI Detector Menus

## The RI Detector Menu Screen

The **RI Detector Menu** screen is used to access the autozero and purge actions for the RI detector, the detector parameters and service options. To access the RI Detector screen, select the **RI Detector** option from the **Main Menu** screen. The items available in the menu are as follows:

### Autozero

This action zeros the RI detector output to the set Zero Offset parameter value.

- |                   |  |
|-------------------|--|
| On Selecting A/Z  | The text <b>RI Autozero</b> is displayed in the System Status Area. The keypad is locked with the Lock icon displayed            |
| On A/Z Completion | The text <b>RI Autozero Completed</b> is displayed in the System Status Area. The keypad is unlocked with the Lock icon removed. |

#### NOTE

It is not possible to cancel the autozero command once selected.

### Purge

This action purges the RI detector for the set Purge Time parameter value.

- |               |   |
|---------------|---|
| On Selecting  | The text <b>RI Purge</b> is displayed in the System Status Area together with a count down clock of the RI purge time remaining. The keypad is locked with the Lock icon displayed. |
| On Completion | The text <b>RI Purge Completed</b> is displayed in the System Status Area. The keypad is unlocked with the Lock icon removed.   |
| On Cancelling | The text <b>RI Purge Cancelled</b> is displayed in the System Status Area. The keypad is unlocked with the Lock icon removed.   |

#### NOTE

To cancel the Purge action press the **CE** key on the keypad.

## 4 Controlling the 1260 Infinity MDS

### The RI Detector Menus

#### **Parameters...**

This sub menu option displays the **RI Parameters Menu** screen.

#### **Service...**

This sub menu option displays the **RI Service Menu** screen.

#### **NOTE**

Only Agilent-qualified engineers should access the RI Service Menu screen.

---

#### **Back**

This option returns the display back to the **Main Menu** screen.

## The RI Parameters Menu Screen

The **RI Parameters Menu** screen is used to access the RI detector parameters. To access the **RI Parameters** screen, select the **Parameters** sub menu option from the **RI Detector Menu** screen. The items available in the menu are as follows:

### Purge Time

This parameter defines the time delay between the RI purge valve opening and closing during a purge action selected from the **RI Detector Menu** screen. The time range and default value are shown below.

Range: 0 – 600 s

Default: 30 s

### Temperature

This parameter sets the temperature of the RI detector in increments of 1 °C. There is also the option to turn the heater off. The temperature range and default value are shown below:

Range: 30 – 60 °C with an Off option

Default: Off

### Polarity

This parameter sets the polarity of the RI detector output. If the polarity is set to "-ve", then the detector signal is flipped, i.e. sample peaks with a negative output in "+ve" mode become positive outputs in "-ve" mode. The polarity range and default value are shown below.

Range: +ve / -ve

Default: +ve

## 4 Controlling the 1260 Infinity MDS

### The RI Detector Menus

#### Signal Time

This parameter sets the applied smoothing level of the RI detector output in increments of 0.1 sec. The larger the value set, the more the signal will be smoothed. The signal time range and default value are shown below:

Range: 0.1 – 5 s

Default: 1 s

#### LED Gain

This parameter is used to adjust the sensitivity of the RI detector in order to bring the peak response back on-scale. Adjustment of this parameter alters the brightness of the LED. The LED gain range and default value are shown below.

Range: 50 – 100 %of full scale

Default: 100 %(Maxium sensitivity)

#### Signal Gain

This parameter sets the factor by which the RI detector output signal is amplified. The signal gain setting does not change the sensitivity of the detector, but merely amplifies the captured signal by the inputted factor. When setting the signal gain, both the signal and noise are simply amplified by the value set, so signal to noise ratios are unaffected. The signal gain range and default value are shown below.

Range: 0.1 – 5.0

Default: 1.0



## Zero Offset

This parameter is used to define the zero offset applied to the RI detector signal baseline after an autozero of the RI detector has been performed. For example setting a zero offset value of 10 mV would mean the signal baseline is autozeroed to 10 mV. The zero offset range and default value are shown below.

Range: -500 – +500 mV

Default: 0.0 mV

## Sum Value

This parameter is the current voltage sum value of the RI Detector in mV. This is the voltage sum of the two light intensities ( $I_1$  and  $I_2$ ) of the two halves of the RI cell. When the reference and sample sides of the RI Cell are flushed into the same solvent and an autozero action performed then typically this value should be  $\approx 1000$  mV. If the sum value is  $< 200$  mV the RI detector will be unable to perform an autozero action (RI Error #3410). Sum values below 200 mV may indicate that there is an air bubble in the cell, the cell is dirty or the RI detector needs servicing. See [“Maintaining and Cleaning the RI Flow Cell”](#) on page 109 for further details. This parameter is read only.

## LFSC Value

This parameter is the set LED Full Scale Current of the RI detector in mA. This parameter is set during manufacture to give the specified detector sensitivity. This parameter is read only.

## Max LED Value

This parameter is the maximum LED current value of the RI detector in mA. This is the absolute maximum current that should be passed through the LED. The value is determined from the manufacturer’s data sheet and includes a temperature de-rating factor. This parameter is read only.

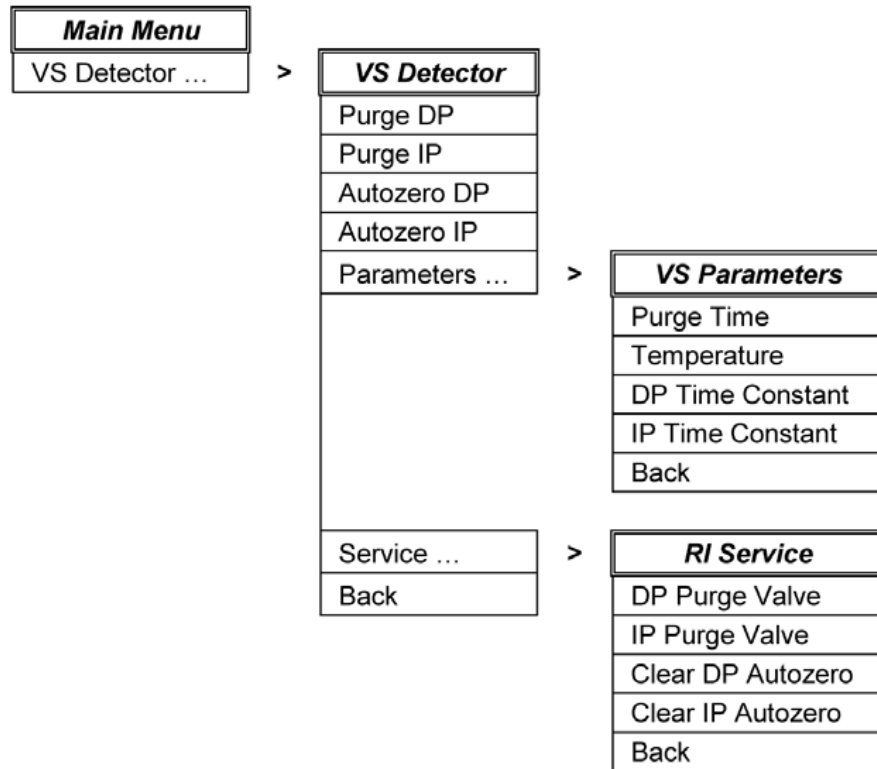
## The Viscometer Menus

The MDS Viscometer menus are used to access the specific options, actions, components and parameters for the viscometer. To access these menus select the **VS Detector** option from the **Main Menu** screen. The Viscometer menu screens that are available are as follows:

- The **VS Detector** Screen
- The **VS Parameters** Screen
- The **VS Service** Screen

User required actions and parameters are located in the **VS Detector** and **VS Parameters** screens. Components for service and checking viscometer are located in the **VS Service** screen.

The complete structures of the MDS RI Detector Menu screens are as follows:



**Figure 18** The MDS Viscometer Menus

## The VS Detector Menu Screen

The **VS Detector Menu** screen is used to access the purge and autozero actions for the viscometer, the detector parameters and service options. To access the **VS Detector** screen, select the **VS Detector** option from the **Main Menu** screen. The items available in the menu are as follows:

### Purge DP

This action purges the DP transducer of the viscometer for the set Purge Time parameter value.

On Selecting	The text <b>DP Purge</b> will be displayed in the System Status Area together with a count down clock of the DP purge time remaining. The keypad is locked with the Lock icon displayed.
On Completion	The text <b>DP Purge Completed</b> will be displayed in the System Status Area. The keypad is unlocked with the Lock icon removed.
On Cancelling	The text <b>DP Purge Cancelled</b> will be displayed in the in the System Status Area. The keypad is unlocked with the Lock icon removed.

### Purge IP

This action purges the IP transducer of the viscometer for the set Purge Time parameter value.

On Selecting	The text <b>IP Purge</b> will be displayed in the System Status Area together with a count down clock of the IP purge time remaining. The keypad is locked with the Lock icon displayed.
On Completion	The text <b>IP Purge Completed</b> will be displayed in the System Status Area. The keypad is unlocked with the Lock icon removed.
On Cancelling	The text <b>IP Purge Cancelled</b> will be displayed in the in the System Status Area. The keypad is unlocked with the Lock icon removed.

### Autozero DP

This action autozeros the DP transducer output of the viscometer to a fixed zero offset of 0 Pa.

On Selecting            The text **DP Autozero Completed** will be displayed in the System Status Area.

### Autozero IP

This action autozeros the IP transducer output of the viscometer to a fixed zero offset of 0 kPa.

On Selecting            The text **IP Autozero Completed** will be displayed in the System Status Area.

#### CAUTION

Autozero IP

Incorrect use of autozero IP can lead to poor results.

→ Only Autozero the IP when flow rate is stopped and with no pressure on the system.

---

### Parameters...

This sub menu option displays the **VS Parameters Menu** screen.

### Service...

This sub menu option displays the **VS Service Menu** screen.

#### NOTE

Only Agilent-qualified engineers should access the VS Service Menu screen.

---

### Back

This option returns the display back to the **Main Menu** screen.

## The VS Parameters Menu Screen

The **VS Parameters Menu** screen is used to access the Viscometer parameters. To access the **VS Parameters** screen, select the **Parameters** sub menu option from the **VS Detector Menu** screen. The items available in the menu are as follows.

### Purge Time

This parameter defines the time delay between the DP and IP purge valves opening and closing during a DP or IP purge action selected from the **VS Detector Menu** screen. The time range and default value are shown below.

Range: 0 – 600 s

Default: 60 s

### Temperature

This parameter sets the temperature of the Viscometer in increments of 1 °C. There is also the option to turn the heater off. The temperature range and default value are shown below.

Range: 30 – 60 °C with an Off Option

Default: Off

### Back

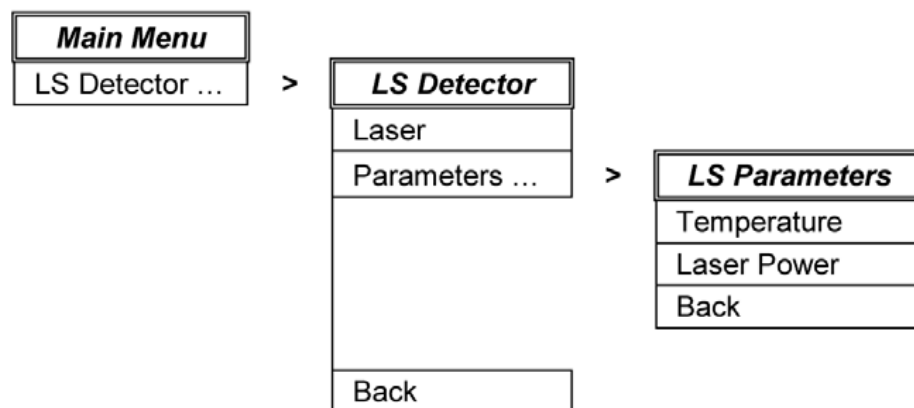
This option returns the display back to the **VS Detector Menu** screen.

## The LS Detector Menus

The 1260 Infinity MDS LS detector menus are used to access the specific options, component and parameters for the LS detector. To access these menus select the **LS Detector** option from the **Main Menu** screen. The **LS Detector menu** screens that are available are as follows:

- The **LS Detector** Screen
- The **LS Parameters** Screen

User required component and parameters are located in the **LS Detector** and **LS Parameters** screens. The complete structure of the MDS LS Detector Menu screens is as follows:



**Figure 19** The MDS LS Detector Menus

## 4 Controlling the 1260 Infinity MDS

### The LS Detector Menus

## The LS and LS/DLS Detector Menu Screen

The **LS Detector Menu** screen is used to access the Laser component for the LS detector and the detector parameters option. To access the **LS Detector** screen, select the **LS Detector** option from the **Main Menu** screen. The items available in the menu are as follows:

### Laser

This component is used to manually turn the LS Laser on and off. The information text displayed is dynamic and is dependent on the state of the Laser.

If the Laser is off (default position)

On Selecting	This will turn the Laser on with the text <b>LS Laser On</b> displayed in the System Status Area. The Laser status icon will be displayed as static in the LS Detector Area of the Home screen.
--------------	---

If the Laser is on

On Selecting	This will turn the Laser off with the text <b>LS Laser Off</b> displayed in the System Status Area. The Laser status icon will be displayed as flashing with a cross in the LS Detector Area of the Home screen.
--------------	--

### Parameters...

This sub menu option displays the **LS Parameters Menu** screen.

### Back

This option returns the display back to the **Main Menu** screen.



## The LS Parameters Menu Screen

The **LS Parameters Menu** screen is used to access the LS detector parameters. To access the **LS Parameters** screen, select the **Parameters** sub menu option from the **LS Detector Menu** screen. The items available in the menu are as follows.

### Temperature

This parameter sets the temperature of the LS detector in increments of 1 °C. There is also the option to turn the heater off. The temperature range and default value are shown below.

Range: 30 – 60 °C

Default: Off

### Laser Power

This parameter sets the power level of the LS Laser as a percentage. If a sample peak response is off-scale then reducing the Laser power from 100 % will bring the peak response back on-scale. The Laser power range and default value are shown below.

Range: 10 – 100 %

Default: 100 %

### Back

This option returns the display back to the **LS Detector Menu** screen.

## The Analog Input Menus

The 1260 Infinity MDS analog input menus are used to access the specific options, actions and parameters for the two independent analog inputs located at the rear of the MDS Control Unit. To access these menus select the **Analog Input** option from the **Main Menu** screen. The Analog Input menu screens that are available are as follows:

- The **Analog Input** Screen
- The **Analog Input Parameters** Screen

User required components and parameters are located in the **Analog Input** and **Analog Input Parameters** screens.

The complete structure of the MDS Analog Input Menu screens is as follows:

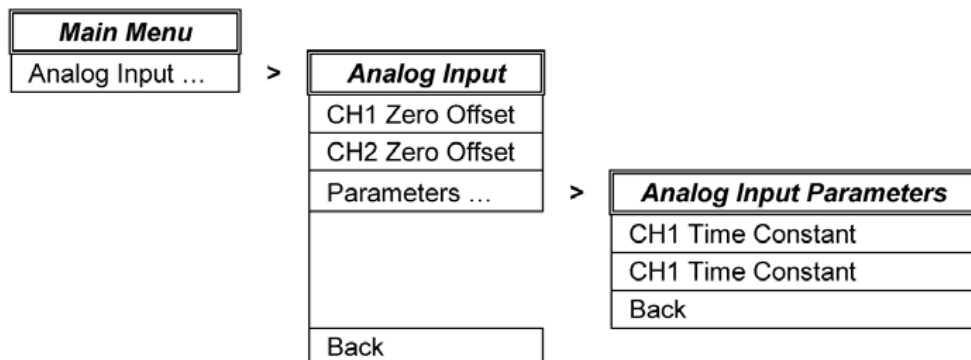


Figure 20 Analog Input Menu Structure

## The Analog Input Menu Screen

The **Analog Input Menu** screen is used to access the zero offset actions for the two analog input channels, the analog input parameters. To access the **Analog Input** screen, select the **Analog Input** option from the **Main Menu** screen. The items available in the menu are as follows:

### CH1 and CH2 Zero Offset

This action is used to apply a fixed zero offset of 10 mV or clear the zero offset to the output of the channel 1 or 2 analog input. When applying the 10 mV zero offset the MDS Control Unit calculates the offset required to bring the current output value for the channel 1 or 2 analog input to 10 mV. This value is stored and applied to the output from then on. The information text displayed is dynamic and is dependent on whether a zero offset has been applied or not.

If no zero offset is being applied (default position):

On Selecting	This will apply the zero offset with the text <b>Zero Offset On</b> displayed in the System Status Area.
--------------	--

If a zero offset is being applied:

On Selecting	This will clear the zero offset with the text <b>Zero Offset Off</b> displayed in the System Status Area.
--------------	---

### Parameters...

This sub menu option displays the **Analog Input Parameters Menu** screen.

### Back

This option returns the display back to the **Main Menu** screen.

## 4 Controlling the 1260 Infinity MDS

### The Analog Input Menus

## The Analog Input Parameters Menu Screen

The **Analog Input Parameters Menu** screen is used to access the parameters for the two analog input channels. To access the **Analog Input Parameters** screen, select the **Parameters** sub menu option from the **Analog Input Menu** screen. The items available in the menu are as follows:

### CH1 and CH2 Time Constant

This parameter sets the applied smoothing level for the output of the channel 1 or 2 analog input in increments of 1 s. The larger the value set, the more the signal will be smoothed. The CH1 and CH2 time constant range and default value are shown below.

Range: 0 – 10 s

Default: 1 s

### Back

This option returns the display back to the **Analog Input Menu** screen.

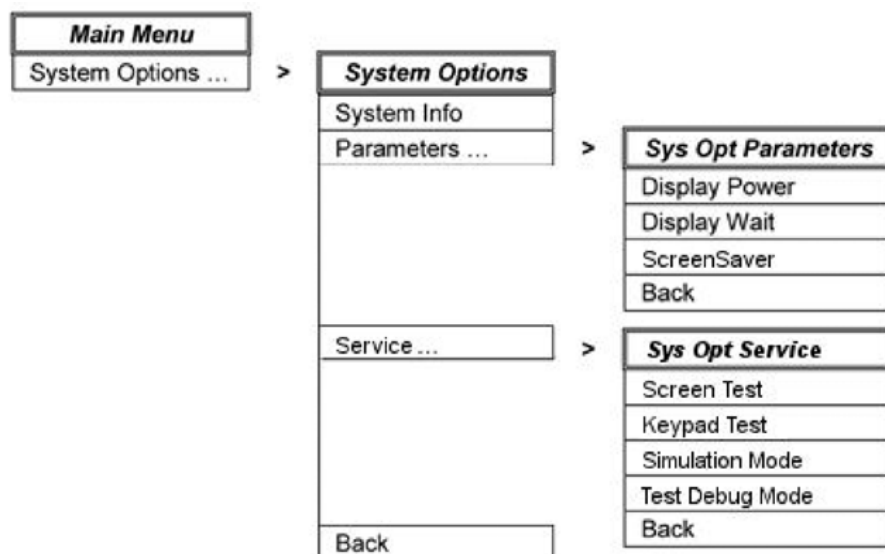
## The System Options Menu

The MDS System Options menus are used to access system information and parameters for the system. To access these menus select the **System Options** option from the **Main Menu** screen. The **System Options menu** screens that are available are as follows:

- The **System Options** screen
- The **System Parameters** screen
- The **System Service** screen

User required parameters are located in the System Options Parameters screen.

The complete structure of the MDS System Options Menu screens is as follows:



**Figure 21** MDS System Options Menu Structure

## The System Options Menu Screen

The **System Options Menu** screen is used to access the system information and the system parameters. To access the **System Options** screen, select the **System Options** option from the **Main Menu** screen. The items available in the menu are as follows:

### System Info

The System Info screen displays the firmware versions of the modules installed in your MDS instrument. It consists of the firmware versions used in the MDS Control Unit and the installed MDS detectors. The full list of modules is shown below.

- MMI – Used in the MDS Control Unit
- STB - Used in the MDS Control Unit
- ACQ - Used in the MDS Control Unit
- RI - Used in the MDS RI Detector
- VS - Used in the MDS Viscometer
- LS - Used in the MDS LS Detector

The information displayed for each module is the Version and Build of the firmware in use. For example MMI 1.0 B89 is firmware version 1.0 build 89 for the MMI module.

### Parameters

This sub menu option displays the **System Options Parameters Menu** screen.

### Service

This sub menu option displays the Service Options Menu screen. To action any of the options within this Service screen a Pass Code is required.

This Service Menu screen should only be accessed by an Agilent engineer.

### Back

This option returns the display back to the **Main Menu** screen.

## The System Options Parameters Menu Screen

The **System Options Parameters Menu** screen is used to access the system options parameters. To access the **System Options Parameters** screen, select the **Parameters** sub menu option from the **System Options Menu** screen. The items available in the menu are as follows:

### Display Power

This parameter sets the display power (brightness) of the display screen in increments of 25 %. Therefore a set value of 100 % would be full brightness. The display power range and default value are shown below.

Range: 25 – 100 %

Default: 75 %

### Display Wait

This parameter sets the display wait of the display screen in increments of 1 min. It is the time of inactivity before the system automatically reduces the display power to minimum, 25 %. This is done to extend the lifetime of the display screen. If required there is the option to turn this off so that the display power is not automatically set to minimum. The display wait range and default value are shown below.

Range: 1 – 120 min with an Off option

Default: 20 min

### Screen Saver

This parameter controls the screen saver behaviour.

Range: On – Off

Default: On

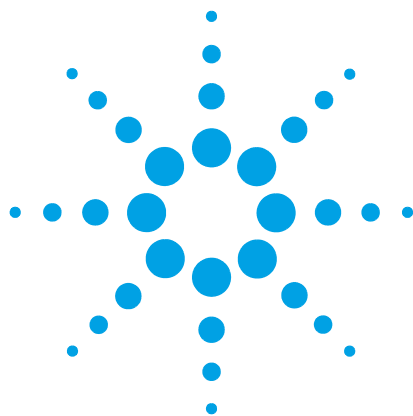
### Back

This option returns the display back to the **System Options Menu** screen.

## **4 Controlling the 1260 Infinity MDS**

### **The System Options Menus**





## 5 Operating the 1260 Infinity Multi-Detector Suite (MDS)

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Preparing the Viscometer	99
Preparing the LS and DLS Detector	100
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Mobile Phase Priming	101
Solvent Information	102

This chapter describes the operational parameters of the instrument.



## Using the Instrument

### Preparing the 1260 Infinity Multi Detector Suite

- Locate the 1260 Infinity MDS in an area that does not experience large temperature changes. See [“Physical Specifications”](#) on page 33 for details.
- Do not place solvent bottles on top of the instrument.
- Always pre-mix and de-gas the mobile phase.
- Do not allow pumps to mix solvents for the mobile phase. Use only isocratic conditions.
- Always use tubing of 1.0 mm i.d. (0.040 in) or greater on the RI detector's/viscometer's purge waste outlet.
- When using buffer solutions, flush the system with water before switching it off. Growth of algae should be avoided.
- Always use an inline filter with Light scattering detection. Use a filter kit for inline filtering (High pressure filter kit (5067-4638)).
- When purging the reference side of the RID cell and viscometer transducers, always use mobile phase that has eluted through the analytical column. Allow mobile phase to flow through at least one column volume before purging the detector/transducers. See [“Purging the Detectors”](#) on page 106.
- Priming and Purging the System: When the solvents have been exchanged or the system has been turned off for a certain time (for example, overnight) oxygen will re-diffuse into the solvent channel. Therefore priming and purging of the system is required before starting an application.
- Always filter solvents through  $\leq 0.1 \mu\text{m}$  filters. Small particles reaching the MDS will result in poor performance of light-scattering based detection.

## Preparing the RI Detector

- Never subject the detector to back pressures greater than 350 kPa (50 psi). High pressures can cause internal fluid leakage or break the cell, which is difficult to replace.
- Always fit the 5PSI back-pressure regulator on the waste outlet so that the reference side of the RI cell can be flushed during a purge.
- Keep the RI cell clean, see [“Maintaining and Cleaning the RI Flow Cell”](#) on page 109.

## Preparing the Viscometer

- Never subject the viscometer to backpressures greater than 50 kPa (7 psi). High pressures can cause internal fluid leakage.
- Never allow the DP transducer to exceed  $\pm 5000$  Pa and the IP transducer to exceed 100 kPa.
- When transferring the viscometer between solvents, always start the pump at reduced flow rates  $< 0.2$  mL/min. See [“Purging the Viscometer”](#) on page 107.
- Note that the DP value may be offset with the autozero function at anytime.
- Autozero should be applied to the IP value only if the flow through the viscometer is zero and the LC pump purge valve is opened to remove pressure in the system.

## 5 Operating the 1260 Infinity Multi-Detector Suite (MDS)

### Using the Instrument

#### Preparing the LS and DLS Detector

- Always triple-filter the mobile phase through  $\leq 0.1 \mu\text{m}$  filters.
- Always filter the samples prior to injection.
- If the solvent is changed, make sure that the two solvents are miscible. If not, use an intermediate solvent such as isopropanol.
- If you are using a buffer, make certain that the buffer salt is soluble in the mobile phase.
- If a new column is used, flush it with five volumes of mobile phase before connecting it to the detector.
- The cell should not be allowed to dry out.
- Keep the LS cell clean, see [“Maintaining and Cleaning the LS Flow Cell”](#) on page 115.

## Pumping Systems

It is recommended to use a high-performance pumping system with no flow pulses to maximise viscometer performance. Inconsistent solvent flow will result in poor system performance.

A backpressure regulator maybe necessary on certain pumps in order to minimize pump pulsation. This can also be achieved by the column itself or a coil of 0.127 mm (0.005 in) i.d. tubing placed between the pump and the injector.

### Mobile Phase Priming

#### NOTE

Make sure that the solvent within the detector(s) is completely miscible with the intended solvent to be used. If this is not the case, the detector(s) must be flushed through with an intermediate solvent (e.g., acetone), which is miscible with both solvents.

---

The entire flow system, consisting of pump, damper, injector, column, MDS, etc., must be primed with the mobile phase. Flush the entire system with 5 – 10 times the system volume until a steady baseline is obtained. The mobile phase should be pre-mixed and degassed before use. Alternatively, the mobile phase can be degassed online by a solvent degassing system.

## **Solvent Information**

The high sensitivity of the instrument makes it imperative that a high standard of solvent purity and general system cleanliness be maintained.

Use solvent reservoir filters to filter solvents. Always degas solvents before use.

Eluents containing non-volatile mineral acids or bases should be avoided.

Do not use strongly basic (pH 10 or higher) solutions, as these will etch the quartz cell window of the MDS RI detector.

Some solvents may corrode the detector(s) installed if they are not removed from the system after use. However, certain solvents can be left in the detector(s) at the end of operation. For example, water, acetonitrile, 2-propanol, and paraffin-derived hydrocarbons are quite innocuous. They may be left in the MDS overnight or over a weekend.

### **Buffers**

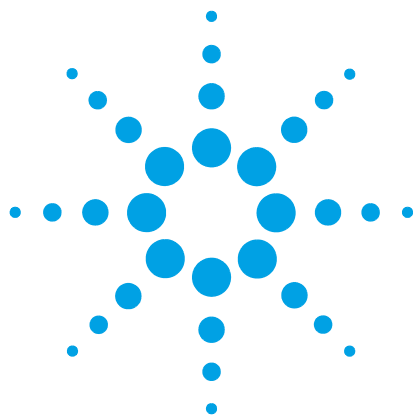
The use of buffers (even non-corrosive buffers) can lead to salt precipitation in the MDS detector(s) and tubings installed. Keep the solvent flowing at a reduced rate to eliminate the possibility of salt precipitation.

### **Tetrahydrofuran**

THF is prone to auto-oxidization. Keeping the solvent flowing at a reduced rate minimizes re-stabilization time upon startup of the chromatographic system.

### **Organohalogenes**

A small amount of flow will minimize the amount of corrosive chloride impurities in the cell, when using solvents such as methylene chloride and chloroform.



## 6 Optimizing Performance

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



## **6** Optimizing Performance

### Location of the 1260 Infinity MDS

## **Location of the 1260 Infinity MDS**

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

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



## Routine Maintenance of the MDS System

A contaminated MDS can lead to noise and drift problems that are often mistakenly attributed to other areas of the system. Many cases of performance degradation in sensitive instruments with flow-through cells are traceable to cell contamination.

The high sensitivity of the instrument makes it imperative that a high standard of solvent purity and general system cleanliness be maintained. To insure this and to decrease the amount of cleaning required, use:

- filtered solvents with solvent reservoir filters,
- an in-line filter between the pump and detector,
- a pre-column to protect the cell from contamination.

However, contamination from trapped particulates, from precipitates, or from thin films of residues can still occur. If the instrument performance deteriorates, we recommend cleaning the cell. See [“Maintaining and Cleaning the RI Flow Cell”](#) on page 109 and [“Maintaining and Cleaning the LS Flow Cell”](#) on page 115.

If cleaning the various detectors does not fix the problems, contact your Agilent service representative.

## Purging the Detectors

To ensure optimum performance of the 1260 Infinity MDS, all detectors must be purged to ensure they have been fully flushed with mobile phase prior to use.

### NOTE

When flushing the detector between immiscible solvents, initial flushing into an intermediate miscible solvent such as acetone or methanol is recommended before final transfer into the new solvent.

---

### Purging the RI Detector

The RI detector can be flushed with any miscible solvent.

- 1 Introduce the solvent at a reduced flow of typically no higher than 0.5 mL/min into the flow cell.
- 2 Flush the sample cell for a minimum of 5 min.

### NOTE

Monitor the pressure during the flushing procedure to ensure that excessive pressure does not occur.

---

- 3 Perform an RI Purge action with a 5 min purge time setting.
- 4 Repeat step 2 and 3 if required.

### NOTE

Depending on the solvent choice, some equilibration of the detector may be required. This may last several hours.

---

## Purging the Viscometer

The viscometer detector can be flushed with any miscible solvent.

- 1 Before starting to flush the viscometer open the IP purge to allow the solvent flow to bypass the bridge and flush out the IP transducer.
- 2 Pumping at 1 mL/min flush the IP transducer for 15 min to ensure the solvent is fully exchanged.
- 3 Now open the DP purge valve and close the IP valve. The flow solvent will now flush the first half of the bridge and the DP transducer, passing to waste from the transducer. Again flush for 15 min at a flow of 1 mL/min .
- 4 Closing the DP purge valve will direct all the flow to the viscometer bridge and start flushing out the delay columns. Each column has a flushing volume of  $\approx 10$  mL and therefore the total delay column flushing time will be approximately one hour at 1 mL/min.
- 5 If lower flow rates are used it is advisable to flush the viscometer overnight.

### NOTE

Monitor the pressure during the flushing procedure.

Do not exceed  $\pm 5000$  Pa (DP) and  $\pm 100$  kPa (IP) as this may damage the pressure transducers.

---

**Before operation the viscometer should be again purged and the DP zeroed:**

- 1 Perform 3x IP Purge action with a 5 min purge time setting.
- 2 Perform 3x DP Purge action with a 5 min purge time setting.
- 3 Zero the DP out-put.

The detector is fully equilibrated and ready to use.

### NOTE

Depending on the solvent choice, some equilibration of the detector may be required. This may last several hours.

---

## Purging the Light-Scattering Detector

The LS detector can be flushed with any miscible solvent.

### NOTE

Monitor the pressure during the flushing procedure to ensure that excessive pressure does not occur.

Depending on the solvent choice, some equilibration of the detector may be required. This may last several hours.

---

- 1 Introduce the solvent at a reduced flow of typically no higher than 0.5 mL/min into the flow cell.
- 2 Flush the sample cell for a minimum of 5 min.
- 3 Increase the flow to the operating flow (typically 1.0 mL/min).

The detector is fully equilibrated and ready to use.

## Purging the Dynamic Light-Scattering Detector

The LS detector can be flushed with any miscible solvent.

### NOTE

Monitor the pressure during the flushing procedure to ensure that excessive pressure does not occur.

Depending on the solvent choice, some equilibration of the detector may be required. This may last several hours.

---

- 1 Introduce the solvent at a reduced flow of typically no higher than 0.5 mL/min into the flow cell.
- 2 Flush the sample cell for a minimum of 5 min.
- 3 Increase the flow to the operating flow (typically 1.0 mL/min).

The detector is fully equilibrated and ready to use.

## Optimizing RI Performance

### Maintaining and Cleaning the RI Flow Cell

The high sensitivity of the RI detector makes it imperative that a high standard of solvent purity and general system cleanliness be maintained. A contaminated flow cell can lead to noise and drift problems that are often mistakenly attributed to other areas of the system.

The use of filtered solvents with solvent reservoir filters, in-line filter between the pump and detector and a pre-column will protect the cell from contamination and decrease the amount of cleaning required.

However, contamination from trapped particulates or bubbles, from precipitates, or from thin films of residues can still occur.

### Flushing the RI Flow Cell

Connect the tubing directly from the pump outlet to the MDS RI Detector INLET port to bypass the column. Connect the outlet port to waste.

#### NOTE

*Do not* connect the MDS detectors in series when performing a cleaning procedure.

---

Some materials should be injected directly into the RI Detector by syringe due to their high corrosivity or safety risk.

If you encounter a large backpressure while flushing the cell, proceed with caution as you could rupture the cell.

The following recommendations may be applicable to a variety of cleaning conditions:

## 6 Optimizing Performance

### Optimizing RI Performance

#### Liquid purging

Clean all internal lines of the RI detector by injecting cleaning solution with Purge off, and inject cleaning solution again with Purge on.

Particulate matter can be removed by forcing liquid through the cell using a syringe. Sometimes it helps to reverse flow and inject in the OUTLET port. If liquid purging does not work, try gas purging.

See “[Purging the Detectors](#)” on page 106 for further details on the correct purge procedure.

#### Gas purging

Replace cell liquid with a volatile solvent (for example acetone). Purge and dry the cells with clean gas at about 15 psi. Gas flowing through the cell will sometimes dislodge particles. Also try reversing the flow direction.

For difficult entrapments, follow the above gas purge procedure with a liquid purge. When liquid hits a particle in a dry cell, it is generally more effective than a continuous liquid purge.

#### Entrapped bubbles

If you suspect that you have a problem with entrapped bubbles, purge with propan-2-ol.

#### NOTE

Heating the flow cell during the cleaning procedure can help remove marginally soluble material.

## Alternative Cell Cleaning Procedures

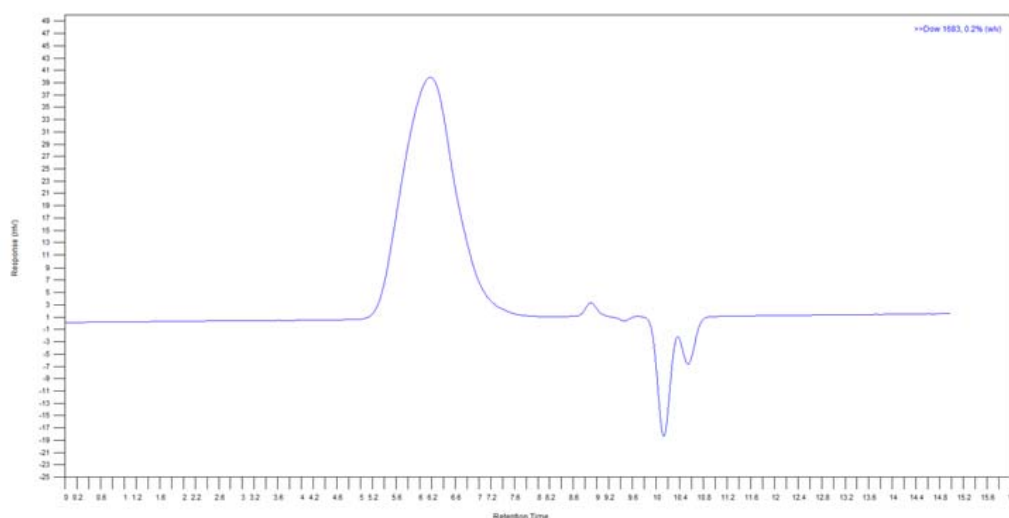
If buffers or solutions of high salt content have been in use, the cells may be contaminated by precipitated salt. Flushing with large amounts of distilled, deionized water at 1 mL/min, for up to several hours is the simplest clean-up procedure. An elevated cell temperature will speed up dissolution.

The water wash can be acidified if the precipitated salt is more soluble in acidic solutions (do not exceed pH 2). However, do not use strongly basic (pH 10 or higher) solutions, as these will etch the refractive index cell.

If contamination is suspected when a non-aqueous solvent is in use, flush the cells with a solvent that is

- miscible with your mobile phase,
- a good solvent for the predicted contaminant.

A typical RI chromatogram is shown below.



**Figure 22** Typical RI chromatogram

## Optimizing Viscometer Performance

The Agilent 1260 MDS Viscometer employs the fluid flow equivalent of the analogous Wheatstone bridge electrical circuit; see “[The 1260 Infinity MDS Viscometer](#)” on page 163 for detailed theory of operation.

When mobile phase is pumped into the viscometer, solvent travels down a bridge of four capillaries of equal resistance. However, when the sample is introduced an imbalance is created. This change in pressure,  $\Delta P$ , is proportional to the specific viscosity and the instrument makes a true differential measurement directly, making it very sensitive to changes in viscosity.

Consequently, the performance of the viscometer is highly dependent on the pump performance. The solvent delivery module must be thoroughly purged, and able to deliver solvent at the desired flow rate without pulsation.

Viscometer performance is also dependent on how thoroughly the detector has been purged. Any air bubbles or residual traces of previous solvent in the system will result in poor results. It is therefore important to follow the purging procedures that are described in this manual and allow enough time for the detector to equilibrate in the desired solvent.



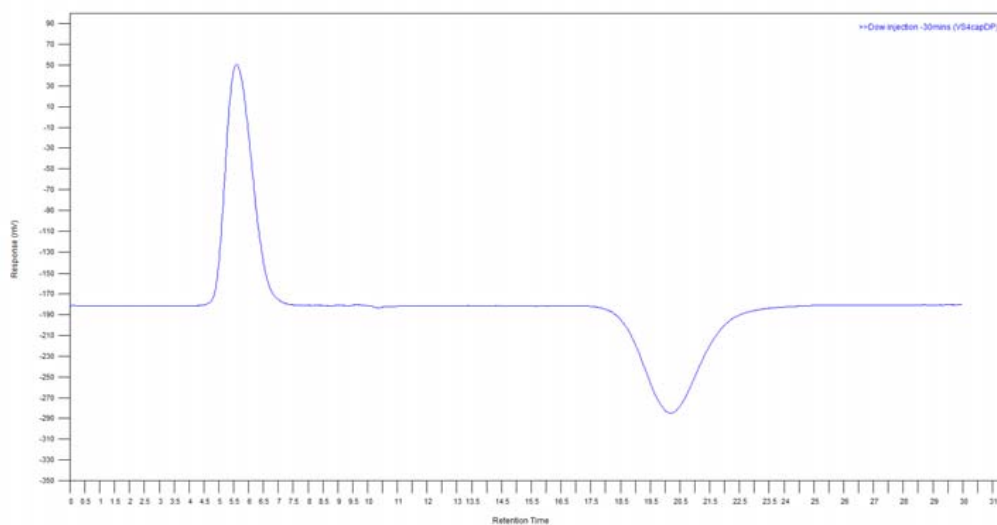
## Delay/Hold-Up Columns

The delay columns play an important role in the viscometer operation. While the polymer sample is passing through the detector's capillaries, the hold-up columns delay the polymer elution across one side of the viscometer bridge leading to an imbalance in the pressure across the viscometer.

Once the sample passes through the delay columns, it creates a "breakthrough" peak. The hold-up columns are of sufficient volume that the breakthrough peak elutes after the sample peak has eluted.

The delay volume is provided by 2x delay columns that are fitted internally to the viscometer assembly.

A typical chromatogram showing the "breakthrough" peak is shown below.



**Figure 23** Typical viscometer chromatogram

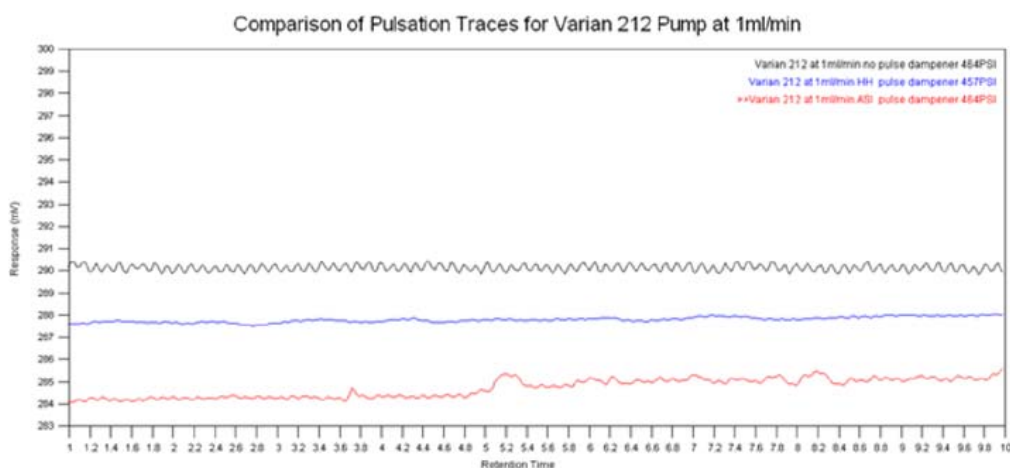
An early eluting "breakthrough" peak (too close to the DP sample peak response) may indicate a problem with the stability of the delay columns.

Typically, the baseline noise that is expected from a viscometer should be 0.25 – 0.5 mV. Greater values are often a result of poor pump performance. The pattern of baseline noise observed on a viscometer can indicate the source of the problem.

## 6 Optimizing Performance

### Optimizing Viscometer Performance

A regular noise pattern as shown by the black trace in [Figure 24](#) on page 114 is typical for a viscometer baseline with a pulsating pump. This pattern can be suppressed by using a pulse dampening device and/or increasing the backpressure on the pump. Liquid chromatography pumps function more effectively when they are working against some pressure (>1.5 MPa).



**Figure 24** Example of typical viscometer baseline problems

High levels of baseline noise can also be seen when the viscometer hasn't been purged correctly (see [“Purging the Detectors”](#) on page 106) and/or the viscometer hasn't been given enough time to equilibrate before starting the analysis.

## Optimizing Light Scattering Performance

### Maintaining and Cleaning the LS Flow Cell

A typical mistake when using light scattering, especially with a new unit, is pumping the fines that are typically present in new columns into the light scattering cell. This causes a high baseline offset and in the worst case a dirty flow cell requiring cleaning. When using light scattering, it is highly recommended to flush new columns with at least 10 column volumes of solvent before connecting them to the light scattering detector.

In addition, another common mistake is to turn the pump on and off without proper flow rate control. Turning the pump from zero flow to 1 mL/min has the effect of shocking the column bed. This again releases fines from the column and radically affects the light scattering baselines – causing increased off-set and spikes. It is therefore recommended to change the flow rate by no more than 0.2 mL/min/min.

### Aqueous Eluents

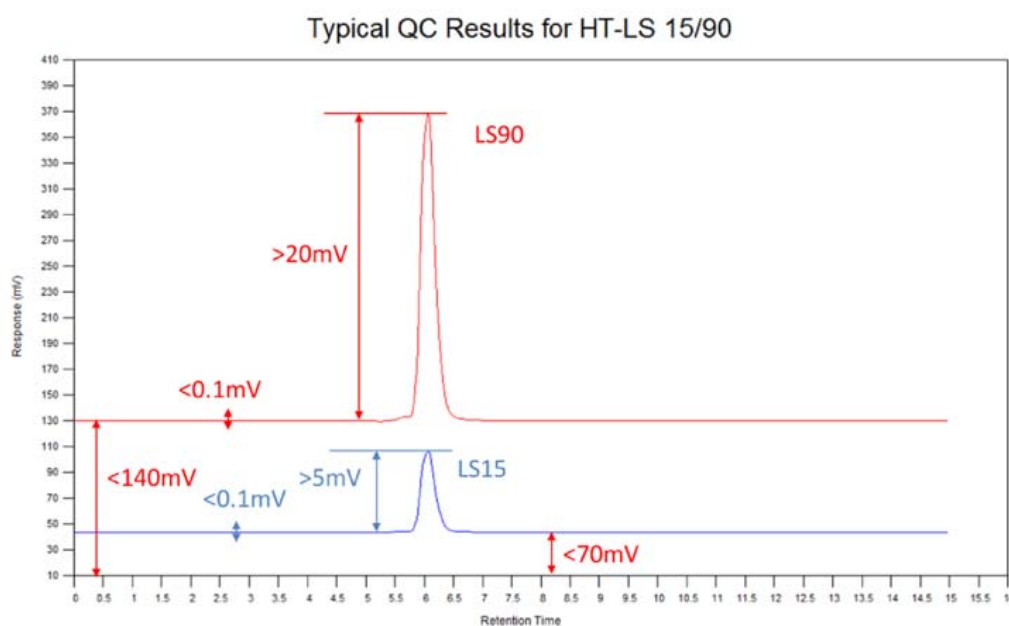
Water is a particularly poor scattering media and although this has the positive effect that the baseline offset is particularly low, the negative effect is that any particle (dust) in the water scatters strongly, giving rise to a spiky baseline. To counteract this spiky baseline it is essential that the eluent is filtered down to 0.02  $\mu\text{m}$ , the columns are thoroughly flushed to waste before being connected to the light scattering detector, and an inline filter is fitted after the column immediately before the light scattering detector to minimize particles reaching the flow cell. The addition of buffers to the water, as long as they are fully soluble, tend to have the effect of suppressing the dust and spiky appearance of the baseline, but again filtration is essential.

The most common symptoms of a contaminated flow cell are high baseline noise and/or the light scattering signals off-scale. Sinusoidal oscillation on the light scattering baselines (if not temperature related) is also a strong indication that the cell is dirty and requires cleaning.

## 6 Optimizing Performance

### Optimizing Light Scattering Performance

The results that are shown in [Figure 25](#) on page 116 demonstrate the typical QC test carried out on all the Agilent light scattering detectors with the indicated pass specifications. The test probe is a polystyrene standard  $M_p=96000$  g/mol with a PDI of 1.03 dissolved in THF. The solution is prepared at 1.0 mg/mL and 100  $\mu$ L injected into a 1 mL/min flow stream of THF on a PLgel MIXED-C column.



**Figure 25** Example QC data from a Light-scattering detector

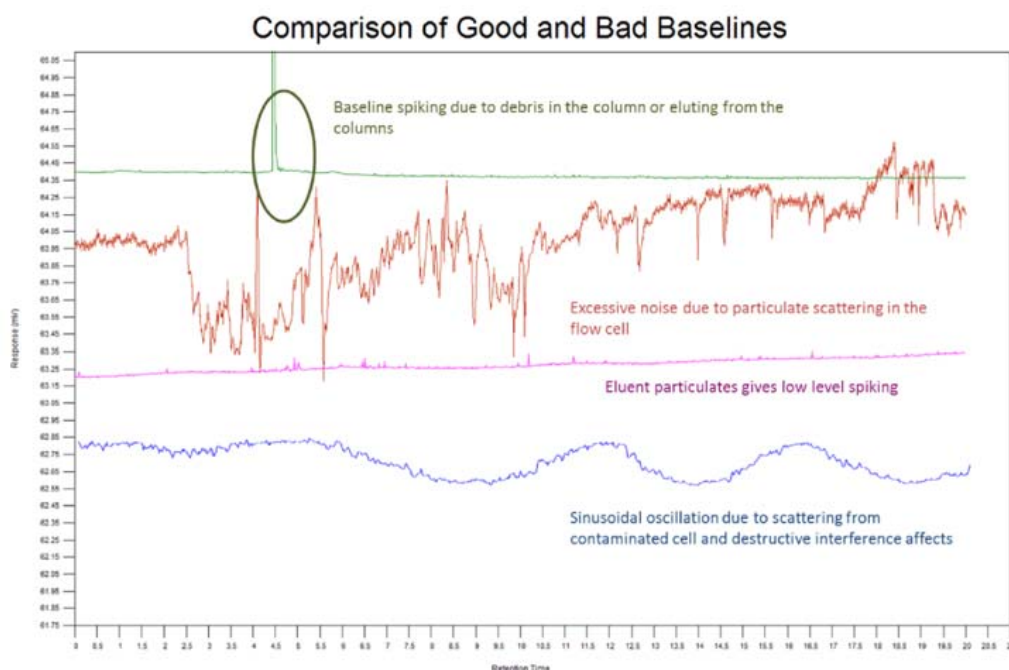
It should be remembered that the light scattering offset is a direct measurement of the solvent or eluent scattering; and if the solvent is “dirty” and contains particulates the scattering will be higher. The table below, compiled from empirical data should be used as a general guideline for the expected offsets of a few commonly used eluents. The best performance and the lowest solvent scattering are generally obtained when the eluent has been filtered to 0.02  $\mu\text{m}$  (best, or  $<0.2$   $\mu\text{m}$  acceptable).

Table of typical solvent scattering values under conditions specified in the comment column.

**Table 12** LS baseline offsets in common solvents

Eluent	LS 15° Baseline	LS 90° Baseline	Comments
Tetrahydrofuran (THF)	<140 mV	<70 mV	1 mL/min flow
HPLC Water (0.02 % Sodium Azide)	<200 mV	<200 mV	

As a visual indication of good and bad baselines see the graph below. The graphic shows some of the symptoms that are prevalent with light scattering and the common reasons for such behavior.



**Figure 26** Example of typical light-scattering baseline problems

In most cases, the cell can be cleaned, and particulate matter can be removed by pumping pure solvent through it for several hours.

## Shutting-down the Instrument

### CAUTION

Corrosive and oxidizing solvents

Some solvents may corrode the detector(s) if they are not fully flushed from the system after use.

→ Remove corrosive solvents thoroughly from the entire system before switching off the instrument.

---

### NOTE

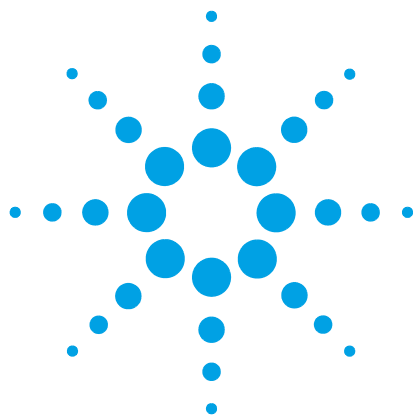
A continuous low flow rate of solvent through the MDS should be maintained during short term shut-down, especially if buffers, tetrahydrofuran or organohalogens are used.

---

**If the instrument is not to be used for short periods of time, such as overnight or weekend, it is recommended to follow the shut-down procedure outlined below:**

- 1 Reduce the flow to 0.5 – 0.1 mL/min.
- 2 Leave the MDS powered on with the installed detector(s) at temperature, if heating is used, on overnight and on weekends.

This will keep the MDS detector(s) close to operating conditions and minimize stabilization time.



## 7 Troubleshooting and Diagnostics

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



## Troubleshooting

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

Malfunctions within the MDS can arise from four general sources:

- the MDS itself is operating outside specification,
- the MDS detector(s) installed can be dirty or operating outside specification,
- the LC system can have a broken, dirty, or non-optimally operating component, but the problem is manifesting itself in the MDS detector(s) installed,
- a mobile phase and/or column problem, which by its very nature is spread throughout the LC system but appears as a malfunction of the MDS detector(s) installed.

To troubleshoot the MDS detector(s) installed, you must be able to separate the MDS detector(s) performance within the LC system from its performance outside the LC system. This section begins with guidelines for testing the MDS, including the installed detector(s), as a stand-alone.

Section “[System Problems](#)” on page 122 lists the observed problems with the detectors along with possible causes and the suggested solutions.



## Troubleshooting an LC System

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

Begin troubleshooting by adding the pump to the MDS first and finish by adding the column last. If the MDS has more than one detector installed or there is another type of detector available use them to aid in troubleshooting.

For example, if the MDS has a MDS RI and a MDS LS detector installed and only the MDS RI Detector has a noisy baseline. One possible implication is that the noise arises from pressure fluctuations to which the refractive index detector is more sensitive. If both detectors are showing noise, a power line current may be indicated. If both detectors show anomalous baseline performance, such as huge peaks that continue indefinitely, a bleed-off problem (material from the column or immiscible solvents trapped in the system) is more likely.

## System Problems

### Excessive baseline noise

#### Probable cause

- 1 Air bubble in pump
- 2 Particulates in the mobile phase
- 3 Formation of gases in the mobile phase reservoir
- 4 Particles shedding from analytical column
- 5 Environmental temperature fluctuations
- 6 Damper has inadequate damping capacity for system
- 7 Damper is operating at a pressure where performance is poor
- 8 Incorrect compressibility setting for mobile phase
- 9 External detector inputs/outputs are faulty

#### Suggested actions

- Purge the pump heads. Use only premixed/degassed mobile phase.
- Filter the mobile phase
  - Flush the system thoroughly with the filtered mobile phase to waste and then allow to stabilise.
- Use only premixed/degassed mobile phase. Use an in-line degasser.
- Condition the columns by flushing with solvent prior to use.
  - Fit an in-line filter after the column.
- Move instrument to a more stable environment, see [“Location of the Instrument”](#) on page 31 for more information.
- Add a second damper.
- Add a restrictor coil/backpressure regulator between pump and injector valve.
- Use correct pump compressibility setting for mobile phase solvent.
- Repair or replace defective recorder or cable. Investigate external detector.

## Excessive baseline drift

### Probable cause

- 1 Environmental temperature fluctuations
- 2 Formation of gases in the mobile phase reservoir
- 3 System not fully equilibrated

### Suggested actions

Move instrument to a more stable environment, see [“Location of the Instrument”](#) on page 31 for more information.

Use only premixed/degassed mobile phase.  
Use an in-line degasser.

Allow sufficient time for the system to equilibrate with the mobile phase and environment.

## Long retention times

### Probable cause

- 1 Pump flow rate fluctuating
- 2 Solvent leak within the system
- 3 System not fully equilibrated

### Suggested actions

Check pump performance.

Check system for solvent leaks.

Allow sufficient time for the system to equilibrate with the mobile phase and environment.

## RI Detector Problems

### Excessive baseline noise

#### Probable cause

- 1 Air bubble in pump
- 2 Air bubble in detector tubing
- 3 Contaminated flow cell
- 4 Environmental temperature fluctuations
- 5 Faulty light source

#### Suggested actions

- Purge the RI detector thoroughly to remove air bubble.
- Use only degassed mobile phase.

Elevate waste reservoir above the level of flow cell.

Clean the flow cell thoroughly. Refer to [“Maintaining and Cleaning the RI Flow Cell”](#) on page 109 for further information.

Move instrument to a more stable environment, see [“Location of the Instrument”](#) on page 31 for more information.

Please contact your Agilent service representative.

### Excessive baseline drift

#### Probable cause

- 1 Detector not fully equilibrated with environment
- 2 Contaminated flow cell
- 3 Damaged flow cell
- 4 Solvent in reference cell has deteriorated/oxidised

#### Suggested actions

Allow sufficient time for the system to thermally equilibrate with its environment

Clean the flow cell thoroughly. Refer to [“Routine Maintenance of the MDS System”](#) on page 105 for further information.

Check for liquid in front drain tube or drip gutter. Contact Agilent for further assistance

Purge the RI detector to refill the reference cell. Add antioxidant to mobile phase

## Flat baseline with no noise

### Probable cause

- 1 Faulty light source

### Suggested actions

Please contact your Agilent service representative.

## Low sensitivity

### Probable cause

- 1 Contaminated flow cell

### Suggested actions

Clean the flow cell thoroughly. Refer to [“Routine Maintenance of the MDS System”](#) on page 105 for further information.

## “Mirrored” peaks

### Probable cause

- 1 Purge valve not closed
- 2 Damaged flow cell

### Suggested actions

Check purge valve is closed.

- Check for liquid in front drain tube or drip gutter.
- Please contact your Agilent service representative.

## 7 Troubleshooting and Diagnostics

### RI Detector Problems

## Unable to autozero detector

#### Probable cause

- 1 Sample and reference cells do not contain identical solutions
- 2 Reference cell contains air bubbles
- 3 Contaminated flow cell
- 4 Faulty light source

#### Suggested actions

- Purge the RI detector thoroughly.
- Purge the RI detector thoroughly.
- Clean the flow cell thoroughly. Refer to [“Routine Maintenance of the MDS System”](#) on page 105 for further information.
- Please contact your Agilent service representative.

## Viscometer Detector Problems

### DP/IP excessive baseline noise

#### Probable cause

- 1 Bubble in DP/IP transducer
- 2 Incorrect compressibility setting for mobile phase
- 3 Pump flow rate fluctuating

#### Suggested actions

- Purge the DP/IP transducers thoroughly to remove air bubble.
  - Use only degassed mobile phase.
  - Repeatedly purge with 3 s purge time.
- Use correct pump compressibility setting for mobile phase solvent.
- Check pump performance.
  - Insufficient pressure on pump.
  - Use pulse damper.

### DP/IP excessive baseline drift

#### Probable cause

- 1 Detector not fully equilibrated with environment
- 2 Environmental temperature fluctuations
- 3 DP/IP transducers not fully flushed with mobile phase

#### Suggested actions

- Allow sufficient time for the viscometer to equilibrate with the mobile phase and environment.
- Move instrument to a more stable environment, see [“Location of the Instrument”](#) on page 31 for more information.
- Purge the DP/IP transducers thoroughly.

## 7 Troubleshooting and Diagnostics

### Viscometer Detector Problems

#### Low sensitivity

##### Probable cause

- 1 DP/IP transducers not fully flushed with mobile phase

##### Suggested actions

Purge the DP/IP transducers thoroughly.

#### Excessive DP Offset

##### Probable cause

- 1 DP transducer leaking
- 2 Partial blockage of the bridge
- 3 Distorted pressure transducer membrane
- 4 Hold-up column defective

##### Suggested actions

Check system for solvent leaks.

Please contact your Agilent service representative.

Please contact your Agilent service representative.

Please contact your Agilent service representative.

#### High IP Output

##### Probable cause

- 1 Partial blockage on front of the bridge

##### Suggested actions

Please contact your Agilent service representative.

#### Low IP Output

##### Probable cause

- 1 Incorrect flow rate
- 2 Leak before viscometer bridge

##### Suggested actions

Check pump performance.

Check system for solvent leaks.



## Light Scattering Detector Problems

### Excessive baseline noise

#### Probable cause

- 1 Particles shedding from analytical column
- 2 Particles floating in the cell that do not exit easily after continuous pumping
- 3 Contaminated flow cell due to salt crystals deposited in the cell and lens after an inappropriate solvent was used to flush the system following use of a salt buffer
- 4 Dried sample and other material when the cell has been allowed to run dry and the solvent has evaporated

#### Suggested actions

- Condition with at least 10 column volumes by flushing with solvent to waste prior to use.
  - Fit an in-line filter after the column.
  - Filter the mobile phase through  $<0.2\ \mu\text{m}$  membrane.
  - Flush the system thoroughly with the filtered mobile phase to waste and then allow to stabilise.
  - Fit an in-line filter between pump and injection valve.
- Flush the system thoroughly with filtered water to re-dissolve the salts in the cell and then allow to stabilise.
- Filter the mobile phase through  $<0.2\ \mu\text{m}$  membrane.
  - Flush the system thoroughly with the filtered mobile phase to waste and then allow to stabilize.
  - Fit an in-line filter between pump and injection valve.

## High baseline offset

### Probable cause

- 1 Particulates in the mobile phase
- 2 Contaminated flow cell

### Suggested actions

- Filter the mobile phase through  $<0.2\ \mu\text{m}$  membrane.
- Flush the system thoroughly with the filtered mobile phase to waste and then allow to stabilize.
- Fit an in-line filter between pump and injection valve.

Clean the flow cell thoroughly. Refer to [“Routine Maintenance of the MDS System”](#) on page 105 for further information.

## No signal outputs

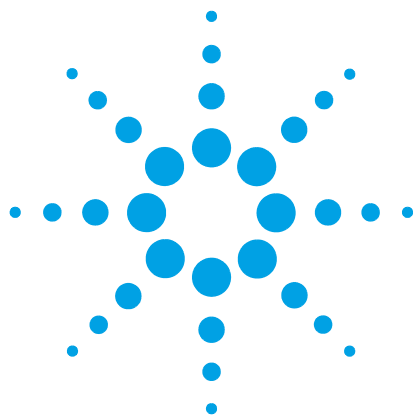
### Probable cause

- 1 Laser not switched on
- 2 Faulty laser

### Suggested actions

Check laser is switched on.

Please contact your Agilent service representative.



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## 8 Error Information

### Light Scattering Detector Problems

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

## What Are Error Messages

If an error occurs on the MDS, it is indicated by the system alarming continuously with the display showing information about the error in the System Status Area.

There are two types of errors that can occur:

- System Errors
- Detector Errors

For system errors, the Home screen is displayed with the keypad locked. The keypad cannot be unlocked manually.

The detector heaters are turned off and a pump stop command is sent via the auxiliary I/O cable.

Pressing the CE key on the keypad once will stop the alarm. Pressing it a second time will clear the error message. If the error has not been rectified, the error message will be displayed again within 5 s. If the keypad has been inactive and no keys are pressed, for a minute the alarm will sound again.

In all cases, if it is not possible to resolve the error, you should make a note of the error information displayed and contact your Agilent service representative for more information.

## System Error Messages

### Upper Leak Sensor Failed

**Error ID: SYSTEM 1010**

The upper thermistor on the leak sensor located in upper enclosure bay has failed.

**Probable cause**

- 1 Leak sensor not connected to the main board in the control unit.
- 2 Defective vapor sensor

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

### Upper Leak Sensor Failed

**Error ID: SYSTEM 1020**

The lower thermistor on the leak sensor located in upper enclosure bay has failed.

**Probable cause**

- 1 Leak sensor not connected to the main board in the control unit.
- 2 Defective vapor sensor

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Lower Leak Sensor Failed

**Error ID: SYSTEM 1030**

The upper thermistor on the leak sensor located in lower enclosure bay has failed

**Probable cause**

- 1 Leak sensor not connected to the main board in the control unit.
- 2 Defective vapor sensor

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Lower Leak Sensor Failed

**Error ID: SYSTEM 1040**

The lower thermistor on the leak sensor located in lower enclosure bay has failed.

**Probable cause**

- 1 Leak sensor not connected to the main board in the control unit.
- 2 Defective vapor sensor

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Leak Detected in Upper Enclosure

**Error ID: SYSTEM 1050**

A leak was detected inside the system in the upper enclosure

**Probable cause**

- 1 Loose connection at upper detector ports

**Suggested actions**

Ensure all fittings are tight

**Probable cause**

- 1 Blocked detector inlet causing leak at capillary fittings

**Suggested actions**

Check flow path is clear of obstruction

## Leak Detected in Bottom Enclosure

**Error ID: SYSTEM 1060**

A leak was detected inside the system in the lower enclosure

**Probable cause**

- 1 Loose connection at upper detector ports

**Suggested actions**

Ensure all fittings are tight

- 2 Blocked detector inlet causing leak at capillary fittings

Check flow path is clear of obstruction



## Rear Fan Failed in Control Unit

**Error ID: SYSTEM 1100**

Thermal shut-down of the cooling fan at rear of control unit.

**Probable cause**

- 1 Fan cable disconnected
- 2 Defective fan
- 3 Obstruction in fan
- 4 Defective main board in control unit

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Fan Failed in Upper Enclosure

**Error ID: SYSTEM 1110**

Thermal shut-down of the cooling fan at rear of upper enclosure.

**Probable cause**

- 1 Fan cable disconnected
- 2 Defective fan
- 3 Obstruction in fan
- 4 Defective main board in control unit

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Fan Failed in Lower Enclosure

**Error ID: SYSTEM 1120**

Thermal shut-down of the cooling fan at rear of lower enclosure.

**Probable cause**

- 1 Fan cable disconnected
- 2 Defective fan
- 3 Obstruction in fan
- 4 Defective main board in control unit

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Fan Failed on Control Unit Power Supply

**Error ID: SYSTEM 1130**

Thermal shut-down of the cooling fan located next to power supply in control unit.

**Probable cause**

- 1 Fan cable disconnected
- 2 Defective fan
- 3 Defective main board in control unit

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Communication to RI Detector Failed

**Error ID: SYSTEM 1230**

Communication between RI detector and Control unit has failed

<b>Probable cause</b>	<b>Suggested actions</b>
<b>1</b> RI Detector not connected to main board in control unit	Please contact your Agilent service representative.
<b>2</b> Defective RI Detector	Please contact your Agilent service representative.
<b>3</b> Defective main board in control unit	Please contact your Agilent service representative.

## Communication to VS Detector Failed

**Error ID: SYSTEM 1240**

Communication between Viscometer and Control unit has failed

<b>Probable cause</b>	<b>Suggested actions</b>
<b>1</b> Viscometer not connected to main board in control unit	Please contact your Agilent service representative.
<b>2</b> Defective Viscometer	Please contact your Agilent service representative.
<b>3</b> Defective main board in control unit	Please contact your Agilent service representative.

## Communication to ADC Failed

**Error ID: SYSTEM 1250**

Communication between ADC and main processor has failed

<b>Probable cause</b>	<b>Suggested actions</b>
1 Main control firmware corrupt	Please contact your Agilent service representative.
2 Defective main board in control unit	Please contact your Agilent service representative.

## Communication to LS Detector Failed

**Error ID: SYSTEM 1260**

Communication between LS or LS/DLS Detector and Control unit has failed

<b>Probable cause</b>	<b>Suggested actions</b>
1 LS Detector not connected to main board in control unit	Please contact your Agilent service representative.
2 Defective LS Detector	Please contact your Agilent service representative.
3 Defective main board in control unit	Please contact your Agilent service representative.

## Communication to Control Unit Failed

**Error ID: SYSTEM 1280**

Communication in Control unit has failed.

<b>Probable cause</b>	<b>Suggested actions</b>
1 Main control firmware corrupt	Restart the instrument
2 Defective main board in control unit	Please contact your Agilent service representative.

## RI Detector Specific Error Messages

### Heated Block exceeded lower limit

**Error ID: RI 3110**

The sample cell block thermistor is reading too low.

**Probable cause**

- 1 Defective RI block assembly

**Suggested actions**

Please contact your Agilent service representative.

### Heated Block exceeded upper limit

**Error ID: RI 3120**

The sample cell block thermistor reading too high.

**Probable cause**

- 1 Defective RI block assembly

**Suggested actions**

Please contact your Agilent service representative.

### LED Light Source Error

**Error ID: RI 3210**

The LED light source has failed.

**Probable cause**

- 1 LED Light source not connected to the RI main board
- 2 Defective LED Light source

**Suggested actions**

Please contact your Agilent service representative.

Please contact your Agilent service representative.

## 8 Error Information

### RI Detector Specific Error Messages

## Autozero Error

**Error ID: RI 3410**

The autozero action has timed out.

### Probable cause

- 1 Detector at false zero
- 2 Zero glass assembly not connected to RI main board
- 3 Defective zero-glass assembly

### Suggested actions

- Purge RI detector thoroughly and repeat autozero
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Viscometer Specific Error Messages

### Temperature Sensor Error

**Error ID: VS 4110**

Temperature sensor reading outside normal limits.

**Probable cause**

- 1 Defective temperature sensor
- 2 Defective main board in viscometer

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

### Unable to reach temperature set-point

**Error ID: VS 4120**

The heater has timed out and is unable to reach set temperature.

**Probable cause**

- 1 Defective heater
- 2 Defective main board in viscometer

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## 8 Error Information

### Viscometer Specific Error Messages

#### Heater Error

**Error ID: VS 4130**

Heater has failed

**Probable cause**

- 1 Defective heater
- 2 Defective main board in viscometer

**Suggested actions**

Please contact your Agilent service representative.

Please contact your Agilent service representative.

#### IP Purge Valve Error

**Error ID: VS 4310**

Purge valve failed to operate.

**Probable cause**

- 1 Purge valve is defective
- 2 Purge valve assembly not connected to viscometer main board
- 3 Defective main board in viscometer

**Suggested actions**

Please contact your Agilent service representative.

Please contact your Agilent service representative.

Please contact your Agilent service representative.



## DP Purge Valve Error

**Error ID: VS 4320**

Purge valve failed to operate.

**Probable cause**

- 1 Purge valve is defective
- 2 Purge valve assembly not connected to viscometer main board
- 3 Defective main board in viscometer

**Suggested actions**

Please contact your Agilent service representative.

Please contact your Agilent service representative.

Please contact your Agilent service representative.

## 8 Error Information

### LS and DLS Detector Specific Error Messages

## LS and DLS Detector Specific Error Messages

### Temperature Sensor Error

**Error ID: LS 6110**

Temperature sensor reading outside normal limits.

**Probable cause**

- 1 Defective temperature sensor
- 2 Defective LS Detector main board

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

### Unable to reach temperature set-point

**Error ID: LS 6120**

Heater has timed out and unable to reach set temperature.

**Probable cause**

- 1 Defective heater
- 2 Defective LS Detector main board

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Heater Error

**Error ID: LS 6130**

Heater has failed.

**Probable cause**

- 1 Defective heater
- 2 Heater not connected to LS detector main board
- 3 Defective LS Detector main board

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## Laser Error

Laser current outside of normal operating range.

**Probable cause**

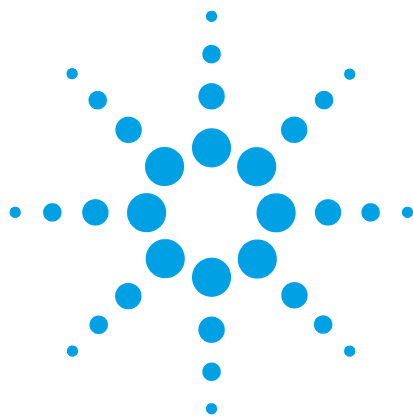
- 1 Laser Assembly is defective
- 2 Laser Assembly not connected to detector main board
- 3 Laser Interlock switch not connected to detector main board
- 4 Laser Interlock switch is defective
- 5 Defective LS Detector main board

**Suggested actions**

- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.
- Please contact your Agilent service representative.

## **8 Error Information**

### **LS and DLS Detector Specific Error Messages**



## 9 Maintenance

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This chapter describes the maintenance of the 1260 Infinity Multidetector Suite.



## **9 Maintenance**

### **Introduction to Maintenance**

## **Introduction to Maintenance**

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

## Information for Service Personnel

Please note that this instrument is double fused.

The following fuses are fitted:

- 2x 250 H 2 A T (20x5 mm)

### **WARNING**

#### **Fire and damage to the module**

##### **Wrong fuses**

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

## Warnings and Cautions

### WARNING

#### Toxic, flammable and hazardous solvents, samples and reagents

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

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

### WARNING

#### Eye Discomfort

**The light source in the RI detector is a class 1 LED product. Temporary discomfort may result from directly viewing the light produced by this source.**

- Do not look into the beam.
- 

### WARNING

#### Eye damage by laser light

**Hazardous laser light can injure eyes.**

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



**WARNING**

**Electrical shock**

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

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

**WARNING**

**Personal injury or damage to the product**

**Agilent is not responsible for any damages caused, in whole or in part, by improper use of the products, unauthorized alterations, adjustments or modifications to the products, failure to comply with procedures in Agilent product user guides, or use of the products in violation of applicable laws, rules or regulations.**

- Use your Agilent products only in the manner described in the Agilent product user guides.
- 

**CAUTION**

**Safety standards for external equipment**

- If you connect external equipment to the instrument, make sure that you only use accessory units tested and approved according to the safety standards appropriate for the type of external equipment.
-

## Cleaning the Module

The exterior of the instrument should be cleaned by wiping down with a soft cloth moistened with dilute detergent solution, followed by wiping down with a cloth moistened with deionized water. Ensure that no moisture enters the instrument.

### WARNING

#### Electrical shock and burns

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

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

## Inspection of Cables

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

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

## 9 Maintenance

### Putting the Instrument into Long-Term Storage

# Putting the Instrument into Long-Term Storage

#### CAUTION

Corrosive and oxidizing solvents

Some solvents may corrode the detector(s) if they are not fully flushed from the system after use.

→ Remove corrosive solvents thoroughly from the entire system before switching off the instrument.

#### NOTE

For flushing, use solvent with the following characteristics:

- No dissolved salts, acids, bases or halides
- Low vapour pressure at storage temperatures
- Does not allow bacterial growth
- Does not oxidize or form peroxides

**Follow this procedure if the instrument is to be stored or not used for an extended period of time:**

- 1 Purge the pump with a mixture of Acetone/Water (50/50) at 1 mL/min for 15 min.
- 2 Flush out all 1260 Infinity Multi-Detector Suite detector lines.
- 3 Flush the reference side of the RI cell and the viscometer transducers thoroughly by using the purge actions.
- 4 Power the MDS off.
- 5 Remove the tubing lines to MDS detector(s) installed and insert a blank plug into the inlet and outlet ports and the purge port if an RI detector is installed.
- 6 Label the instrument with a tag describing the storage solvent.



## 10 Parts and Materials for Maintenance

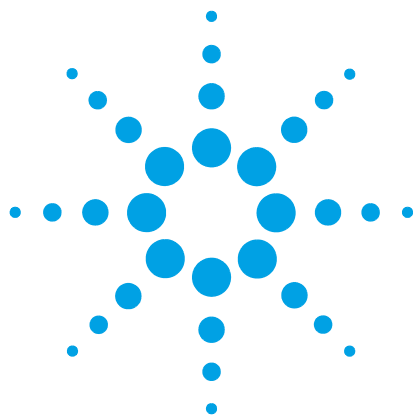
Parts and Materials for Maintenance 158

This chapter provides information on parts for maintenance.



## Parts and Materials for Maintenance

<b>p/n</b>	<b>Description</b>
G7801A	Agilent 1260 Infinity GPC/SEC MDS RI Detector
G7802A	Agilent 1260 Infinity GPC/SEC MDS Viscometer
G7803A	Agilent 1260 Infinity GPC/SEC GPC MDS Dual Angle LSD
G7808A	Agilent 1260 Infinity Bio-SEC LSD
G7809A	Agilent 1260 Infinity Bio-SEC LS / DLS
G7800-68000	Agilent 1260 Infinity MDS Installation Kit
G7800-64003	1260 Infinity GPC/SEC MDS Enclosure Expansion
G7800-68001	1260 Infinity GPC/SEC MDS 1200 Accessory Kit
G7800-63001	Agilent 1260 Infinity MDS USB Cable
G7800-87000	PTFE Tubing 0.25 mm ID x 1.6 mm OD (2 m)
G7800-87001	PTFE Tubing 1.0 mm ID x 1.6 mm OD (2 m)
G7800-63002	Agilent 1200 to 1260 MDS Analogue cable
G7800-63003	Agilent 1260 Infinity MDS Auxiliary I/O cable
G7809-68000	Agilent 1260 Infinity MDS LS/DLS Installation Kit
5062-2463	Corrugated tubing, PP, 6.5 mm id, 5 m
5022-2184	Union, stand LD flow, no fitting
PL1-24950	RS232 Serial Cable
PL1-28327	MDS to 1200 Series Auxiliary Cable
PL0100-3601	Valco Nut, 1/16 inch, 5 EA
PL0100-3602	Valco Ferrule, 1/16 inch, 5 EA



## 11 Appendix 1— Theory of Operation

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This chapter provides an overview of the principles and theory of RI, Viscometry and Light Scattering detection.



## 11 Appendix 1– Theory of Operation

### The 1260 Infinity MDS Refractive Index Detector

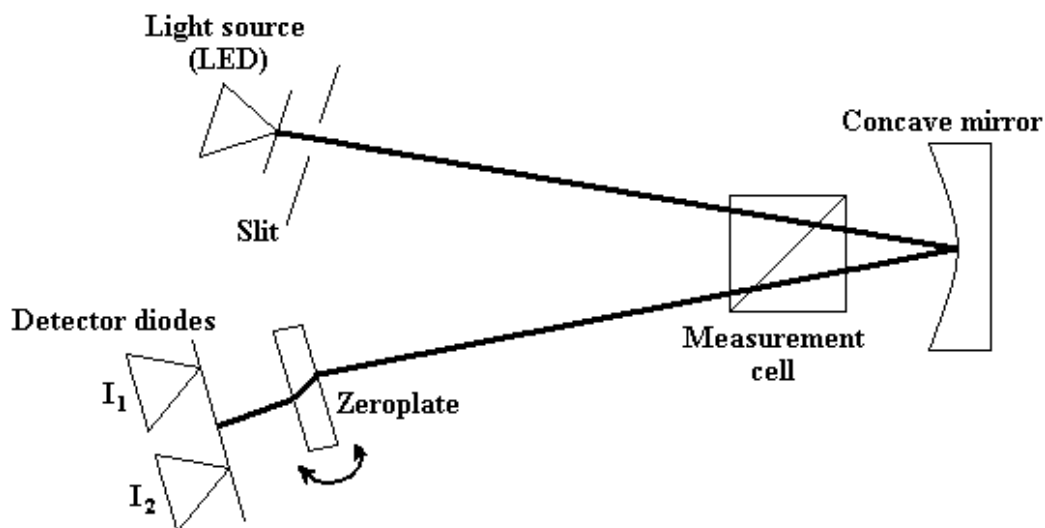
## The 1260 Infinity MDS Refractive Index Detector

The MDS differential refractive index detector is a high performance device specifically designed for GPC. The RI detector features high signal-to-noise and low levels of drift and is fitted with a low dispersion cell to maintain peak shapes throughout the sample injection. The heated cell environment means that excellent temperature stability is maintained, giving ultra stable baselines.

### Measurement Principle and Light Path

A light beam emitted from an LED crosses the sample and the reference cell of the RI detector twice. When both cells contain pure solvent, the system is calibrated to zero by means of a parallel zero-plate that positions the beam on the two detector diodes 1 and 2 in such a manner that the measured light intensities ( $I_1$  and  $I_2$ ) of the two diodes are virtually identical. When the sample cell contains a solution with a different refractive index to that of the solvent, the light beam is geometrically proportionally deflected depending on the relative change of the refractive index (according to Snell's refraction law).





**Figure 27** Optical Diagram of the Refractive Index Detector

This results in a change of the light intensities  $I_1$  and  $I_2$ , proportional to concentration and refractive index of the sample solution. From these intensity changes the signal value is calculated and indicated on the output display.

The built-in measurement cell of the RI detector operates with a measuring angle of  $45^\circ$ . Measurements are performed at a wavelength of 658 nm. The signal detection and signal processing method allows the refractive index to be given out in the “online” mode without absorption components. The autozero-range includes the complete measurement range.

**NOTE**

The refractive index is highly temperature dependent. The change per  $1^\circ\text{C}$  for pure water is  $\approx 1 \cdot 10^{-4}$  RIU and for typical organic solvents is  $\approx 5 \cdot 10^{-4}$  RIU.

## Using Refractive Index Measurements to Measure Concentration

Refractive index measurements can be used to determine the compound concentration in the chromatographic slice.

The difference in refractive index between the solution under study and the pure solvent is measured by passing a light beam through two cells. One of the cells contains pure solvent and the other contains the eluent as it passes through the cell after the column. The cell with the pure solvent should be filled with the solvent before the separation and should have the same temperature as the eluent.

The observed signal (RI), which corresponds to the light beam deviation is proportional to the difference in the refractive index of the fluid in the two cells as shown in the following equation:

$$RI = K (dn/dc) * c$$

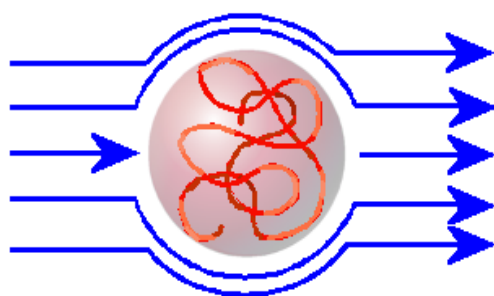
where:

- K  
instrument constant
- c  
sample concentration in the solvent
- $dn / dc$   
the change in the refractive index as a function of the change in concentration. It is considered to be a constant for any specified solvent-solute pair under constant operating conditions.

## The 1260 Infinity MDS Viscometer

### Theory of Online Viscosity Measurements

When a polymer dissolves in a liquid, the interaction of the two components stimulates an increase in polymer dimensions over that of the unsolved state. Due to the vast difference in size between solvent and solute, the frictional properties of the solvent in the mixture are drastically altered, and an increase in viscosity occurs which should reflect the size and shape of the dissolved solute, even in dilute solutions.



The dissolved polymer coil disturbs the linear flow of solvent, resulting in a change in viscosity.

This was first recognized in 1930 by Staudinger, who found an empirical relation between the relative magnitude of increase in viscosity and the molar mass of the polymer.

The Intrinsic viscosity  $[\eta]$  is a measure of the viscosity of a polymer solution, as the concentration tends to zero, i.e. at very low concentrations. It is this parameter therefore that is a characteristic of isolated polymer chains in solution and can be considered to be proportional to the density of the polymer coil.

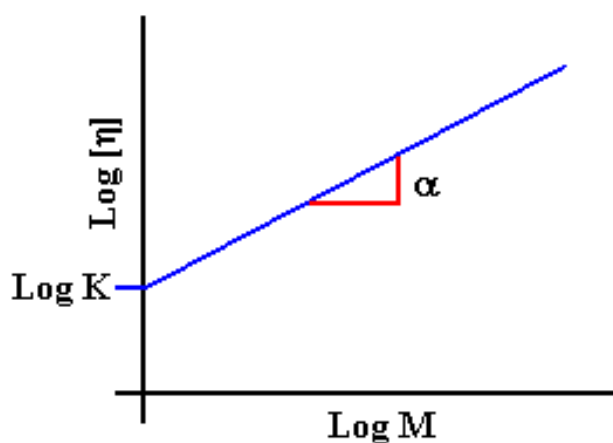
## 11 Appendix 1– Theory of Operation

### The 1260 Infinity MDS Viscometer

For a given polymer and solvent system at a specified temperature,  $[\eta]$  can be related to molecular weight,  $M$ , through the Mark-Houwink-Sakurada equation.

$$[\eta] = KM^\alpha$$

Where  $K$  and  $\alpha$  are coefficients specific to the polymer solvent system and temperature. These coefficients can be determined by calibrating with polymer standards of known molecular weights. A plot of  $\text{Log}([\eta])$  versus  $\text{Log}(M)$ , a Mark-Houwink-Sakurada plot, will be a straight line for a linear homopolymer as long as the Universal Calibration is obeyed.

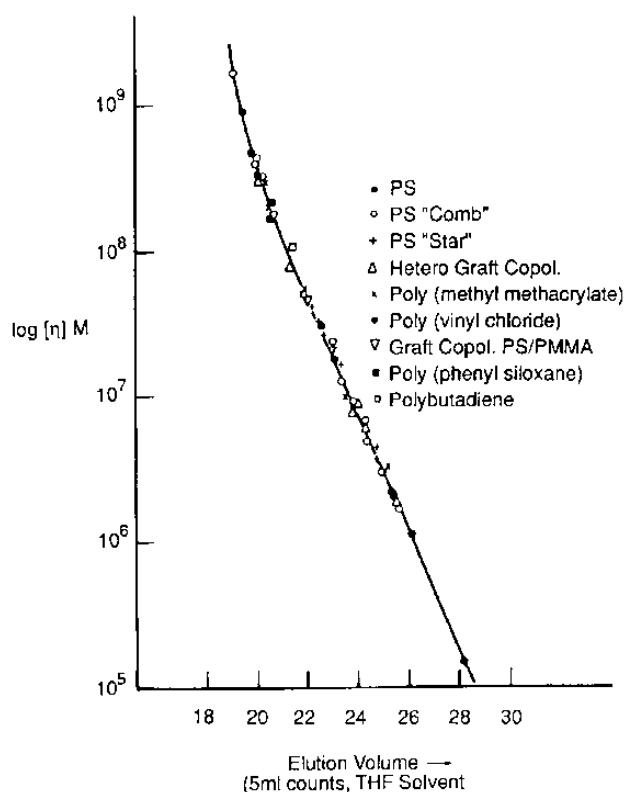


**Figure 28** Mark-Houwink-Sakurada Plot

Values of  $\alpha$  reflect the size of the molecule in solution ranging between 0.5 for a polymer behaving as a hard-sphere to about 2.0 for rigid-rods.

## Universal Calibration

Benoit (1967) showed that polymers of different structure fall on the same calibration curve if the intrinsic viscosity is included as a calibration parameter. A calibration plot of the product of intrinsic viscosity and molecular weight ( $[\eta].M$ ) versus elution volume is a "Universal Calibration", as shown below in Figure 29 on page 165. This allows the system to be calibrated with polymer standards of one type and then analyze polymers of another type and still obtain accurate molecular weight distributions.



**Figure 29** A Universal Calibration Plot

Therefore for an unknown polymer sample the Universal Calibration is used to convert the intrinsic viscosity, determined for each slice across the distribution from the viscometer, to molecular weight.

## Polymer Branching

The determination of the degree of long chain branching has long occupied polymer chemists. In polymer solutions, the branching factor,  $g$ , is the starting point for many branching calculations.

$$g = \left( \frac{[\eta_b]}{[\eta_l]} \right)^{1/\epsilon}$$

Where  $\eta_b$  is the intrinsic viscosity of the branched polymer and  $\eta_l$  is the intrinsic viscosity of the linear polymer of the same molecular weight.  $\epsilon$  is the “structural model” parameter, which is user defined.

## Four Capillary Viscometry Theory

The MDS viscometer employs the fluid flow equivalent of the analogous Wheatstone bridge electrical circuit. Solvent travels down a bridge of four capillaries of equal resistance arranged as shown in Figure 30 on page 166.

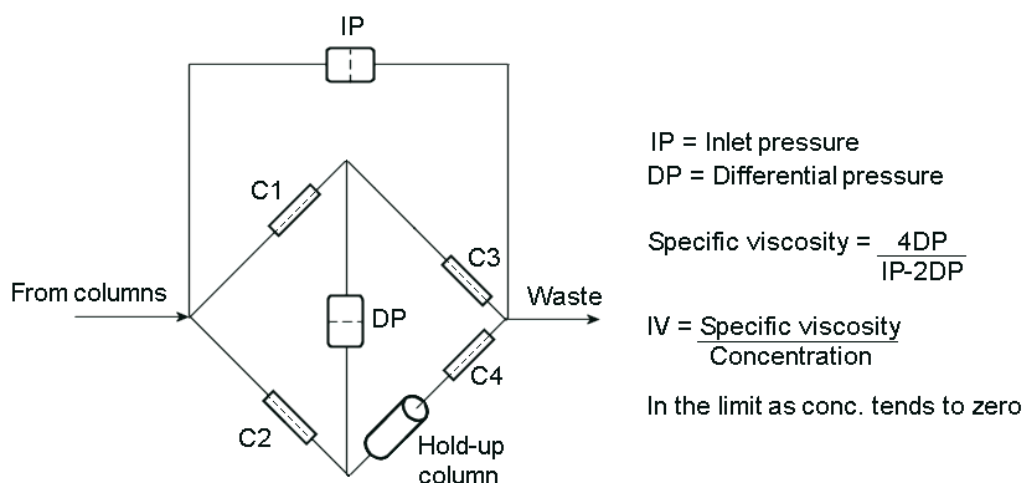


Figure 30 Plumbing Diagram of the MDS Viscometer

The differential pressure transducer monitors the pressure drop across the bridge,  $\Delta P$ , and the inlet pressure transducer measures the pressure drop through the bridge,  $P_i$ . When the sample is introduced across capillary 4 (C4) of the bridge, an imbalance is caused due to the holdup columns. This change in pressure,  $\Delta P$  is proportional to the specific viscosity,  $\eta_{sp}$ , according to the following equation.

$$\eta_{sp} = \frac{4\Delta P}{P_i - 2\Delta P}$$

Where  $\Delta P$  is the differential pressure across the bridge and  $P_i$ , is the pressure of the flow through the capillary bridge. The instrument makes a true differential measurement directly, making it very sensitive to dilute solution viscosity measurements.

The intrinsic viscosity,  $[\eta]$  is ideally obtained by measuring the specific viscosity values at several finite concentrations and extrapolate to zero concentration.

$$[\eta] = \lim_{c \rightarrow 0} (\eta_{sp}/c)$$

However, the concentrations used in GPC are low enough for the equation to be valid over a typical chromatogram, and thus an extrapolation to the true intrinsic viscosity is not required, since the concentration is close enough to the limiting concentration.

All of the data acquisition and data processing are accomplished using Agilent GPC-SEC software, which enables calculation of molecular weight and intrinsic viscosity distributions using algorithms for Universal Calibration. Branching information can also be obtained from Agilent GPC-SEC software.

## Viscometer Troubleshooting Charts

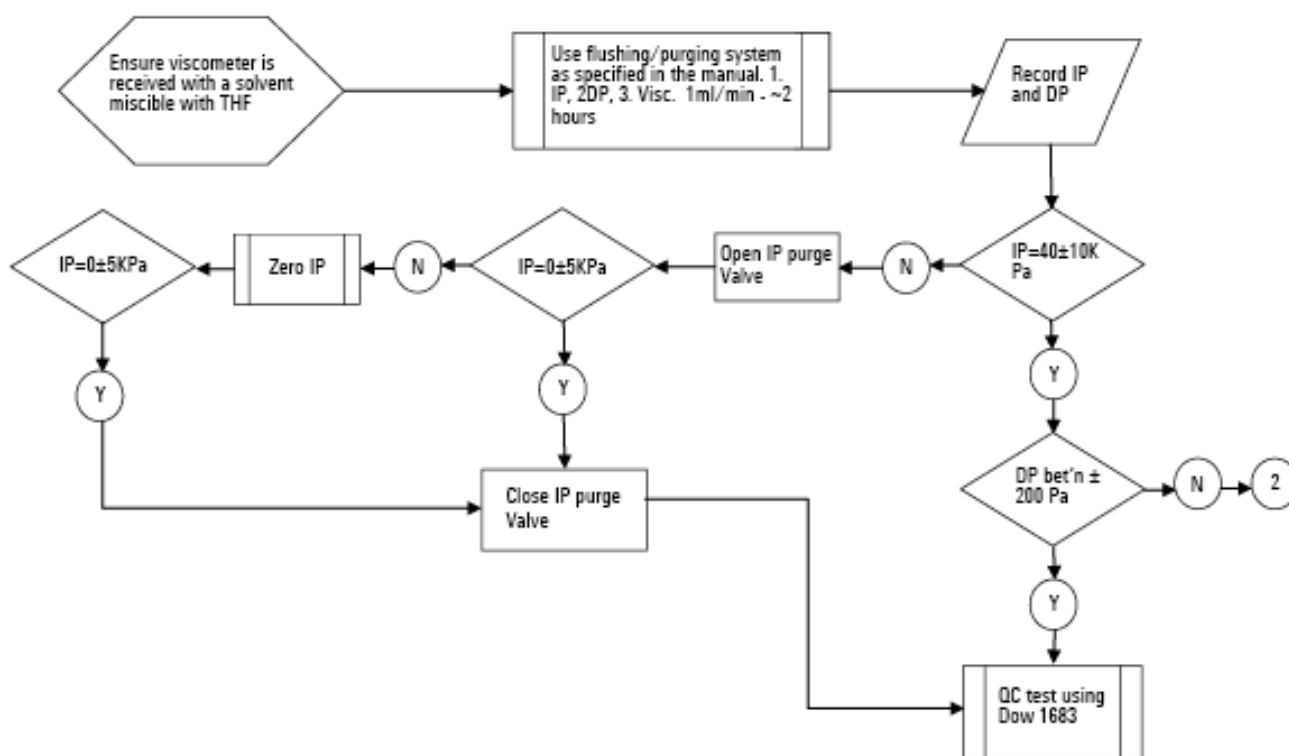


Figure 31 Viscometer Troubleshooting Chart 1



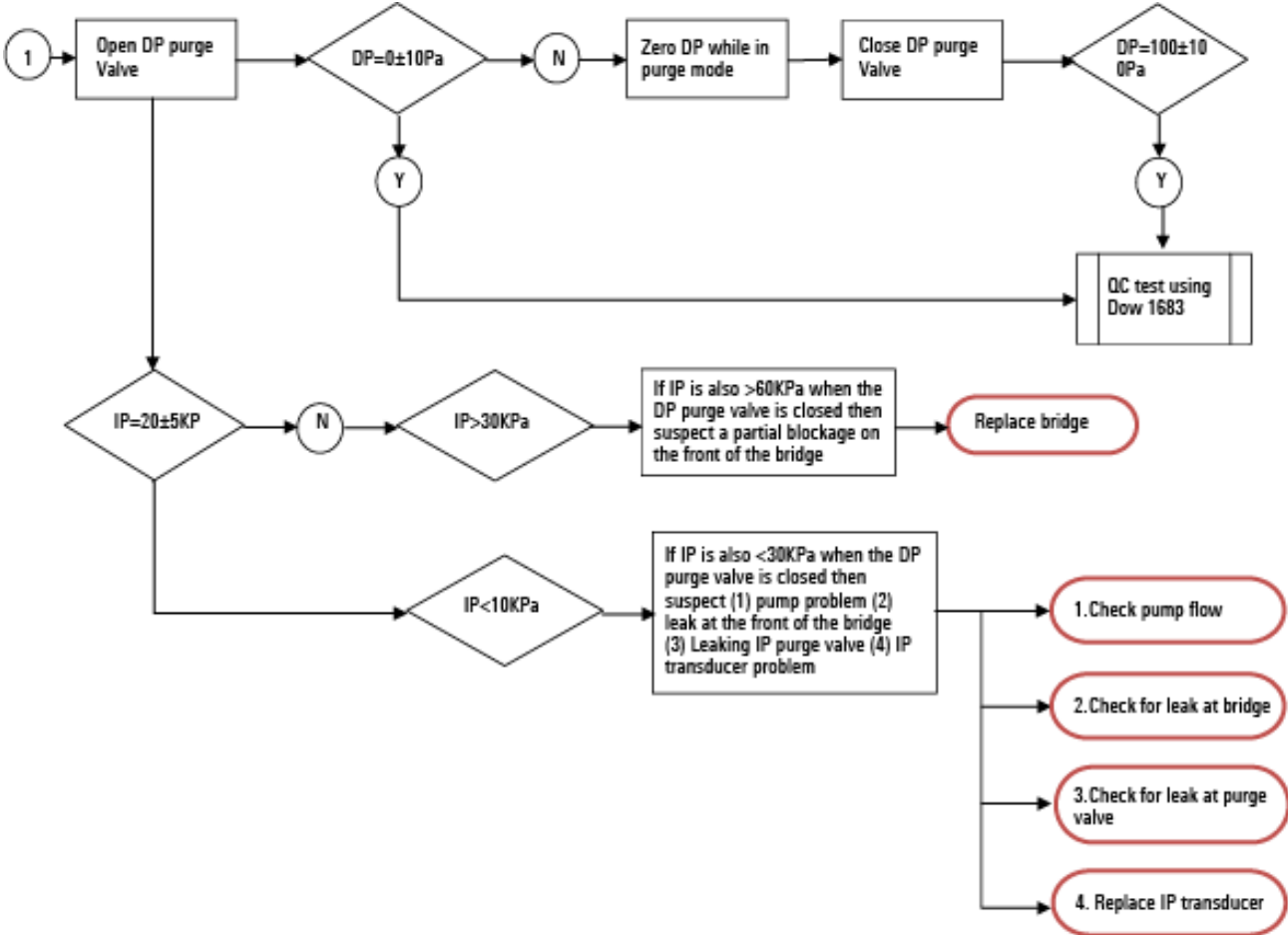
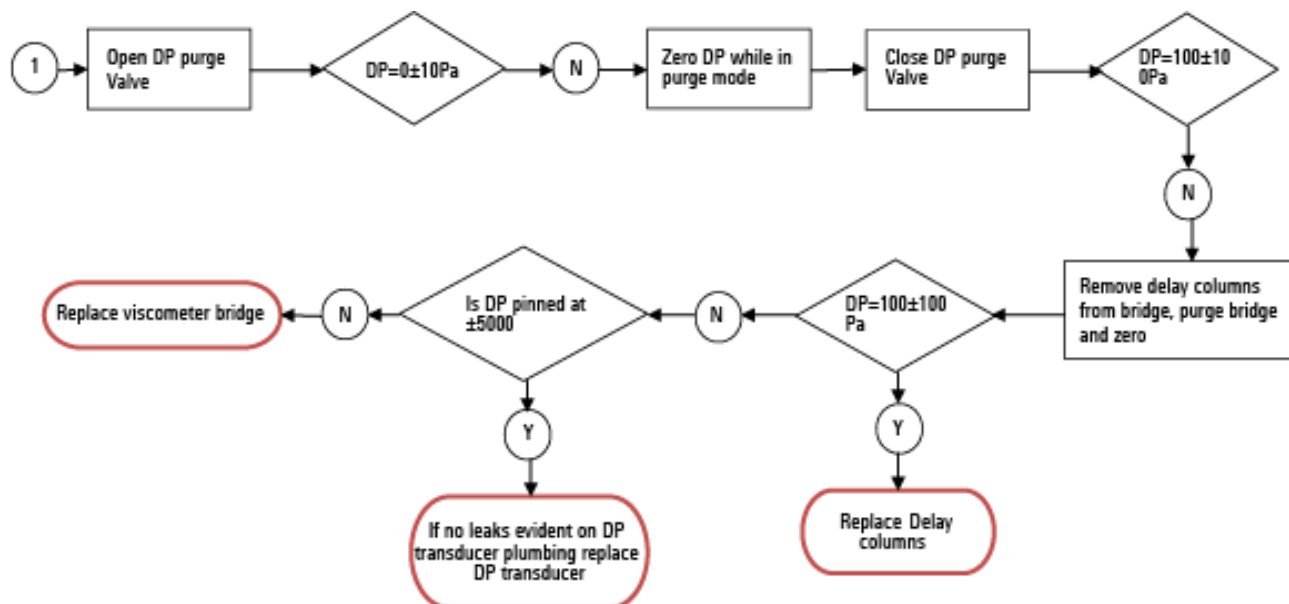


Figure 32 Viscometer Troubleshooting Chart 2

**11 Appendix 1– Theory of Operation**  
The 1260 Infinity MDS Viscometer



**Figure 33** Viscometer Troubleshooting Chart 3

## The 1260 Infinity MDS Light-Scattering Detector

### Overview

Light Scattering refers to the process in which light from an incident polarized laser beam is scattered in all directions when it interacts with a molecule or particle. Light scattering is an everyday occurrence and was first described by Lord Rayleigh in the late 1800's. An example of light scattering is the scattering of sunlight by particles in the atmosphere: the sky is blue because shorter visible wavelength radiation (blue light) is scattered more strongly by the gas molecules in air than light of longer wavelengths (red light). There are two general techniques for the measurement of physical properties of polymers (synthetic polymers and natural polymers such as proteins and polysaccharides), and particles.

#### Static Light Scattering

Also known as Rayleigh scattering or classical light scattering - the intensity of the scattered light from the sample dissolved in the solvent. This difference between the two measurements can be used to determine the average molecular weight and  $R_g$  (radius of gyration). To complete the molecular weight determination, the concentration of the compound of interest is required (typically obtained from a refractive index detector or an absorbance detector). Static Light Scattering is described in [“Static Light Scattering”](#) on page 173.

#### Dynamic Light Scattering

Also known as Quasi-elastic scattering, Photocorrelation Spectroscopy, or Beat Spectroscopy - the fluctuations of the intensity of the scattered light is used to determine the diffusion coefficient of the molecules as they move in solution (Brownian motion). The Stokes-Einstein equation is used to determine  $R_h$  (the hydrodynamic radius of the molecule). Dynamic Light Scattering is described in [“Measuring  \$R\_h\$  Using Dynamic Light Scattering”](#) on page 179.

Static and Dynamic measurements can be made on a sample in a cuvette or in a flowing stream such as that found in gel permeation chromatography (GPC). In flowing streams, measurements are made on each elution slice using a unique light scattering design and very fast digital signal processors.

## The Physical Basis Of Light Scattering

Light consists of perpendicular electric and magnetic fields that oscillate in a direction that is perpendicular to the direction of propagation of the light as shown in Figure 34 on page 172. When light strikes a molecule, the electrons will experience a force due to the electric field and will move slightly. This movement will induce an oscillating dipole moment that will radiate light in all directions at the oscillating frequency. This radiated light is the scattered light that is detected and processed as described below.

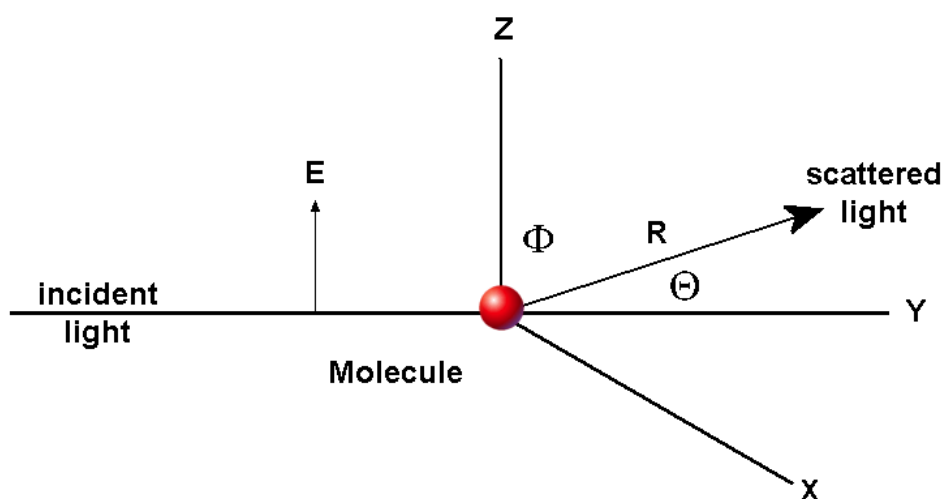


Figure 34 Light Scattered by a Molecule

Figure 34 on page 172 describes the spatial arrangement of the incident light and scattered light in the light scattering experiment. The light is polarized in the vertical direction, thus the electric field will oscillate in the Z direction and the magnetic field will oscillate in the X direction.

A diode laser, which is a monochromatic source of light that can be focused to a very small point in the centre of the sample cell, is used in the light scattering system. Typical sources include a semiconductor laser that can provide 20 to 30 mW at 685 nm and another, which provides 100 to 150 mW at 800 nm. The scattered light is collected at a given angle and orientation (e.g. 15 ° or 90 °) from the incident radiation, and is used to deduce the desired molecular properties.

## Static Light Scattering

### Measuring the Molecular Weight

#### NOTE

Agilent Technologies systems include polarized laser light sources, the equations presented in this chapter will be slightly different than those presented in discussions of light scattering when non-polarized sources are used.

The electric dipole moment that is induced is shown in equation 1:

$$\vec{p} = \alpha \vec{E} \quad 1$$

where:  $p$  is the dipole moment  
 $\alpha$  is the polarizability  
 $E$  is the electric field

The polarizability can be related to measurable parameters via equation 2:

$$A = M_w \left( \frac{dn/dc}{2 \cdot \pi \cdot N_A} \right) \quad 2$$

where:  $M_w$  is the molecular weight  
 $N_A$  is Avogadro's number ( $6.02 \cdot 10^{23}$  molecules per mol)  
 $dn/dc$  is the change in the index of refraction as a function of the change in concentration. It is considered to be a constant for any specified solvent-solute pair under constant operating conditions.

## 11 Appendix 1– Theory of Operation

### The 1260 Infinity MDS Light-Scattering Detector

The oscillating dipole will radiate light in all directions at the oscillating frequency. This is the origin of scattered light. If a single molecule has dimensions that are small with respect to the wavelength of the incident light, the intensity of the light can be defined by equation 3:

$$I_S = \frac{[4 \cdot \pi^2 \cdot M_w \cdot \sin^2 \Phi \cdot (dn/dc)^2 \cdot I_0]}{N_A^2 \cdot \lambda_0^4 \cdot R^2} \quad 3$$

where  $I_S$  is the intensity of the radiated light  
 $I_0$  is the intensity of the incident light  
 $\lambda_0$  is the wavelength of light in a vacuum  
 $\Phi$  as defined in [Figure 34](#) on page 172  
 and  
 $R$  Radius of molecule in solution

If we collect light from a volume V of a solution with a concentration c (gm/mL), the intensity of scattered light can be found by multiplying equation 3 by the number of molecules in the volume V.

The number of molecules can be expressed by

$$N_A \cdot c \cdot \frac{V}{M_w}$$

Now if we solve for  $M_w$ , we obtain equation 4:

$$M_w = \frac{[N_A^2 \cdot \lambda_0^4 \cdot R^2 \cdot I_S]}{4 \cdot \pi^2 \cdot M_w \cdot \sin^2 \Phi \cdot c \cdot (dn/dc)^2 \cdot I_0 \cdot V} \quad 4$$

Collecting all the constants and instrumental parameters into an overall instrumental constant, A, we obtain equation 5:

$$M_w = \frac{[I_s]}{A \cdot c \cdot (dn/dc)^2 \cdot I_0} \quad 5$$

Equation 5 can be used to measure the molecular weight  $M_w$  of small molecules at any scattering angle  $\Theta$ . It should be noted, however, that larger molecules scatter less light at high values of  $\Theta$  than at low angles because of interference effects caused by the fact that light scattered from one part of the molecule travels a different distance from another part of the molecule, and is not in phase with light scattered. This phenomenon can be quantified by defining the light scattering form factor (equation 6). A more detailed discussion of the form factor is presented in “[The Form Factor](#)” on page 177 sub section.

$$P(\Theta) = \frac{\text{scattering intensity at angle } \Theta}{\text{scattering intensity at angle } \Phi} \quad 6$$

It should be noted that  $P(\Theta)$  can be written as a series as shown in equation 7:

$$P(\Theta) = 1 - \frac{1}{3}(q^2 \cdot R_g^2) + \dots \quad 7$$

where

- $q = 4 \pi \cdot n \cdot \sin(\Theta / 2) / \lambda_0$
- $R_g$  is the radius of gyration of the molecule
- $n$  is the index of refraction of the liquid
- $\lambda_0$  is the wavelength of light in a vacuum

## 11 Appendix 1– Theory of Operation

### The 1260 Infinity MDS Light-Scattering Detector

For scattering at 15 ° and 90 °, equation 7 can be expressed as equations 8 and 9, respectively.

$$P(\Theta) = 1 - 26.3 \left( \frac{R_g \cdot n}{\lambda_0} \right)^2 \quad 8$$

$$P(\Theta) = 1 - 0.0897 \left( \frac{R_g \cdot n}{\lambda_0} \right)^2 \quad 9$$

Table 13 on page 176 shows values of P for 15 ° and 90 ° degrees as a function of molecular weight. These values assume that the molecules are random coils.

**Table 13** Values of P as a function of molecular weight

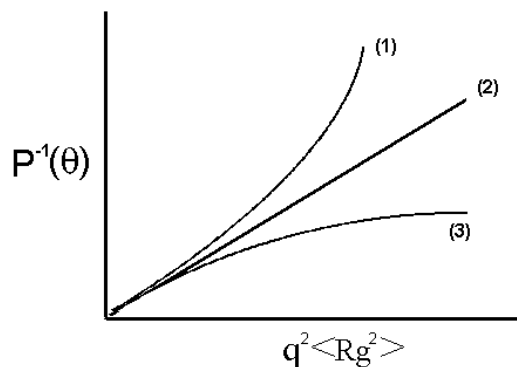
<b>M<sub>w</sub></b>	<b>R<sub>g</sub> (approx.) nm</b>	<b>P(90 °)<sup>1</sup></b>	<b>P(15 °)<sup>1</sup></b>
5·10 <sup>3</sup>	2.3	0.9993	1.0000
5·10 <sup>4</sup>	7	0.9993	0.9928
5·10 <sup>5</sup>	23	0.9993	0.9976
5·10 <sup>6</sup>	70	2	0.9880
5·10 <sup>7</sup>	230	2	0.7622

<sup>1</sup> Value of P(90°) and P(15°) for λ<sub>0</sub> = 685 nm, n = 1.5

<sup>2</sup> values depend on shape



If the value of  $P(\Theta)$  found in equation 7 is below 0.7, higher order components become important. In this case,  $P(\Theta)$  depends on  $R_g$  and also on the shape of the molecule as shown in Figure 35 on page 177.



**Figure 35** Values of  $p^{-1}(\Theta)$  as a function of  $q^2 \langle R_g^2 \rangle$  for various particle shapes

1	Sphere
2	Gaussian Coils
3	Rods

It is clear that all three molecular shapes yield the same value of  $P(\Theta)$  when  $q^2 \langle R_g^2 \rangle$  is less than approximately 1. As the value of  $P(\Theta)$  increases, the shape of the molecule clearly influences the light scattering intensity.

### The Form Factor

The form factor at a particular angle is the ratio of the signal at that angle when compared to the signal expected at the theoretical angle of  $0^\circ$  (where there is no form factor) as indicated by equation 6. The importance of the form factor is that small molecules (e.g. those which have a radius that is  $<10$  nm, which is small when compared to the wavelength of the incident light) are studied, generate comparable signals at all angles, while large molecules generate signals that are smaller at higher angles and larger at small angles.

## 11 Appendix 1– Theory of Operation

### The 1260 Infinity MDS Light-Scattering Detector

#### Characteristics of Low-Angle (15°) Light Scattering

Low-angle light scattering data is collected at a 15 ° angle to the incident beam and is typically used for determination of the molecular weights of large molecules. This measurement angle is especially useful for the study of proteins with molecular weight of greater than 1·10<sup>6</sup> Da and for random coils with molecular weight between 2·10<sup>6</sup> Da and about 1·10<sup>7</sup> Da.

In addition, 15 ° data is also used with 90 ° data to measure R<sub>g</sub>, the radius of gyration of molecules over a limited range of sizes (12 – 150 nm) using static light scattering analysis.

#### High-Angle Light Scattering

High-angle light scattering data is collected at a 90 ° angle to the incident beam and is typically used with static light scattering analysis to measure the molecular weight of smaller molecules such as proteins with a molecular weight below 1·10<sup>6</sup> Da, random coils with molecular weight below 2·10<sup>5</sup> Da and for lower molecular weight non-spherical coil polymers. In addition, it is used with dynamic light scattering analysis to measure R<sub>h</sub>, the hydrodynamic radius of molecules and particles from 1 to 1000 nm.

$$[\eta] = \lim_{c \rightarrow 0} (\eta_{sp}/c)$$

## Measuring Rh Using Dynamic Light Scattering

### Basics of Dynamic Light Scattering

The fundamental measurement for dynamic light scattering is the fluctuation of the intensity of the scattered light. This data is analyzed as described to yield the diffusion of the molecules or particles moving under Brownian movement.

When polarized laser light is scattered, the scattered light is at the same wavelength as the incident beam. However, it should be noted that when the light is monitored over extremely short time increments (in the microsecond time regime), a Doppler shift occurs and the frequency of the light appears to be changing. As the particle moves toward the detector, the frequency increases, and as it moves away the frequency decreases. The amount of the increase (decrease) of the change in the frequency is related to the diffusion rate of the scattering molecule in the solvent.

While the diffusion coefficient could be obtained from the frequency spectrum, it is easier to measure the small intensity fluctuations and then compute the autocorrelation function. The autocorrelation function is related to the frequency spectrum by the Fourier transform (i.e. the data processing is performed in the time domain rather than the frequency domain).

From an experimental perspective, the intensity of the scattered light is detected by counting the photons scattered via an avalanche photodiode, an electronic device that emits a pulse every time a photon passes through its detector. This high sensitivity detector sees a time varying signal rather than a relatively constant (in batch mode) or slowly varying “total intensity” signal.

It should be noted that even when measurements are taken in flow mode, the sample can be considered as slowly varying. This is due to the time frame of the scattered light measurement, relative to the rate of elution of the light scattering molecules from the column.

The photon counting measurement is performed over a very short period of time to record the very rapid diffusion that is taking place. Small particles or biomolecules diffuse quickly with the scattered light showing rapid small fluctuations while larger particles such as protein aggregates, nanoparticles, polymers, etc., diffuse more slowly, resulting in lower frequency fluctuations.

## 11 Appendix 1– Theory of Operation

### The 1260 Infinity MDS Light-Scattering Detector

#### The Autocorrelator

The autocorrelation function of these short interval counts is computed by an autocorrelator, which is a special purpose proprietary parallel computer that has been specially designed by Agilent Technologies. The correlator uses 256 channels that can be distributed over a maximum “channel space” of 1024 channels. Each channel can be considered as a separate “bucket” in which the emitted photons are counted during the sample period (which is in the order of a few  $\mu$ s). The 1024 channel spaces occupy a total “time-space” of 1024 equal sampling times that can be set by the operator according to the size and shape of the molecule of interest. Counts are collected in up to 256 of these channel spaces, which are arranged logarithmically throughout the total channel space by the software, the higher density being at the start.

The autocorrelator function over the 1024 channel space is computed by correlating the counts in each of the channels. Observation of the correlation function provides information that is useful to optimize the analysis. In addition, the display indicates the fraction of the sample with the indicated particle size.

#### Using Diffusion Coefficient to Determine Rh

Rh, the hydrodynamic radius of the compound of interest can be determined using the Stokes-Einstein equation (equation 11):

$$Rh = \frac{k \cdot T}{6 \cdot \pi \cdot \nu \cdot D} \quad 11$$

where	$k$	is Boltzman’s constant
	$T$	is the temperature of the eluent
	$\nu$	is the viscosity of the sample
	$D$	is the Diffusion coefficient calculated from the autocorrelation function



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



## General Safety Information

### Safety Symbols

Table 14 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.
	Indicates dangerous voltages.
	Indicates a protected ground terminal.
	The apparatus is marked with this symbol when hot surfaces are available and the user should not touch it when heated up.
	External Laser warning label located on rear of LS and DLS detector
	Internal Laser warning label located on inner box of LS and DLS detector

### WARNING

#### A WARNING

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

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

### CAUTION

#### A CAUTION

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

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

## General Safety Information

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

### **WARNING**

**Ensure the proper usage of the equipment.**

**The protection provided by the equipment may be impaired.**

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

---

## Safety Standards

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

## Operation

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

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

Make sure that only fuses with the required rated current and of the specified type (normal blow, time delay, and so on) are used for

## 12 Appendix 2 – General Safety Information

### General Safety Information

replacement. The use of repaired fuses and the short-circuiting of fuse holders must be avoided.

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

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

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

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

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

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



## The Waste Electrical and Electronic Equipment Directive

### Abstract

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

#### NOTE

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

Product Category:

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

---



#### NOTE

Do not dispose off in domestic household waste

To return unwanted products, contact your local Agilent office, or see [www.agilent.com](http://www.agilent.com) for more information.

---

## **Radio Interference**

Cables supplied by Agilent Technologies are screened to provide optimized protection against radio interference. All cables are in compliance with safety or EMC regulations.

### **Test and Measurement**

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

## **Agilent Technologies on Internet**

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<http://www.agilent.com>

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## In This Book

This manual contains information on:

- Agilent 1260 Infinity GPC/SEC MDS (G7800A)
- Agilent 1260 Infinity Bio-SEC MDS (G7805A)

The manual describes the following:

- Introduction,
- specifications,
- installation,
- controlling and operating,
- optimization,
- troubleshooting and diagnostic,
- error information,
- maintenance,
- parts information,
- hardware information.

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