TECHNICAL NOTE

Gas Chromatography/
Mass Spectrometry

Swafer Micro-Channel Flow Technology – Automated Inlet Selection for GC/MS

Abstract

This technical note describes an automated method for switching back and forth between two injection techniques in a dual-channel capillary column gas chromatograph/mass spectrometer (GC/MS) system. Developments in the micro-fluidic switching technology help analysts to maximize their return on investment. The system under consideration allows the injector/column system coupled to the GC/MS to be switched between a headspace and liquid injection in a completely automated way to maximize the use of this important analytical tool. This is demonstrated in Table 1.

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Table 1. **Section of an Automated Inlet Switching Sequence.**

Injector	Start Time	
VOC Standard 001	Headspace	8:13:35 PM
VOC Standard 002	Headspace	8:43:37 PM
VOC Standard 003	Headspace	9:13:39 PM
VOC Standard 004	Headspace	9:43:41 PM
VOC Standard 005	Headspace	10:13:35 PM
VOC Standard 006	Headspace	10:43:36 PM
VOC Standard 007	Headspace	11:13:34 PM
VOC Standard 008	**Headspace**	**11:43:34 PM**
Jet Fuel 001	**Capillary**	**12:11:32 AM**
Jet Fuel 002	Capillary	12:45:29 AM
Jet Fuel 003	Capillary	1:19:38 AM
Jet Fuel 004	Capillary	1:53:53 AM
Jet Fuel 005	Capillary	2:28:47 AM
Jet Fuel 006	Capillary	3:03:22 AM
Jet Fuel 007	Capillary	3:38:21 AM
Jet Fuel 008	Capillary	4:12:44 AM

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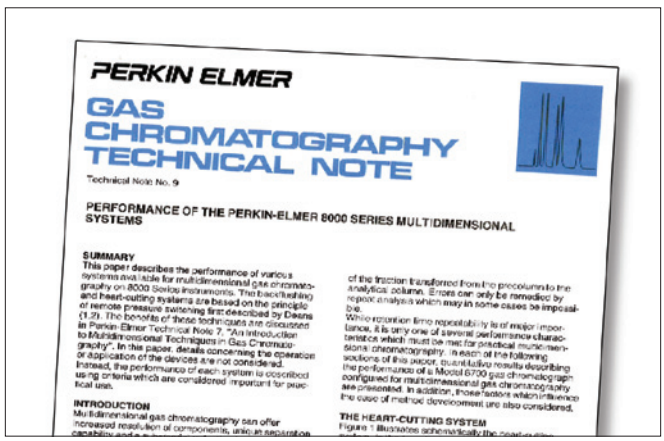
Introduction

GC/MS is considered to be one of the most useful and powerful general-purpose techniques in the analytical laboratory. It represents an ideal system to be coupled to a standard autosampler for liquid injection or other, more specialized sample-introduction systems such as headspace (HS), purge and trap (P&T), pyrolysis, or automated thermal desorption (ATD) systems. The ability to switch quickly and easily between multiple inlet techniques provides optimum application flexibility. For example, the ability to quickly switch from a specialized purge and trap or automated thermal desorption injection to a simple automated liquid injection, or vice versa, could significantly increase laboratory throughput for many users.

This note discusses a column-switching technology with numerous possible configurations that permit techniques such as column backflushing, heartcutting, polarity tuning and detector switching. However, we will limit discussion to the example that has been described above: how do we maximize the use of a GC/MS, so as to automatically switch from one autosampler/injector combination, to another? Although flow-switching technology is far from new, there has been a resurgence of interest in micro-fluidic switching devices to enable techniques such as heartcutting. PerkinElmer first introduced heartcutting devices more than 25 years ago and Figure 1 shows a technical note dating back to 1987, showing the layout of the then-current heartcutting system.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GAS CHROMATOGRAPHY TECHNICAL NOTE

Technical Note No. 9

PERFORMANCE OF THE PERKIN-ELMER 8000 SERIES MULTIDIMENSIONAL SYSTEMS

SUMMARY

INTRODUCTION

THE HEART-CUTTING SYSTEM

Figure 1. A technical note published more than 25 years ago describing PerkinElmer's then-current heartcutting system.

Several technologies have contributed to the growing interest in flow-switching technology. Pneumatic control of the carrier gas using programmable electronic systems provides the user with long-term reproducibility and ultimate ease of use. As will be seen later, this technical note reports a simple set of experiments that puts this claim to an extreme test. Micro-machining using various techniques can generate accurate, reproducible flow channels of any design in a steel substrate. Modern deactivation techniques can then be used to passivate the steel surface, making the channels chromatographically equivalent in inertness to that of fused silica or glass.

The primary concern with all of these typically robust, modern designs concerns the possibility of blockage of the internal (out of sight) sample pathways. This could prove disastrous with the entire switching device having to be replaced due to contamination with particles of either crushed fused silica or ferrules.

The data reported in this technical note was generated utilizing hardware in which the entire 'active switching region' is built into a low-cost, reproducible, exchangeable wafer; even in the worst case scenario, the potential effort to remediate the 'damage' is little greater than changing the filament on a mass spectrometer, or changing the syringe on a liquid autosampler.

Table 2. **Conditions used for Headspace Work.**

Setting	Value
Oven	30 °C for 1 min then 8.0 °C/min to 150 °C then 40 °C/min to 270 °C and hold for 1 min
Injector Used	Programmable Split Splitless at 150 °C, 22.4 psig He, Split flow = 5 mL/min
Injector not in Use	Capillary injector at 280 °C, 21.8 psig He, Split flow = 25 mL/min
MS	Source at 180 °C, Transfer line at 300 °C, 45-280 u at 4 Hz (0.18 sec scan time / 0.07 sec inter-scan delay)
D-Swafer	22.0 psig He
Column	20 m x 0.18 mm x 0.4 µm Elite-1
Headspace	Constant Mode, Vial Shaker ON, Vial Venting OFF
Thermostatting Time	25.00 min
Pressurization Time	3.0 min
Inject Time	0.03 min
Thermostatting Oven	80 °C
Needle Temp	110 °C
Transfer Line Temp	120 °C
Carrier Pressure at HS	25.0 psig

Instrumentation

A PerkinElmer® Clarus® GC/MS system was equipped with a D-Swafer™ Dean's Switch (PerkinElmer Part No. N6520273) configured to allow the user to select which injector of the GC is being used with the mass spectrometer. The switch from utilizing one injector to another is performed entirely within the instrument software and this may be fully automated, so that this switchover can be programmed to occur between injections or sample lists.

This facility could be used to allow liquid samples to be analyzed by GC/MS using different injector types, but in this case, we are using one channel (A) for headspace-GC/MS analysis of VOCs and the second channel (B) for automated liquid injection of aviation jet fuel. Both applications can be performed on a single, non-polar capillary column.

Figures 2 and 3 show the basic D-Swafer hardware arrangement for injector selection. The dimensions of the two restrictors were matched. However, this was not optimized for this application. It is recommended that the restrictor length be chosen with the setup required, based on the optimum pressures or flows required.

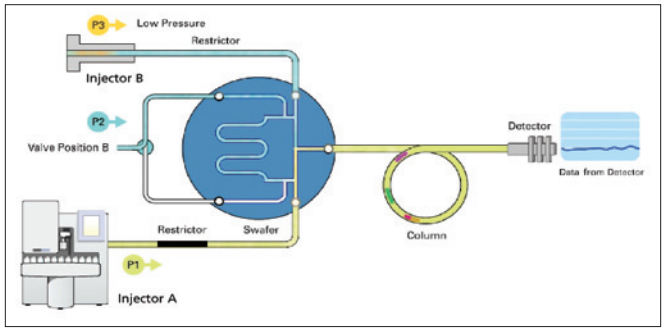

Figure 2. Sample introduced at Injector A reaches the MS detector.

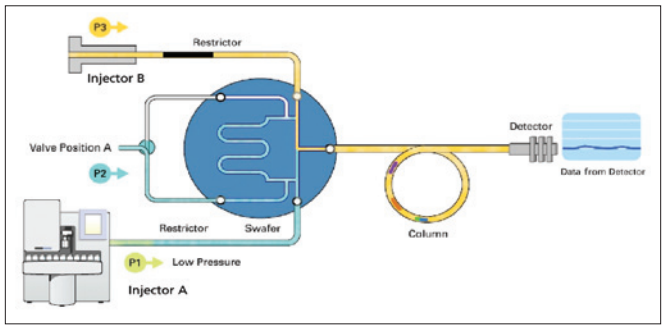

Figure 3. Sample introduced at Injector B reaches the MS detector.

The carrier-gas pressure at the injector that is not in use is usually kept slightly lower than the carrier-gas pressure set at the switching device. This ensures that there is a reverse trickle flow of carrier gas within the restrictor that is not in use. It also ensures that if this injector were contaminated and was being 'baked out', material would not reach either the column or the mass spectrometer. Similarly, if the user decides to lower the injector carrier-gas pressure to a negligible setting and then perform injector maintenance, no air will get to the mass spectrometer.

The dimensions of the restrictors used were: 50.0 cm length of 0.18 mm i.d. fused silica. This was simply a matter of convenience at the time, but the setup would be greatly improved by using 0.10 mm id fused silica. In the current setup, the column installed between the D-Swafer and the MS is a PerkinElmer Elite-1 20 m x 0.18 mm x 0.40 µm. The injector in use was maintained at 0.4 psig higher than the pressure set at the switching device. The pressure set at the injector that was not in use was just 0.2 psig below that set at the D-Swafer. With more appropriate (narrower or longer) fused-silica restrictors, the same flows could be achieved with larger, more-reproducible pressure differences. Nevertheless, this setup does represent a very demanding challenge for this technology and is very revealing of how precisely these small pressure differences can be maintained. Whatever the results achieved with these conditions, it should be fairly easy to improve upon them.

***Table 3.* Conditions used for Automated Liquid Injection Work.**

Setting	Value
Oven	30 °C for 1 min then 10.0 °C/min to 200 °C then 40 °C/min to 280 °C and hold for 5 min
Injector not in Use	Programmable Split Splitless at 150 °C, 21.8 psig He, Split flow = 25 mL/min
Injector Used	Capillary injector at 280 °C, 22.4 psig He, Split flow set by timed events
MS	Source at 180 °C, Transfer line at 300 °C, 45-420 Da at 4 Hz (0.15 sec scan time / 0.10 sec inter-scan delay)
D-Swafer	22.0 psig He
Column	20 m x 0.18 mm x 0.4 µm Elite-1 (PerkinElmer Part No. N9316004)
Timed Events	Split (injector in use) = 250 mL/min @ -0.50 min and 50 mL/min at 0.50 min
Autosampler	0.1 µL injected fast

What follows is a brief description of two quite different applications based on the single hardware configuration that includes the D-Swafer Dean's Switch illustrated in Figures 2 and 3.

VOCs in Water by HS-GC/MS (Injector A) / Analysis of Jet Fuel by Liquid Injection (Injector B)

Figure 4 shows the use of Injector A which is connected to a PerkinElmer TurboMatrix™ HS 110 operated in constant mode. This particular injection is the last in a short sequence of replicates illustrating this analysis. Worthy of note is the date and time of injection – February 12th at 11:43 PM.

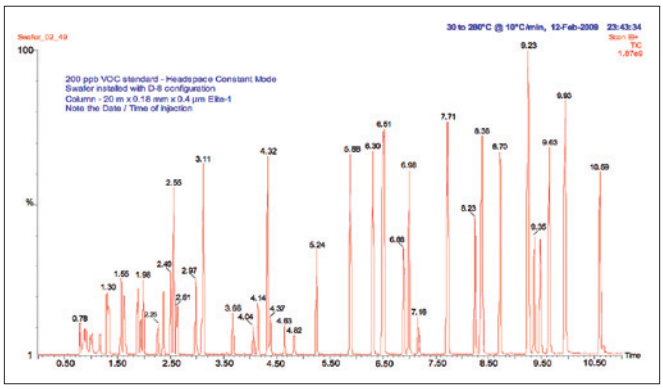

Figure 4. HS-GC/MS analysis of 5.0 mL of 200 ppb VOC standard.

Despite the fact that the pressure drop across the restrictor linking the injector to the D-Swafer device is only 0.4 psig, the chromatography does not appear degraded in either application. In Figure 5, six replicate injections of the VOC standard are presented as an overlay with linked vertical axes. Annotated on each peak is the %RSD based on absolute peak area. The precision is quite reasonable.

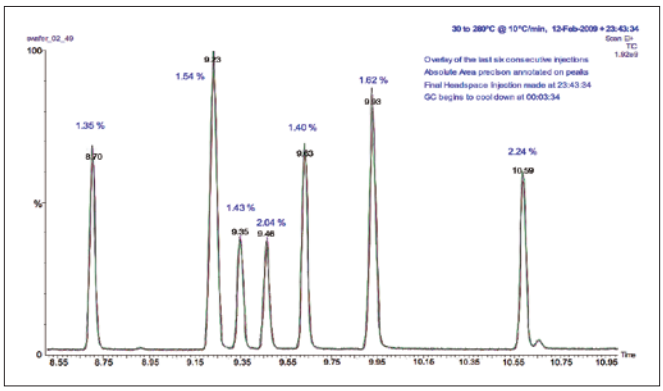

Figure 5. Enlargement of the final six headspace injections with linked vertical axes.

Figure 6 shows the first injection of the jet fuel. The final headspace autosampler injection occurred at 11:43:34 PM and, with an analysis time of exactly 20.0 minutes, this means that the GC oven begins its cool-down from 270 ˚C to 30 ˚C at 00:03:34 AM (of the following day). Despite a 2.0-minute equilibration time, the first analysis of the jet fuel begins less than 8.00 minutes after the headspace analysis ended. This was configured earlier in the day using a sequence of different methods. The sequence was started and the operator left for the day.

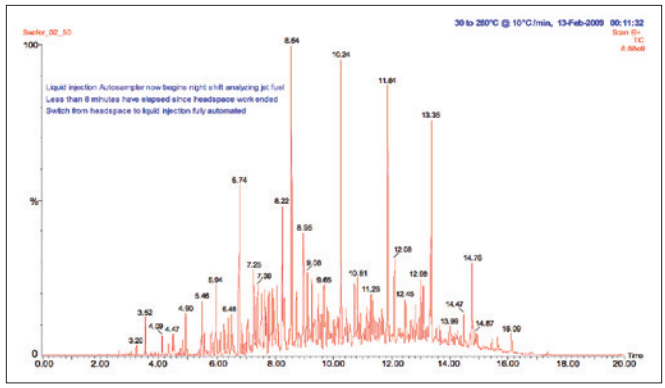

Figure 6. The first liquid injection of jet fuel – introduced less than 8 minutes after the HS work finished.

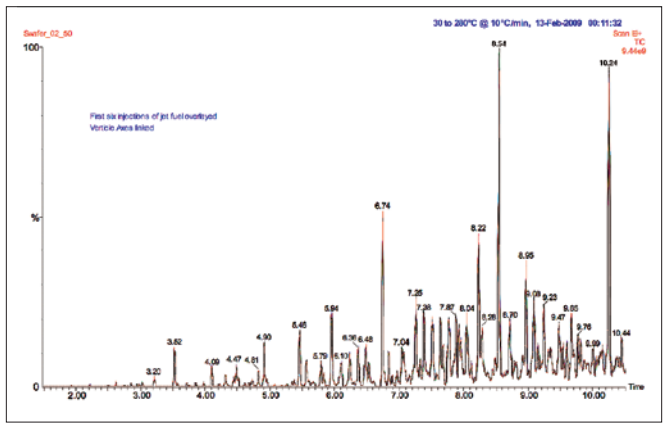

Figure 7. Enlargement of the first six replicate liquid injections of the jet fuel with synchronized vertical axes.

The chromatogram illustrated in Figure 8 shows both the TIC and an extracted mass chromatogram (*m/z* 166) for the analysis of the final headspace injection of the VOC standard. The rather unique signature of tetrachloroethene eluting at 5.24 minutes, is unmistakable. Figure 9 shows the first injection of the jet fuel using the liquid autosampler. There is no evidence of any carryover of the tetrachloroethene indicating that the automated switchover of injection channels is occurring exactly as planned.

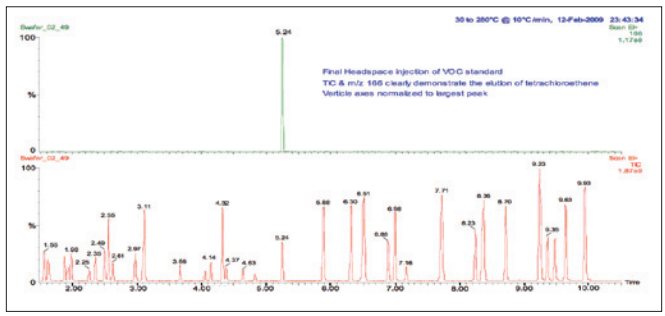

Figure 8. The final injection of the voc standard – TIC plus a mass chromatogram at *m/z* 166.

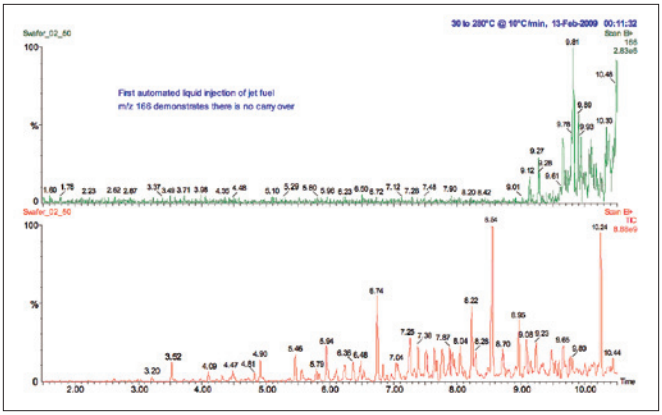

Figure 9. The first injection of the jet fuel – TIC plus a mass chromatogram at *m/z* 166.

Conclusions

The D-Swafer Micro-Channel Flow Technology System enables users to choose between a variety of sample inlet devices in a completely automated fashion. In addition, the D-Swafer demonstrates reliability, precision, and no carryover. The cost of the micro-channel option for the Clarus GC is small relative to the total financial investment in a GC/MS, a HS-GC/MS or an ATD-GC/MS. However, the D-Swafer can make a major contribution to the end users' return on investment.

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