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Tim Anderson was raised in Texas where it was completely too hot, then moved to Pennsylvania and Ohio where it was entirely too cold. He finally settled on California where the weather is just right.

Increased Resolution of Propylene Glycol Impurities with High-Efficiency GC Column Dimensions and Zebron™ ZB-WAX*PLUS*™

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Changes in GC column efficiency can significantly impact separations, even when selectivity is held constant. This work explores the impact of changes in column dimensions and therefore efficiency on column resolution, using propylene glycol impurity testing as an example.

Introduction

Separation of a complex mixture by GC is easiest when using a high efficiency column, as higher efficiency equals greater resolving power. Selectivity of course plays an important role. When there is a large number of structurally-related compounds however, changing from one selectivity to another can be hit or miss. That is, the selectivity change may improve resolution between a certain pair of analytes while simultaneously causing resolution problems in a different area of the chromatogram.

The inherent characteristics of a particular sample may limit the pool of compatible stationary phase selectivities to choose from. In such cases, it is important to choose high efficiency GC column dimensions for the best separation the phase can provide. To maximize efficiency for a GC column, the dimensions (including length, internal diameter (ID) and film thickness) each have a significant effect. A simplified explanation of how this is accomplished is highlighted below:

- Increasing column length proportionally increases column efficiency. For example, the longer the column the greater the resolving power.
- Decreasing the column ID increases column efficiency. The reason for this is that an analyte travelling through the column will spend more time interacting with the stationary phase because there is less distance between column walls.
- Decreasing film thickness increases column efficiency. A thinner film thickness causes greater mass transfer, resulting in less analyte diffusion from injection to detection.

Materials and Methods

The propylene glycol impurity sample was supplied by Archer Daniels Midland, Chicago, Illinois, USA.

Experimental Conditions

GC/FID analysis was performed using a Zebron ZB-WAX*PLUS* GC column on an Agilent® 6890 GC system (Agilent Technologies, Palo Alto, CA, USA) and a Shimadzu® GC-2010 Plus GC system (Shimadzu, Maryland, CA, USA). Helium was used as the carrier gas. Small adjustments between comparative methods were made to account for changes in retention. Details of each method can be seen in **Tables 1** and **2**.

Results and Discussion

As an example, propylene glycol impurity testing analyzes multiple structurally-related compounds that are best resolved with a high-efficiency Zebron ZB-WAX*PLUS* GC column. The sample matrix is mostly aqueous, which immediately rules out the use of non-aqueous-stable stationary phases. The ZB-WAX*PLUS* polymer is stabilized for aqueous injections during manufacturing; multiple or routine injections will therefore not alter the efficiency or polarity of the phase over time and makes it ideal for this testing.

Chromatogram A of **Figure 1** shows the relatively poor resolution of three pentanediol isomers obtained with a 60 meter x 0.53 mm x 1.00 µm ZB-WAX*PLUS* column. By simultaneously decreasing both the column ID and the film thickness, the resolution significantly improves as shown in chromatogram B.

To go one step further, the assay was then transferred to a 60 meter x 0.25 mm x 0.15 µm ZB-WAX*PLUS* column. As shown in **Figure 2**, the higher-efficiency column dimensions result in greater resolution between the low molecular-weight alcohols (methanol and 2-propanol) and dramatically improves resolution between 2-methyl-1-propanol and 1,4-dioxane, which were previously co-eluting.

These propylene glycol impurity examples demonstrate that higher efficiency column dimensions can drastically improve resolution between closely eluting peaks. However, it is important to keep in mind that higher efficiency column dimensions are routinely applied to improve assays with sufficient resolution for additional benefits. For instance, transferring to a narrower ID and thinner film thickness allows the analyst to use a shorter column, resulting in a faster analysis time.

Conclusion

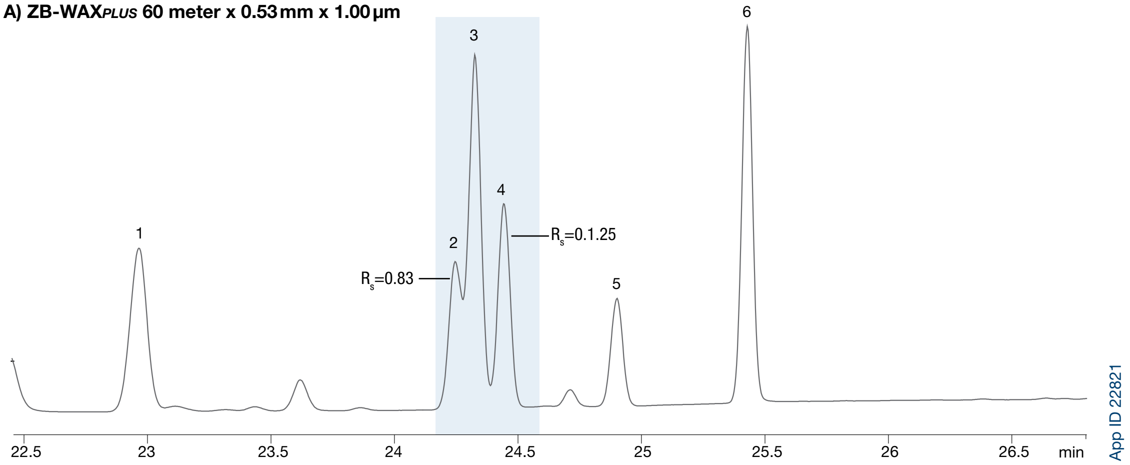
Regardless of selectivity, using higher efficiency GC column dimensions provides numerous benefits. These benefits are obvious during method development, since it makes difficult separations less challenging. But, the benefits are also highly applicable to routine testing methods, since high efficiency GC column dimensions can result in faster analysis times and lower detection limits.

Table 1. Analytical conditions used to collect column efficiency comparison data for pentanediol isomers.

	Chromatogram A	Chromatogram B
Column:	Zebron ZB-WAX*PLUS*™	Zebron ZB-WAX*PLUS*
Dimensions:	60 meter x 0.53 mm x 1.00 µm	60 meter x 0.32 mm x 0.50 µm
Part No.:	7KK-G013-22	7KM-G013-17
Injection :	Split 40:1 @ 240 °C, 1 µL	Split 40:1 @ 240 °C, 1 µL
Oven Program:	100 °C for 4 min to 120 °C @ 50 °C/min for 3 min to 140 °C @ 5 °C/min for 10 min to 220 °C @ 20 °C/min for 12 min	100 °C for 5.5 min to 150 °C @ 30 °C/min for 15 min to 240 °C @ 50 °C/min for 18 min
Carrier Gas:	Helium @ 25 cm/sec (constant flow)	Helium @ 25 cm/sec (constant flow)
Detector:	FID @ 250 °C	FID @ 250 °C
Instrument:	Agilent® 6890	Shimadzu® GC-2010 Plus
Sample:	1. 2,3-Pentanediol-2 2. 2,4-Pentanediol-1 3. 1,2-Butanediol 4. 2,4-Pentanediol-2 5. 2,3-Hexanediol 6. 2,2,2-Trichloroethanol 7. 1,3-Butanediol	1. 2,3-Pentanediol-2 2. 2,4-Pentanediol-1 3. 1,2-Butanediol 4. 2,4-Pentanediol-2 5. 2,3-Hexanediol 6. 2,2,2-Trichloroethanol 7. 1,3-Butanediol

Figure 1. Resolution comparison of pentanediol isomers between (A) 60 meter x 0.53 mm x 1.00 µm and (B) 60 meter x 0.32 mm x 0.50 µm ZB-WAX*PLUS* GC columns.

A) ZB-WAX*PLUS* 60 meter x 0.53 mm x 1.00 µm



B) ZB-WAX*PLUS* 60 meter x 0.32 mm x 0.50 µm

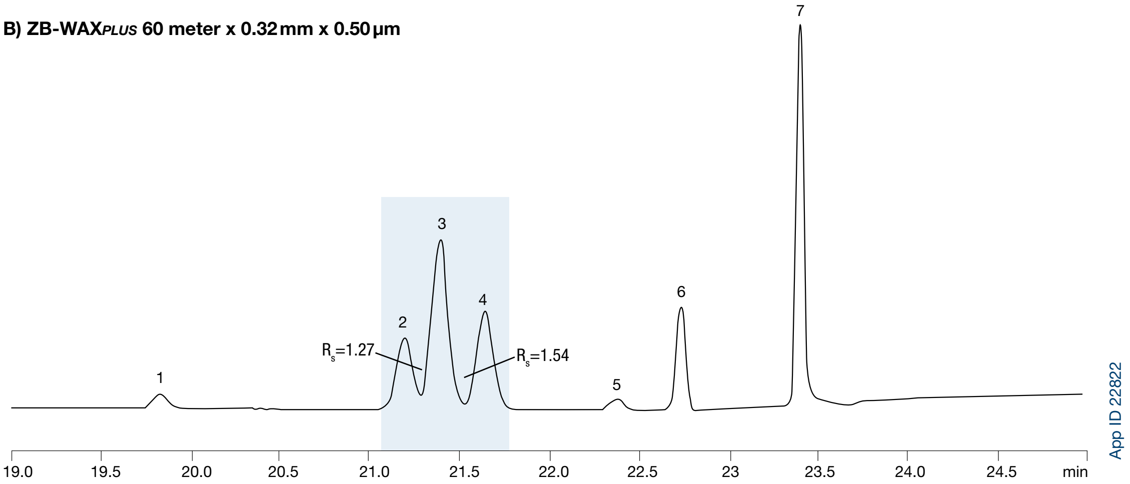
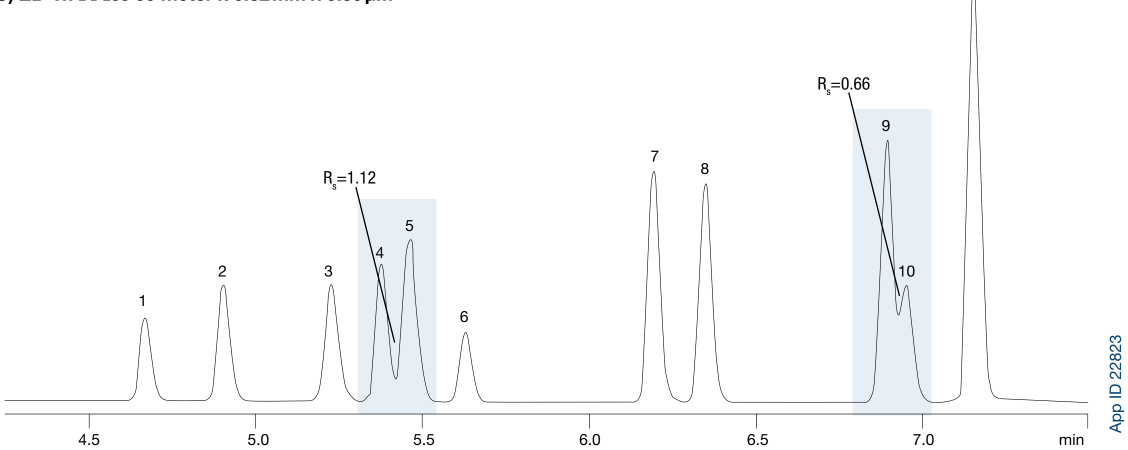


Table 2. Analytical conditions used to collect column efficiency comparison data for pentanediol isomers.

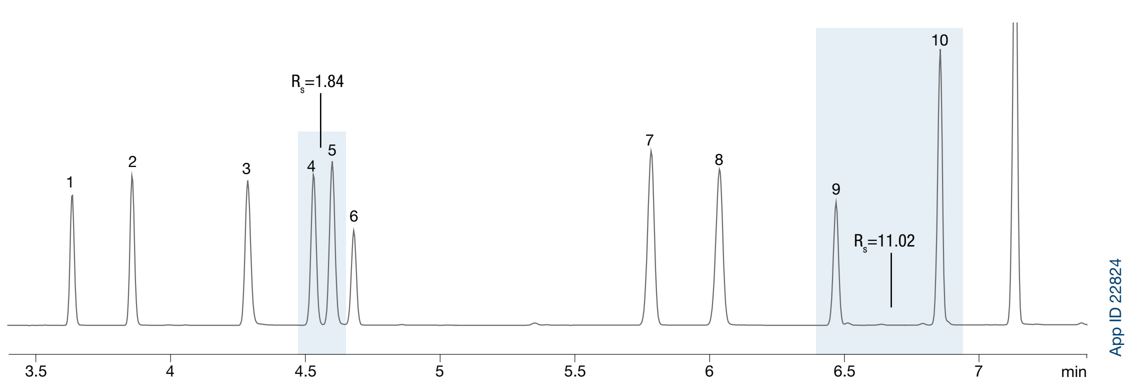
	Chromatogram C	Chromatogram D
Column:	Zebron ZB-WAX*PLUS*™	Zebron ZB-WAX*PLUS*
Dimensions:	60 meter x 0.32 mm x 0.50 µm	60 meter x 0.25 mm x 0.15 µm
Part No.:	7KM-G013-17	7KG-G013-05
Injection :	Split 40:1 @ 240 °C, 1 µL	Split 80:1 @ 240 °C, 0.2 µL
Oven Program:	100 °C for 5.5 min to 150 °C @ 30 °C/min for 15 min to 240 °C @ 50 °C/min for 18 min	60 °C for 6 min to 120 °C @ 50 °C/min for 10 min to 220 °C @ 20 °C/min for 12 min
Carrier Gas:	Helium @ 25 cm/sec (constant flow)	Helium @ 30 cm/sec (constant flow)
Detector:	FID @ 250 °C	FID @ 250 °C
Instrument:	Shimadzu® GC-2010 Plus	Agilent® 6890
Sample:	1. Propylene oxide 2. Acetone 3. Methanol 4. 2-Propanol 5. Ethanol 6. Unknown (Impurity) 7. 2-Butanol 8. 1-Propanol 9. 2-Methyl-1-propanol 10. 1,4-Dioxane 11. Allyl alcohol	1. Propylene oxide 2. Acetone 3. Methanol 4. 2-Propanol 5. Ethanol 6. Unknown (Impurity) 7. 2-Butanol 8. 1-Propanol 9. 2-Methyl-1-propanol 10. 1,4-Dioxane 11. Allyl alcohol

Figure 2. Resolution comparison of pentanediol isomers between (C) 60 meter x 0.32 mm x 0.50 µm and (D) 60 meter x 0.25 mm x 0.15 µm ZB-WAX*PLUS* GC columns.

C) ZB-WAX*PLUS* 60 meter x 0.32 mm x 0.50 µm



D) ZB-WAX*PLUS* 60 meter x 0.25 mm x 0.15 µm



Ordering Information

Zebron ZB-WAX*PLUS*™ GC Columns

ID(mm)	df(µm)	Temp. Limits °C	Part No.
10-Meter			
0.10	0.10	20 to 250/260	7CB-G013-02
15-Meter			
0.25	0.25	20 to 250/260	7EG-G013-11
0.53	1.00	20 to 230/240	7EK-G013-22
20-Meter			
0.18	0.18	20 to 250/260	7FD-G013-08
30-Meter			
0.25	0.25	20 to 250/260	7HG-G013-11
0.25	0.50	20 to 250/260	7HG-G013-17
0.32	0.25	20 to 250/260	7HM-G013-11
0.32	0.50	20 to 250/260	7HM-G013-17
0.32	1.00	20 to 230/240	7HM-G013-22
0.53	1.00	20 to 230/240	7HK-G013-22
60-Meter			
0.25	0.15	20 to 250/260	7KG-G013-05
0.25	0.25	20 to 250/260	7KG-G013-11
0.25	0.50	20 to 250/260	7KG-G013-17
0.32	0.25	20 to 250/260	7KM-G013-11
0.32	0.50	20 to 250/260	7KM-G013-17
0.53	1.00	20 to 230/240	7KK-G013-22

* Thcker films (≥1.0 µm df) are rated to 230/240 °C.

Note: If you need a 5 in. cage, simply add a (-B) after the part number, e.g., 7HG-G013-11-B. Some exceptions may apply. Agilent 6850 and some SRI and process GC systems use only 5 in. cages.

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guarantee

If Zebron GC columns do not provide you with equivalent separations as compared to any other GC column of the same phase and dimensions, return the column with comparative data within 45 days for a FULL REFUND.

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