Reducing Helium Costs more than 10-fold with the PCT Gas Saver mode

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Technical Overview

This note describes a technique for minimizing helium carrier flow when the GC-MS system is not in use. Helium has become expensive and costs of operation are always a concern. Using the G1472A Rapid & Universal GC-MS Backflushing kit in the Pressure Controlled Tee (PCT) configuration, total helium use can be lowered to < 3 mL/min while the GC-MS is dormant. The MS analyzer is still under vacuum, at operating temperatures and can be rapidly called into use. Total helium savings over the course of a month or year are substantial using this mode.

Introduction

Recently the price of helium carrier for gas chromatography has dramatically increased and supply has become limited. Many instrument users have become concerned about saving this gas whenever possible. Saving helium carrier by cooling and venting GC-MS instruments (single quadrupole and triple quadrupole) is not an efficient approach because air and water enter the vacuum manifold. Although the instruments can rapidly reach analyzer operating temperatures, the removal of chemisorbed water is a slow process; therefore the time necessary to reach stable operation is lost analytical time. In addition, the frequent venting and pumpdown cycles wear out the pump system components and tend to be time consuming. The optimum situation has always been to keep an MS system under vacuum and at temperature to allow the system background to continuously improve or at least remain stable. This philosophy is also true for other GC detectors. Employing GC instrument parameters to minimize gas use, such as standby methods using low split ratios with low column flows and septum purge settings, risk air intrusion with consequent damage to the inlet or column. These methods can produce a gas usage rate of about 7 to 10 mL/min at best. While this is lower than the typical use of greater than 24 mL/min during operations, it still represents a substantial quantity of helium expended that is not available for analysis.

This note describes a state of the GC instrument enabled by the Pressure Controlled Tee (PCT) and the Agilent Rapid Universal GC/MS Backflushing Kit (G1472A) in which total helium carrier gas use is < 3 mL/min during the archived state of the GC-MS instrument. This state is referred to as the “PCT Gas Saver mode” and, while saving helium, the MS system remains under vacuum and at temperature and rapidly can be brought into operation. In terms of helium carrier savings, nearly every hour the instrument is placed in this PCT Gas Saver mode is equal to a saved hour of analysis when compared to typical (default) operating conditions (24 mL/min). Over the course of a year this can result in considerable helium gas savings. For example, archiving a GC-MS instrument with the PCT Gas Saver through every weekend in a year will result in a helium savings equivalent to more than an additional 100 days or almost half of year of analytical operating time. This technical overview contains specific directions for enabling this PCT Gas Saver mode for PCT with the GC-MS. The approach can be easily generalized to other PCT configurations to enable carrier gas savings on all GC systems.

Why the PCT for GC/MS Operation?

A simple schematic and an illustration of the PCT configuration are shown in Figures 1 and 2. The analytical capillary column is split into two sections by a tee, called the Purged Ultimate Union. Many configurations of the PCT are possible because the column sections do not need to be of equal lengths. Using the PCT improves GC-MS operation by providing rapid backflushing and rapid GC servicing.

Rapid backflushing removes late eluting matrix contaminants to:

- Avoid fouling the MS source thereby increasing instrument uptime and eliminating frequent source cleaning
- Avoid carryover, baseline rise, and compound retention time shifts over the course of sample sequences

The PCT enables both post acquisition or Post Run backflushing and Concurrent backflushing modes to minimize run time and cycle time. Concurrent backflushing takes place while sample data acquisition is still underway.

Rapid servicing without venting the MS system allows:

- Quick capillary analytical column cutbacks to quickly restore chromatographic performance
- Fast inlet maintenance including liner change and septum change
- Simple column servicing or the more efficient approach of guard or coated precolumn exchange to restore compound chromatographic performance and maintain compound retention times

PCT operation is simple to understand (Figure 1). During GC analysis, the pressure applied at the Purged Ultimate Union (P_{epc}) is just a little higher than that needed to prevent backflow into the electronic pneumatic control (EPC) module. After the GC-MS analysis is completed, the pressure is raised at the tee (Purged Ultimate Union) and lowered in the front section of column to push matrix out the split vent. This is Post Run backflushing mode. In Concurrent backflushing mode, after the last analyte of interest has passed through the front column and the tee (Purged Ultimate Union) and entered the second section of column, the head pressure at the inlet can be dropped (P_{inlet}) so that the later eluting components will begin to retreat in column 1. The last analytes then proceed to column 2 and enter the MS detector. The MS system can be any of the Agilent systems including diffusion pumped systems.

Details and evidence of PCT performance are given in several application notes [1-4] and instructions for the installation and operation are given in the *Agilent G1472A Rapid Universal GC/MS Backflushing Kit Pressure Controlled Tee* manual (G1472-90001).

For simplicity, this note describes work performed with the midpoint PCT configuration in which the Purged Ultimate Union is inserted between two 15-m columns (0.25-mm id). The concept is easily generalized to other column arrangements.

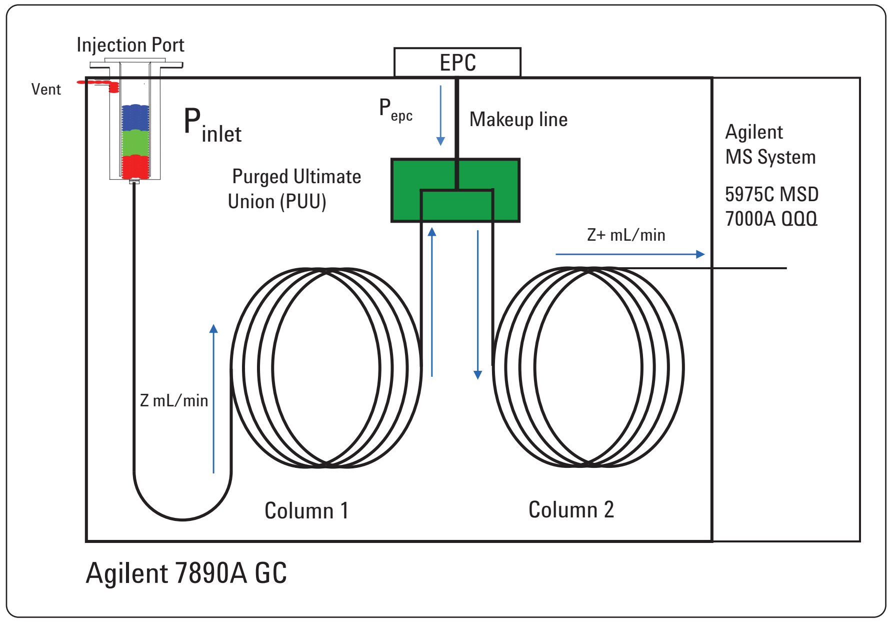

Figure 1A. The PCT configuration schematically shown in the forward flow mode operating during analysis. The EPC device supplies just enough pressure to prevent backflow into the connecting EPC device line ($P_{inlet} > P_{epc}$).

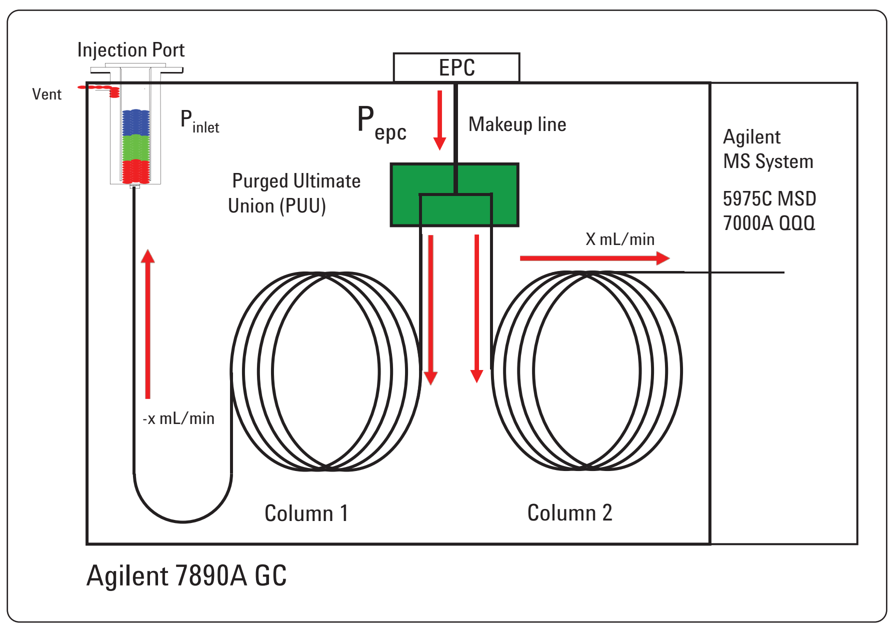

Figure 1B. The PCT configuration schematically shown in backflush mode. After completing the analysis the EPC pressure has been raised to send flow back through the forward section of the column into the inlet and out the split vent ($P_{inlet} < P_{epc}$).

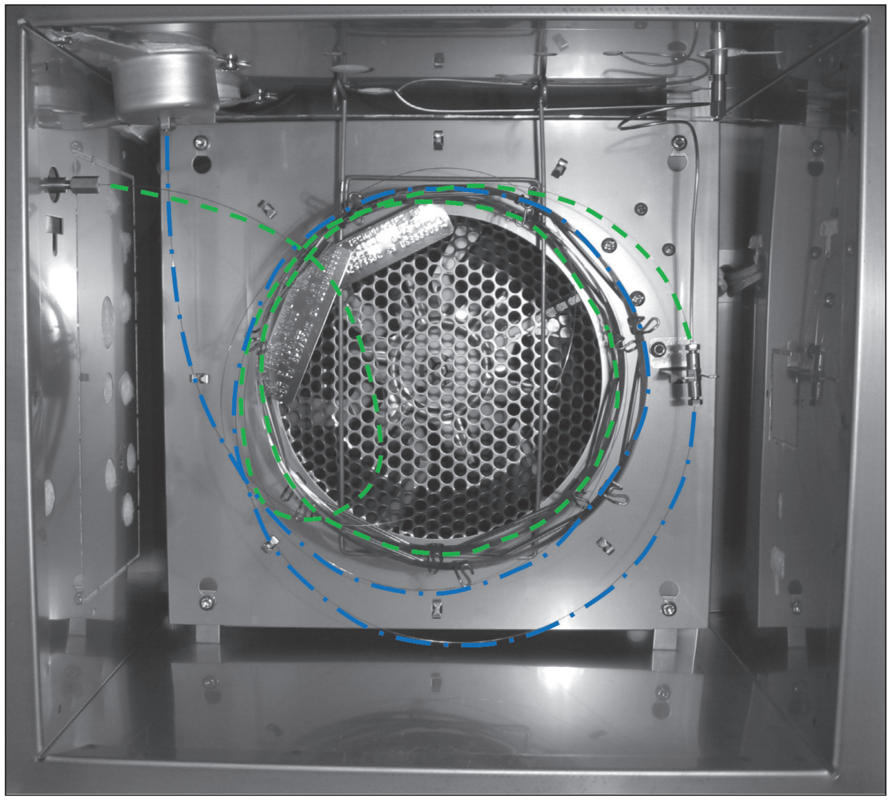

Figure 2. Picture of an installed PCT: Column 1 connects (dash-dot-dash) the injection port to the Purged Ultimate Union (at right) and Column 2 connects (dash) the MS system to the Purged Ultimate Union.

Procedure for PCT Gas Saver Mode

To put the system in PCT Gas Saver mode:

1. Set the injection port temperature to OFF and allow it to cool to room temperature. The port will cool faster with the GC oven set to 20 °C. After the oven has reached room temperature it can be turned OFF to save power.
2. Set the Column 2 flow to 1.5 mL/min (constant flow) (Figure 3). (Note: for columns narrower than the 0.25 mm id used in this example, set the pressure at Column 2 to 3 psi. For example, a 10 m × 0.18 mm id Column 2 would only require a flow of ~0.6 mL/min which results in even higher gas savings.)

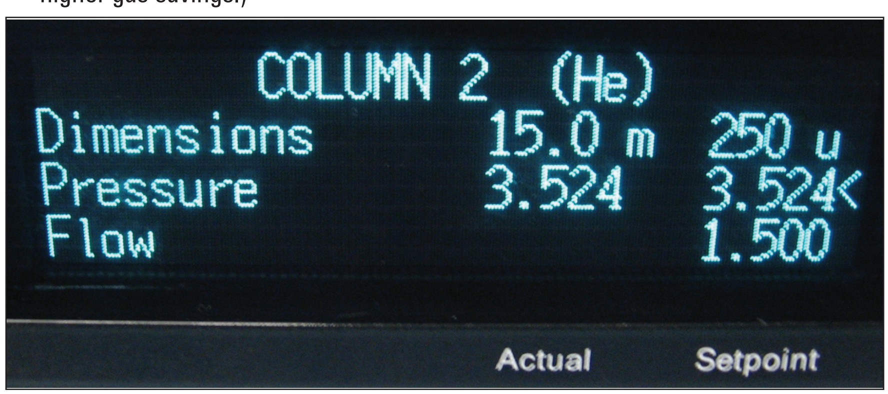

Figure 3. Column 2 (AUX → MSD) conditions: column flow setpoint 1.5 mL/min.

3. When the injection port and oven have cooled, set the inlet pressure to OFF. The port must then be opened by either removing the septum and septum nut or rotating the twist-top to release the liner connection (Figure 4). This releases the pressure inside the port and the inlet. The Column 1 display should read zero or close to it.
(Alternatively, if the user does not want to open the injection port, then: set the 7890 Gas Saver to OFF, the inlet pressure to 0, total flow to 0 and septum purge to 0. The 7890 system will beep as it can not achieve the setpoints.)

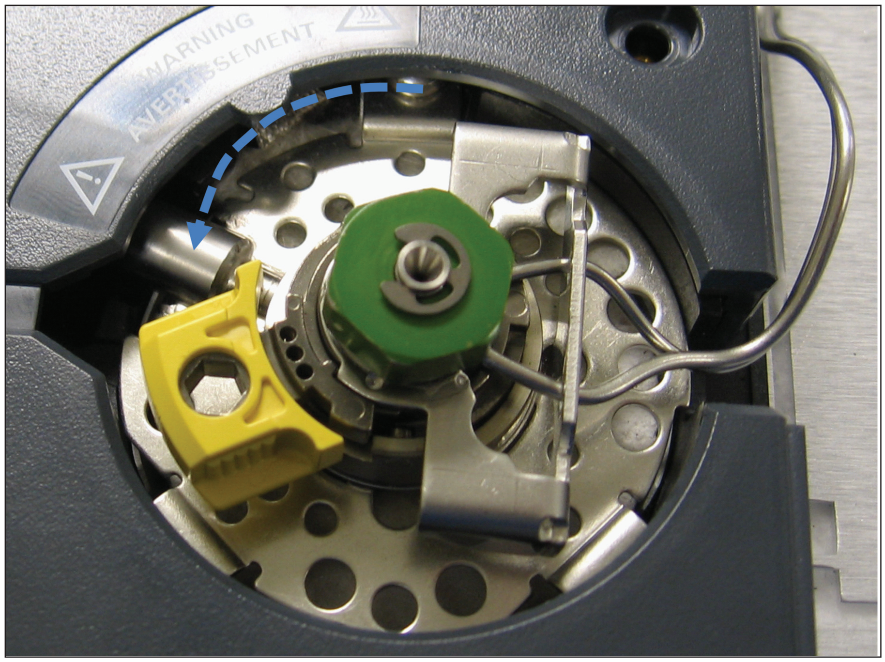

Figure 4. When the injection port is cold, loosen the injection twist-top by counter-clockwise rotation of the yellow handle. Make sure to lift the top slightly to insure the seal is broken.

4. In this 15 m × 15 m column configuration, the calculated flow displayed on the GC panel for Column 1 near the inlet should be negative and approximately –0.53 mL/min (Figure 5). This shows column flow is back from the Purged Ultimate Union into the inlet.

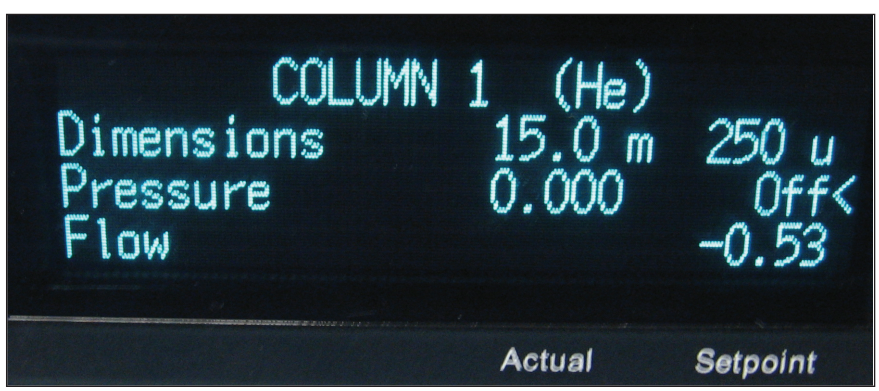

Figure 5. Column 1 (Inlet → Aux) conditions: pressure OFF, the flow calculation result is shown and the negative sign indicates flow is back into the injection port.

To return the system back to use:

1. Set the Column 2 flow to 3 mL/min, for diffusion pump systems, or 8 mL/min for turbo systems. See the PCT User's Manual
2. Reseal the injection port and set the inlet pressure to 2 psi.
3. Set the septum purge to ON at ≥3 mL/min and gas saver on with a split flow of ≥20 mL/min.
4. Turn on the injection port temperature.

The Column 1 flow will be negative (–1.7 mL/min in 15 m × 0.25 mm id configuration) and as the port warms up to operating temperature, contaminants accumulated during the idle period will be backflushed out the split vent and not transferred into the analytical column.

Results

Comparing the air/water background in the mass spectrometer reveals no measureable increase due to air intrusion between the operating state and this PCT Gas Saver mode even when compared over a > 72-hour weekend period (Figure 6).

Total gas use is about 2.6 mL/min for this configuration. The default 7890 Gas Saver mode is a split flow of 20 mL/min, an additional flow used by the septum purge is 3 mL/min (minimum), and the column flow of about 1.2 mL/min means that the minimum use in operation is 24 mL/min. If the user were to apply this PCT Gas Saver mode over only weekends (from Friday at 6 pm to 8 am Monday morning, 62 hours), they would gain about 55 hours of operation every week. If this is applied every weekend of a year, the helium savings is over 2600 hours or 100 days of operation each year. Even overnight from 6 pm to 8 am saves helium carrier equivalent to ~12.5 hours of operating time or more than an additional 8-hour day of operation.

Also worth noting is that PCT Gas Saver mode, unlike 7890 Gas Saver mode, causes a very small backward flow through the port. Therefore, trace carrier contaminants do not accumulate as rapidly as they would at higher split flow. This is similar to the PCT Standby mode where the system is in backflush and column is kept clean [5].

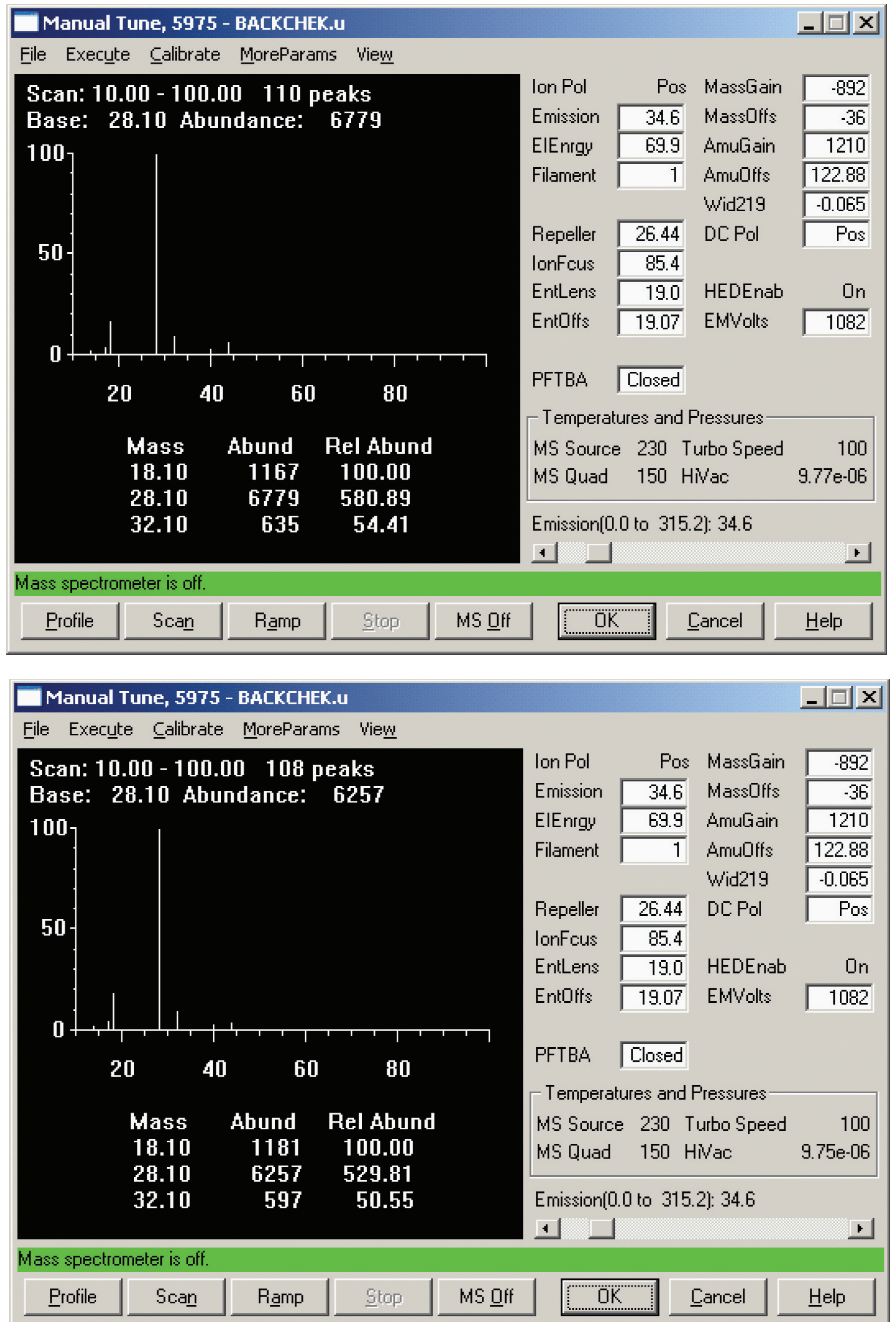

Figure 6. MS background with 1.5-mL/min helium carrier and no flow contribution from front split/splitless injection port. Upper panel at time zero and lower panel at 72 hours later. No change in the 18, 28 or 32 background is detected over the period. (Note: a small air leak does exist in this system).

Conclusions

The PCT configuration provides a number of features that improve GC-MS operation. These include rapid backflushing to protect the column and MS source and increase productivity, and quick, ventless servicing of the column and inlet to maintain data quality. The PCT Gas Saver mode is an additional capability which "archives" the GC-MS system with extremely low helium carrier gas use. This allows the MS analyzer to remain at temperatures where the background and stability of the system improves, or maintains its integrity. The GC system can be quickly called into use so there is less time required than in venting the system and returning it to service.

PCT Gas Saving Mode uses carrier at < 3 mL/min. When compared to typical GC-MS use, every hour the system is in PCT Gas Saver Mode is an additional hour of analytical time and carrier saved. Routinely archiving the system in this mode for every weekend of a year equates to more than 100 days of analytical time recovered. Since the PCT can be enabled on any GC-MS (or GC) system, an entire laboratory can employ these savings to dramatically reduce one of the major costs of operation. Table 1 shows a summary of the additional analytical time that can be provided by PCT Gas Saver mode over various durations.

Table 1. Additional Operating Analytical Time (Days) Supplied by the PCT Gas Saver Mode as a Function of the Duration Applied and Period of Consistent Use.

	PCT Gas Saver Mode Use		
Period	**Every night**	**Only weekends**	**Evening and weekends**
1 Week	3.6 days	2.3 days	4.4 days
1 Month	14.6 days	9.2 days	17.5 days
1 Year	174.6 days	110.5 days	210.3 days

Note: For example, using PCT Gas Saver Mode only on the weekends will add an additional 110 days/year of operation compared to the default Gas Savers Settings of 20 mL/min (plus column and septum flows of 1.2 mL/min and 3 mL/min, respectively).

References

1. "Capillary Flow Technology for GC/MS: A Simple Tee Configuration for Analysis at Trace Concentrations with Rapid Backflushing for Matrix Elimination," Agilent Technologies publication (5989-8664EN)

2. "Capillary Flow Technology for GC/MS: Efficacy of the Simple Tee Configuration for Robust Analysis Using Rapid Backflushing for Matrix Elimination," Agilent Technologies publication (5989-9359EN)

3. "Maintaining Compound Retention Times with the Backflush enabled Pressure Controlled Tee Configuration for Agilent 7890A GCs with Agilent 5975 Series MSD and Agilent 7000 Series Triple Quadrupole MS Systems," Agilent Technologies publication (5990-4643EN)

4. "Implementation of the Pressure Controlled Tee for Backflushing for the 7000 Series Triple Quadrupole Mass Spectrometer: Implications for Sensitivity," Agilent Technologies publication (5990-4504EN)

5. "User Quick Guide to Pressure Controlled Tee (PCT) Operation - Post Run Backflushing," Agilent Technologies publication (5990-5484EN)

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