

Agilent InfinityLab Online SPE Solution

Instructions

Technical Information about the Agilent InfinityLab Online SPE Solution.

The following possible set-ups are described:

- G4742A Online SPE Starter Set
- G4743A Online SPE Multi Cartridge Kit
- G4744A Online SPE Direct Inject Kit
- G4744B Online SPE Direct Inject Kit, 1300 bar
- G4745A Online SPE High Volume Injection Kit

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Typical Applications

G4742A Online SPE Starter Set

The Agilent InfinityLab Online SPE Solutions are based on the 1290 Infinity Flexible Cube that houses re-usable SPE (solid phase extraction) cartridges and up to two valves. Combined with the Agilent 6400 Series Triple Quadrupole mass spectrometer the InfinityLab Online SPE Solution allows ultra-low, trace-level detection.

The G4742A Online SPE Starter Set builds the basis for all possible Online SPE solutions.

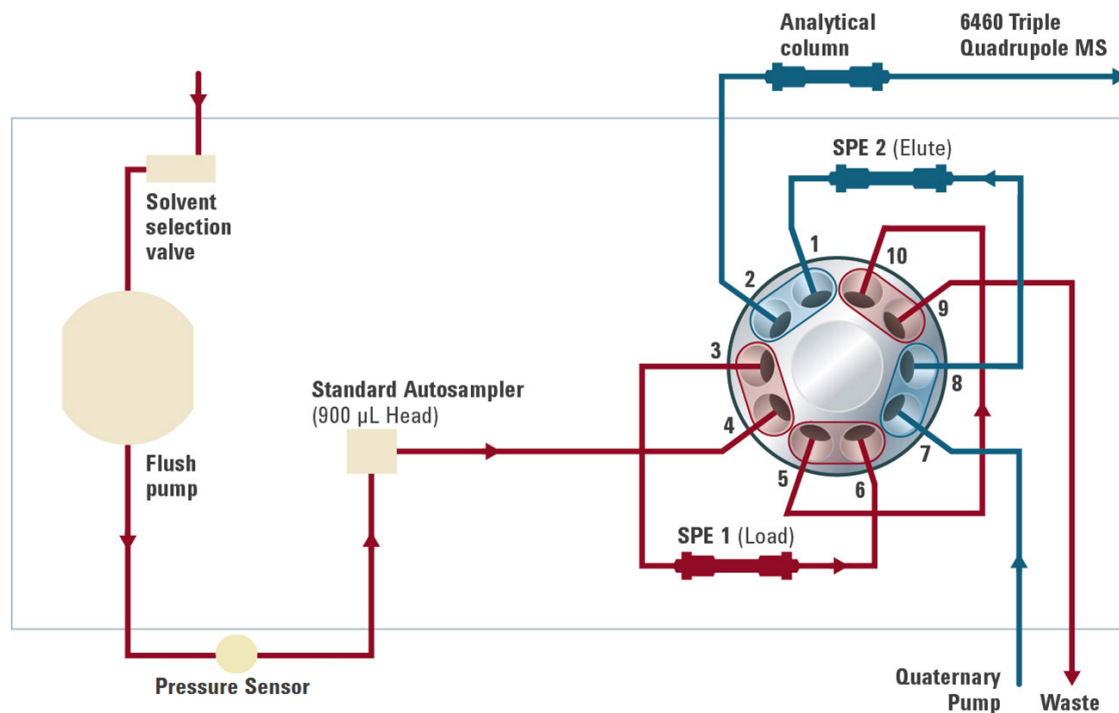


Figure 1: Example of the flow path

G4744A/B Online SPE Direct Inject Kit

NOTE

The maximum system pressure depends on the component, e.g. cartridge, which has the lowest pressure limit.

In addition to the Online SPE Starter Set G4742A the Online SPE Direct Injection Kit G4744A/B allows also to bypass the SPE cartridges and instead inject directly onto the analytical column.

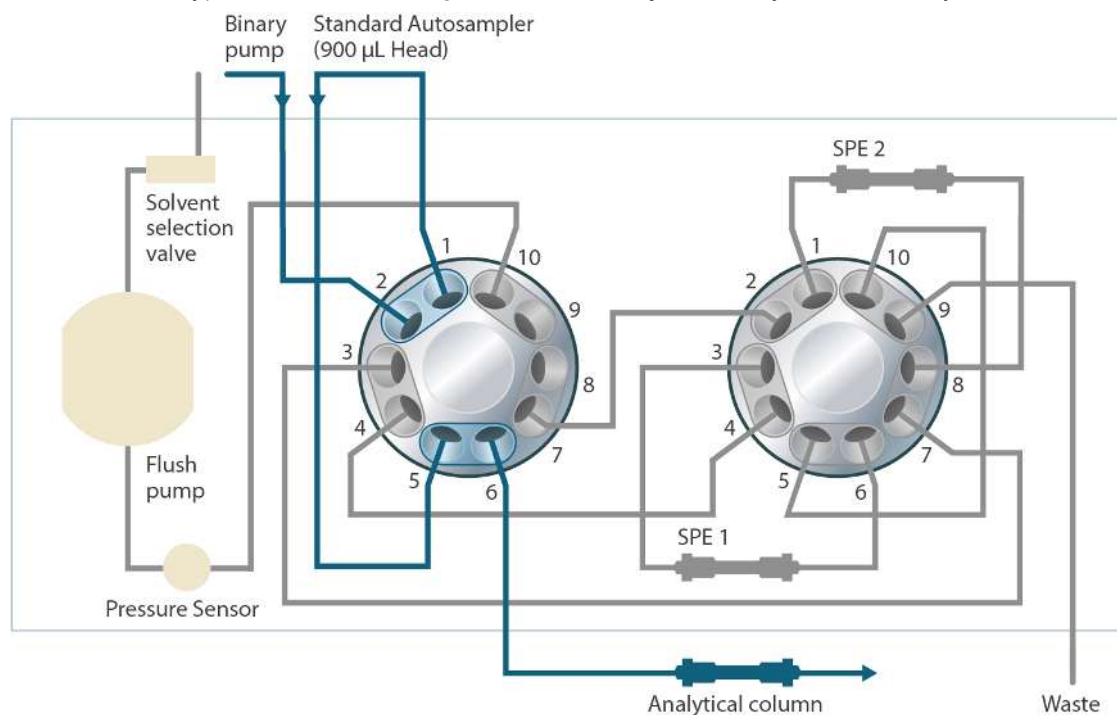


Figure 2: Direct Injection (Valve A in Position 2)

Valve A (left) in position 2 connects the flow path to valve B (right). From valve B, SPE cartridges can be chosen for either loading or elution.

Typical Applications

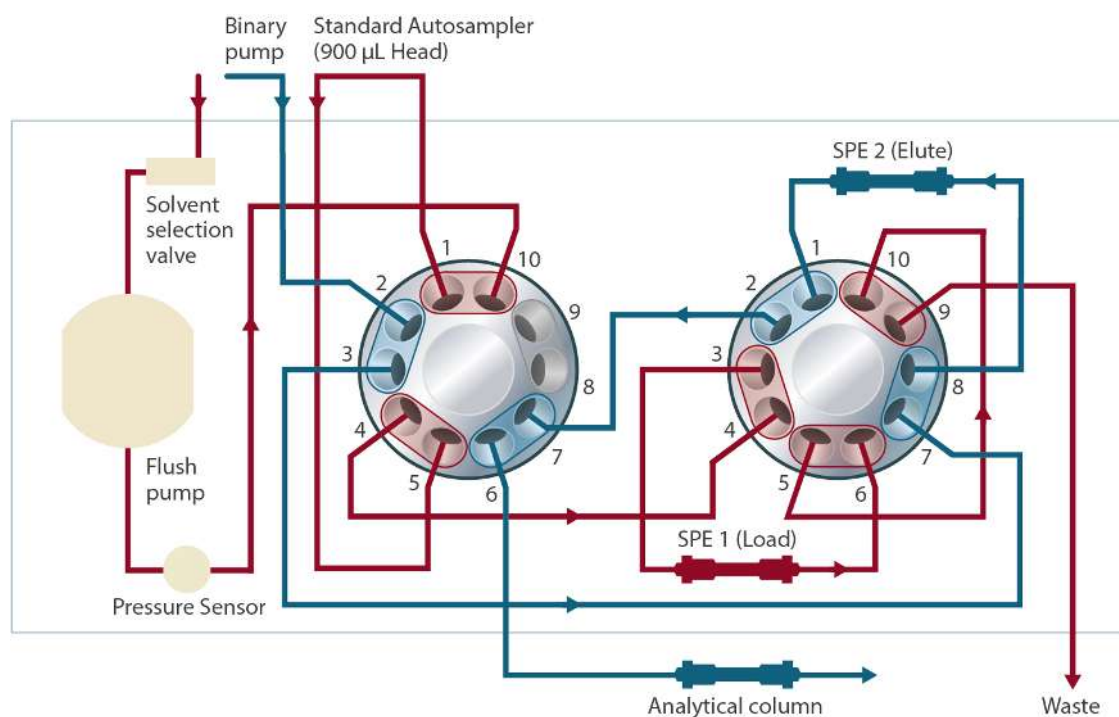


Figure 3: Valve A in Position 1 and Valve B in Position 1

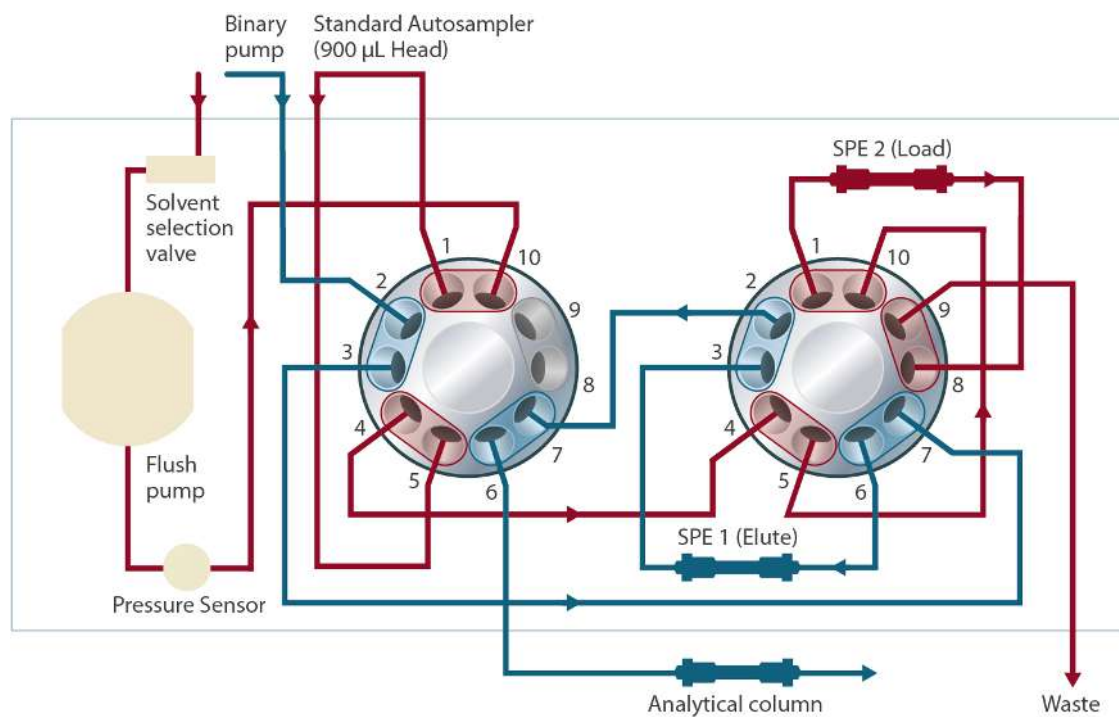


Figure 4: Valve A in Position 1 and Valve B in Position 2

G4743A Online SPE Multi Cartridge Kit

The Agilent InfinityLab Online SPE Solution offers a method development kit for online SPE cartridges.

This Online SPE Multi Cartridge Kit (G4743A) is based on the Online SPE Starter Set (G4742A) and is recommended in combination with the Direct Injection Kit (G4744A/B). It adds a column selection valve (6ps/14pt valve head, 800 bar) for SPE method development. This 6ps/14pt valve is installed next to the 1290 Infinity Flexible Cube in an external valve drive (G1170A). The Flexible Cube itself hosts two 2ps/10pt valves to support direct injections and online SPE.

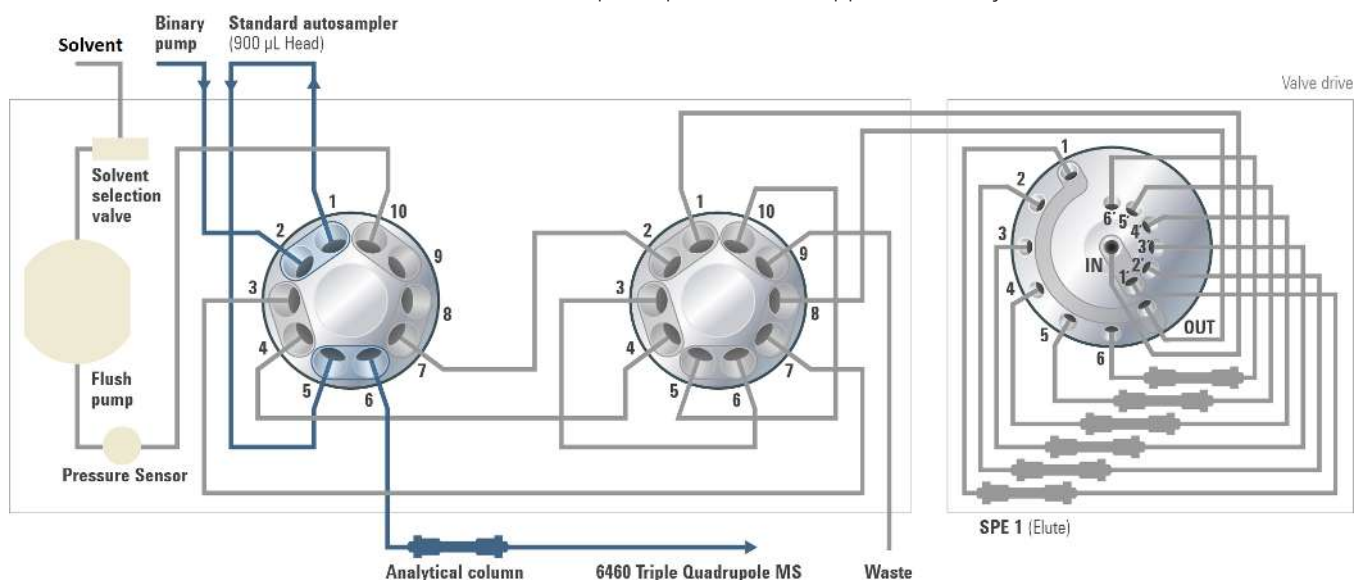


Figure 5: Flow path of the direct injection run

The Agilent 1290 Infinity Flexible Cube offers the possibility of direct injections and online SPE. For the flow path of the direct injection run, see [Figure 5](#) on page 5. In this position, the right 2ps/10pt valve and the additional 6-column selection valve in the external valve drive are disabled and not busy.

With the built-in solvent selection valve in the 1290 Infinity Flexible Cube, up to three solvents can be used to condition and to regenerate the cartridges, see [Figure 6](#) on page 6. It is recommended to use the quaternary pump as flush pump. Also, the right 2ps/10pt valve can be switched between online SPE and bypass, for cleaning the system.

Typical Applications

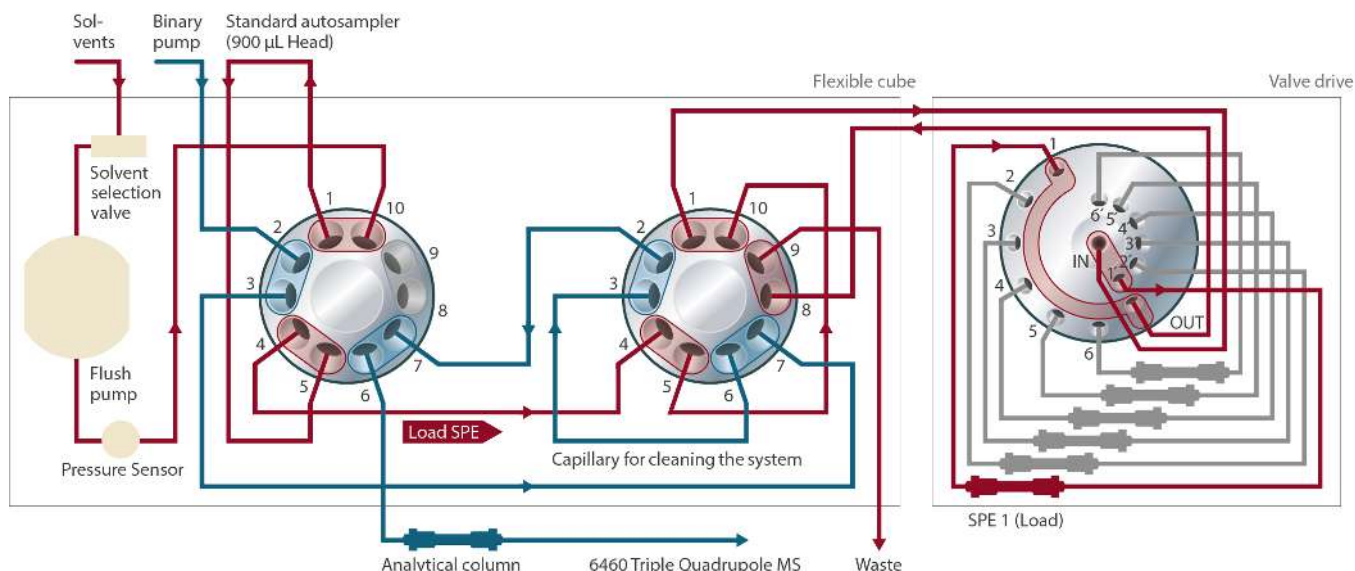


Figure 6: Flow path for online SPE

For online SPE, the flow path of the Agilent 1290 Infinity Flexible Cube goes through the left and right 2ps/10pt valve and afterwards to the 6-column selection valve. One out of the six cartridges is loaded with the sample.

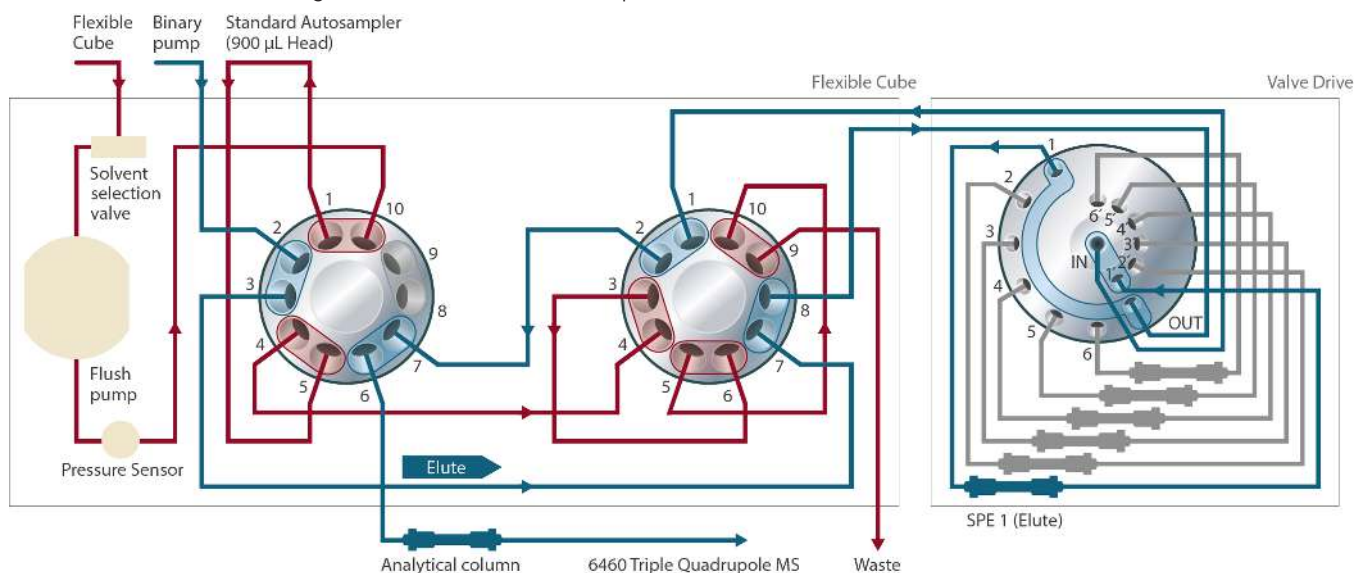


Figure 7: Flow path to elute the loaded cartridge

To elute the loaded cartridge in the previous step, the right 2ps/10pt valve is switched, see [Figure 7](#) on page 6.

NOTE

Additional information can be found in 5991-4005EN (Online SPE Multi-Cartridge Kit for Fast and Easy Method Development - The Agilent 1200 Infinity Series Online SPE Solution) .

G4745A Online SPE High Volume Injection Kit

This Technical Note describes an additional high volume injection kit with a 5 mL sample loop. The 5 mL sample loop is connected to an external 2-position/6-port valve, which makes it possible to switch the sample loop out of the flow path to run normal HPLC methods in direct inject mode without the delay volume of the external loop.

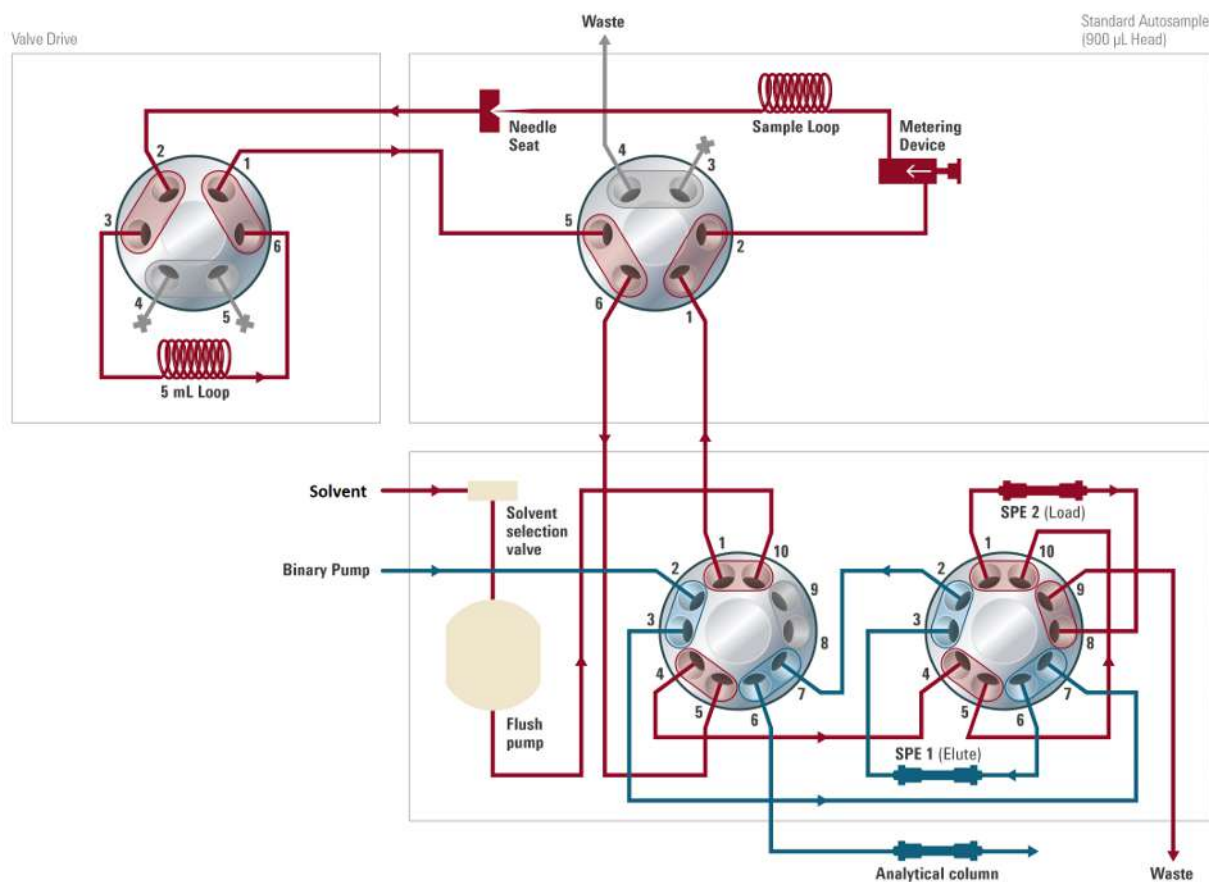


Figure 8: Switch diagram step 1

The Agilent 1290 Infinity Flexible Cube with two 2-position/10-port valves, the autosampler configuration, and the external valve drive with the 5 mL loop. The sample is drawn and ejected with the autosampler into the external loop. After the ejection, the 1290 Infinity Flexible Cube loads the samples from the loop to one of the trapping columns.

Typical Applications

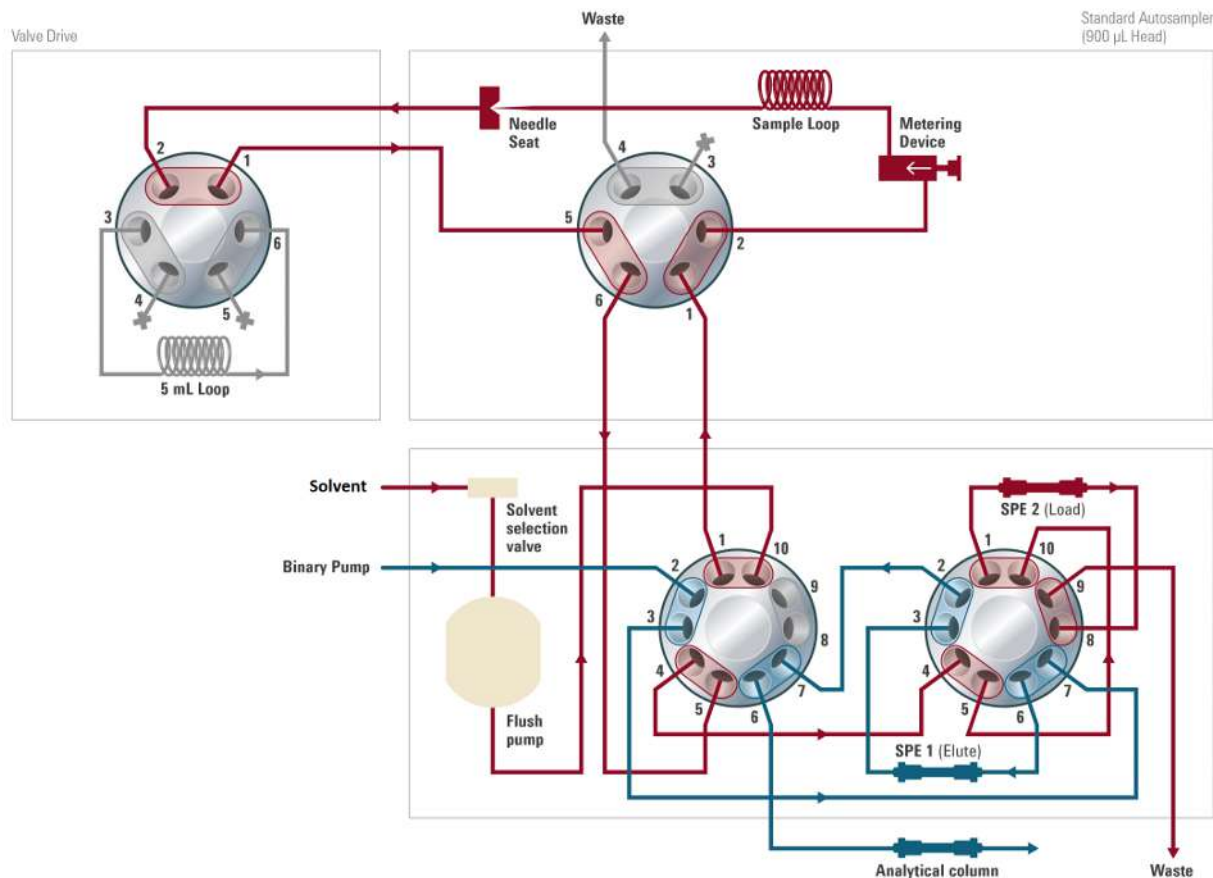


Figure 9: Switch diagram step 2

The 5 mL loop can be easily disabled if the position of the 2-position/6-port valve is changed. Hence, additional dead volume is saved.

NOTE

Additional information can be found in 5991-3173EN (High Volume Injection up to 5 mL with the Agilent 1200 Infinity Series Online SPE Solution for Highest Sensitivity) .


Delivery Checklist

Check the content of the delivery. You should have received the following:



G4742A Online SPE Starter Set

p/n	Description
 5067-4283	2-position/10-port valve, 800 bar
 5067-4294	Online SPE Capillary Starter Kit



5067-4294 (Online SPE Capillary Starter Kit)

Qty.	p/n	Description
2	 0890-1713	Tubing, PTFE, ID/OD 0.8 /1.6 mm
2	 0100-1516	Finger-tight fitting PEEK, 2/pk
1	 5982-1271	BE online PLRP-S 15 - 20 µm 2.1 mm x 12.5 mm 3/pk
1	 5065-9963	Capillary stainless steel 0.17 x 900 mm no fittings
1	 G1316-87319	Capillary ST 0.12 mm x 340 mm S/S
2	 5500-1237	Capillary 0.17 mm x 700 mm, ns/ns
1	 5065-9979	Capillary ST 0.25 mm x 250 mm
5	 5500-1262	Capillary 0.25 mm x 150 mm, ns/ns
2	 5065-9967	Fitting screw extra long
1	 G4742-90101	Online SPE Solutions Technical Note
1	 820999-901	Guard Column Hardware Kit

G4744A Online SPE Direct Inject Kit









p/n	Description
 5067-4283	2-position/10-port valve, 800 bar
 5067-4295	Online SPE Direct Inject Capillary Kit

G4744B Online SPE Direct Inject Kit 1300 bar




p/n	Description
 5067-4240	2 position/10 port valve head, 1300 bar
 5067-4295	Online SPE Direct Inject Capillary Kit

Delivery Checklist






5067-4295 (Online SPE Direct Inject Capillary Kit)

Qty.	p/n	Description
2	 5043-0277	PEEK blank nut for bio-compatible devices
1	 0100-1516	Finger-tight fitting PEEK, 2/pk
1	 5500-1262	Capillary 0.25 mm x 150 mm, ns/ns
1	 5022-6508	Capillary, 0.25 mm x 280 mm no fittings
2	 5021-1822	Capillary, 0.12 mm x 280 mm
1	 G4742-90101	Online SPE Solutions Technical Note
1	 5500-1192	Capillary ST 0.12 mm x 500 mm, long socket
1	 5065-9967	Fitting screw extra long



G4743A Online SPE Multi Cartridge Kit

p/n	Description
 5067-4284	6-column selector valve head, 800 bar In combination with the G4744A Online SPE Direct Injection Kit a 3rd valve drive is needed, for example an additional valve drive (G1170A).
 5067-4296	Online SPE Multi Cartridge Capillary Kit
 820999-901	Guard Column Hardware Kit





5067-4296 (Online SPE Multi Cartridge Capillary Kit)

Qty.	p/n	Description
2	 5067-5112	Capillary ST 0.17 mm x 280 mm SV/M
2	 5067-5120	Capillary ST 0.17 mm x 700 mm SL/M
12	 5067-5109	Capillary ST 0.17 mm x 90 mm SL/M
1	 5023-2504	Hex driver SW-4 slitted
1	 G4742-90101	Online SPE Solutions Technical Note

G4745A Online SPE High Volume Injection Kit

p/n	Description
 5067-4282	2-position/6-port valve head, 800 bar
 5067-4242	Online SPE High Volume Injection Capillary Kit

5067-4242 (Online SPE High Volume Injection Capillary Kit)

p/n	Description
 G1312-87303	StS Capillary 400 x 0.17 mm, pre-swaged (both ends)
 5022-2184	Union, stand LC flow, no fitting
 G2260-68711	5 mL Multi draw loop, for use with G2260A
 G4742-90101	Online SPE Solutions Technical Note

System Setup and Installation

This chapter provides information on system setup, installation of valve heads, and capillaries, and installation of solvent selection parts.

Documentation of the Individual Modules

The Online SPE System works in lots of different hardware configurations. For details of the individual modules mentioned in this guide, see Agilent InfinityLab LC Series User Documentation.

System Setup

NOTE

Online SPE is used for online purification and concentration of target analytes. A quick and easy check using Pesticides Checkout is available to verify the system.

The Online SPE System works in lots of different hardware configurations. Examples of two different Online SPE System setups are shown, see [Figure 10](#) on page 11 and [Figure 11](#) on page 12.

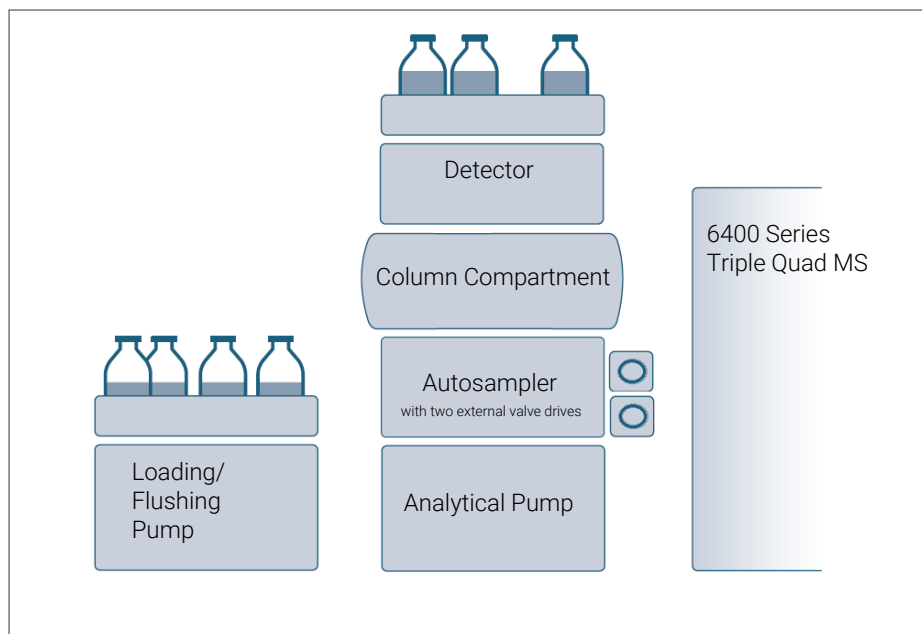


Figure 10: Online SPE System setup example with two external valve drives

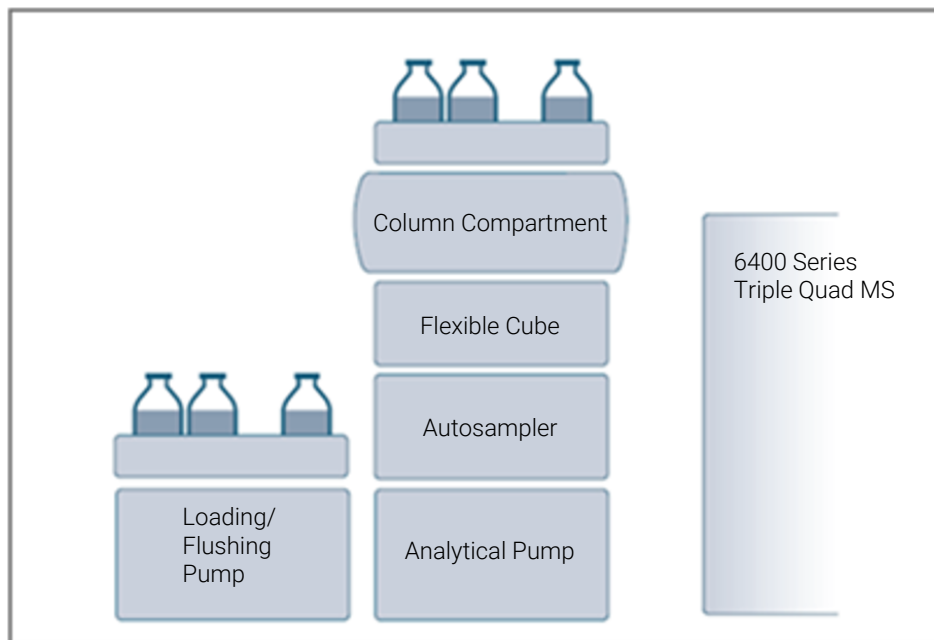


Figure 11: Online SPE System setup example with Flexible Cube

In principle, the connecting capillaries should be kept as short as possible to reduce extra-column band-broadening, and to keep the backpressure small. It is very important to keep the distance to the valve solution and the detector as short as possible. The next important connection is from the autosampler to the chosen Online SPE solution. Several set-ups taking these considerations into account are covered with the available capillary kits.

System Setup and Installation

System Labeling

The online SPE system is available in various hardware configurations. Label the system to identify the various pumps and valves as well as their position and flow direction on the capillaries from the outside.

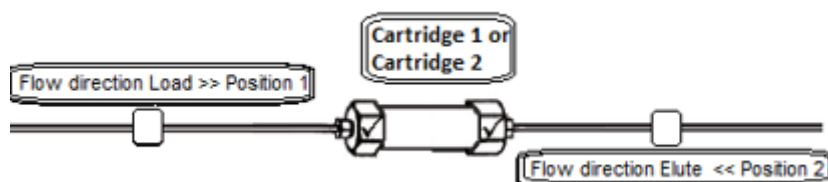


Figure 12: Example for outside labeling of capillaries

This information such as the different valve positions should also be visible in the CDS.

An example of labeling in the CDS would be e.g.:

- **Direct Inject valve**
 - Direct Inject Position 1 (Port1<2),
 - Online SPE Position 2 (Port1<10)
- **Online SPE valve**
 - Position 1 SPE2 Load SP1 Elute (Port1>10)
 - Position 2 SPE2 Elute > SP1 Load (Port 1> 2)

Instrument Status

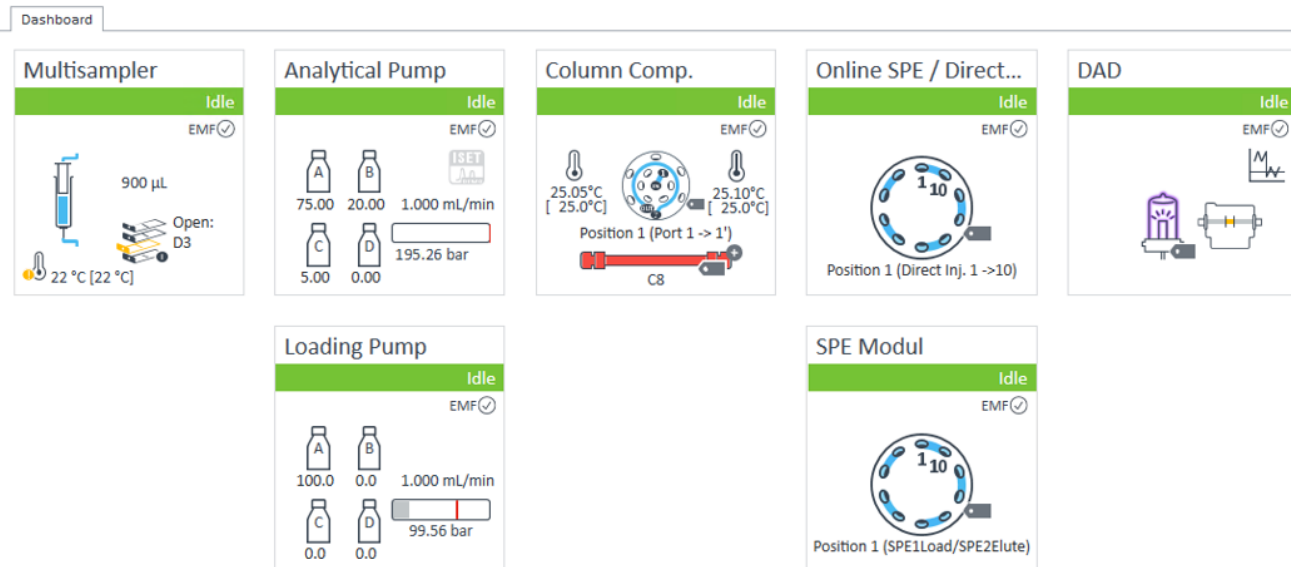


Figure 13: Example for CDS labeling

Online SPE Solution Setup

Setting up the Starter Set G4742A

NOTE

This section describes the setup of the Starter Set only. Refer for the following sections if used in combination with complementary Online SPE kits.

NOTE

More application details can be found in the Online SPE Application Note (p/n 5991-1738EN).

Setup the hydraulic path of the Online SPE Starter Set.

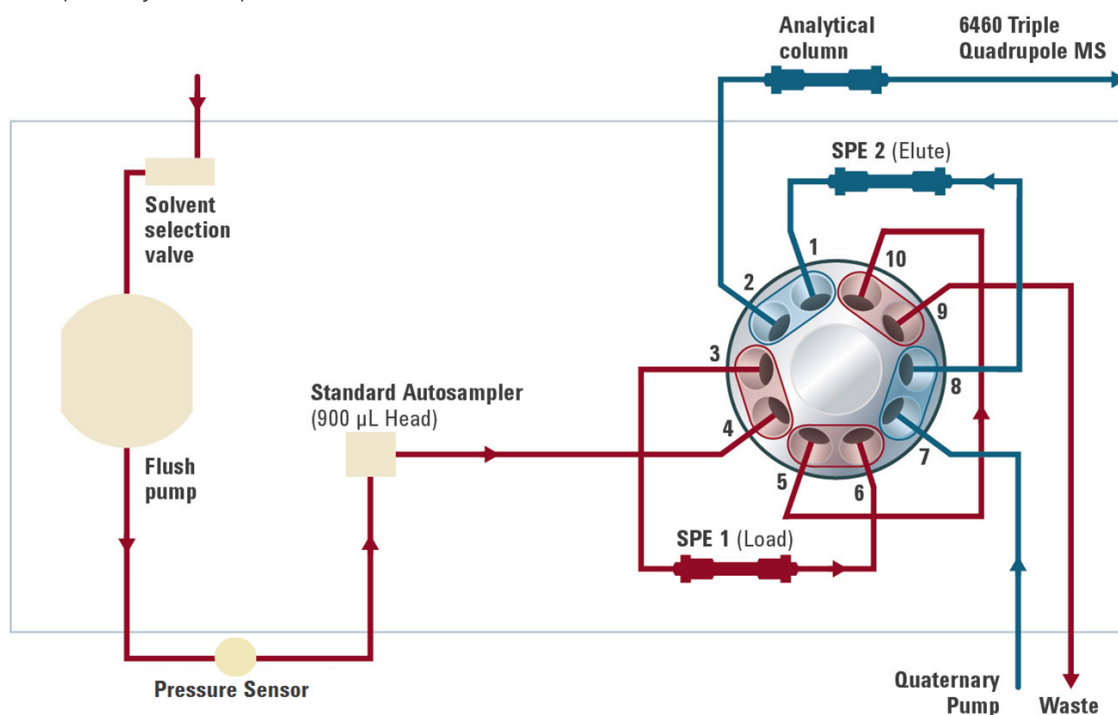


Figure 14: Flow path in an Online SPE Starter setup

Table 1: Example Capillary Connections for the Online SPE Starter Set (5067-4294)

#	P/N	Part Description	Connection
5	5500-1262	Capillary 0.25 mm x 150 mm, ns/ns	Valve - cartridges (4x) Valve crossing (Port 5 – Port 10)
1	G1316-87319	Capillary ST 0.12 mm x 340 mm S/S	Valve (port 2) - column use long fittings on valve
1	5065-9963	Capillary stainless steel 0.17 x 900 mm no fittings	LC pump - valve (Port 7)
2	5500-1237	Capillary 0.17 mm x 700 mm, ns/ns	Pressure Sensor - Autosampler Autosampler - Valve (port 4)
1	5065-9979	Capillary ST 0.25 mm x 250 mm	Flush pump - pressure sensor (Use PEEK fitting for Flush pump)
1	0890-1713	Tubing, PTFE, ID/OD 0.8 /1.6 mm	valve (port 9)

Setting up the Direct Inject Kit G4744A/B

NOTE

The Direct Inject Kit can only be used in combination with the G4742A Online SPE Starter Set.

Setup the hydraulic path of the Online SPE Direct Inject Kit.

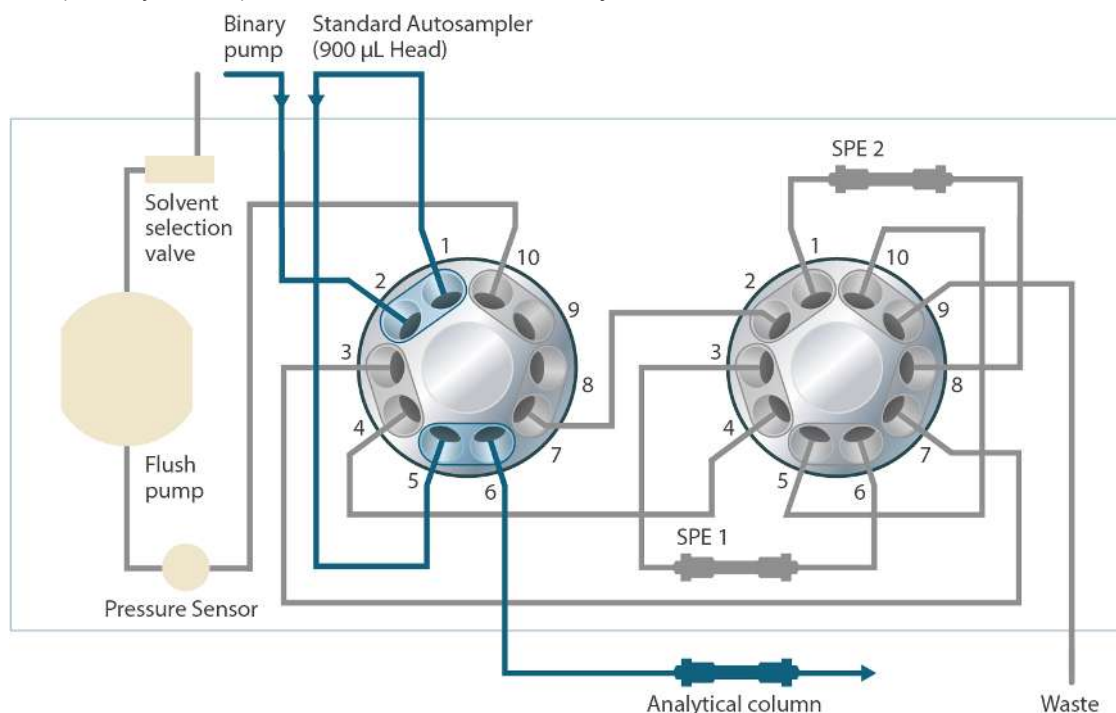


Figure 15: Direct Injection (Valve A in Position 2)

Use the capillaries and parts as described in [Table 2](#) on page 15

Table 2: Connecting the Online SPE Direct Inject Solution by using capillaries from both kits, the Online SPE Capillary Starter Kit (p/n 5067-4294) and Online SPE Direct Inject Capillary Kit (p/n 5067-4295)

P/N	Part Description	Connection
5022-6508	Capillary, 0.25 mm x 280 mm no fittings	Valve A / Port 4 - Valve B / Port 4
5021-1822	Capillary, 0.12 mm x 280 mm	Valve A / Port 3 - Valve B / Port 7 Valve A / Port 7 - Valve B / Port 2
5500-1262	Capillary 0.25 mm x 150 mm, ns/ns	Pressure Sensor - Valve A / Port 10 Valve to cartridges (4x) Valve crossing, Valve B (Port 5 – Port 10)
G1316-87319	Capillary ST 0.12 mm x 340 mm S/S	Valve A (port 6) - column Autosampler – Valve A / Port 5 use long fittings on valve
5065-9963	5065-9963	LC pump to valve A (Port 2)
5500-1237	5500-1237	Valve A (port 1) - Autosampler
5065-9979	Capillary ST 0.25 mm x 250 mm	Flush pump - pressure sensor (Use PEEK fitting for Flush pump)
0890-1713	Tubing, PTFE, ID/OD 0.8 /1.6 mm	valve B (port 9)
5043-0277	PEEK blank nut for bio-compatible devices	Valve A (port 8, port 9)

Setting up the SPE Multi Cartridge Kit G4743A

NOTE

The Online SPE Multi Cartridge Kit can only be used in combination with the Online SPE Starter Set (G4742A) and the Online SPE Direct Inject Kit (G4744A/B).

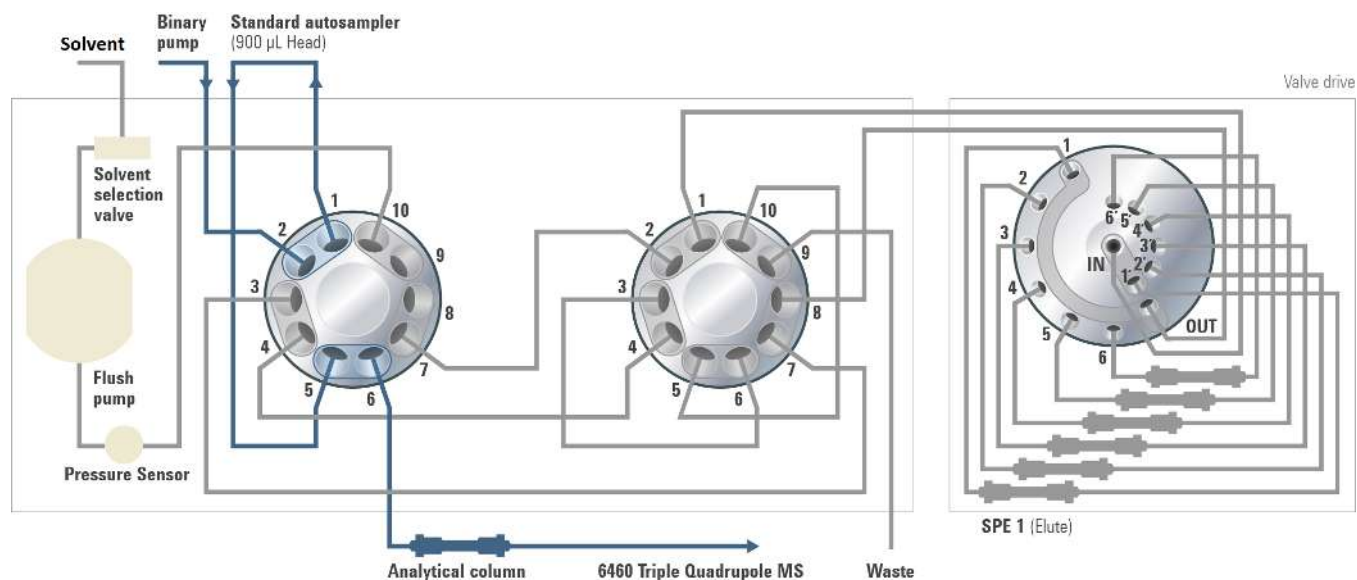


Figure 16: Flow path of the direct injection run

Table 3: Capillary Connections for the Multi Cartridge Solution (5067-4296)

P/N	Part Description	Connection
5022-6508	Capillary, 0.25 mm x 280 mm no fittings	Valve A / Port 4 - Valve B / Port 4
5021-1822	Capillary, 0.12 mm x 280 mm	Valve A / Port 3 - Valve B / Port 7 Valve A / Port 7 - Valve B / Port 2
5500-1262	Capillary 0.25 mm x 150 mm, ns/ns	Pressure Sensor - Valve A / Port 10 Valve crossing, Valve B (Port 3 – Port 6) Valve crossing, Valve B (Port 5 – Port 10)
G1316-87319	Capillary ST 0.12 mm x 340 mm S/S	Valve A (port 6) - column Autosampler – Valve A / Port 5 use long fittings on valve
5065-9963	Capillary stainless steel 0.17 x 900 mm no fittings	LC pump - valve A (Port 2)
5500-1237	Capillary 0.17 mm x 700 mm, ns/ns	Valve A (port 1) - Autosampler
5065-9979	Capillary ST 0.25 mm x 250 mm	Flush pump - Pressure sensor (Use PEEK fitting for Flush pump)
0890-1713	Tubing, PTFE, ID/OD 0.8 /1.6 mm	Valve B (port 9)
5043-0277	PEEK blank nut for bio-compatible devices	Valve A (port 8, port 9)
5067-5112	Capillary ST 0.17 mm x 280 mm SV/M	Valve B (port 1) - Selection valve 'IN' port Selection valve 'OUT' port - Valve B (port 8)
5067-5109	Capillary ST 0.17 mm x 90 mm SL/M	Cartridges - Selection valve
5067-5120	Capillary ST 0.17 mm x 700 mm SL/M	In case of combining two Multi Cartridge Solutions: Valve B / Port 3 - 2nd Selection Valve / IN Port Valve B / Port 6 - 2nd Selection Valve / OUT Port

Setting Up the SPE High Volume Injection Kit G4745A

NOTE

The High volume Injection Kit can only be used in combination with the G4742A Online SPE Starter Set.
If the G4744A Online SPE Direct Inject Kit is used additionally in combination a third valve drive is required in addition (for example a G1170A) as host for the 2pos/6port valve head.

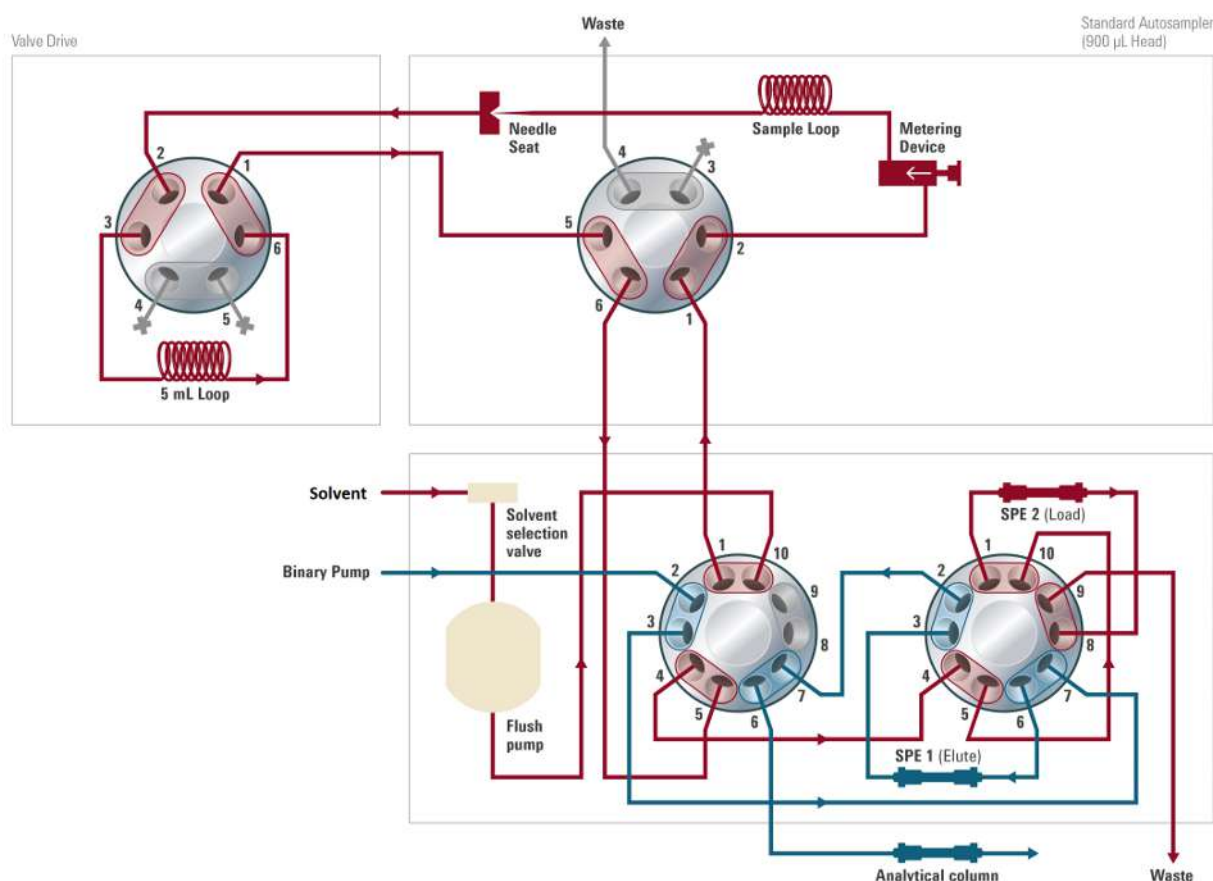


Figure 17: Flow path of High Volume Kit in combination with the Direct Inject Kit

Table 4: Additional capillary connections for the High Volume Injection Kit (5067-4242)

P/N	Part Description	Connection
G1312-87303	StS Capillary 400 x 0.17 mm, pre-swaged (both ends)	Needle Seat / Valve Port 2 Valve Port 1/ ALS Injection Valve Port 5
5022-2184	Union, stand LC flow, no fitting	Connector Needle seat to capillary G1312-87303
G2260-68711	5 mL Multi draw loop, for use with G2260A (for use with G2260A)	Valve Port 3 / Valve Port 6

Autosampler-Related Information

When injecting larger volumes with an autosampler (e.g. with the multidraw technique or with a high-volume loop), the following recommendations will help to ensure high precision and reproducibility:

- **Adjust the aspiration and ejection speed. Select the correct slotted needle assembly.**

Using the 900- μ L analysis head may lead to loss of accuracy, due to large sampling volumes, high speeds, and negative pressure. In that case the methods should be adjusted to allow sufficient time for pressure equalization. The following recommended values (**Draw Speed**, **Eject Speed**, **Wait Time After Draw**) are a good starting point for a standard HPLC sample.

Multisampler (G7167A)	
Advanced	
Sampling Speed	
Draw Speed:	900.0 μ L/min
Eject Speed:	3,000.0 μ L/min
Wait Time After Draw:	30.0 s

Figure 18: Recommended values for Sampling Speed

There are the following options:

- **Draw Speed** of 900 μ L/min and **Wait Time After Draw** of at least 30 s, **OR**
- **Draw Speed** of 600 μ L/min or less and default **Wait Time After Draw** of 1.2 s

If more viscous samples or special techniques are used, the corresponding values should be determined and adjusted experimentally, as incorrect settings are often responsible for problems with area accuracy.

- **Set the needle height according to the dimensions of the vial.**

Information on needle height can be found in your autosampler manual.

- **Handle and select the correct sample vial.**

If more than one injection is taken from the same vial (e.g. injections of standards or volumes larger than 900 μ L), we recommend using 6 mL vials. As there is only a limited amount of solution in the vial, the total volume of injections withdrawn must not exceed the capacity of the vial. For large volumes, we also recommend the use of preslit septa.

- **Avoid overfilling the sample vial.**

Sample at the underside of the septum can lead to carry over. To avoid carrying over:

- Fill the vial only to a maximum of 2/3 of the specified volume.
- Never shake the vial.

NOTE

The max pressure for an autosampler with the 900 μ L option is 400 bar.

Installing the Valve Heads

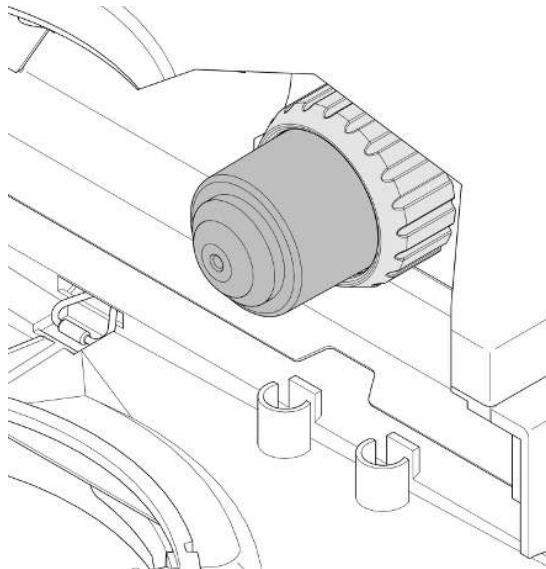
The valve drives are factory-installed in the Thermostatted Column Compartment, in the Flexible Cube, and in the 1290 Infinity Valve Drive. The valve heads are interchangeable and can be easily mounted.

At the first installation, the dummy valve has to be removed, see [Removing the Valve Dummy](#) on page 19. The valve heads can be installed by mounting the valve heads onto the valve drives and fastening the nut manually (do not use any tools).

Be sure that the guide pin snaps into the groove of the valve drive thread.

Removing the Valve Dummy

- 1 To remove the valve dummy, loosen the nut manually.



Installing the Valve Head and Connecting Capillaries

CAUTION

The valve actuator contains sensitive optical parts, which need to be protected from dust and other pollution. Pollution of these parts can impair the accurate selection of valve ports and therefore bias measurement results.

- Always install a valve head for operation and storage. For protecting the actuator, a dummy valve head can be used instead of a functional valve. Do not touch parts inside the actuator.

CAUTION

Column Damage or Bias Measurement Results

Switching the valve to a wrong position can damage the column or bias measurement results.

- Fit the lobe to the groove to make sure the valve is switched to the correct position.

Installing the Valve Heads

CAUTION

Valve Damage

Using a low pressure valve on the high pressure side can damage the valve.

- When using multiple column compartments as part of a method development solution, make sure that the high pressure valve head is connected to the autosampler and the low pressure valve head is connected to the detector.

NOTE

The tag reader reads the valve head properties from the valve head RFID tag during initialization of the module. Valve properties will not be updated, if the valve head is replaced while the module is on. Selection of valve port positions can fail, if the instrument does not know the properties of the installed valve.

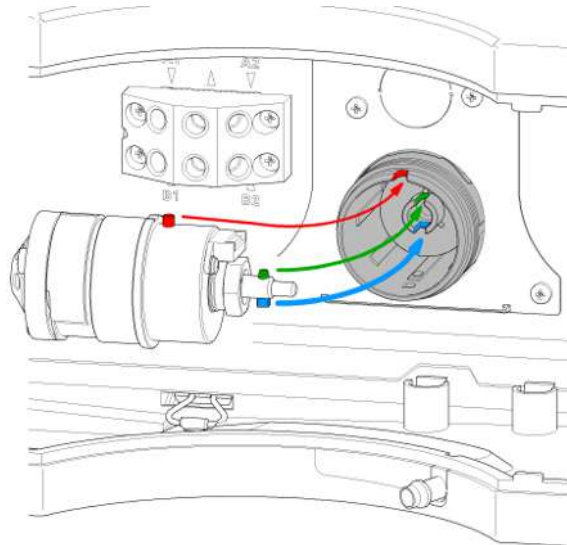
NOTE

To have the valve correctly recognized by the Flex Cube you must have the module powered off for at least 10 s.

NOTE

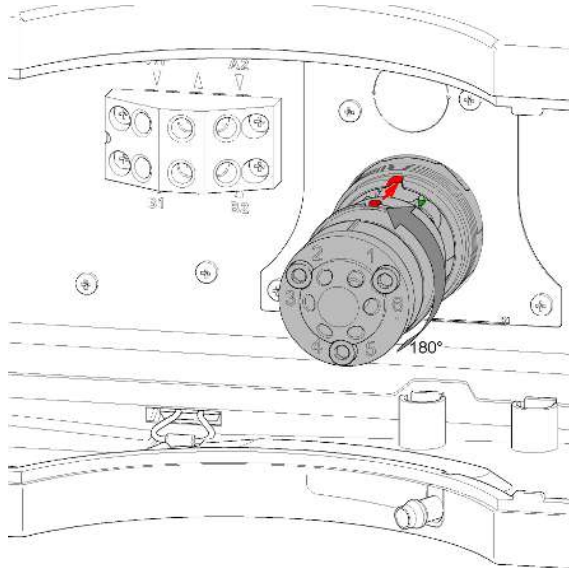
For firmware requirements see 01200-90134 (Information on RFID Tag Technical Note) which is included to each valve head.

- 1 The two different pins (blue & green) on the valve shaft must fit into the corresponding grooves of the actuator axis. The outside pin (red) must fit into the outside groove.

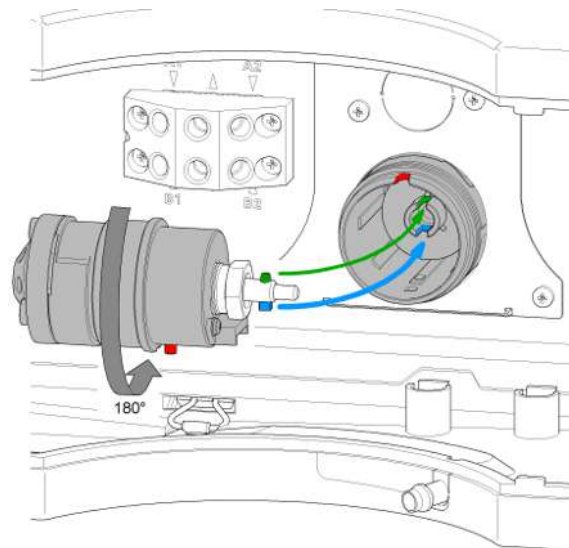


Installing the Valve Heads

- 2 It can be possible that you have to turn the valve head until the outside pin completely locks in. While turning the valve you should feel some resistance from the drive.

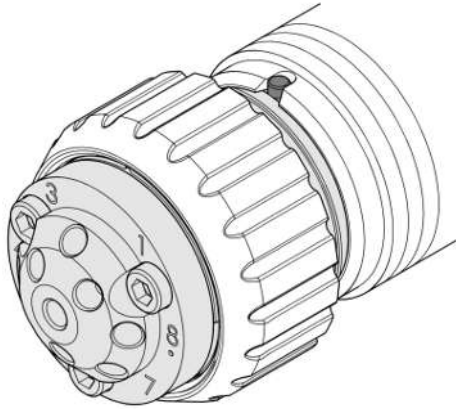


Otherwise the pins on the valve shaft are not properly fitted into the corresponding adapters on the valve drive.



Installing the Valve Heads

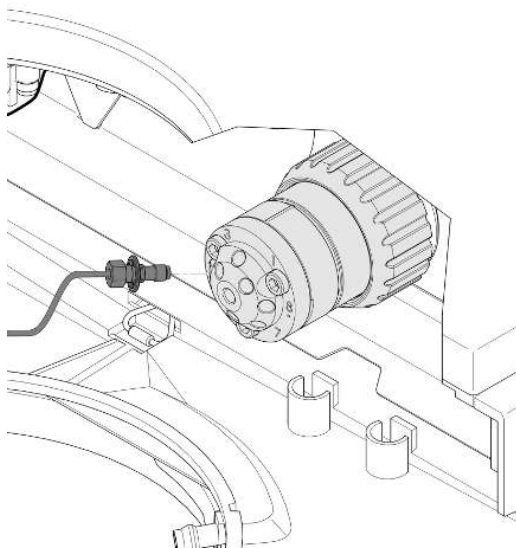
- 3 When the outer pin is locked into the groove, manually screw the nut onto the valve head.



NOTE

Fasten the nut manually. Do not use any tools.

- 4 Install all required capillary connections to the valve.



- 5 Power on or power-cycle your module, so the valve head gets recognized during module initialization.

NOTE

Power Off the Infinity valve drive for at least 10 s.

Valve Parts

Replacement Parts

Table 5: Replacement parts

Valve	Rotor Seal	Stator Head	Stator Screws	Stator Ring
5067-4283 2ps/10pt valve head, 800 bar	0101-1415 (PEEK)	5068-0165	5068-0019	n.a.
5067-4284 6 column selector valve head, 800 bar	5068-0298 (PEEK)	5068-0241	5068-0089	n.a.
5067-4240 2ps/10pt valve head, 1300 bar	5068-0205	5068-0011	5068-0019	1535-4045

Valve Head Parts

NOTE

The figure below illustrates replacement parts for the valve heads, with the 12-position/13-port selector valve as an example. The valves can vary in their appearance and do not necessarily include all of the illustrated parts. Neither, every spare part is available for each flavor of the valve.

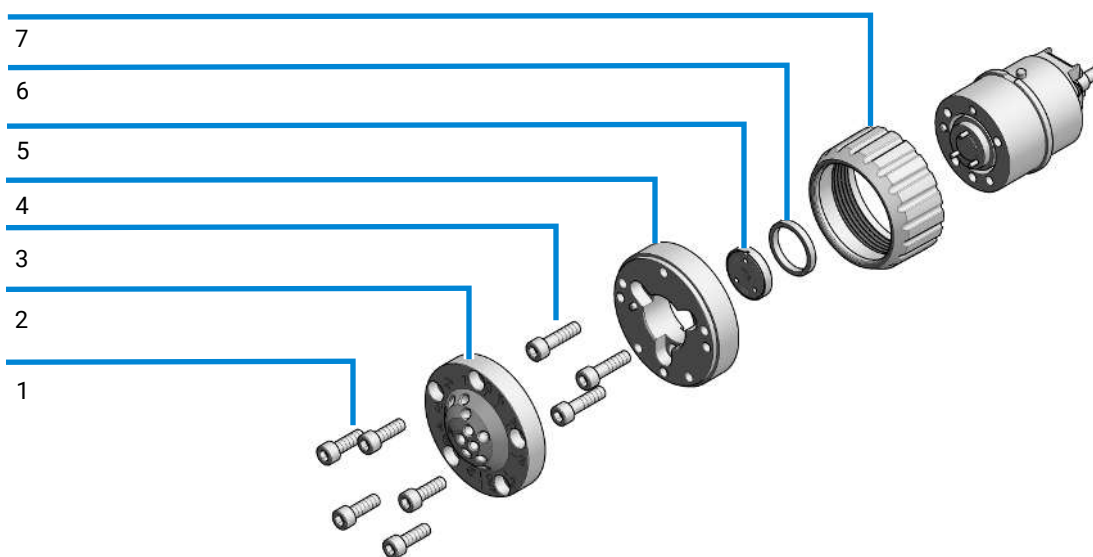


Figure 19: Valve Head Parts (example)

1	Stator screws
2	Stator head assembly
3	Stator ring screws (not available)
4	Stator ring (available for service only)
5	Rotor seal
6	Bearing ring
7	Spanner nut (available for service only)

