A Better Way to Guard Your GC Column:
An Evaluation of GC Columns with Integrated Guards

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Overview

Removing part of the inlet end of a GC column to maintain its performance is common, but this can reduce the column's performance and its lifetime. Even using fused silica guard columns and column unions can adversely affect the separation. We demonstrate the lifetime and performance advantages of an integrated guard and analytical column design over the use of a separate guard column.

Introduction

Gas chromatography columns can accumulate involatile contaminants, which can affect their chromatographic performance. A typical approach to column maintenance or stage in troubleshooting a separation might involve removing a small amount of the inlet end, with the aim of removing contaminants that may have accumulated with use, and restore the quality of the separation. However, the removal of part of the analytical column also removes some of the stationary phase, which will have an adverse affect on the separation. Instead, a guard column, consisting of a length of fused silica, can be used in front of the analytical column in order to protect this from contamination. The guard is typically connected to the analytical column with a column union. The disadvantage of using such unions is that these can introduce unwanted activity and dead volume into the system, which can adversely affect the peak shape of the analytes.

Thermo Scientific TraceGOLD GC Columns with SafeGuard combine an analytical column and a deactivated fused silica guard column. We have compared the chromatographic results of cutting standard columns and columns fitted with integrated guards. We have also compared the performance of columns with integrated guards and those connected to a separate a fused silica guard with a column union.

Methods

1) The Effect of Cutting a GC Column: Analysis of Polycyclic Aromatic Hydrocarbons (PAHs)

Instrumentation:
Thermo Scientific Trace GC with DSQII Mass Spectrometer detector

Column(s):
1. Thermo Scientific Trace TR-5 30m x 0.25mm x 0.25μm
2. Thermo Scientific TraceGOLD TG-5SILMS 30m x 0.25mm x 0.25μm with SafeGuard (10m)

Septum: BTO 17mm
Liner: Splitless Straight Liner 3mm ID 105mm length
Column ferrules: Graphite ferrules to for 0.25mm ID column
Injection syringe: 10μL Fixed needle 50mm Cone tip

Carrier gas: Helium
Column flow: 1.2mL/min, constant flow
Oven temperature: 80°C hold 1 minute, ramp 40°C/min to 200°C, ramp 15°C/min to 325°C hold 3 minutes

Injector type: Split/Splitless
Injector mode: SSL
Injection details: Splitless time 1.5mins
Injector temperature: 300°C
Detector details: MS detection

MS Conditions:
Instrumentation:
Thermo Scientific DSQII in Selected Ion Monitoring (SIM)
Transfer line temperature: 300°C
Ionization conditions: EI

PAH Test Mix:

Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(ghi)perylene
Chrysene
Dibenzo(a,h)anthracene
Fluoranthene
Fluorine
Indeno(1,2,3-cd)pyrene
Naphthalene
Phenanthrene
Pyrene

2) Sensitivity of SafeGuard vs Standard GC Column: Toothpaste Ingredients

Instrumentation:
Thermo Scientific TRACE GC Ultra with Thermo Scientific ISQ Mass Spectrometer

Columns:
1. Thermo Scientific TG-WAXMS 30m x 0.25mm x 0.25μm with SafeGuard 5m
2. Thermo Scientific TG-WAXMS 30m x 0.25mm x 0.25μm with TraceGOLD capillary guard column 5m x 0.25mm connected with a column union

Septum: BTO, 17mm
Liner: Focus Splitless liner, 5 x 8mm
Column ferrules: 100% Graphite ferrules for TRACE injector/detector
Injection syringe: 10μL Fixed needle syringe for a TriPlus Autosampler
Vials and caps: PTFE lined 2mL Screw Vial Kit

Carrier gas: Helium
Split flow: 20mL/min
Column flow: 1.0mL/min, constant flow
Split ratio: 20:1
Oven temperature: 100°C (1 min), 10°C/min, 250°C (4 min)

Injector type: Split/Splitless
Injector mode: Split
Injector temperature: 250°C

Transfer line Temperature: 250°C
Source Temperature: 230°C
Ionization Mode: EI
Scan Parameters: 29-400 amu
Scan time: 0.25 seconds
Solvent Delay: 4 minutes

Thermo Scientific TriPlus Autosampler
Injection Volume: 1μL
Pre and Post Injection Dwell Time: 5 seconds

Results

1) By removing 13 metres from a standard GC column (without any guard) we reproduced several attempts to improve column performance by trimming the column inlet. Typically, half to one metre of column might be removed in any single column trimming. The removal of 13 metres constitutes a loss of > 40% of the stationary phase, which we would expect to result in an equivalent loss in the same number of theoretical plates or 25% loss in resolution. In this experiment we observed a 21% loss (approx.) in resolution, measured using the separation of phenanthrene and pyrene in a mixture of PAHs (figure 1).

When 13 metres of column were removed from a 30 metre column with an integrated guard we observed an 9 % loss (approx.) in resolution, which is close to our predicted loss of 5%, given that we removed 3m of the 30m total length (10%) of stationary phase.

2) When we compared the same separation of 5 toothpaste components (table 5) on a TraceGOLD™ TG-WAX Column fitted with integrated guard and the same column connected to a separate fused silica guard with a column union, there was a loss of almost half the signal-to-noise for glycerin (figure 3). This is believed to be due to the extra dead volume in the column union.

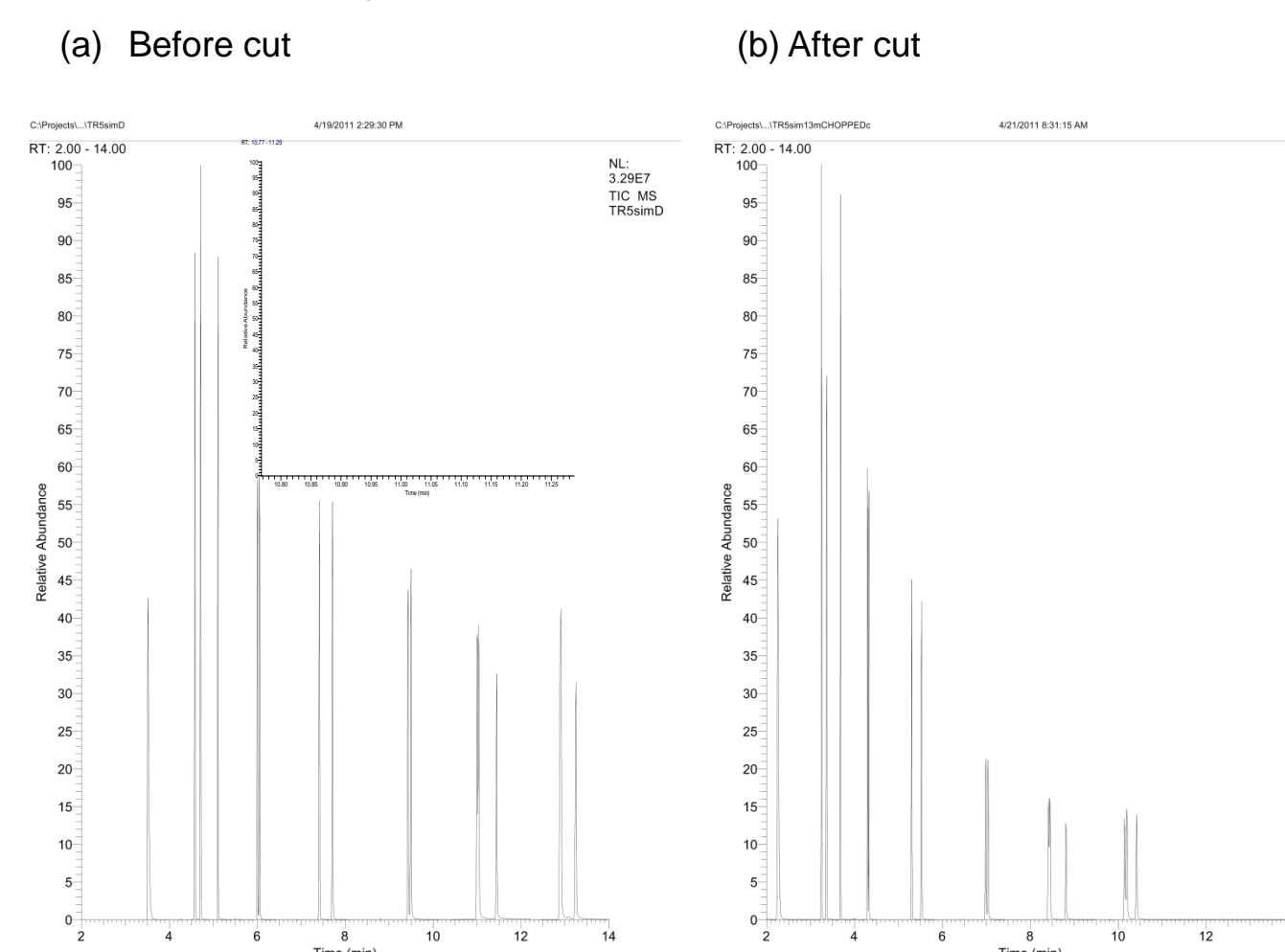FIGURE 1 - Cutting 13m off of a standard column

(a) Before cut   (b) After cut

FIGURE 2 - Cutting 13m off a column fitted with integrated 10m guard
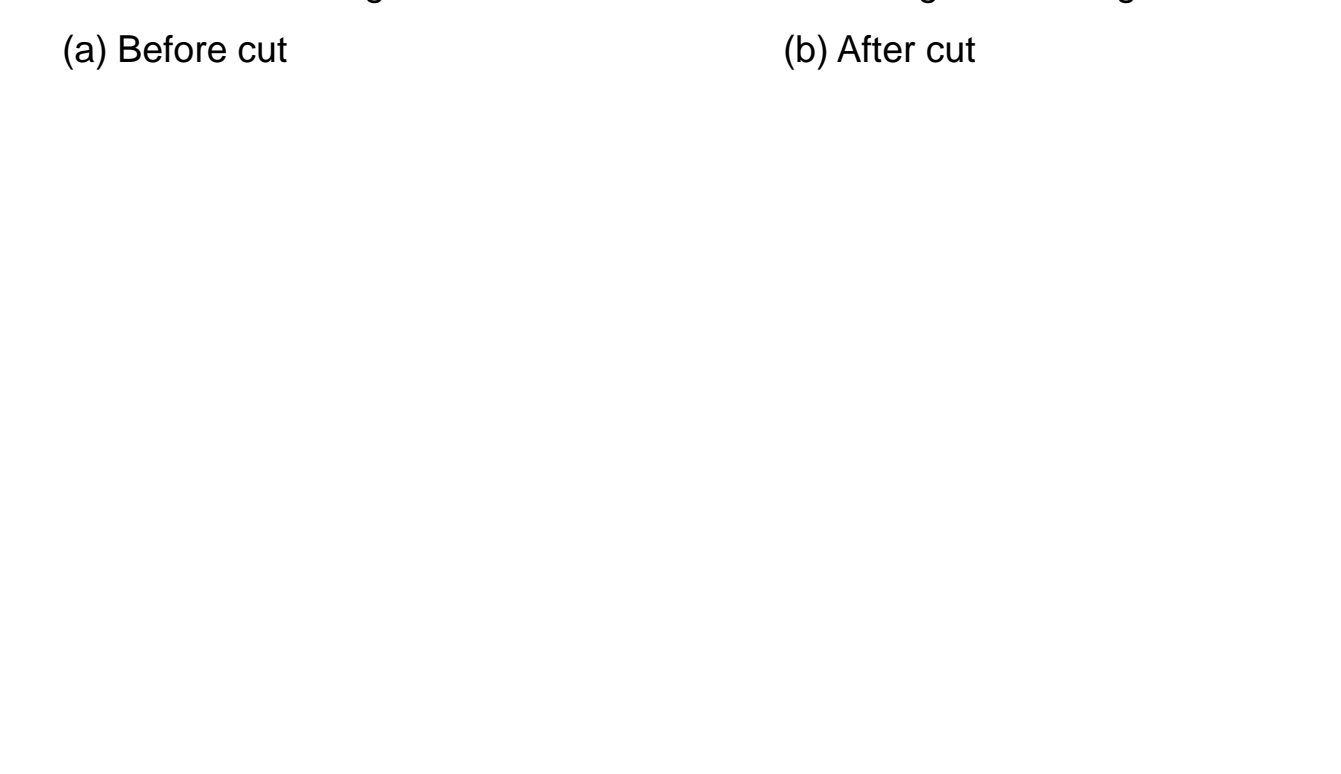
(a) Before cut   (b) After cut

Peak Number	Compound	Tr (min) (Figure 1)	Tr (min) (Figure 2)
1	1,2-Propane diol	5.99	5.71
2	Ethylene Glycol	6.37	6.07
3	1,3-propane diol (IS)	7.99	7.68
4	Diethylene glycol	10.00	9.66
5	Glycerin	13.15	12.90

FIGURE 3 - Comparison of chromatography from a Wax column fitted with a separate guard column, connected with a fused silica column union and a similar column fitted with an integrated guard column

(a) Column with separate guard and column union and (b) with integrated guard

(a) s/n 25 rms

(b) s/n 47 rms
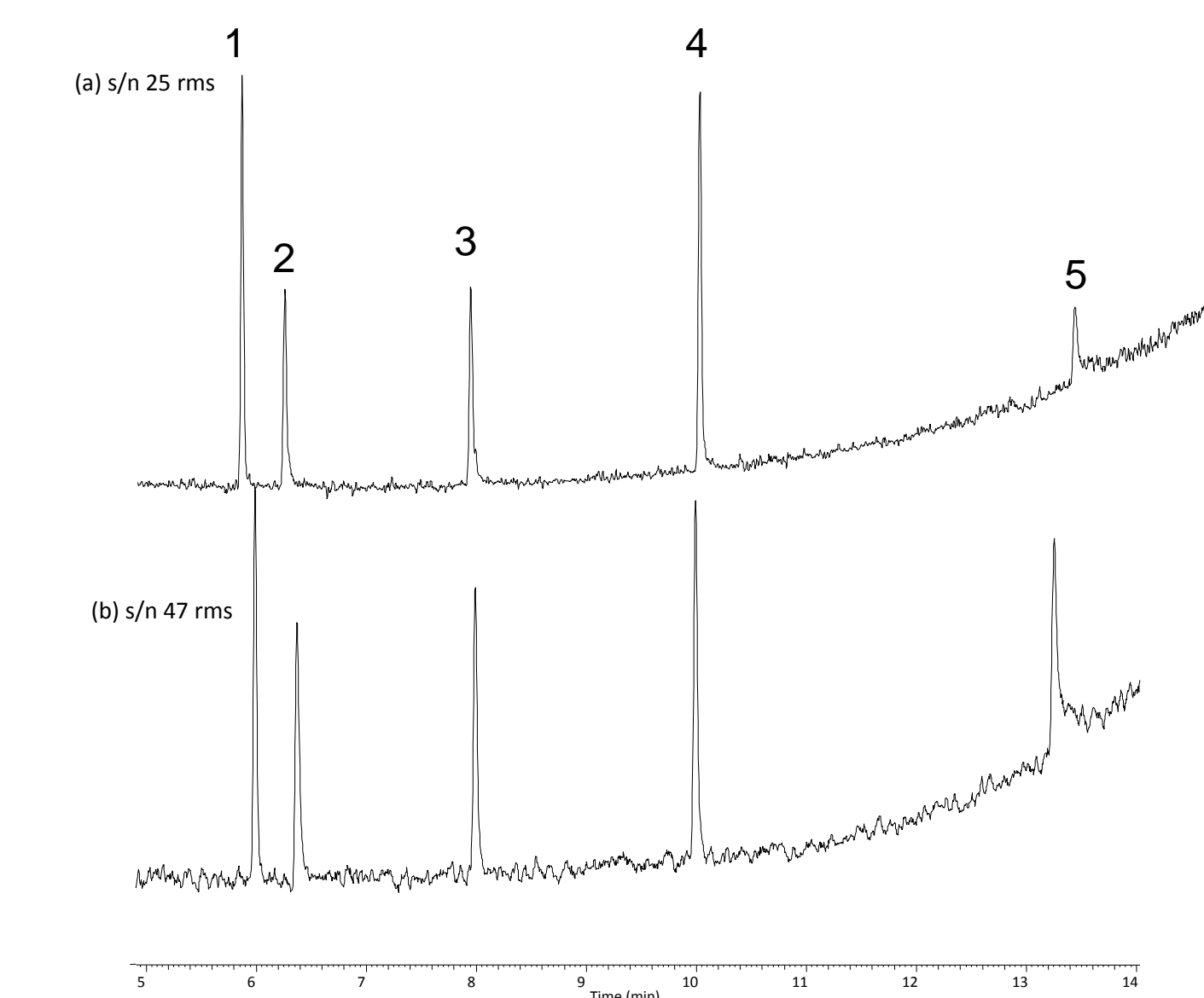
Conclusion

TraceGOLD GC columns with SafeGuard feature an integrated guard column and improve column lifetime by providing the capacity to trim the inlet end of the column without removing the stationary phase and therefore reducing any changes to a separation. The removal of any need to use a column union can also improve peak shape by reducing dead volume, and increases the sensitivity for some compounds by reducing the presence of active silica surface.