Setting up SilFlow® for backflush in your GC

What is backflush and why use it?

The backflush system eliminates the need to 'bake' heavy sample fractions off the capillary column. Oils, tars and other semivolatile matter can be flushed back out of the injection port while the oven remains at a relatively low temperature. This increases column lifetime dramatically.

Run times will also be significantly shorter because the system allows the run to be terminated as soon as the compounds of interest have eluted from the column, with everything else being flushed out the split vent. This results in shorter cycle times, lower maximum oven temperatures, longer column life and no carryover between samples.

Backflushing saves time and keeps detectors clean. Using the SilFlow® three or four port microchannel device (MCD) (Figure 1) for backflushing through a precolumn before the midpoint auxillary automated pressure control line, keeps the analytical column in pristine condition as chemicals of low volatility are backflushed before they even reach the analytical column.

SilFlow features

SilFlow, is an innovation in design and fabrication resulting in a highly efficient and reliable microchannel device for gas chromatography.

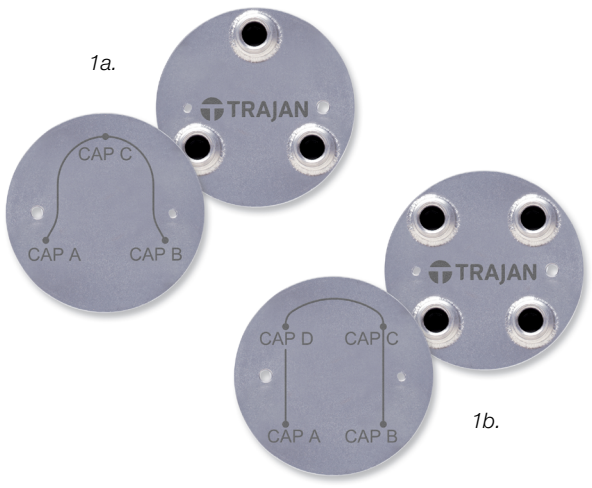

Figure 1a. SilFlow GC 3 port splitter.
Figure 1b. SilFlow GC 4 port splitter.

- Chemically deactivated internal channels result in analysis with enhanced quantitative accuracy and high reproducibility. When incorporated into the GC chromatography system there is no impact on routine chromatography.
- Low dead volume connections - SilFlow FingerTite metal ferrules result in a reliable zero dead volume connection, giving you optimized peak shapes.
- Excellent operational stability - thermal lag is negligible as the device tracks the oven temperature up to 20°C/min. The design alleviates cold spots and sample condensation. Maximum temperature – No practical temperature limit. Limited only by the temperature rating of the GC column being used (≤ 420°C).
- Easy to install and leak free, each kit incorporates SilFlow FingerTite fittings that are easy to set up and can be tightened using finger force to achieve a perfect, reliable seal, even for the most sensitive MS systems – no wrenches are required.

Using SilFlow for backflush

This article demonstrates three different configurations that can be used for backflushing using SilFlow. How to program a backflush run will be outlined, then the options for a pre-column, postcolumn, or dual detector set up will be discussed.

Programming a backflush run

Backflushing can be achieved by the following sequence:

1. Calculate the correct midpoint pressure required to give the desired average linear velocity in the analytical column using a GC flow and pressure software application. Enter the analytical column dimensions, temperature and desired linear velocity to calculate the midpoint pressure.
2. Turn off the auxiliary carrier gas supply to the midpoint and adjust the injection port pressure until the midpoint has reached the required pressure to give the desired average linear velocity in the analytical column.
3. With the auxiliary pressure (i.e. midpoint) off, perform a run without backflushing and estimate from the chromatogram the time that you require

to backflush - this estimated time is when the last analyte of interest passes the endpoint.

4. To backflush, the auxiliary pressure should be turned down until all required analytes have reached the detector, at which time it must be turned on to provide pressure to the analytical column to backflush to the injector and out the split vent. The head pressure must be switched to the lowest possible value the system is capable of performing when the backflush commences. The injector must be in split mode.

Example 1 - Backflush experiment using a 3 port SilFlow MCD – pre-column

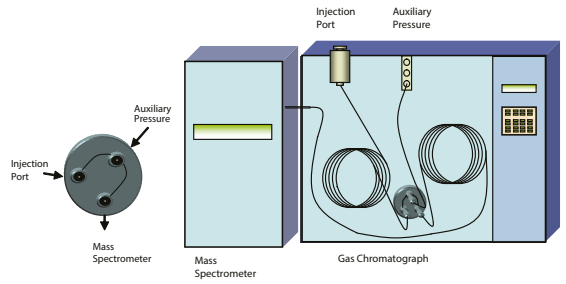

Figure 2. Diagram of backflush system with gas chromatograph, mass spectrometer and auxiliary automated pressure control.

Conditions

MCD part number	123722
Oven	
Initial temperature	40°C for 2 min
Rate	20°C/min
Final temperature	300°C for 10 min
Run time	25 min
Injector	
Injection mode	Split (100:1), 280°C
Carrier gas	He, 17.4 psi
Detector	**MS**
MS solvent delay	2.6 min
MS transfer line temperature	280°C
Scan parameters	Low mass: 50.0, High mass: 650.0
MS quad temperature	150°C
MS source temperature	230°C
Backflush device	**3 port SilFlow MCD**
Analytical column (MCD to MS)	BPX5 30 m x 0.25 mm x 0.25 µm
MCD midpoint to auxiliary pressure connection	0.25 mm deactivated fused silica
Pre column (Injector to MCD)	BPX5 10 m x 0.25 mm x 0.25 µm
Carrier gas program	
Carrier gas	He
Backflush mode	On
Agilent column program	
Initial pressure	17.4 psi for 6 min
Ramp pressure	150 psi/min to 0.01 psi for 19 min
Agilent auxiliary control	10.4 psi for 20 min
Backflush mode	Off
Agilent column program	
Pressure	17.4 psi for 20 min
Agilent auxiliary control	10.4 psi for 20 min
Injection	Alkane mix in pentane

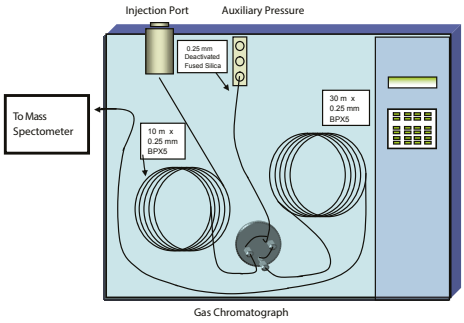

Figure 3. Example of a backflush system with capillary tubing and analytical column details.

Figure 3 - This system consists of a midpoint Auxillary EPC (electric pressure control) line to a three port MCD, a 10 m pre SilFlow column, and a 30 m post SilFlow analytical column.

The objective was to exclude any alkane greater in mass than C11 reaching the analytical column and mass spectrometer.

The following trace (Figure 4), shows the MS chromatogram without backflushing.

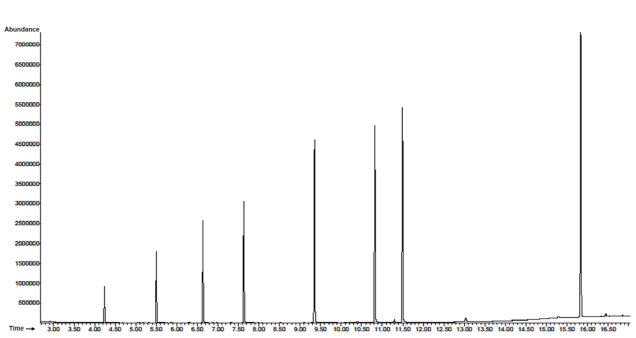

Figure 4. Alkane test mix response on MS pre-column backflush.

The same test mix was injected with the backflush initiated at 6 minutes (Figure 5). The backflush timing has to be estimated taking into account the type of carrier gas used, the type of analytes being tested, as well as the temperature, dimensions, and pressure of the pre-column and the analytical column. A few analyses will give an experimentally obtained optimum backflush time.

In the following example all of the less volatile alkanes have been backflushed out the injection port split vent.

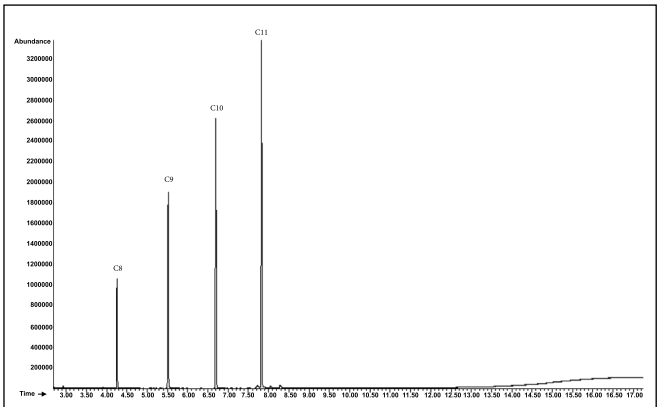

Figure 5. Alkane test mix with pre-column backflush initiated at 6 minutes.

Example 2 - Backflush experiment using a 3 port SilFlow MCD – post column

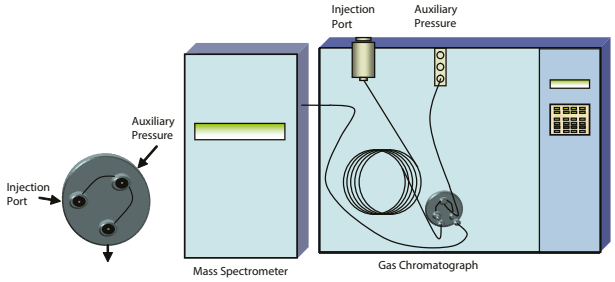

Figure 6. Diagram of backflush system with gas chromatograph, mass spectrometer, and auxiliary automated pressure control.

Figure 7- This system consists of a midpoint auxiliary EPC line, a pre-splitter 30 m analytical column, and a post splitter restrictor connected to the mass spectrometer.

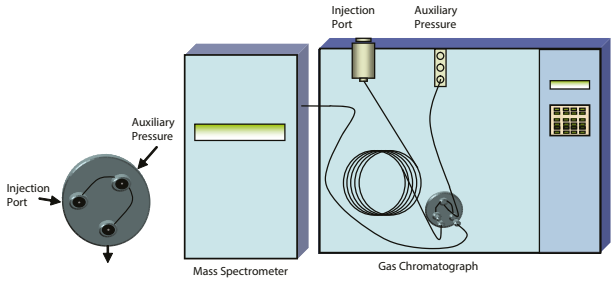

Figure 7. Example of a backflush system with capillary tubing and analytical column details.

The same objective of excluding any alkane greater in mass than C8 reaching the analytical column and mass spectrometer was tested.

Conditions

MCD part number	123722
Oven	
Initial temperature	40°C for 2 min
Rate	20°C/min
Final temperature	300°C for 10 min
Run time	25 min
Injector	
Injection mode	Split (100:1), 280°C
Carrier gas	He, 17.4 psi
Detector	**MS**
MS solvent delay	2.6 min
MS transfer line temperature	280°C
Scan parameters	Low mass: 50.0, High mass: 650.0
MS quad temperature	150°C
MS source temperature	230°C
Backflush device	**3 port SilFlow MCD**
Analytical column (MCD to MS)	BPX5 30 m x 0.25 mm x 0.25 µm
MCD midpoint to auxiliary pressure connection	0.25 mm deactivated fused silica
Pre column (Injector to MCD)	0.65 m x 0.11 mm deactivated fused silica column
Carrier gas program	
Carrier gas	He
Backflush mode	On
Agilent column program	
Initial pressure	17.4 psi for 6 min
Ramp pressure	150 psi/min to 0.01 psi for 19 min
Agilent auxiliary control	Set midpoint EPC pressure to 'off' (at start of run)
Agilent auxiliary runtime midpoint control	Set runtime event to start midpoint EPC pressure setting at 17.4 psi
Backflush mode	Off
Agilent column program	
Agilent auxiliary control	Leave midpoint pressure off and injection port pressure on throughout whole run
Injection	Alkane mix in pentane

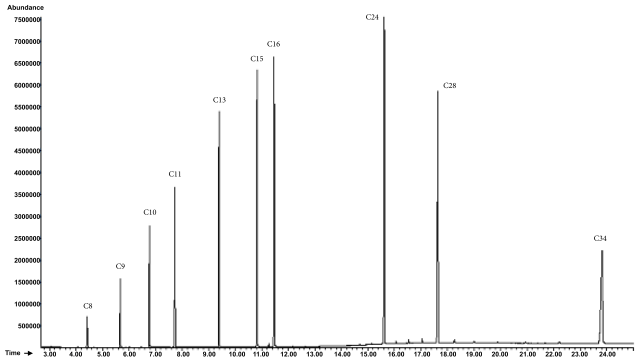

Figure 8. MS chromatogram without backflushing on alkane test mix post-column backflush.

Figure 8 trace shows the MS chromatogram without backflushing. The same test mix was injected with the backflush initiated at 6 minutes (Figure 9).

The backflush timing has to be estimated taking into account the type of carrier gas used, the type of analytes being tested, as well as the temperature, dimensions, and pressure of the precolumn and the analytical column. A few analyses will give an experimentally obtained optimum backflush time.

In the following example all of the less volatile alkanes have been backflushed out the injection port split vent.

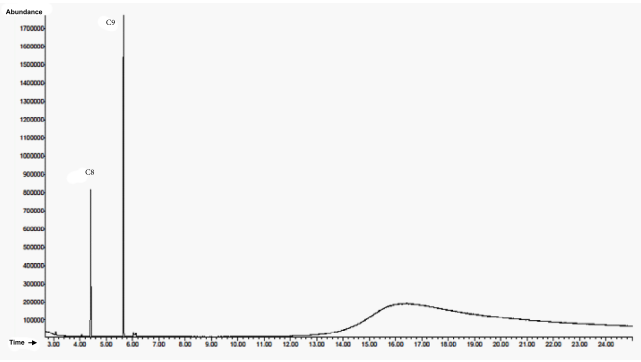

Figure 9. Alkane test mix response on MS backflush initiated at 6 minutes.

Example 3 - Backflush experiment with 4 port SilFlow MCD – two detector backflush

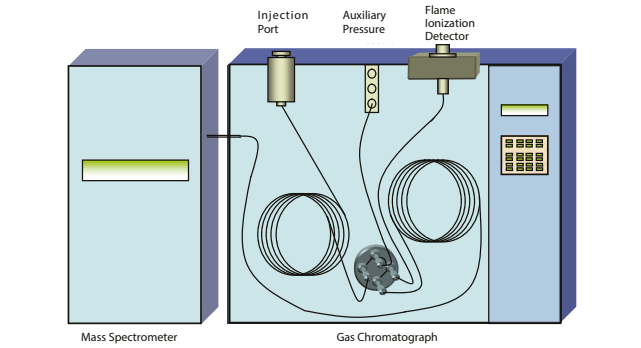

Figure 10. Diagram of backflush system with gas chromatograph, mass spectrometer, flame ionization detector, and auxiliary automated pressure control.

This system uses two detectors. A flame ionization detector (FID) and a mass spectrometer (MS). A 30 meter analytical column is connected to the midpoint four port SilFlow and the MS (see Figure 12).

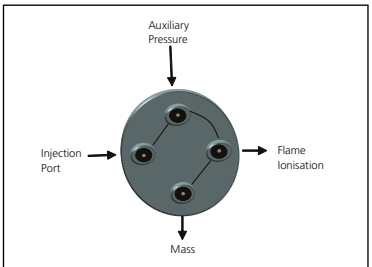

Figure 11. Four port SilFlow set up.

Conditions

MCD part number	123722
Oven	
Initial temperature	40°C for 2 min
Rate	20°C/min
Final temperature	300°C for 10 min
Run time	25 min
Injector	
Injection mode	Split (100:1), 280°C
Carrier gas	He, 17.4 psi
Detector	**MS**
FID	
Temperature	400°C
Gas flows	Hydrogen 40.0 mL/min, Air 450 mL/min, Nitrogen make up flow 45 mL/min
MS	
Solvent delay	2.6 min
Transfer line temperature	280°C
Scan parameters	Low mass: 50.0, High mass: 650.0
MS quad temperature	150°C
MS source temperature	230°C
Backflush device	**4 Port SilFlow MCD**
Analytical column (MCD to MS)	BPX5 30 m x 0.25 mm x 0.25 µm
MCD midpoint to auxiliary pressure connection	0.25 mm deactivated fused silica
MCD to FID connection restrictor	0.65 m x 0.11 mm deactivated fused silica column
Injection port to MCD	BPX5 10 m x 0.25 mm x 0.25 µm
Carrier gas program	
Carrier gas	He
Backflush mode	On
Agilent column program	
Initial pressure	17.4 psi for 6 min
Ramp pressure	150 psi/min to 0.01 psi for 19 min
Agilent auxiliary control	10.4 psi for 20 min
Backflush mode	Off
Agilent column program	
Pressure	17.4 psi for 20 min
Agilent auxiliary control	10.4 psi for 20 min
Injection	Alkane mix in pentane

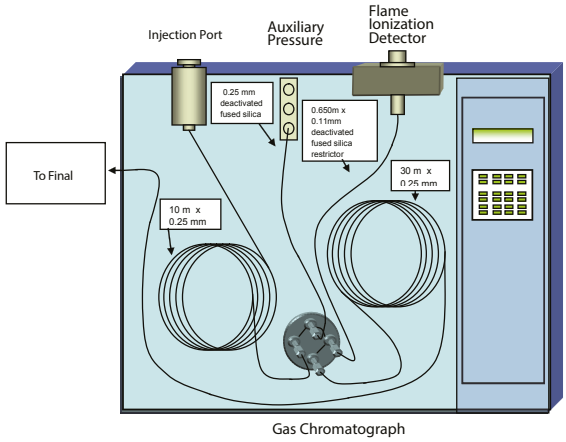

Figure 12. Example of a backflush system with capillary tubing and analytical column details.

A 10 meter pre-column is connected to the injection port and the midpoint SilFlow. The auxiliary EPC is connected to the midpoint. The flow rate (mL/min) from the midpoint is balanced between the FID and the MS detectors. A restrictor that has the appropriate dimensions to balance the flow is installed between the midpoint and the FID. This will ensure that good signals will be recorded on both detectors.

Prior to the backflush experiment a sample of alkanes was injected to establish the 'observed time' for the backflush event. The objective was to exclude any alkane greater in mass than C11 reaching the analytical column and mass spectrometer.

Figure 13 trace shows the MS chromatogram on top and the midpoint FID chromatogram underneath. While the most volatile alkane, octane, appears on the MS trace, the octane peak is missing from the FID trace due to the FID data acquisition only commencing once the MS solvent delay had finished. Based on the observed elution time at the FID, the backflush event was timed for 6 minutes.

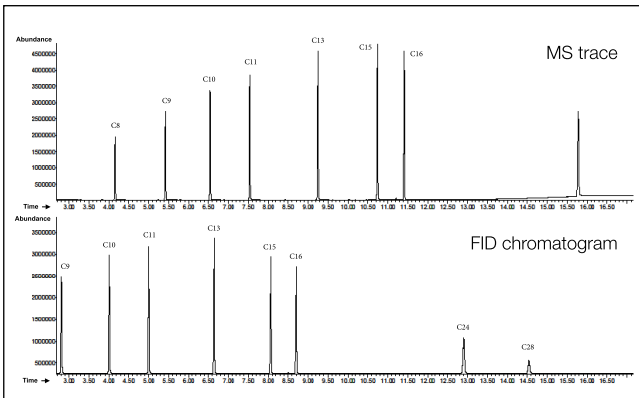

Figure 13. Alkane test mix response on MS and FID

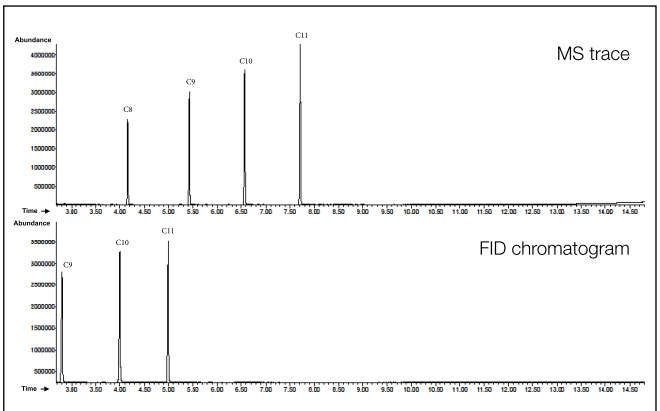

Figure 14. Alkane test mix response on MS and FID with backflush initiated at 6 minutes

The same test mix was injected with the backflush initiated at 6 minutes. In Figure 14, the top MS trace shows that the backflush was successful and no alkane greater in mass than C11 made it onto the analytical column – all of the less volatile alkanes have been backflushed out the injection port split vent.

Taking care of your SilFlow MCD

Tubing to the SilFlow MCD can be disconnected and reconnected many times without removing the pre-swaged ferrules. However, it is very important to inspect the capillary end carefully before reconnecting to the SilFlow MCD. Make sure the end of the capillary is intact. If the capillary does not have a clean square end, the column end has to be cut again and a new ferrule to be pre-swaged.

When it is not connected, protect the SilFlow MCD from particulates and dust getting into the internal channels. Make sure to block the bosses using either self-sealing nuts or pre-swaged ferrule to pieces of metal wire and appropriate FingerTite nuts.

Table 1. Part numbers for SilFlow 3 and 4 port MCD kits for backflushing.

Initial installation kits have all components required to get you started. Other items referenced in this article are also listed.

Part number	Part description and detail
Kits	
123722	SilFlow GC 3 port splitter kit, port A 0.25/0.32 mm OD, ports B and C 0.25/0.32 mm ID
123732	SilFlow GC 4 port splitter kit, port A 0.25/0.32 mm OD, ports B, C and D 0.25/0.32 mm ID
Tubing	
123751	2 m x 100 µm/363 µm VSD tubing
0624431	2 m x 250 µm/363 µm VSD tubing
0624459	2 m x 110 µm/310 µm VSD tubing
Ferrule	
123713	SilFlow ferrules 0.35 mm ID, PK10
GC capillary column	
054101	BPX5 30 m x 0.25 mm x 0.25 µm

Information and support

Visit www.trajanscimed.com or contact techsupport@trajanscimed.com

Specifications are subject to change without notice.