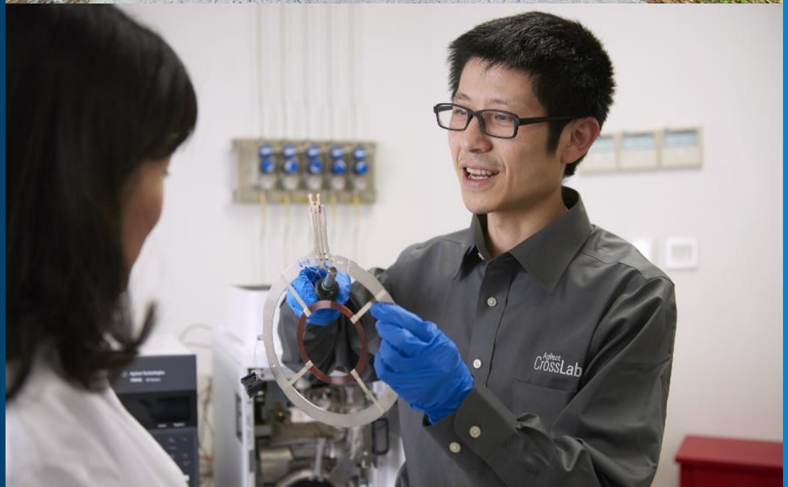
Overwhelmed with Too Many GC Column Options? Let Us Help



Mark Sinnott
Online Application Engineer
August 30th , 2023

JW Column Portfolio- DB, HP, CP, VF

Low Polarity			Mid Polarity			High Polarity		
CP-Sil 2	DB-1ms UI HP-1ms UI	DB & HP-5ms UI	DB-XLB	DB-225ms	DB-ALC1	HP-88	DB-WAX	DB-WAX UI
DB-MTBE	DB-1ms HP-1ms	DB & HP-5ms	VF-Xms	DB-225	DB-Dioxin	CP-Sil 88	DB-WAXetr	DB-HeavyWAX
CP-Select CB MTBE	VF-1ms	VF-5ms	DB-35ms UI	CP-Sil 43 CB	DB-200	DB-23	HP-INNOWax	DB-FATWAX UI
	DB & HP-1	DB & HP-5	DB & VF-35ms	VF-1701ms	VF-200ms	VF-23ms	VF-WAXms	
	CP-Sil 5 CB	CP-Sil 8 CB	DB & HP-35	DB-1701	DB-210		CP-Wax 57 CB	
	Ultra 1	Ultra 2	DB & VF-17ms	CP-Sil 19 CB	DX-4		DB-FFAP HP-FFAP	
	DB-1ht	VF-DA	DB-17	HP-Blood Alcohol			DB-WAX FF	
	DB-2887	DB-5.625	HP-50+	DB-ALC2			CP-FFAP CB	
	DB-Petro/PONA	DB & VF-5ht	DB-17ht	DX-1			CP-WAX 58 FFAP CB	
	CP-Sil PONA CB	CP-Sil PAH CB	DB-608				CP-Wax 52 CB	
	DB-HT SimDis	Select Biodiesel	DB-TPH				CP-WAX 51	
	CP-SimDis	SE-54	DB-502.2				CP-Carbowax 400	
	CP-Volamine		HP-VOC				Carbowax 20M	
	Select Mineral Oil		DB-VRX				HP-20M	
	HP-101		DB-624				CAM	
	SE-30		VF-624ms				CP-TCEP	
			CP-Select 624 CB					
			DB-1301					
			VF-1301ms					
			CP-Sil 13 CB					

Agilent J&W has over 50 different stationary phase offerings

Things to Consider When Choosing a Column

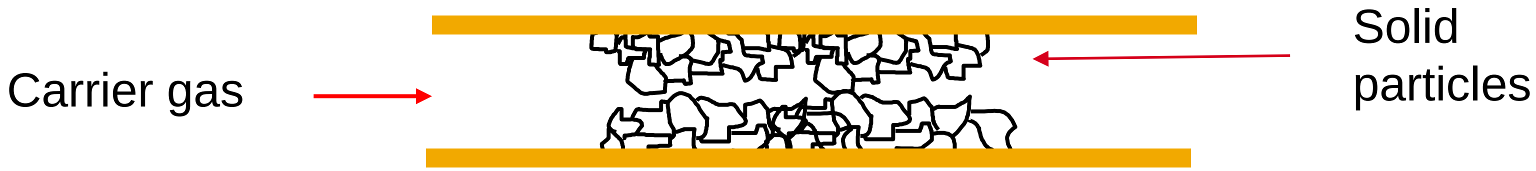
- Is it volatile enough to chromatograph by GC?
- Is it thermally stable?
- Is it a gas or a liquid?
- How is the sample being introduced?
- What is the sample matrix?
    - Can we do sample cleanup?
- Is it an established method?
- What do we know about the analytes?
- What else *may* be present in the sample?

Stationary Phase Selection

- Existing information
  - Literature search
- Selectivity
- Polarity
- Critical separations
- Temperature limits
  - Consider all columns in the GC oven!

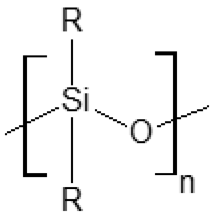
Capillary Column Types

Porous Layer Open Tube (PLOT)



Wall Coated Open Tube (WCOT)

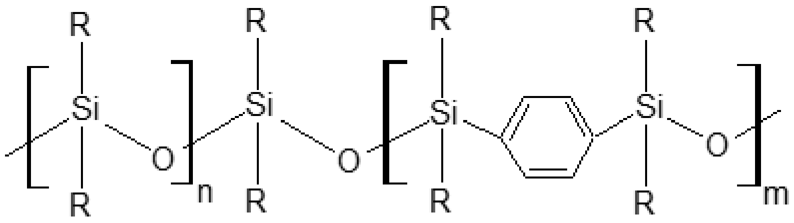
Stationary Phase Polymers



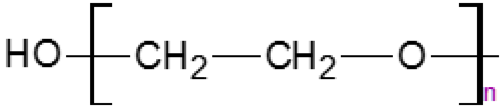
Siloxane

R= methyl, phenyl, cyanopropyl, trifluoropropyl

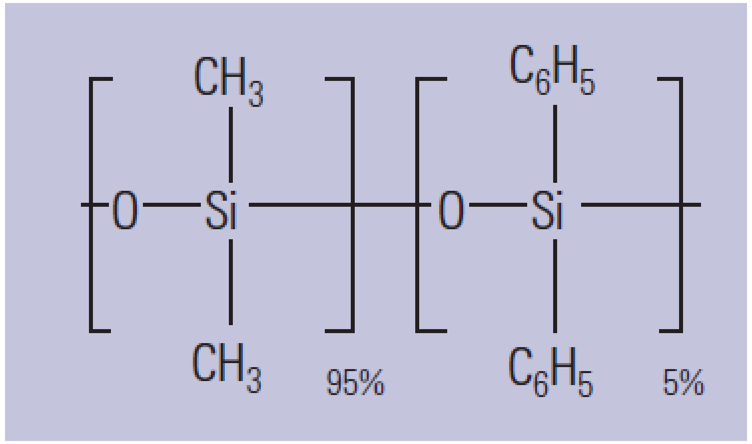
% = number of sites on silicon atoms occupied



Silarylene backbone



Polyethylene glycol

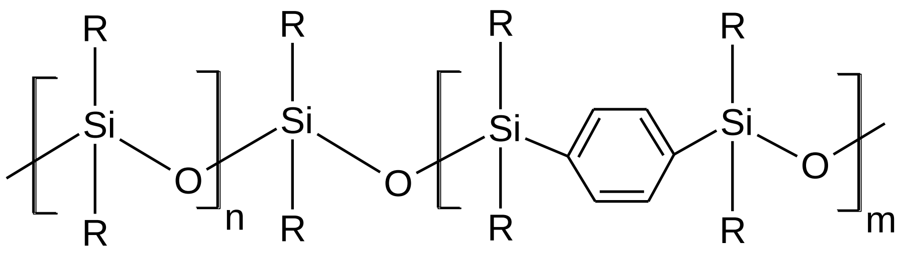


Structure of Agilent J&W HP-5ms

5% phenyl / 95% methyl

Low Bleed Phases-("ms Phases")

- Phases tailored to 'mimic' currently existing polymers
  Examples: DB-5ms, DB-35ms, DB-17ms, VF-1701ms



Siarylene backbone

- New phases unrelated to any previously existing polymers
  Examples: DB-XLB

- Optimized manufacturing processes
  Examples: DB-1ms, HP-1ms, HP-5ms, VF-5ms

Stationary Phase Bleed

A thermodynamic equilibrium process that occurs to some degree in all columns, and is proportional to the mass amount of stationary phase inside the capillary tubing/carrier gas flow path

Polysiloxane backbone releases low molecular weight, cyclic fragments

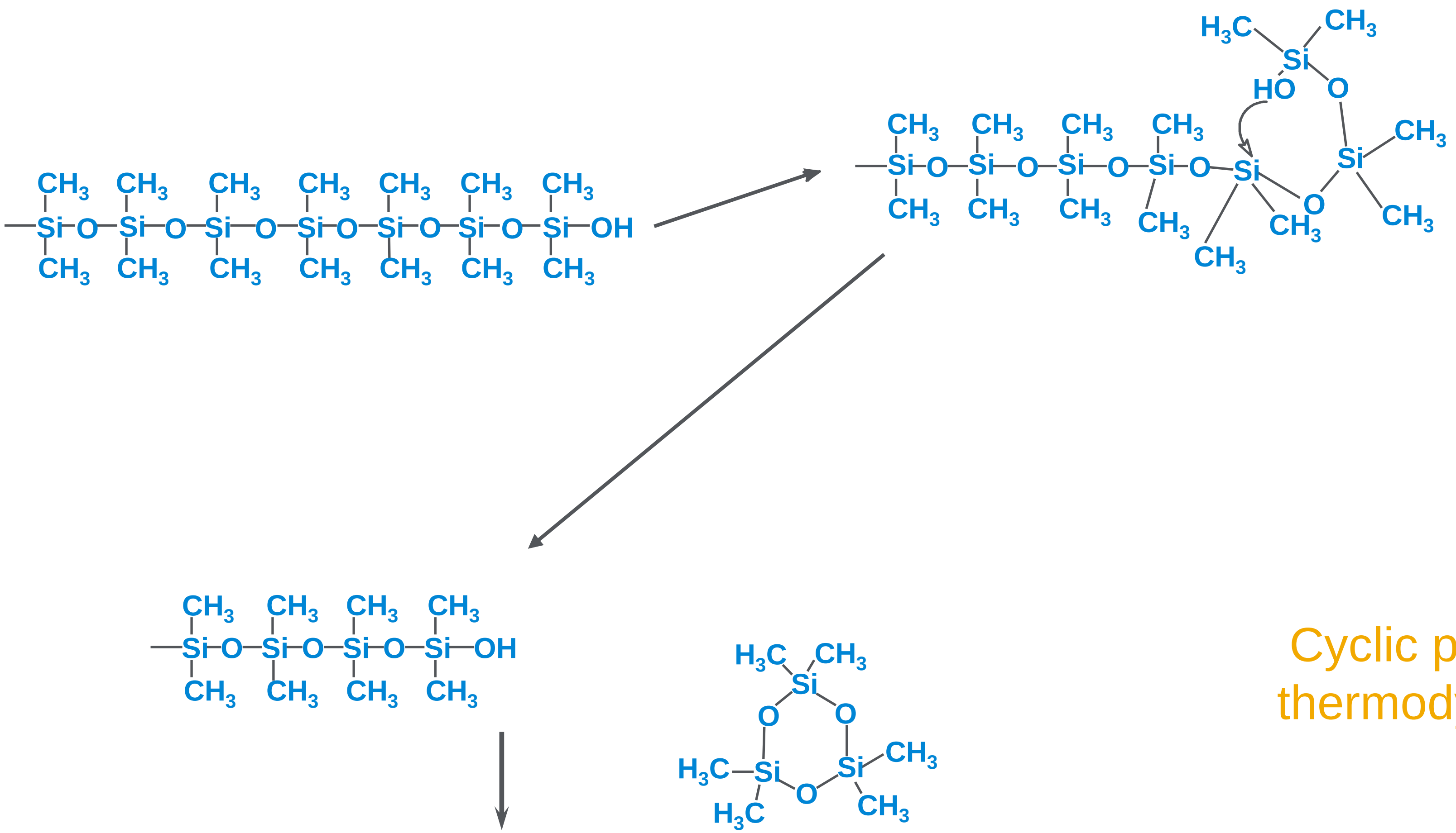
Is negligible in low temperature

Increased by increased temperature, oxygen exposure, or chemical damage

It does not manifest as discreet peaks!

What is Column Bleed?

“Back biting” mechanism of product formation

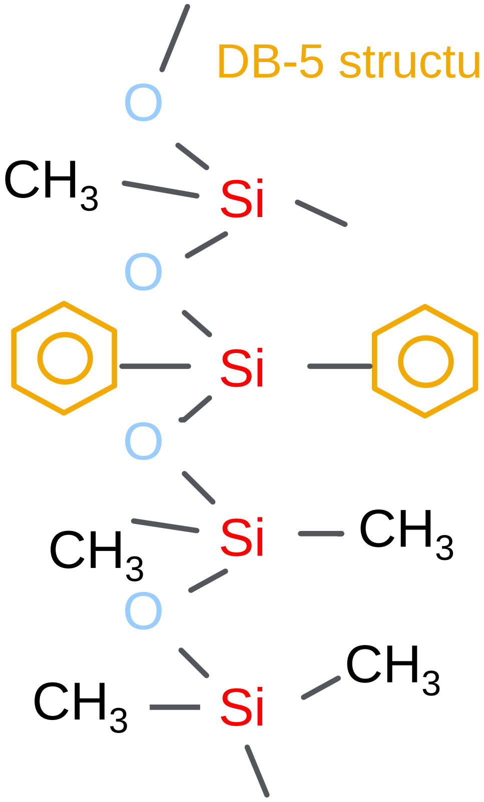


Repeat

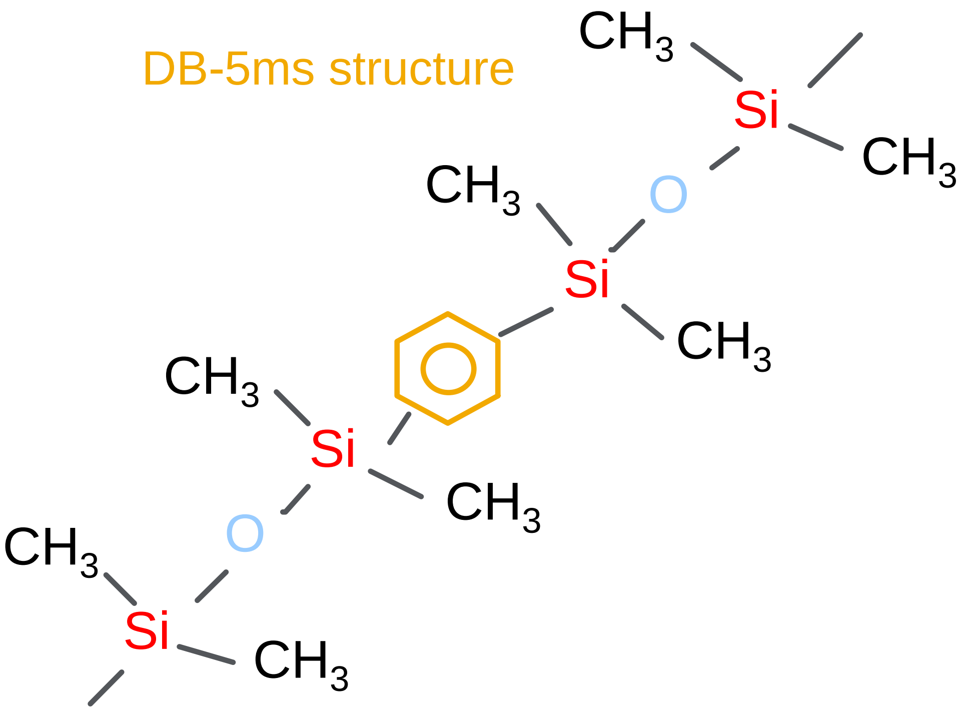
Cyclic products are more thermodynamically stable!

Agilent J&W DB-5ms Structure

DB-5 structure



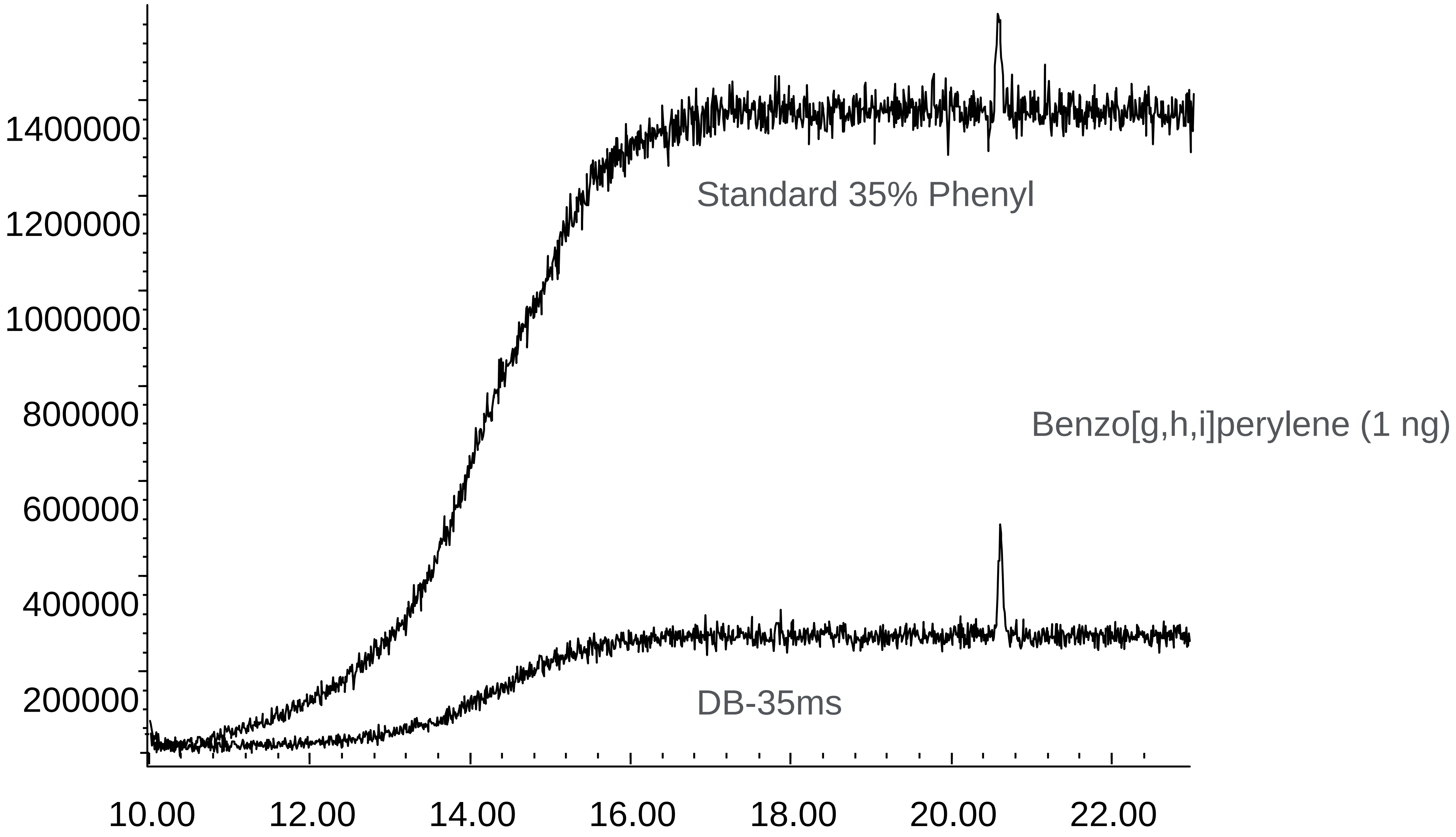
DB-5ms structure



DB-5ms:

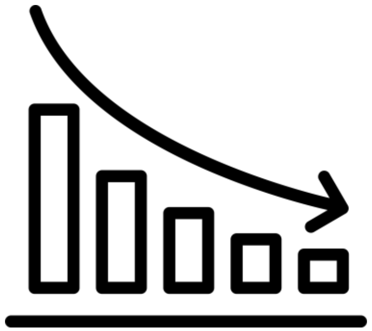
- Increased stability
- Stiffer polymer backbone reduces back-biting
- ***Different selectivity***
- Optimized to match DB-5 as much as possible

Comparison of Agilent J&W DB-35ms Versus Standard DB-35



Polyethylene Glycol/WAX Phases

$$HO-(CH_2-CH_2-O)-H$$

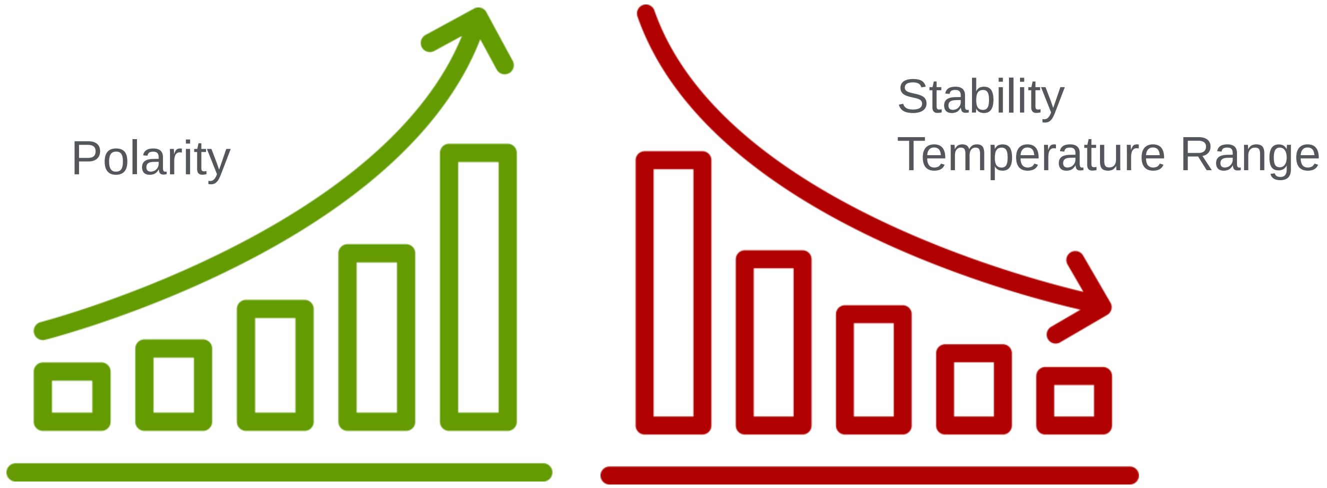


Less Stable than Polysiloxanes

Unique Separation Characteristics

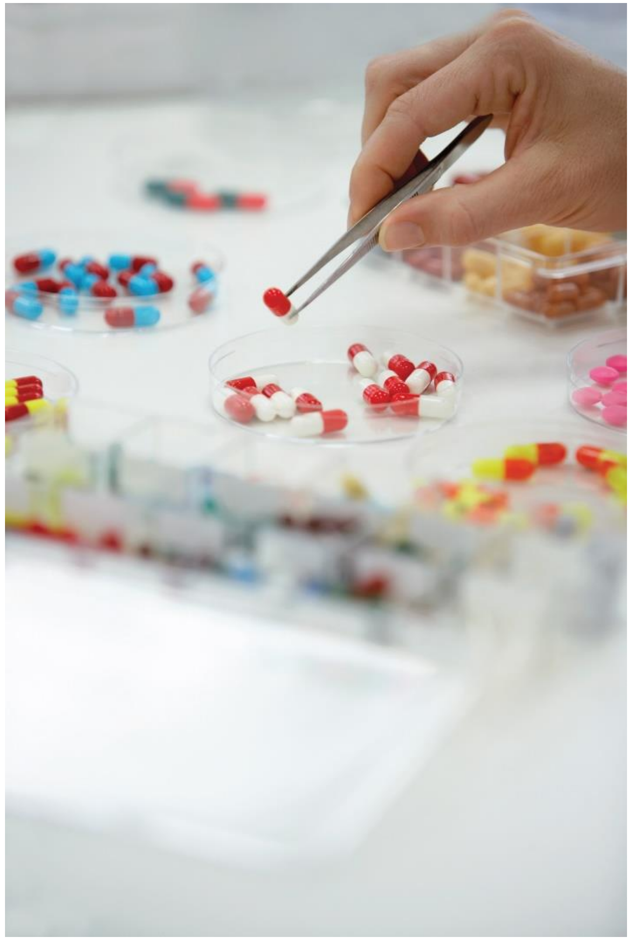
DB-WAX UI
DB-HeavyWAX
DB-FATWAX UI
VF-WAXms
CP-WAX CB

Polarity vs. Stability



Selectivity

- Relative spacing of the chromatographic peaks
- The result of all nonpolar, polarizable, and polar interactions that cause a stationary phase to be more or less retentive to one analyte than another



Why is Stationary Phase Type Important?

$$R_s = \frac{\sqrt{N}}{4}\left(\frac{k}{k+1}\right)\left(\frac{\alpha-1}{\alpha}\right)$$

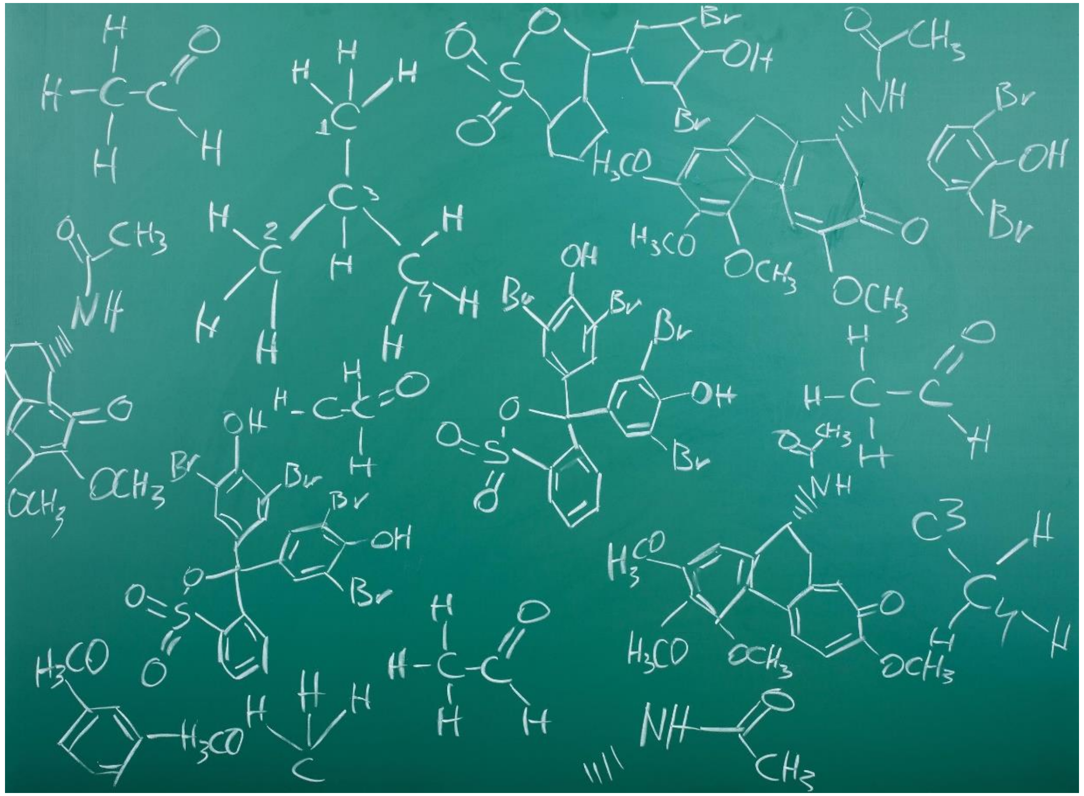
Influence on α *(separation factor)*

$$\alpha = \frac{k_2}{k_1}$$

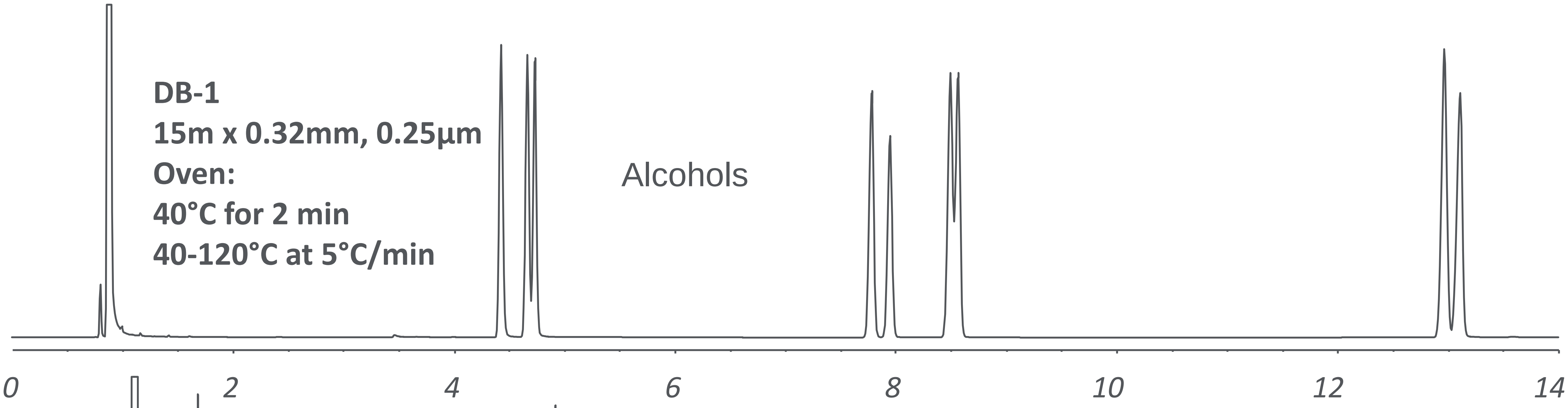
k_2 = partition ratio of 2nd peak
k_1 = partition ratio of 1st peak

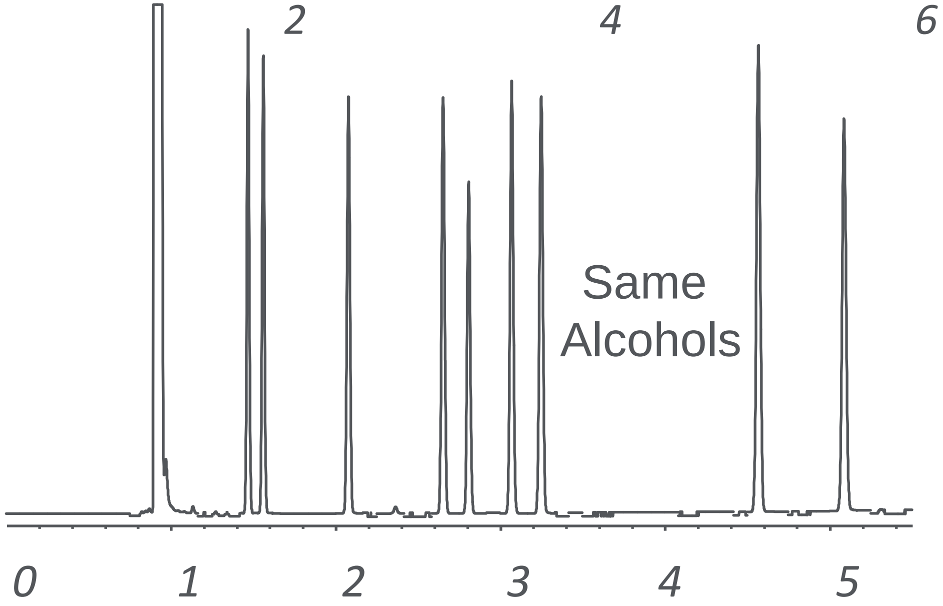
Optimizing Selectivity (α)

- Match analyte polarity to stationary phase polarity for complex mixtures
  - “Like dissolves like”
- Take advantage of unique interactions between analyte and stationary phase functional groups



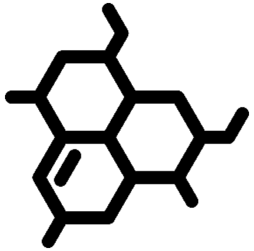
Start with the Right Phase – “like dissolves like”

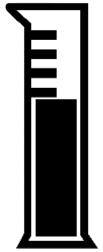




DB-Wax
15m, 0.32mm, 0.25µm
Oven:
80-190°C at 20°C/min

Analyte Polarity

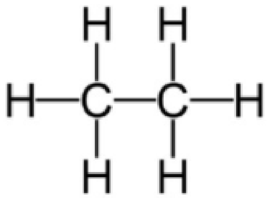


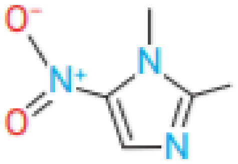


Nonpolar

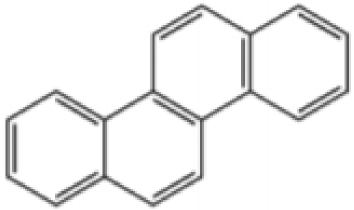
Polar

Polarizable





Dimetridazole (DMZ)
(1,2-dimethyl-5-nitroimidazole)

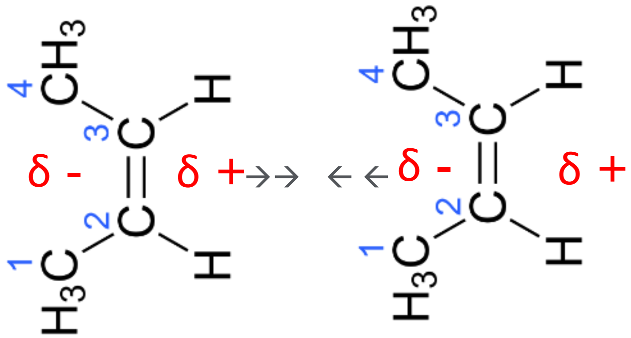


Chrysene

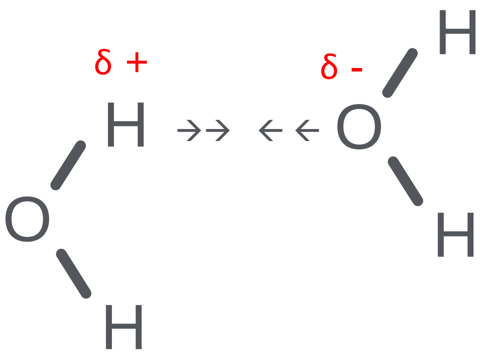
Selectivity Interactions – Intermolecular Forces

Dispersion

Dipole

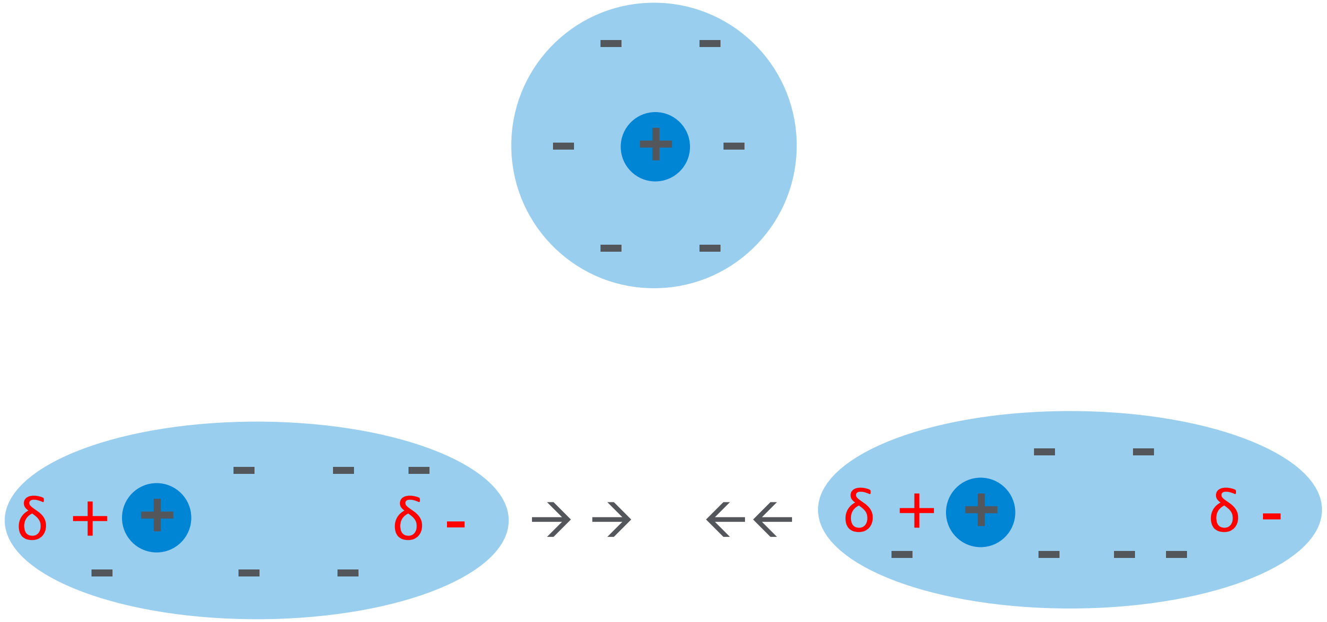


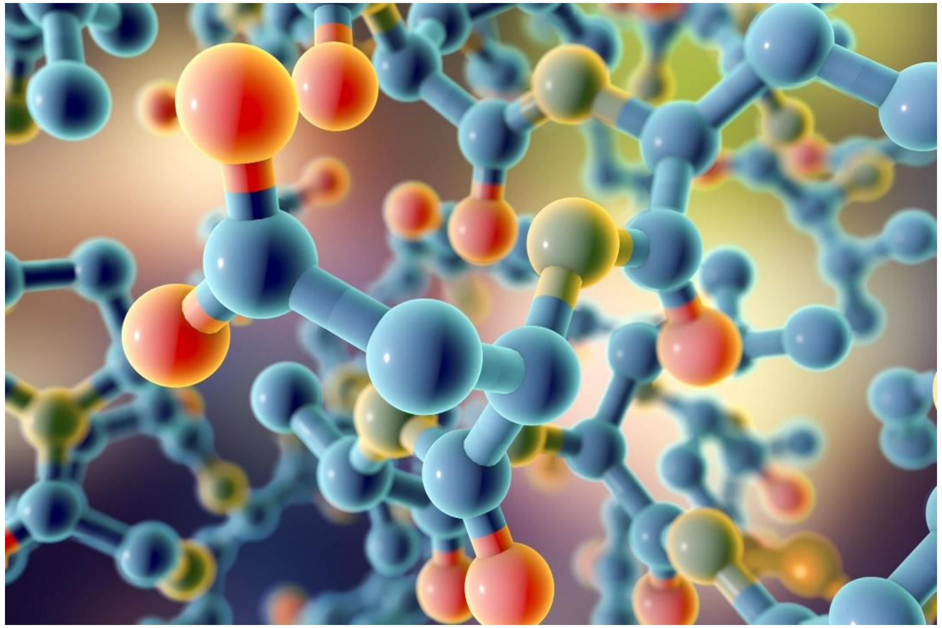
Hydrogen bonding



Dispersion Interaction

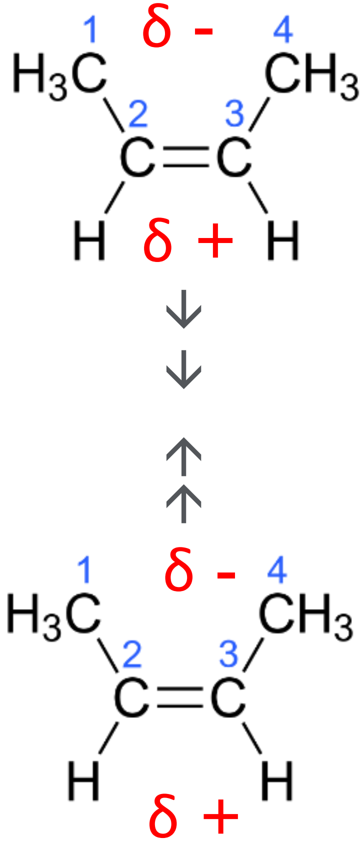
- Molecules are generally electrically symmetrical
- Can be polarized when electrons are distributed unsymmetrically…
- This can cause an induced dipole for near-by molecules
- Weakest intermolecular force



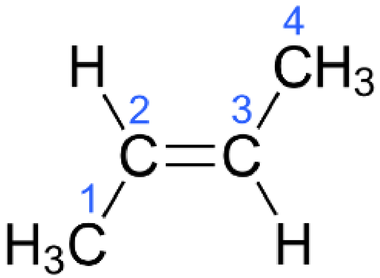


Dipole Interaction

- Molecules are electrically asymmetrical
- Natural attraction between negative and positive ends



Smaller differences require a stronger dipole phase



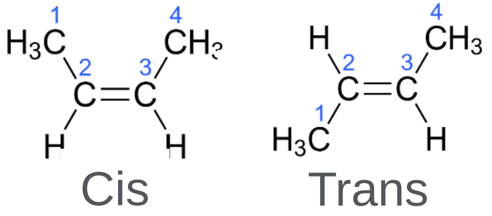
Trans- has no permanent dipole

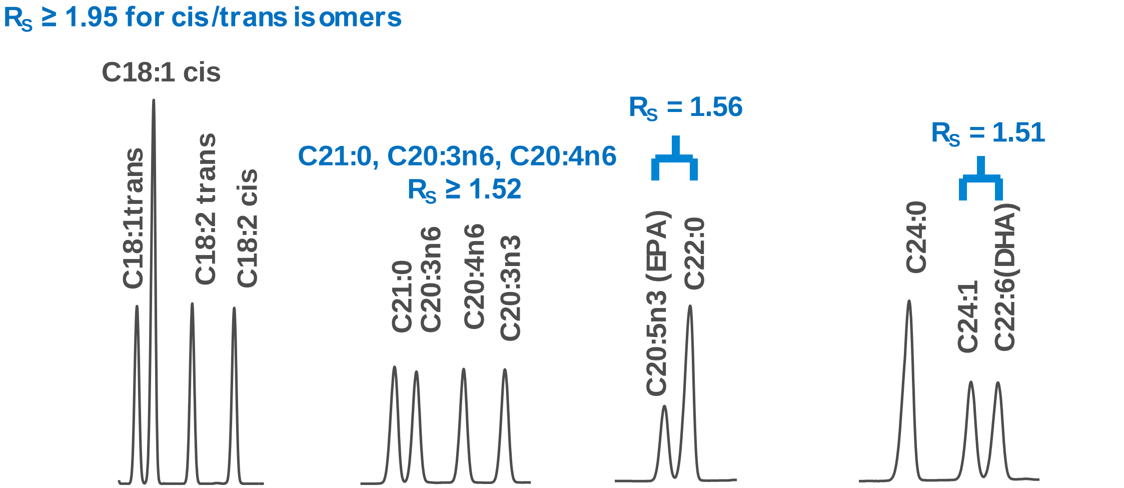
DB-FastFAME

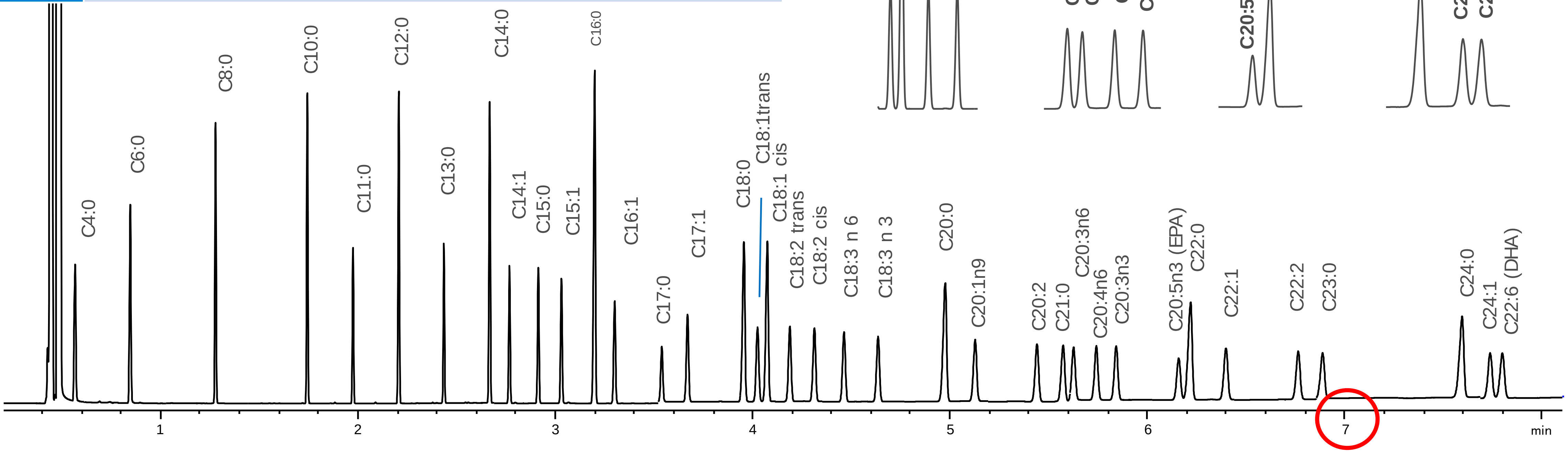
20 m x 0.18 mm x 0.20 µm

Column	**Agilent J&W DB-FastFAME, 20 m x 0.18 mm, 0.20 µm**
Gas	**Hydrogen**, 28 psi, constant pressure mode
Inlet	Split/splitless, 250 °C, split ratio 50:1
Oven	80 °C (0.5 min), 65 °C/min to 175 °C, 10 °C/min to 185 °C (0.5 min), 7 °C/min to 230 °C
FID	280 °C, Hydrogen: 40 mL/min; Air: 400 mL/min; make-up gas: 25 mL/min.
Injection	1 µL

Strong interaction between cis isomers and the dipoles of the cyanopropyl ligands.
This forces the ***cis*** isomer to elute after the ***trans*** isomer.



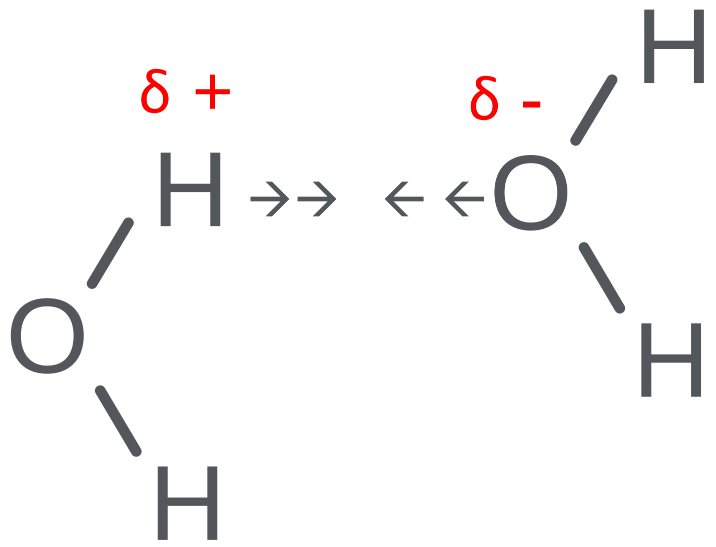




Application note: 5991-8706EN

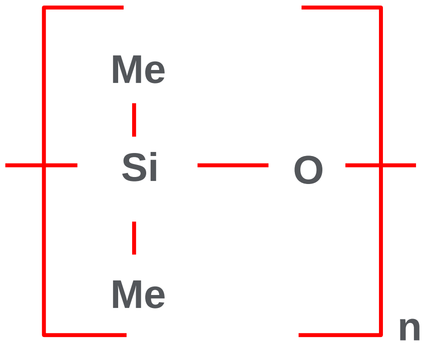
Hydrogen Bonding Interaction

Dipole-Dipole interaction with hydrogen bound to oxygen or nitrogen interacting with an oxygen or nitrogen-atom



Non-polar Phases

Characterized by 100% polydimethylsiloxanes such as HP-1, DB-1, DB-1ms, HP-1ms, VF-1ms, CP-Sil 5 CB

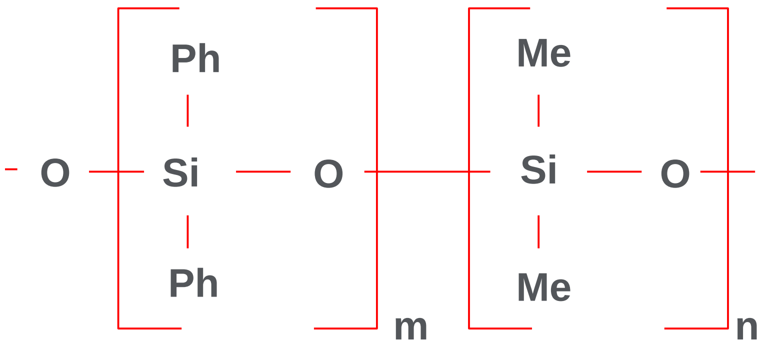


Separation mechanisms:

- Dispersion only

Polarizable Phases

Typified by phenyl substituted siloxanes, substituted at 5–50%
(HP-5, HP-5ms, DB-35, DB-35ms, DB-17, DB-17ms)



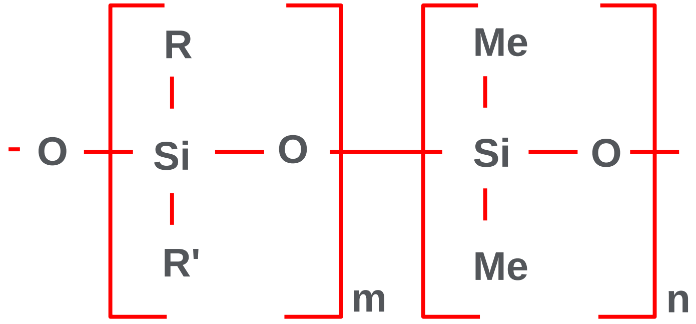
5%--weakly polar,
50%-- midpolar

Separation mechanisms:

- Dispersion
- Inducible dipole at phenyl groups

Strong Dipole Phases

Typified by cyanopropyl or trifluoropropyl substituted DMPS, substituted 6–50% (DB-1701, DB-1301, DB-200, DB-23, DB-225, HP/CP-88, DB-FASTFAME, Select FAME)



R = cyanopropyl or trifluoropropyl
R′ = phenyl or methyl

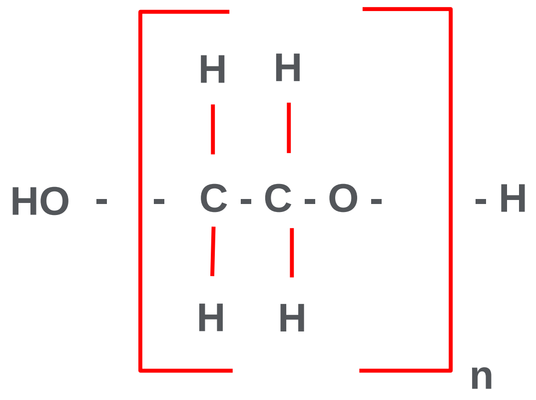
Separation mechanisms:

- Dispersion
- Inducible dipole at phenyl groups
- Strong permanent dipole
- Hydrogen bonding

An excellent guide to FAMES: 5991-8763EN

Hydrogen Bonding Phases

Typified by polyethylene glycol polymers (HP-INNOWax, DB-WAX UI, DB-HeavyWAX, DB-FFAP, VF-WAXms, CP-Wax 52 CB…)



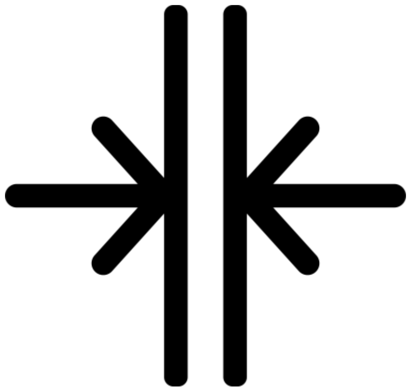
Separation mechanisms:

- Dispersion
- Strong permanent dipole
- Hydrogen bonding

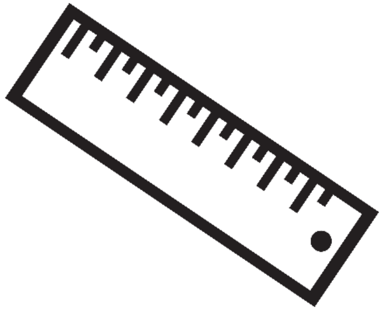
Selectivity
Interaction strengths summarized

Phase	Dispersion	Dipole	H Bonding
Methyl	Strong	None	None
Phenyl	Strong	None	Weak
Cyanopropyl	Strong	Very Strong	Moderate
Trifluoropropyl	Strong	Moderate	Weak
PEG	Strong	Strong	Moderate

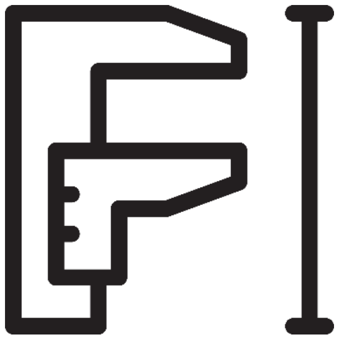
Column Dimensions



Inner Diameter



Length



Film thickness

Column Diameter

Capillary Columns

id (mm)	Common Name
0.53	Megabore
0.45	High speed mega-bore
0.32	Wide
0.20–0.25	Narrow
0.18	Mini-bore

Column Diameter

Theoretical Efficiency

id (mm)	N/m
0.10	11905
0.18	6666
0.20	5941
0.25	4762
0.32	3717
0.53	2242

k = 5

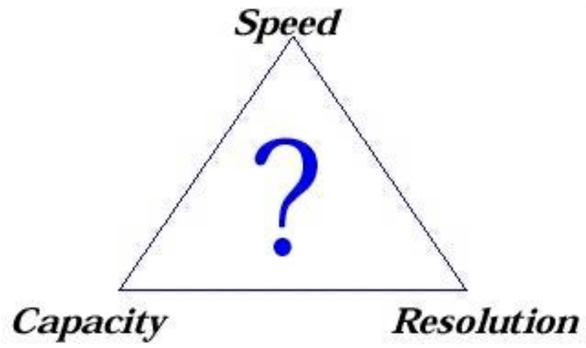
Efficiency and Resolution
Relationship

$$\sqrt{N} \propto R_s$$

efficiency x 4 = resolution x 2

Column Diameter

Resolution (180 °C isothermal)

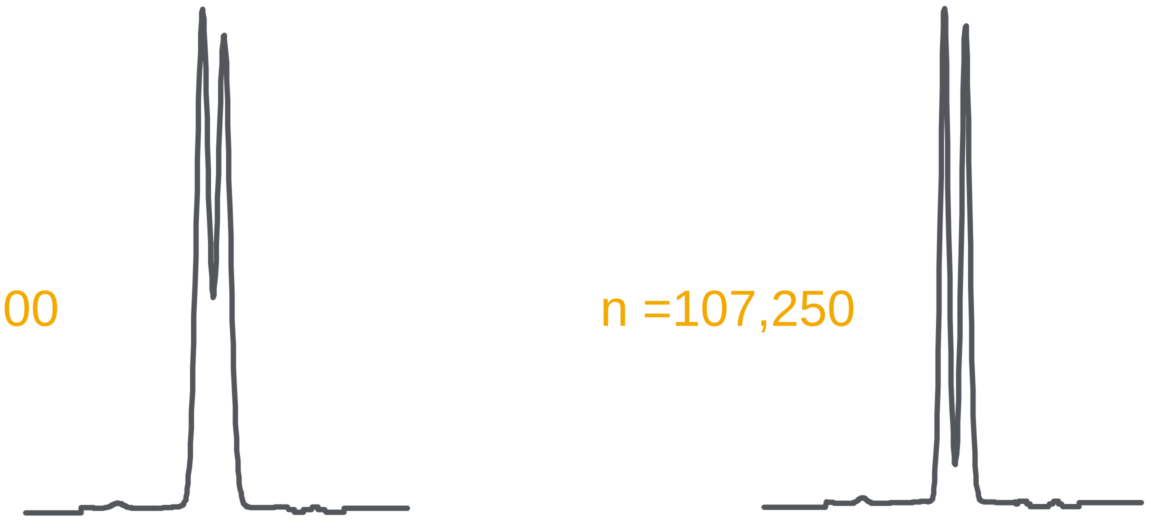


R= 0.87

R= 1.01

n =58,700

n =107,250



0.53 mm

0.32 mm

Square root of resolution is inversely proportional to column diameter

Column Diameter

Capacity (0.25 µm film thickness)

id (mm)	Capacity (ng)
0.20	50–100
0.25	75–150
0.32	125–250
0.53	200–400

Like polarity phase/solute

Column Diameter
Carrier gas flow rate

Smaller diameters for low flow situations
(e.g., GC/MS, SCD/NCD)

Larger diameters for high flow situations
(e.g., purge & trap, headspace, gas sample valve)

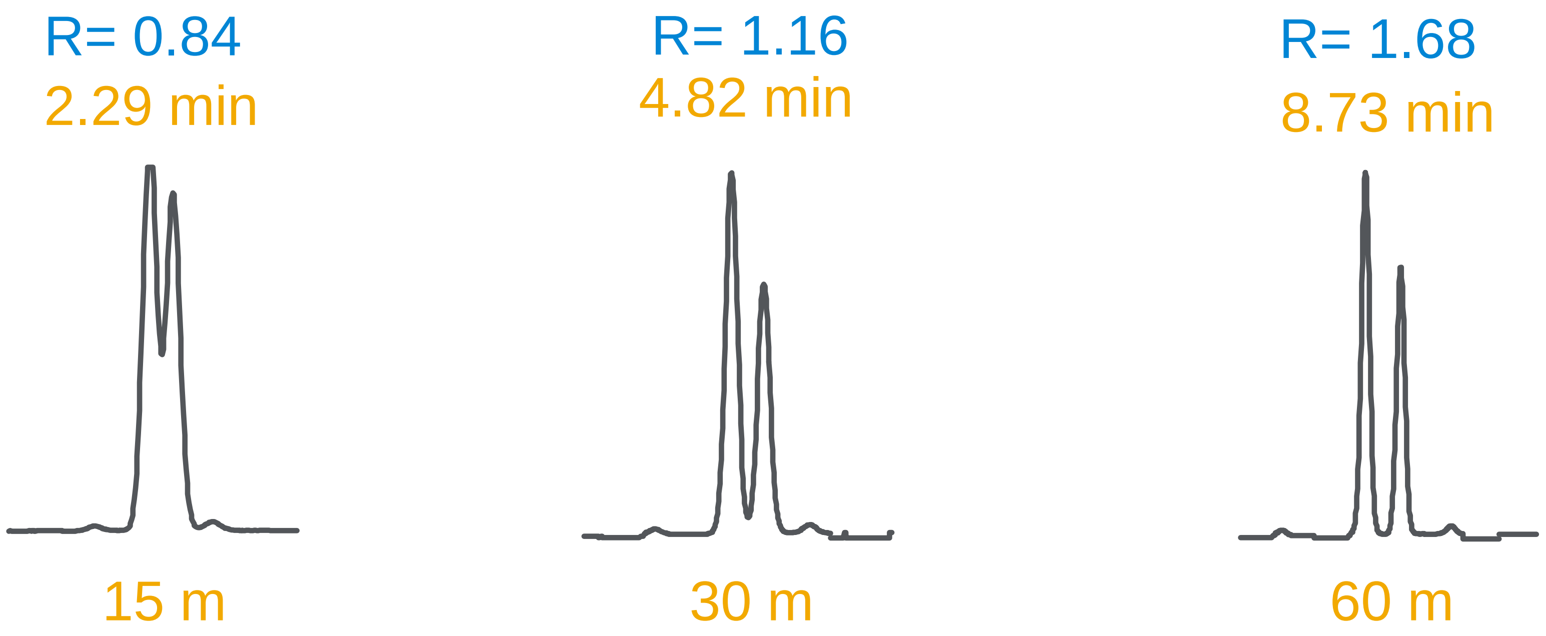
Column Length

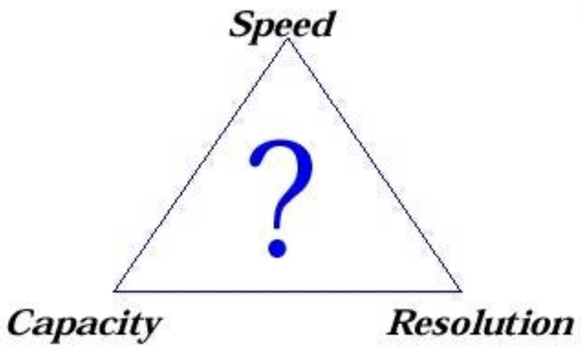
Most common: 15–60 meters

Available: 5–200 meters

Column Length

Resolution and retention 210 °C isothermal





Resolution is proportional to the square root of column length

Isothermal: retention is proportional to length

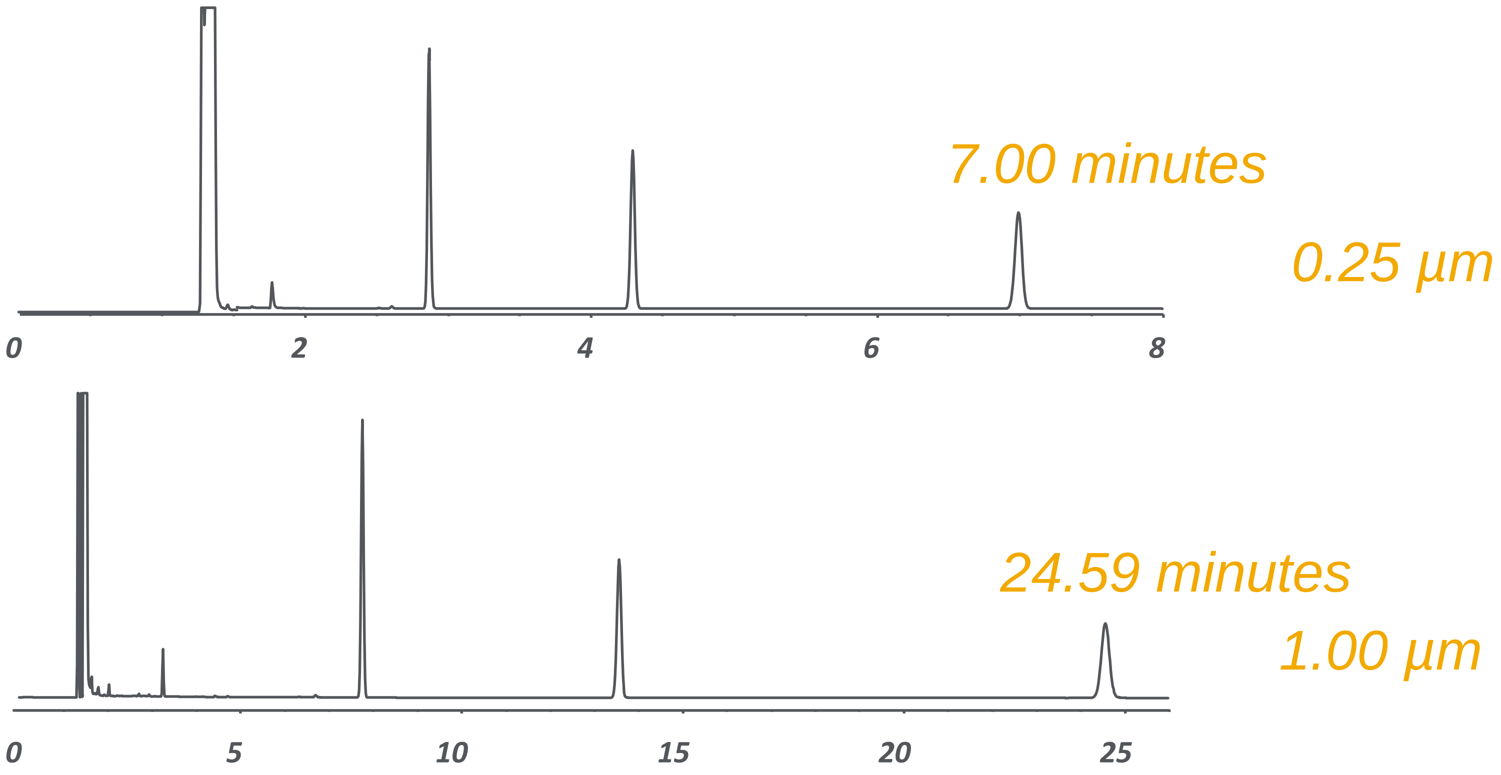
Temperature program: 1/3–1/2 of isothermal values

Film Thickness

Most common: 0.1–3.0 µm

Available: 0.1–10.0 µm

Film Thickness and Retention (100 °C Isothermal)



Isothermal: Retention is proportional to film thickness
Temperature program: 1/3–1/2 of isothermal values

Film Thickness and Resolution

When solute k < 5

(early eluters)

d_f R

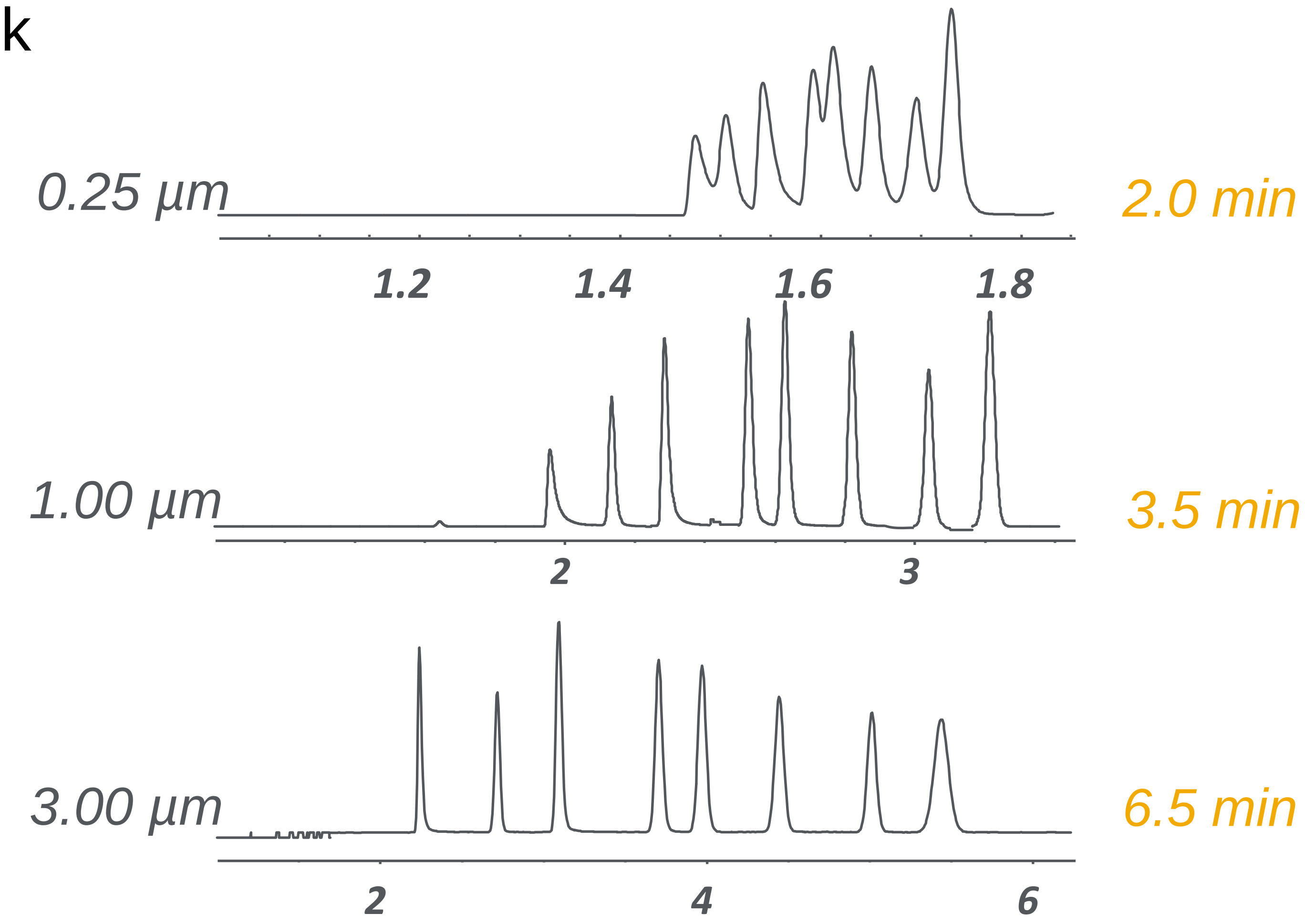
When solute k > 5

(later eluters)

d_f R

Film Thickness

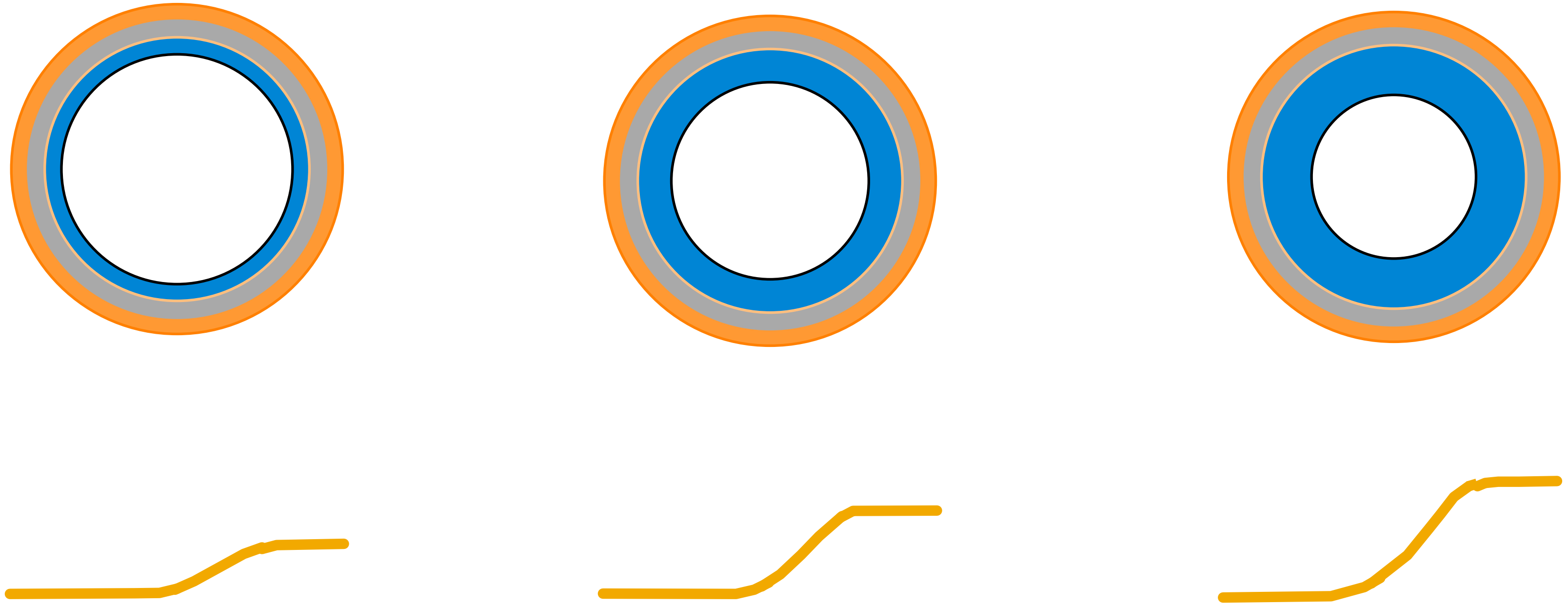
Resolution at low k



DB-1, 30 m x 0.32 mm id
40 °C isothermal, He at 35 cm/sec
Solvent mixture

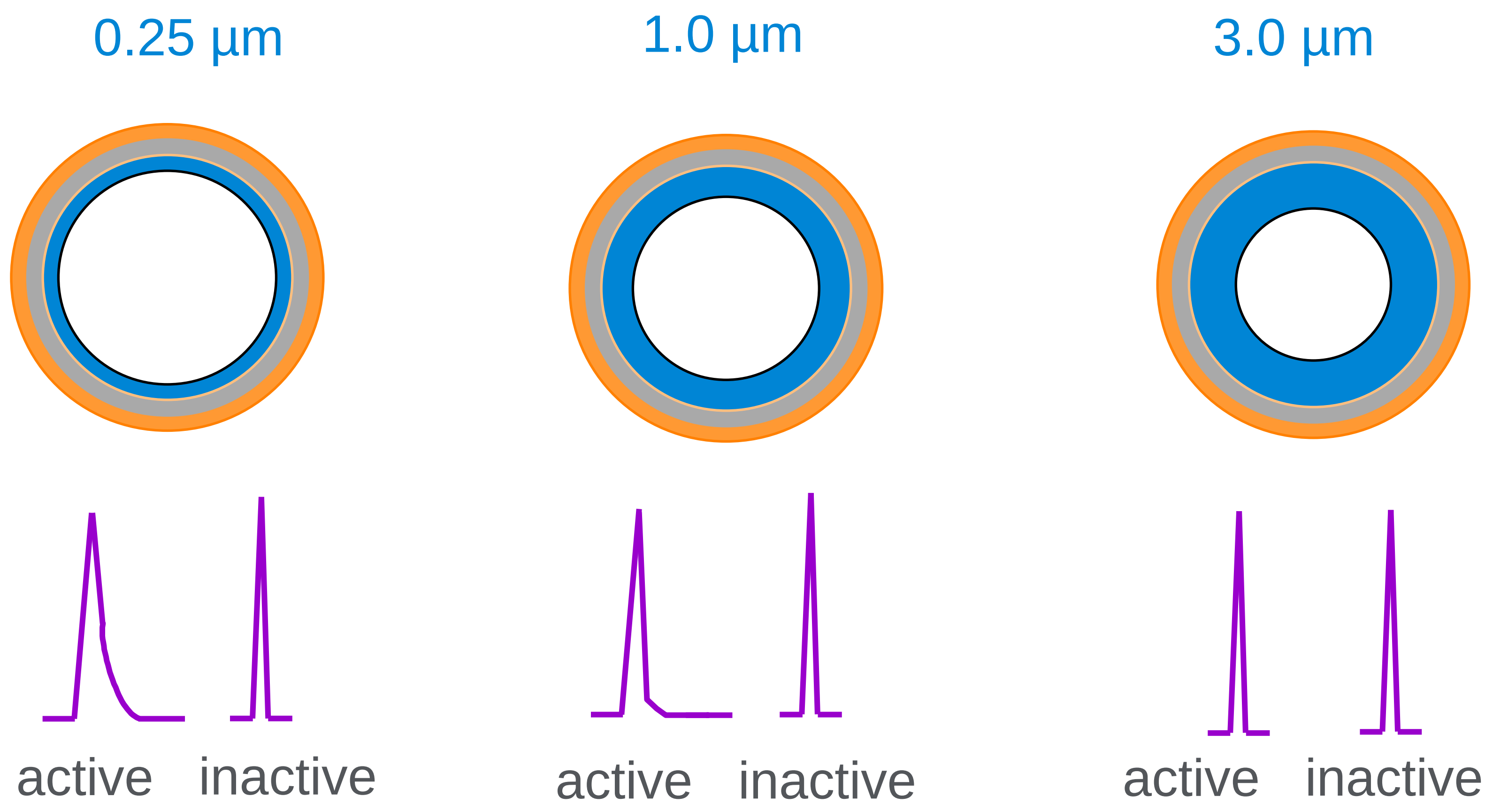
Film Thickness (Bleed)

More stationary phase = More degradation products



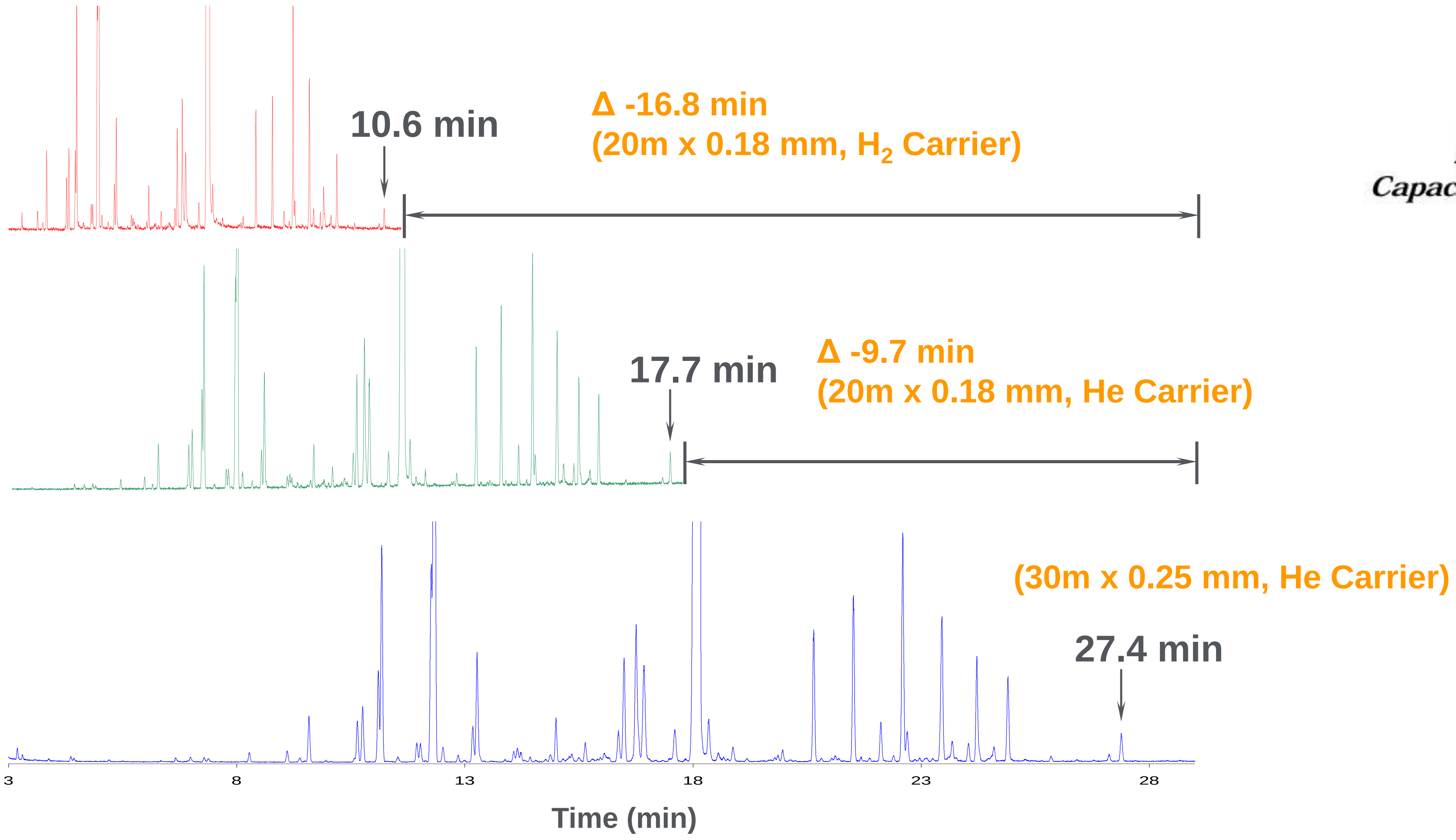
**Longer and wider columns also bleed more.*

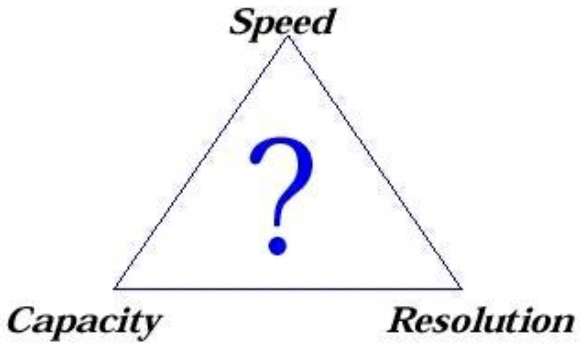
Film Thickness (Inertness)



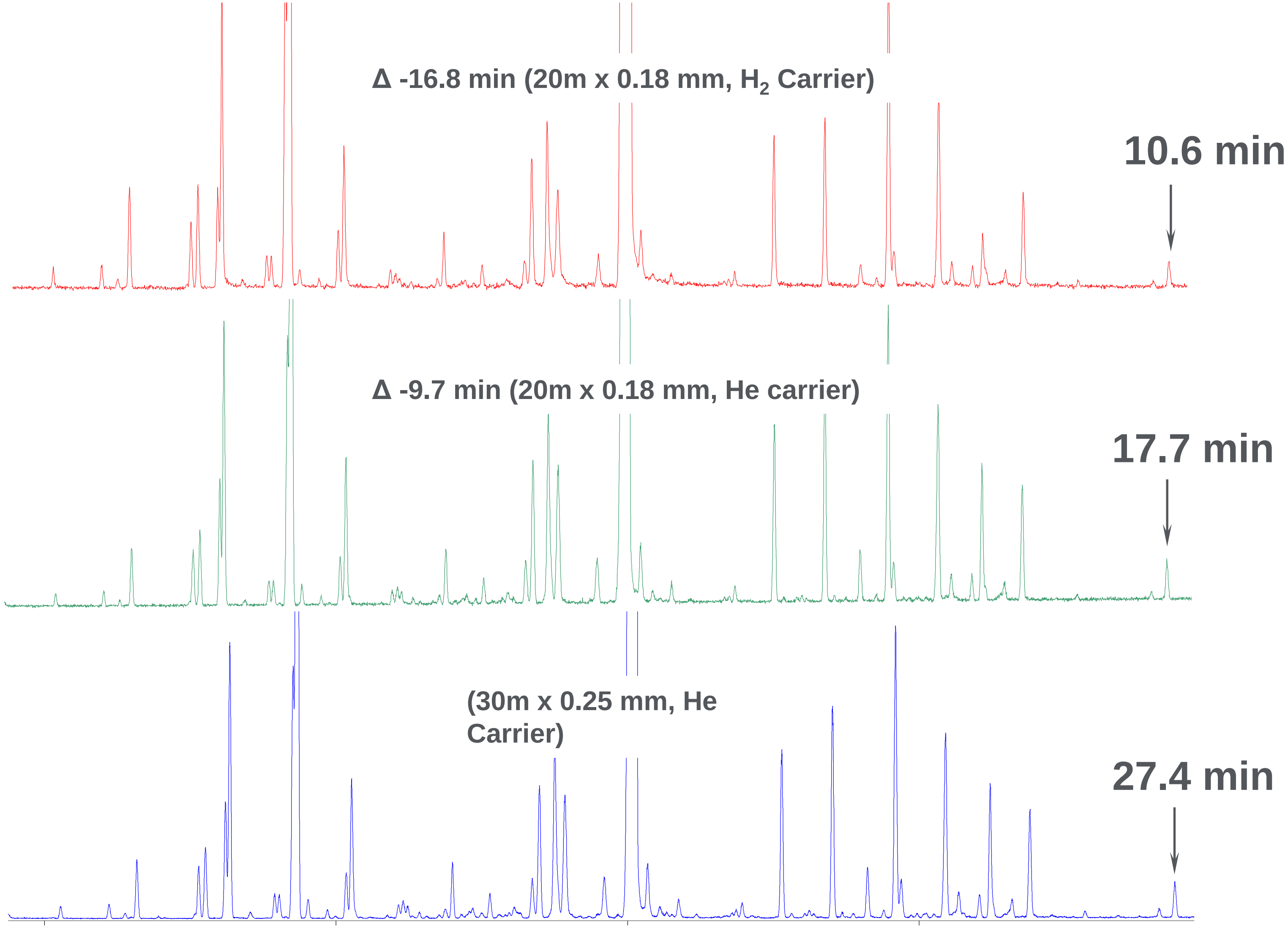
So You Want To Go Faster…

Spearmint Oil





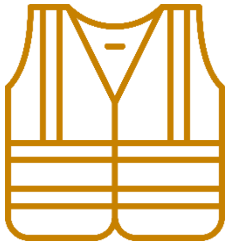
Spearmint Oil – Resolution Check



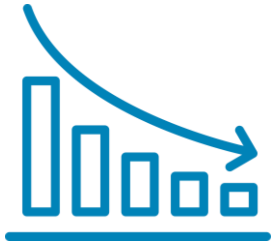
Another New Column: J&W DB-HeavyWAX

The WAX column you've been waiting for!

Increased temperature limit (280 °C / 290 °C)



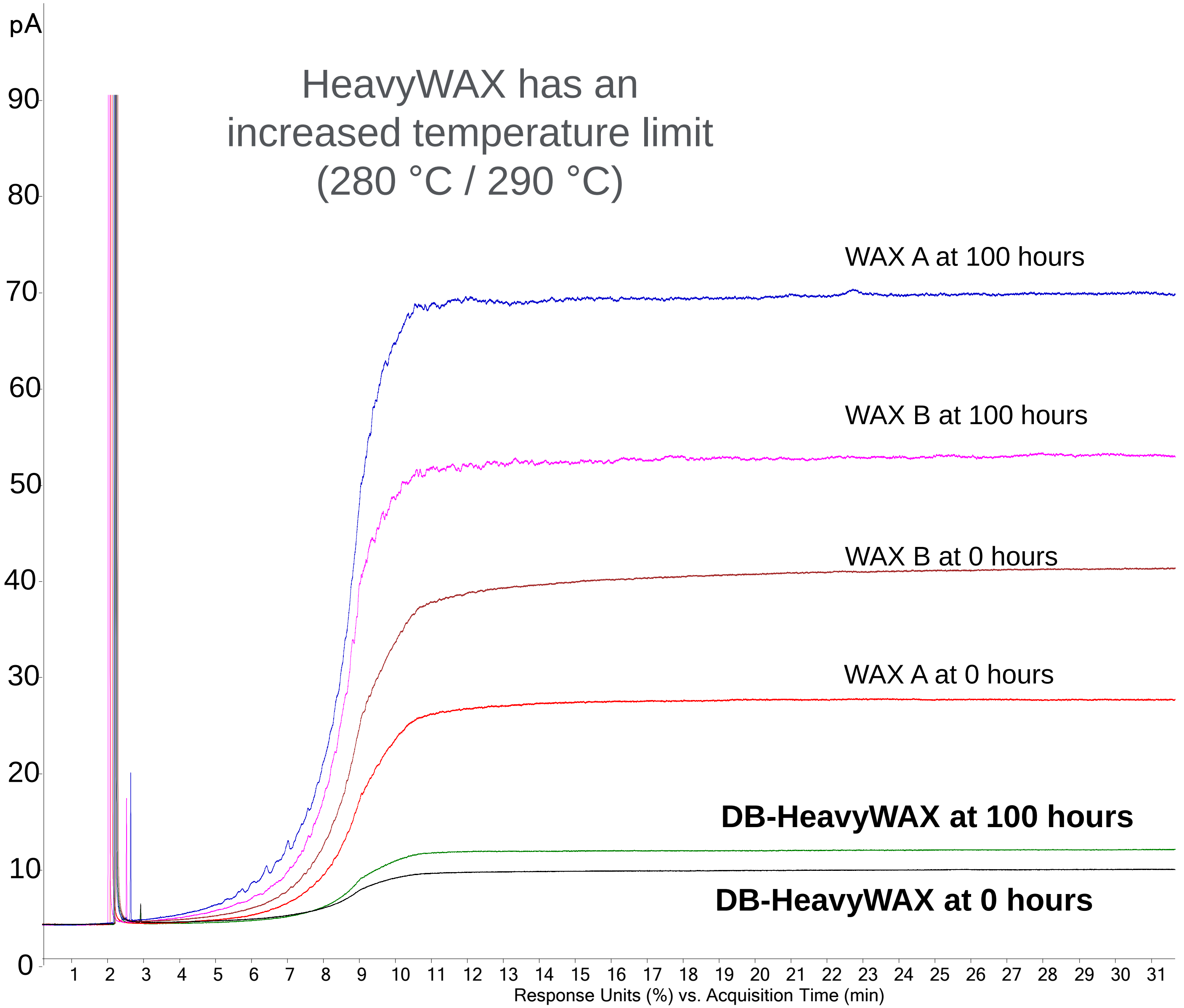
Increased thermal stability

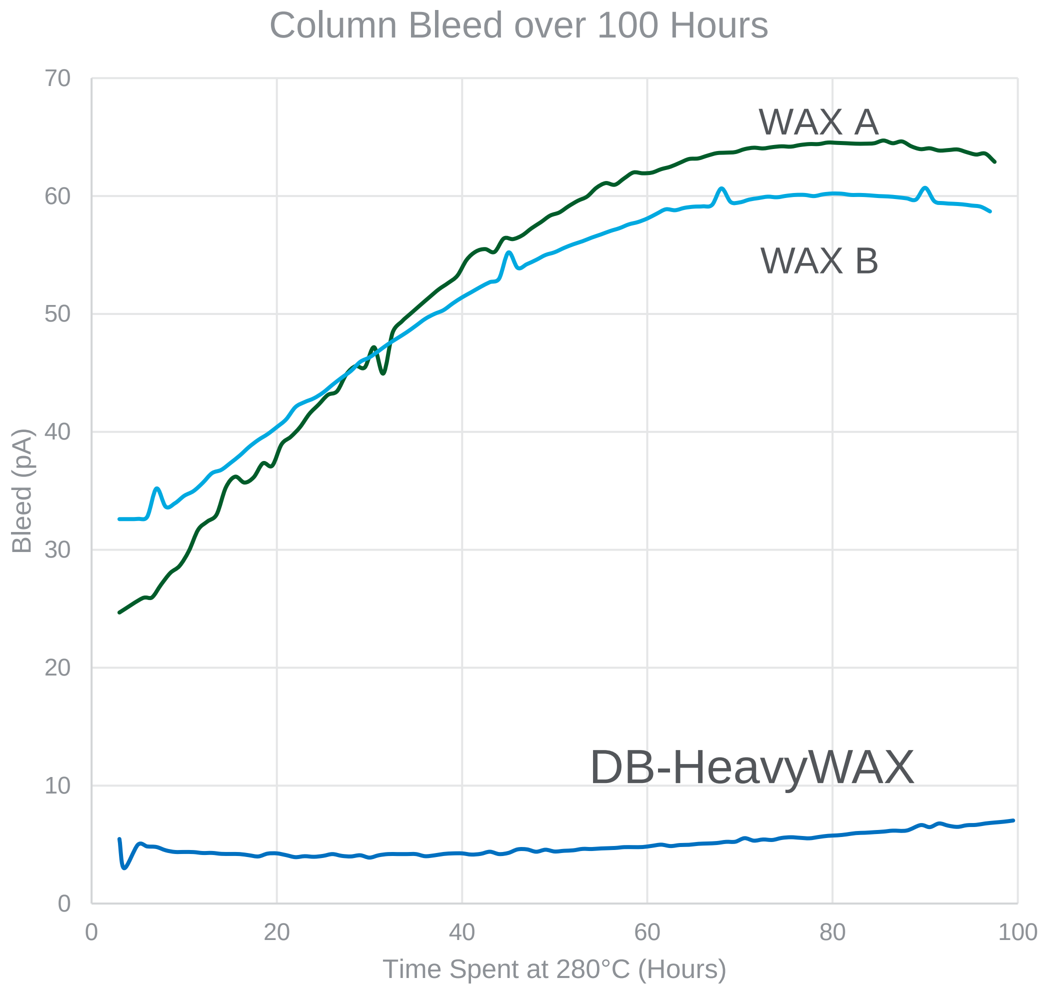


Lower bleed

www.agilent.com/chem/db-heavywax

Bleed Summary at 280 °C Over 100 Hours





Agilent Ultra Inert GC Columns

Column inertness: What does it mean?

- Easier to describe "lack of inertness"
  - Peak tailing (reversible interaction)
  - Complete loss of compound (irreversible interaction)

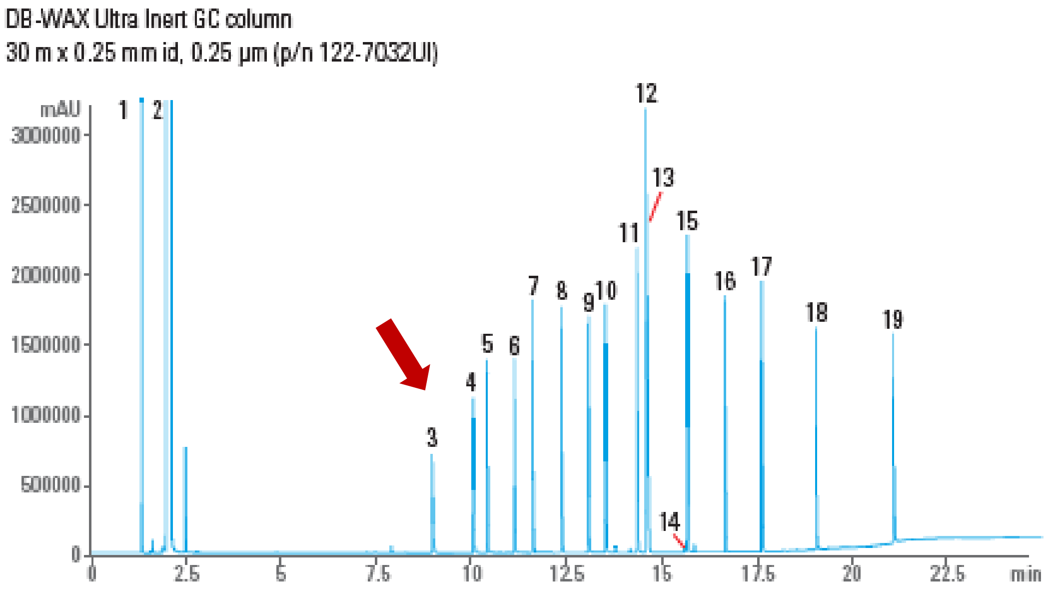
- A high level of flow path inertness will produce peaks from active compounds that are not degraded and will look "normal"/symmetrical.

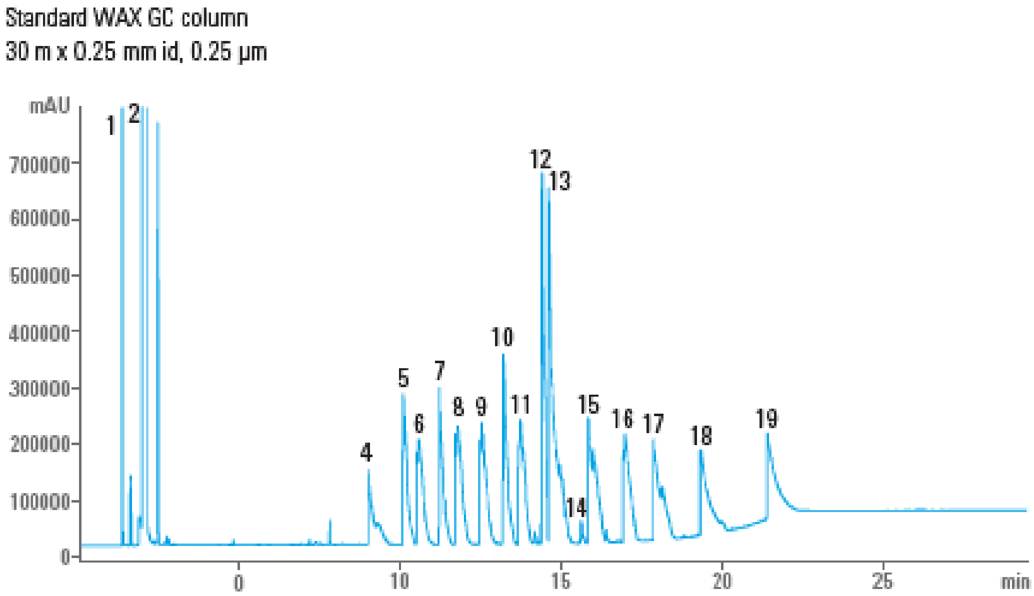
- The negative effects the column has towards challenging compounds:
  - Acids
  - Bases
  - Hydrogen bonding
  - Compounds such as 2,4-DNP, Endrin, etc.

DB-WAX Ultra Inert and Free Fatty Acid

Peak identification:
1. Methane
2. Acetone (solvent)
3. Acetic acid
4. Propionic acid
5. Isobutyric acid
6. Butyric acid
7. Isovaleric acid
8. Valeric acid
9. 4-Methylvaleric acid
10. Hexanoic acid
11. 4-Methylhexanoic acid
12. 2-Ethylhexanoic acid
13. Heptanoic acid
14. Pyruvic acid
15. Octanoic acid
16. Nonanoic acid
17. Decanoic acid
18. Undecylenic acid
19. Myristic acid (Tetradecanoic)

Competitive comparison: free fatty acids

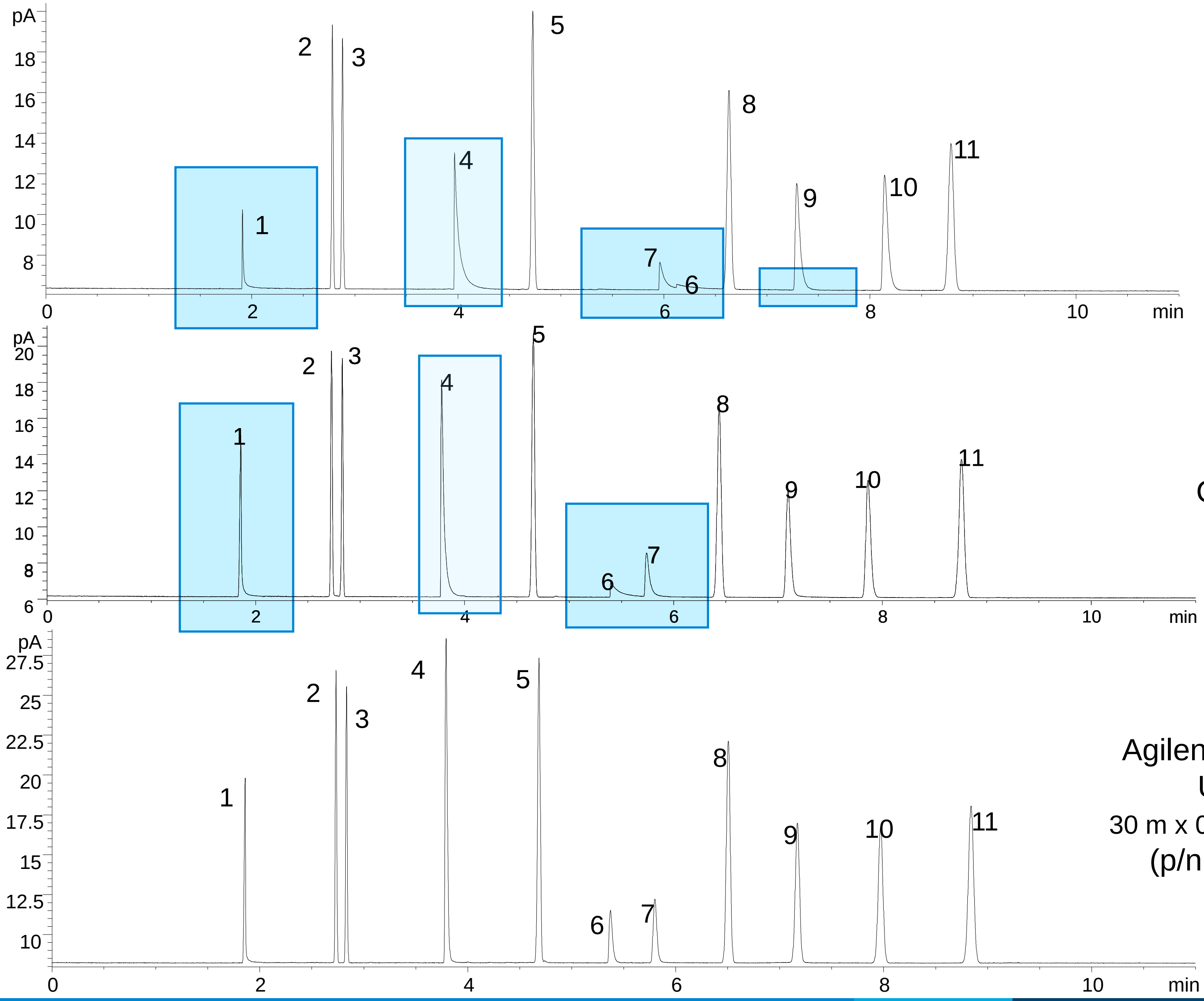




Application notes:
5991-8763EN
5991-6709EN

Ultra Inert Test Mix – DB-5MS Ultra Inert versus Competitors

1. **1-Propionic acid**
2. **1-Octene**
3. **n-Octane**
4 **4-Picoline**
5. **n-Nonane**
6. **Trimethyl phosphate**
7. **1,2-Pentanediol**
8. **n-Propylbenzene**
9. **1-Heptanol**
10. **3-Octanone**
11. **n-Decane**



Competitor column

Competitor column

Agilent J&W DB-5ms Ultra Inert
30 m x 0.25 mm x 0.25 µm
(p/n 122-5532UI)

Ultra Inert Flow Path

Touchless Packaging

5190-6144 (EA)
5190-6145 (10 pk)

Specialty Phases

Columns developed for specific applications

- Examples:
  - DB-8270D UI
  - DB-624 UI <467>
  - DB-MTBE
  - DB-TPH
  - DB-BAC1 UI & DB-BAC2 UI
  - DB-HT SimDis
  - Select Low Sulfur
  - CP-Volamine
  - Select PAH
  - DB-EUPAH
  - DB-CLP1 & DB-CLP2
  - DB-Select 624 UI <467>
  - CP-LowOx
  - Select Permanent Gases
  - …and more!

Conclusions

- Understand the sample
- Is it volatile and thermally stable enough to chromatograph by GC?
- Match polarity when necessary – oil and water don’t mix!
- Look for unique characteristics of compounds and match them to a phase
- If you have the correct selectivity, change the dimensions to improve resolution or shorten run time – consider a shorter / smaller id column
- If you need better peak shape for difficult compounds, try the ‘UI’ version
  - Use UI liners and seals as well…
- Look for available information for a particular application

Call Tech Support!

Contact Agilent Chemistries and Supplies Technical Support

1-800-227-9770 Option 3, Option 3:

Option 1 for GC and GC/MS columns and supplies

Option 2 for LC and LC/MS columns and supplies

Option 3 for sample preparation, filtration, and QuEChERS

Option 4 for spectroscopy supplies

Option 5 for chemical standards

Available in the USA and Canada 8–5, all time zones

gc-column-support@agilent.com

lc-column-support@agilent.com

spp-support@agilent.com

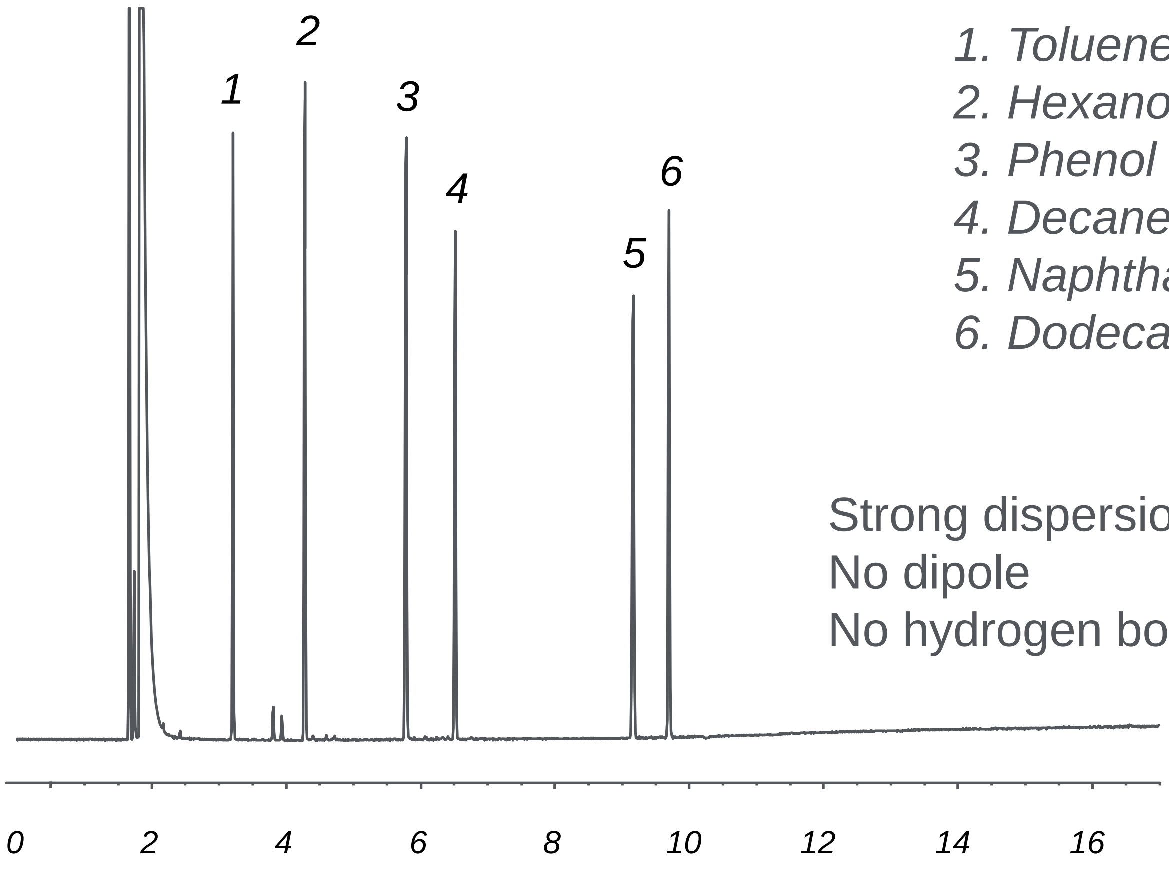
spectro-supplies-support@agilent.com

chem-standards-support@agilent.com

Compounds and Properties

Compounds	Polar	Aromatic	Hydrogen Bonding	Dipole
Toluene	no	yes	no	induced
Hexanol	yes	no	yes	yes
Phenol	yes	yes	yes	yes
Decane	no	no	no	no
Naphthalene	no	yes	no	induced
Dodecane	no	no	no	no

100% Methyl Polysiloxane

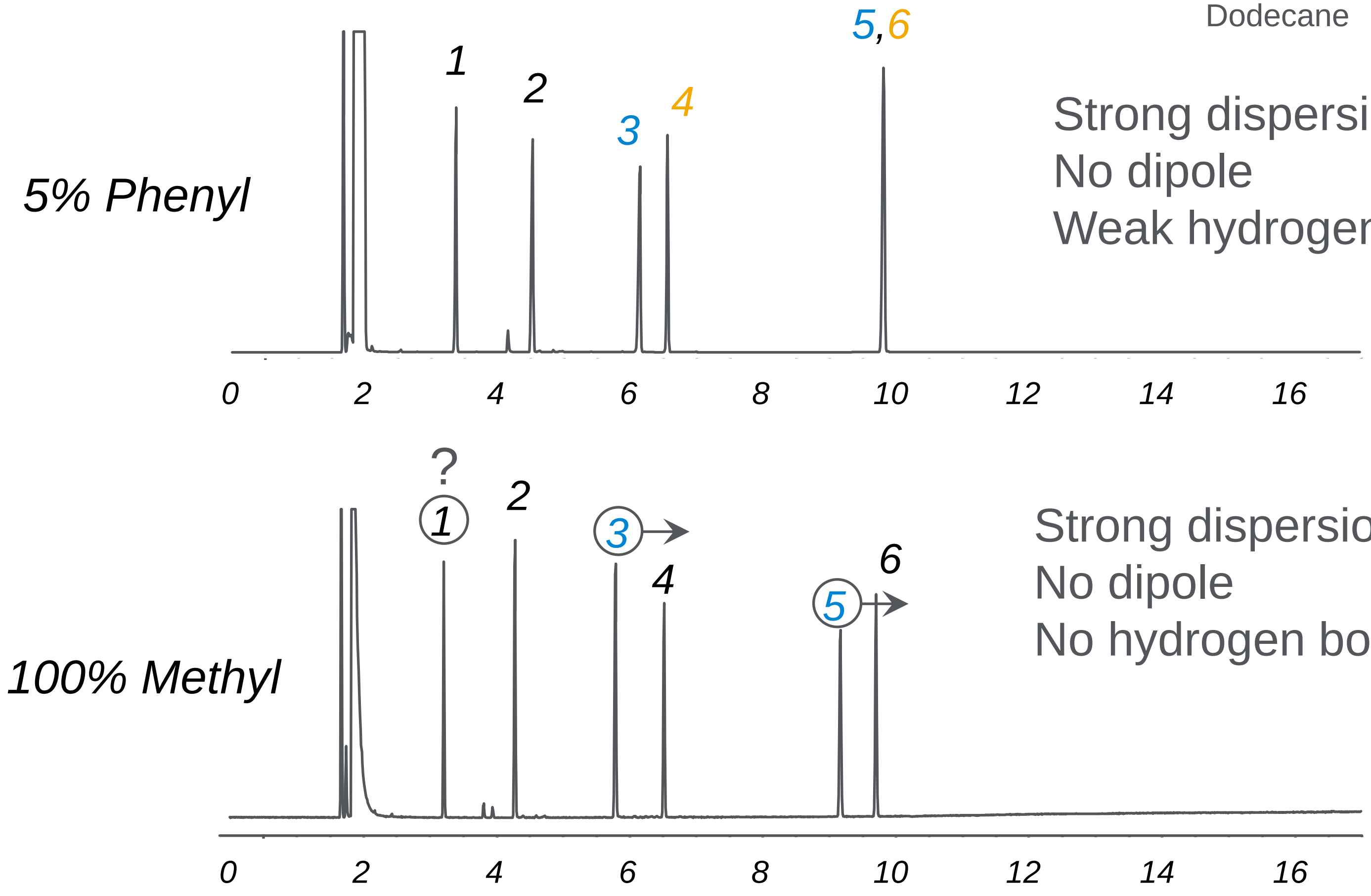


1. Toluene *110* °C
2. Hexanol *156* °C
3. Phenol *182 °C*
4. Decane (C10) *174* °C
5. Naphthalene *218 °C*
6. Dodecane (C12) *216* °C

Strong dispersion
No dipole
No hydrogen bonding

5% Phenyl

Compounds	Polar	Aromatic	Hydrogen Bonding	Dipole
Toluene	no	yes	no	induced
Hexanol	yes	no	yes	yes
Phenol	yes	**yes**	**yes**	yes
Decane	no	no	no	no
Naphthalene	no	**yes**	no	induced
Dodecane	no	no	no	no



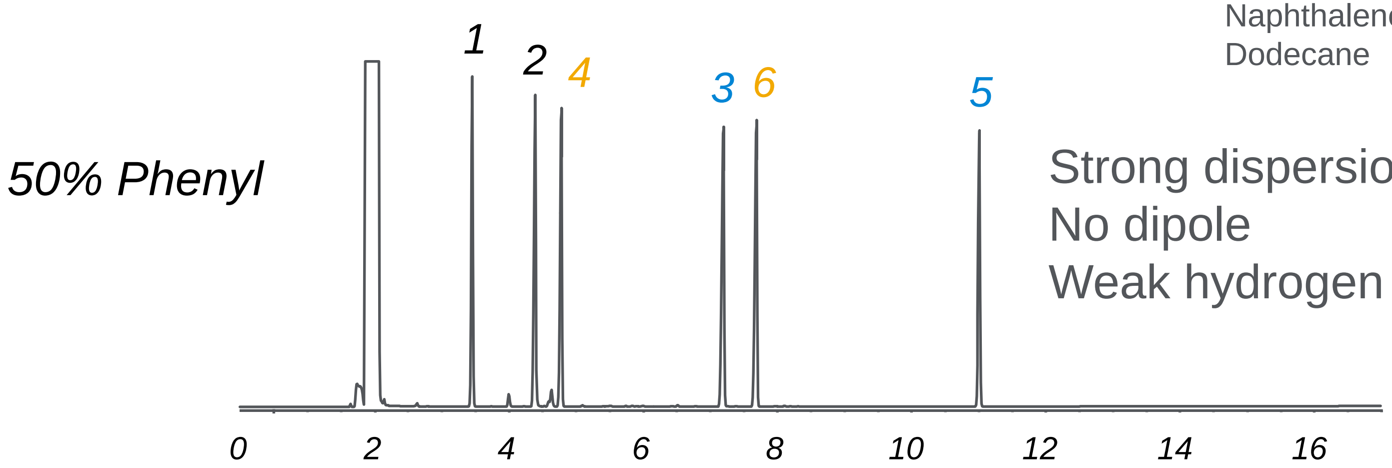
Strong dispersion
No dipole
Weak hydrogen bonding

Strong dispersion
No dipole
No hydrogen bonding

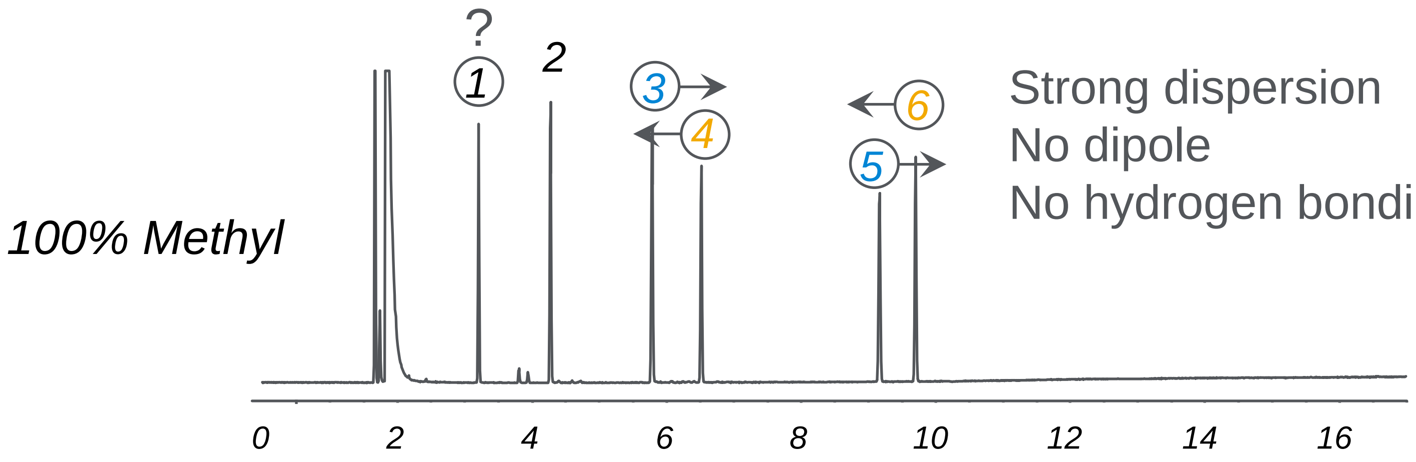
1. Toluene
2. Hexanol
3. Phenol
4. Decane (C10)
5. Naphthalene
6. Dodecane (C12)

50% Phenyl

Compounds	Polar	Aromatic	Hydrogen Bonding	Dipole
Toluene	no	yes	no	induced
Hexanol	yes	no	yes	yes
Phenol	yes	**yes**	**yes**	yes
Decane	**no**	**no**	**no**	no
Naphthalene	no	**yes**	no	induced
Dodecane	**no**	**no**	**no**	no



Strong dispersion
No dipole
Weak hydrogen bonding



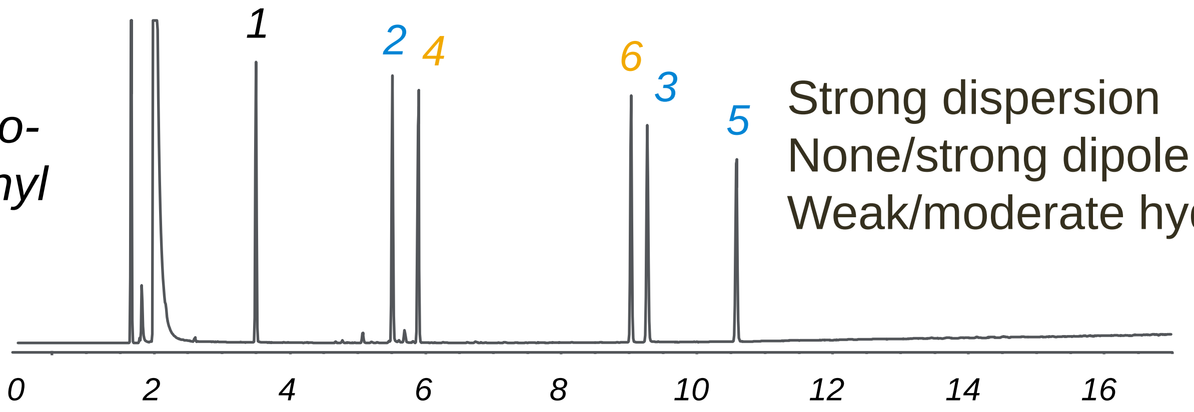
Strong dispersion
No dipole
No hydrogen bonding

1. Toluene *110 °C*
2. Hexanol *156 °C*
3. Phenol *182 °C*
4. Decane (C10) *174 °C*
5. Naphthalene *218 °C*
6. Dodecane (C12) *216 °C*

14% Cyanopropylphenyl

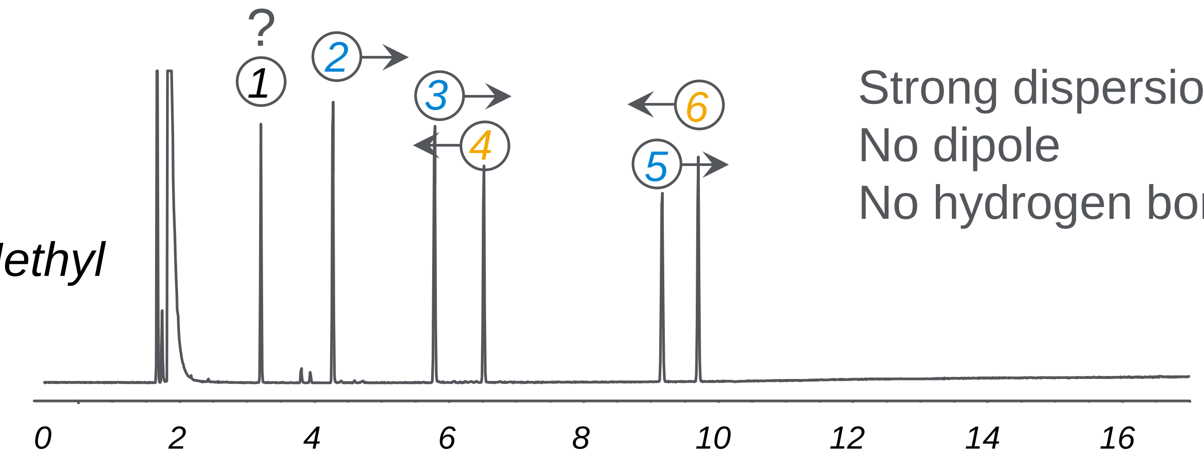
Compounds	Polar	Aromatic	Hydrogen Bonding	Dipole
Toluene	no	yes	no	induced
Hexanol	**yes**	no	**yes**	**yes**
Phenol	**yes**	yes	**yes**	**yes**
Decane	**no**	no	**no**	**no**
Naphthalene	no	**yes**	no	**induced**
Dodecane	**no**	no	**no**	**no**

14% Cyano-propylphenyl



Strong dispersion
None/strong dipole (phenyl/cyanopropyl)
Weak/moderate hydrogen bonding (phenyl/cyanopropyl)

100% Methyl

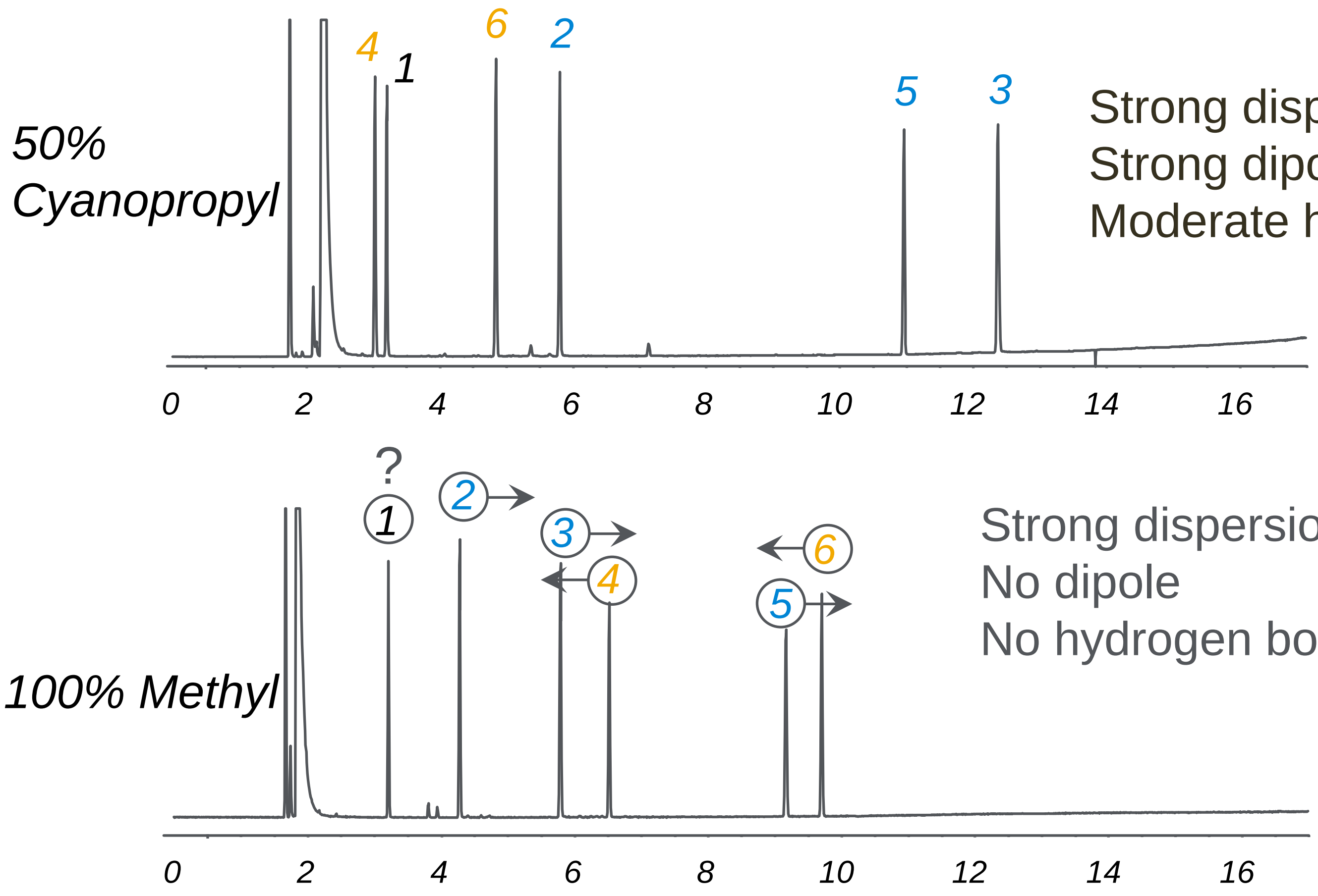


Strong dispersion
No dipole
No hydrogen bonding

1. Toluene
2. Hexanol
3. Phenol
4. Decane (C10)
5. Naphthalene
6. Dodecane (C12)

50% Cyanopropyl

Compounds	Polar	Aromatic	Hydrogen Bonding	Dipole
Toluene	no	yes	no	induced
Hexanol	**yes**	no	**yes**	**yes**
Phenol	**yes**	yes	**yes**	**yes**
Decane	no	no	**no**	**no**
Naphthalene	no	yes	no	induced
Dodecane	no	no	**no**	**no**



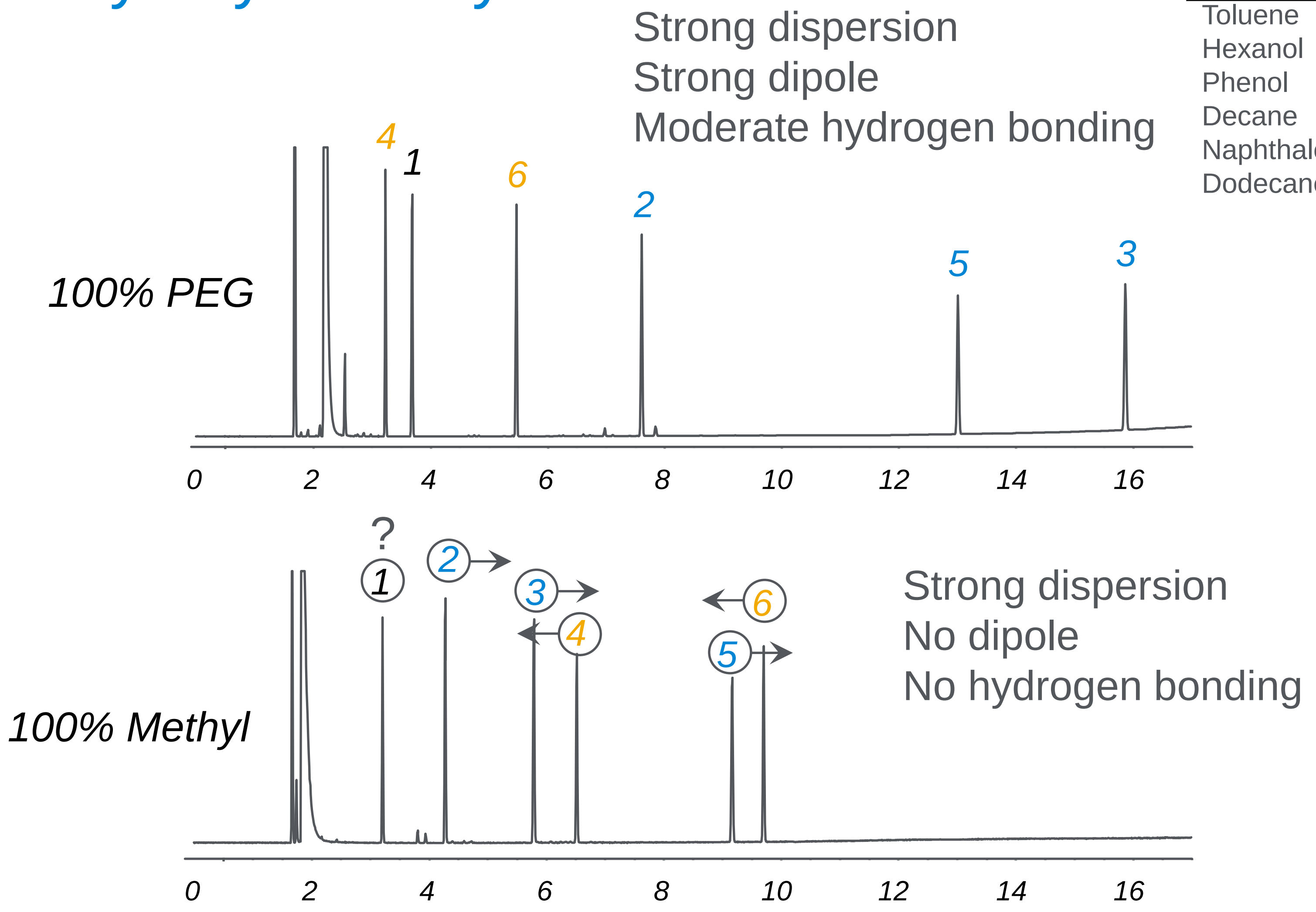
Strong dispersion
Strong dipole
Moderate hydrogen bonding

Strong dispersion
No dipole
No hydrogen bonding

1. *Toluene*
2. *Hexanol*
3. *Phenol*
4. *Decane (C10)*
5. *Naphthalene*
6. *Dodecane (C12)*

100% Polyethylene Glycol

Compounds	Polar	Aromatic	Hydrogen Bonding	Dipole
Toluene	no	yes	no	induced
Hexanol	**yes**	no	**yes**	**yes**
Phenol	**yes**	yes	**yes**	**yes**
Decane	**no**	no	**no**	**no**
Naphthalene	no	yes	no	induced
Dodecane	**no**	no	**no**	**no**



1. *Toluene*
2. *Hexanol*
3. *Phenol*
4. *Decane (C10)*
5. *Naphthalene*
6. *Dodecane (C12)*

Column Diameter

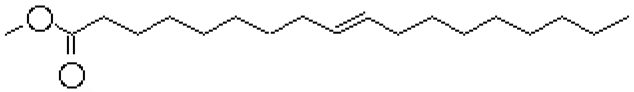
Inlet head pressures for 30 meter column (helium)

id (mm)	Pressure (psig)
0.10	225–250
0.20	25–35
0.25	15–25
0.32	10–20
0.53	2–4

Hydrogen will require about half the amount of pressure

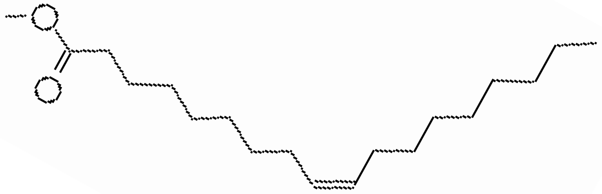
Dipole Interaction

- Molecules are electrically asymmetrical
- Natural attraction between negative and positive ends



C18:1 (Methyl *trans*-9-octadecenoate)

B.Pt. 186 °C



C18:1 (Methyl *cis*-9-octadecenoate)

B.Pt. 186 °C

Smaller differences require a stronger dipole phase

Film Thickness (Capacity)

Thickness (µm)	Capacity (ng)
0.10	50-100
0.25	125-250
1.0	500-1000
3.0	1500-3000
5.0	2500-5000

0.32 mm column id

Like polarity phase/solute

Test Your Knowledge

Q Which one of these Agilent HP-5ms columns have the highest theoretically efficiency?

A. 0.53 i.d. X 30 meters X 3.00 um

B. 0.18 i.d. X 20 meters X 0.18 um

C. 0.18 i.d. x 40 meters X 0.18 um

D. 0.25 i.d. X 15 meters X 0.25 um

Test Your Knowledge

Q Which column below is the most inert?

A. Agilent DB-5ms UI 0.25 i.d. X 30 meters X 0.25 um

B. Agilent HP-5ms 0.25 X 60 meters X 0.25 um

C. Agilent DB-WAX 0.32 i.d. X 30 meters X 0.50 um

D. Agilent DB-5ms UI 0.25 X 30 meters X 0.50 um

Test Your Knowledge

Q If I double my column length, my resolution will also double.

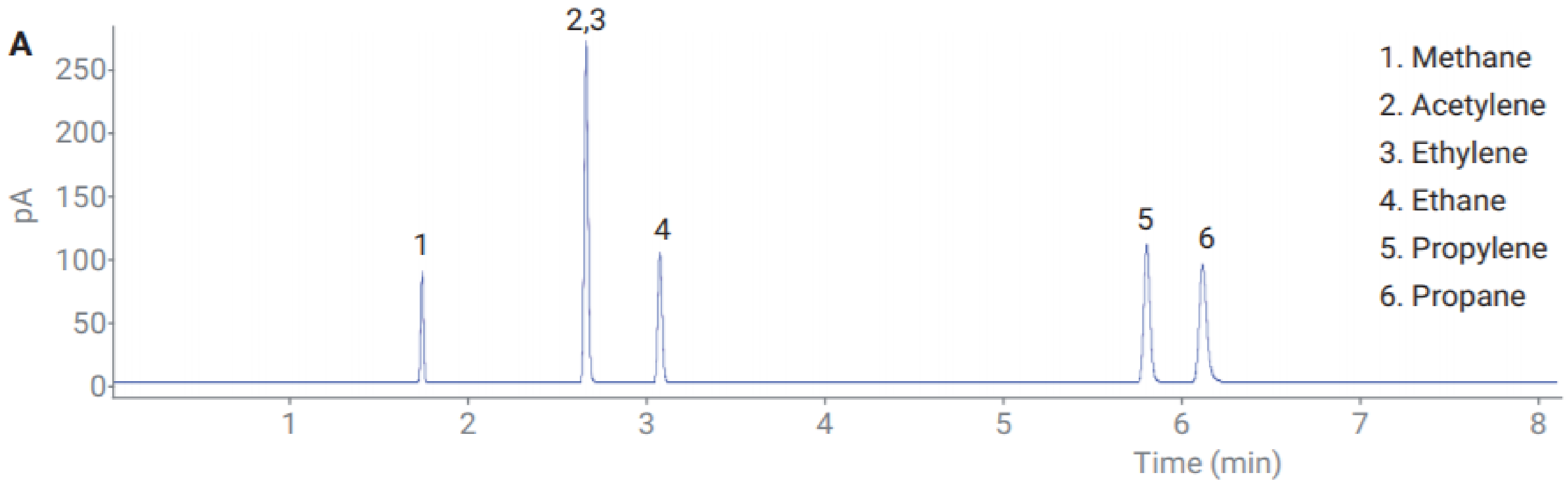
True

Or

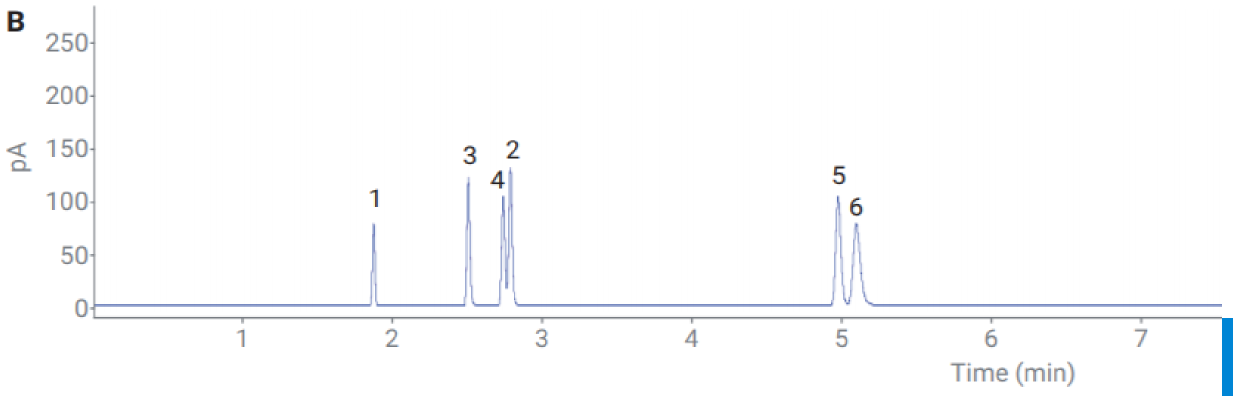
False

Separation of Volatile Organic Hydrocarbons with Agilent J&W PLOT GC Columns and Selectivity Tuning (5994-3485EN)

PoraPLOT Q



PoraPLOT S



PoraPLOT U