

Agilent InfinityLab Online SPE Solution

Instruction

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

Contents

Typical Applications 2

| | |
|---|---|
| G4742A Online SPE Starter Set | 2 |
| G4744A/B Online SPE Direct Inject Kit | 3 |
| G4743A Online SPE Multi Cartridge Kit | 5 |
| G4745A Online SPE High Volume Injection Kit | 7 |

Delivery Checklist 9

System Setup and Installation 12

| | |
|---|----|
| Documentation of the Individual Modules | 12 |
| System Setup | 12 |

Online SPE Solution Setup 14

| | |
|---|----|
| Setting up the Starter Set G4742A | 14 |
| Setting up the Direct Inject Kit G4744A/B | 16 |
| Setting up the SPE Multi Cartridge Kit G4743A | 18 |
| Setting Up the SPE High Volume Injection Kit G4745A | 19 |

Installing the Valve Heads 20

Valve Parts 23



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.

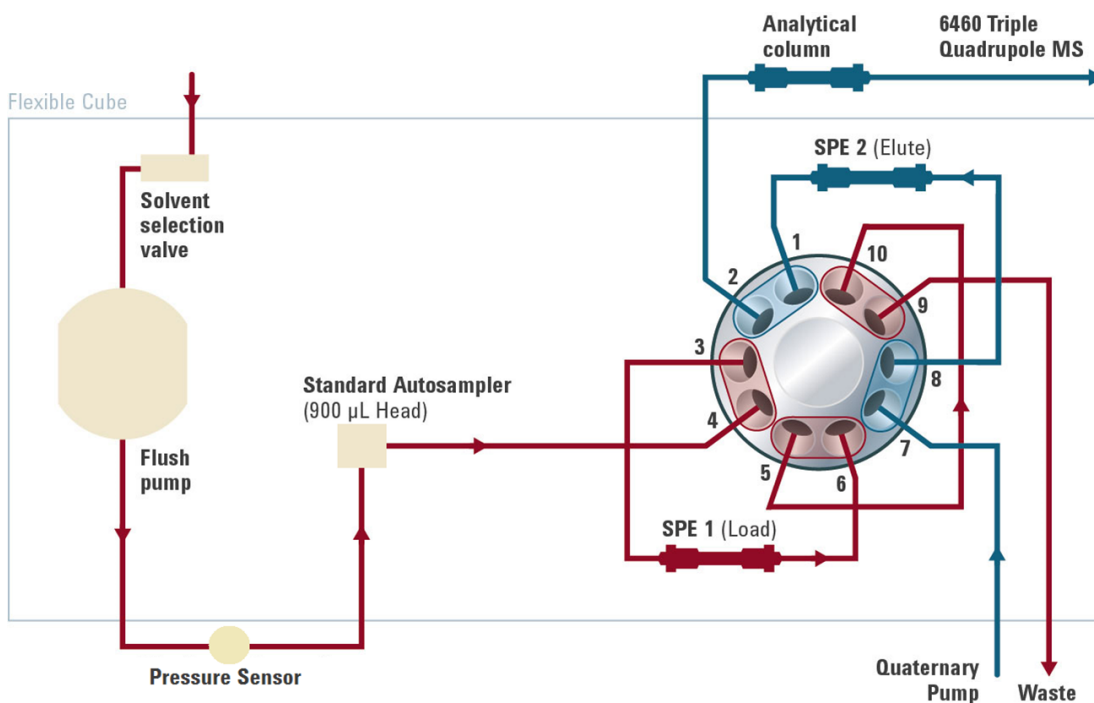


Figure 1 Example of the flow path

G4744A/B Online SPE Direct Inject Kit

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.

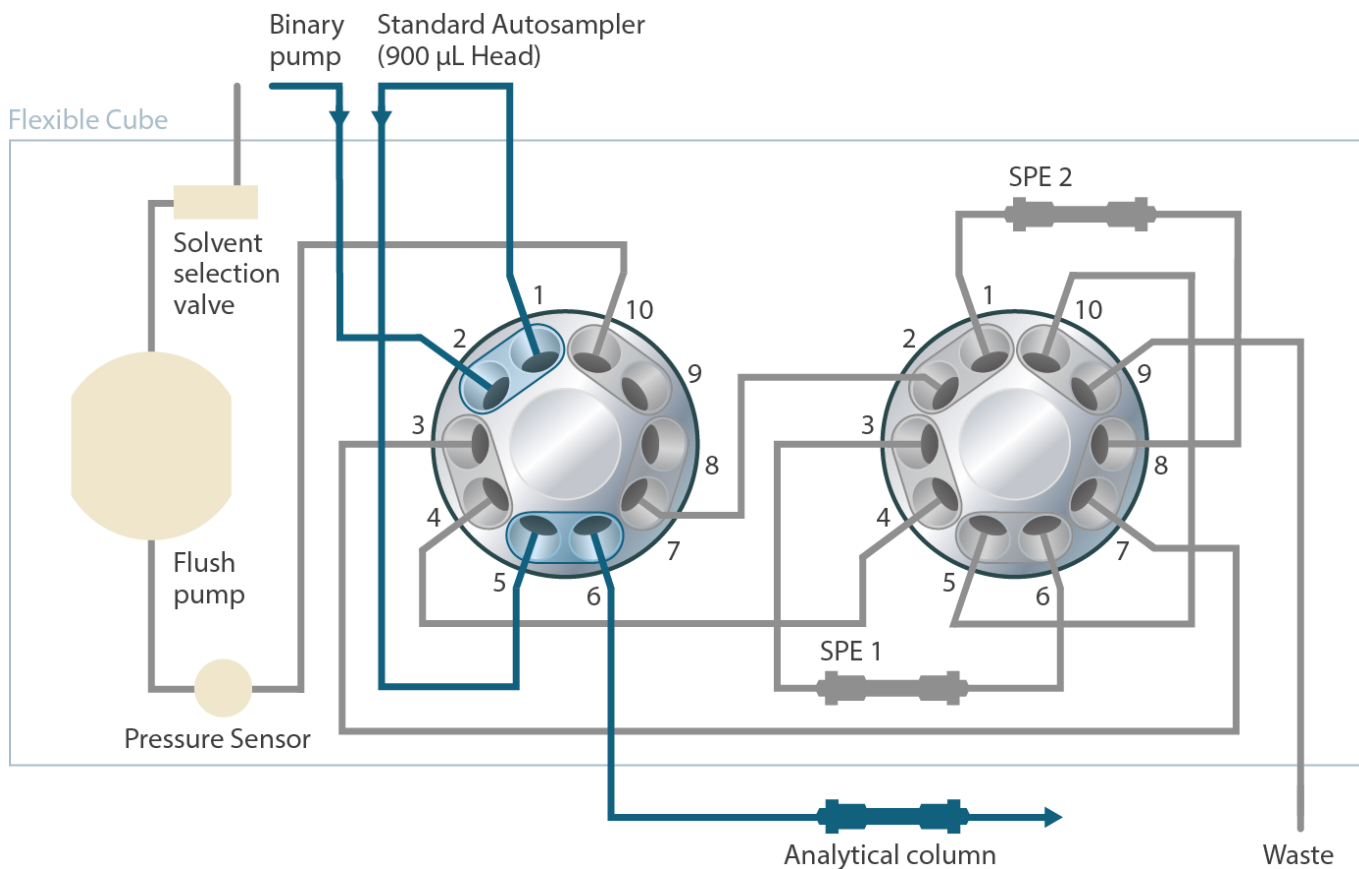


Figure 2 Direct Injection (Valve A in Position 1)

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
G4744A/B Online SPE Direct Inject Kit

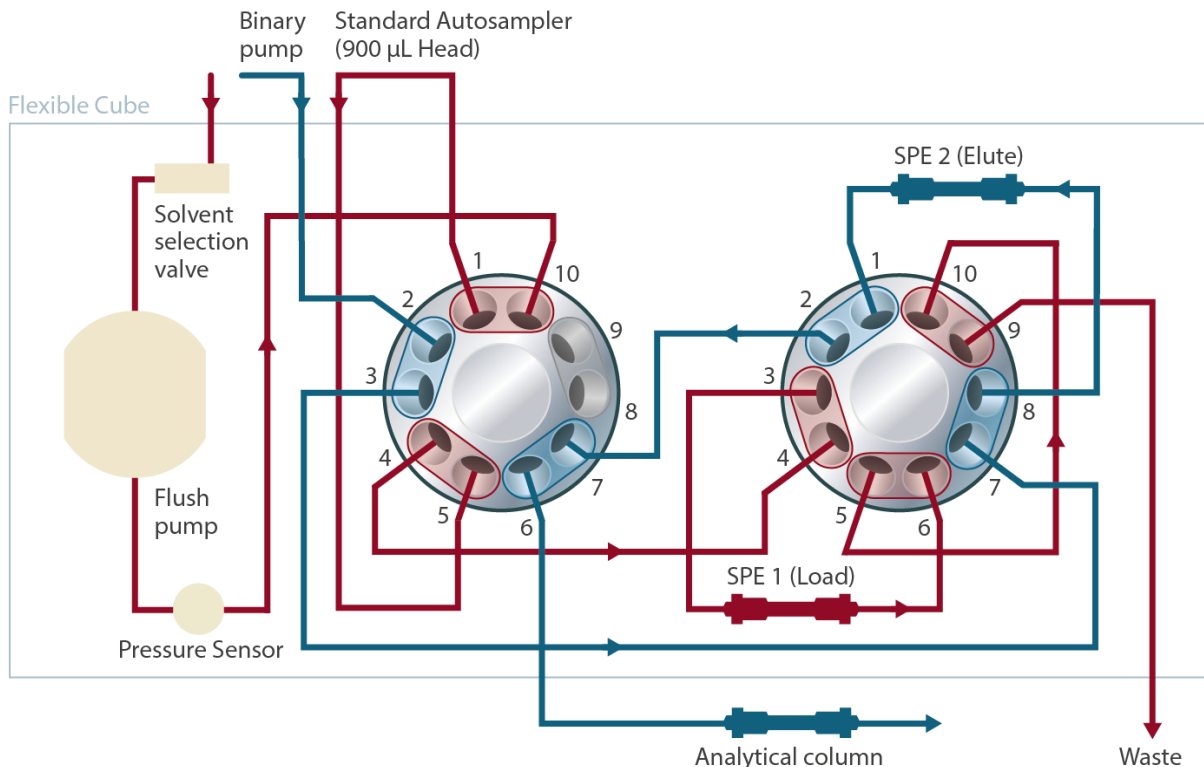


Figure 3 Valve A in Position 2 and Valve B in Position 1

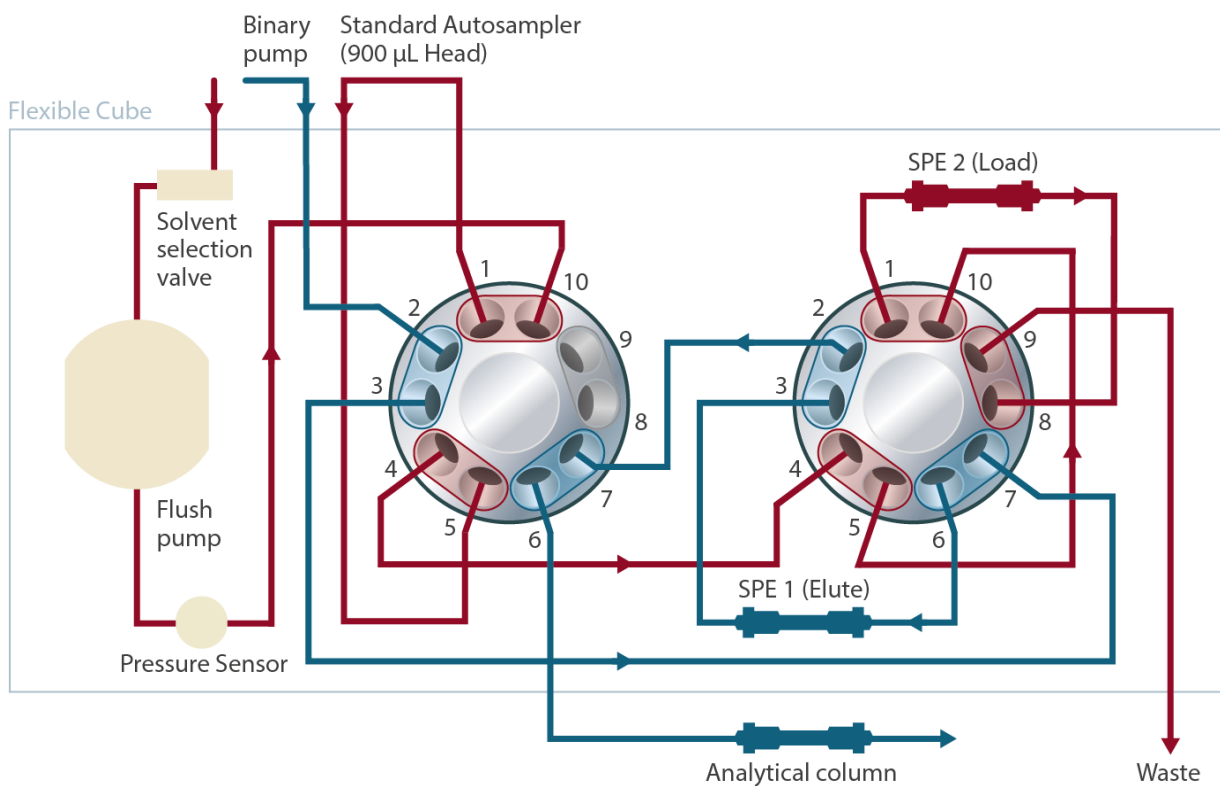


Figure 4 Valve A in Position 2 and Valve B in Position 2

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.

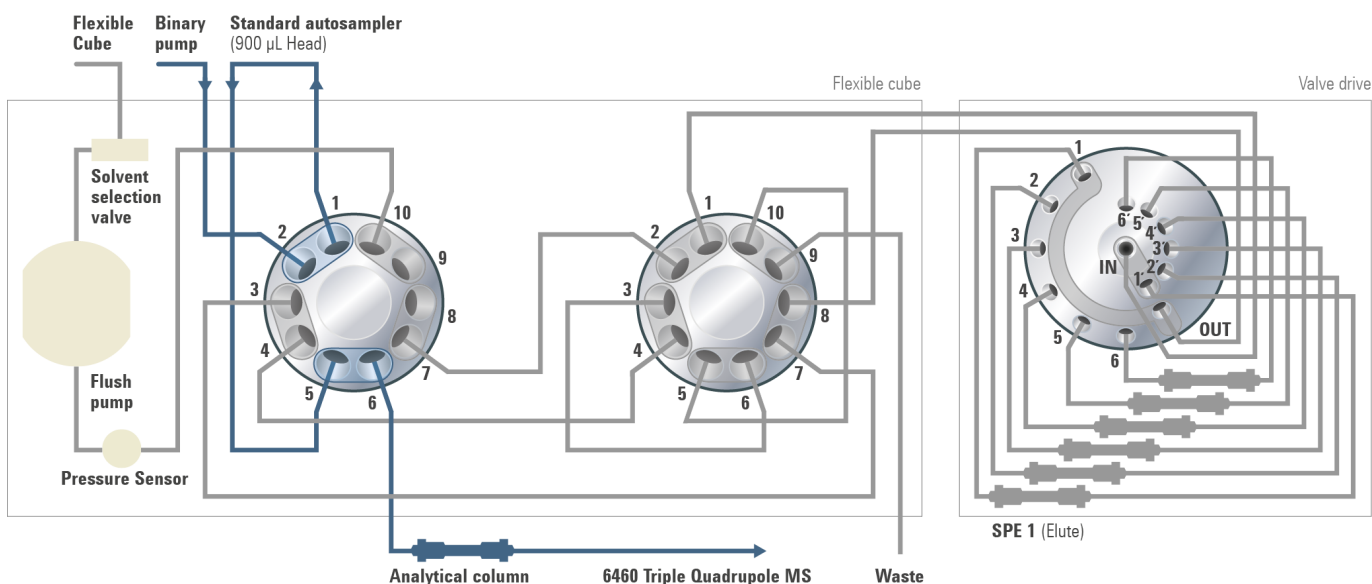


Figure 5 Flow path of the direct injection run

The Agilent 1290 Infinity Flexible Cube offers the possibility of direct injections and online SPE. In [Figure 5](#) on page 5, the flow path of the direct injection run is shown. 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 ([Figure 6](#) on page 6). It is recommended to use the quarternary pump as flush pump. Also, the right 2ps/10pt valve can be switched between online SPE and bypass, for cleaning the system.

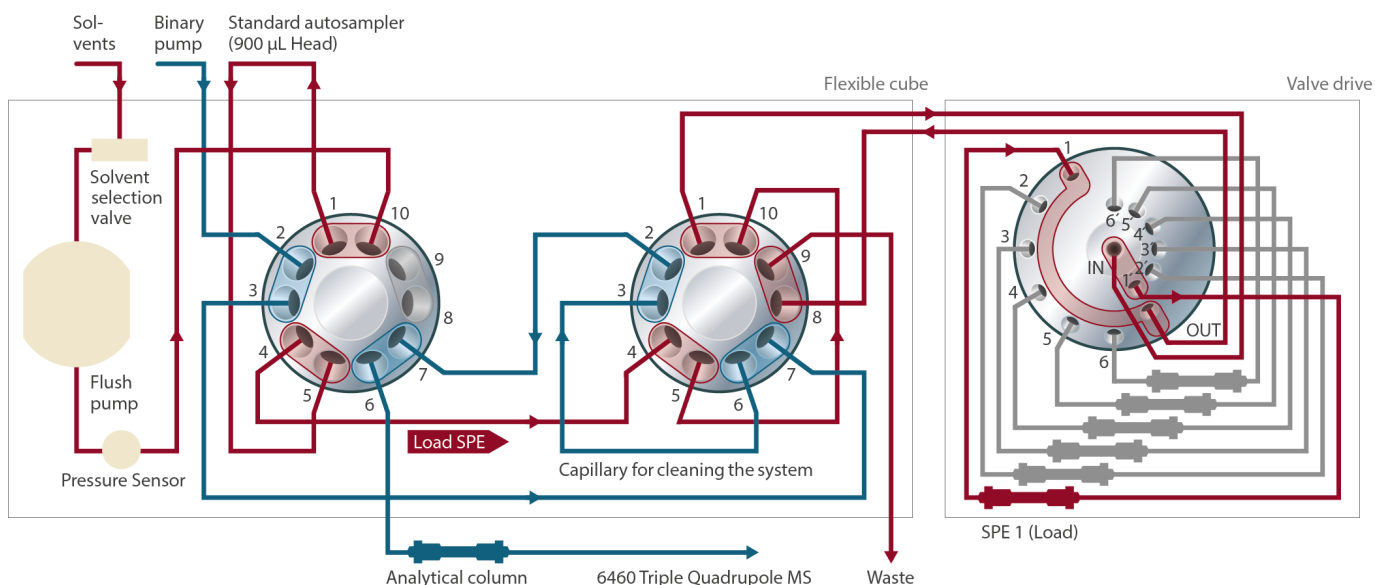


Figure 6 Flow path for online SPE

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.

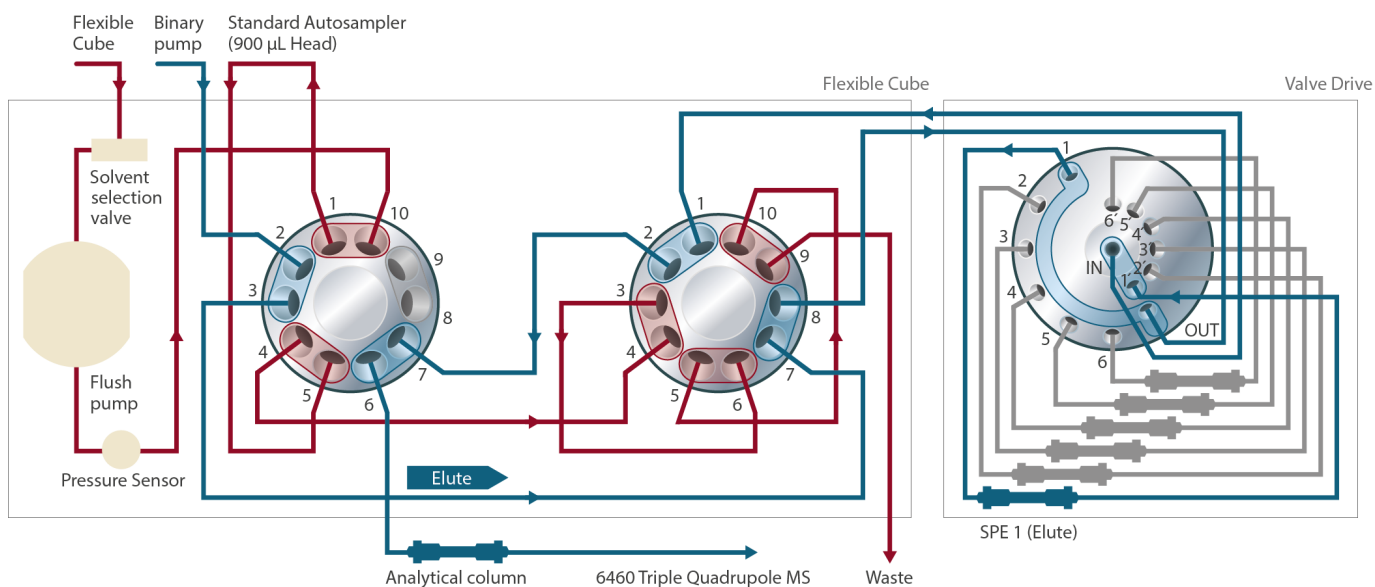


Figure 7 Flow path to elute the loaded cartridge

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

NOTE

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

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.

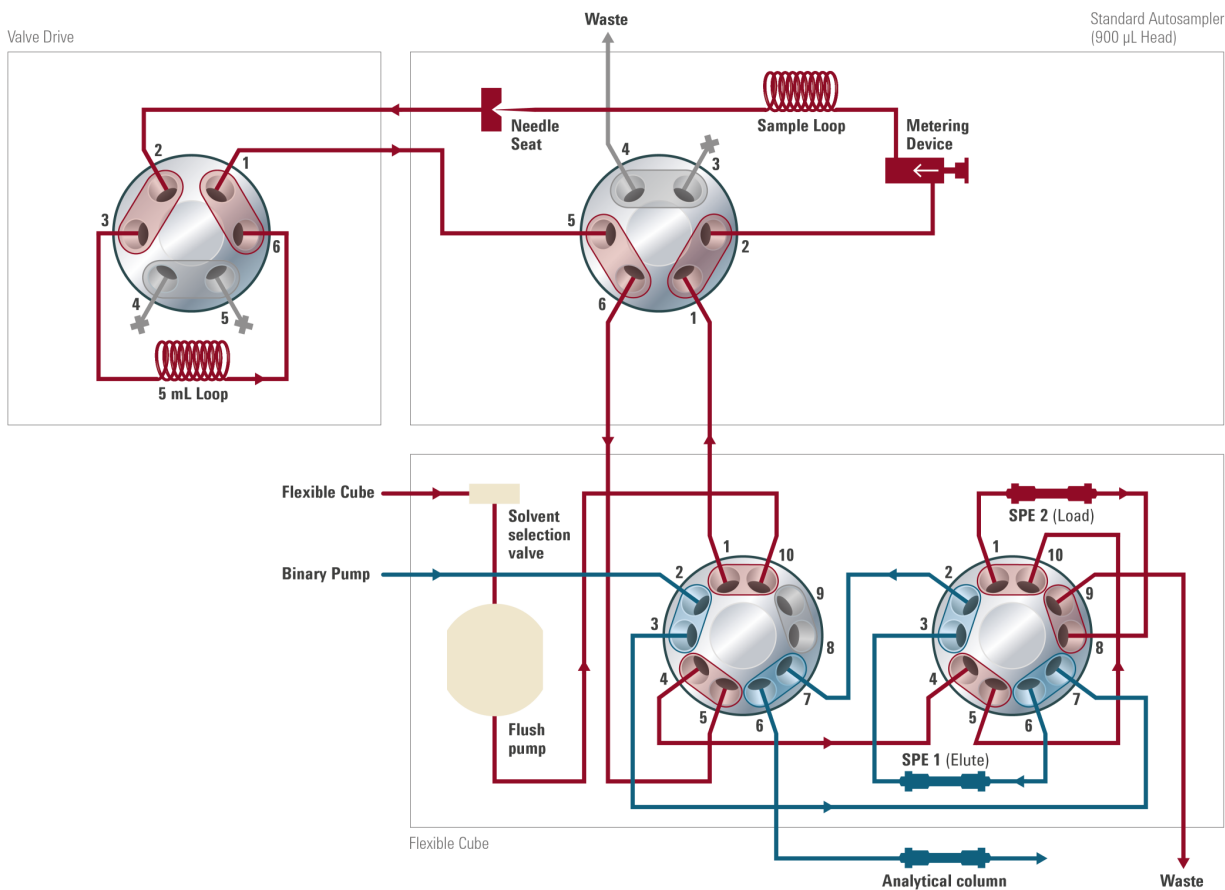


Figure 8 Switch diagram step 1

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.

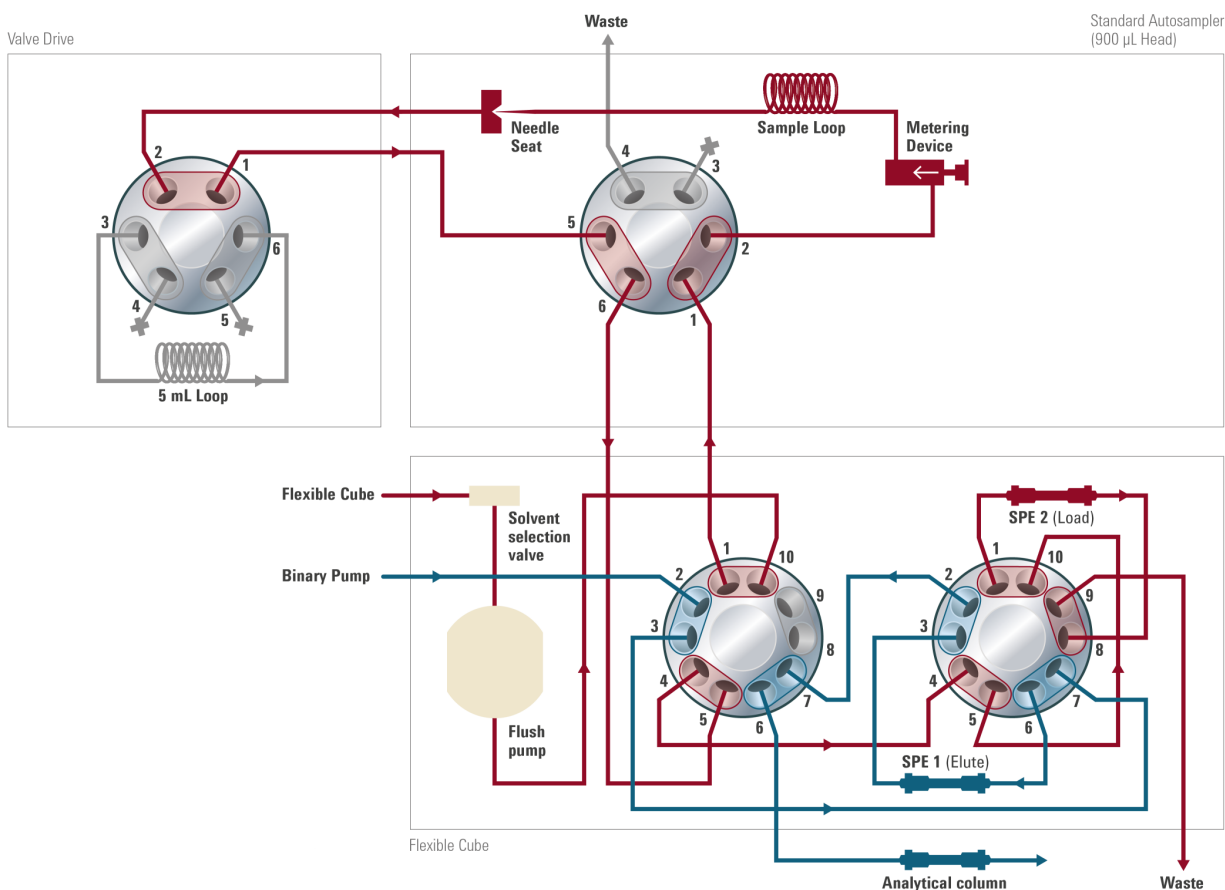


Figure 9 Switch diagram step 2

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

Delivery Checklist

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

G4742A Online SPE Starter Set

| p/n | Description |
|-----------|----------------------------------|
| 5067-4283 | 2ps/10pt valve head, 800 bar |
| 5067-4294 | Online SPE Capillary Starter Kit |

Online SPE Capillary Starter Kit (5067-4294)

| # | p/n | Description |
|---|-------------|--|
| 2 | 0890-1713 | Tube PTFE, 2 m |
| 2 | 0100-1516 | Fitting male PEEK, 2/pk |
| 1 | 5982-1271 | BE online PLRP-S 15 – 20 μ m 2.1 mm x 12.5 mm 3/pk |
| 1 | 5065-9963 | Capillary, 0.17 mm x 900 mm, ns/ns |
| 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 10/pk |
| 1 | G4742-90101 | Online SPE Solutions Technical Note |

G4744A Online SPE Direct Inject Kit

| p/n | Description |
|-----------|--|
| 5067-4283 | 2ps/10pt valve head, 800 bar |
| 5067-4295 | Online SPE Direct Inject Capillary Kit |

G4744B Online SPE Direct Inject Kit 1300 bar

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

Delivery Checklist

G4745A Online SPE High Volume Injection Kit

Online SPE Direct Inject Capillary Kit (5067-4295)

| # | p/n | Description |
|---|-------------|--|
| 2 | 5043-0277 | Plug Black Long 10-32 |
| 1 | 0100-1516 | Fitting male 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 10/pk |

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 |

Online SPE Multi Cartridge Capillary Kit (5067-4296)

| # | 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 | 2ps/6pt valve head, 800 bar |
| 5067-4242 | Online SPE High Volume Injection Capillary Kit |

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

| p/n | Description |
|-------------|---|
| G1312-87303 | Capillary ST 0.17 mm x 400 mm S/S |
| 5022-2184 | Union ZDV |
| 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, refer to the Agilent Information Center (AIC).

System Setup

The Online SPE System works in lots of different hardware configurations. Examples of two different Online SPE System setups are shown in [Figure 10](#) on page 12 and [Figure 11](#) on page 13 below.

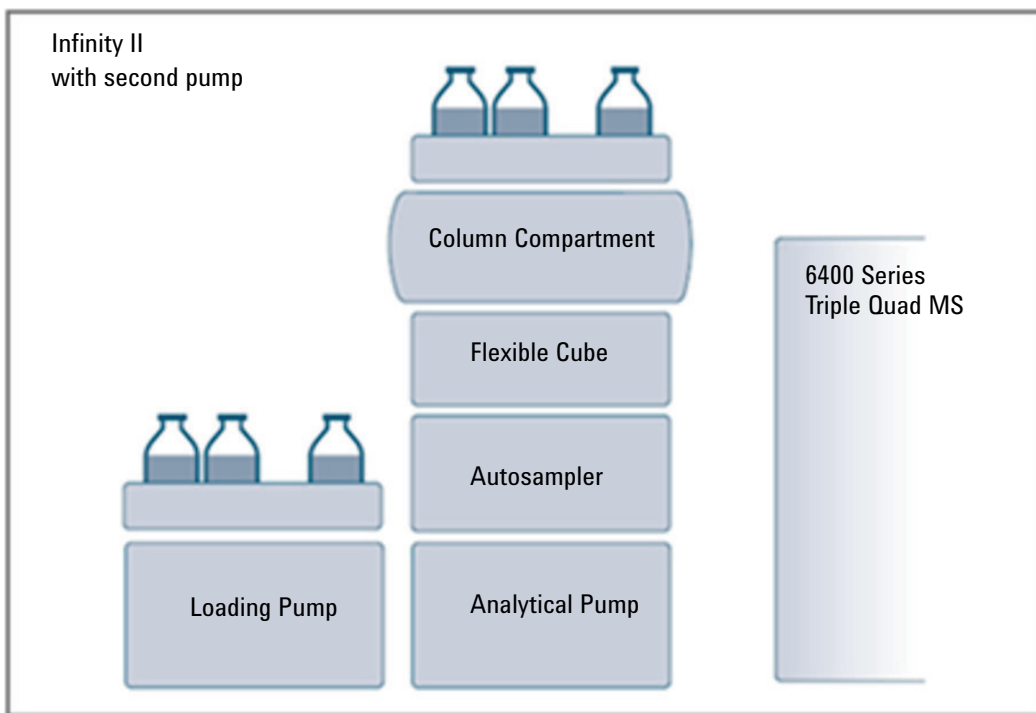


Figure 10 Recommended Online SPE System setup

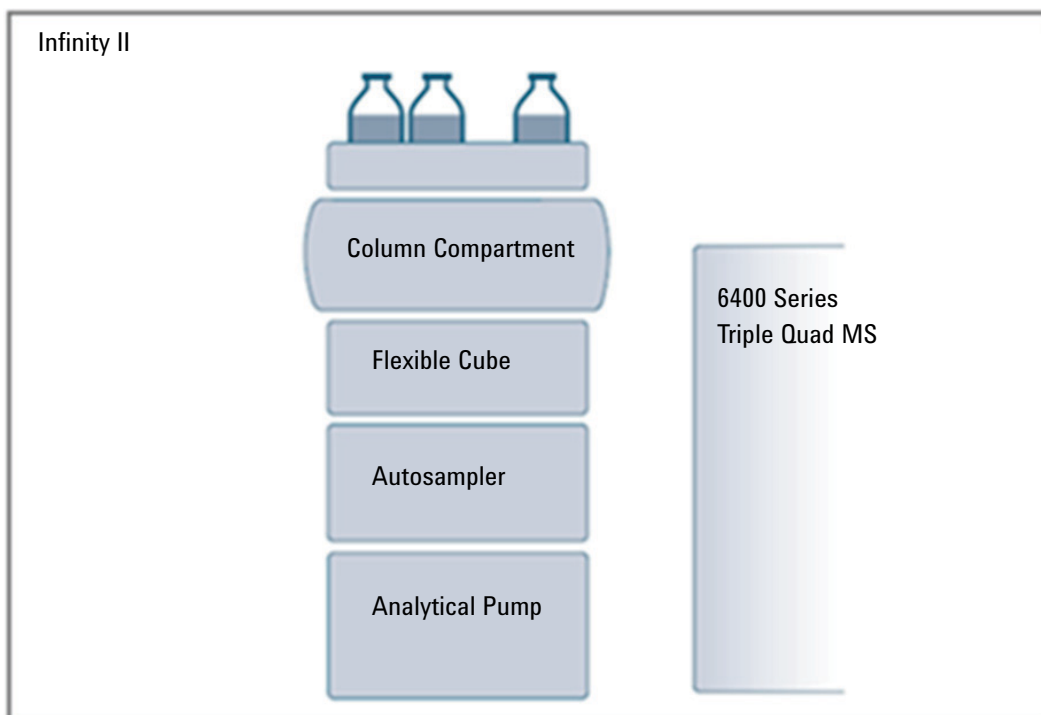


Figure 11 Legacy Online SPE System setup

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.

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.

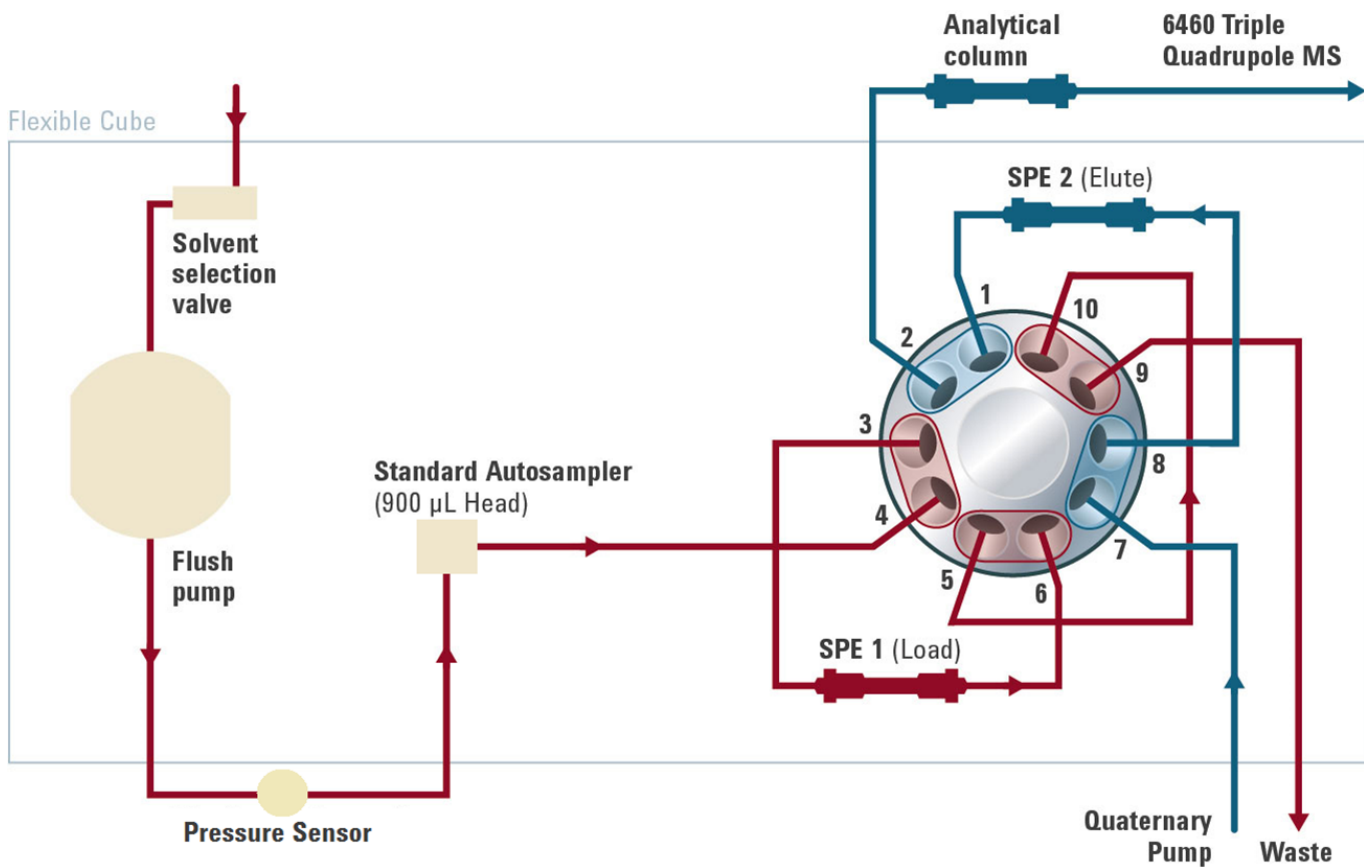


Figure 12 Flow path in an Online SPE Starter setup

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

| # | Part Description | Connection |
|----------|---|---|
| 5 | Capillary 0.25 mm x 150 mm, ns/ns (5500-1262) | Valve - cartridges (4x) Valve crossing (Port 5 – Port 10) |
| 1 | Capillary ST 0.12 mm x 340 mm S/S (G1316-87319) | Valve (port 2) - column use long fittings on valve |
| 1 | Capillary, 0.17 mm x 900 mm, ns/ns (5065-9963) | LC pump - valve (Port 7) |
| 2 | Capillary 0.17 mm x 700 mm, ns/ns (5500-1237) | Pressure Sensor - Autosampler Autosampler - Valve (port 4) |
| 1 | Capillary ST 0.25 mm x 250 mm (5065-9979) | Flush pump - pressure sensor (Use PEEK fitting for Flush pump) |
| 1 | Waste tubing, 2 m (0890-1713) | 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.

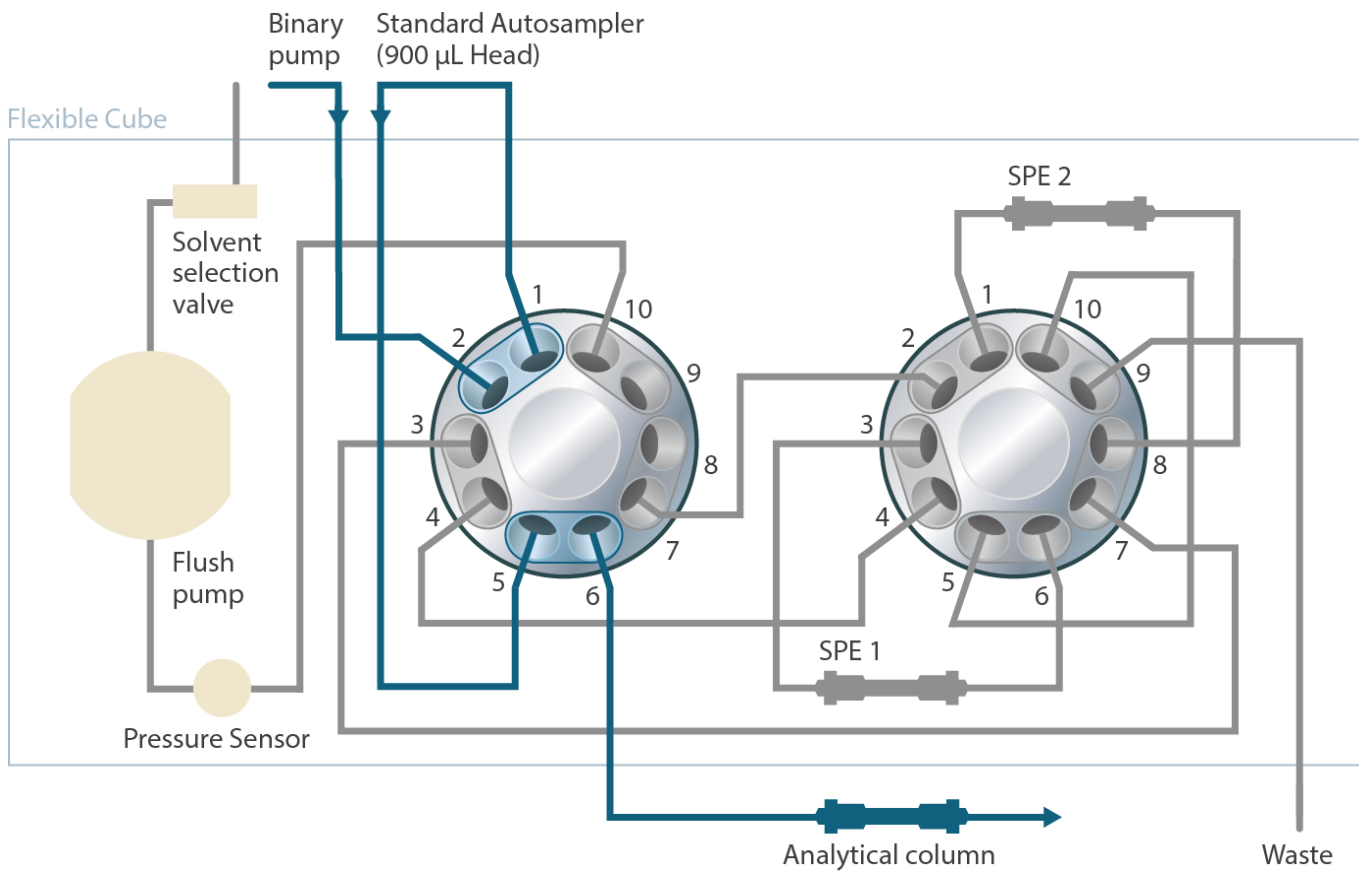


Figure 13 Direct Injection (Valve A in Position 1)

Use the capillaries and parts as described in [Table 2](#) on page 17

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)

| Part Description | Connection |
|---|---|
| Capillary, 0.25 mm x 280 mm no fittings (5022-6508) | Valve A / Port 4 - Valve B / Port 4 |
| Capillary, 0.12 mm x 280 mm (5021-1822) | Valve A / Port 3 - Valve B / Port 7 Valve A / Port 7 - Valve B / Port 2 |
| Capillary 0.25 mm x 150 mm, ns/ns (5500-1262) | Pressure Sensor - Valve A / Port 10 Valve to cartridges (4x) Valve crossing, Valve B (Port 5 – Port 10) |
| Capillary ST 0.12 mm x 340 mm S/S (G1316-87319) | Valve A (port 6) - column Autosampler – Valve A / Port 5 use long fittings on valve |
| Capillary, 0.17 mm x 900 mm, ns/ns (5065-9963) | LC pump to valve A (Port 2) |
| Capillary 0.17 mm x 700 mm, ns/ns (5500-1237) | Valve A (port 1) - Autosampler |
| Capillary ST 0.25 mm x 250 mm (5065-9979) | Flush pump - pressure sensor (Use PEEK fitting for Flush pump) |
| Waste tubing, 2 m (0890-1713) | valve B (port 9) |
| Plug Black Long 10-32 (5043-0277) | 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).

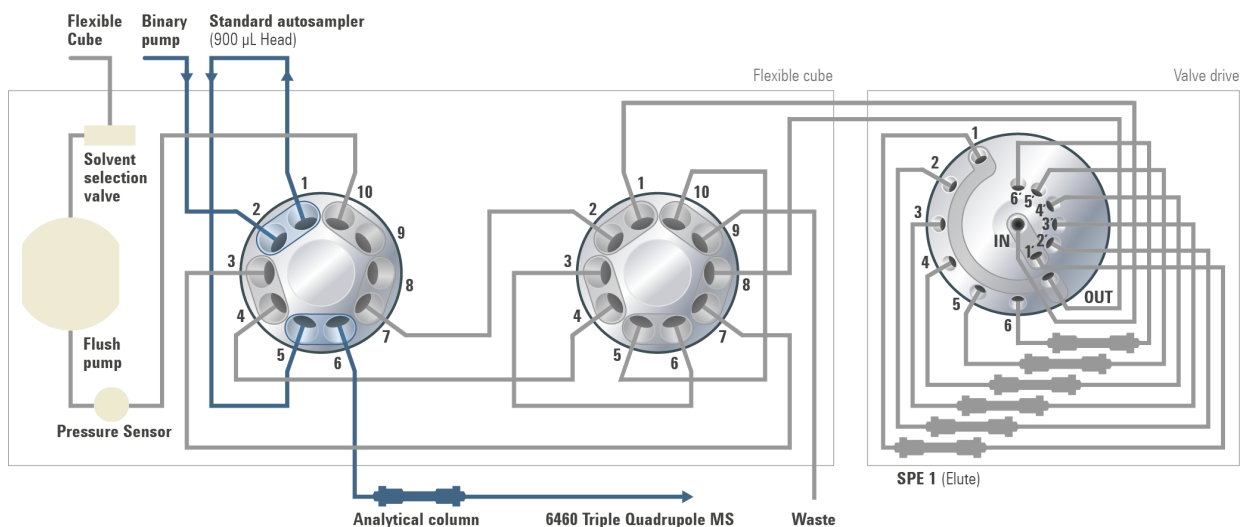


Figure 14 Flow path of the direct injection run

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

| Part Description | Connection |
|---|--|
| Capillary, 0.25 mm x 280 mm no fittings (5022-6508) | Valve A / Port 4 - Valve B / Port 4 |
| Capillary, 0.12 mm x 280 mm (5021-1822) | Valve A / Port 3 - Valve B / Port 7 Valve A / Port 7 - Valve B / Port 2 |
| Capillary 0.25 mm x 150 mm, ns/ns (5500-1262) | Pressure Sensor - Valve A / Port 10 Valve crossing, Valve B (Port 3 – Port 6) Valve crossing, Valve B (Port 5 – Port 10) |
| Capillary ST 0.12 mm x 340 mm S/S (G1316-87319) | Valve A (port 6) - column Autosampler – Valve A / Port 5 use long fittings on valve |
| Capillary, 0.17 mm x 900 mm, ns/ns (5065-9963) | LC pump - valve A (Port 2) |
| Capillary 0.17 mm x 700 mm, ns/ns (5500-1237) | Valve A (port 1) - Autosampler |
| Capillary ST 0.25 mm x 250 mm (5065-9979) | Flush pump - Pressure sensor (Use PEEK fitting for Flush pump) |
| Waste tubing, 2 m (0890-1713) | Valve B (port 9) |
| Plug Black Long 10-32 (5043-0277) | Valve A (port 8, port 9) |
| Capillary ST 0.17 mm x 280 mm SV/M (5067-5112) | Valve B (port 1) - Selection valve 'IN' port Selection valve 'OUT' port - Valve B (port 8) |
| Capillary ST 90 mm x 0.17 mm SW-1/16/M4 (5067-5109) | Cartridges - Selection valve |
| Capillary ST 0.17 mm x 700 mm SL/M (5067-5120) | 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.

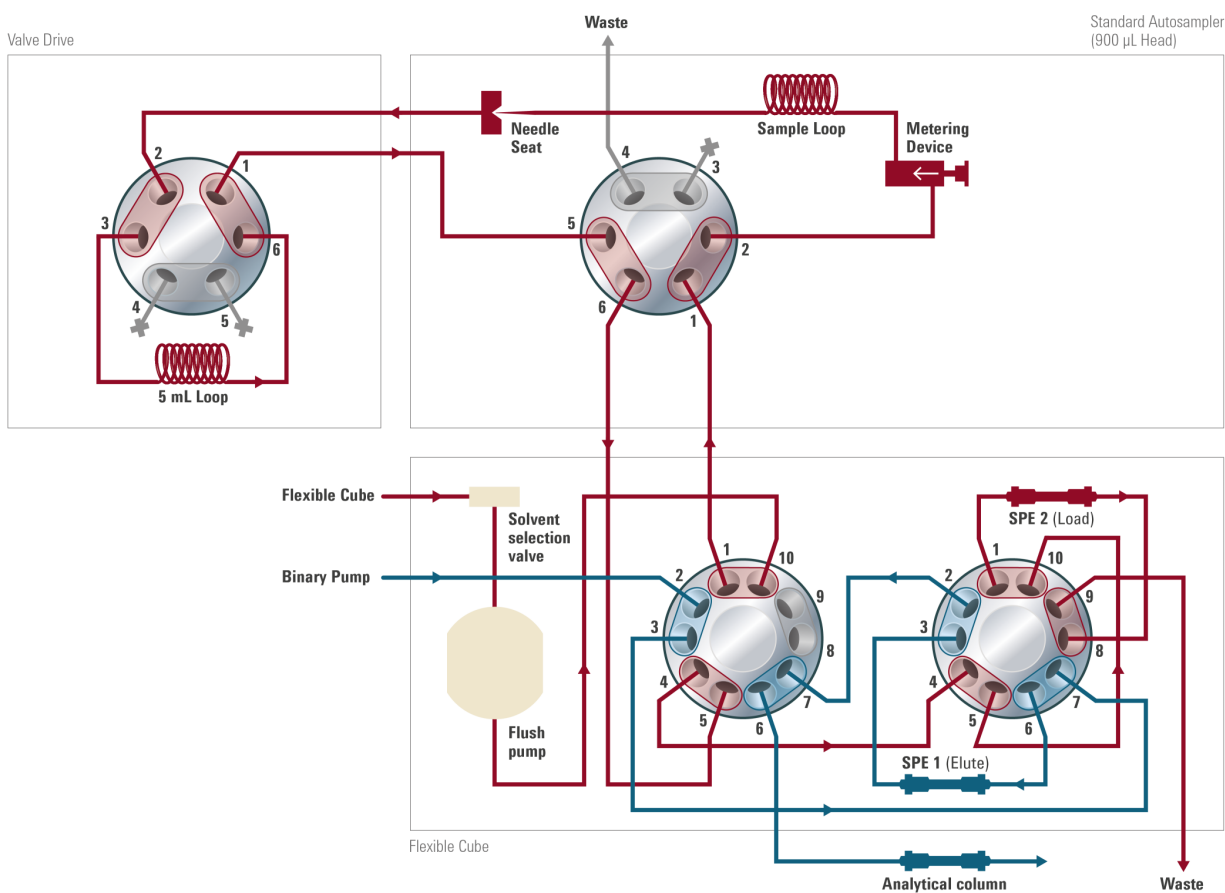


Figure 15 Flow path of High Volume Kit in combination with the Direct Inject Kit

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

| Part Description | Connection |
|---|---|
| Capillary ST 0.17 mm x 400 mm S/S (G1312-87303) | Needle Seat / Valve Port 2 Valve Port 1 / ALS Injection Valve Port 5 |
| Union, stand LC flow, no fitting (5022-2184) | Connector Needle seat to capillary G1312-87303 |
| Loop 5 mL (G2260-68711) | Valve Port 3 / Valve Port 6 |

Installing the Valve Heads

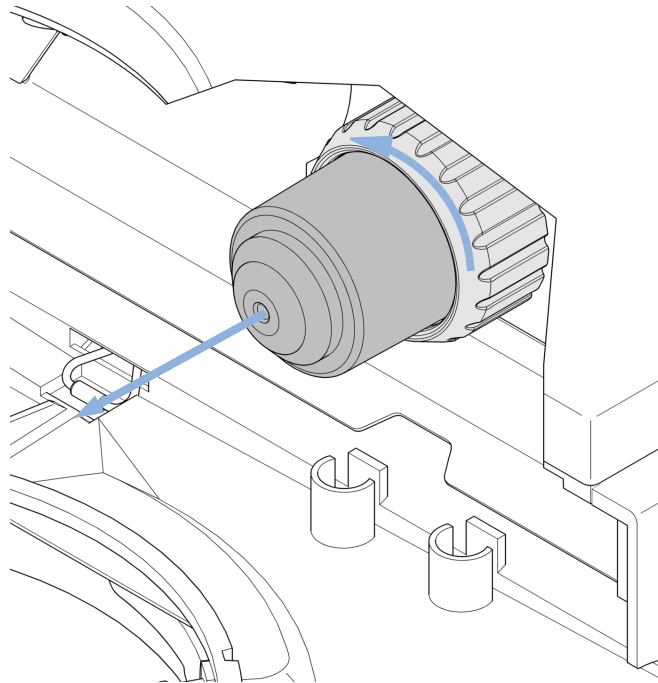
The valve drives are factory-installed in the Thermostatted Column Compartment, in the Flexible Cube, and in the 1290 Infinity Valve Drive. The valve heads are interchangeable and can be easily mounted.

At the first installation, the dummy valve has to be removed, see “[Removing the Valve Dummy](#)” on page 20. 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 pollutions. 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.

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.

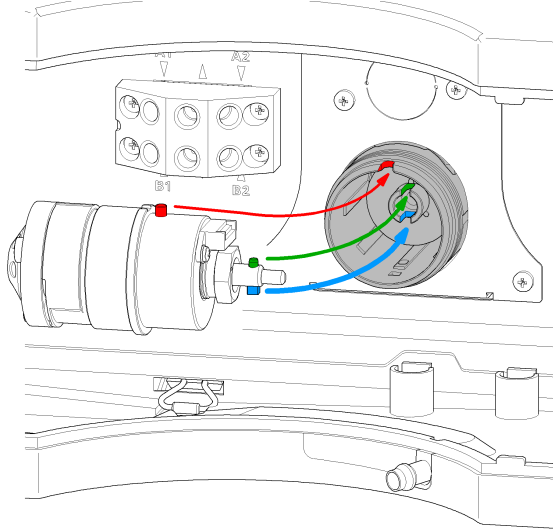
NOTE

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

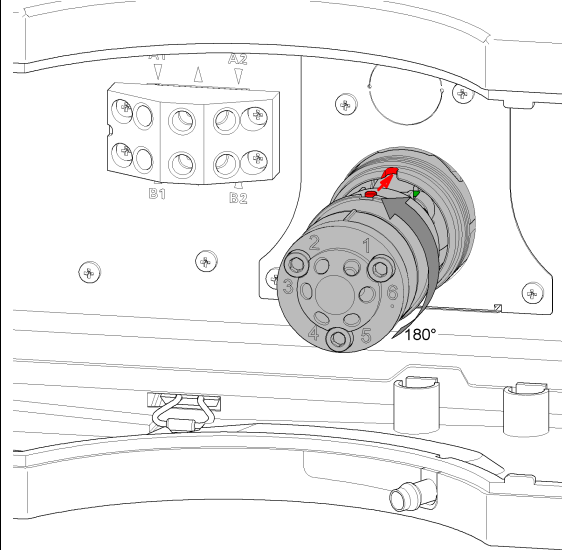
Installing the Valve Heads

Setting Up the SPE High Volume Injection Kit G4745A

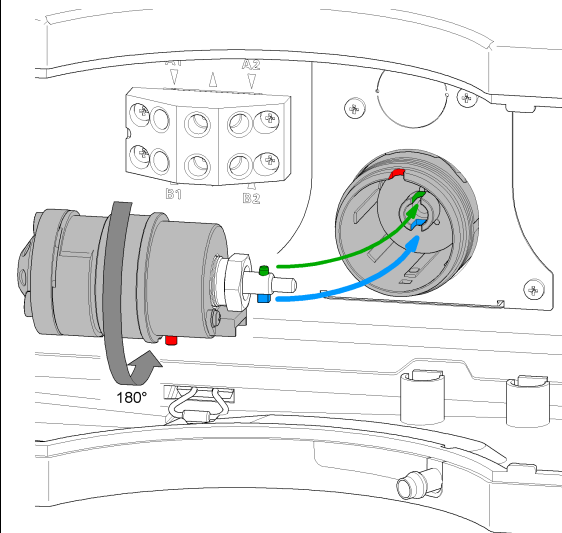
- 1** The two different pins (blue & green) on the valve shaft must fit into the corresponding grooves of the actuator axis. The outside pin (red) must fit into the outside groove.



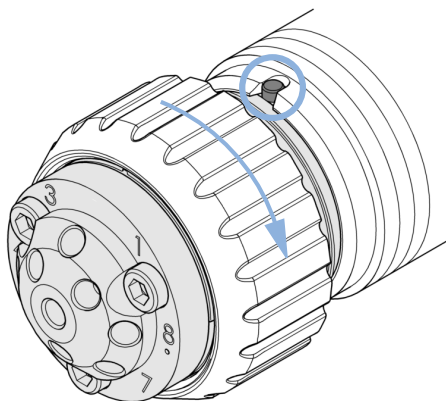
- 2** It can be possible that you have to turn the valve head until the outside pin completely locks in. While turning the valve you should feel some resistance from the drive.



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



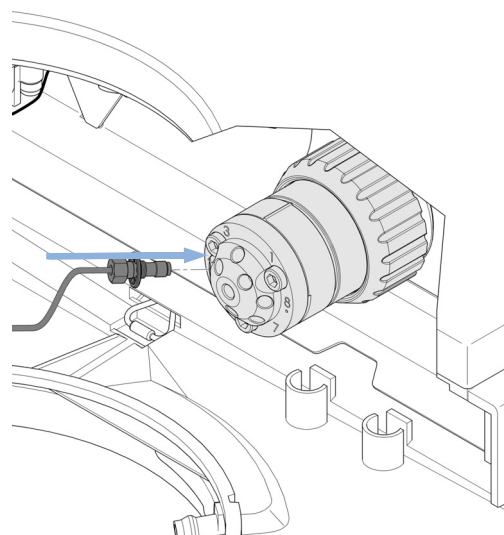
3 When the outer pin is locked into the groove, manually screw the nut onto the valve head.



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 12ps/13pt 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.

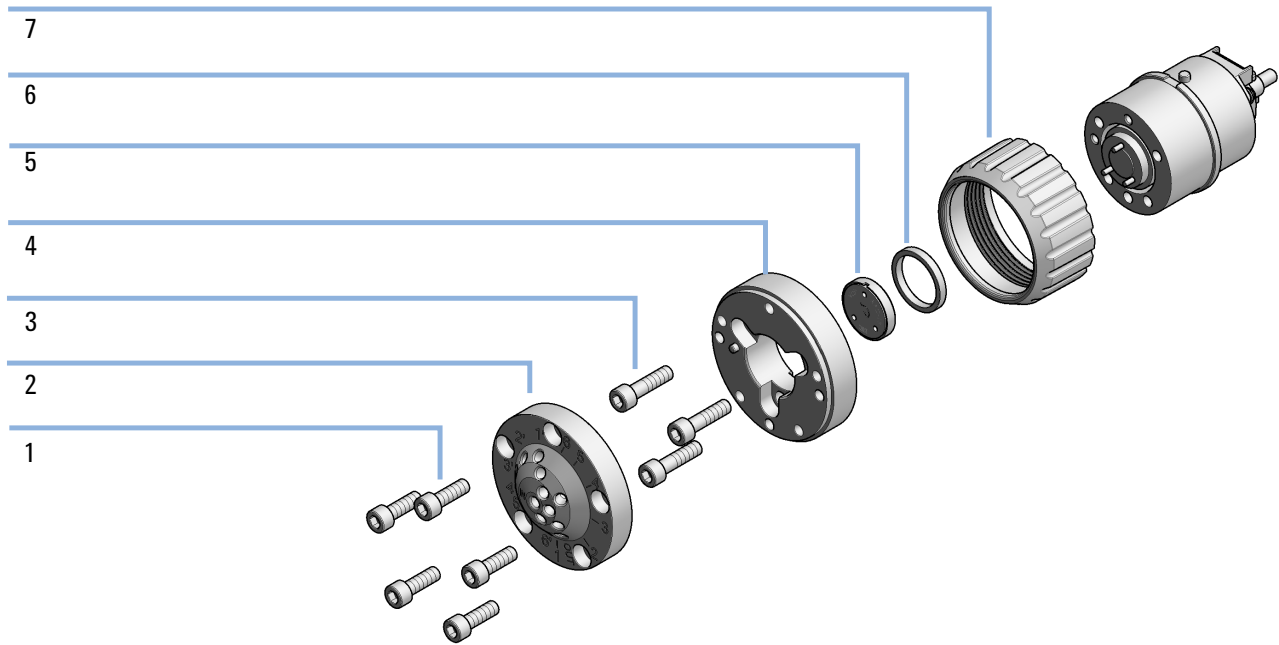


Figure 16 Valve Head Parts (example)

| | |
|---|--|
| 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) |

