Capillary Flow Technology—Backflush

Small device.

Big boost in results and productivity.

Our measure is your success.
A simple way to reduce cycle times and increase your lab’s productivity.

GC and GC/MS analyses are often performed on complex samples containing late-eluting compounds or sample matrix; many of those analyses require detection and quantitation of peaks found early in the chromatogram. In these cases, higher temperatures and longer run times are needed to elute the high boilers before starting the next run.

How about a more efficient alternative?

Backflushing—reversing column flow immediately after the last compound of interest has eluted—can improve the quality of your analysis and save you time and money. The technique eliminates long bake-out times and high temperatures used to elute highly retained sample components. Instead, these materials are swept backwards through the column and out the split vent—preventing carryover, column fouling, retention time shifts, and MSD source contamination.

**Backflushing improves your GC analysis.**
**Agilent Capillary Flow Technology makes it easy.**

- **Faster cycle times**—Shorter analytical run times and faster cool-down allow faster results and more samples per day per instrument.
- **More uptime**—Removing high boilers means less frequent column trimming and detector maintenance, as well as less need for recalibration.
- **Lower costs**—Columns last longer because high boilers are removed and columns are not exposed to high bake-out temperatures.
- **Better results**—Backflushing significantly reduces column bleed, eliminates ghost peaks, and improves the quality of peak integration.
An Auxiliary EPC (electronic pneumatics control) module or a PCM (pneumatics control module) provides a second source of gas. In normal operation, the Aux EPC or PCM pressure provides a slight incremental flow through the Capillary Flow Technology device. During backflush, the inlet pressure is lowered and/or the Aux EPC or PCM pressure is increased, which forces the flow to reverse through the column and out the inlet split vent.

**How backflushing works**

Conventional backflush configurations require rotary valves, stainless-steel tubing, and fittings in the sample flow path. All this plumbing—with its dead volume, high thermal mass and activity—can cause peak broadening, analyte loss, and other problems. And sooner or later, it’s going to leak.

Agilent’s proprietary Capillary Flow Technology makes it easy to make reliable, gas-tight connections, every time. The inert, low mass, low dead volume modules can stand up to the temperature extremes of any GC oven. They also save on electricity and require less carrier gas per sample.

- **Low thermal mass** allows the modules to closely follow the oven ramp, avoiding peak tailing frequently seen with conventional approaches.
- **Small, well-swept dead volumes** eliminate peak distortion and sample losses.
- **Capillary Flow Technology ferrules and fittings** eliminate leaks—even after many oven temperature cycles—optimizing uptime and increasing the accuracy of results.
- **Inert surfaces** across the entire flow path minimize loss of labile analytes.

*To learn more about backflushing and other techniques enabled by Agilent Capillary Flow Technology, visit www.agilent.com/chem/backflush*
Better results, faster.

**Significantly reduced run time**

In analysis of food extracts, run times and temperatures are often increased to elute matrix components that elute after the last target compound.

By backflushing for 7 minutes after the last component of interest has eluted, the original (top) run time was reduced from 75 minutes to 40 minutes, and the upper temperature reduced from 320°C to 280°C.

Total time saved: 35 minutes, a run time reduction of 46%. The column did not have to be exposed to the higher bake-out temperature, and excess column bleed and heavy residues were excluded from the MSD.

**Elimination of sample carryover and retention time shift**

Sometimes, extended run times and temperatures do not remove all late-eluting sample components, such as with this fish oil extract.

**Without backflush.** In the figure at left, the top chromatogram shows a GC/FID analysis from a 1 µL splitless injection. The red arrow indicates the end of target compound elution. Holding oven temperature at 290°C for an extra 25 minutes was ineffective at removing highly retained sample matrix before the next run, as shown by the two subsequent blank runs to 310°C for 30 minutes. Residue from the fish oil injection can clearly be seen and has led to significant retention time instability.

**With backflush.** The figure at right shows the first (top) and sixth (inverted) injections of fish oil in a sequence. The column was backflushed after each run, preventing build-up of fish oil residue. The comparison demonstrates that there was baseline upset or shift in retention times caused by fish oil accumulation.

*(Agilent 7890A GC/5975C MSD with Capillary Flow Technology purged 3-way splitter. See Agilent Application Note 5989-6018EN.)*
Elimination of ghost peaks, carryover, and baseline instability

GC/MS analysis of fragrances in cosmetics can suffer because of less volatile or non-volatile matrix components, such as detergents, waxes, lipids, etc. In this example, shampoo samples were directly injected, and a Capillary Flow Technology QuickSwap device was used to effectively backflush the low-volatility components.

**Without backflush.** The bottom GC chromatogram on the left shows the sixth analysis of a shampoo extract, stopped at approximately 8 minutes (240°C), after the last compound of interest had eluted. The middle chromatogram over the same time period shows the first blank run after the six runs; sample carryover is clearly evident as baseline disturbances and ghost peaks. There are other issues as well. The top chromatogram shows the second blank run extended to 320°C where the extent of highly-retained matrix peaks can clearly be seen. Over time, these can cause column deterioration, difficulty in detecting and quantifying minor sample components, reduced MS performance, and more frequent source cleaning.

**With backflush.** On the right is an overlay of 10 consecutive analyses with backflush performed after each run, showing excellent retention time stability and peak area reproducibility—and no evidence of carryover, ghost peaks, or increasing baseline. An added benefit was a 20% run time reduction and faster oven recycle times.

(Agilent 5975C GC/MSD with Capillary Flow Technology QuikSwap module. See Agilent Application Note 5989-6460EN.)
GC/MS: Pre-column backflush maintains MSD sensitivity

For trace analyses with extremely flow-sensitive detectors, pre-column backflushing can offer several advantages over post-column backflush.

Using a pressure-controlled tee Capillary Flow device between a pre-column and the analytical column, the Aux EPC supplying the purged union is configured so that the flow of the second column is equal to that of the original method. Concurrent backflush is accomplished by reducing the inlet pressure of the first column while analysis continues with the second column. Alternately, post-run backflush can be initiated after the last compound of interest has eluted by decreasing the inlet pressure and increasing the pressure of the purged union. This configuration also enables pre-column and inlet maintenance without having to vent the MSD and can be used with both turbo and diffusion pump MSD systems.

**Simple Tee configuration for high sensitivity GC/MS analysis with rapid backflushing**

Solid lines indicate the forward flow during the analysis, and dashed lines indicate the backflushing flows.

**Backflushing with pressure-controlled tee configuration**

Sample - no backflush

Sample - with backflush

Solvent blank - no backflush

In the example above, the top chromatogram shows a six-standard chromatogram, where the third peak is considered the last analyte of interest and the fourth peak is the first of the late-eluting interferences. The middle chromatogram shows (a) the same standard with backflushing beginning at 10.1 minutes, where flow is dropped in the pre-column and increased in the second column. Note that the last analyte is retained, but the late-eluters never enter the MSD. The bottom chromatogram is a blank run, demonstrating no evidence of carryover.

(Capillary Flow Technology for GC/MS. Agilent Application Notes 5989-6460EN and 5989-9359EN)
It’s easy to take advantage of Agilent’s backflush capability

Whether you’re bringing a new instrument into your lab or adding capability to an installed 7890 or 6890 Series GC with EPC, you can get all the benefits of Agilent Capillary Flow Technology with minimal investment of time and money. Here’s what you need to get started.

**Purged Capillary Flow Technology accessory kit or GC option** — one of the following
- Pressure-controlled tee
- Purged Ultimate Union
- Purged (2-way or 3-way) splitter
- QuickSwap
- Deans switch
- GC x GC Flow Modulator

**Flow source** — Aux EPC module or PCM

Both PCM and Aux EPC provide real-time ambient temperature compensation for retention time (RT) repeatability and stability; PCM also provides real-time atmospheric pressure compensation for even more precise RT repeatability and stability. Aux EPC provides three channels of pressure control.

<table>
<thead>
<tr>
<th>Description</th>
<th>Part Number</th>
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<tbody>
<tr>
<td>Accessory kits for 7890A/6890N and options for new 7890A GC</td>
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<tr>
<td>Capillary Flow Technology Deans Switch</td>
<td>G2855B, G3440A option 888</td>
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<td>Capillary Flow Technology Two-way Splitter with Makeup Gas</td>
<td>G3180B, G3440A option 889</td>
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<td>Capillary Flow Technology Three-way Splitter with Makeup Gas</td>
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<td>Capillary Flow Technology QuickSwap</td>
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<td>Capillary Flow Technology Purged Union</td>
<td>G3186B</td>
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<td>Capillary Flow Technology GC x GC Flow Modulator</td>
<td>G3486A, G3440A option 887</td>
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<td>PCM for 7890A GC</td>
<td>G3471A, G3440A option 309</td>
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<td>PCM for 6890 GC</td>
<td>G2317A</td>
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<td>Aux EPC for 7890A GC</td>
<td>G3470A, G3440A option 301</td>
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<td>Aux EPC for 6890 GC</td>
<td>G1570A</td>
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**Spare parts**

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<tr>
<td>SilTite Metal Ferrules, 1/16 inch x 0.4 mm id, and 2 column nuts (for 0.20-0.25 mm id columns); 10/pack</td>
<td>5184-3569</td>
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<tr>
<td>SilTite Metal Ferrules, 1/16 inch x 0.5 mm id (for 0.32 mm id columns); 10/pack</td>
<td>5184-3570</td>
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<tr>
<td>SilTite Metal Ferrules, 1/16 inch x 0.8 mm id, and 2 column nuts (for 0.53 mm id columns); 10/pack</td>
<td>5188-2789</td>
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<tr>
<td>Fused Silica, Deactivated — 0.100 mm x 5 m (for restrictor)</td>
<td>160-2635-5</td>
</tr>
<tr>
<td>Fused Silica, Deactivated — 0.200 mm x 5 m (for restrictor)</td>
<td>160-2205-5</td>
</tr>
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High performance Agilent J&W GC columns and supplies for every analytical need.

Agilent GC consumables, including our J&W columns, are designed, manufactured, and packaged to deliver maximum productivity from your Agilent GC and GC/MSD systems. We strive to provide you with the cleanest, most inert flow path. From our proprietary deactivated inlet liners to our injection-molded inlet gold seal through the J&W low-bleed columns, your samples are protected from exposure to active sites or outgassed contaminants that can alter your results.

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© Agilent Technologies, Inc. 2010
Printed in USA February 18, 2010
5990-5358EN