

Agilent InfinityLab LC Series 1260 Infinity II SFC System

User Manual





Agilent Technologies

Notices

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

In This Guide...

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

1 Introduction to Supercritical Fluid Chromatography (SFC)

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

2 Site Requirements and Specifications

This chapter provides information on environmental requirements, physical and performance specifications only for the Agilent 1260 Infinity II SFC System.

3 Installing the Agilent 1260 Infinity II SFC System

This chapter provides an overview of the installation and setup of the hardware and software

4 Configuring the System

How to configure the Agilent 1260 Infinity II SFC System and Agilent 1260 Infinity II SFC Control Module in OpenLAB CDS.

5 Using the Agilent 1260 Infinity II SFC Control Module

This chapter provides information and hints on the use of the SFC System.

6 Maintenance and Repair

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)

7 Parts for Maintenance

This chapter provides information on parts for maintenance and repair.

8 Identifying Cables

This chapter provides information on cables used with the modules.

9 Appendix

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

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



1 Introduction to Supercritical Fluid Chromatography (SFC) History of SFC

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 II SFC Control Module coupled to an Agilent 1260 Infinity II Binary LC system optimized for SFC.

Introduction to Supercritical Fluid Chromatography (SFC) 1 Theory of 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 Figure 1 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.*

1 Introduction to Supercritical Fluid Chromatography (SFC) Benefits of SFC

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.

1

The Agilent 1260 Infinity II SFC System

The Agilent 1260 Infinity II SFC System consists of a binary SFC pump (G4782A), an SFC Multisampler (4767A), a thermostatted column compartment (G7116A), and either a DAD (G7115A) or MWD (G7165A) equipped with a high pressure SFC flow cell and the SFC control module (G4301A).

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 Agilent 1260 Infinity II SFC system is completely controlled by Agilent OpenLAB CDS software. The system diagram is shown below:



Figure 2 Agilent 1260 Infinity II Analytical SFC System

The Agilent 1260 Infinity II SFC Control Module (G4301A)

The Agilent 1260 Infinity II 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 Agilent 1260 Infinity II 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 Agilent 1260 Infinity II 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.

The Agilent 1260 Infinity II SFC Binary Pump (G4782A)

The 1260 Infinity II SFC Binary pump is equipped with passive inlet valves and with special seals and pistons 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.

The Agilent 1260 Infinity II 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* 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 $0.1 - 90 \ \mu\text{L}$ with highest precision, and excellent linearity over a broad volume range.



Figure 4 Overview of the Multisampler

The Agilent 1260 Infinity II Multicolumn Thermostat (G7116A)

Product Description

The Agilent 1260 Infinity II Multicolumn Thermostat (MCT) allows precise column thermostatting over a broad temperature range: from cooling down to 10 below ambient temperature to heating up to 85 °C, thus providing high flexibility for optimized speed and selectivity in standard LC separation.

Ultrahigh pressure valves enable a wide range of applications such as column selection from 4 columns in a single MCT, sample preparation for analyte enrichment or matrix removal, alternating column regeneration, etc.



Leak drain



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

The Agilent 1260 Infinity II Diode Array Detector WR (G7115A) and Agilent 1260 Infinity II 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 μ L 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

1 Introduction to Supercritical Fluid Chromatography (SFC)

The Agilent 1260 Infinity II SFC System



Site Requirements and Specifications

Site Requirements 22 Power Considerations 22 Power Cords 23 Bench space 23 Environment 25 Ventilation 25 Specifications 26 System Specifications 26 Specifications 26 Agilent 1260 Infinity II System 30

This chapter provides information on environmental requirements, physical and performance specifications only for the Agilent 1260 Infinity II SFC System.



2 Site Requirements and Specifications Site Requirements

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" on page 26. Consequently there is no voltage selector in the rear of the module.

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

WARNING

The module is partially energized when switched off, as long as the power cord is plugged in.

Repair work at the module can lead to personal injuries, e.g. electrical shock, when the cover is opened and the module is connected to power.

- → Always unplug the power cable before opening the cover.
- → Do not connect the power cable to the instrument while the covers are removed.

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

Different power cords are offered as options with the module. The female end of all power cords is identical. It plugs into the power-input socket at the rear of the module. The male end of each power cord is different and designed to match the wall socket of a particular country or region.

WARNING

Absence of ground connection or use of unspecified power cord

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

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

WARNING

Use of unsupplied cables

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

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

Bench space

Agilent 1260 Infinity II SFC Control Module

The Agilent 1260 Infinity II SFC Control Module requires approximately 30 cm (1 ft) of linear bench space immediately adjacent to the target Agilent 1260 Infinity II 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 1260 Infinity II system is also required to install cables and interface cards. As mentioned earlier, for optimal performance, the rear air space should not be heated significantly above room temperature by the exhaust of other instrumentation in the lab; rather, hot exhaust should be vented or directed upward from the instrument.

The module is designed to be installed on either side of the 1260 Infinity II system stack with sufficient high pressure transfer tubing to attach to a double-stacked system. If the 1260 Infinity II system is attached to a split-flow detector such as Mass Spec or ELSD, the Agilent 1260 Infinity II SFC Control Module should be positioned on the opposite side of the stack. Shelves overhanging the Agilent 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 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 Agilent 1260 Infinity II SFC Control Module can exist on either side of the 1260 Infinity II system, it is often easier to locate it on the left side. For more specification details see "Agilent 1260 Infinity II SFC Control Module" on page 26.

Agilent modules

The dimensions and weight of your module (see "Agilent 1260 Infinity II SFC Control Module" on page 26) 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 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 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.

Environment

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

System Specifications

Table 1	1260 Infinity II	SFC System	Specifications
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Туре	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.	

Agilent 1260 Infinity II SFC Control Module

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 Agilent 1260 Infinity II SFC Control Module. Make sure that your laboratory bench can support this weight.

WARNING

Personal injury

The module is heavy.

→ Enlist the aid of a co-worker to share the lifting load to avoid personal injury.

Туре	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)	adapted to new data sheet units
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)	
Non-operating altitude	up to 4600 m (15092 ft)	For storing the module
Safety standards	IEC, EN, CSA, UL	Installation Category II, Pollution Degree II For Indoor Use only
ISM Classification	ISM Group 1 Class A	According to CISPR 11

 Table 2
 Physical Specifications (G4301A)

The Agilent 1260 Infinity II 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. It should be positioned on a sturdy bench capable of holding the total weight of the Agilent 1260 Infinity II SFC Control Module plus the Agilent 1260 Infinity II System.

2 Site Requirements and Specifications Specifications

Туре	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 3 Chemical Specifications (G4301A)

Table 4 Wetted Materials Specifications (G4301A)

Туре	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

¹ Only when a legacy wash pump is installed.

Performance Specifications

Table 5	Performance S	pecifications	(G4301A))

Туре	Specifications
Hydraulic system	Single piston with proprietary motor control
Total hydraulic volume	<5 mL @ pressure <70 bar <25 mL @ 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)
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]

2 Site Requirements and Specifications Specifications

Agilent 1260 Infinity II System

WARNING

Unspecified Conditions

Operating the instrumentation under conditions other than its intended use might result in a potential safety hazard or might damage the instrumentation.

→ Never operate your instrumentation under conditions other than those specified by the vendor.



Installing the Agilent 1260 Infinity II SFC System

Hardware Installation 32 General Procedures 32 Installing the Agilent 1260 Infinity II SFC Control Module (G4301A) 35 Preparing the HPLC 53

This chapter provides an overview of the installation and setup of the hardware and software



3 Installing the Agilent 1260 Infinity II SFC System Hardware Installation

Hardware Installation

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 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.

3 Installing the Agilent 1260 Infinity II SFC System

Hardware Installation



Installing the Agilent 1260 Infinity II SFC Control Module (G4301A)

CAUTION

Early connection may damage the instrument

→ Do not connect AC power or interconnection cables or gas tubing to the Agilent 1260 Infinity II SFC Control Module until these installation procedures direct you to do so.

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.

Unpacking the Agilent 1260 Infinity II SFC Control Module

Damaged Packaging

When you receive your Agilent 1260 Infinity II SFC Control Module (G4301A), inspect the shipping boxes for any signs of damage. If the shipping container or cushioning material is damaged, notify the carrier and save the shipping material for inspection. Save all materials until the contents have been checked for completeness and the instrument has been mechanically and electrically checked.

CAUTION Signs of damage

→ If there are signs of damage to the module, please do not attempt to install or use the instrument.

Delivery Checklist

Compare the delivery checklist with the contents of the shipping boxes to ensure completeness of the shipment.

Connecting the Booster to the Agilent 1260 Infinity II 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 10 Connecting the Booster outlet line
This tube can be routed through the tee slots on the side of the Agilent 1260 Infinity II SFC Control Module. The tube is routed upward between the SFC control module and the Agilent 1260 Infinity II 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 the 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 11 Plumbing of the SFC Binary Pump

3 Installing the Agilent 1260 Infinity II SFC System Hardware Installation

Install the Multisampler

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



Figure 12 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 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 Agilent 1260 Infinity II 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 13 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 14 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 flow cell to high pressures will result in leakage or damage to the cell.

Installing the Agilent 1260 Infinity II SFC System 3 Hardware Installation



41

Connecting the BPR to the 1260 Infinity II stack

Connect the Agilent 1260 Infinity II 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 1260 Infinity 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 16 Connecting the SFC control module return Line

Connecting the Waste system to the SFC control module BPR Outlet

The Agilent 1260 Infinity II 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 17 Installing the Waste Container

3 Installing the Agilent 1260 Infinity II SFC System

Hardware Installation

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 Agilent 1260 Infinity II 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 Agilent 1260 Infinity II 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 18 Leak Tray Waste Line

3 Installing the Agilent 1260 Infinity II SFC System Hardware Installation

Cable connections to the HPLC

WARNING

Personal injury

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



Figure 19 Remote Connection 5188-8045 Remote Cable APG-ERI

0	Power off
I	Power on

Lastly, a cable should be connected to the Agilent 1260 Infinity II SFC Control Module USB port. This can be run to any convenient, compatible USB port on the PC.

CAN cabling between the Agilent 1260 Infinity II modules is unchanged from the normal, recommended means of interconnecting HPLC devices.

The Agilent 1260 Infinity 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 Agilent 1260 Infinity II SFC Control Module installation.

Connecting and operating multiple instruments on a single PC is not supported. Both the Agilent 1260 Infinity II SFC Control Module USB and Agilent 1260 Infinity II LAN connections must be made on the PC on which ChemStation is installed and which is used for instrument control.

3 Installing the Agilent 1260 Infinity II SFC System Hardware Installation

Connecting the Agilent 1260 Infinity II SFC Control Module to a Source of Carbon Dioxide

The Agilent 1260 Infinity II 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 1260 Infinity II 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

CO₂ 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 1260 Infinity II 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 Agilent 1260 Infinity II 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

3 Installing the Agilent 1260 Infinity II SFC System

Hardware Installation

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.



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 CO₂ supply.

CAUTION

Overtightening the fitting could damage the filter

→ Use two wrenches when installing the CO₂ 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 Agilent 1260 Infinity II 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 Agilent 1260 Infinity II SFC Control Module easily condenses any fluid that is present as a vapor, and prevent pump cavitations.

3 Installing the Agilent 1260 Infinity II SFC System Hardware Installation

System Flow Diagram

Figure 21 on page 52 schematically shows the correct plumbing of the SFC system.



Figure 21 SFC System flow diagram

Preparing the HPLC

Preferred Stack Arrangement

To minimize delays and broadening caused by excessive tube lengths, we recommend the following stack layout (the Agilent 1260 Infinity II SFC Control Module needs to be immediately adjacent to the LC stack, but can be placed on either side):



Figure 22 Examplary stack configuration for an Agilent 1260 Infinity II SFC System

3 Installing the Agilent 1260 Infinity II SFC System Hardware Installation

Agilent 1260 Infinity II 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.



How to configure the Agilent 1260 Infinity II SFC System and Agilent 1260 Infinity II SFC Control Module in OpenLAB CDS.



4 Configuring the System

Configuring the SFC system in OpenLAB CDS

Configuring the SFC system in OpenLAB CDS

NOTE

The Agilent 1260 Infinity II 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 Agilent OpenLAB CDS ChemStation Edition Instrument Configuration Guide 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*.

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

🕼 Configure Instrument						
Agilent LC System						
Use classic drivers						

4 Click Auto Configuration.

NOTE

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

Instrument Configuration: SFC								
Configurable Modules Agitent LC Modules and Systems Image: State of the sta	Selected Modules Auto Agilent LC Modules and Systems Configuration Binary Pump (G4302A) (PP00000002) Sampler (G4303A) (DEABF00102) Courne Come, (G1316C) (DE33001217) Courne Come, (G1315C) (DE73453347)	↑ ↓ Configure						
OK Cancel Help								

4 Configuring the System

Configuring the SFC system in OpenLAB CDS

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.

Instrument Configuration: SFC		
Configurable Modules C.C System (G4293C) C.C System (G4293C) C.C System (G4293C) C.C System (G4293C) C.C System (G42948) Generic Modules Agilert LT-ELSD (G4218A) 35900E v	Selected Modules Auto Agient LC Modules and Systems Configuration Binay Pump (G4302A) (PP00000002) Image: Sampler (G4303A) (DEABF00102) Image: Sampler (G4303A) (DEABF00102) Image: Column Comp. (G1316C] (DE33001217) Image: Column Comp. (G1315C] (DE73459347) Image: Column Comp. (G1315C] (DE73459347) Image: Column Comp. (G1315C] (DE73459347)	↑ ↓ Configure
OK	Cancel Help	

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

Instrument Configuration: SFC									
Instrument Configurable Modules	Auto Configuration Selected Modules Agilent LC Modules and Systems Signary Pump [G4302A] (PP0000002) Sampler [G4303A] (DEABF00102) Image: Sampler [G4303A] (DEABF00102) Image: Sampler [G4301A] (DEABF00102) Image: Sampler [G4301A] (DEABF00102)								
Agilent LT ELSD (G4218A)									
OK Cancel Help									

Configuring the SFC system in OpenLAB CDS

Device name	SFC		
Type ID	G4301A	Ŧ	
Serial number	1110US000409		
COM Port	COM7	-	
Firmware Revision	A.03.06 [0015]		
Options			
Pressure Unit	bar		
T TOSSARD OT IK	Dai		

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:

Setup Method					
🚆 Binary Pump 🌋 SFC 🦠 Sampler l Sampler Injector Program 🖋 Colu	mn Comp. 🛛 😻 Ir	nstrument Curves			
				SFC	G4301A
SFC Mode (uncheck for LC Mode)					
Back Pressure Regulator (BPR)					
BPR Pressure: 100.00 📜 bar					
BPR Temperature: 60.00 1 *C					
Stoptime					
C As Pump/Injector C 650.00 ; min					
Enable analysis with broader pressure range					
Show timetable graph					
- ·		OK	Apply	Cancel	Help

Setup Method	
🔛 Binary Pump 🖉 SFC 🧇 Sampler l 🎭 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.	
V Direction of Piston B	
SFC:	
✓ BackPressure (bar)	
⊘ AunPressure (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

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

4 Configuring the System Status

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.

Γ	Instrument Control Run Queue Easy Se	equence Easy Se	quence Setup								
	Sequence 🕕 Pause 🕞 Resu	ume 💿 Stop	DEF_SFC.N					Ĵ			0
	Sampler ? _ =	Binary	Pump ? _ 🗖		SFC	? _ =	Column C 💿 💿 EMF	Comp. ? _ =	🤝 D/ ⊙ @ EMF⊗	AD ? _ =	
	3.0 x loop	A1 B1 80.0 20.0	3.000 mL/min			133.13 bar -5.8 °C 100.04 bar		l			a Analysis
		00	141.50 par		-	60.1 °C	37.49 °C	40.00 °C	ġ.	4 4	
				Reference	Pressure	141.30 bar					
				Delta Press	sure	8.1/bar					_
				RDR Drossi	ire ire	100.04 bar					
				BPR Temp	erature	60.1 °C					
				Booster Pr	essure	133.13 bar					
				BoosterTer	mperatur	e -5.8 °C					
	0.00 / 0.00						Inst	trument Idle	1	⊖ 0n 0 0ff	

Click to hide the instrument actuals display.

3 Use ^③ and ^④ 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 Configuring the System

Control



Using the Agilent 1260 Infinity II SFC Control Module

Powering up the Module 66 Power-up Sequence and Operational Control States 67 **Operational Control States** 68 The OFF State 68 The STANDBY State 69 The ON State 70 Controlling the Agilent 1260 Infinity II SFC Control Module through the Agilent OpenLAB CDS 71 Running a method on the SFC system 73 Shutting Down the SFC System 75 Partial Shutdown 75

This chapter provides information and hints on the use of the SFC System.



5 Using the Agilent 1260 Infinity II SFC Control Module Powering up the Module

Powering up the Module

The Agilent 1260 Infinity II 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 Agilent 1260 Infinity II 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 68. 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.

5 Using the Agilent 1260 Infinity II SFC Control Module Operational Control States

Operational Control States

The Agilent 1260 Infinity II 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 Agilent 1260 Infinity II 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 Agilent 1260 Infinity II SFC Control Module control state.

The OFF State

The **OFF** state is characterized as follows:

- \checkmark The CO₂ 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 Agilent 1260 Infinity II 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:

- \checkmark The CO₂ 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 Agilent 1260 Infinity II 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.

5 Using the Agilent 1260 Infinity II SFC Control Module

Operational Control States

The ON State

The **ON** state is characterized as follows:

- \checkmark The CO₂ supply valve is open (powered).
- ✓ The booster pump drive is powered and begins to pump CO₂ 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 Agilent 1260 Infinity II 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 Agilent 1260 Infinity II 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.

Controlling the Agilent 1260 Infinity II SFC Control Module through the Agilent OpenLAB CDS

Method of G4301A (14)				_		×	
				SFC G4	301A		
SFC Mode (uncheck for LC Mode) Back Pressure Regulator (BPR) BPR Pressure: 130.00 ; bar		e (empty)					
		Time [min] 🗠 Function			Parameter		
BPR Temperature: 60.00 - C							
Stoptime							
As pump/injector							
Extended Ready Band							
Enable analysis with broader pressure range	<u>A</u> dd Cut	<u>R</u> emove Copy	Clear all Paste				
			<u>O</u> k	Apply	<u>C</u> ar	icel	

Figure 23 Editing the SFC control module Method Parameters

Editable method parameters for the Agilent 1260 Infinity II 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).

5 Using the Agilent 1260 Infinity II SFC Control Module

Controlling the Agilent 1260 Infinity II SFC Control Module through the Agilent OpenLAB CDS

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.
Running a method on the SFC system

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.

Method of G4767A (this.is.mt.Serial.Number)	- 🗆 X
	Multisampler (G4767A)
Operation Mode	Advanced
● SFC O LC	Sampling Speed
Injection	Draw Speed: 100.0 ; µL/min Eject Speed: 400.0 ; µL/min
Injection volume: 5.00 ¢ µL	Wart Time After Draw: 1.2 . s
Maradia Maradia	Feed Injection
Needle wash	Feed Speed: 400.0 → µL/min
Standard Wash 👻	Overfeed Volume: 4.00 ¢ µL
Stoptime Posttime	
As Pump/No Limit	Needle Height Position
	Offset: 0.0 🛟 mm
	Use Vial/Well Bottom Sensing
	High Throughput
	Sample Flush-Out Factor: 5.0 🛟
	Injection Valve to Bypass for Delay Volume Reduction
	Enable Overlapped Injection
	When Sample is Flushed Out
	After Period of Time
	0.00 ‡ minutes after injection
	Injection Path Cleaning
	Ok Apply Cancel

Figure 24 SFC Sampler method parameters

Running a method on the SFC system

Default Pump setting is 80 $\%~{\rm CO}_2$: 20 % modifier. When you select ${\rm CO}_2$ as the solvent, the **Compressibility** is set automatically. Typical **Flow** rate in SFC is 3 mL/min.

Method of G4782A (this.is.mt.Serial.Number)		-	\Box ×
		Binary Pump (G4782A)	
Flow	Advanced		
3.000 + mL/min	Minimum Stroke		
	Channel A:	Channel B:	
Solvents	Automatic	Automatic	
A: 100.0 1 % CO2 extended V.01 •	Ο 20 ; μL	Ο μL	
B: ☑ 0.0 ‡ % MeOH ▼ MeOH/POH/PNH2	Compressibility		
Pressure Limits	Use Solvent Types		
Min: 0.00 t bar Max: 400.00 t bar	Maximum Flow Gradient		
Stoptime Posttime	100 000		
	100.000 - MEMIR		
O As Injector/No Limit O Off			
<u>3.00</u> min <u>1.00</u> min			
	Timetable (empty)		
		<u>Ok</u> <u>Apply</u>	<u>C</u> ancel

Figure 25 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 CO₂

→ 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 CO₂ 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.

5 Using the Agilent 1260 Infinity II SFC Control Module

Shutting Down the SFC System

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 Agilent 1260 Infinity II SFC Control Module booster (the standby state will be selected) check the **Depressurize** box and click **OK**.

NOTE If the Agilent 1260 Infinity II 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 Agilent 1260 Infinity II 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 CO₂

→ Never inhale vapor issuing from an SFC flow line. Exposure to concentrations of CO₂ over 5 % in air can be lethal. Always keep tubes directed away your face. CO₂ 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 Agilent 1260 Infinity II SFC Control Module unit contains approximately 25 mL of liquid CO₂. The CO₂ 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.



6

Maintenance and Repair

Inspection and Preventative Maintenance Intervals 78 **Daily Procedure** 78 Every 3 months 79 As needed (Corrective) 79 **General Maintenance procedures** 80 **Booster Drawer** 80 **Replacing Fuses** 87 **Standard Decontamination** 88 **Plugged BPR Decontamination** 89 Preparing for storage or shipping 89

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)



Inspection and Preventative Maintenance Intervals

Inspection and Preventative Maintenance Intervals

Inspection and maintenance of the Agilent 1260 Infinity II 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).

Maintenance and Repair General Maintenance procedures

6

General Maintenance procedures

Booster Drawer



Figure 26 Exploded view of Booster Components

Removing the vapor shield

The vapor shield is not shown in the graphic Figure 26 on page 80.

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

Tools required • 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.

HINT

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.

General Maintenance procedures

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.

Tools required	• A • A • S • L • Is • E	A 1/4" open-end wre A 9/16" open-end wr A 3/16" hex-drive wr eal insertion/remova Iltrasonic bath sopropanol Deionized water	ench rench rench al tool
Parts required	# 1	p/n G4301-20200	Description Seal SFC Booster
NOTE	Each time the booster pump head is removed, the piston seal should be exchanged the seal surface may be easily scratched or distorted during removal. For this reaso 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 n tools or paper toweling to wipe, probe or contact these surfaces.		
	1 S	et the SFC conti	rol module mode to OFF .

Removing the Pump head

- **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:
 - a Remove inlet and outlet check valves.
 - **b** 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.
 - c Replace the check valves.
- **3** Wipe off any residual liquid from external (non-sealing) surfaces with a soft cloth such as a microfiber towel.

General Maintenance procedures

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	 Tools for removing the vapor shield (see "Removing the vapor shield" on page 81) and pump head (see "Removing the Pump head" on page 82) A 9/64" hex-drive wrench 			
Parts required	# p/n	Description		
	1 G4301-20101	Piston SFC Booster		
ΝΟΤΕ	In most cases, this proce	dure will be used only to replace a broken piston. Pumping CO_2		
NOTE	does not tend to leave deposits on the piston. Development of such deposits warran			
	materials. Release of extr	aneous materials into the CO_2 supply system may cause		
	contamination of the Agil	ent 1260 Infinity II Analytical SFC System.		
	1 Remove the Vapor S	Shield.		
	2 Remove the pump head.			
	3 Clean the pump hea	ad.		
CAUTION	Take care not to break c	oolant tubes		
	→ Use care removing th connected to a circula hard as this may loos or cause leaks in the	e chiller assembly from the mounting posts. The assembly is ation pump behind the drawer panel. Do not pull the flow lines en or crimp the tubes and cause the chiller to lose efficiency secondary cooler system.		
	4 With a gentle rockin	ng motion, loosen the chiller plate assembly and		
	carefully slide it for the assembly out of	ward off the pump head mounting posts. Carefully twist the way.		
	5 Use the 9/64" hex w spacer and very can fully exposes the pi	vrench to unscrew the two cap screws attaching the refully remove the spacer by pulling straight back. This ston 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.

6 Maintenance and Repair

General Maintenance procedures

- **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 CO₂ 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 Agilent 1260 Infinity II 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	#	Description
	1	Fuses with 8A 250V
	1 P	ower down the unit.
	2 D	isconnect the power cable from the power entry module.
	3 D	Pepress the release lever of the fuse drawer and pull the drawer straight ack 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 ScrubTM. 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.

6 Maintenance and Repair

General Maintenance procedures

- 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" on page 32. Store the components along with the original software disc and any upgrades with the module.



This chapter provides information on parts for maintenance and repair.

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7 Parts for Maintenance

Agilent 1260 Infinity II SFC Control Module Parts

Agilent 1260 Infinity II SFC Control Module Parts

G4301A SFC		
Control Module	p/n	Description
	G4301-65000	Mainboard G4301A
	G4301-65010	Motor Drive Board
	G4301-60501	Heated Nozzle Assembly LD
	G4301-00200	Thermal Isolation Cover Assy
	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	CO ₂ Inlet valve
	5188-9074	Tool 3/16"

Parts for Maintenance 7

Agilent 1260 Infinity II SFC Control Module Parts

G4301-67028		
Booster Drawer	p/n	Description
parts	G4301-60056	Capillary SFC Booster Pump out
	G4302-60066	Inlet valve SFC Pump
	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 1260 N-Type

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	Tubing Valve to Wash Pump

7 Parts for Maintenance Agilent 1260 Infinity II SFC DAD Parts

Agilent 1260 Infinity II SFC DAD Parts



Figure 27 DAD Flow Cell SFC

ltem	p/n	Description
1	G4301-60100	SFC Flow Cell
2	G4301-60200	SFC Low Dispersion Flow Cell
3	79883-68700	High Pressure Flow cell refurbishment Kit

NOTE

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



Identifying Cables

Cable Overview96Analog Cables98Remote Cables100CAN/LAN Cables104RS-232 Cables105USB106

This chapter provides information on cables used with the modules.





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 Description p/n 35900-60750 Agilent 35900A A/D converter 01046-60105 Analog cable (BNC to general purpose, spade lugs) **Remote cables** Description p/n 5188-8029 ERI to general purpose Remote Cable ERI – ERI 5188-8044 Remote Cable APG – ERI 5188-8045 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 Description p/n 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		
(not for FUSION	p/n	Description
Doard)	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's 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

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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.

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

Agilent Module to 35900 A/D converters

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
52.	2	Black	Analog -
	3	Red	Analog +
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Remote Cables

ERI (Enhanced Remote Interface)

5188-8029 ERI to general purpose

p⁄n 51	88-8029	pin	Color code	Enhanced Remote	Classic Remote	Active (TTL)
	D-Sub female 15way user's view to connector	1	white	101	START REQUEST	Low
		2	brown	102	STOP	Low
_		3	green	103	READY	High
\bigcirc	15	4	yellow	104	POWER ON	High
	1WI DGI +5V PGN +24	5	grey	105	NOT USED	
	tV ND ND FPron	6	pink	106	SHUT DOWN	Low
	2	7	blue	107	START	Low
		8	red	108	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			

5188-8044 ERI to ERI (Connector D_Subminiature 15 pin)

p/n 5188-8044	Pin (ERI)	Signal	Pin (ERI)	Active (TTL)
F7	10	GND	10	
	1	Start Request	1	Low
	2	Stop	2	Low
	3	Ready	3	High
	5	Power on	5	High
	4	Future	4	
	6	Shut Down	6	Low
	7	Start	7	Low
	8	Prepare	8	Low
	Ground	Cable Shielding	NC	

Table 6 5188-8044 ERI to ERI

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))

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		

Table 75188-8057 ERI to APG and RJ45



One end of these cables provides a 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.

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
50 00	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 Agilent 35900 A/D Converters

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
S 0 15	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



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's 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

8 Identifying Cables USB

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)



Appendix

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



General Safety Information

Safety Symbols

Symbol	Description
<u>/!</u>	The apparatus is marked with this symbol when the user should refer to the instruction manual in order to protect risk of harm to the operator and to protect the apparatus against damage.
	Indicates dangerous voltages.
	Indicates a protected ground terminal.
	Indicates eye damage may result from directly viewing the light produced by the deuterium lamp used in this product.
	The apparatus is marked with this symbol when hot surfaces are available and the user should not touch it when heated up.

Table 8 Safety Symbols

WARNING

A WARNING

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

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

CAUTION

A CAUTION

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

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

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

WARNING

Ensure the proper usage of the equipment.

The protection provided by the equipment may be impaired.

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

Safety Standards

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

General Safety Information

Operation

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

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

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

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

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

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

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

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

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

9 Appendix

The Waste Electrical and Electronic Equipment (WEEE) Directive (2002-96-EC)

The Waste Electrical and Electronic Equipment (WEEE) Directive (2002-96-EC)

Abstract

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

NOTE



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

Product Category: With reference to the equipment types in the WEEE Directive Annex I, this product is classed as a "Monitoring and Control instrumentation" product.

Do not dispose off in domestic household waste

To return unwanted products, contact your local Agilent office, or see www.agilent.com for more information.

Radio Interference

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

Test and Measurement

If test and measurement equipment is operated with equipment unscreened cables and/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.

Sound Emission

Manufacturer's Declaration

This statement is provided to comply with the requirements of the German Sound Emission Directive of 18 January 1991.

This product has a sound pressure emission (at the operator position) < 70 dB.

- Sound Pressure Lp < 70 dB (A)
- At Operator Position
- Normal Operation
- According to ISO 7779:1988/EN 27779/1991 (Type Test)

Solvent Information

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.

Use of Solvents

Observe the following recommendations on the use of solvents.

- Brown glass ware can avoid growth of algae.
- 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:

 $2\mathrm{CHCl}_3 \texttt{+} \mathrm{O}_2 \rightarrow 2\mathrm{COCl}_2 \texttt{+} 2\mathrm{HCl}$

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, di-isopropyl ether) such ethers 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.

Appendix 9 Agilent Technologies on Internet

Agilent Technologies on Internet

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

http://www.agilent.com

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- Site Requirements and Specifications
- Installing the System
- Configuring the System
- Using the Agilent 1260 Infinity SFC Control Module
- Maintenance
- Parts for Maintenance
- Identifying Cables
- Appendix

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