

Use of the SFC-MS Splitter Kit G4309-68715

Technical Note

Using the SFC-MS Splitter Kit to couple an Agilent 1260 Infinity Analytical Supercritical Fluid Chromatography (SFC) System to the Ion Source of a Mass Spectrometer is beneficial, because a makeup flow can easily be added to enhance ionization, eluting peaks are not dispersed, and overloading the ion source caused by high flow rates can be avoided. This note describes the use and plumbing of the splitter kit.



Parts

Item	p/n	Description
1		Splitter assembly
2	5067-5939	Restriction capillary 50µm ID x 1m
3	5067-5941	Restriction capillary 50µm ID x 0.5m
4	0100-1516	1/16 in finger-tight PEEK fitting, 2/pk
5	5067-5403	UHP-FF fitting
6	5043-0915	Mounting tool for fitting
7	5065-9963 (2x)	Flex capillary 0.17x900mm
8	5021-1822	Flexible capillary 0.12x280mm
9	0100-0969	Tee, Zero 1/16"SS Low dead volume
10	5062-2418	1/16in Fittings and Ferrules, 10/Pk
11	0890-1763	PEEK Tubing 0.18x1500mm yellow

Plumbing of the splitter kit

The splitter kit consists of 4 major parts. The makeup flow enters the first adaptor block with the high pressure filter and then it is guided to adaptor block 2 which contains a check valve (Part No: G1312-60067), which prevents back flush of CO₂ to the pump. In the double Tee block makeup flow and analytical flow are first combined, then split to the Back Pressure Regulator (BPR) and via the stainless steel restriction capillary (50µm ID x 1m or 50µm ID x 0.5m) to the ion source of the mass spectrometer.

- 1 Use the removable UHP-FF fitting 5067-5403 to connect the restriction capillary to the splitter and the finger-tight PEEK fitting to connect to the MS-sprayer.

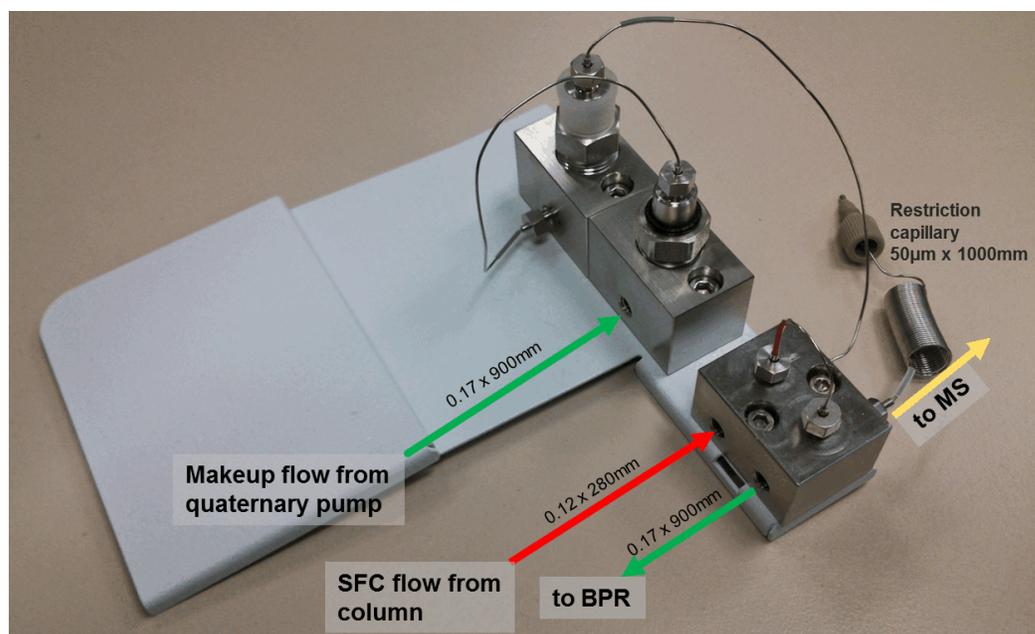


Figure 1 Plumbing of the splitter kit

Position of the splitter kit in the SFC-LC stack

- 1 Place the splitter between two modules on the right front corner. Typically, this is placed on top of the UV (DAD) detector. When a UV detector is not present, locate on the TCC containing the outlet column selection valve. Make sure, the position is close enough to reach the ion source with the stainless steel restriction capillary.

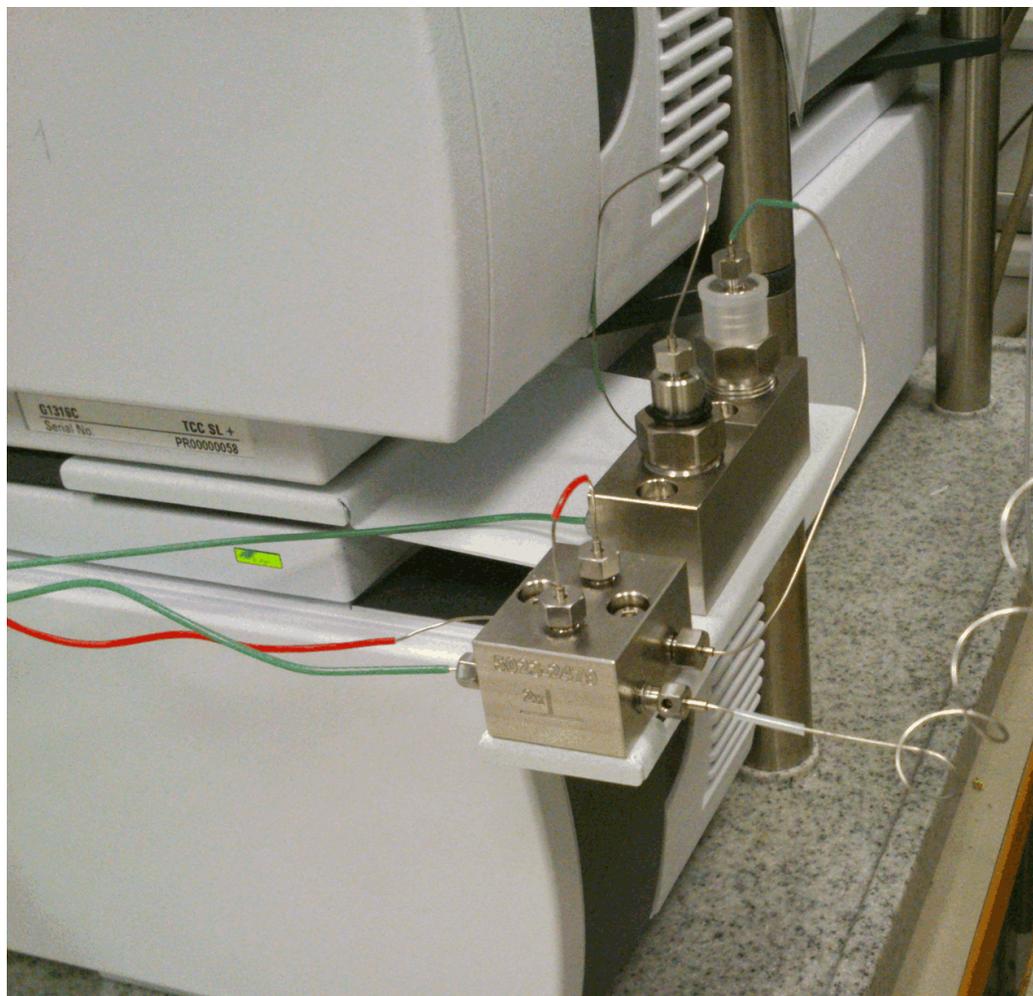


Figure 2 Position of the splitter in the LC stack

CAUTION

Fragile restriction capillary

Over bending of the restriction capillary may cause damages

→ Do not over bend the restriction capillary when attaching it to the ion source, it is very fragile and easily breaks. Especially when bent behind the steel sleeves at the ends.

- 2 Use the removable UHP-FF fitting 5067-5403 to connect the restriction capillary to the splitter block. The mounting tool 5043-0915 for this fitting is part of the Splitter Kit. To connect the low pressure side of the restriction capillary to the MS-sprayer, the Swagelok 1.6 mm PEEK finger-tight fitting SPF 0100-1516 can be used.



Figure 3 Position of the splitter in the LC stack

Estimated flow through the split capillary

The flow through the split capillary is mainly independent from the primary flow. Split flow, and thus split ratio, depend on the resistance of the split capillary (ID and length), solvent viscosity and Back Pressure Regulator setting. Some variance in splitter flow rates is expected between individual splitters based on each splitter's actual resistance.

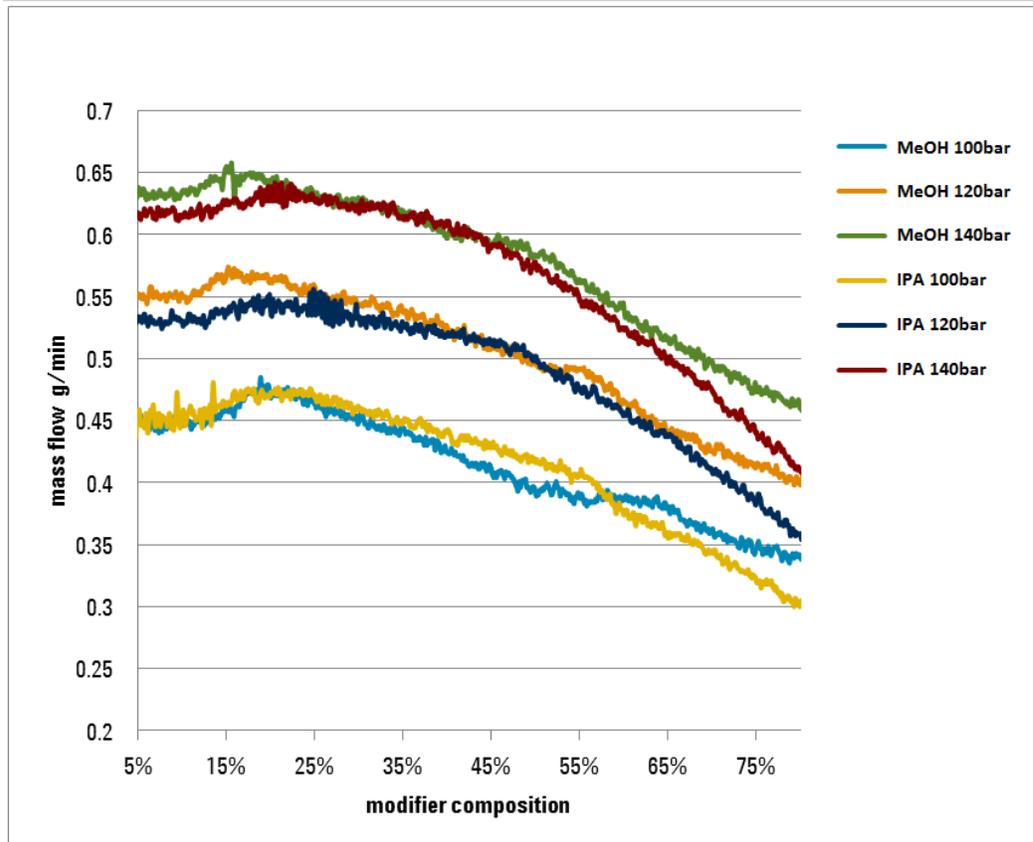


Figure 4 Split mass flow versus total composition and solvent type

As shown in [Figure 4](#) on page 6, the total composition of all fluids (column effluent plus any additional makeup flow) through the splitter capillary affects the split flow. Increases in back pressure increase the flow rate through the splitter capillary. Increasing liquid solvent, from both makeup flow and mobile phase composition will decrease the split capillary flow rate.

Representative differences between solvent types (methanol and isopropanol shown) are more predominant at the higher total composition where liquid solvent viscosity has a more pronounced effect.

Figure 5 on page 7 shows the split flow for three 50µm ID steel capillaries of different length, 50, 75 and 100cm, for different BPR settings. Solvent was CO₂ with ~10% Methanol. So, to a certain extent, the split flow can be adjusted by changing the back pressure. But eventually, selectivity of the column and retention times may also change with pressure. This data was generated with a primary flow of 1200µl/min CO₂ + 2% MeOH and 100µl/min Methanol as makeup flow.

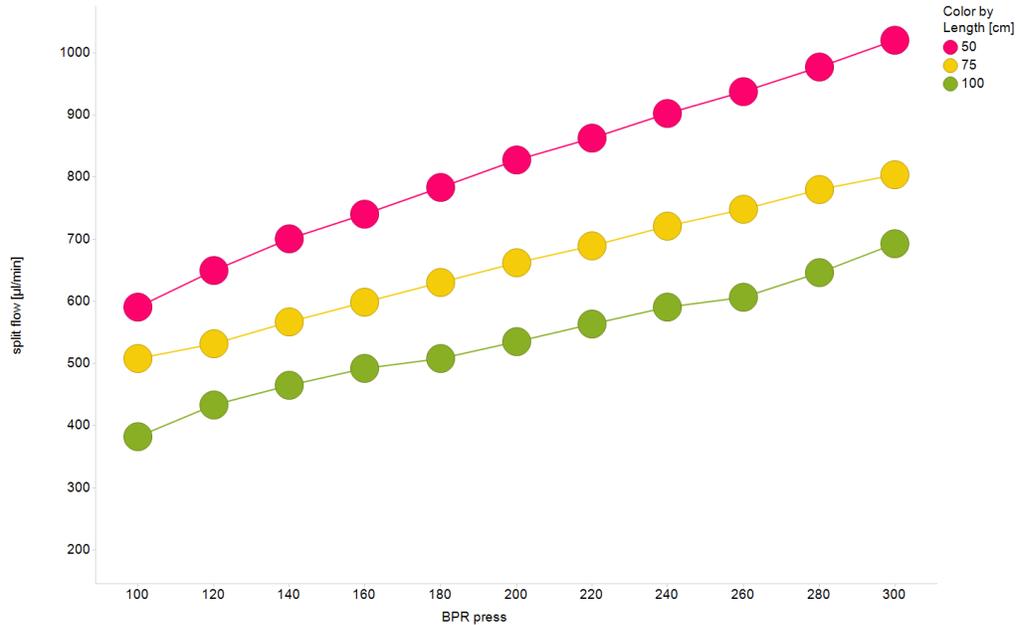


Figure 5 Split flow versus set BPR pressure

Plumbing for full flow connection to the MS

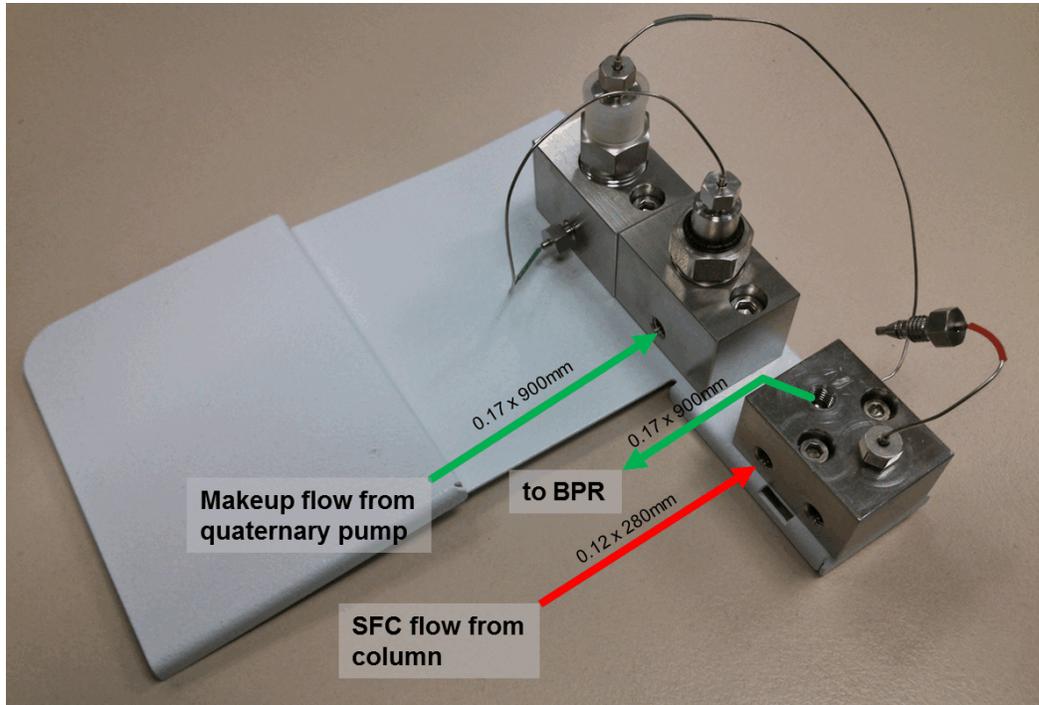


Figure 6 Plumbing of the splitter kit for full flow connection to the MS

- 1 For full flow connection to the MS source, only one of the splitter tees is used to combine the makeup flow. For this operation mode, unscrew the connection capillary of the double tee as shown in [Figure 6](#) on page 8 and attach the BPR connection capillary instead.
- 2 Use the PEEK Tubing 0.18x1500mm yellow, 0890-1763 to connect the BPR to the ion source of the mass spectrometer. To minimize peak broadening due to dispersion, shorten the PEEK Tubing to the necessary length.



G4309-90130

Part Number: G4309-90130

Edition: 03/2015
Printed in Germany

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