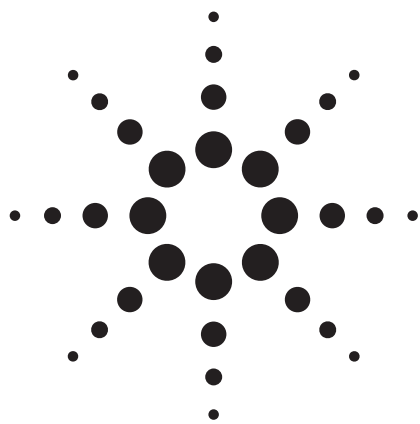




Implementation of the Pressure Controlled Tee for Backflushing for the 7000 Series Triple Quadrupole Mass Spectrometer: Implications for Sensitivity

Application Note

Environmental, Drug testing, Forensics and Metabolomics

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Abstract

The 7000 Series Triple Quadrupole Mass Spectrometer is designed to provide the most sensitive and specific analytical determinations in gas chromatography. The enhanced specificity of GC/MS/MS analysis encourages a more relaxed or minimized sample preparation strategy with the aim of higher sample throughput and sample turnaround time. However these more complex samples can rapidly degrade the gas chromatograph (GC) and mass spectrometer (MS) performance. Applying backflushing helps maintain the GC and the MS source by removing later eluting compounds from the column before reaching the MS, and prior to the next sample injection. This note cites the results of the Pressure Controlled Tee (PCT) configuration for rapid and universal GC/MS backflushing applied to selected reaction monitoring experiments on the Agilent 7890 GC used with the Agilent 7000 Series Triple Quadrupole Mass Spectrometer. The results show no loss in sensitivity and precision as measured for the peak area, height or signal-to-noise ratio in the 272→241 m/z for octafluoronaphthalene. This means that rapid backflushing and quick column and inlet maintenance without venting the MS system can be incorporated without a loss in detection limits using a simple PCT configuration.



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Introduction

Agilent's GC Capillary Flow Technologies (CFTs) have greatly expanded the capability and flexibility of gas chromatographic analysis. CFT can provide rapid column and inlet maintenance without MS venting as well as backflushing of the accumulated sample matrix preventing carryover which compromises the column performance. One such CFT arrangement, the Pressure Controlled Tee [1], has been shown to provide rapid backflushing and enhance analytical robustness in GC/MSD single quadrupole analysis in a very challenging sample matrix [2]. The expectation of reduced sample preparation steps for analyses using GC/MS/MS suggests that backflushing is essential to prevent source and column fouling and maintain sample throughput. In fact, MS/MS analysis of complex hydrocarbon samples has already demonstrated rising baselines and carryover in the absence of comprehensive backflushing [3]. However, concerns exist that implementing additional "plumbing" may compromise the signal and therefore the detection limits in MS/MS analysis. This note presents sensitivity results for the 7000A Triple Quadrupole GC/MS in multiple reaction monitoring (MRM) mode using the Pressure Controlled Tee (PCT) configuration.

Experimental

The target compound is the checkout compound, octafluoronaphthalene (OFN), which represents a model compound for demonstrating MRM detection thresholds. Studies were conducted at concentrations of 100 fg using the Pressure Controlled Tee (PCT) configuration.

The experimental arrangement is shown schematically in Figure 1. The typical 30-m analytical column is replaced by two 15-m columns joined by the Purged Ultimate Union. Makeup gas can be supplied by either an Auxiliary Electronic Pneumatic Control (Aux EPC) or Pressure Control module (PCM) but here an Aux EPC was used. The installation and use of the PCT are described in the G1472A kit and included documents [4]. It should be noted that the PCT is a very flexible configuration and many column combinations are possible. The one in this demonstration is the simplest to illustrate.

The same method MRM acquisition parameters were applied for the OFN as the standard instrument checkout method however the GC parameters were altered to include a backflushing period and to terminate the GC at 200 °C (Table 1). This shortened the runtime by about one third compared with the standard method. Data was also collected using a continuous 30-m column as in the checkout with the same tune file and standard checkout GC parameters.

Table 1. GC Parameters for OFN Testing with Backflush

Configuration:	(the actual arrangement is two – 15-m columns but the GC is configured as follows)
Column 1:	Front Inlet to MSD Outlet: HP-5ms 30 m × 0.25 mm, 0.25 µm film, helium carrier at 1.20 mL/min.
Column 2:	AUX EPC Channel 4 to MSD Outlet: HP-5ms 15 m × 0.25 mm, 0.25 µm film, helium carrier at 1.22 mL/min.
GC oven:	45 °C for 2.25 min, 40 °C/min to 195 °C (6 minutes runtime)
Post run:	200 °C for 2 minutes
Column 1:	0.25 mL/min (flow is actually reversed)
Column 2:	5 mL/min

Results

Replicate injections showed no statistically significant difference between the peak areas or heights for the 272 *m/z* → 241 *m/z* transition (Table 2). Further, retention time reproducibility was unaffected by addition of the PCT and a standard deviation of less than 0.1 secs was achieved both with and without the PCT. (The absolute retention times can shift slightly due to a slight difference in the absolute flows and lengths). Signal-to-noise was also indistinguishable between the two configurations although noise is uniformly low in selected reaction mode so variation in the signal-to-noise ratio can be high. The degree of correspondence is best illustrated in Figure 2.

Conclusions

Since part of the strategy for sample analysis by MRM is minimized sample cleanup to increase sample throughput, protecting the mass spectrometer source integrity and GC column phase through backflushing will help achieve system robustness. Introducing the Pressure Controlled Tee configuration into the 7890 GC combined with the 7000 Series MS maintains signal height and area without degrading compound retention time reproducibility or other performance metrics. This means that rapid backflushing and service of the injection port and column (without venting the mass spectrometer) can be introduced without compromising analytical integrity. In this way the PCT is a powerful addition to the analytical strategy for tandem mass spectrometry.

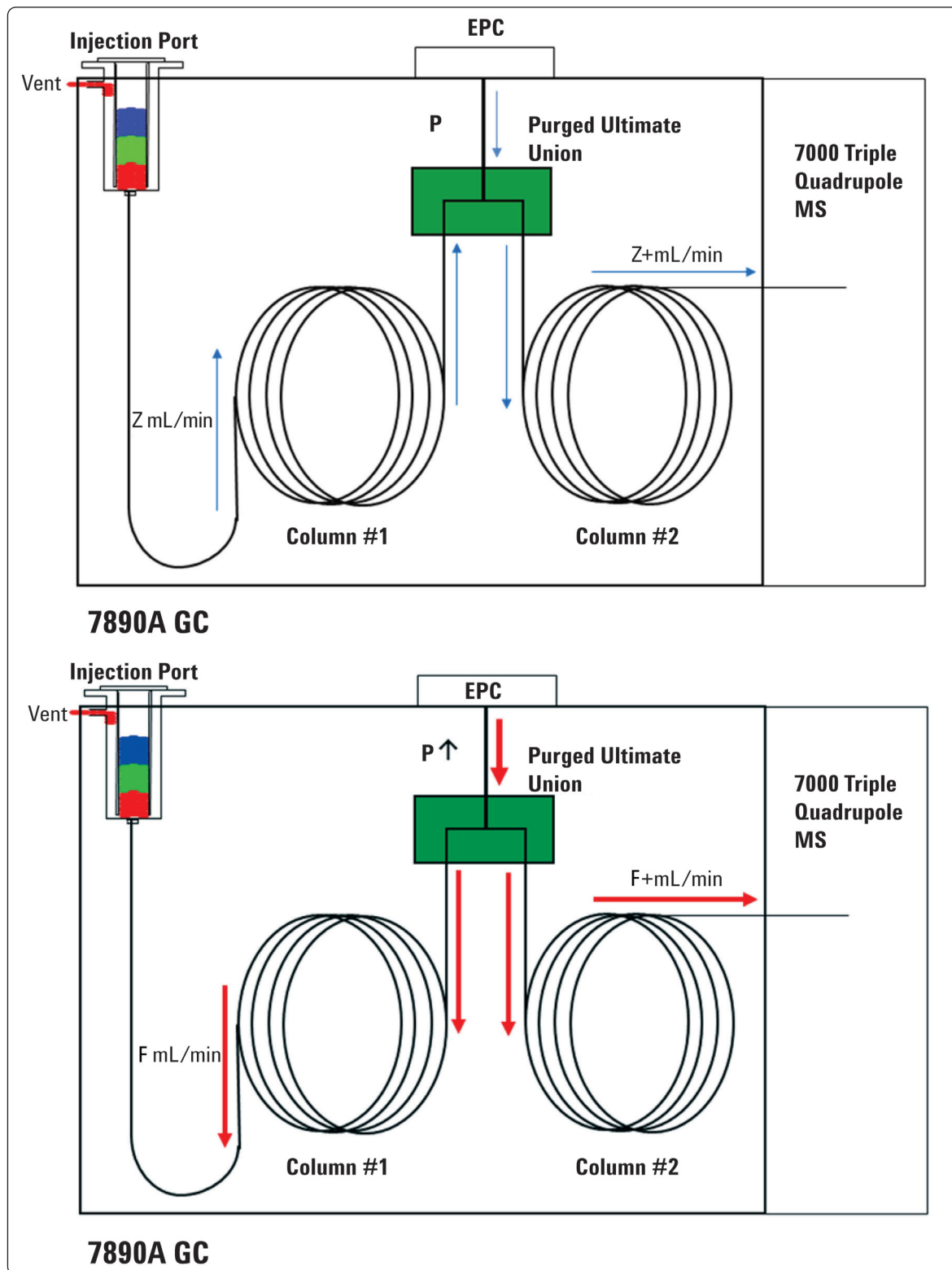


Figure 1 The Pressure Controlled Tee arrangement. Upper panel shows the forward flow during analysis and the lower panel shows the column flows during backflushing.

Table 2. Comparison between PCT and Continuous Column configurations for MRM 272→241 m/z transition for octafluoronaphthalene (OFN). To simplify the comparison, the results for the peak area, height and signal-to-noise (S/N) ratio have been normalized against the standard configuration.

GC configuration	RT variation (s)	Normalized MRM Signal area	Normalized MRM RSD (%)	Normalized MRM Signal height	Normalized MRM RSD (%)	Normalized MRM S/N
Continuous column without PCT	0.063	1	14	1	15	1
With PCT	0.098	1.1	15	1.0	16	1.2

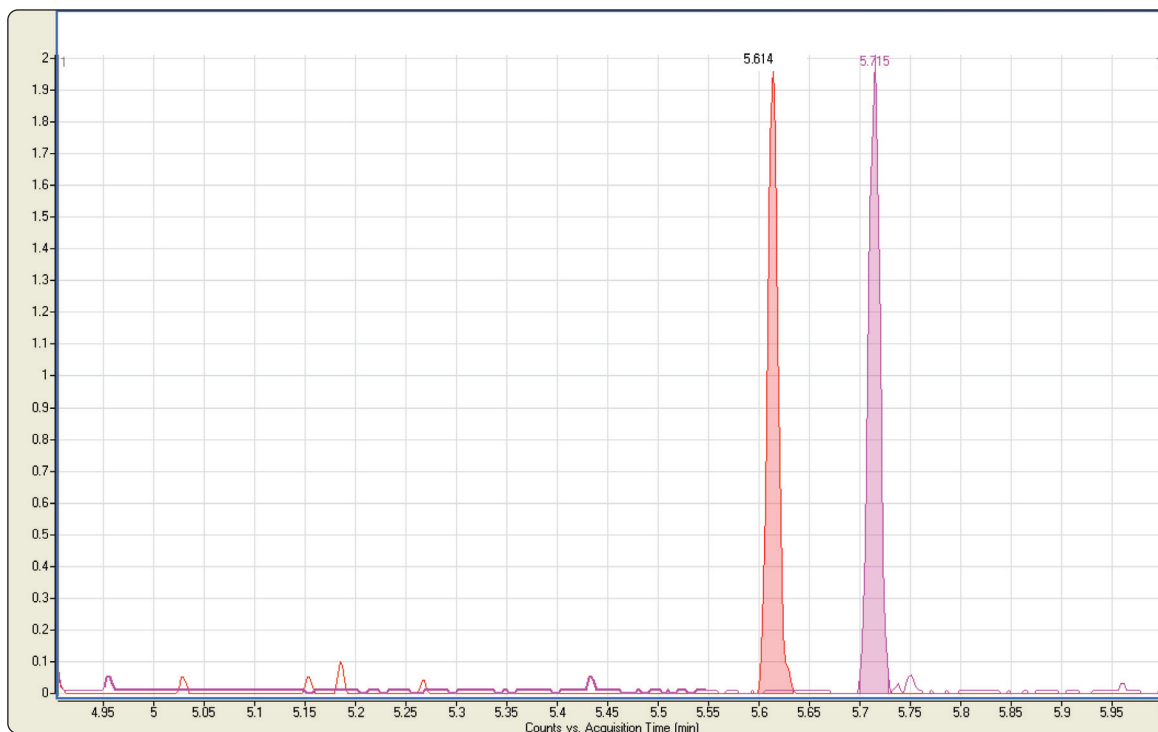


Figure 2. OFN 272 → 241 m/z transition without the PCT (5.614 min) and using the PCT (5.715 min). The same absolute abundance scale is applied to both.

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 "Enhanced Sensitivity for Biomarker Characterization in Crude Oil by GC-SRM," in press
- 4 "The Rapid Universal GC/MS Backflushing Kit," part number G1472A and contains the manual (G1472-90001) which describes this configuration and operation for 5973 or 5975 or 7000 series Mass Selective Detectors.

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Printed in the USA
October 13, 2009
5990-4504EN



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