GC BackFlush
Installation & Operations Instructions

OVERVIEW

The gc-BackFlush system is a device that is used to shorten run times and eliminate carryover. It is designed to reverse the flow through the column and flush contents out the injection port instead of the detector. This results in much shorter run times than would normally be expected for the analysis because "baking off" heavy compounds is no longer necessary.

The BackFlush system consists of:

A control unit that monitors internal and downstream carrier gas pressures and supplies carrier gas to the outlet of the capillary column

A low dead volume tee piece that allows carrier gas to be switched in the forward and reverse directions

Mounting brackets, 1/16" stainless steel tubing, spare nuts and ferrules, this instruction booklet and fused silica restrictors to suit most column types.

INSTALLING THE BackFlush SYSTEM

1. Turn off the gas cylinder that supplies the carrier gas to the GC.

2. De-pressurize the line until the pressure gauge on the cylinder reads zero.

3. Locate the 1/8" copper tubing that supplies the carrier gas to the back of the GC and cut it with an appropriate tube cutter.

4. Connect the 1/8" Parker™ tee to the carrier gas line as shown in Figure 1 using the nuts and ferrules provided.

5. Connect the piece of 1/8" copper tubing, provided in the BackFlush kit, to the sidearm of the 1/8" tee.

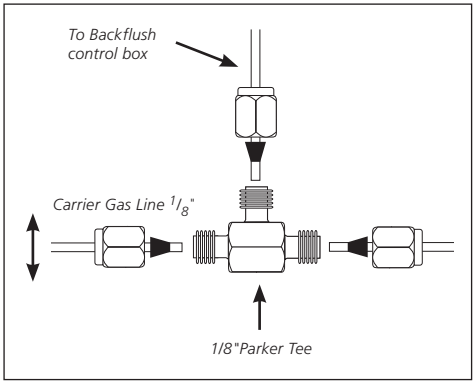

Figure 1.

6. Place the BackFlush control module on the top of the GC (or somewhere safe and dry) within reach of the copper tubing connected to the tee.

7. Connect the copper tubing to the "Carrier Gas In" port on the BackFlush control module with the 1/8" nuts and ferrules provided.

8. Connect the short 20 cm piece of stainless steel tubing to the "Carrier Gas Out" port on the BackFlush control module. Use the nut and Graphite/Vespel® sealing ring (VSR-16) provided.

9. Connect one side the adsorption tube (P/N 103489) to the remaining end of the short piece of stainless steel tubing. Use the nut 1/16" nut and the Graphite/Vespel® sealing ring (VSR-16) provided.

10. In the BackFlush kit there is a 1/16" stainless steel tube with a red marker on one end. Connect the non-marked end to the remaining side of the adsorption tube that is connected to the BackFlush control module.

11. Feed the other end of the 1/16" tube through a hole in the top of the oven wall and into the GC oven. NB: Leave the red cap on the end of the tubing when doing so. It will help protect the tube from blocking with insulating material when it's passed through through oven wall.

12. Place the BackFlush tee stand in the oven. Attach the 1/16" stainless steel union to the stand using the lock nut and washer located on the longest side of the union as shown in Figure 2.

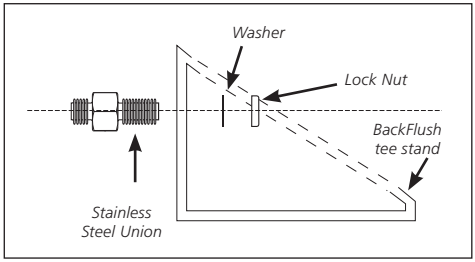

Figure 2.

13. Choose the correct restrictor for your capillary column using table 1.

Column	Restrictor Type
0.1 mm ID x 5 m	Type 1
0.20 mm ID x 15 m	Type 2
0.20 mm ID x 30 m	Type 1
0.22 mm ID x 12 m	Type 2
0.22 mm ID x 25 m	Type 1
0.25 mm ID x 15 m	Type 2
0.25 mm ID x 30 m	Type 1
0.25 mm ID x 60 m	Type 1
0.32 mm ID x 15 m	Type 3
0.32 mm ID x 30 m	Type 3
0.32 mm ID x 60 m	Type 2

Table 1.

Type 1
50 cm of 0.10 mm ID
Methyl Deactivated Fused Silica

Type 2
50 cm of 0.11 mm ID
Methyl Deactivated Fused Silica

Type 3
50 cm of 0.125 mm ID
Methyl Deactivated Fused Silica

14. Slide the long end of the restrictor through the union as shown in Figure 3.

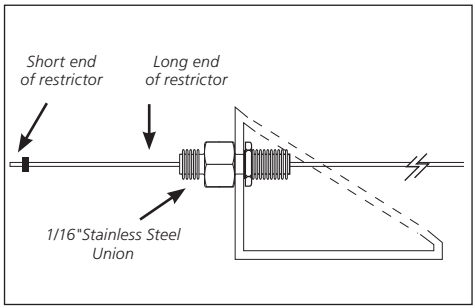

Figure 3.

15. Carefully Insert the short end of the restrictor into the female side of the BackFlush tee. The restrictor will have to be positioned so that the fused silica is inserted into the hole in the center of the tee. The SilTite™ sealing ring should then sit flat against the inner surface of the tee. See Figure 4.

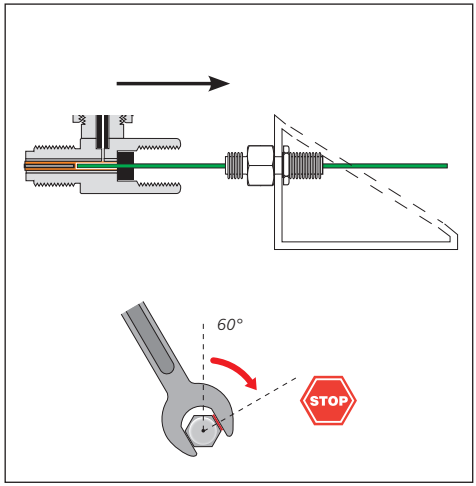

Figure 4.

16. Screw the BackFlush tee (with restrictor inserted) onto the short end of the stainless steel union attached to the bracket. Be careful not to let the restrictor fall out of the hole in the tee.

17. With a spanner, tighten the tee onto the union 60 ° only using a spanner to hold both parts.

18. Install the other end of the restrictor in the GC detector (FID, ECD, MS…etc).

19. Connect the 1/16" stainless steel tube (from the BackFlush control unit) to the side arm of the BackFlush tee using the 1/16" SilTite™ nut and ferrule supplied in the BackFlush kit. Be sure to take note of the orientation of the ferrule as shown in Figure 5.

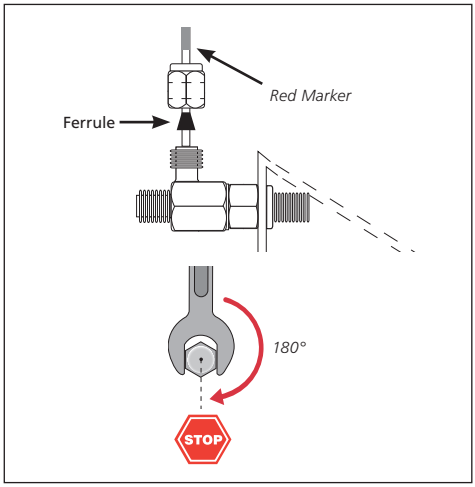

Figure 5.

20. Tighten the 1/16" nut finger tight and then turn 180 ° with a spanner.
21. Turn the carrier gas back on at the bottle and check for leaks at the 1/8" Parker™ tee that was inserted in the carrier gas line.
22. In the BackFlush kit there are two white valve input cables that can connect the control module to the GC. For the Agilent 6890, the cable with the 8 pin DIN plug is used, on all other instruments the cable without the DIN plug is used. Take the appropriate cable for your instrument and plug it into the "Valve Input" connection on the BackFlush control module.
23. Plug the other end of the cable into the external events output of the GC.

    On the Agilent 6890, the DIN plug connects to the "EXT EVNT" port on the back panel of the instrument.

    For the Varian 3800, the two wires at the end of the cable connect to the external events screw terminal block, labeled "E.E.". The screw terminal block is located under the left-hand top panel and is usually orange. Connect the wires from the valve input cable into a pair of connectors, eg. Valve 4.
24. Plug the 12V power supply into the "Power In" port on the BackFlush unit.
25. Plug the power supply into the mains power outlet and turn the power on.
26. Locate the ON/OFF switch on BackFlush unit and switch it on. The LCD display will light up and run through a brief starting procedure that will end with two pressure readings being displayed on the screen. The top line will display the pressure in the BackFlush unit and the lower line will display the pressure in the BackFlush tee.

CONNECTING THE CAPILLARY COLUMN TO THE BackFlush TEE:

If a capillary column is not installed in the GC, install one end in the injection port only.

1. Slide the SilTite™ nut and ferrule onto the outlet of the capillary column as shown in Figure 6. Be sure to take note of the orientation of the ferrule.
2. Place the end of the column onto the flat face of the tee and slide the nut and ferrule onto the male thread. See Figure 6.
3. Finger tighten the nut then use a spanner to tighten it another 60 °. NB.

   Over tightening may cause the column to break.

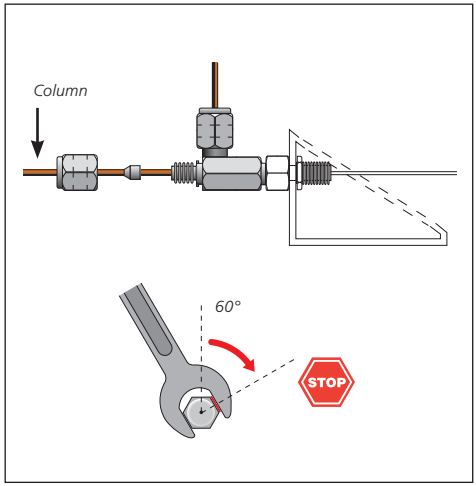

Figure 6.

OPERATING INSTRUCTIONS

How does the BackFlush system work?

When the compounds of interest have eluted from the analytical column, the BackFlush control unit receives an instruction from the GC that enables it to enter a state called "BackFlush Mode". At this point the system will terminate the analysis by reversing the direction of the carrier gas flow through the column, causing all the remaining compounds to be flushed out of the injection port instead of the detector.

This action can be set in the method and triggered by a run time event. For most instrument types a valve turning on symbolizes this event. For example, on a Varian 3800 or Agilent 6890, "Valve #5 ON at 5.1 minutes", will instruct the BackFlush unit to flush the column at 5.1 minutes into a run. In the software, this is under "sample delivery" in Varian® Star Workstation and "runtime" in HP/ ChemStation™. The BackFlush system has two modes of operation:

1.1 Analysis Mode

In analysis mode the injection port pressure is on and the BackFlush valve is off. The carrier gas flows through the column in the normal direction and passes through the BackFlush tee before reaching the detector, as shown in Figure 7.

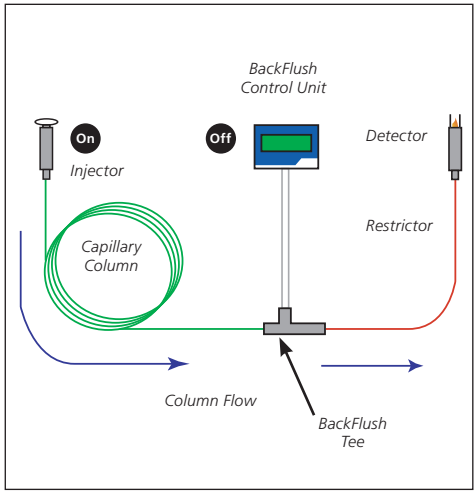

Figure 7 - The BackFlush system in analysis mode

1.2 BackFlush Mode

In BackFlush mode the column head pressure is set to 0.1 psi* (via inlet pressure programming) and the BackFlush valve is switched on (via timed events). This reverses the flow through the column, but the flow through the restrictor remains in the forward direction, so the detector is unaffected. Figure 8 shows the operation of the system in BackFlush mode.

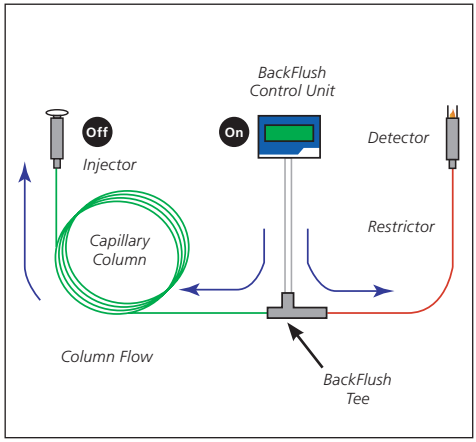

Figure 8 - The system in BackFlush Mode

It is important to note that the injection port pressure must be set to 0.1 when the BackFlush valve is on. This allows the injection port to act as the outlet (via the split vent) while the BackFlush control unit becomes the inlet.

* On some instruments (eg HP/Agilent 6890/6850), setting the pressure to 0 psi or Off causes the EPC to close the split vent. If the pressure is set to 0.1 psi the split vent will remain open.

1.3 Pressure changes that need to be made before operating the BackFlush system.

The restrictor that is installed with the BackFlush unit almost acts like another capillary column. It has no effect on the chromatography but it will change the pressure that is needed to operate the analytical column at its optimum linear velocity. The new pressure that is needed to operate the column + restrictor combination will always be higher than the pressure that was required to operate the column only. Table 2 contains the pressure increase factor that can be used to calculate new injection port pressure when the column and restrictor are both installed.

To calculate the new injection port pressure, multiply the old injection port pressure with the pressure increase factor for the column being used. For example, a 0.25 mm ID x 30 m capillary column will need 18.8 psi of Helium to run at 35 cm/sec at 100 °C. When this column and a type 1 restrictor are joined together the new pressure needed for 35 cm/sec at 100 °C will be 18.8 * 1.65 = 31.02.

Column	Restrictor Type	Old Pressure (psi) (Eg.)	Pressure Increase Factor	New Pressure Factor
0.1 mm ID x 5 m	Type 1	19.6	1.10	21.56
0.20 mm ID x 15 m	Type 2	14.5	1.36	19.72
0.20 mm ID x 30 m	Type 1	30.3	1.27	38.48
0.22 mm ID x 12 m	Type 2	9.4	1.67	15.70
0.22 mm ID x 25 m	Type 1	20.3	1.47	29.84
0.25 mm ID x 15 m	Type 2	9.1	1.89	17.20
0.25 mm ID x 30 m	Type 1	18.8	1.65	31.02
0.25 mm ID x 60 m	Type 1	39.1	1.33	52.00
0.32 mm ID x 15 m	Type 3	5.5	2.43	7.87
0.32 mm ID x 30 m	Type 3	11.2	1.72	19.26
0.32 mm ID x 60 m	Type 2	23.2	1.60	37.12

Table 2 - Pressure increase factors and examples of old and new column head pressures

The table values of "Old Pressure" and "New Pressure" are based on figures obtained from a detector at atmospheric pressure (FID, ECD, NPD…) and an oven temperature of 100 °C. The pressure increase factor is independent of these conditions and can be used to calculate the new column head pressure under different conditions than the examples.

2.0 Setting up a basic BackFlushing Method

One of the most critical parts of any back flushing method is the length of time the system spends in BackFlush mode. The amount of time is important because the compounds that are being removed from the column need enough time to exit, or carry over will occur. Changes in oven temperature and BackFlush pressure will affect the amount of time that is needed to remove the sample from the column, but these can be used to speed up the process if they are set correctly. A basic back flush can be performed using the steps in 2.1, 2.2 and 2.3.

2.1 Runnng a normal chromatogram and deciding when to BackFlush

To determine the exact time during a run when back flushing should occur, a chromatogram should be run without back flushing, on the same GC that the BackFlush system is installed. If another GC is used, the retention time of the appropriate point is likely to be slightly different, so it is highly recommended that the GC used has the BackFlush system installed. To install the BackFlush system, see the "Installation" section of this manual.

When running a previous method with the BackFlush system installed, remember to check that the new injection port pressure is being used (see section 1.3). This compensates for the restrictor that is installed with the capillary column, and ensures that the linear velocity of the carrier gas will be corrected.

The normal chromatogram should be run under the conditions that are usually used for the analysis (temperature program etc.) and the chromatogram must contain every peak that would usually be seen if the run was left to completion. Figure 9 shows a chromatogram of a gasoline sample that has been run under standard conditions where the diesel fraction is baked off the column as normal. The analytes of interest in the chromatogram are the volatile components of the gasoline. The purpose is to screen for C5 to C9 volatile contamination, so the heavier fractions of each sample are not needed.

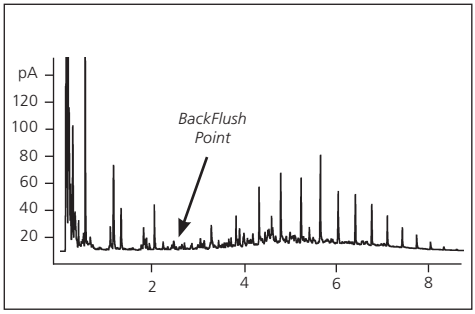

Figure 9 - Gasoline and Diesel sample run under standard conditions

Back flushing can occur immediately after the compounds of interest have eluted from the column. But it is a good idea to wait another ten seconds or so to allow for any future retention time variations. This makes sure that you will not accidentally flush out any compounds that need to be analysed. In figure 9, the BackFlush point was chosen to be at 2.25 min, which is after the C5 to C9 volatiles are eluted from the column.

Figure 10 shows a chromatogram of the same gasoline sample with a flush occurring at the point chosen in the first chromatogram.

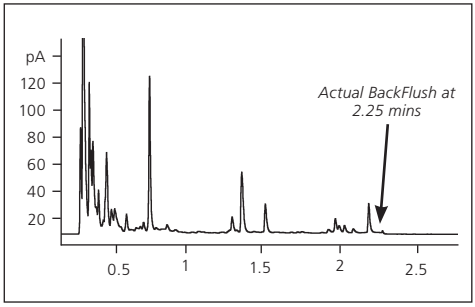

Figure 10 - Gasoline and Diesel sample with BackFlushing

The BackFlush control box displays two pressures. The top line of the display, labelled "set", is a reading of the pressure that will be supplied by the unit when the BackFlush valve is on. This is the carrier gas pressure that will flush the remaining sample out of the capillary column but is only active when the system is in BackFlush mode. The lower line of the display, labelled "outlet", is a direct reading of the pressure at the outlet of the capillary column. This reading will change depending on the type of column (and restrictor) installed, the injection port pressure and the current oven temperature, so it is likely to be different in every method. The "outlet" pressure is important because it is used to calculate the pressure drop across the column, which defines the minimum BackFlush pressure.

To calculate the pressure drop across the column:

Injection Port Pressure – "outlet" Pressure = Pressure Drop*

eg. If the injection port pressure is 19.3 psi and the "outlet" reading on the BackFlush unit is 9.7 psi, the pressure drop across the column is 19.3 – 9.7 = 9.6 psi

The pressure drop across the column is the minimum pressure required to BackFlush the remaining compounds out of the column when the flow is reversed. Using the example above, the injection port pressure would be 19.3 psi. This would cause the "outlet" reading on the BackFlush unit to be 9.7 psi because of the natural pressure drop across the column. The pressure in the BackFlush unit should then be set to a minimum of 9.6 psi by adjusting pressure control knob on the back of the unit until the "set" pressure on the front LCD reads the correct value.

*Remember to ensure that the injection port pressure is in the same units as the "outlet" pressure when calculating the pressure drop.

2.3 Determining the length of time to leave the system in BackFlush mode

When using the minimum BackFlush pressure, the time that it takes to flush the remaining sample out of the column will be equal to the time the compounds have spent in the column (this is assuming that the oven temperature remains constant once the system enters BackFlush mode). Therefore as a basic rule, when using the minimum back flush pressure, the total run time of the chromatogram is equal to twice the amount of time the chromatogram was running when the BackFlush unit was turned on. ie. If the method is set up to enter BackFlush mode 10 minutes into a run, the system should be left in BackFlush mode for at least another 10 minutes before the run is completed. This ensures that whatever sample is left in the column gets totally flushed out.

3.0 Fast Back Flushing

The procedures described in section 2.0 to 2.3 can be used to set up a basic BackFlush method but the run times will be much longer than what can be achieved when the system is optimized. When a BackFlush method is running at it's optimum, the run time can be up to 80 % shorter than the original analysis. The following steps outline how to speed up the back flushing process.

3.1 Using the oven temperature to speed up a BackFlushing method

It is possible to reduce run times by ramping oven temperature while the system is in BackFlush mode. If the temperature is higher when back flushing, the sample that remains in the column will be retained less by the phase on the way out than it was on the way in. So it will take less time to exit than it took to enter the column. Therefore, the time that is needed to completely flush the column will be less than the current run time when the system enters BackFlush mode.

i.e. If the BackFlush unit is turned on at 4 minutes, it does not have to be left on for another 4 minutes to completely flush out the remaining fraction of the sample. The higher temperature may reduce the flush time to 2 or 3 minutes.

Conversely there is no need to "bake out" the column at a very high temperature, so column life can be increased if the oven temperature is kept well within the columns limits. The high boiling point compounds do not have to travel large distances through the column once the flow is reversed, so it is usually unnecessary to take the column to its maximum operating temperature.

The best way of taking full advantage of the effects that temperature has on back flushing is to leave the oven temperature ramp on (if one is being used) when the system first enters BackFlush mode. The best method is to leave the temperature ramp as normal during the analysis and when the system enters BackFlush mode, quickly ramp the oven temperature up another 100 °C*, then run isothermally until the compounds have been flushed out of the column. The value of 100 °C could be different depending on the type of analysis, if in doubt, find examples of similar BackFlush methods (www.sge.com) and use them as a reference.

*In methods where back flushing occurs near the maximum column temperature, there is usually no need to raise the oven temperature any further. Beware of the maximum column temperature if doing so.

3.2 Using the BackFlush pressure to speed up a back flushing method

Another way to decrease the time it takes to flush out the remaining sample is to increase the (reverse) flow of the column. By having a faster linear velocity in BackFlush mode than in analysis mode, the flush time can be reduced, which in turn, reduces the overall run time. The easiest way to do this is to double the minimum BackFlush pressure (described in section 2.2), this will double the linear velocity of the carrier gas when the system is in BackFlush mode.

For example, if a 5 m x 0.1 mm ID capillary column and a type 1 restrictor were installed, the injection port pressure would have to be about 21.6 psi (from table 2). The "outlet" pressure on the BackFlush unit will read 3.1psi because of the pressure drop across the column. So the minimum BackFlush pressure will be 21.6 – 3.1 = 18.5 psi. If we double this pressure we get 37psi. This would be the maximum recommended BackFlush pressure in this example. Figure 10 shows a chromatogram with back flushing that was run with the same column and restrictor setup. The BackFlush pressure in that example was 30 psi which was enough to achieve fast back flushing when combined with a temperature ramp (as described in section 3.1).

Packing list

gc-BackFlush Control Module	1
Valve input cable for connection to GC	2
Adsorption tube (chemical trap)	1
gc-BackFlush Tee Piece with SilTite Nuts	1
1/16" SS Union with lock nut and washer	1
gc-BackFlush Tee Piece Mounting Bracket	1
1/16" Stainless Steel Tubing with Restricted End	1
1/16" Stainless Steel Tubing 0.8 mm ID x 20 cm	1
SilTite 1/16" Ferrule	5
Spare SilTite™ Column Nut (0.8 mm ID)	1
SilTite 0.4mm ID Ferrule	5
SilTite 0.5mm ID Ferrule	5
1/8" Parker Tee Fitting	1
1/8" Copper Tubing	1
1/8" Graphite/Vespel® Ferrule	5
1/16" Graphite/Vespel® Sealing Ring	5
Restrictor Kit Type 1	1
Restrictor Kit Type 2	1
Restrictor Kit Type 3	1
5/16" Spanner	2
7/16" Spanner	1
AC/DC Power Adapter	1
Instructions	1

Re-Order list

SilTite Kit 10/32", for 1/16"	073203
SilTite Kit 10/32", for 0.25 mm ID Columns	073200
SilTite Kit 10/32", for 0.32 mm ID Columns	073201
Graphite/Vespel® Sealing Ring 1/16" (ptk 10)	072653
Graphite/Vespel® Ferrule 1/8" (pkt 10)	072652
Restrictor Kit Type 1	093355
Restrictor Kit Type 2	093356
Restrictor Kit Type 3	093357
gc-BackFlush Tee Piece	093354
Adsorption tube (chemical trap)	103489
1/16" SS tube with restricted end (red marker)	113417

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