Fast Analysis of Phenols Using Conventional GC Instrumentation

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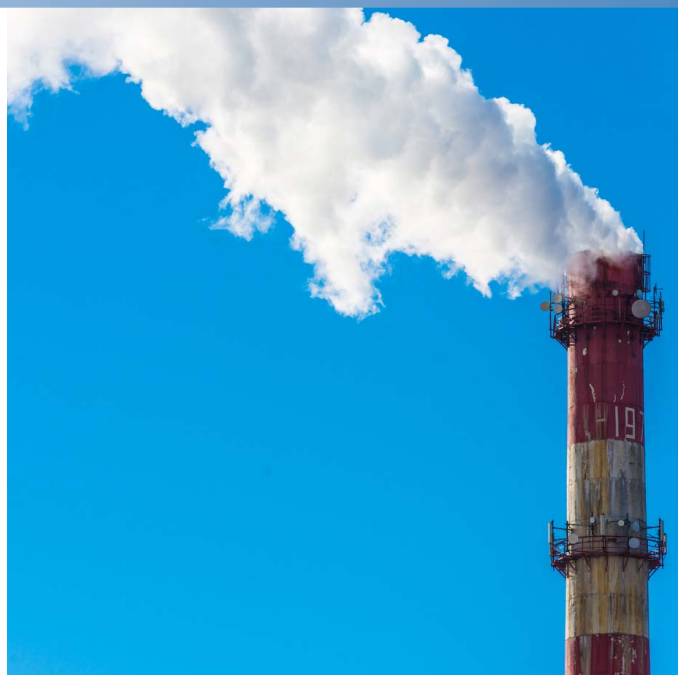

Key Words
Fast GC analysis, Phenols, EPA Method 625, 5% diphenyl dimethyl polysiloxane, Fast GC column, TraceGOLD TG-5MS

Abstract
This application note compares the performance of a 0.15 mm internal diameter (i.d.) GC column with a 0.25 mm i.d. GC column, and demonstrates an increase in speed of analysis for a phenol standard mix according to EPA Method 625. There was no compromise in the separation capability of the method and conversion of the conventional method was easily achieved.

Introduction

An important consideration in many laboratories is speed and sample throughput. A GC separation on a 30 m column with a 0.25 mm i.d. at a 1.0 mL/min flow rate can take 30 minutes or more of analysis time, depending on the mixture of analytes being separated. There are some parameters that can be adjusted in a GC method to reduce run time, including increasing the column temperature, increasing the temperature ramp rate, or reducing the column length. However, these changes can be detrimental to the resolution of the components.

Shorter columns can be used to reduce analysis time without loss of resolution as long as the column diameter is also reduced so that faster mass transfer and better efficiency can be achieved.

There are some practical considerations that need to be considered when reducing column length and internal diameter:

- The ratio of column length to the internal diameter should be kept the same.
- The column stationary phase should remain the same.
- The phase ratio (β) of the columns should be kept the same where possible.

This application note describes the transfer of a method for a phenol standard according to the EPA Method 625 from a standard GC column to a Thermo Scientific™ TraceGOLD™ TG-5MS Fast GC column with an equivalent phase.

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Experimental Details

Consumables		Part Number
Fast GC column:	TraceGOLD TG-5MS, 20 m × 0.15 mm × 0.15 µm	26098-2760
Standard GC column:	Equivalent 5% diphenyl dimethyl polysiloxane, 30 m × 0.25 mm × 0.25 µm	
Injection port septum:	Thermo Scientific 17 mm BTO Septa	31303211
Liner:	Thermo Scientific™ Split FocusLiner™ for 50 mm needle, 5 × 8 × 105 mm	453T1905
Column ferrules:	100% graphite ferrules for Thermo Scientifc™ TRACE™ injector, 0.1–0.25 mm i.d.	29053488
Injection syringe:	50 mm 25s gauge, 10 µL fixed needle syringe for Thermo Scientifc™ TriPlus™ Autosampler	36500525
Vials and closures:	Thermo Scientific 9 mm Wide Opening Screw Thread Vials Convenience Kit, Clear glass vial with blue silicone/PTFE septum	60180-599

Sample Preparation

A stock standard of EPA Method 625 phenol mix was obtained commercially. The standard contained varied concentrations of 500 –2500 µg/mL. This was then diluted ten fold in dichloromethane and used as a working standard solution.

GC Conditions

Instrumentation:	Thermo Scientific TRACE GC Ultra
Injector type:	Split/Splitless
Injector mode:	Split, constant septum purge
Injector temperature:	250 °C
Detector type:	Flame ionization detector (FID)
Detector temperature:	280 °C
Detector air flow:	350 mL/min
Detector hydrogen flow:	35 mL/min
Detector nitrogen flow:	30 mL/min

Data Processing

Software:	Thermo Scientific™ Xcalibur™ software

Method Transfer Equations

The following calculations were used to determine the system parameters required to optimize performance using a TraceGOLD Fast GC column:

$$t_{g2} = t_{g1} \frac{\nu_2}{\nu_1} \frac{\beta_2}{\beta_1} \frac{l_1}{l_2} \qquad T_2 = T_1 \frac{\nu_1}{\nu_2} \frac{\beta_1}{\beta_2} \frac{l_2}{l_1}$$

Where;

t_{g1}, t_{g2}	- temperature gradient for original and new conditions
ν_1, ν_2	- linear velocity of gas for original and new conditions
T_1, T_2	- hold time for isothermal part of separation for original and new conditions
β_1, β_2	- phase ratio for original and new conditions
l_1, l_2	- length of column for original and new conditions

Standard method (I):	Equivalent 5MS phase column 30 m × 0.25 mm × 0.25 µm, β = 250
Carrier gas:	1.2 mL/min helium flow rate, linear velocity 30 cm/s, constant flow
Split injection:	80:1, 1.0 µL
Oven:	60 °C (1 min), 8 °C/min, 240 °C, 23.50 min total run time

New fast method (II):	TG-5MS 20 m × 0.15 mm × 0.15 µm, β = 250
Carrier gas:	0.6 mL/min helium flow rate, linear velocity 30cm/s, constant flow
Split injection:	80:1, 0.5 µL
Oven:	60 °C (0.7 min), 12 °C/min, 240 °C, 15.70 min total run time

New faster method (III):	TG-5MS 20 m × 0.15 mm × 0.15 µm, β = 250
Carrier gas:	1.0 mL/min helium flow rate, linear velocity 43 cm/s, constant flow
Split injection:	80:1, 0.5 µL
Oven:	60 °C (0.5 min), 16 °C/min, 240 °C, 11.75 min total run time

Results

Figure 1 illustrates that the analysis time decreased by 30% on the Fast GC column (II) compared to the standard column (I), with a minimal reduction in resolution of approximately 11%. The method (II) was then further modified by increasing the linear velocity by approximately 40–50%. As a result, the speed of separation was further increased as shown in the faster method (III). Overall, the analysis time was reduced by approximately 50% compared to the original method (I), with no changes to resolution compared to method (II).

The column head pressure on the Fast GC column was 310 kPa (method II) and the standard GC column was 166 kPa (method I) at an oven temperature of 250 °C and linear velocity of 30 cm/s. By increasing the linear velocity to 43 cm/s, the column head pressure increased to 420 kPa on Fast GC column (method III). The increase in performance is gained with an increase in column head pressure but is still within the operating limits of a conventional GC system with a maximum pressure input of 1000 kPa.

Six replicate injections were carried on standard and Fast GC columns, the latter at two linear velocities. The data illustrates excellent retention time reproducibility for all components of the EPA Method 625 phenol standard mix (Table 1).

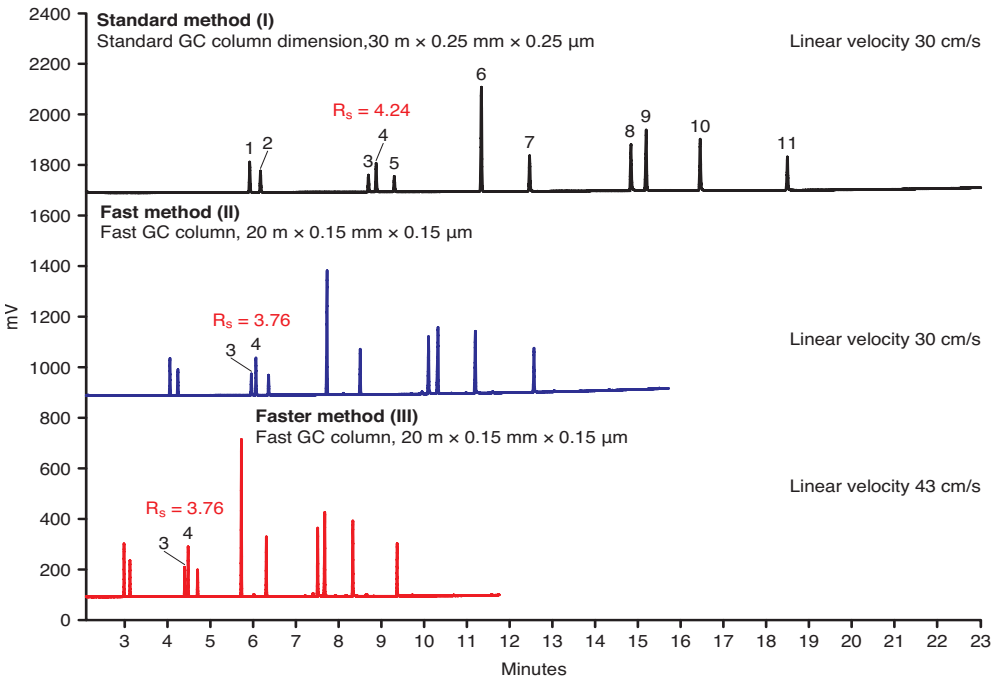

Figure 1: Chromatograms for EPA 625 phenol mix analyzed on a standard GC column and Fast GC column. Average resolution values of six replicate injections were compared on peaks 3 and 4.

Compound	Linear Velocity 30 cm/s				Linear Velocity 43 cm/s	
	Standard GC Column (I) Mean t_R (min)	%RSD (n=6)	Fast GC Column (II) Mean t_R (min)	%RSD (n=6)	Fast GC Column (III) Mean t_R (min)	%RSD (n=6)
1. Phenol	5.92	0.02	4.06	0.01	2.98	0.05
2. 2-Chlorophenol	6.17	0.01	4.24	0.00	3.12	0.06
3. 2-Nitrophenol	8.69	0.01	5.96	0.02	4.40	0.02
4. 2,4-Dimethylphenol	8.87	0.01	6.07	0.01	4.48	0.05
5. 2,4-Dichlorophenol	9.30	0.01	6.36	0.01	4.70	0.01
6. 4-Chloro-3-methylphenol	11.33	0.01	7.73	0.00	5.73	0.01
7. 2,4,6-Trichlorophenol	12.46	0.01	8.50	0.01	6.31	0.01
8. 2,4-Dinitrophenol	14.83	0.01	10.10	0.01	7.51	0.02
9. 4-Nitrophenol	15.19	0.01	10.32	0.01	7.68	0.02
10. 2-Methyl-4,6-dinitrophenol	16.45	0.01	11.19	0.00	8.33	0.02
11. Pentachlorophenol	18.48	0.00	12.56	0.01	9.36	0.01

Table 1: Mean retention time and reproducibility data from six replicate injections

Conclusion

The use of a Fast GC column gave a reduction in the runtime of 30% over a standard GC column following a method transfer, with no changes to the system configuration. Further reduction in run time was observed when linear velocity was increased by 50% with small loss of resolution of approximately 10%. Data on the Fast GC column shows excellent retention time reproducibility at 30 and 43 cm/s linear velocity.

GC analysis time can be reduced by transferring a method to a Fast GC column, without compromising performance, however it is necessary to consider:

- Column length
- Column i.d.
- Column film thickness
- Carrier gas linear velocity
- Temperature ramp rate

This approach has been used to transfer a phenol standard mix according to the EPA 625 from a standard 30 m × 0.25 mm × 0.25 µm GC column to a Fast GC column giving up to 50% faster analysis time, with little change to resolution and with no changes to system configuration.

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