Agilent InfinityLab LC Series

1260 Infinity III SFC System

User Manual

Notices

Document Information

The information in this document also applies to 1260 Infinity II and 1290 Infinity II modules.

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In This Book

This manual covers the Agilent InfinityLab LC Series 1260 Infinity III SFC System.

1 Introduction

This chapter provides an overview of the history, theory and benefits of SFC.

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History of SFC

Supercritical fluid chromatography (SFC) was first introduced by Klesper et al. in 1962 (Klesper, E.; Corwin, A. H.; Turner, D. A. J. Org. Chem. 1962, 27,700.) for the separation and analysis of a porphyrin mixture using open tubular SFC. The first commercial instruments using packed columns were available from Hewlett-Packard (HP) in 1982. Since then, several vendors have developed and commercialized packed column SFC instrumentation for analytical as well as for preparative separation. SFC is widely accepted for the separation of chiral compounds and increased user interest has been observed for a wide spectrum of small to medium sized molecules due to the analysis speed achieved and the low solvent consumption.

The latest introduction of analytical SFC instrumentation, the Agilent 1260 Infinity III SFC Control Module can be coupled to a Binary LC system optimized for SFC.

Theory of SFC

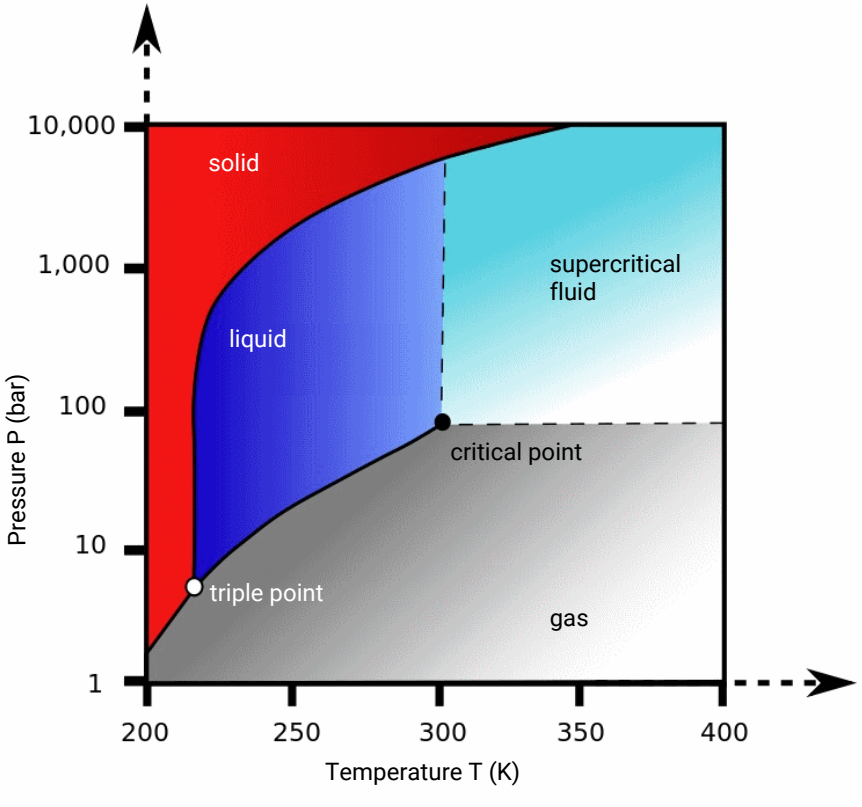

Figure 1: State of a solvent

The superior separation properties achieved by SFC can be explained best by the thermodynamics of liquids and gases (see the phase diagram in State of solvent on page 9). Above a critical pressure, liquids can no longer enter the gaseous state; similarly, above a critical temperature, gases cannot be converted to liquids. Above both the critical pressure and temperature (characterized by the critical point), solvents are in the supercritical state. Under these conditions, the mobile phases exhibit gaseous as well as liquid-like properties. *The major*

advantages of this state related to chromatography are improved diffusion characteristics and mass transfer and low viscosity, which result in high separation efficiency and fast separation capability.

Benefits of SFC

SFC is widely accepted for the analysis and separation of chiral compounds. In addition, it gains increasing acceptance as a complementary liquid-based separation technique to HPLC for high-throughput and high-resolution analysis of complex mixtures. This is due to the thermodynamic properties of supercritical fluids, which can be exploited for high throughput and high efficiency. In addition, the mild thermal conditions also allow for the analysis of thermally labile compounds. Typically, analysis times and column re-equilibration are decreased by a factor of 3–5 compared to standard HPLC. With the increasing costs of organic solvents and the environmental awareness to minimize toxic waste, production SFC is increasingly accepted as the “green alternative” to normal phase or reversed phase chromatography, gaining popularity in method development and UV- and MS-based separation and purification. A variety of parameters, such as stationary phase selection, mobile phase composition, modifier type and concentration, column temperature and system pressure, can be easily manipulated to fulfill separation requirements by influencing, optimizing and exploiting selectivity in SFC.

Common Flow Path Overview for Packed Column SFC Instrumentation

In commercially available SFC systems, CO_2 is initially pumped in liquid state and is brought into the supercritical state by heating it above the critical temperature before it enters the high-pressure area of the LC instrument. After high-pressure mixing with a modifier, the mobile phase passes into an injector, where the sample is introduced into the supercritical stream, and further transported to the separation column. The high pressure of the mobile phase must be maintained downstream of the detector outlet using a back-pressure regulator to keep the mobile phase in its supercritical condition over the complete flow path.

SFC System Components

The Agilent 1260 Infinity III SFC System consists of a G4782A SFC Binary Pump, G4301A SFC Control Module, G4767A SFC Multisampler, G7116A Multicolumn Thermostat, and either a G7115A Diode Array Detector WR or G7165A Multiple Wavelength Detector equipped with a high pressure SFC flow cell.

The SFC control module, redestills and preconditions the CO_2 by boosting the pressure to just below the column head pressure, relieving the SFC pump of any compression requirements. This results in low detector noise and significantly higher sensitivity. Therefore, the Agilent SFC pump receives pre-conditioned CO_2, and acts only as metering device for the mobile phase flow and to form the gradient with the second pump head by adding the appropriate amount of modifier solvent.

Downstream of the detector, the mobile phase is redirected back into the SFC control module to an integrated back-pressure regulator that maintains the back-pressure over the system.

The SFC system is designed for use with InfinityLab Level Sensing and Sample ID Reader and completely controlled by Agilent OpenLAB CDS software. The system diagram is shown below:

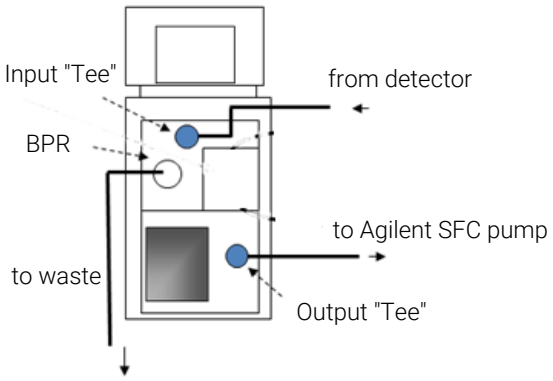

Figure 2: Agilent 1260 Infinity III SFC System

SFC Control Module (G4301A)

The Agilent 1260 Infinity III SFC Control Module is responsible for all tasks connected to pre- and post-conditioning of the mobile phase. In contrast, flow rate, mobile phase composition, detection, column temperature and data analysis are controlled by the modules of the SFC system in combination with the ChemStation software. This includes metering the carbon dioxide flow and mixing the modifier into the mobile phase by the SFC binary pump.

In detail, the SFC Control Module uses vapor-phase carbon dioxide, redestills it to the liquid state and boosts its pressure to just under the column head pressure. Since the CO_2 gas is a very poor solvent, most contaminants in the carbon dioxide are left in the source, which allows for the use of inexpensive, beverage-grade CO_2, unlike in any other commercially available instrument. The SFC control module further recollects the effluent from the UV (or other) detector and controls the back-pressure up to 400 bar over the complete system.

SFC Binary Pump (G4782A)

The Agilent 1260 Infinity III SFC Binary Pump is equipped with passive inlet valves and with special seals and valves to allow for CO_2 pumping in channel A while channel B adds organic modifier for either isocratic or gradient performance. Pump head B is also equipped with a purge valve to allow for quick changeover of the organic modifier. In addition, it has an integrated solvent selection 2-channel degasser and built-in active seal-wash on channel A for increased uptime.

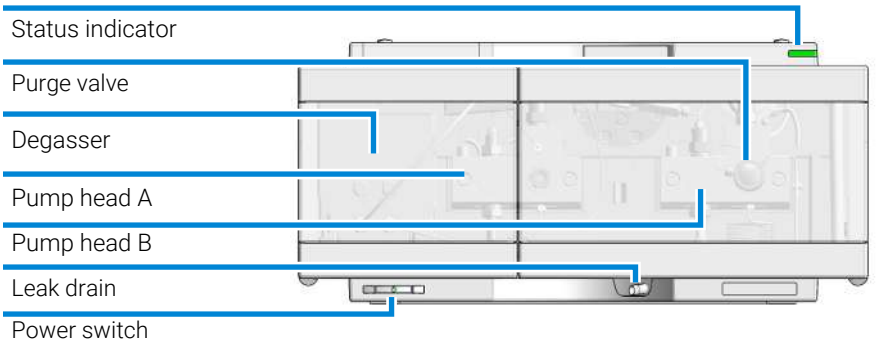

Figure 3: Overview of the pump

SFC Multisampler (G4767A)

In SFC, the complete solvent flow path needs to be pressurized under all conditions to avoid expansion of the supercritical fluid. With the *Feed Injection Technology* of the Agilent 1260 Infinity III SFC Multisampler, the sample volume is drawn under atmospheric pressure conditions, pressurized to system pressure, and injected by an ultrafast syringing process. This enables the injection of flexible sample volumes from 0.1 – 90 µL with highest precision, and excellent linearity over a broad volume range.

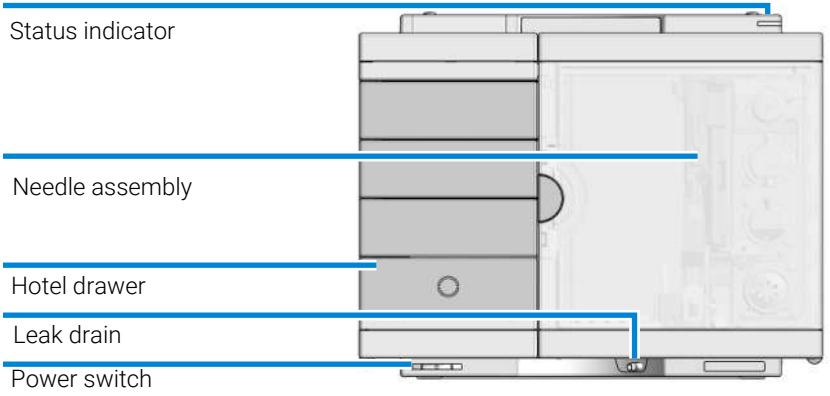

Figure 4: Overview of the Multisampler

Multicolumn Thermostat (G7116A)

The Agilent 1260 Infinity III Multicolumn Thermostat (MCT) facilitates precise column thermostatting over a broad temperature range with cooling down to 10 °C below ambient temperature and heating up to 85 °C.

This capability provides robust and reliable separations for maximum application flexibility. Exchangeable high-pressure valves enable a wide range of applications such as column selection of up to four columns, sample preparation for analyte enrichment or matrix removal, or alternating column regeneration.

The MCT matches perfectly with all InfinityLab LC Series systems and can also be combined with 1290 Infinity II/III Series modules as well as with previous 1260 Infinity and 1290 Infinity Series modules.

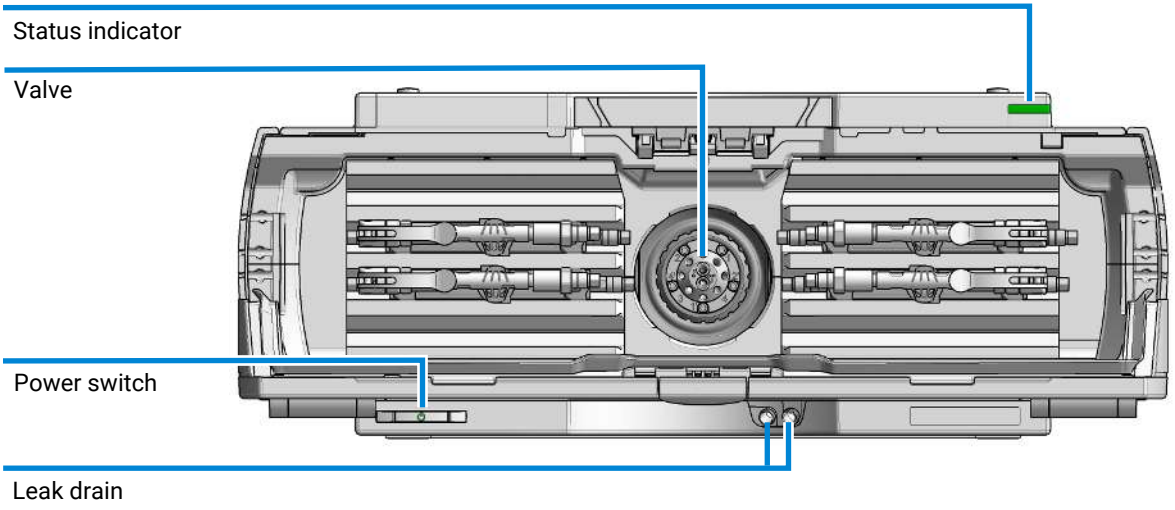

Figure 5: Overview of the Multicolumn Thermostat

Recommendations

The temperature of the mobile phase prior to detection is a critical parameter for minimizing baseline noise recorded in the detector flow cell. The heat exchanger on the right side of the column compartment is used to pre- heat the mobile phase before it enters the column, indirectly heating the column. The heat exchanger on the left side is used to change the temperature of the mobile phase to achieve minimum noise. This is of crucial importance, since the refractive index of carbon dioxide responds up to 50 times stronger than water- based mobile phases; thus, even small changes in temperature can significantly affect noise levels. Temperature changes of mobile phase with the left side heat exchanger e.g. between 38 °C and 49 °C resulted in a variation in peak-to-peak noise of over an order of magnitude.

The recommended temperature setting on the left heat exchanger in order to achieve minimum noise is typically between 37 °C and 40 °C for G7115A or G7165A detectors.

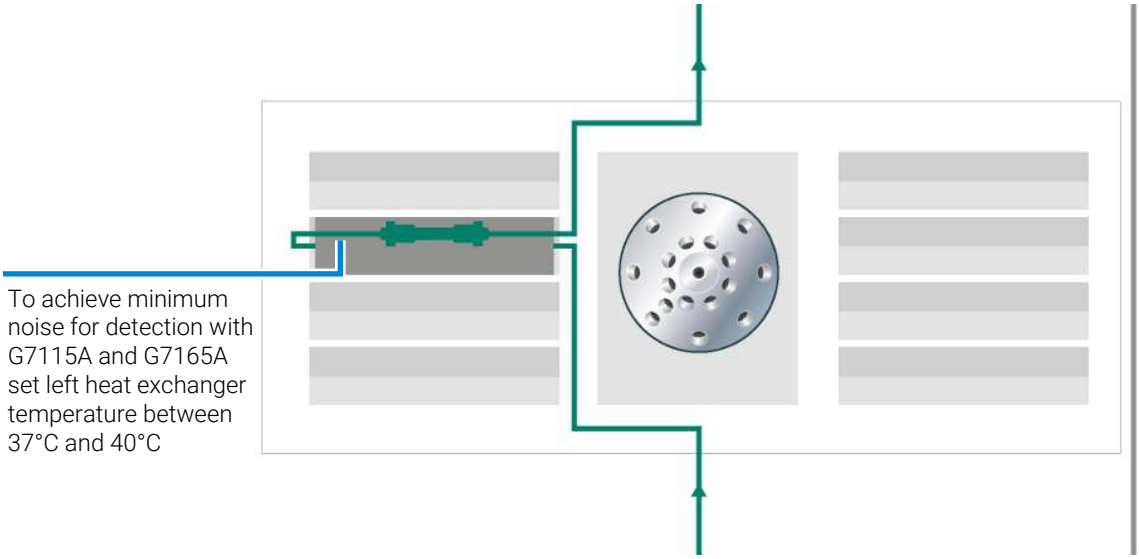

Figure 6: Recommended temperature setting on the left heat exchanger

Diode Array Detector WR (G7115A) and Multi Wavelength Detector (G7165A)

The system can be equipped either with a DAD or an MWD using a high pressure detector flow cell suitable for SFC (10 mm path length, 13 µL volume), with short transfer tubing to minimize peak broadening. Electronic temperature control provides highest baseline stability and stable sensitivity values under fluctuating temperature and humidity conditions. This feature aids greatly in minimizing detector noise, and now enables impurity and EE (enantiomeric excess) analysis by SFC, particularly when using elevated temperatures.

Applications

SFC has gained a wide interest and acceptance in many small molecule applications because of its high separation speed and efficiency, selectivity, low operating costs, and due to low generation of organic solvent waste.

Important applications have been developed for the analysis of pharmaceutical drugs, natural products, fatty acids, vitamins, pesticides, lipids and chiral compounds. See Figure 7 on page 19.

Columns

In contrast to reversed phase separation, there is no universal stationary phase available for SFC separations. Most typically used stationary phases are ethyl pyridine, diol, cyano, amino, Silica and SCX columns. This usually leads to additional effort to screen different columns in order to achieve optimum separation. On the other hand, it provides a valuable tool for achieving different selectivities for a given analyte mixture. A review of column developments for SFC was recently published by *T.Berger, B.Berger & R.E.Majors* in LCGC North America, May 1, 2010.

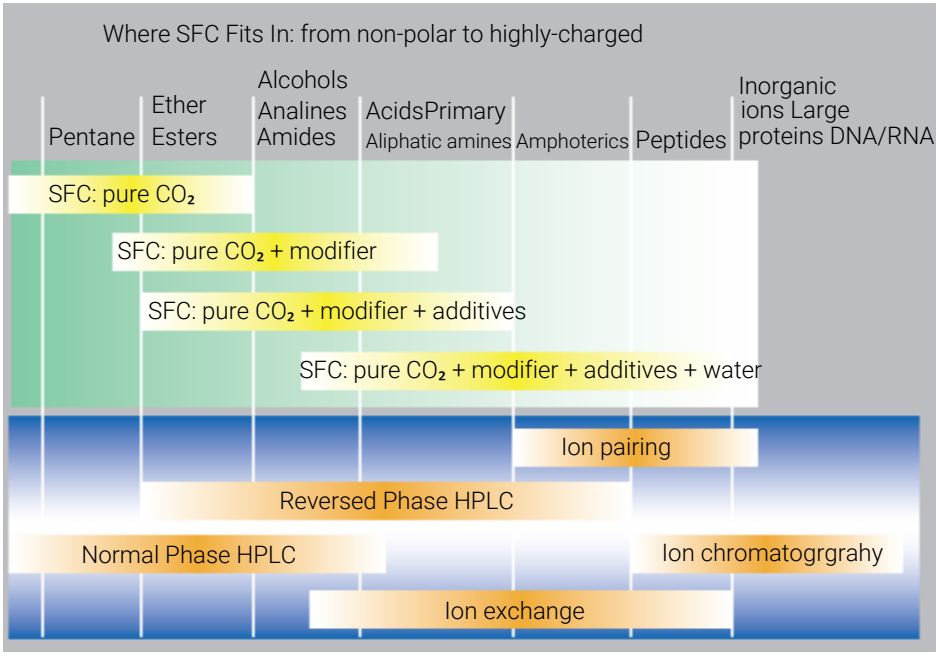

Figure 7: Where SFC Fits In: from non-polar to highly-charged

2 Site Requirements and Specifications

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

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Site Requirements

A suitable environment is important to ensure optimal performance of the instrument.

Power Considerations

The module power supply has wide ranging capability. It accepts any line voltage in the range described in System Specifications. Consequently there is no voltage selector in the rear of the module.

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.**

WARNING **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

Country-specific power cords are available for the module. The female end of all power cords is identical. It plugs into the power-input socket at the rear. The male end of each power cord is different and designed to match the wall socket of a particular country or region.

Agilent makes sure that your instrument is shipped with the power cord that is suitable for your particular country or region.

WARNING **Unintended use of power cords**

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

- **Never use a power cord other than the one that Agilent shipped with this instrument.**
- **Never use the power cords that Agilent Technologies supplies with this instrument for any other equipment.**
- **Never use cables other than the ones supplied by Agilent Technologies to ensure proper functionality and compliance with safety or EMC regulations.**

WARNING **Absence of ground connection**

The absence of ground connection can lead to electric shock or short circuit.

- **Never operate your instrumentation from a power outlet that has no ground connection.**

WARNING **Electrical shock hazard**

Solvents may damage electrical cables.

- **Prevent electrical cables from getting in contact with solvents.**
- **Exchange electrical cables after contact with solvents.**

Bench Space

Agilent G4301A SFC Control Module

The Agilent 1260 Infinity III SFC Control Module requires approximately 30 cm (1 ft) of linear bench space immediately adjacent to the target 1260 Infinity III system stack. Approximately 13 cm (5 inches) of free space is required behind the instrument for cable access and adequate air flow for ventilation. Similar access to the rear of the system is also required to install cables and interface cards. For optimal performance, the rear air space should not be heated significantly above room temperature by the exhaust of other instrumentation in the lab. Hot exhaust should be vented or directed upward from the instrument.

The module is designed to be installed on either side of the system stack with sufficient high pressure transfer tubing to attach to a double-stacked system. If the system is attached to a split-flow detector such as Mass Spec or ELSD, the G4301A SFC Control Module should be positioned on the opposite side of the stack. Shelves overhanging the G4301A SFC Control Module should provide a minimum of 15 cm (6 inches) of clearance to allow access to the rear power switch. Finally, the PC system interface to the G4301A SFC Control Module is USB 2.0. A 1.8 m (6 ft) cable is supplied with the system. The CPU must be placed within range of this cable. Alternately, the user may supply an extended length USB cable not to exceed 5 m (16 feet).

While the G4301A SFC Control Module can exist on either side of the 1260 Infinity III system, it is often easier to locate it on the left side. For more specification details see Agilent 1260 Infinity II SFC Control Module.

Other Agilent Modules

The dimensions and weight of your module (see Agilent 1260 Infinity II SFC Control Module) allow it to be placed on almost any laboratory bench. It needs an additional 2.5 cm (1.0 inches) of space on either side and approximately 8 cm (3.1 inches) at the rear for the circulation of air and electric connections.

The G4301A SFC Control Module should be operated in a vertical, the other modules in a horizontal position.

If a Column Compartment is installed, an additional 25 cm (10 inches) of space on either side for the circulation of air, and approximately 8 cm (3.1 inches) at the rear is required for electrical connections.

If a complete 1260 Infinity III system is to be installed on the bench, make sure that the bench is designed to carry the weight of all the modules. For a system including the Thermostatted Autosampler it is recommended to position the modules in two stacks.

Condensation

CAUTION

Condensation within the module

Condensation can damage the system electronics.

- Do not store, ship or use your module under conditions where temperature fluctuations could cause condensation within the module.
- If your module was shipped in cold weather, leave it in its box and allow it to warm slowly to room temperature to avoid condensation.

Ventilation

WARNING

Waste tube has to be connected to hood or vent

- **The effluent from a supercritical fluid chromatograph may contain vaporized, toxic solvents. Never vent into an enclosed, occupied space. Always vent into a fume hood or vent to the outside.**

Specifications of the 1260 Infinity III SFC System

Table 1: Specifications of the 1260 Infinity III SFC System

Type	Specifications	Comments
Settable flow range	0.001 mL/min to 5 mL/min	recommended: 1 mL/min to 5 mL/min
Maximum operating pressure	600 bar up to 5 mL/min	
Upgrade of existing Infinity and Infinity II setups	Yes	
Option to run SFC/ UHPLC in one system	Yes	
Unattended operation	Leak sensors, diagnostic software features	
Control and data evaluation	Agilent OpenLAB CDS ChemStation Edition; OpenLAB CDS 2; OpenLAB CDS EZChrom Edition; Instrument Control Framework.	

Specifications of the 1260 Infinity III SFC Control Module (G4301A)

The Agilent 1260 Infinity III SFC Control Module is heavy (approximately 26 kg or 56 lbs). Enlist the aid of a co-worker to share the lifting load in order to avoid possible injury. Select the laboratory bench space before your system arrives. Pay special attention to the total height requirements. Avoid bench space with overhanging shelves. Pay special attention to the total weight of the modules and solvents you have in addition to the G4301A SFC Control Module. Make sure that your laboratory bench can support this weight.

Table 2: Physical Specifications of the 1260 Infinity III SFC Control Module (G4301A)

Type	Specification	Comments
Weight	26 kg (56 lbs)	
Dimensions (height × width × depth)	60 cm x 26 cm x 48 cm (23 in x 10 in x 18 in)	
Line voltage	100 - 240 VAC, ±10 %	
Line frequency	50 - 60 Hz, ±5 %	
Power consumption	700 VA max	
Operating temperature	15 – 30 °C (59 – 86 °F)	
Non-operating temperature	-40 – 70 °C (-40 – 158 °F)	
Humidity	<95 %, at 25 – 40 °C	Non-condensing
Laboratory ventilation	Minimum 6 air exchanges/hr for lab air;	CO_2 monitor recommended w/ alarm @5000 ppm
Exhaust vent capacity	>20 L/min with sustained negative pressure	
Operating altitude	up to 3000 m (9842 ft)	
Safety standards: IEC, EN, CSA, UL	Overvoltage Category II, Pollution Degree 2	For Indoor Use only
ISM Classification	ISM Group 1 Class A	According to CISPR 11

Table 3: Chemical Specifications (G4301A)

Type	Specifications
Inlet CO_2 bulk purity	>99.99 % vapor; >99.999 % liquid
Inlet CO_2 phase	vapor from non-dip-tube high pressure cylinder; liquid from commercial CO_2 delivery system
Inlet CO_2 supply pressure	40 - 70 bar (580 - 1000 psi)
Inlet CO_2 temperature	15 - 30 °C (59 - 86 °F)
Liquid coolant	30 % propylene glycol in deionized water; proprietary antioxidants; red dye added for safety
Coolant volume	< 280 mL

Table 4: Wetted Materials Specifications (G4301A)

Type	Specifications
High pressure flow path	300 and 400 series stainless steel, carbon filled PEEK, UHMW PE, sapphire, ceramic, gold
Low pressure flow paths [waste, wash pump, leak tray] [1]	316 stainless steel, PEEK PTFE, FEP, CTFE CPE; LDPE Tygon PVC
Vapor exhaust	Tygon PVC

Table 5: Performance Specifications (G4301A)

Type	Specifications
Hydraulic system	Single piston with proprietary motor control
Total hydraulic volume	<5 mL at pressure <70 bar <25 mL at pressure up to 400 bar
Chiller system	Thermoelectric cooling with secondary air/liquid cooling circuit
Chiller temperature	-20 – 9 °C (-4 – 48.2 °F)
Back-Pressure Regulation (BPR) system	Low volume diaphragm type with proprietary drive control; replaceable BPR head assy; No recalibration required after head replacement
Booster pump pressure range	100 – 400 bar up to 5 mL/min demand
BPR thermal range	40 – 70 °C (104 – 158 °F)

1 Only when a legacy wash pump is installed.

Type	Specifications
BPR thermal precision	±1 K
BPR settable pressure range	100 – 400 bar
Back-pressure precision	±0.25 bar
Back-pressure thermal precision	±1 K
Programmable back-pressure gradient	Yes. Up to 20 timetable entries.
Communications	USB 2.0; APG Remote: ready, start, stop and shut-down signals; relay contact closure (wash pump only)

3 Installation

The installation of the module will be done by an Agilent service representative. In this chapter, only installation of user-installable options and accessories are described.

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General Procedures

Proper use of wrenches

Some of the plumbing connections require a nut to be tightened onto a fitting. There are often two sets of flats next to each other. Attempting to tighten the nut without securing the other part of the fitting with a second wrench can result in loosening yet another connection upstream or downstream. It is best practice to always hold the fitting with one wrench while tightening or loosening another connection.

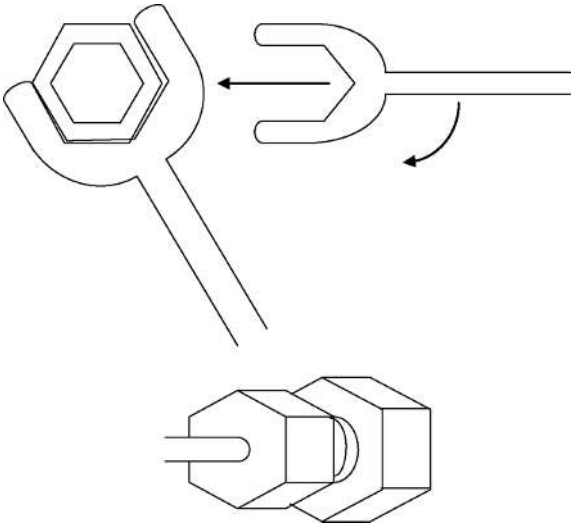


Figure 8: Proper tightening of Fittings

Compression (Swaged Fittings)

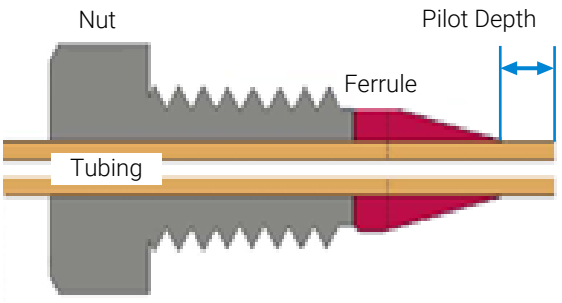
The fittings used in the Agilent 1260 Infinity II/III SFC Control Module are Valco. Fittings used in all Agilent modules are Swagelok. Use the appropriate fitting as recommended by the equipment manufacturer. The recommended tightening

procedure to install new fittings is to tighten the nut finger tight, then an additional 1/4 to 1/2 turn to seal. In general, previously swaged fittings need only an additional 1/8th turn once finger tight.

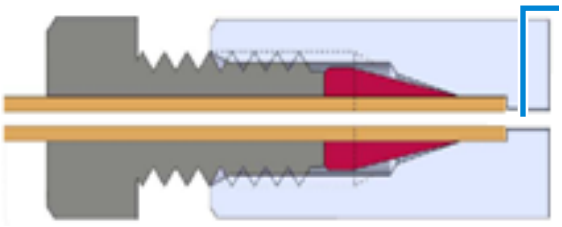
In Supercritical Fluid Chromatography, the fluid has 1/10th the viscosity of water, so this may not be tight enough. All connections should be checked for leaks and tightened further if necessary. Soapy water or Snoop make it easy to find leaks if carbon dioxide is in the fluid. Tiny bubbles appear in the liquid around the fitting.

Each fitting should be individually and carefully installed. The depth of the tube inside the fitting is very important. If the tube pilot (length beyond the ferrule end) is too long, the fitting can leak or, after excessive tightening, bind permanently. If the pilot is too short, a poorly swept volume can be created. This poorly swept volume will create noticeable chromatographic tailing. If the pilot is much too short, the fitting could fail under use. Pilot depths are not always interchangeable between fittings. It is a best practice to swage a tube in the fitting in which it will be used. It is best to provide some light force to hold the tube in the fitting and prevent the tube from exiting while tightening the fitting.

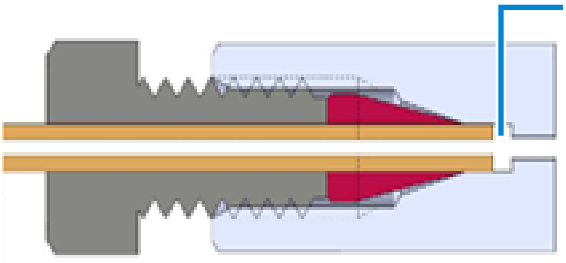
Excessive force can result in breakage of some components, and should obviously be avoided. It may be more expedient to replace the whole fitting if one of the connections fails to seal. You may notice that some of the more expensive components (such as a pressure transducer) have a less expensive fitting mounted to them to act as a sacrificial fitting. Connections should be made to the less expensive component, and repetitive removal and replacement to one of the more expensive fittings should be avoided.



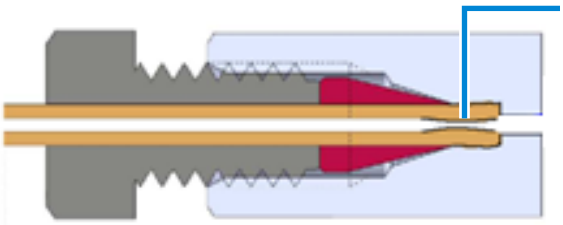
Compression fittings used in the SFC control module use a nut and ferrule. As the nut is tightened the ferrule compresses on the tubing creating a leak-tight seal



When correctly installed, there is minimal additional volume. All volume is well swept.



When improperly set, the pilot does not fill the fitting, leading to additional poorly swept volumes.



If over-tightened, the ferrule can compress the tubing and cause sufficient deformation to prevent removal of the fitting.

Compression fittings

Installing the G4301A SFC Control Module

Preparation

Locate all modules, devices and supporting equipment before continuing. Ensure that the supply tubing can reach a physically secured source of CO_2. Ensure that adequate venting is available and within reach of supplied waste systems.

This document describes a particular order of plumbing the system, with plumbing and electrical connections described last. These operations are performed at the rear of the systems. Depending upon your individual installation, you may wish to perform operations at the rear of the instruments first. This is perfectly acceptable, provided you can maintain access to supply connections to ensure integrity and leak tightness of fittings and connections.

Connect the Booster to the SFC System

Locate the stainless steel booster transfer line. Connect one end to the top port of the output tee on the center right side of the booster drawer. Tighten finger tight with an additional 1/8th turn as needed.

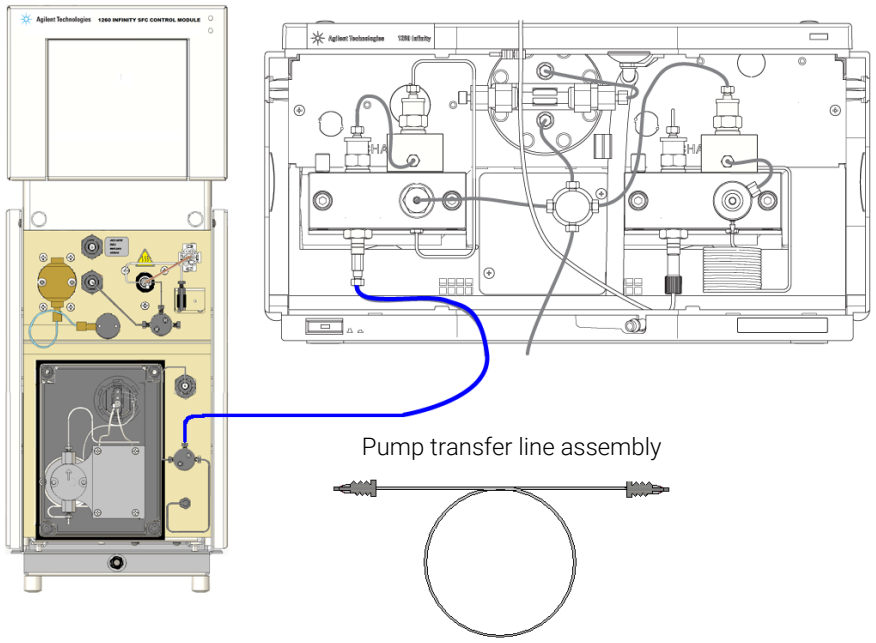


Figure 9: Connecting the Booster outlet line

This tube can be routed through the tee slots on the side of the SFC control module. The tube is routed upward between the SFC control module and the Agilent 1260 Infinity II/III stack to the bottom side of the binary pump. The tube is then routed horizontally to the bottom center of the binary pump where it can enter the concave opening behind the cover. The tube should then be routed to the left side of the pump beneath channel A (left side). The end of the booster transfer tube can then be installed with the adapter in the passive inlet valve.

Any spare tubing can be located between the SFC control module and HPLC stack.

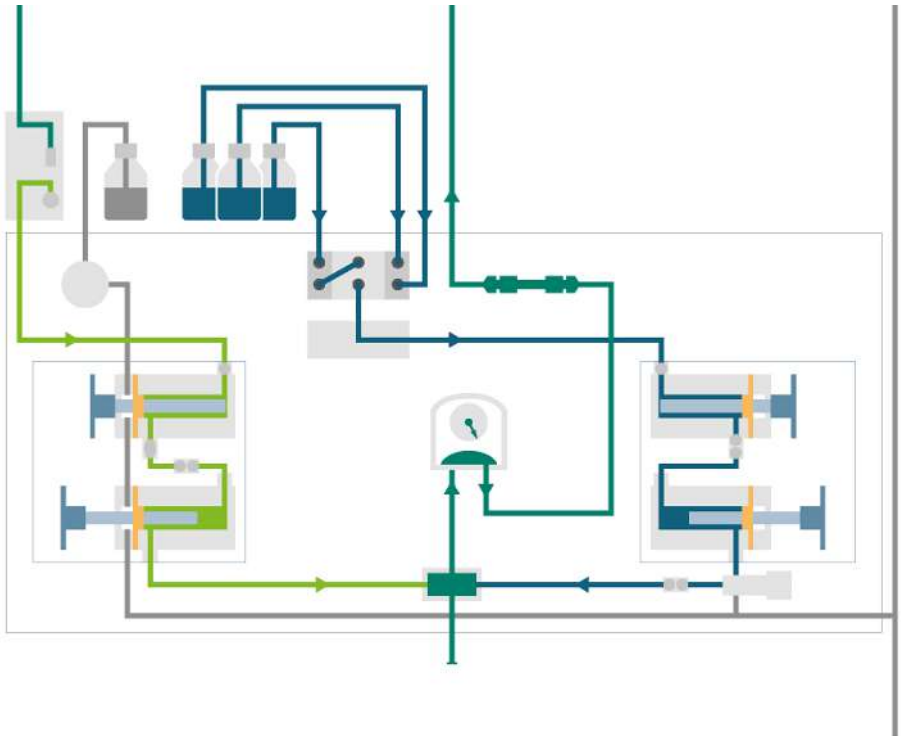


Figure 10: Plumbing of the SFC Binary Pump

Installing the G4767A SFC Multisampler

Connect the capillary from the SFC binary pump outlet to port 1 in the SFC Multisampler's injection valve and from port 6 to the MCT.

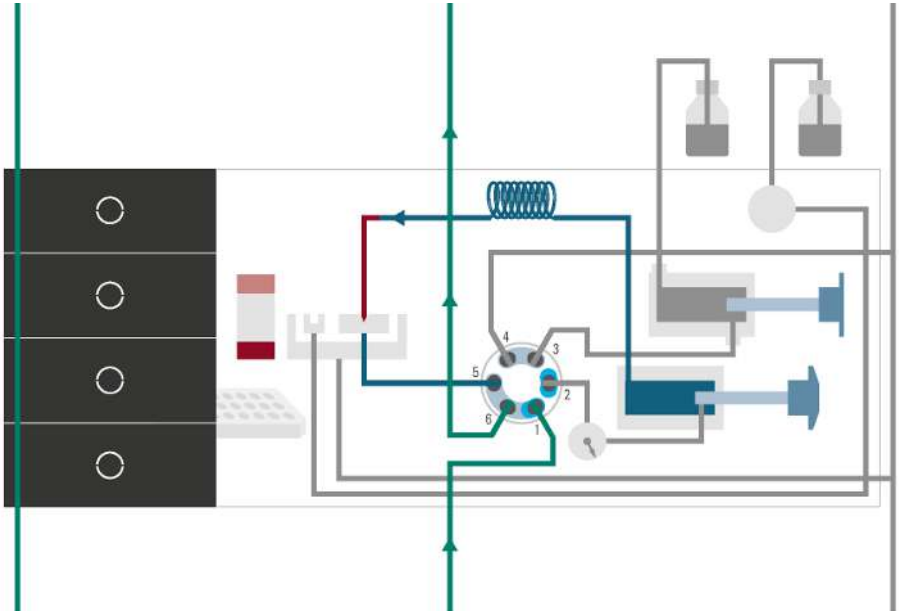


Figure 11: Plumbing of the SFC Multisampler

Optimized Thermostatted Column Compartment Plumbing

Supercritical Fluid Chromatography (SFC) is susceptible to increased noise due to poor thermal matching of components within the Agilent 1260 Infinity II/III stack. The refractive index of carbon dioxide is 50 times more susceptible to temperature changes than water. Consequently, thermal control in SFC is extremely important. The Multicolumn Thermostat (MCT) contains two thermal conditioning zones that can greatly increase system performance by matching temperatures of the mobile phase to the modules being used.

Each of these zones contains heat exchangers that can be used to thermally condition the fluid flowing through them. The two zones exist on the left and right side blocks within the MCT.

The right zone is used to precondition the mobile phase before it enters the column. When using 150 mm or shorter columns, place them in the right side of the oven. This zone is used to precondition the fluid to column temperature and provide thermal control of the column.

The left zone is plumbed with the effluent from the column. The purpose of the left side block is to independently match the temperature of the mobile phase to the optimum temperature for the detector. In order to achieve minimum noise it is important to use the optimum conditioning temperature prior to detection.

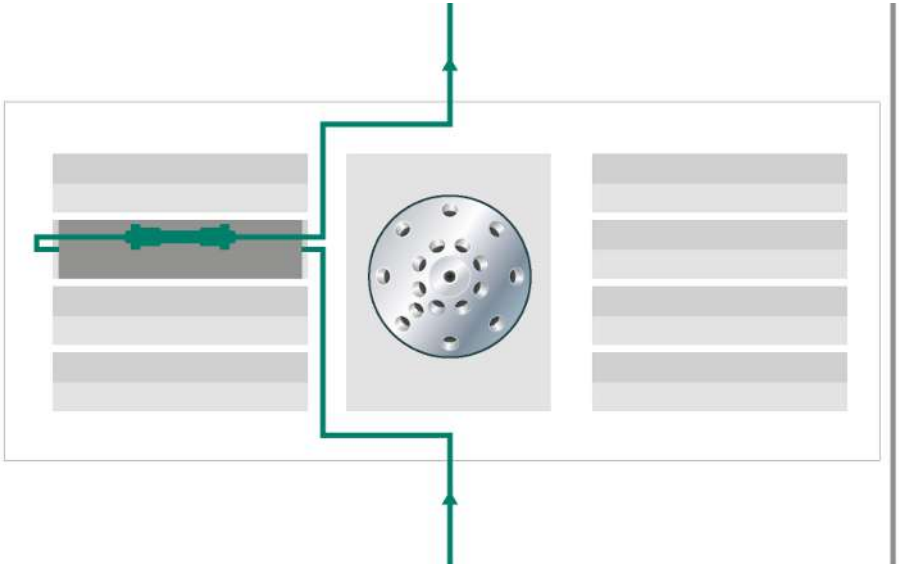


Figure 12: Optimized plumbing of the MCT

Installing the Flow Cell

The back-pressure regulator exists after any detectors in the HPLC system. Thus, the detector flow cells (or splitter in the case on an ELSD or Mass Spectrometer), operate at an elevated pressure relative to HPLC.

Agilent Technologies offers a Diode Array Detector (DAD) flow cell that has been extensively optimized for use in Supercritical Fluid Chromatography (SFC). This cell is pressure-rated and tested to 400 bar. It contains extensive thermal conditioning not found in standard HPLC flow cells. Agilent Technology's cell is highly recommended for SFC usage.

The flow cell should already be installed in the DAD/MWD. Carefully examine the inlet and outlet ports of the cells to ensure that flow is in the correct direction. In the DAD/MWD, the outlet port is normally located below the inlet port on the connection block.

The inlet port of the DAD/MWD flow cell enters the stationary portion of the handle. This stationary bar acts as an initial thermal conditioning zone. Normally, this port has a male fitting. The outlet port connects directly to the cylindrical portion of the flow cell. Normally, this connection has a female fitting.

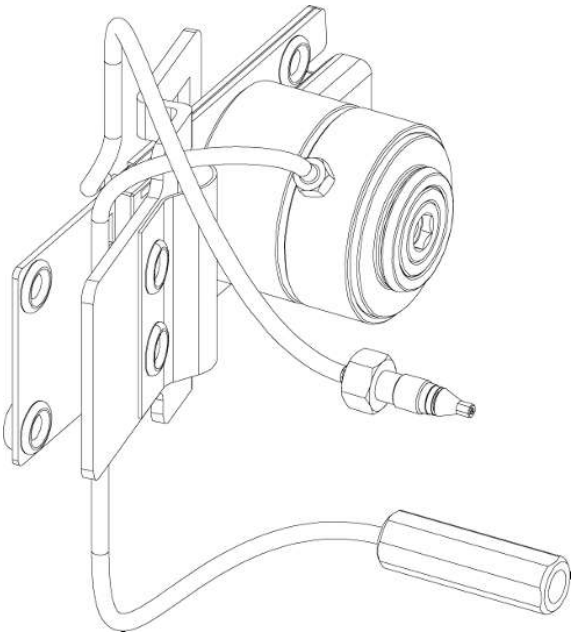


Figure 13: DAD SFC Flow Cell

CAUTION

Damage to the flow cell

— Verify that the cell installed in the detector is capable of the high pressures used in Supercritical Fluid Chromatography (SFC). Exposing a standard 1260 Infinity II/III flow cell to high pressures will result in leakage or damage to the cell.

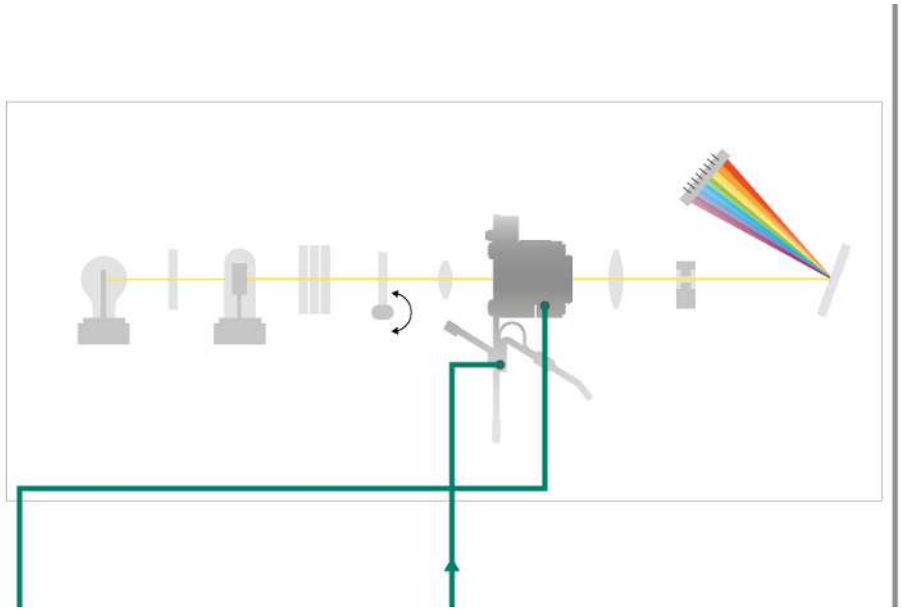


Figure 14: Plumbing of the flow cell

Connecting the BPR to the HPLC Stack

Connect the SFC control module return transfer tube to the outlet block of the detector. This tube can then be routed out the concave opening in the bottom of the detector behind the detector cover. The return transfer tube should then be routed to the space between the HPLC stack and the SFC control module. Move the tube upward between the units and through the upper tee-slot on the side cover of the SFC control module. The return transfer tube can then be fastened in the right port of the tee in the lower center of the BPR drawer.

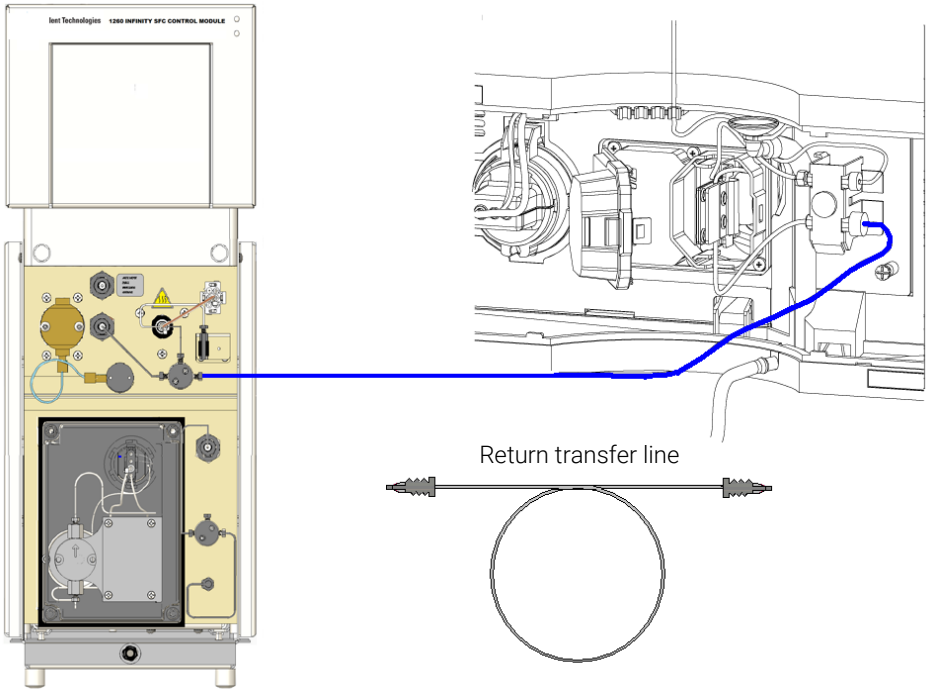


Figure 15: Connecting the SFC control module return line

Connecting the Waste System to the Fusion BPR Outlet

The SFC Control Module has a waste bottle located outside of the cabinet. It can be located anywhere easily accessible and visible within the range of the supplied tubing. The waste bottle serves multiple purposes and collects liquid waste from multiple sources. The primary purpose is to separate the gaseous and liquid waste from the outlet of the BPR (system) in such a manner that the gaseous waste can be appropriately vented outside of the lab environment. The waste bottle has input and output ports located above any collected liquids. The mixed stream enters the waste bottle and the gaseous stream exits from the spout.

NOTE

Proper system operation requires adequate space in the waste bottle to allow gaseous exit. It is the responsibility of the operator to ensure that the waste bottle is empty before beginning operation of the SFC control module, and to monitor and empty the waste bottle as needed during usage. This is not a warning.

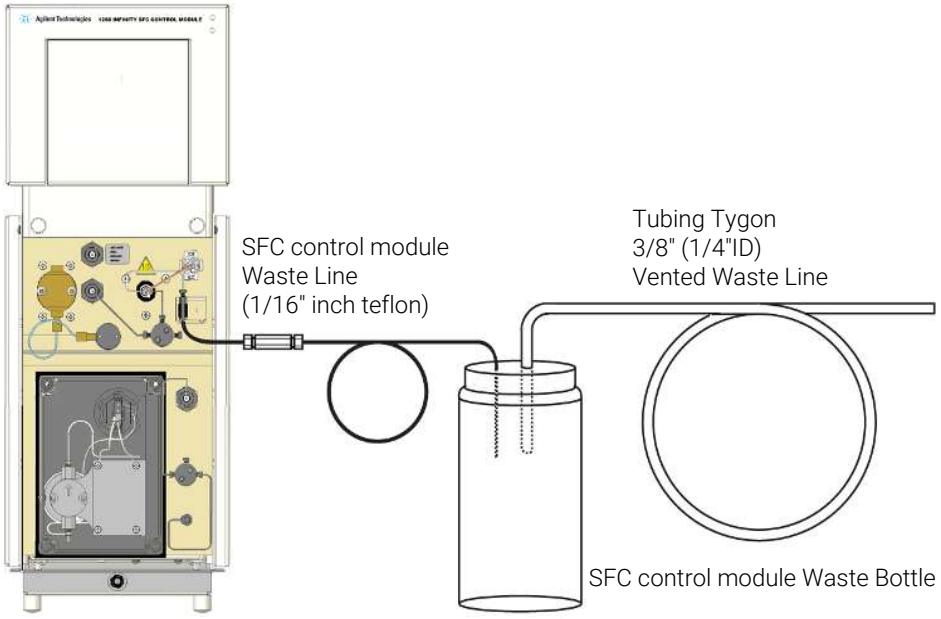


Figure 16: Installing the Waste Container

WARNING **Exposure to toxic substances**

— **The vapor exiting the module may contain several percent organic solvent. The effluent should NEVER be vented directly into an enclosed space occupied by humans because of the potential for long-term exposure to toxic substances.**

Locate and assemble the waste bottle and Tygon vent tubing. The vent tubing can be placed over the spout on the top of the waste bottle. Route the Tygon tubing to an appropriate vent. The system must be actively vented.

Locate the SFC control module Waste line. Insert the free end through a hole in the top of the waste bottle cap. Insert the tube half-way into the waste bottle. Connect the fitting end to the outlet union on the BPR. This union uses a 10-32 CPI fitting. Tighten snugly.

Connecting the Leak Tray Waste Line

The SFC control module contains a leak tray on the bottom of the instrument to collect and sense any liquid spills that may occur in the SFC control module cabinet. In the bottom of this leak tray is an active sensor that continuously monitors for the presence of liquid.

The drip tray contains an overflow drain to divert any large amounts of collected liquid to an external collection container. The overflow tube incorporates a simple push-to-connect fitting. It is connected to the port on the bottom center on the front of the instrument. Pushing on the outside ring of the port allows this line to be removed.

Since this liquid may be organic solvents, you should supply an appropriate collection container.

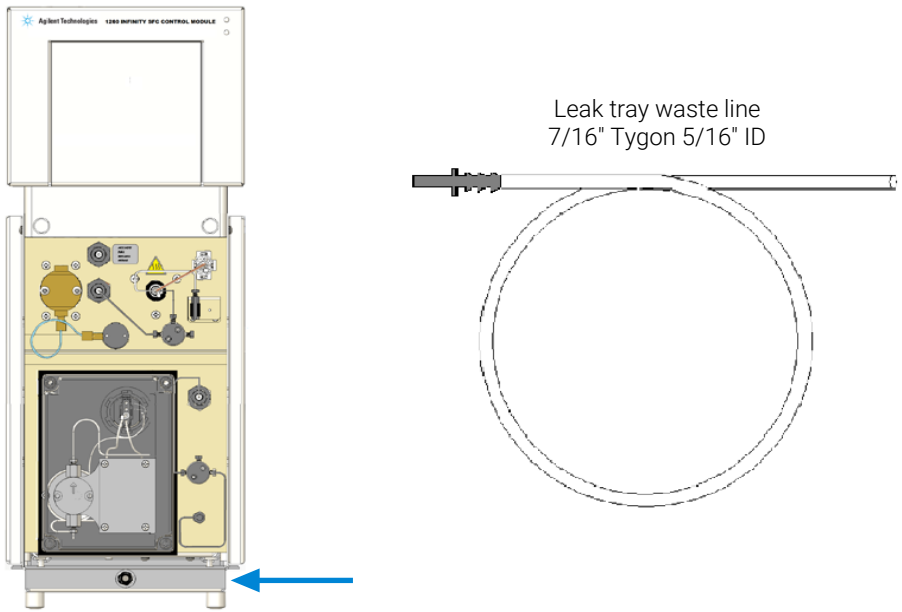


Figure 17: Leak Tray Waste Line

Cable Connections to the HPLC

WARNING

Personal injury

— Ensure that the AC power cord is NOT yet connected to the instrument.

WARNING

Electric shock due to insufficient insulation of connected instruments

Personal injury or damage to the instrument

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

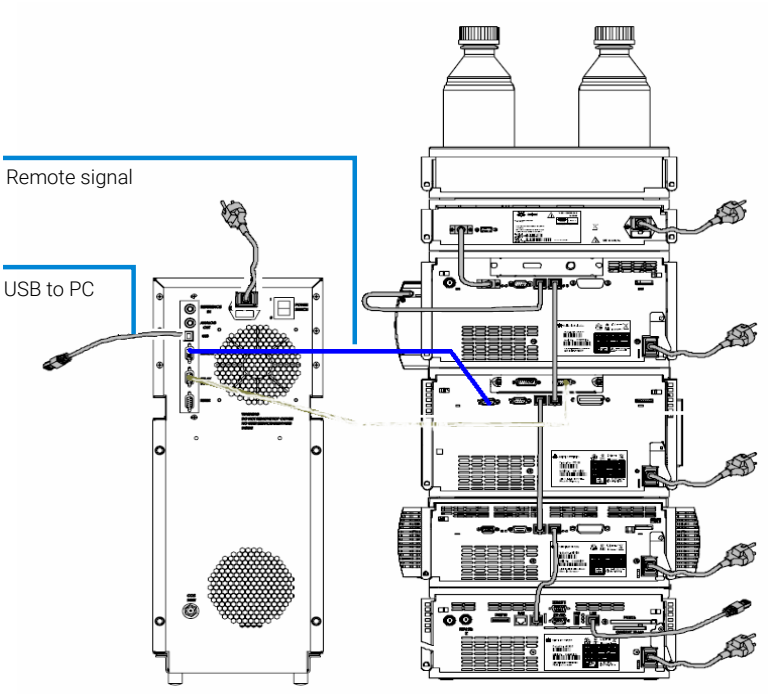


Figure 18: Remote Connection 5188-8045 Remote Cable APG-ERI

	Power off
	Power on

Table 6: Electrical Connectors

Reference IN	Analog signal in, not used
Analog OUT	Analog signal out, not used
USB	USB connector to control PC, used for communication with controlling software
Remote	Remote connector for Start/Stop signal
Relay Input	Relay connector to control wash pump, not used anymore (legacy from old system)
RS232	RS232 connector, used in manufacuring to flash first Firmware on board

Lastly, a cable should be connected to the SFC control module USB port. This can be run to any convenient, compatible USB port on the PC.

CAN cabling between the modules is unchanged from the normal, recommended means of interconnecting HPLC devices.

The HPLC stack requires a LAN connection to the PC. This LAN connection requires all the normal HPLC/ChemStation properties such as BOOTP, Firewall, and IP Address settings. These remain unchanged in an SFC Control Module installation.

Connecting and operating multiple instruments on a single PC is not supported. Both the SFC control module USB and LAN connections must be made on the PC on which ChemStation is installed and which is used for instrument control.

Connecting the SFC Control Module to a Source of Carbon Dioxide

The SFC Control Module has a 1/8 inch tube inlet connection on the lower left side on the back of the module. This connection is actually part of a very high surface area filter intended to intercept catastrophic levels of particulates. You need to connect this input to a source of carbon dioxide. The most common source of carbon dioxide is liquefied carbon dioxide from a room-temperature cylinder. At room temperature, the pressure in the cylinder could change from a little above 50 bar to just below 70 bar. Unlike most SFCs, Agilent's G4301A SFC Control Module is immune to the variations in flow resulting from cylinder pressure.

WARNING **Creating severe frostbite in a short time**

- **Expanding carbon dioxide can become extremely cold, capable of creating severe frostbite in a short time. Avoid contact with expanding gases. Do not vent substantial quantities into the laboratory.**

WARNING **Use the system in a well ventilated area**

- **Carbon dioxide is poisonous at high concentrations and should only be used in well ventilated areas. The system effluent should be vented into a fume hood or to the outside. Evacuate if a large spill occurs. A carbon dioxide sensor/alarm is recommended.**

Individual Cylinders

WARNING

CO_2 cylinders can be dangerous if handled improperly

- **Carbon dioxide in cylinders is partially liquefied under high pressure and contains a great deal of energy. If containment is breached (a break in the line or cylinder) the entire contents will vaporize and quickly expand up to 500 times in volume and create very forceful high velocity gas streams. Cylinders must be properly constrained, and proper tubing used, to avoid damage that could generate projectiles.**

Any industrial grade of carbon dioxide is acceptable provided it is supplied in a cylinder without a DIP tube. Drawing off the vapor phase leaves non-volatile contaminants behind in the cylinder. Using cylinders with a DIP tube subjects the chromatograph to contaminants soluble in the dense, liquid layer.

Larger tanks are more convenient in that they require to be changed less frequently. Cylinders can contain up to 35 kg of CO_2. Generally, 4.6 mm columns are run at 3 – 5 mL/min, which is approximately 2.5 – 4 g/min of carbon dioxide. This is equivalent to 150 – 250 g/h; 1.2 – 6 kg/day. Thus, a 15 kg cylinder should last 2.2 to 11 days; a 25 kg cylinder would last 4 to 19 days; and a 35 kg cylinder could last 5.3 to 27 days - all depending on use (3 – 5 mL/min; 8 – 24 h/day). For individual users, particularly new users, the use of cylinders is perfectly acceptable. Larger groups should consider installing a gas delivery system and a bulk storage tank.

Locate the cylinder as close to the instrument as possible. In the past, the cylinders were stored at much higher temperature than the lab temperature, which resulted in vaporization in the supply line coming into the lab. Most SFC pumps cannot condense this vapor and therefore, cannot deliver CO_2. Agilent's SFC control module has a very powerful condenser designed to accept vapor phase CO_2. Nevertheless, it is always advisable to not stress any equipment.

Facilities and safety personnel often wish to store and mount the cylinders outside the lab - sometimes quite far from the intended location of the instrument. They should recognize that the transfer lines can hold large volumes, equivalent to a large fraction of a cylinder, particularly if tubing with large ID is used. Shut-off valves at both ends of a transfer line are not recommended, unless one or both has a pressure relief valve or burst disk.

Be sure the cylinder is properly constrained and cannot tip over. Suitable chains or cylinder straps are required.

Cylinders in the USA and Canada use a CGA 320 cylinder adapter. The filter fitting sticking out the back of the SFC control module contains 1/8 inch nut and ferrule(s), which could be used with the supplied 1.8 m (6 feet) tube, or a longer, user-supplied tube, to connect a cylinder to the SFC control module.

There are at least four different European standards for the connection of carbon dioxide cylinders. They do not appear to change at national borders (some users in one country have different supply connections from other users in the same country). Agilent Technologies recommends that customers in Europe contact their gas supply companies and ask them how to mate the cylinders they supply with an American 1/8 OD supply line. This should require a cylinder connector and a reducing union down to a 1/8 inch compression fitting.

WARNING

Leaks will not be sensed or protected

- **Carefully check supply cylinders and inlet fittings for leaks. Any leaks present in the supply line and inlet fitting will not be sensed or protected by safety features in the module.**

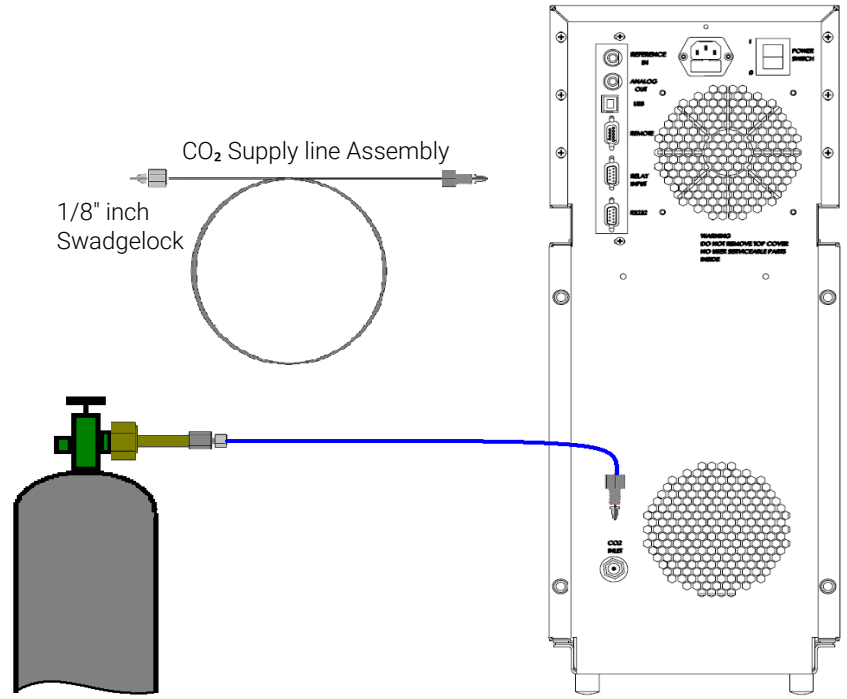


Figure 19: Figure 3.25

O	Power off
I	Power on

WARNING

Improper plumbing can cause leaks

- **It is imperative to use two wrenches to install the supply fitting in the bulkhead filter (entry connection) on the back of the module. Turning or twisting the bulkhead supply filter could cause failures or leaks in tubing within the module.**
- **All supply fittings need to be thoroughly checked for leaks. Any leaks in the supply fittings can vent the CO_2 supply.**

CAUTION

Overtightening the fitting could damage the filter

— Use two wrenches when installing the CO_2 supply line to the bulkhead filter (entry connection) on the back of the module. Although the filter is captured and should not rotate, there is a remote possibility that fittings inside the module could be loosened by severe stress on the nut on the supply line. Leakage inside the cabinet requires service by trained personnel.

Gas Delivery Systems (GDS)

Anyone performing semi-prep SFC has probably been convinced that operation without a GDS is problematic. Thus, many laboratories are now plumbed with carbon dioxide boosted to 70 – 80 bar outlet pressure. Even though an analytical system does not need such a GDS, it is perfectly adapted to its use.

The G4301A SFC Control Module has an inlet safety shut-off valve rated to 1500 psi (>100 bar). While this is rating is well above the outlet pressure of any typical GDS, past experience suggests that it is wise to allow for some extra margin. Agilent Technologies suggests setting the local output of any GDS between 60 bar and 70 bar, through local outlet pressure regulators.

Under these conditions, the GDS will almost always provide liquid carbon dioxide to the chromatograph. Unlike some earlier systems, the G4301A SFC Control Module easily condenses any fluid that is present as a vapor, and prevent pump cavitations.

System Flow Diagram

Figure 20 on page 54 schematically shows the correct plumbing of the SFC system.

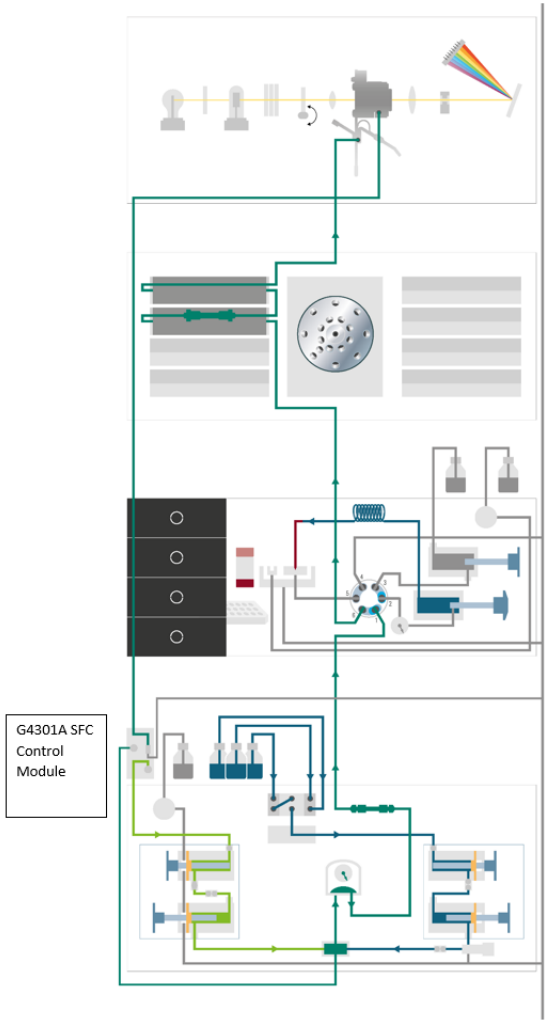


Figure 20: SFC System flow diagram

Handling Leak and Waste

The Agilent InfinityLab LC Series has been designed for safe leak and waste handling. It is important that all security concepts are understood and instructions are carefully followed.

The solvent cabinet is designed to store a maximum volume of 8 L solvent. The maximum volume for an individual bottle stored in the solvent cabinet should not exceed 2 L. For details, see the usage guideline for the Agilent Infinity III Solvent Cabinets (a printed copy of the guideline has been shipped with the solvent cabinet, electronic copies are available on the Internet).

All leak plane outlets are situated in a consistent position so that all Infinity and Infinity II/III modules can be stacked on top of each other. Waste tubes are guided through a channel on the right hand side of the instrument, keeping the front access clear from tubes.

The leak plane provides leak management by catching all internal liquid leaks, guiding them to the leak sensor for leak detection, and passing them on to the next module below, if the leak sensor fails. The leak sensor in the leak plane stops the running system as soon as the leak detection level is reached.

Solvent and condensate is guided through the waste channel into the waste container:

- from the detector's flow cell outlet
- from the Multisampler needle wash port
- from the Sample Thermostat (condensate)
- from the pump's Seal Wash Sensor (if applicable)
- from the pump's Purge Valve or Multipurpose Valve

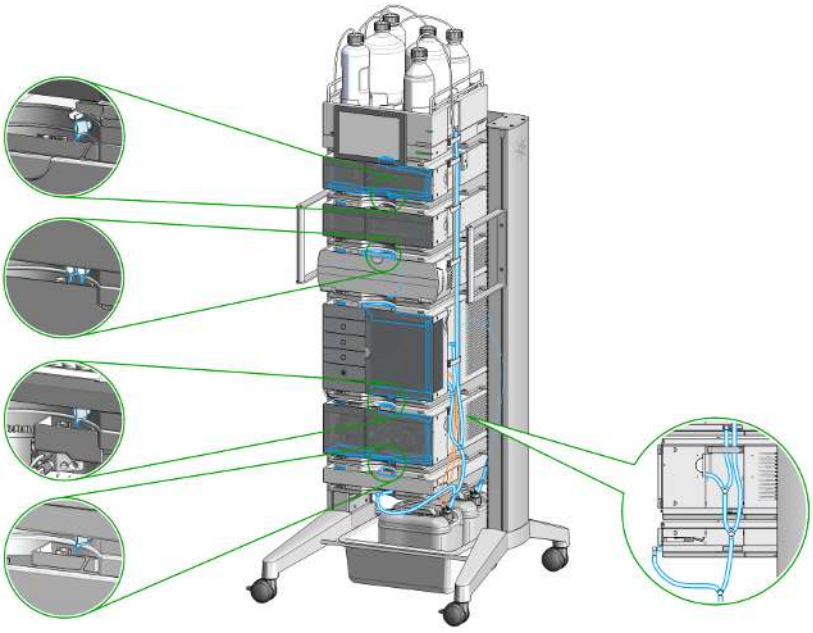


Figure 21: Infinity III Leak Waste Concept (Flex Bench installation)

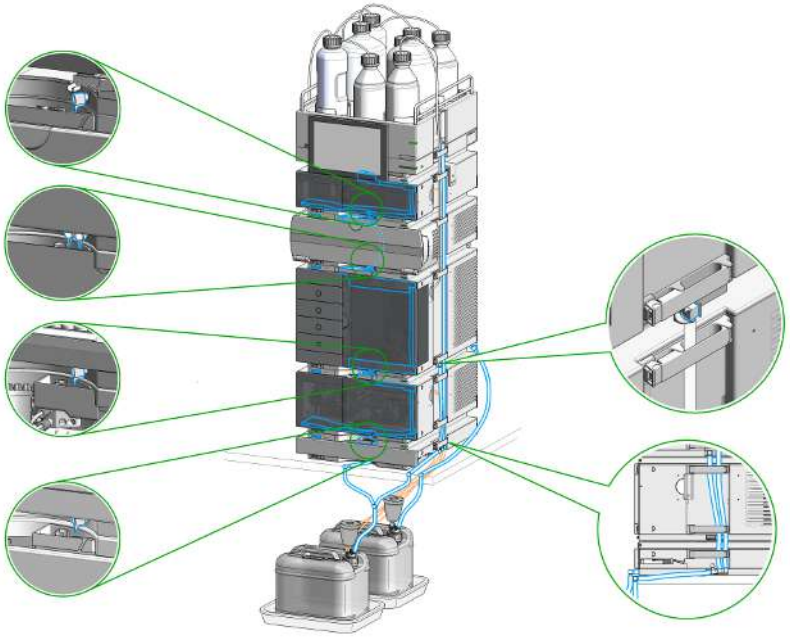


Figure 22: Infinity III Single Stack Leak Waste Concept (bench installation)

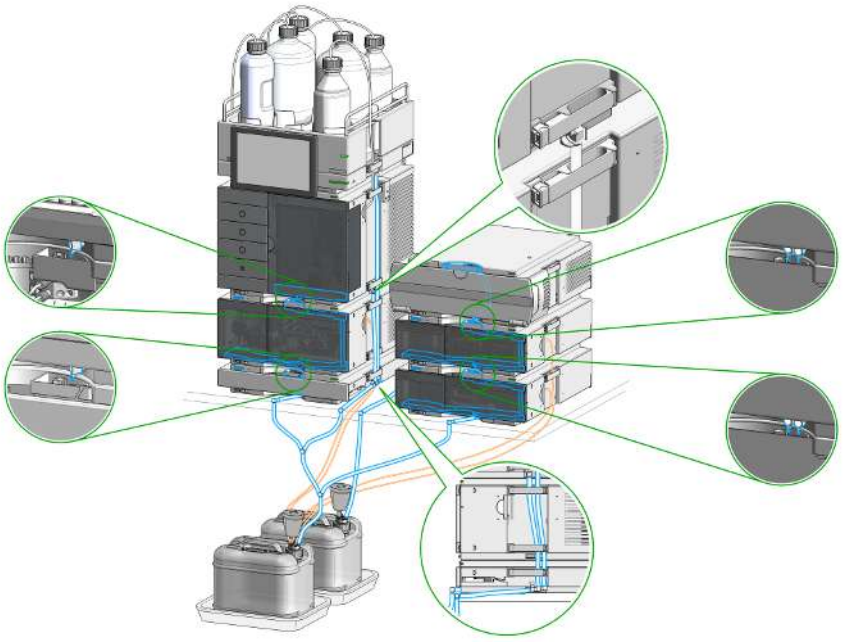


Figure 23: Infinity III Two Stack Leak Waste Concept (bench installation)

The waste tube connected to the leak plane outlet on each of the bottom instruments guides the solvent to a suitable waste container.

Drain Connectors Installation

Drain Connectors have been developed to improve leak drainage for low flow leaks of high viscosity solvents (for example, isopropanol) in Agilent InfinityLab LC Series Systems. Install these parts to modules where they are missing (usually preinstalled).

- Make sure that dripping adapters are correctly installed on each module in the LC stack, excluding lowest module.
- Remove the dripping adapter if it is appeared to be installed on the lowest module in the LC stack and connect waste tube instead.
- Consider 5004-0000 (Drain Connectors Kit) if drain adaptor is missing on some module(s).

For illustration, see Handling Leak and Waste on page 55.

Parts required

Qty.	p/n	Description
	5004-0000	Drain Connectors Kit

Content of Drain Connectors Kit (p/n 5004-0000)

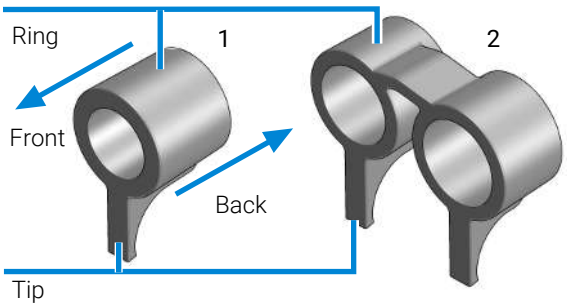


Figure 24: Overview of Drain Connectors: Single (left) and Double (right)

Qty.	p/n	Description
Parts can be ordered only as a complete kit.		
3	5043-1834	Single Drain Connector ID3.0-Long
1	5043-1836	Double Drain Connector-Long

Table 7: Compatibility of drain connectors and modules

Drain Connector Type	Compatible Module	Compatible Module Type
Double	G7116A/B	Column Compartment
Single	G7114A/B	Detector
	G7115A	
	G7117A/B/C	
	G7121A/B	
	G7162A/B	
	G7165A	
	G7129A/B/C	Sampler
	G7167A/B/C	
	G5668A	
	G7137A	
	G7157A	
	G4767A	
	G7122A	Degasser
	G7104A/C	Pump
	G7110B	
	G7111A/B	
	G7112B	
	G7120A	
	G7131A/C	
	G7132A	
	G5654A	
	G4782A	

Preparations

- Leak drains of LC modules are clean and free of salt or solvent residuals.

NOTE

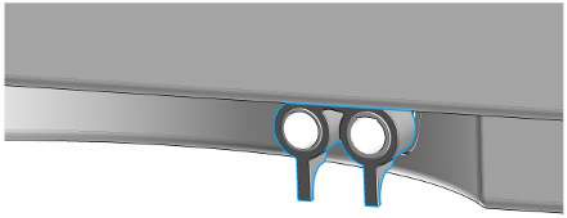
Do not install drain connectors on the bottom modules of the stack. Drain outlet of the bottom module has to be connected via waste tubing to a suitable waste container (see Leak and Waste Handling in the manual for a respective module).

NOTE In case of incorrect installation, drain connectors cannot fully perform the intended function.

NOTE It is not required to power off the HPLC stack to install Single and Double Drain Connectors. The installation of the connectors does not affect the analysis performed during the installation.

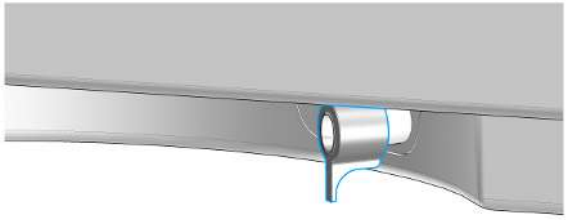
Install the Double Drain Connector on the leak drain of the 1260 Infinity III Multicolumn Thermostat (G7116A)/ 1290 Infinity III Multicolumn Thermostat (G7116B)

1 Align the rings with the leak drain outlets of the module, press slightly with the fingers, and slide the connector along the leak drain outlets until it is aligned with the front of the leak drain.



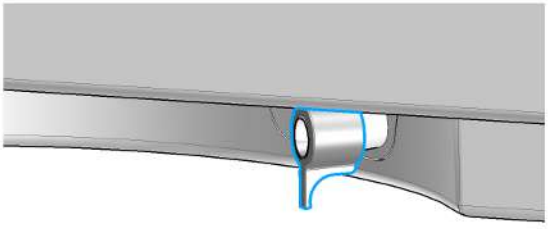
Install Single Drain Connectors on other modules in the LC stack

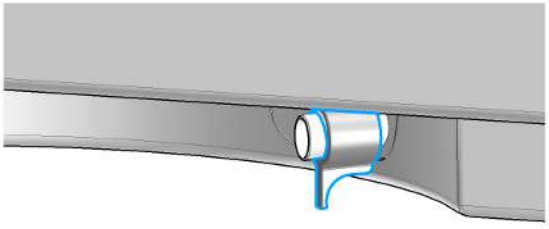
1 Align the ring with the leak drain outlet of the module, press slightly with the fingers, and slide the connector along the leak drain outlet until it is aligned with the front of the leak drain.



Make sure that the following requirements are covered:

- The tip of the drain connector points straight down.
- The leak drain outlets and the drain connectors are aligned properly.



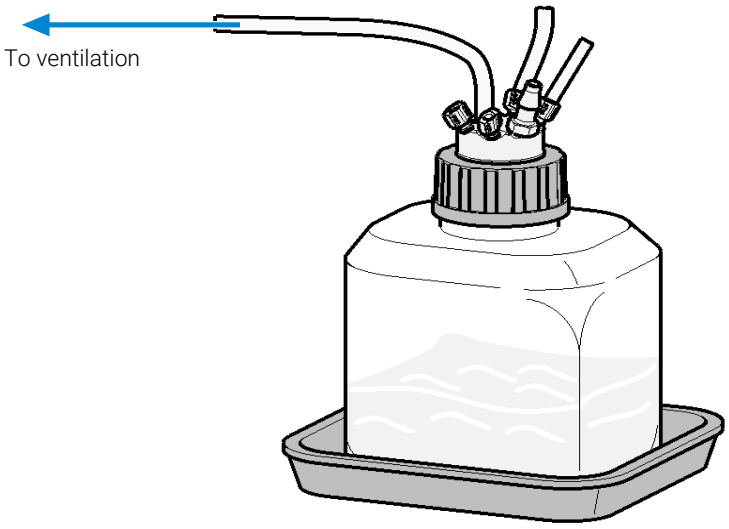


Waste Concept

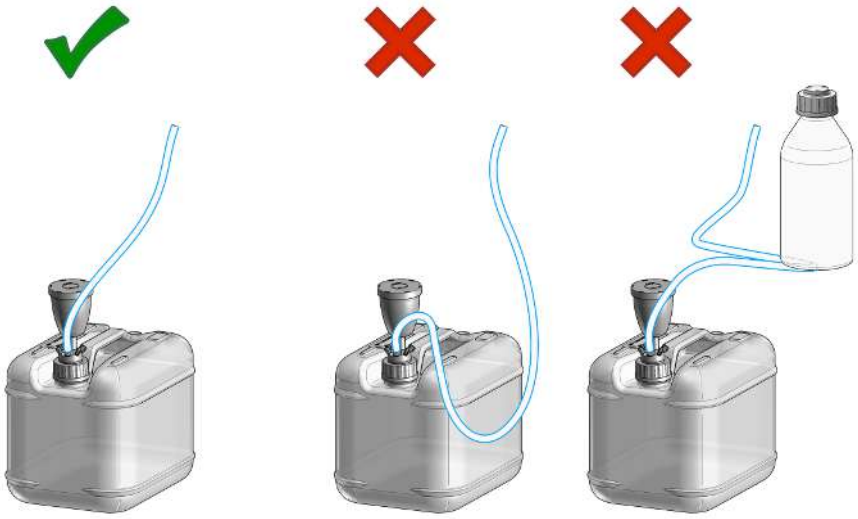
Agilent recommends using the 5043-1221 (6 L waste can with 1 Stay Safe cap GL45 with 4 ports) for optimal and safe waste disposal. If you decide to use your own waste solution, make sure that the tubes don't immerse in the liquid.

SFC Waste Concept

1 Agilent recommends using the 5043-1221 (6 L waste can with 1 Stay Safe cap GL45 with 4 ports) for optimal and safe waste disposal. If you decide to use your own waste solution, make sure that the tubes don't immerse in the liquid.



Waste Guidance



NOTE

The waste drainage must go straight into the waste containers. The waste flow must not be restricted at bends or joints.

Leak Sensor

CAUTION

Solvent incompatibility

The solvent DMF (dimethylformamide) leads to corrosion of the leak sensor. The material of the leak sensor, PVDF (polyvinylidene fluoride), is incompatible with DMF.

- Do not use DMF as mobile phase.
- Check the leak sensor regularly for corrosion.

Preparing the HPLC

To minimize delays and broadening caused by excessive tube lengths, we recommend the following stack layout (the SFC Control Module needs to be immediately adjacent to the LC stack, but can be placed on either side):

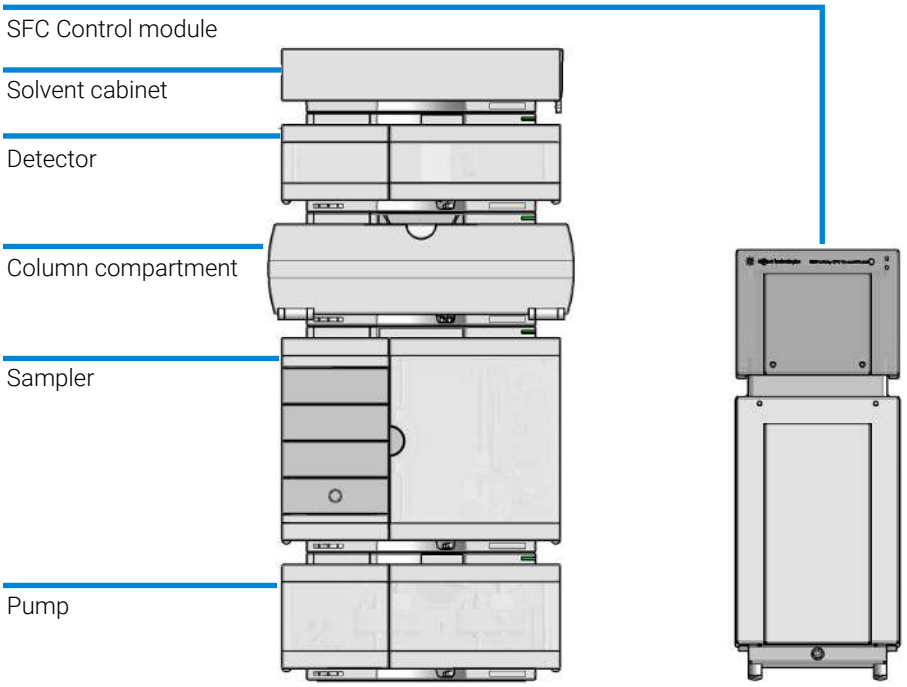


Figure 25: Examplary stack configuration for an SFC System

SFC Binary Pump

It is highly recommended that you familiarize yourself with standard maintenance functions and terminology used in the binary pump. This information is available in the Agilent Binary pump manual.

Less Polar Sample Solvents Help

Replacing the sample solvent with a much less polar solvent can allow injection of much larger volumes. However, the new solvent must be significantly less polar than the modifier used. Replacing methanol with ethanol or even isopropanol has minimal effect. Solvents such as chloroform or methylene chloride tend to cause significant focusing (NO broadening), but since they are chlorinated should probably be avoided.

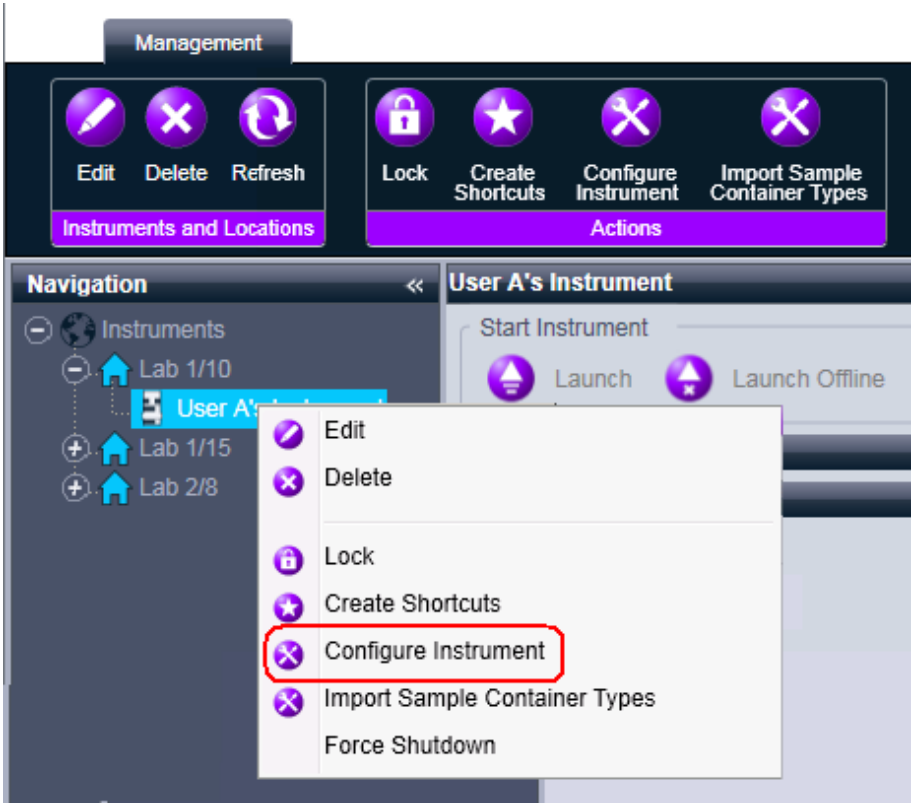
Configuring the SFC System in OpenLAB CDS

NOTE

The Agilent 1260 Infinity III SFC system requires the ChemStation **Modular LC System**, not with **Modular LC System (Classic)**. The **Modular LC System (Classic)** does not support the RC.Net drivers, which are necessary for the SFC modules.

1 Open the OpenLAB Control panel and create a new instrument. Refer to *Adding an LC System* in Chapter 3 of the *OpenLab ChemStation Configuration Guide (CDS_CS_Configure_C.01.10-U3.pdf, M8301-90060)* for detailed instructions.

2 Select **Configure Instrument** from the newly created instrument's context menu (right-click) or click **Configure Instrument** in the toolbar.

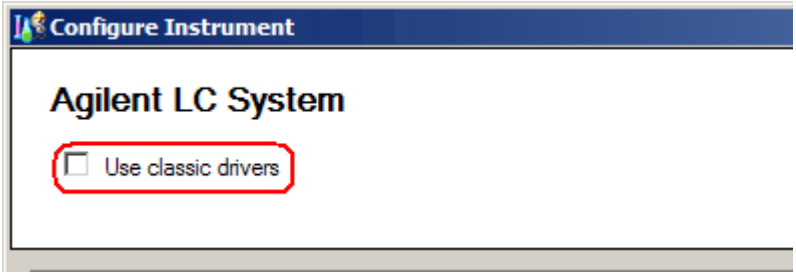


The dialog box is displayed.

NOTE Access to instrument configuration is disabled if the **Instrument Type** or the **Agilent Instrument Controller** are not specified.

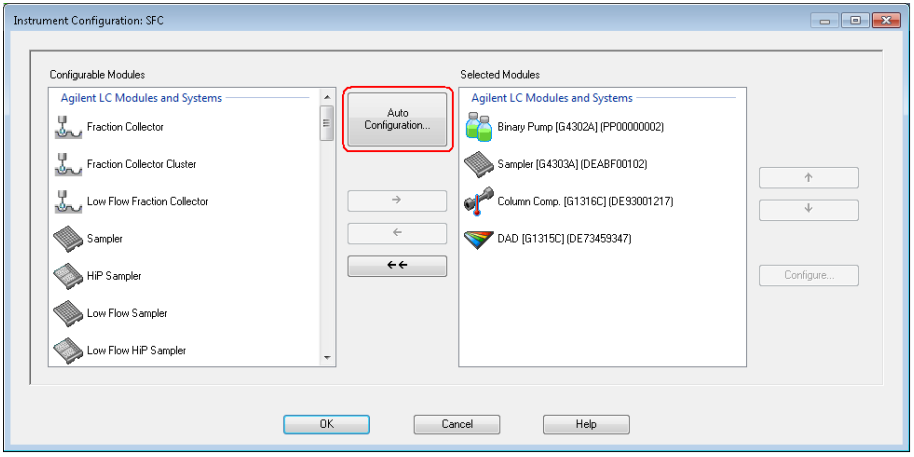
3 In the upper panel of the **Configure Instrument** dialog box, ensure that the **Classic drivers** check box is *cleared*.

NOTE If your instrument is not supported by the classic ChemStation Edition drivers, the **Use classic drivers** check box is unavailable.

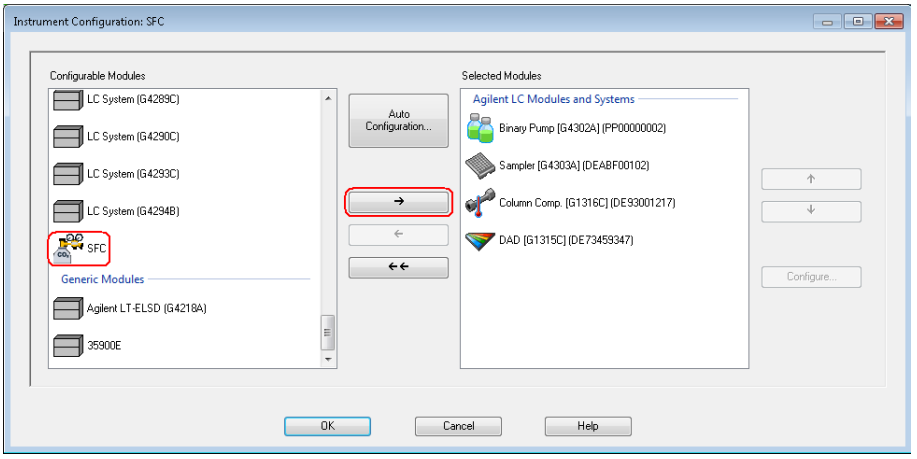


4 Click **Auto Configuration**.

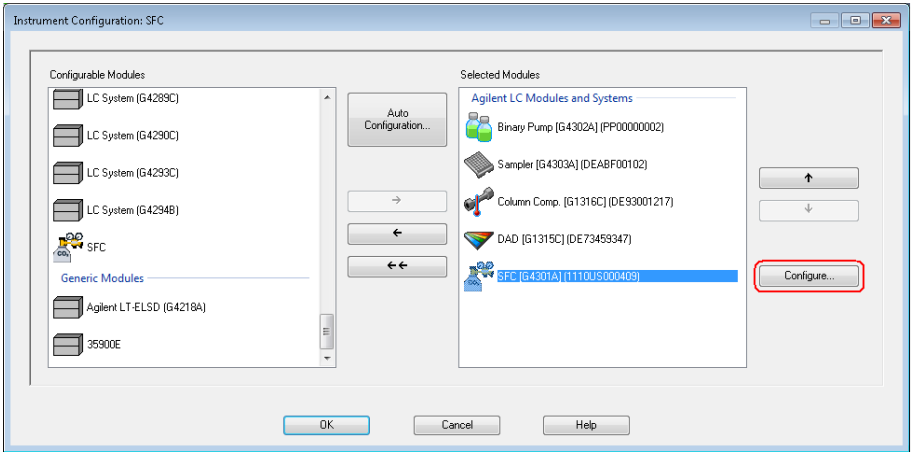
Autoconfiguration automatically recognizes the pump, autosampler, column compartment and detector, but does not recognize the SFC Control Module.



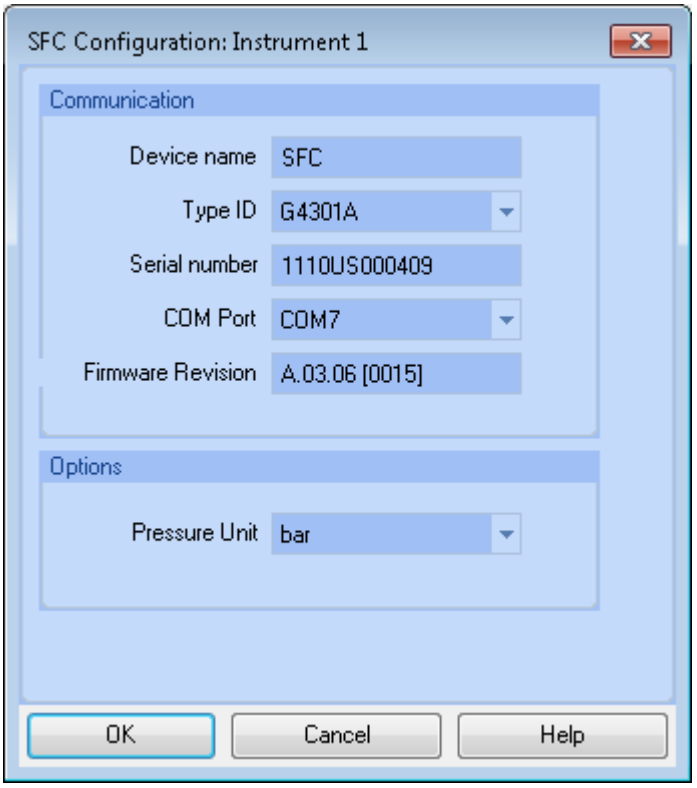
5 To set up the SFC module, scroll down the **Configurable Modules** panel until you see the SFC icon. Move it to the **Selected Modules** panel either by double-clicking it or selecting it and clicking the right-arrow button.



6 Select the SFC module in the **Selected Modules** panel and click **Configure.**



The SFC Configuration dialog box is displayed.



7 Click the COM Port down-arrow and select the COM port through which the SFC Control Module communicates with the ChemStation.

The Device name is the name that is shown in all ChemStation reports. You can edit the default name if you wish.

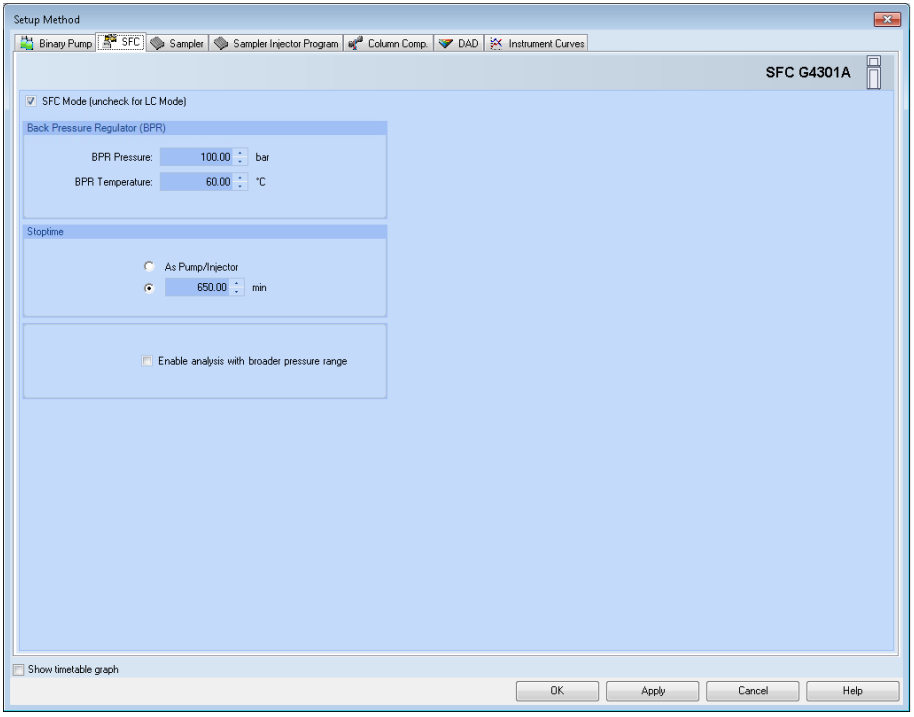
8 Click OK to accept the settings and close the SFC Configuration dialog box.

9 Click OK to close the Instrument Configuration dialog box.

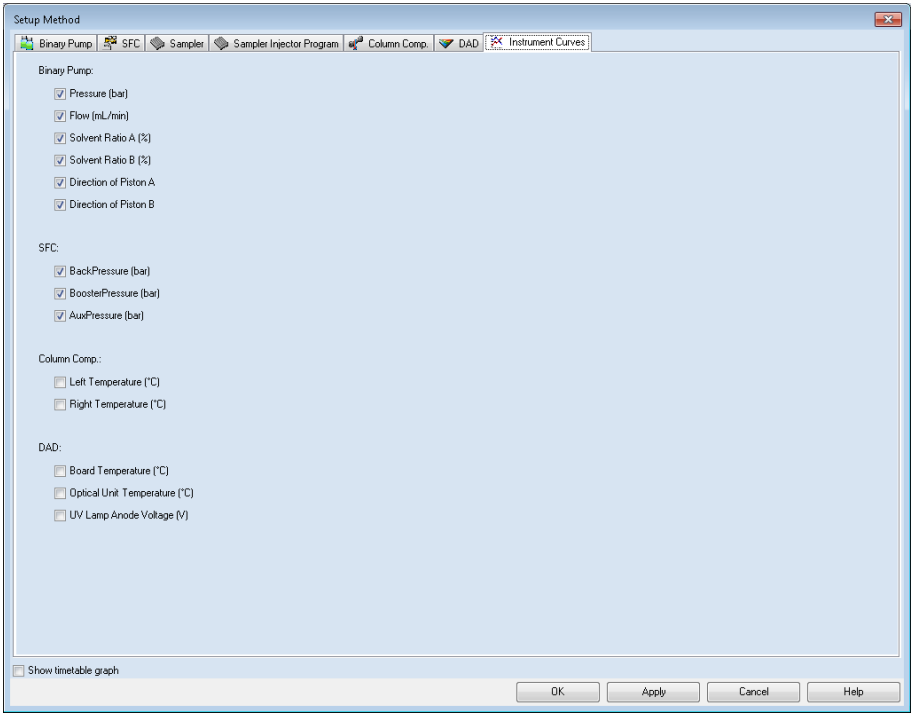
Your SFC system is now configured and ready to use.

Setting Up the Method

1 In the Agilent ChemStation, the method parameters for all RC.Net devices, including SFC settings, are combined into a single tabbed dialog box:

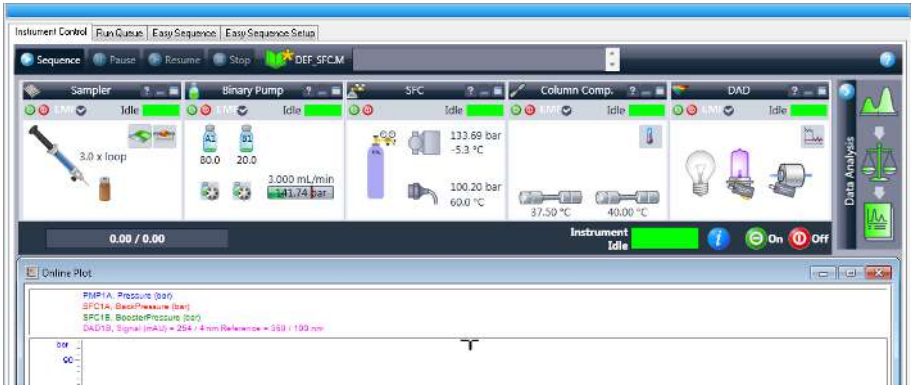


2 The **Instrument Curves** tab shows all instrument curves (monitor traces) available for storing with the acquired data file:

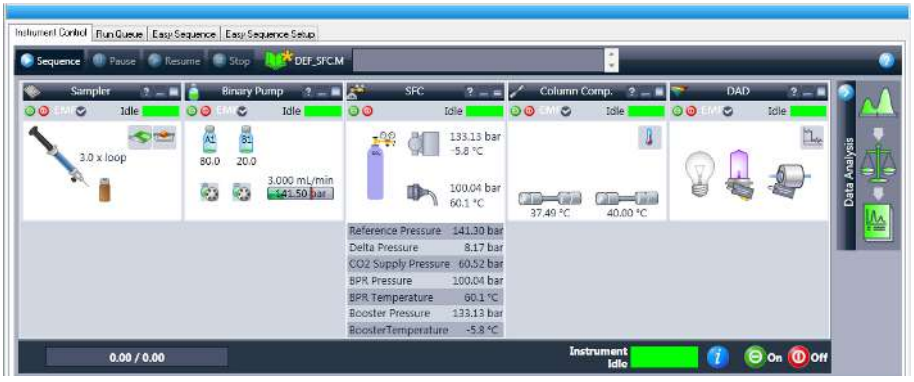

Setup Method
Binary Pump
SFC
Sampler
Sampler Injector Program
Column Comp.
DAD
Instrument Curves
Binary Pump:
Pressure (bar)
Flow (mL/min)
Solvent Ratio A (%)
Solvent Ratio B (%)
Direction of Piston A
Direction of Piston B
SFC:
BackPressure (bar)
BoosterPressure (bar)
AuxPressure (bar)
Column Comp.:
Left Temperature (°C)
Right Temperature (°C)
DAD:
Board Temperature (°C)
Optical Unit Temperature (°C)
UV Lamp Anode Voltage (V)
Show timetable graph
OK
Apply
Cancel
Help

Status

1 The SFC System status is displayed in the Dashboard. You can toggle the display of the Dashboard using **View** > **System diagram** .



2 Click in the module's title bar to display the instrument actuals.



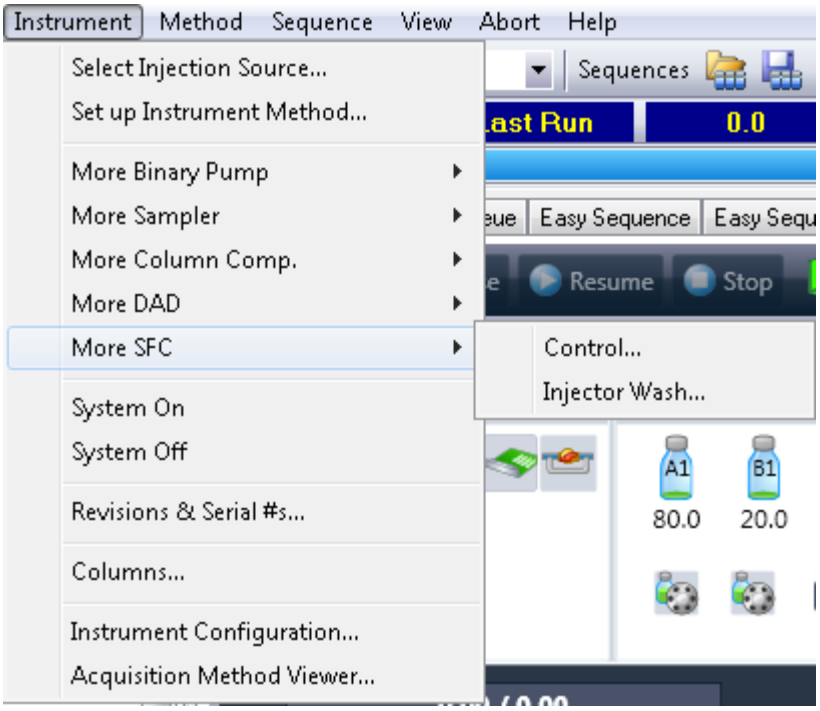
Click to hide the instrument actuals display.

3 Use and at the top left of the module display to switch the device on and off.

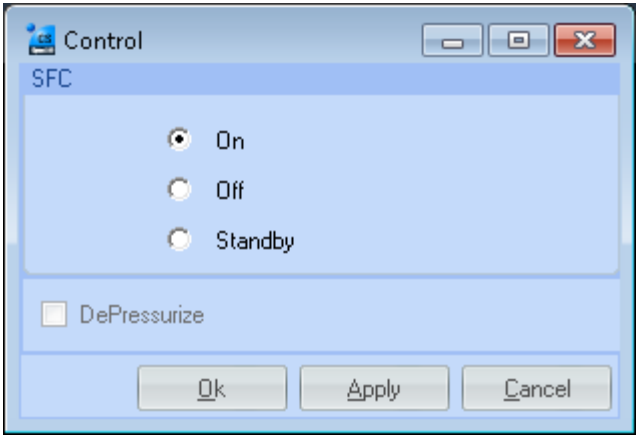
Control

The following steps show you how you can control your SFC instrument in the Agilent OpenLAB CDS ChemStation Edition.

1 In Instrument > More SFC , and in the context menu (right-click) of the SFC Dashboard panel are more menu items that allow you to control the device directly.



2 To change the status of the SFC device, or to depressurize the system, go to Instrument > More SFC > Control . This displays the SFC Control dialog box.



4 Using the Module

This chapter provides information on how to use the module.

Powering Up the Module 79

Power-Up Sequence and Operational Control States 80

Operational Control States 81
The OFF State 81
The STANDBY State 82
The ON State 82

Exploring the Module Graphical User Interface (GUI) 84

Loading the Default Method 86

Shutting Down the SFC System 88
Partial Shutdown 88

Powering Up the Module

The SFC Control Module is powered on by pressing the top of the rocker-type power switch located on the upper right rear of the module. Once the rocker is pressed, the module responds by entering its power-up sequence. The power button of the module must remain accessible at all times. Never arrange equipment so that the switch cannot be accessed.

WARNING

The power switch has to be reachable for emergency

— **The power switch of the module must remain accessible at all times. Never arrange equipment so that the switch cannot be accessed.**

Power-Up Sequence and Operational Control States

When power is applied to the SFC Control Module, a series of events is initiated. The order of these events is designed to safely initialize and test individual component functionality. The module power-up sequence executes the following steps:

1. Power is automatically applied to the processor and the two module fans.
2. The processor initializes:
   - **a** Any temporary configuration or calibration data that has not been stored in flash memory is lost.
   - **b** A checksum validation is made of data stored in flash memory.
   - **c** A self test is run to test power supply voltage levels and sensor readings for in-range values
   - **d** Stored calibration and configuration data are downloaded to RAM.
   - **e** The event logbook is updated
3. The booster pump drive is rotated to find its index pulse.
4. Index pulses are tested for module fans and the coolant pump.
5. The BPR is homed to its fully open (depressurized) position.
6. If installed, the wash pump is rotated to its index pulse.

At the completion of a successful power-up sequence, the processor places the module in the OFF operational state, described in Operational Control States on page 81. If an error is encountered and unresolved after multiple attempts, the module is placed into the ERROR state and a notation is stored in the event log.

Operational Control States

The SFC control module has three defined operational control states: OFF, STANDBY and ON. You control the three states by selecting the Control option from either the Open Lab CDS or from the SFC Test Utility Software.

Some components of the SFC control module are not governed by the three described states but are continuously on. These include:

- The processor.
  - Continuously records and transmits sensor data to the host control system.
  - Handles status and command requests from the host controller.
  - Monitors sensors for safety-related parameters.
  - Updates the event log.
- Pressure and temperature sensors are continuously powered and sensed.
- Coolant and electronics bay fans are continuously powered.
- The wash pump is activated by contact closure of the external contacted board placed in the SFC Autosampler, independent of the SFC control module control state.

The OFF State

The OFF state is characterized as follows:

- The CO_2 supply valve is closed (unpowered).
- The booster pump drive is unpowered.
- The BRP drive is unpowered.
- The BRP heater is unpowered.
- The chiller is unpowered.
- The secondary cooling circuit pump is unpowered.

The OFF state is always entered after a successful power-up sequence. It can also be entered by selecting the Off option in the control window, by a timeout from the STANDBY state or by pressing Off in the Agilent ChemStation graphical user interface twice in succession.

When the SFC control module is in the OFF state, the top “power” status light is constant and the bottom “ready” status light is off.

The STANDBY State

The STANDBY state is characterized as follows:

- The CO_2 supply valve is closed (unpowered).
- The booster pump drive is unpowered.
- The BRP drive is powered.
- The BRP heater is powered.
- The chiller is powered.
- The secondary cooling circuit pump is powered.

The STANDBY state can also be entered by selecting the Standby option in the control window, by pressing the power button associated with the SFC icon of the GUI, or by pressing Off in the ChemStation graphical user interface once while the system is running.

When the STANDBY state is entered from the OFF state, the BPR must be homed. Once this is accomplished, the BPR drive is active but in a hold state.

When the SFC Control Module is in the STANDBY state, the two status lights flash alternately. The STANDBY state remains active for up to three hours. If no user-initiated action is taken to change or renew the state within this time, a timeout occurs and the processor automatically enters the OFF state.

The ON State

The ON state is characterized as follows:

- The CO_2 supply valve is open (powered).

- The booster pump drive is powered and begins to pump CO_2 to the pressure target.
- The BRP drive is powered and the BRP begins the process of regulating to its setpoint.
- The BRP heater is powered.
- The chiller is powered.
- The secondary cooling circuit pump is powered.

The ON state can also be entered by selecting the On option in the control window, by pressing the power button associated with the SFC icon of the GUI, or by pressing On in the ChemStation graphical user interface.

When the SFC Control Module is in the ON state, the top status light is continuously lit. The bottom status light is lit when the system reaches a "ready" state, indicating that back-pressure and booster pump pressure are under control and stabilized within their control band.

Modules that start from the OFF state generally must first perform an initialization routine before moving to ON. In the case of the SFC Control Module, initialization causes the BPR first to home, then move to a default initialization position. The booster pump delays operation until the chiller passes below a threshold temperature value.

Exploring the Module Graphical User Interface (GUI)

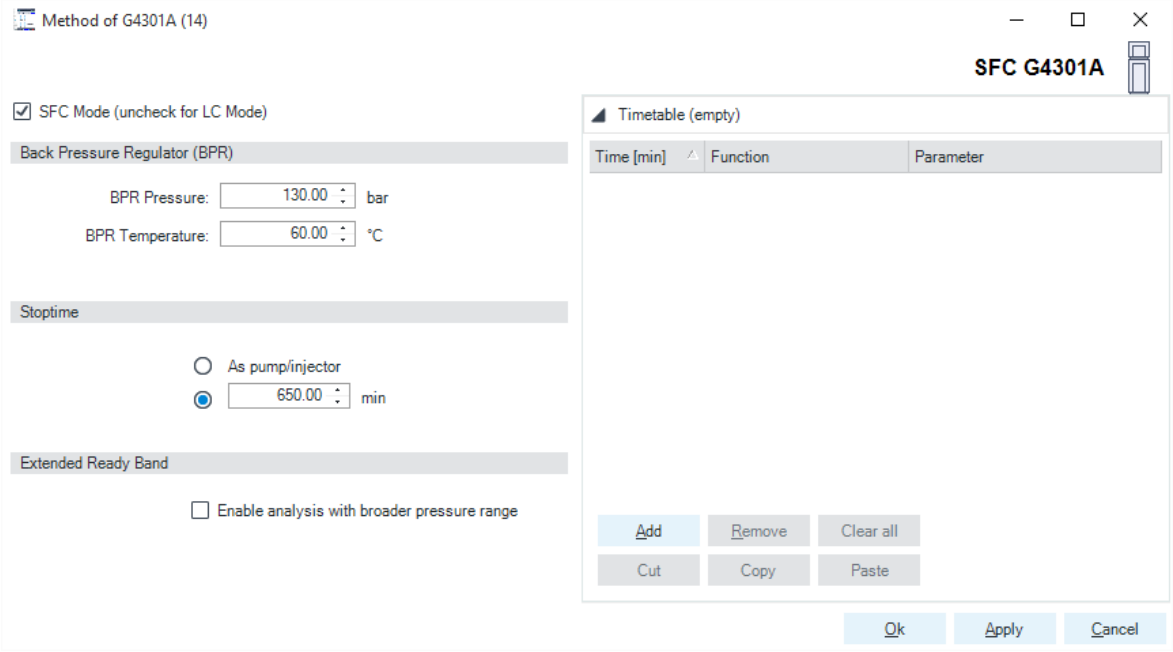


Figure 26: Editing the SFC control module Method Parameters

Editable method parameters for the SFC Control Module are the **BPR Pressure** (system back-pressure) and the **BPR Temperature** (temperature of the back-pressure regulator). Via timetable the **BPR Pressure** can be changed within the run and **BPR Pressure** gradients can be performed.

In addition, you can set a **Stoptime** for the analysis, as for all other modules.

For Hybrid Systems (SFC and HPLC in one system) there is the possibility to choose the mode how the SFC Control module should behave. This function should only be used in Hybrid systems, in pure SFC Systems the **SFC Mode** check box has always to be checked. When the **SFC Mode** check box is checked the SFC Control Module performs under SFC conditions; the **BPR Pressure** and **BPR Temperature** are applied and the Booster Pump provides compressed CO_2 (8 bar below system pressure).

When the **SFC Mode** check box is unchecked the SFC Control Module goes to Standby but reports Ready state to the System. In this mode HPLC runs can be made without any CO_2 flow. SFC Binpump flow should be set to zero ml/min to save solvent consumption.

Loading the Default Method

The OpenLAB CDS ChemStation installation includes a default method, `DEF_LC.M`, which you should use as the basis of your SFC method. `DEF_LC.M` cannot be overwritten; save your SFC method with a new name.

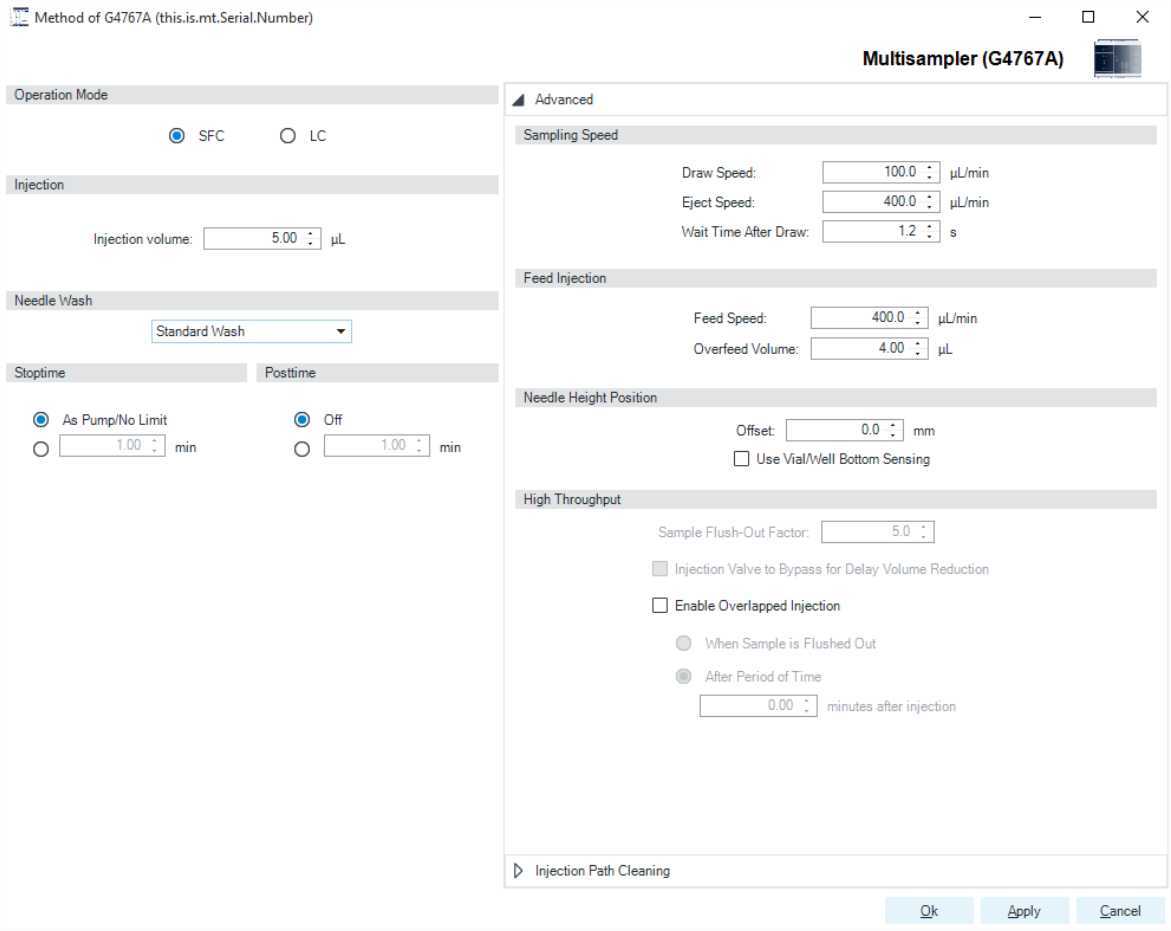


Figure 27: SFC Sampler method parameters

Default Pump setting is 80 % CO_2 : 20 % modifier. When you select CO_2 as the solvent, the **Compressibility** is set automatically. Typical **Flow** rate in SFC is 3 mL/min.

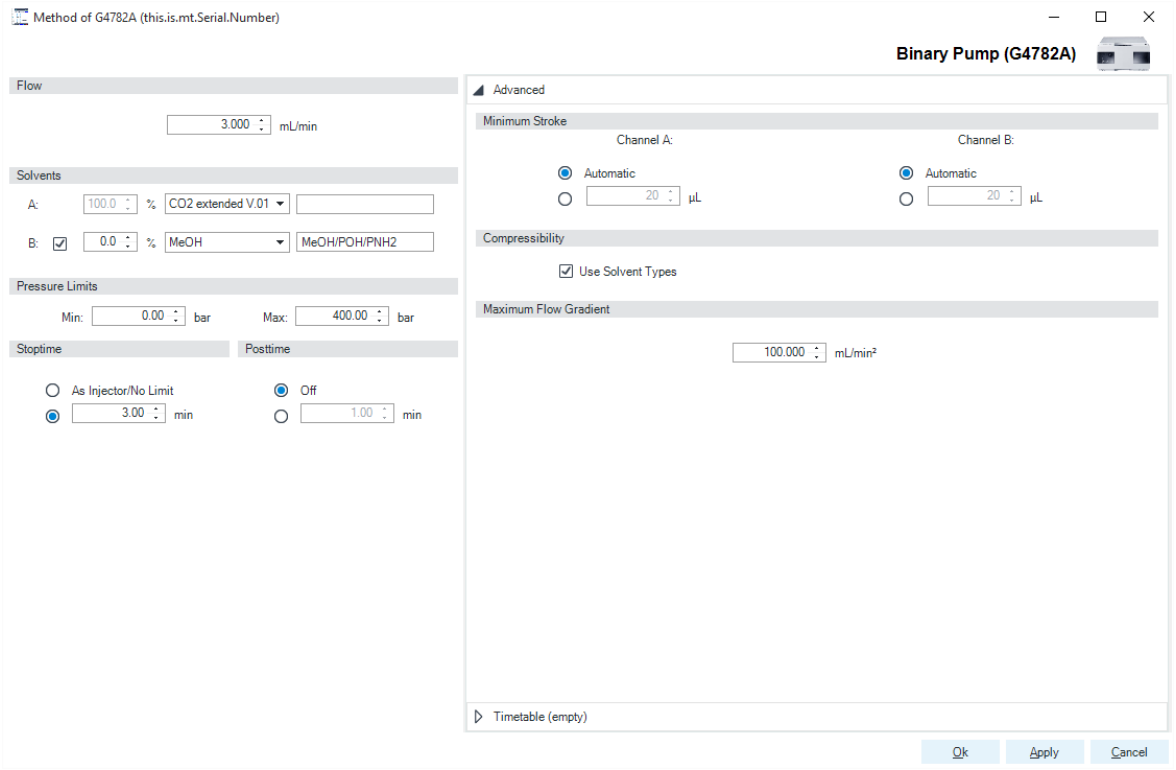


Figure 28: SFC Pump method parameters

Shutting Down the SFC System

The manner of shutting down the SFC depends on the requirement for rapid equilibration of the system on the next startup, and the duration of the shutdown. If the system will be shut down for some time, it is probably best to shut down all components including the DAD and column oven. These components tend to take longer to reach their stable operating conditions than the other components in the system. You should always shut down both the SFC control module and the binary pump if the system is to be idle for a long time.

Partial Shutdown

WARNING

Injuries from pressurized CO_2

- **Setting the pump to STANDBY does not depressurize the system. Do not attempt to loosen fittings or perform maintenance under these conditions. Serious skin and eye injuries can occur as the result of sudden release of CO_2 in the liquid or supercritical state. Always wear gloves and eye protection when maintaining the module.**

Leaving the system pressurized

If the system is to be shut down for less than two hours, a partial pressurized shutdown is recommended. Press the control buttons (for example, button 2) to switch the SFC control module and binary pump states to STANDBY. In this case, the system remains pressurized, and slowly bleeds pressure through the nozzle. A residual pressure remains in the system when the nozzle closes fully at lower pressure. The booster remains chilled, and much of the startup CO_2 is preserved. You should be aware that the system is pressurized and not to attempt maintenance under these conditions.

The detector and column oven are left in the ON state, to maintain their readiness. You may elect to exit the ChemStation in this state, and should answer NO to the “Shutdown Lamps…” query that appears during shutdown. Restarting the ChemStation brings the system to this same state. If more than two hours elapse in STANDBY mode, the SFC control module enters the OFF state, and pressure may be lost at a more rapid rate.

Depressurizing the System

When maintenance is required on the SFC system, such as replacing the column, the system should be depressurized. It is not necessary to shut down all modules, but only the pumps and any other devices undergoing maintenance. To depressurize the system, stop the SFC control module and the binary pump. In the **Control** menu of the SFC Control Module Booster (the standby state will be selected) check the **Depressurize** box and click **OK**.

NOTE

If the G4301A SFC Control Module unit itself is to be serviced, select **OFF** in the **Control** window. This shuts off the BPR heater and booster chiller and allows them to move toward ambient temperature.

This causes the BPR to home and fully open the CO_2 path to depressurize. The G4301A SFC Control Module unit contains approximately 25 mL of stored CO_2. This amount of CO_2 should be vented properly, which takes several minutes. You should allow the system to drain below 40 bar before cracking any fittings. At this point the, CO_2 is in the vapor state, and represents a small expanded volume. However, do not inhale vapor directly from a cracked fitting. The concentration of CO_2 emerging from a flow line, even at low pressure, can be dangerous or even lethal.

WARNING

Avoid inhaling high concentration of CO_2

- **Never inhale vapor issuing from an SFC flow line. Exposure to concentrations of CO_2 over 5 % in air can be lethal. Always keep tubes directed away your face. CO_2 is ubiquitous in the atmosphere, but at high levels should be treated with the same care as other toxic chemicals. Always wear gloves and eye protection for safety. Avoid inhaling venting gas near open fittings.**

Alternatively, if the column oven contains a column switching valve, one path may be jumpered without a column. The reduced restriction will allow the system to depressurize much faster. Further, the isolated column can be exchanged immediately since the contained volume of CO_2 is small.

WARNING

System contains always 25 ml liquid CO2 under pressure

- **The G4301A SFC Control Module unit contains approximately 25 mL of liquid CO_2. The CO_2 must be vented properly since the expanded volume will allow local concentrations exceeding the OSHA PEL. Always allow the system to depressurize to below 40 bar before cracking any fittings. Always keep fittings directed away from the face.**

5 Diagnostics and Troubleshooting

This chapter gives an overview of the maintenance, troubleshooting, and diagnostic features available.

Diagnostic Features 91
User Interfaces 91
Troubleshooting With HPLC Advisor 91

Agilent Lab Advisor Software 92

Diagnostic Features

This section gives an overview of the diagnostic features available.

User Interfaces

InfinityLab Assist

InfinityLab Assist provides you with assisted troubleshooting and maintenance at your instrument.

If the system in use supports the InfinityLab Assist, follow the instructions provided. Else, the preferred solution is to use Agilent Lab Advisor Software.

- Depending on the user interface, the available tests and the screens/reports may vary.
- The preferred tool for troubleshooting and diagnostics should be Agilent Lab Advisor Software, see Agilent Lab Advisor Software on page 92.
- The Agilent OpenLab ChemStation C.01.03 and above do not include any maintenance/test functions.
- Screenshots used within these procedures are based on the Agilent Lab Advisor Software.

Troubleshooting With HPLC Advisor

Baseline, Peak Shape, Pressure, Retention related issues, can be solved using the HPLC Advisor App. For more information, see Troubleshooting Reversed-Phase Chromatographic Techniques With HPLC Advisor.

If using an InfinityLab Assist, navigate to **Health** > **Troubleshooting** to help solve baseline, peak shape, pressure, and retention related issues.

Agilent Lab Advisor Software

The Agilent Lab Advisor Software (basic license, shipped with an Agilent LC pump) is a standalone product that can be used with or without a chromatographic data system. Agilent Lab Advisor helps to manage the lab for high-quality chromatographic results by providing a detailed system overview of all connected analytical instruments with instrument status, Early Maintenance Feedback counters (EMF), instrument configuration information, and diagnostic tests. With the push of a button, a detailed diagnostic report can be generated. Upon request, the user can send this report to Agilent for a significantly improved troubleshooting and repair process.

The Agilent Lab Advisor software is available in two versions:

- Lab Advisor Basic
- Lab Advisor Advanced

Lab Advisor Basic is included with every Agilent 1200 Infinity Series and Agilent InfinityLab LC Series instrument.

The Lab Advisor Advanced features can be unlocked by purchasing a license key, and include real-time monitoring of instrument actuals, all various instrument signals, and state machines. In addition, all diagnostic test results, calibration results, and acquired signal data can be uploaded to a shared network folder. The Review Client included in Lab Advisor Advanced makes it possible to load and examine the uploaded data no matter on which instrument it was generated. This makes Data Sharing an ideal tool for internal support groups and users who want to track the instrument history of their analytical systems.

The optional Agilent Maintenance Wizard Add-on provides an easy-to-use, step-by-step multimedia guide for performing preventive maintenance on Agilent 1200 Infinity LC Series instrument.

The tests and diagnostic features that are provided by the Agilent Lab Advisor software may differ from the descriptions in this manual. For details, refer to the Agilent Lab Advisor software help files.

6 Maintenance

In this chapter only the SFC specific procedures are described. For procedures similar to the Agilent module procedures, please refer to the single module manuals (G4782A, G4767A, G7116A, G7115/65A).

Safety Information Related to Maintenance 94

Cleaning the Module 97

Inspection and Preventative Maintenance Intervals 98
Daily Procedure 98
Every 3 Months 99
As Needed (Corrective) 99

General Maintenance Procedures 100
Booster Drawer 100
Replacing Fuses 110
Standard Decontamination 111
Plugged BPR Decontamination 112
Preparing for Storage or Shipping 113

Safety Information Related to Maintenance

WARNING Eye damage by detector light

Eye damage may result from directly viewing the UV-light produced by the lamp of the optical system used in this product.

— Always turn the lamp of the optical system off before removing it.

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.

WARNING Heavy weight

The module is heavy.

— Carry the module at least with 2 people.

— Avoid back strain or injury by following all precautions for lifting heavy objects.

— Ensure that the load is as close to your body as possible.

— Ensure that you can cope with the weight of your load.

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.

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 Sharp metal edges

Sharp-edged parts of the equipment may cause injuries.

- **To prevent personal injury, be careful when getting in contact with sharp metal areas.**

WARNING Hot heat exchangers

The column compartment has two heat exchanger assemblies that might be hot.

- **Allow them to cool down before starting repairs.**

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.**

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.

CAUTION

Sample degradation and contamination of the instrument

Metal parts in the flow path can interact with the bio-molecules in the sample leading to sample degradation and contamination.

- For bio applications, always use dedicated bio parts, which can be identified by the bio-inert symbol or other markers described in this manual.
- Do not mix bio, and non-bio modules or parts in a bio system.

Cleaning the Module

To keep the module case clean, use a soft cloth slightly dampened with water, or a solution of water and mild detergent. Avoid using organic solvents for cleaning purposes. They can cause damage to plastic parts.

WARNING **Liquid dripping into the electronic compartment of your module can cause shock hazard and damage the module**

- **Do not use an excessively damp cloth during cleaning.**
- **Drain all solvent lines before opening any connections in the flow path.**

NOTE A solution of 70 % isopropanol and 30 % water might be used if the surface of the module needs to be disinfected.

Inspection and Preventative Maintenance Intervals

Inspection and maintenance of the Analytical SFC System are critical elements of long term reliability and performance of the system. Maintenance falls into two categories, preventative and corrective. Preventative maintenance intervals can vary based on the system use. The intervals offered in this section are for systems with average use of approximately 30 hours per week. Infrequently used systems may extend these intervals, while heavily used systems may require more frequent preventative maintenance. Most service can be performed directly by the user or in-house maintenance technicians.

Daily Procedure

System switch on:

- Turn on SFC Control Module first.
- Wait until temperatures are reached and booster pump starts pumping.
- 70 bar booster pressure is reached.
- Switch on binary pump flow.

Daily Inspection and Maintenance

- Verify that power and signal cables are firmly connected and not under strain.
- Inspect all user- serviceable high pressure tubes and transfer lines for crimping or very tight bends. Replace as necessary.
- Wipe up any visible liquid spills or condensation on or near the instrument.
- Verify that all covers are securely fastened to the frame.
- Inspect all reservoirs to ensure an adequate solvent supply.
- Prime the wash pump and modifier pumps. Check that the purge valve reseals without leaking.
- Empty all waste containers.
- Check that the exhaust line is attached to a ventilation system, and that the ventilation system is drawing.

- If an inlet step- down regulator is used with a gas supply system, check that the inlet pressure is between 40 and 70 bar.
- Check the integrity of the SFC Flow path (that is, that column, flow cell etc have not been removed).
- With the system running, visually inspect unions and tees for leaks. Run diagnostic leak test for added sensitivity.
- Check CO_2 air monitor, if available, for suitable exposure level [< 5000 ppm CO_2].

Every 3 Months

- Run nozzle diagnostic test.
- Run system leak test.
- Evaluate system calibration.
- Check chiller efficiency curve.
- If CO_2 cylinders are used as supply, change the cylinder seal (approximately every 10 cylinders) at the next tank change.
- Remove visible dust accumulation in the area of the module.

As Needed (Corrective)

- Change high pressure transfer lines with metal ferrules or PEEK end fittings after 10 – 20 reseals or when leaking.
- Change Booster piston (rare).
- Exchange BPR head (rare).

General Maintenance Procedures

Booster Drawer

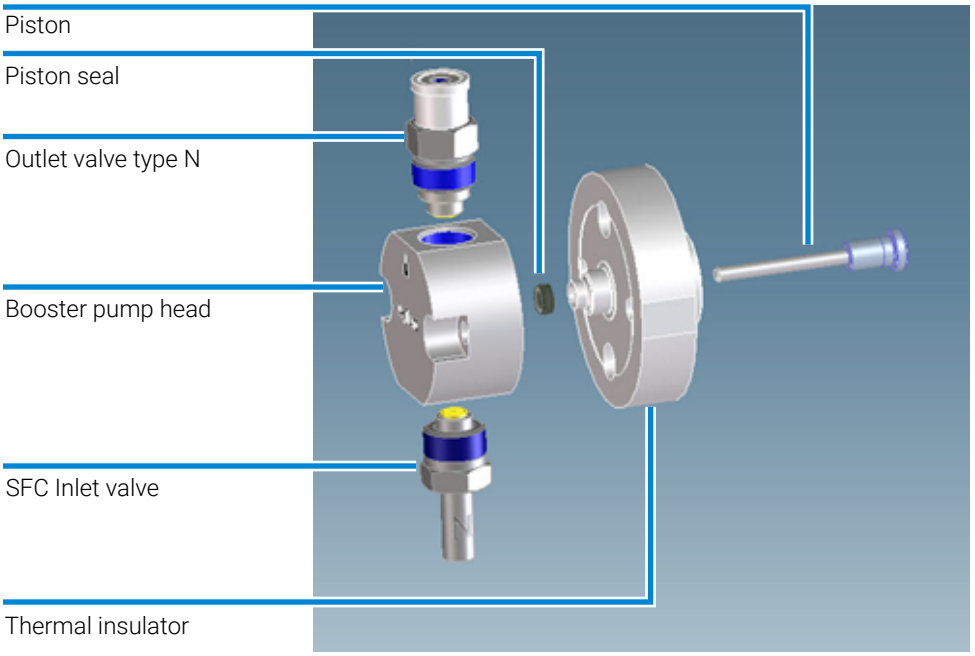


Figure 29: Exploded view of Booster Components

Removing the Vapor Shield

The vapor shield is not shown in the graphic Figure 29 on page 100.

Most maintenance procedures require removal of the vapor shield to access the underlying pump unit.

Tools required

Qty.	p/n	Description
1		A 3/16” hex-drive wrench (mounted on the back of the front cover)

1 Set the Control state to **OFF** on the SFC control module.

2 Wait for the chiller temperature to approach room temperature to prevent significant condensation on the chiller assembly and pump head.

3 Turn the power *OFF* on the SFC control module. Unplug the power cord.

4 Remove the front cover of the SFC control module unit by pulling gently at the upper left and right indents to the rear of the cover. The cover will release from its magnetic catch. Lift the cover upward to clear the two mounting pins at the base and set it aside.

NOTE

The 3/16” hex wrench used to remove the vapor shield and pump head is stored inside the removable front cover.

5 While holding the vapor shield with one hand, use the 3/16” hex wrench to loosen the four cap screws attaching each corner of the shield until they each disengage from the front panel. The screws are captured in the shield; do not try to remove them completely.

6 Remove the shield and store it in a safe location. Do not use a container for disassembled parts. This will scratch the plastic and impair visibility of the pump head during operation.

Replacing the Vapor Shield

1. Locate the vapor shield approximately over the mounting holes in the booster drawer front panel.
2. Engage each screw approximately one turn.
3. Inspect the border of the vapor shield to make sure it is in sealing contact with the foam seal of the drawer face. Adjust as necessary.
4. Tighten the mounting screws to ensure at least 50 % compression of the foam seal by the shield.
5. Replace the front cover by aligning the two base mounting pins and tilting forward to engage the magnetic catches.

Removing the Pump Head

Tools required	Qty.	p/n	Description
	1		A 1/4” open-end wrench
	1		A 9/16” open-end wrench
	1		A 3/16” hex-drive wrench
	1		seal insertion/removal tool
	1		Ultrasonic bath
	1		Isopropanol
	1		Deionized water

Parts required	Qty.	p/n	Description
	1	G4301-20200	Seal SFC Booster

NOTE

Each time the booster pump head is removed, the piston seal should be exchanged, since the seal surface may be easily scratched or distorted during removal. For this reason, the procedures are bundled. Cleaning the pump head is optional after visual inspection. Sealing surfaces of the pump head are critical to successful operation. Never use metal tools or paper toweling to wipe, probe or contact these surfaces.

1 Set the SFC control module mode to **OFF**.

2 Wait for the pump head to reach room temperature.

3 Power off the SFC control module.

4 Remove the vapor shield

5 Using the 9/16” and 1/4” wrenches, remove the inlet line from the inlet check valve holder and the outlet line from the outlet check valve holder.

CAUTION

Danger of piston breakage

— **Be careful not to break the piston when removing the pump head. Twisting the pump head can cause the piston to break.**

6 Using the 3/16” hex-drive, carefully remove the two knurled nuts at the front of the pump head.

7 Carefully separate the pump head from the pump. Move the pump head straight out from the pump and remove it from the piston. Be careful not to break or damage the piston. Also remove the seal from the piston if it did not stay in the pump head.

8 If the seal remains with the pump head, insert the flanged end of the seal insertion/removal tool into the seal cavity. Tilt it slightly so that flange is under the seal and pull out the seal.

Inspecting and Cleaning the Pump Head

1 Visually inspect the piston seal cavity in the pump head. Use magnification if necessary. Remove any foreign material using a cotton swab, or equivalent, and avoid scratching the sealing surfaces. Be sure no fibers from the cleaning swab remain in the components.

2 The pump head, may be further cleaned as follows:

   1. Remove inlet and outlet check valves.
   2. Clean with 50 % isopropanol in water in an ultrasonic bath for at least 30 min, followed by rinsing for at least 10 min in 100 % isopropanol. Be sure that all particles loosened by the above procedures have been removed from the components before re-assembly.
   3. Replace the check valves.

3 Wipe off any residual liquid from external (non-sealing) surfaces with a soft cloth such as a microfiber towel.

Replacing the Piston Seal

1. Sonicate or soak the new seal in isopropanol for 15 min to clean and provide lubrication for installing.
2. Place the replacement seal on the rod-shaped end of the seal insertion/removal tool so that the spring is visible when the seal is fully seated on the tool.
3. Insert the tool into the pump head so that the open side of the seal enters first, facing the high-pressure cavity of the pump head.
4. Be careful to line up the seal with the cavity while inserting. Withdraw the tool, leaving the seal in the pump head.

   When you look into the pump head cavity, only the polymer portion of the seal should be visible.

Replacing the Pump Head

1. Fill the pump head cavity about one third full with isopropyl alcohol.
2. Wet the piston tip with a few drops of isopropyl alcohol.
3. Holding an absorbent towel beneath the pump head assembly, line up the pump head and carefully slide it into place. Be sure that the inlet valve is on the bottom and the outlet valve is on the top. Do not force the pump head into place.
4. Finger tighten both knurled nuts into place. To tighten firmly, alternately turn nuts 1/4 turn while gently wiggling the pump head to center it.
5. Re-attach the inlet and outlet lines.

Cleaning or Replacing Booster Pump Piston

Tools required	Qty.	p/n	Description
	1		Tools for removing the vapor shield (see) and pump head (see)
	1		A 9/64" hex-drive wrench

Parts required	Qty.	p/n	Description
	1	G4301-20101	Piston SFC Booster

NOTE

In most cases, this procedure will be used only to replace a broken piston. Pumping CO_2 does not tend to leave deposits on the piston. Development of such deposits warrants examination of the CO_2 supply system and corrects the source of the deposited materials. Release of extraneous materials into the CO_2 supply system may cause contamination of the Agilent 1260 Infinity III SFC System.

1 Remove the Vapor Shield.

2 Remove the pump head.

3 Clean the pump head.

CAUTION

Take care not to break coolant tubes

— **Use care removing the chiller assembly from the mounting posts. The assembly is connected to a circulation pump behind the drawer panel. Do not pull the flow lines hard as this may loosen or crimp the tubes and cause the chiller to lose efficiency or cause leaks in the secondary cooler system.**

4 With a gentle rocking motion, loosen the chiller plate assembly and carefully slide it forward off the pump head mounting posts. Carefully twist the assembly out of the way.

5 Use the 9/64" hex wrench to unscrew the two cap screws attaching the spacer and very carefully remove the spacer by pulling straight back. This fully exposes the piston and retaining ring.

6 Remove the retaining ring by prying it out with a small blunt instrument or tweezers at the slot provided.

7 Grasp the metal base of the piston assembly so that you avoid exerting any side load on the sapphire rod, and remove the piston from the slot in the carrier by sliding it up.

8 Grasp the metal base of the replacement piston assembly, and insert it into the slot in the piston carrier until it bottoms in the slot.

9 Replace the retaining ring and spacer. Reattach the spacer mounting screws. If properly positioned, the spacer should be pressed into the foam wall seal.

10 Gently slide the chiller back onto the pump mounting posts and firmly press it onto the spacer. If properly positioned, the chiller heat exchanger should now be pressed into the foam wall seal.

11 Replace the piston seal

12 Replace the pump head

13 Replace the vapor shield

Replacing the CO_2 Inlet Filter

1. Unscrew the filter closure from the filter housing.
2. Use a seal insertion/removal tool or a non-metallic object (such as a wooden toothpick) to remove the large seal that remains in the housing
3. Unscrew the old filter and remove the small seal from the filter closure.
4. Place one of the small seals included in the replacement element kit over one of the new filters from the kit. Screw the new filter into the filter closure (finger tight).
5. Place one of the large seals from the replacement kit on the filter closure. Insert the filter closure into the housing and tighten.

Replacing Fuses

The power entry module of the SFC Control Module unit contains an external fuse drawer that is user serviceable.

To replace fuses: (before replacing fuses, first try to determine cause of fuse activation and repair)

Parts required

Qty.	p/n	Description
1		Fuses with 8A 250V

1. Power down the unit.
2. Disconnect the power cable from the power entry module.
3. Depress the release lever of the fuse drawer and pull the drawer straight back to remove.
4. Replace blown fuses with 8 A250 V Time Delay fuses of matching size. (A set of replacement fuses is included shipping kit).
5. Replace the fuse drawer by sliding it into the power entry module until it locks into place.

Standard Decontamination

Cleaning

External surfaces of the enclosure can be wiped with a damp soft cloth. More stubborn marks can be removed with a 50 % isopropanol:water mixture or mild cleanser such as Soft Scrub™. The latter may also be used to remove surface paint blemishes that may result from normal use.

The vapor shield of the booster drawer should be wiped only with a very soft cloth such as a microfiber polypropylene cloth, otherwise the surface may be scratched. Other user-accessible internal surfaces can be cleaned with a damp cloth.

BPR

The BPR head contacts CO_2, modifiers and sample material. To decontaminate, rinse with 50 % modifier flow at 5 mL/min for 15 min followed by pure CO_2 for 5 min.

Plugged BPR Decontamination

Decontamination of plugged BPR heads may require more aggressive solvents. In this case use the following procedure:

1. Depressurize the SFC Control Module completely.
2. Disconnect the BPR inlet and outlet tubes from the BPR drawer.
3. Attach the Inlet tube via a transfer line to waste.
4. Attach a solvent pump to the outlet tube of the BPR head
5. Prime the pump with a suitable solvent for the obstructing material.
6. Flush backwards with strong solvent at 1 mL/min for 20 minutes. Do not exceed a pressure of 400 bar.
7. If the pump cannot transfer fluid at less than 400 bar discontinue the operation and perform steps to exchange the BPR head.
8. If the backflush is successful, rinse the BPR head with Isopropanol for 10 minutes at 1 mL/min to clear the strong solvent.
9. Reconnect the BPR inlet and outlet lines.
10. Complete the standard decontamination procedure listed above

Preparing for Storage or Shipping

If the SFC control module needs to be stored in other than its operational location, it is best to store it in the original factory packaging. This packaging can also be used to reship the device to a secondary location. If the original packaging is unavailable, the unit should be stored upright and preferably covered in a plastic bag or wrap to prevent exposure to dust.

To prepare the unit for storage use the following procedure:

1. Follow the standard decontamination procedure.
2. Depressurize the SFC system completely.
3. Power off the unit.
4. Remove the front panel.
5. Disconnect the wash pump transfer line from the autosampler.
6. Drain the wash pump lines of fluid.
7. Coil the lines to fit in the SFC control module behind the removable front panel.
8. Disconnect the booster pump transfer line from the binary pump at the pump inlet check valve.
9. Disconnect the BPR return line from the detector.
10. Coil both lines to fit inside the SFC control module behind the removable front panel.
11. Replace the front panel
12. Disconnect the power cord and all signal cables from both ends of the connection.
13. Store cables and cords in a large plastic zip-lock bag.
14. Cover the unit with a large plastic bag.
15. If the original container is available, place the unit with its left side down in the packaging. Otherwise, store the unit upright in the storage area.

16 If the HPLC will also be stored or shipped, and will be reconfigured as an SFC system, the upgrade components can remain in the system.

17 If the two systems are to be permanently separated, uninstall the check valves, modifier purge valve, 3-groove rotor and high pressure flow cell by reversing the installation procedures in Hardware Installation. Store the components along with the original software disc and any upgrades with the module.

7 Parts and Materials for Maintenance

This chapter provides information on parts for maintenance.

SFC Control Module Parts 117

SFC DAD Parts 119

SFC Control Module Parts

G4301A SFC Control Module

p/n	Description
G4301-65000	Mainboard
G4301-65010	Motor Drive Board
G4301-60501	Heated Nozzle Assembly LD
G4301-00200	Plastic cover with screws
G4301-67009	BPR Drawer
G4301-67028	Booster Drawer
G4301-60300	CO2 Supply Line
G4301-60310	Pump Transfer Line
G4301-60320	Return Transfer Line
G4301-85000	Coolant 280 mL (12 oz)
G4301-65020	Circuit Board, Break Out
G4301-60330	Pressure sensor Witness line long
G4301-60340	Pressure sensor Witness line short
G4301-60350	Tee (Valco)
G4301-60550	Pressure Sensor Kit
G4301-60560	Cross and Cap. Kit for Aux Pres.Sensor
5973-2413	Inlet Screw + Inlet Filter
G4301-20007	CO2 Inlet valve
5188-9074	Tool 3/16"

G4301-67028 Booster Drawer parts

p/n	Description
G4301-60056	Capillary SFC Booster Pump out
G4302-60066	Inlet Valve SFC

p/n	Description
G4301-60101	Thermal Insulator Assy
G4301-20200	Seal SFC Booster
G4301-20201	Piston SFC Booster
G4301-20100	Pump Head SFC Booster
G1312-60167	Outlet Valve Type N/SFC

G4301-60012 Wash Pump

p/n	Description
5188-9073	Screw Wash Pump
G4301-60120	Wash Pump Check Valve PKG OF 2
G4301-60130	Wash Pump Piston Assy
G4301-60140	Wash Pump Seal Kit
5190-5754	Anti Syphon Valve
5188-9081	Screw for Valve
5067-5378	Connecting tube, DCGV to PIV

SFC DAD Parts

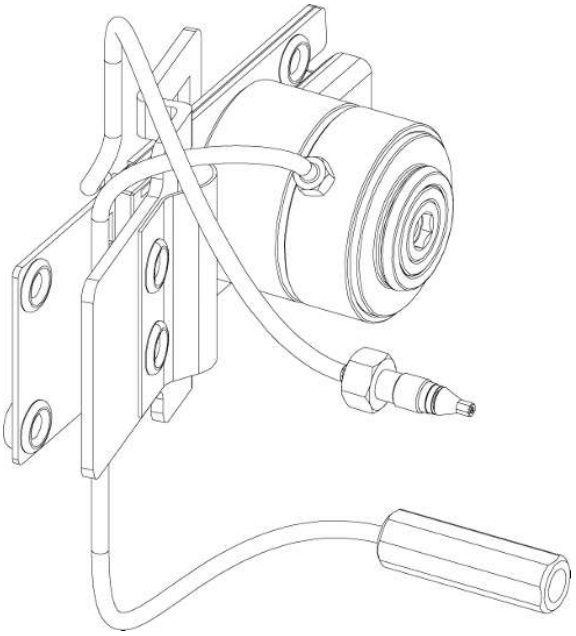


Figure 30: DAD Flow Cell SFC

Qty.	p/n	Description
1	G4301-60100	SFC Flow Cell
1	G4301-60200	SFC Low Dispersion Flow Cell
1	79883-68700	High pressure cell repair kit (includes 1 quartz window, 1 compression washer, 5 spring washers, 2 seal rings)

NOTE For all other Part Numbers please refer to the G7115/65A User manual.

8 Identifying Cables

This chapter provides information on cables used with the modules.

Cable Overview 121

Analog Cables 123

Remote Cables 125

BCD Cables 129

CAN/LAN Cables 131

RS-232 Cables 132

USB 133

Cable Overview

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

Analog cables

p/n	Description
35900-60750	Agilent 35900A A/D converter
01046-60105	Analog cable (BNC to general purpose, spade lugs)

Remote cables

p/n	Description
5188-8029	ERI to general purpose
5188-8044	Remote Cable ERI – ERI
5188-8045	Remote Cable APG – ERI
5188-8059	ERI-Extension-Cable 1.2 m
5061-3378	Remote Cable to 35900 A/D converter
01046-60201	Agilent module to general purpose
5188-8057	Fraction Collection ERI remote Y-cable

CAN cables

p/n	Description
5181-1516	CAN cable, Agilent module to module, 0.5 m
5181-1519	CAN cable, Agilent module to module, 1 m

LAN cables

p/n	Description
5023-0203	Cross-over network cable, shielded, 3 m (for point to point connection)
5023-0202	Twisted pair network cable, shielded, 7 m (for point to point connection)

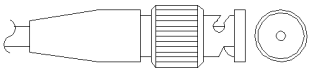
RS-232 cables

p/n	Description
RS232-61601	RS-232 cable, 2.5 m Instrument to PC, 9-to-9 pin (female). This cable has special pin-out, and is not compatible with connecting printers and plotters. It is also called "Null Modem Cable" with full handshaking where the wiring is made between pins 1-1, 2-3, 3-2, 4-6, 5-5, 6-4, 7-8, 8-7, 9-9.
5181-1561	RS-232 cable, 8 m

USB cables

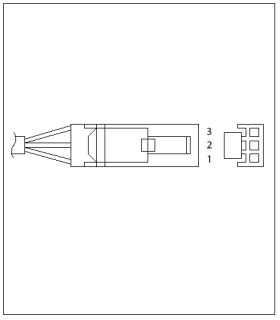
p/n	Description
5188-8050	USB A M-USB Mini B 3 m (PC-Module)
5188-8049	USB A F-USB Mini B M OTG (Module to Flash Drive)

Analog Cables

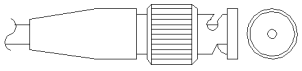


One end of these cables provides a BNC connector to be connected to Agilent modules. The other end depends on the instrument to which connection is being made.

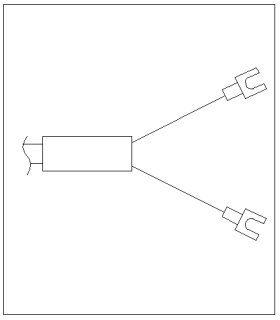
Agilent Module to 35900 A/D converters

p/n 35900-60750	35900	Pin Agilent module	Signal Name
	1		Not connected
	2	Shield	Analog -
	3	Center	Analog +

Agilent Module to BNC Connector

p/n 8120-1840	Pin BNC	Pin Agilent module	Signal Name
	Shield	Shield	Analog -
	Center	Center	Analog +

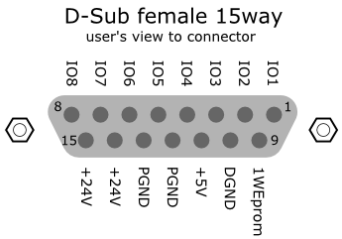
Agilent Module to General Purpose

p/n 01046-60105	Pin	Pin Agilent module	Signal Name
	1		Not connected
	2	Black	Analog -
	3	Red	Analog +

Remote Cables

ERI (Enhanced Remote Interface)

- 5188-8029 ERI to general purpose (D-Sub 15 pin male - open end)
- 5188-8044 ERI to ERI (D_Sub 15 pin male - male)
- 5188-8059 ERI-Extension-Cable 1.2 m (D-Sub15 pin male / female)

p/n 5188-8029	pin	Color code	Enhanced Remote	Classic Remote	Active (TTL)
	1	white	IO1	START REQUEST	Low
	2	brown	IO2	STOP	Low
	3	green	IO3	READY	High
	4	yellow	IO4	PEAK DETECT	Low
	5	grey	IO5	POWER ON	High
	6	pink	IO6	SHUT DOWN	Low
	7	blue	IO7	START	Low
	8	red	IO8	PREPARE	Low
	9	black	1wire DATA		
	10	violet	DGND		
	11	grey-pink	+5V ERI out		
	12	red-blue	PGND		
	13	white-green	PGND		
	14	brown-green	+24V ERI out		
	15	white-yellow	+24V ERI out		
	NC	yellow-brown			

NOTE Configuration is different with old firmware revisions.
The configuration for IO4 and IO5 is swapped for modules with firmware lower than D.07.10.

NOTE Peak Detection is used for LCMS systems connected with the Fraction Collection Remote Y-Cable (5188-8057).

- 5188-8045 ERI to APG (Connector D_Subminiature 15 pin (ERI), Connector D_Subminiature 9 pin (APG))

p/n 5188-8045	Pin (ERI)	Signal	Pin (APG)	Active (TTL)
	10	GND	1	
	1	Start Request	9	Low
	2	Stop	8	Low
	3	Ready	7	High
	5	Power on	6	High
	4	Future	5	
	6	Shut Down	4	Low
	7	Start	3	Low
	8	Prepare	2	Low
	Ground	Cable Shielding	NC	

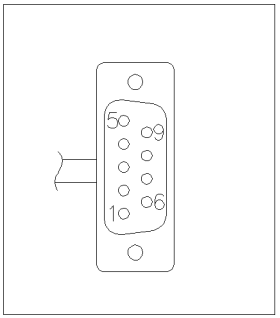
- 5188-8057 ERI to APG and RJ45 (Connector D_Subminiature 15 pin (ERI), Connector D_Subminiature 9 pin (APG), Connector plug Cat5e (RJ45))

Table 8: 5188-8057 ERI to APG and RJ45

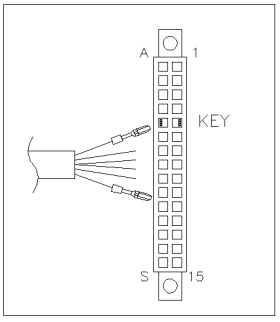
p/n 5188-8057	Pin (ERI)	Signal	Pin (APG)	Active (TTL)	Pin (RJ45)
					
	10	GND	1		5
	1	Start Request	9	High	
	2	Stop	8	High	
	3	Ready	7	High	
	4	Fraction Trigger	5	High	4
	5	Power on	6	High	
	6	Shut Down	4	High	
	7	Start	3	High	
	8	Prepare	2	High	
	Ground	Cable Shielding	NC		

One end of these cables provides an Agilent Technologies APG (Analytical Products Group) remote connector to be connected to Agilent modules. The other end depends on the instrument to be connected to.

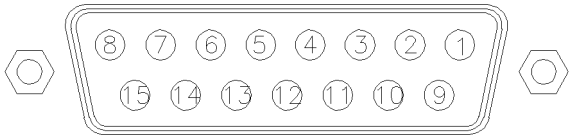
Agilent Module to Agilent 35900 A/D Converters

p/n 5061-3378	Pin 35900 A/D	Pin Agilent module	Signal Name	Active (TTL)
	1 - White	1 - White	Digital ground	
	2 - Brown	2 - Brown	Prepare run	Low
	3 - Gray	3 - Gray	Start	Low
	4 - Blue	4 - Blue	Shut down	Low
	5 - Pink	5 - Pink	Not connected	
	6 - Yellow	6 - Yellow	Power on	High
	7 - Red	7 - Red	Ready	High
	8 - Green	8 - Green	Stop	Low
	9 - Black	9 - Black	Start request	Low

Agilent Module to General Purpose

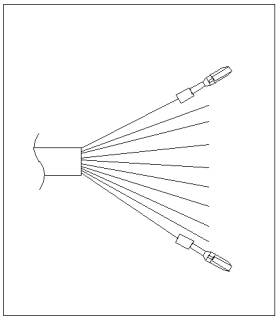
p/n 01046-60201	Wire Color	Pin Agilent module	Signal Name	Active (TTL)
	White	1	Digital ground	
	Brown	2	Prepare run	Low
	Gray	3	Start	Low
	Blue	4	Shut down	Low
	Pink	5	Not connected	
	Yellow	6	Power on	High
	Red	7	Ready	High
	Green	8	Stop	Low
	Black	9	Start request	Low

BCD Cables

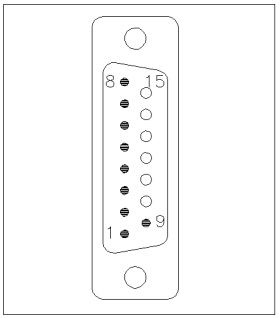


One end of these cables provides a 15-pin BCD connector to be connected to the Agilent modules. The other end depends on the instrument to be connected to

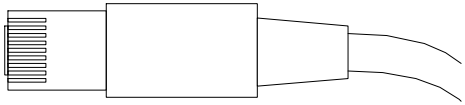
Agilent Module to General Purpose

p/n G1351-81600	Wire Color	Pin Agilent module	Signal Name	BCD Digit
	Green	1	BCD 5	20
	Violet	2	BCD 7	80
	Blue	3	BCD 6	40
	Yellow	4	BCD 4	10
	Black	5	BCD 0	1
	Orange	6	BCD 3	8
	Red	7	BCD 2	4
	Brown	8	BCD 1	2
	Gray	9	Digital ground	Gray
	Gray/pink	10	BCD 11	800
	Red/blue	11	BCD 10	400
	White/green	12	BCD 9	200
	Brown/green	13	BCD 8	100
	not connected	14		
	not connected	15	+ 5 V	Low

Agilent Module to 3396 Integrators

p/n 03396-60560	Pin 3396	Pin Agilent module	Signal Name	BCD Digit
	1	1	BCD 5	20
	2	2	BCD 7	80
	3	3	BCD 6	40
	4	4	BCD 4	10
	5	5	BCD0	1
	6	6	BCD 3	8
	7	7	BCD 2	4
	8	8	BCD 1	2
	9	9	Digital ground	
	NC	15	+ 5 V	Low

CAN/LAN Cables



Both ends of this cable provide a modular plug to be connected to Agilent modules CAN or LAN connectors.

Can Cables

p/n	Description
5181-1516	CAN cable, Agilent module to module, 0.5 m
5181-1519	CAN cable, Agilent module to module, 1 m

LAN Cables

p/n	Description
5023-0203	Cross-over network cable, shielded, 3 m (for point to point connection)
5023-0202	Twisted pair network cable, shielded, 7 m (for point to point connection)

RS-232 Cables

p/n	Description
RS232-61601	RS-232 cable, 2.5 m Instrument to PC, 9-to-9 pin (female). This cable has special pin-out, and is not compatible with connecting printers and plotters. It is also called "Null Modem Cable" with full handshaking where the wiring is made between pins 1-1, 2-3, 3-2, 4-6, 5-5, 6-4, 7-8, 8-7, 9-9.
5181-1561	RS-232 cable, 8 m

USB

To connect a USB Flash Drive use a USB OTG cable with Mini-B plug and A socket.

p/n	Description
5188-8050	USB A M-USB Mini B 3 m (PC-Module)
5188-8049	USB A F-USB Mini B M OTG (Module to Flash Drive)

9 Appendix

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

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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.

General

Do not use this product in any manner not specified by the manufacturer. The protective features of this product may be impaired if it is used in a manner not specified in the operation instructions.

Before Applying Power

WARNING

Wrong voltage range, frequency or cabling

Personal injury or damage to the instrument

- **Verify that the voltage range and frequency of your power distribution matches to the power specification of the individual instrument.**
- **Never use cables other than the ones supplied by Agilent Technologies to ensure proper functionality and compliance with safety or EMC regulations.**
- **Make all connections to the unit before applying power.**

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.**

NOTE

Note the instrument's external markings described under Safety Symbols on page 145.

Ground the Instrument

WARNING

Missing electrical ground

Electrical shock

- **If your product is provided with a grounding type power plug, the instrument chassis and cover must be connected to an electrical ground to minimize shock hazard.**
- **The ground pin must be firmly connected to an electrical ground (safety ground) terminal at the power outlet. Any interruption of the protective (grounding) conductor or disconnection of the protective earth terminal will cause a potential shock hazard that could result in personal injury.**

Do Not Operate in an Explosive Atmosphere

WARNING

Presence of flammable gases or fumes

Explosion hazard

- **Do not operate the instrument in the presence of flammable gases or fumes.**

Do Not Remove the Instrument Cover

WARNING

Instrument covers removed

Electrical shock

- **Do Not Remove the Instrument Cover**
- **Only Agilent authorized personnel are allowed to remove instrument covers. Always disconnect the power cables and any external circuits before removing the instrument cover.**

Do Not Modify the Instrument

Do not install substitute parts or perform any unauthorized modification to the product. Return the product to an Agilent Sales and Service Office for service and repair to ensure that safety features are maintained.

In Case of Damage

WARNING

Damage to the module

Personal injury (for example electrical shock, intoxication)

- **Instruments that appear damaged or defective should be made inoperative and secured against unintended operation until they can be repaired by qualified service personnel.**

Solvent Information

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.**
- **Do not use solvents with an auto-ignition temperature below 200 °C (392 °F). Do not use solvents with a boiling point below 56 °C (133 °F).**
- **Avoid high vapor concentrations. Keep the solvent temperature at least 40 °C (72 °F) below the boiling point of the solvent used. This includes the solvent temperature in the sample compartment. For the solvents methanol and ethanol keep the solvent temperature at least 25 °C (45 °F) below the boiling point.**
- **Do not operate the instrument in an explosive atmosphere.**
- **Do not use solvents of ignition Class IIC according IEC 60079-20-1 (for example, carbon disulfide).**
- **Reduce the volume of substances to the minimum required for the analysis.**
- **Never exceed the maximum permissible volume of solvents (8 L) in the solvent cabinet. Do not use bottles that exceed the maximum permissible volume as specified in the usage guideline for solvent cabinet.**
- **Ground the waste container.**
- **Regularly check the filling level of the waste container. The residual free volume in the waste container must be large enough to collect the waste liquid.**
- **To achieve maximal safety, regularly check the tubing for correct installation.**

NOTE For details, see the usage guideline for the solvent cabinet. A printed copy of the guideline has been shipped with the solvent cabinet, electronic copies are available in the Agilent Information Center or via the Internet.

Recommendations on the Use of Solvents

Observe the following recommendations on the use of solvents.

- Brown glass ware can avoid growth of algae.
- Follow the recommendations for avoiding the growth of algae, see the pump manuals.
- Follow the recommendations for avoiding the growth of algae, see Algae Growth in HPLC Systems on page 142
- Small particles can permanently block capillaries and valves. Therefore, always filter solvents through 0.22 µm filters.
- Avoid or minimize the use of solvents that may corrode parts in the flow path. Consider specifications for the pH range given for different materials such as flow cells, valve materials etc. and recommendations in subsequent sections.
- Avoid the use of the following steel-corrosive solvents:
  - solutions of alkali halides and their respective acids (for example, lithium iodide, potassium chloride, and so on),
  - high concentrations of inorganic acids like sulfuric acid and nitric acid, especially at higher temperatures (if your chromatography method allows, replace by phosphoric acid or phosphate buffer which are less corrosive against stainless steel),
  - halogenated solvents or mixtures which form radicals and/or acids, for example:

    $2CHCl_3 + O_2 \rightarrow 2COCl_2 + 2HCl$

    This reaction, in which stainless steel probably acts as a catalyst, occurs quickly with dried chloroform if the drying process removes the stabilizing alcohol,
  - chromatographic grade ethers, which can contain peroxides (for example, THF, dioxane, diisopropyl ether) should be filtered through dry aluminium oxide which adsorbs the peroxides,
  - solvents containing strong complexing agents (e.g. EDTA),
  - mixtures of carbon tetrachloride with 2-propanol or THF.
- Avoid the use of dimethyl formamide (DMF). Polyvinylidene fluoride (PVDF), which is used in leak sensors, is not resistant to DMF.

Recommended Wash Solvents

- water

- ethanol
- methanol
- water/acid (especially for basic compounds)
- water/base (especially for acidic compounds)
- water/acetonitrile

NOTE For different wash solvents as mentioned above, verify that the wash solvent is suitable for the silicone wash tubing.

Solvent Compatibility of Tubings for Peristaltic Pumps

The table shows the chemical resistance properties of Silicone and PharMed tubing to different needle wash solvents:

Table 9: Solvent Compatibility of Silicone and PharMed Tubing

	Silicone	PharMed
Acids		
• weak	• good	• very good
• medium	• unsatisfactory	• good
• strong	• not recommended	• not recommended
Alkaline solution		
• weak	• good	• very good
• medium	• unsatisfactory	• very good
• strong	• not recommended	• good
Hydrocarbons		
• aliphatic	• not recommended	• not recommended
• aromatizised	• not recommended	• not recommended
• halogenated	• not recommended	• not recommended

Flow cell

To protect optimal functionality of your flow-cell:

- Avoid the use of alkaline solutions (pH > 9.5) which can attack quartz and thus impair the optical properties of the flow cell.

Algae Growth in HPLC Systems

The presence of algae in HPLC systems can cause many problems that may be incorrectly diagnosed as instrument or application problems. Algae grow in aqueous media, preferably in a pH range from 4 to 8. Their growth is accelerated by buffers, for example phosphate or acetate. Since algae grow through photosynthesis, light will also stimulate their growth. Even in distilled water small-sized algae grow after some time.

Instrumental Problems Associated With Algae

Algae deposit and grow everywhere within the HPLC system, causing the following problems:

- Blocked solvent filters, or deposits on inlet or outlet valves, resulting in unstable flow, composition or gradient problems, or a complete failure of the pump.
- Plugging of small-pore, high-pressure solvent filters, usually placed before the injector, resulting in high system pressure.
- Blockage of PTFE frits, leading to increased system pressure.
- Plugging of column filters, giving high system pressure.
- Dirty flow cell windows of detectors, resulting in higher noise levels (since the detector is the last module in the flow path, this problem is less common).

How to Prevent and/or Reduce the Algae Problem

- Always use freshly prepared solvents, especially use demineralized water, which was filtered through 0.2 µm filters.
- Never leave mobile phase in the instrument for several days without flow.
- Always discard old mobile phase.
- Use the amber solvent bottle (9301-6526 (Solvent bottle, amber, 1000 mL)) supplied with the instrument for your aqueous mobile phase.
- If possible add a few mg/L sodium azide or a few percent organic solvent to the aqueous mobile phase.

Refrigerant

Table 10: Physical properties of refrigerant R600a (isobutane)

Molecular weight	58.12
Critical temperature	134.98 °C
Critical pressure	36.6 bar
Boiling point	-11.7 °C

CAUTION

General hazards and improper disposal

Improper disposal of the media and components used pollutes the environment.

- The disposal or scrapping of the Sample Thermostat must be carried out by a qualified disposal company.
- All media must be disposed of in accordance with national and local regulations.
- Please contact your local Agilent Service Center in regard to safe environmental disposal of the appliance or check www.agilent.com for more info.

CAUTION

Risk of fire or explosion

- Dispose of properly in accordance with federal or local regulations. Flammable Refrigerant Used.
- Do not dispose of in domestic household waste.
- To return unwanted products, contact your local Agilent office, or see http://www.agilent.com for more information.

Magnets

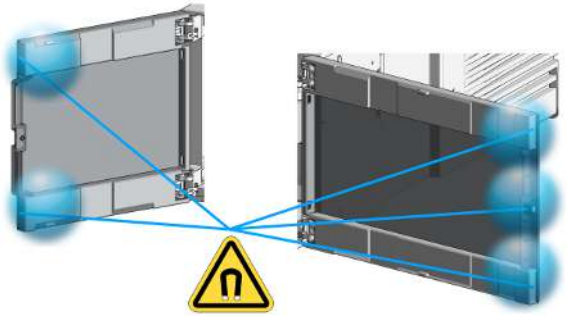

Figure 31: Magnets in doors of pumps, autosamplers, detectors, and fraction collectors

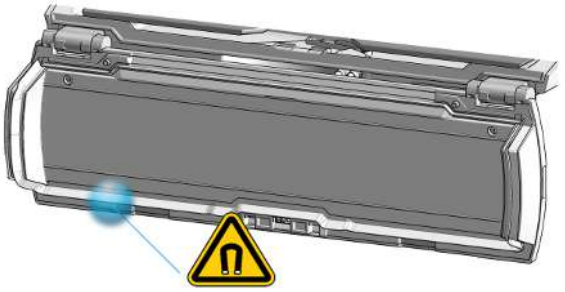

Figure 32: Magnet in the front door of the Multicolumn Thermostat

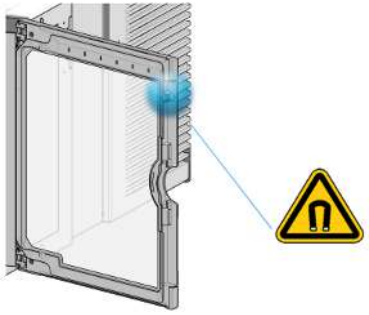

Figure 33: Magnet in door of the multisampler

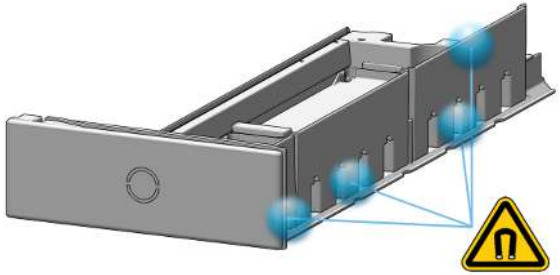

Figure 34: Magnets in drawers of the multisampler

Safety Symbols

Table 11: Symbols

	The apparatus is marked with this symbol when the user shall 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.
	Indicates flammable material used. Consult the Agilent Information Center / User Manual before attempting to install or service this equipment. Follow all safety precautions.
	Confirms that a manufactured product complies with all applicable European Community directives. The European Declaration of Conformity is available at: http://regulations.corporate.agilent.com/DoC/search.htm
	Manufacturing date.

REF	Product Number
SN	Serial Number
	Power symbol indicates On/Off. The apparatus is not completely disconnected from the mains supply when the on/off switch is in the Off position
	Pacemaker Magnets could affect the functioning of pacemakers and implanted heart defibrillators. A pacemaker could switch into test mode and cause illness. A heart defibrillator may stop working. If you wear these devices keep at least 55 mm distance to magnets. Warn others who wear these devices from getting too close to magnets.
	Magnetic field Magnets produce a far-reaching, strong magnetic field. They could damage TVs and laptops, computer hard drives, credit and ATM cards, data storage media, mechanical watches, hearing aids and speakers. Keep magnets at least 25 mm away from devices and objects that could be damaged by strong magnetic fields.
	Indicates a pinching or crushing hazard
	Indicates a piercing or cutting hazard.

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.

Material Information

This section provides detailed information about materials used in the HPLC system and general information about solvent/material compatibility.

Materials Used in the Bio-inert LC System

For the Bio-inert LC system, Agilent Technologies uses highest-quality materials in the flow path (also referred to as wetted parts), which are widely accepted by life science scientists, as they are known for optimum inertness to biological samples and ensure best compatibility with common samples and solvents over a wide pH range. Explicitly, the complete flow path is free of stainless steel and free of other alloys containing metals such as iron, nickel, cobalt, chromium, molybdenum, or copper, which can interfere with biological samples. The flow downstream of the sample introduction contains no metals whatsoever.

Table 12: Used bio-inert materials

Module	Materials
Agilent 1260 Infinity III Bio-inert Pump (G5654A)	Titanium, gold, platinum-iridium, ceramic, ruby, PTFE, PEEK
Agilent 1260 Infinity III Bio-inert Multisampler (G5668A)	Upstream of sample introduction: • Titanium, gold, PTFE, PEEK, ceramic Downstream of sample introduction: • PEEK, ceramic
Agilent 1260 Infinity III Bio-inert Manual Injector (G5628A)	PEEK, ceramic
Agilent 1260 Infinity III Bio-inert Analytical Fraction Collector (G5664B)	PEEK, ceramic, PTFE
Bio-inert Flow Cells:	
G5615-60022 (Standard flow cell bio-inert, 10 mm, 13 µL, 120 bar (12 MPa) for MWD/DAD, includes 0890-1763 – 0.18 x 1500 mm PEEK capillary and 5063-6591 – PEEK fittings) (*for Agilent 1260 Infinity III DAD G7115A, and MWD G7165A*)	PEEK, ceramic, sapphire, PTFE
G5615-60005 (Bio-inert flow cell, 8 µL, 20 bar) (*for Agilent 1260 Infinity III FLD G7121A/B*)	PEEK, fused silica, PTFE
Bio-inert Heat Exchangers, Valves and Capillaries:	
G7116-60041 (Quick Connect Heat Exchanger Bio-inert) (*for Agilent 1260 Infinity III Multicolumn Thermostat G7116A*)	PEEK (steel-cladded)
Bio-inert Valve heads (G4235A, G5631A, G5632A, G5639A)	PEEK, ceramic (Al_2O_3 based)
Bio-inert Connection capillaries	Upstream of sample introduction: • Titanium Downstream of sample introduction: • Agilent uses stainless-steel-cladded PEEK capillaries, which keep the flow path free of steel and provide pressure stability up to 600 bar.

NOTE To ensure optimum biocompatibility of your Bio-inert LC system, do not include non-inert standard modules or parts to the flow path. Do not use any parts that are not labeled as Agilent "Bio-inert". For solvent compatibility of these materials, see General Information About Solvent/Material Compatibility on page 149.

Materials in Flow Path

Following materials are used in the flow path of this module:

Table 13: Materials in flow path

Part	Materials
Degasser chamber	TFE/PDD Copolymer, PFA (internal tubings), PEEK (inlets), FEP (tubings), ETFE (fittings)
SSV	PEEK, FFKM
Active inlet valve	SST, sapphire, ruby, ceramic, PTFE
Outlet valve	SST, gold, ruby, ZrO_2-based ceramic, tantalum
Adapter	SST, gold
Pump head (body)	SST
Pistons	Sapphire
Piston seals/wash seals	PTFE, SST (reversed phase) or UHMW-PE, SST (normal phase)
Pressure sensor	SST
Purge valve	SST, gold, PTFE, ceramic
Damping unit	SST, gold
Capillaries/fittings	SST
Tubings	PTFE

General Information About Solvent/Material Compatibility

Materials in the flow path are carefully selected based on Agilent's experiences in developing highest-quality instruments for HPLC analysis over several decades. These materials exhibit excellent robustness under typical HPLC conditions. For any special condition, please consult the material information section or contact Agilent.

Disclaimer

Subsequent data was collected from external resources and is meant as a reference. Agilent cannot guarantee the correctness and completeness of such information. Data is based on compatibility libraries, which are not specific for estimating the long-term life time under specific but highly variable conditions of UHPLC systems, solvents, solvent mixtures, and samples. Information also cannot be generalized due to catalytic effects of impurities like metal ions, complexing agents, oxygen etc. Apart from pure chemical corrosion, other effects like electro corrosion, electrostatic charging (especially for nonconductive organic solvents), swelling of polymer parts etc. need to be considered. Most data available refers to room temperature (typically 20 – 25 °C, 68 – 77 °F). If corrosion is possible, it usually accelerates at higher temperatures. If in doubt, please consult technical literature on chemical compatibility of materials.

MP35N

MP35N is a nonmagnetic, nickel-cobalt-chromium-molybdenum alloy demonstrating excellent corrosion resistance (for example, against nitric and sulfuric acids, sodium hydroxide, and seawater) over a wide range of concentrations and temperatures. In addition, this alloy shows exceptional resistance to high-temperature oxidation. Due to excellent chemical resistance and toughness, the alloy is used in diverse applications: dental products, medical devices, nonmagnetic electrical components, chemical and food processing equipment, marine equipment. Treatment of MP35N alloy samples with 10 % NaCl in HCl (pH 2.0) does not reveal any detectable corrosion. MP35N also demonstrates excellent corrosion resistance in a humid environment. Although the influence of a broad variety of solvents and conditions has been tested, users should keep in mind that multiple factors can affect corrosion rates, such as temperature, concentration, pH, impurities, stress, surface finish, and dissimilar metal contacts.

Polyphenylene Sulfide (PPS)

Polyphenylene sulfide has outstanding stability even at elevated temperatures. It is resistant to dilute solutions of most inorganic acids, but it can be attacked by some organic compounds and oxidizing reagents. Nonoxidizing inorganic acids, such as sulfuric acid and phosphoric acid, have little effect on polyphenylene sulfide, but at high concentrations and temperatures, they can still cause material damage. Nonoxidizing organic chemicals generally have little effect on polyphenylene sulfide stability, but amines, aromatic compounds, and halogenated compounds may cause some swelling and softening over extended

periods of time at elevated temperatures. Strong oxidizing acids, such as nitric acid (> 0.1 %), hydrogen halides (> 0.1 %), peroxy acids (> 1 %), or chlorosulfuric acid degrade polyphenylene sulfide. It is not recommended to use polyphenylene sulfide with oxidizing material, such as sodium hypochlorite and hydrogen peroxide. However, under mild environmental conditions, at low concentrations and for short exposure times, polyphenylene sulfide can withstand these chemicals, for example, as ingredients of common disinfectant solutions.

PEEK

PEEK (Polyether-Ether Ketones) combines excellent properties regarding biocompatibility, chemical resistance, mechanical and thermal stability. PEEK is therefore the material of choice for UHPLC and biochemical instrumentation.

It is stable in the specified pH range (for the Bio-Inert LC system: pH 1 – 13 , see bio-inert module manuals for details), and inert to many common solvents.

There are still some known incompatibilities with chemicals such as chloroform, methylene chloride, THF, DMSO, strong acids (nitric acid > 10 %, sulfuric acid > 10 %, sulfonic acids, trichloroacetic acid), halogens or aqueous halogen solutions, phenol and derivatives (cresols, salicylic acid, and so on).

When used above room temperature, PEEK is sensitive to bases and various organic solvents, which can cause it to swell. Under such conditions, normal PEEK capillaries are sensitive to high pressure. Therefore, Agilent uses stainless steel clad PEEK capillaries in bio-inert systems. The use of stainless steel clad PEEK capillaries keeps the flow path free of steel and ensures pressure stability up to 600 bar. If in doubt, consult the available literature about the chemical compatibility of PEEK.

Polyimide

Agilent uses semi-crystalline polyimide for rotor seals in valves and needle seats in autosamplers. One supplier of polyimide is DuPont, which brands polyimide as Vespel, which is also used by Agilent.

Polyimide is stable in a pH range between 1 and 10 and in most organic solvents. It is incompatible with concentrated mineral acids (e.g. sulphuric acid), glacial acetic acid, DMSO and THF. It is also degraded by nucleophilic substances like ammonia (e.g. ammonium salts in basic conditions) or acetates.

Polyethylene (PE)

Agilent uses UHMW (ultra-high molecular weight)-PE/PTFE blends for yellow piston and wash seals, which are used in 1290 Infinity pumps, 1290 Infinity II/III pumps, the G7104C and for normal phase applications in 1260 Infinity pumps.

Polyethylene has a good stability for most common inorganic solvents including acids and bases in a pH range of 1 to 12.5 . It is compatible with many organic solvents used in chromatographic systems like methanol, acetonitrile and isopropanol. It has limited stability with aliphatic, aromatic and halogenated hydrocarbons, THF, phenol and derivatives, concentrated acids and bases. For normal phase applications, the maximum pressure should be limited to 200 bar.

Tantalum (Ta)

Tantalum is inert to most common HPLC solvents and almost all acids except fluoric acid and acids with free sulfur trioxide. It can be corroded by strong bases (e.g. hydroxide solutions > 10 %, diethylamine). It is not recommended for the use with fluoric acid and fluorides.

Stainless Steel (SST)

Stainless steel is inert against many common solvents. It is stable in the presence of acids and bases in a pH range of 1 to 12.5 . It can be corroded by acids below pH 2.3 . It can also corrode in following solvents:

- Solutions of alkali halides, their respective acids (for example, lithium iodide, potassium chloride) and aqueous solutions of halogens.
- High concentrations of inorganic acids like nitric acid, sulfuric acid, and organic solvents especially at higher temperatures (replace, if your chromatography method allows, by phosphoric acid or phosphate buffer, which are less corrosive against stainless steel).
- Halogenated solvents or mixtures, which form radicals and/or acids, for example:

  $2\ CHCl_3 + O_2 \rightarrow 2\ COCl_2 + 2\ HCl$

  This reaction, in which stainless steel probably acts as a catalyst, occurs quickly with dried chloroform if the drying process removes the stabilizing alcohol.
- Chromatographic grade ethers, which can contain peroxides (for example, THF, dioxane, diisopropyl ether). Such ethers should be filtered through dry aluminum oxide, which adsorbs the peroxides.

- Solutions of organic acids (acetic acid, formic acid, and so on) in organic solvents. For example, a 1 % solution of acetic acid in methanol will attack steel.
- Solutions containing strong complexing agents (for example, EDTA, ethylenediaminetetraacetic acid).
- Mixtures of carbon tetrachloride with isopropanol or THF.

Titanium (Ti)

Titanium is highly resistant to oxidizing acids (for example, nitric, perchloric and hypochlorous acid) over a wide range of concentrations and temperatures. This is due to a thin oxide layer on the surface, which is stabilized by oxidizing compounds. Non-oxidizing acids (for example, hydrochloric, sulfuric and phosphoric acid) can cause slight corrosion, which increases with acid concentration and temperature. For example, the corrosion rate with 3 % HCl (about pH 0.1) at room temperature is about 13 µm/year. At room temperature, titanium is resistant to concentrations of about 5 % sulfuric acid (about pH 0.3). Addition of nitric acid to hydrochloric or sulfuric acids significantly reduces corrosion rates. Titanium is sensitive to acidic metal chlorides like $FeCl_3$ or $CuCl_2$. Titanium is subject to corrosion in anhydrous methanol, which can be avoided by adding a small amount of water (about 3 %). Slight corrosion is possible with ammonia > 10 %.

Diamond-Like Carbon (DLC)

Diamond-Like Carbon is inert to almost all common acids, bases, and solvents. There are no documented incompatibilities for HPLC applications.

Fused Silica and Quartz (SiO_2)

Fused silica is used in Max Light Cartridges. Quartz is used for classical flow cell windows. It is inert against all common solvents and acids except hydrofluoric acid and acidic solvents containing fluorides. It is corroded by strong bases and should not be used above pH 12 at room temperature. The corrosion of flow cell windows can negatively affect measurement results. For a pH greater than 12, the use of flow cells with sapphire windows is recommended.

Gold

Gold is inert to all common HPLC solvents, acids, and bases within the specified pH range. It can be corroded by complexing cyanides and concentrated acids like aqua regia.

Zirconium Oxide (ZrO_2)

Zirconium Oxide is inert to almost all common acids, bases, and solvents. There are no documented incompatibilities for HPLC applications.

Platinum/Iridium

Platinum/Iridium is inert to almost all common acids, bases, and solvents. There are no documented incompatibilities for HPLC applications.

Fluorinated Polymers (PTFE, PFA, FEP, FFKM, PVDF)

Fluorinated polymers like PTFE (polytetrafluorethylene), PFA (perfluoroalkoxy), and FEP (fluorinated ethylene propylene) are inert to almost all common acids, bases, and solvents. FFKM is perfluorinated rubber, which is also resistant to most chemicals. As an elastomer, it may swell in some organic solvents like halogenated hydrocarbons.

TFE/PDD copolymer tubings, which are used in all Agilent degassers except G1322A/G7122A, are not compatible with fluorinated solvents like Freon, Fluorinert, or Vertrel. They have limited life time in the presence of hexafluoroisopropanol (HFIP). To ensure the longest possible life with HFIP, it is best to dedicate a particular chamber to this solvent, not to switch solvents, and not to let dry out the chamber. For optimizing the life of the pressure sensor, do not leave HFIP in the chamber when the unit is off.

The tubing of the leak sensor is made of PVDF (polyvinylidene fluoride), which is incompatible with the solvent DMF (dimethylformamide).

Sapphire, Ruby, and Al_2O_3-Based Ceramics

Sapphire, ruby, and ceramics based on aluminum oxide Al_2O_3 are inert to almost all common acids, bases, and solvents. There are no documented incompatibilities for HPLC applications.

Flow Cell

To protect optimal functionality of your flow cell:

- G5615-60022 (Standard flow cell bio-inert, 10 mm, 13 µL, 120 bar (12 MPa) for MWD/DAD, includes 0890-1763 – 0.18 x 1500 mm PEEK capillary and 5063-6591 – PEEK fittings) (PEEK, ceramic, sapphire, PTFE) for 1260 Infinity III Diode Array Detectors (G7115A):

  The recommended pH range of the cell is 1 – 13 (short term 14)

- G5615-60005 (Bio-inert flow cell, 8 µL, 20 bar) , (PEEK, fused silica, PTFE) for 1260 Infinity III Fluorescence Detector (G7121A/B)

  The recommended pH range of the cell is 1 – 12 (solvent dependent).

- If the flow cell is transported while temperatures are below 5 °C, it must be ensured that the cell is filled with alcohol to avoid damage by freezing water.
- Aqueous solvents in the flow cell can build up algae. Therefore, do not leave aqueous solvents sitting in the flow cell. Add a small percentage of organic solvents (for example, about 5 % of acetonitrile or methanol).

At-a-Glance Details About Agilent Capillaries

The following section provides useful information about Agilent capillaries and its characteristics.

Syntax for capillary description

Type - Material - Capillary dimensions - Fitting Left/Fitting right

Table 14: Example for a capillary description

Code provided with the part	Meaing of the code
Color code: 	Material of the product is MP35N, the inner diameter is 0.20 or 0.25 mm
Capillary	The part is a connection capillary
MP35N	Material of the part is MP35N
0.25 x 80 mm	The part has an inner diameter of 0.25 mm and a length of 80 mm
SI/SI	Left fitting: Swagelok + 1.6 mm Port id, Intermediate Right fitting: Swagelok + 1.6 mm Port id, Intermediate

To get an overview of the code in use, see

- Color: Table 15 on page 157
- Type: Table 16 on page 157
- Material: Table 17 on page 158
- Dimension: Table 18 on page 158
- Fittings: Table 19 on page 159

Color Coding Guide

Table 15: Color-coding key for Agilent capillary tubing

Internal diameter in mm		Color code
0.015		Orange
0.025		Yellow
0.05		Beige
0.075		Black
0.075	MP35N	Black with orange stripe
0.1		Purple
0.12		Red
0.12	MP35N	Red with orange stripe
0.17		Green
0.17	MP35N	Green with orange stripe
0.20 /0.25		Blue
0.20 /0.25	MP35N	Blue with orange stripe
0.3		Grey
0.50		Bone White

NOTE

As you move to smaller-volume, high efficiency columns, you'll want to use narrow id tubing, as opposed to the wider id tubing used for conventional HPLC instruments.

Abbreviation Guide for Type

Table 16: Type (gives some indication on the primary function, like a loop or a connection capillary)

Key	Description
Capillary	Connection capillaries
Loop	Loop capillaries
Seat	Autosampler needle seats

Key	Description
Tube	Tubing
Heat exchanger	Heat exchanger

Abbreviation Guide for Material

Table 17: Material (indicates which raw material is used for the capillary)

Key	Description
ST	Stainless steel
Ti	Titanium
PK	PEEK
FS/PK	PEEK-coated fused silica [2]
PK/ST	Stainless steel-coated PEEK [3]
PFFE	PTFE
FS	Fused silica
MP35N	Nickel-cobalt-chromium-molybdenium alloy

Abbreviation Guide for Capillary Dimensions

Table 18: Capillary dimensions (indicates inner diameter (id), length, and volume of the capillary)

Description
id (mm) x Length (mm)
Volume (µL)

2 Fused silica in contact with solvent

3 Stainless steel-coated PEEK

Abbreviation Guide for Fitting Left/Fitting Right

Table 19: Fitting left/fitting right (indicates which fitting is used on both ends of the capillary)

Key	Description
W	Swagelok + 0.8 mm Port id
S	Swagelok + 1.6 mm Port id
M	Metric M4 + 0.8 mm Port id
E	Metric M3 + 1.6 mm Port id
U	Swagelok union
L	Long
X	Extra long
H	Long head
G	Small head SW 4
N	Small head SW 5
F	Finger-tight
V	1200 bar
B	Bio
P	PEEK
I	Intermediate

Waste Electrical and Electronic Equipment (WEEE) Directive

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

NOTE

Do not dispose of in domestic household waste
To return unwanted products, contact your local Agilent office, or see https://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.

RFID Statement

Brasil

Este equipamento não tem direito à proteção contra interferência prejudicial e não pode causar interferência em sistemas devidamente autorizados. Para mais informações, consulte o site da Anatel: https://www.gov.br/anatel/pt-br.

Este produto não é apropriado para uso em ambientes domésticos, pois poderá causar interferências eletromagnéticas que obrigam o usuário a tomar medidas necessárias para minimizar estas interferências.

Canada

Statement according to RSS GEN Issue 5:

This device contains licence-exempt transmitter(s)/receiver(s) that comply with Innovation, Science and Economic Development Canada's licence-exempt RSS(s). Operation is subject to the following two conditions:

1. This device may not cause interference
2. This device must accept any interference, including interference that may cause undesired operation of the device.

Cet appareil contient des émetteurs / récepteurs exemptés de licence conformes aux RSS (RSS) d'Innovation, Sciences et Développement économique Canada. Le fonctionnement est soumis aux deux conditions suivantes:

1. Cet appareil ne doit pas causer d'interférences
2. Cet appareil doit accepter toutes les interférences, y compris celles susceptibles de provoquer un fonctionnement indésirable de l'appareil.

Mexico

La operación de este equipo está sujeta a las siguientes dos condiciones:

1. es posible que este equipo o dispositivo no cause interferencia perjudicial y
2. este equipo o dispositivo debe aceptar cualquier interferencia, incluyendo la que pueda causar su operación no deseada.

USA

1. User Information according to FCC 15.21:Changes or modifications not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment.
2. Part 15 Statement according to FCC 15.19:

   This device complies with Part 15 of the FCC Rules. Operation is subject to the following two conditions:

   - This device may not cause interference.
   - This device must accept any interference, including interference that may cause undesired operation.

CAUTION

Do not change or modify the equipment.

Changes or modifications not expressly approved by Agilent could void your authority to operate the equipment.

NOTE

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules.

These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Table 20: Operating frequencies and maximum power levels

Technology	Operating Frequencies/ Bands	Maximum Transmit Power Level
RFID	865 MHz - 868 MHz	33 dBm

Sound Emission

Sound Pressure

Sound pressure Lp < 70 db(A) according to DIN EN ISO 7779

Schalldruckpegel

Schalldruckpegel Lp < 70 db(A) nach DIN EN ISO 7779

UV-Radiation

NOTE

This information is only valid for UV-lamps without cover (e.g. 2140-0590 and 2140-0813).

Emissions of ultraviolet radiation (200-315 nm) from this product is limited such that radiant exposure incident upon the unprotected skin or eye of operator or service personnel is limited to the following TLVs (Threshold Limit Values) according to the American Conference of Governmental Industrial Hygienists:

Table 21: UV-Radiation Limits

Exposure/day	Effective Irradiance
8 hours	0.1 µW/cm2
10 minutes	5.0 µW/cm2

Typically the radiation values are much smaller than these limits:

Table 22: UV-Radiation Typical Values

Position	Effective Irradiance
Lamp installed, 50 cm distance	Average 0.016 µW/cm2
Lamp installed, 50 cm distance	Maximum 0.14 µW/cm2

Declaration of Conformity for HOX2 Filter

Declaration of Conformity

We herewith inform you that the

Holmium Oxide Glass Filter

used in Agilents absorbance detectors listed in the table below meets the requirements of National Institute of Standards and Technology (NIST) to be applied as certified wavelength standard.

According to the publication of NIST in J. Res. Natl. Inst. Stand. Technol. 112, 303-306 (2007) the holmium oxide glass filters are inherently stable with respect to the wavelength scale and need no recertification. The expanded uncertainty of the certified wavelength values is 0.2 nm.

Agilent Technologies guarantees, as required by NIST, that the material of the filters is holmium oxide glass representing the inherently existent holmium oxide absorption bands.

Test wavelengths:

Where "x" can be any alphanumeric character

Product Number	Series	Measured Wavelength *	Wavelength Accuracy	Optical Bandwidth
G1315x, G1365x	1100, 1200, 1260	361.0 nm 418.9 nm 453.7 nm 536.7 nm	+/- 1 nm	2 nm
G7115x, G7165x	1260			
G1600x, G7100x	CE			
G1314x	1100, 1200, 1260, 1290	360.8nm 418.5nm 536.4nm	+/- 1 nm	6 nm
G7114x	1260, 1290			
G4286x,..., 94x	1120, 1220			

*) The variation in Measured Wavelength depends on the different Optical Bandwidth.

28-Oct-2014
(Date)

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https://www.agilent.com

In This Book

This manual contains technical reference information about the Agilent 1260 Infinity III SFC System.

The manual describes the following:

- Introduction,
- site requirements and specifications,
- using the module,
- optimizing performance,
- troubleshooting and diagnostics,
- error information,
- maintenance,
- parts and materials,
- hardware information,
- LAN configuration,
- safety and related information.

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