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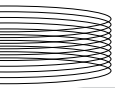
GC users guide

Fused Silica GC Capillary Columns

Zebron™



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General Considerations for Column Selection

To define a capillary column, four parameters must be specified:

1. **LENGTH:** Length is directly related to overall efficiency of the column and to overall analysis time. The relation between length and resolution is a square root relation, while analysis time is directly related to column length.

2. **INTERNAL DIAMETER:** Column internal diameter has a major impact on column efficiency (and thus on resolution) and on the sample capacity of the column. It can also dictate limitations on the injection and detection techniques used and vice versa. Smaller (Internal Diameter) gives higher k, but lower capacity.

3. **FILM THICKNESS:** Film thickness determines solute retention and thus solute elution temperatures. It will also play an important role in the sample capacity of the column. Thin films are faster with higher resolution, but offer lower capacity.

4. **PHASE:** The stationary phase is the most influential column parameter. It not only determines the final resolution obtained (it determines the relative retention of the solutes), but due to its specific characteristics, it will influence virtually every column selection parameter.

Other criteria like instrument availability, feasibility, budget considerations, etc. can also impose limitations on column selection. Experience, literature data and your Phenomenex Technical Representative are invaluable tools to guide you through the labyrinth of column selection.

General Considerations for Column Selection (continued)

I. Length

$\sqrt{L} \sim R$

A. Influence of length on resolution.
Increasing the length will increase resolution in a square root relation. Due to disadvantages of very long columns, increasing resolution by increasing the length is limited.

$\sqrt{L} \sim SC$

B. Influence of length on sample capacity.
Long columns have a higher sample capacity (square root relation).

$L \sim t_R$

C. Influence of length on analysis time.
In order to increase resolution or sample capacity by increasing the length, the total analysis time will also increase. Decreasing analysis time by increasing carrier gas velocity will cause the column efficiency to decrease.

General Guidelines

The length of the column should be chosen primarily to obtain an adequate efficiency.

- Short columns for sample screening.
- Longer columns when better separation is needed.
- Long columns for complex samples or closely eluting peaks.

STARTING ADVICE: Length 30 Meter

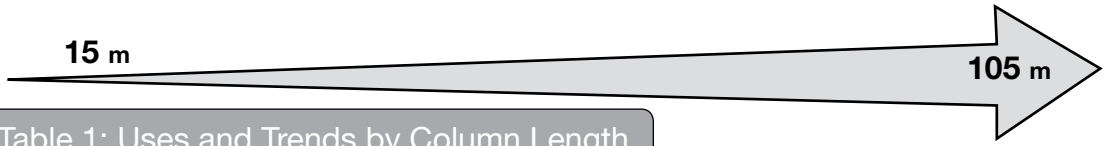


Table 1: Uses and Trends by Column Length

15 m Applications	Chromatographic Trends as Length Increases	105 m Applications
Rapid analysis	Increased retention times	Low boilers
High efficiency separations	Increased efficiency	More complex mixtures
Screening	Greater resolution	Less active samples
Simple mixtures		Programmed temperature analysis
High molecular weight compounds		
More chemically active components		

II. Internal Diameter (ID)

The new developments of capillary column technology, and specifically in column ID, are toward internal smaller diameters. These developments increase capillary column applicability.

A. Influence of internal diameter on resolution.

The smaller the internal diameter, the higher the efficiency.

The relation between efficiency and resolution is a square root relation. The evolution of ever-smaller diameters results in extremely high efficiency columns, ideally suited for high speed analysis and high detection sensitivity. Note that the influence of internal diameter on efficiency is most important with large β values (thin film columns).

B. Influence of internal diameter on sample capacity.

Narrow-bore columns have low sample capacity.

$$SC \sim r^2$$

Wide-bore columns have high sample capacity.

One of the drawbacks of capillary columns compared to packed columns was reduced sample capacity. The development of wide bore FSOT (0.53 mm ID) gave an extra dimension to capillary chromatography. The wide-bore columns combine the general advantages of capillaries with the high sample capacity of packed columns. It is not without reason these are referred to as “the packed column alternative”. Even a small increase of internal diameter (from e.g., 0.25 to 0.32 mm) results in a significant increase in sample capacity.

C. Influence of internal diameter on analysis time.

As a general rule, the smaller the internal diameter, the shorter the analysis time will be for a given resolution.

1. Wide-bore columns are less efficient (less theoretical plates / length). A longer column is needed to obtain the same column efficiency and $L \sim t_R$.
2. The internal diameter determines the optimal velocity (u_{opt}) of your column. Narrow-bore columns have high optimal velocities and the loss in efficiency at velocities higher than u_{opt} is minimal (see Figure 11, p.40).

General Considerations for Column Selection (continued)

D. Other factors to take into consideration when selecting the internal diameter.

ID (mm)	Instrument Selection / Constraints
0.10	High operating pressures are needed. Might require system modifications. Easily overloaded, requires skilled technicians.
0.18	Similar operating parameters to the 0.25 mm ID. Increased efficiency allows for shorter run times. Good first step when looking to use narrow bore columns. Suited for use with mass spectrometers.
0.20	Smalll increase in efficiency vs. 0.25 mm ID columns. Suited for mass spectrometers.
0.25	Most popular dimension. On-column injection possible. Suited for mass spectrometric detection.
0.32	Easy to work with; good compromise between resolution and stability. Routine on-column injection. Compatible with newer mass spectrometers.
0.53	Can be installed in an adapted packed gas chromatograph. Easy on-column injection. Good for use with dirty or high level samples.

* Smaller columns yield higher detection sensitivity but lower sample capacity.

STARTING ADVICE: 0.25 mm Internal Diameter Column

0.10 mm → **0.53 mm**

Table 2: Uses and Trends by Column ID

0.10 mm Applications	Chromatographic Trends as ID Increases	0.53 mm Applications
Complex samples	Increased sample capacity	Less complex samples
High resolution analysis	Decreased resolving power Lower efficiency	Sample components with wide range of concentrations
Low sample concentrations	Lower detectability	High sample concentrations
Can use only capillary column inlet	Lower optimal carrier gas velocities	Suitable with either packed or capillary column inlets

General Considerations for Column Selection (continued)

III. Film Thickness

A. Influence of film thickness on resolution (see equation)

1. Influence on capacity factor (k).

$$k = \frac{K_D}{\beta} = \frac{K_D 2d_f}{r} \text{ and } \frac{k}{k+1} \sim R$$

The retention of the solute by the stationary phase largely depends on the temperature ($K_D \sim T$) and on the β value. For low k solutes (volatile components), retention can be increased through the β value (higher film thickness). Note that the gain in resolution through increasing the capacity factor is limited.

$$d_f \sim 1/N \text{ and } \sqrt{N} \sim R$$

2. Influence on efficiency (see equation).

Two general rules can be applied:

Thick films are less efficient. For thick film columns (low β value), the resistance to mass transfer in the liquid phase (C_L) can no longer be ignored.

Non-polar thick film columns have a higher efficiency than polar thick film columns. The diffusivity in the liquid phase (D_L) is determined by the nature of the solute, the temperature and the nature of the phase. In general, the more polar the stationary phase, the more D_L will decrease. Very thick film coatings with good efficiency can be made for non-polar columns. The maximal film thickness of polar columns is restrained due to the overwhelming loss in efficiency.

$$d_f \sim SC$$

B. Influence on sample capacity.

Thick film columns have greater sample capacity than thin film columns.

$$d_f \sim k$$

C. Influence on analysis time.

The thicker the film, the longer the retention time of the solutes will be. For highly volatile components, thick film columns are a necessity. Less volatile components may require very thin films (0.1 µm) to enable them to elute from the column.

General Considerations for Column Selection (continued)

D. Other factors to take into consideration.

- For analyses where normally subambient temperatures are required, a thick film column may allow the analyst to work at ambient oven temperatures. However, the boiling point range of the sample components should not be too wide, otherwise those with the highest boiling point will not elute from the column, even at maximal operating temperatures.
- A thick film coating will result in a more inert column. This is especially important if the solute components have reactive functional groups.
- Thick films have a higher bleed and lower maximal operating temperatures. This makes them less suited to use in combination with mass spectrometric detection when high oven temperatures are required.

General Guidelines

Thin film columns for the analysis of high boiling compounds.

Medium film columns for:
- solutes with wide boiling point range.
- medium boiling compounds.

Thick film columns for volatile compounds.

STARTING ADVICE: Thin Film: 0.10 µm Medium Film: 0.25 µm Thick Film: 1.0 µm

Table 3: Uses and Trends by Column Film Thickness

0.10 µm Applications	Chromatographic Trends as Film Thickness Increases	5.0 µm Applications
Rapid analysis	Increased retention times	Low boilers, e.g., gases, solvents, purgeables (BP<25 °C)
High efficiency separations	Increased resolution (for low boiling compounds)	Used more with wide ID columns
High MW compounds	Upper temperature limit decreases	More chemically active components
Less chemically active components	Bleed increases	
MS applications	More narrow boiling point range for analytes	

IV. The Right Phase

A. Influence of the stationary phase on resolution.

1. Stationary phase determines selectivity.

$$R \sim \frac{\alpha - 1}{\alpha}$$

Resolution between two components is mainly determined by the selectivity (α) of the stationary phase. This selectivity depends on the nature of the components, on the nature of the stationary phase and on the temperature.

Selectivity is based on differences in interaction between analytes and the stationary phase. Interactive forces include:

- Dispersive forces
- Dipole (permanent or induced) interactions
- Acid-base interactions
- Non-polar phases offer primarily dispersive interactions, and separation between components will be based on boiling point
- More polar phases (with different chemical groups) will have more possible ways of interaction. Separation is not only based solely on boiling point, but also due to interactions between functional groups. Retention will occur according to relative chemical functionality

2. Intrinsic phase characteristics determine the achievable efficiency.

Greater efficiency will lead to greater resolution, provided that selectivity is available. Non-polar stationary phases will provide the highest efficiencies due to a better diffusivity and a higher coating efficiency.

B. Influence of stationary phase on sample capacity.

The sample capacity for a given solute also depends on the sort of interactions which occur, and thus on the nature of the stationary phase and of the solute (i.e., on a non-polar phase, very polar components will show easy overloading).

General Considerations for Column Selection (continued)

C. Influence of stationary phase on analysis time.

To obtain a certain resolution, the analysis time can be decreased with a higher selectivity and efficiency. As gas chromatography is based on volatility, make sure to take the temperature range of your column into consideration.

D. Other factors to take into consideration when selecting the stationary phase.

1. Non-polar phases offer several advantages:

- inertness and low bleed: useful for MS detection
- wide temperature range
- wide range of film thicknesses
- user-friendly columns
- more resistant to traces of oxygen and water in the carrier gas

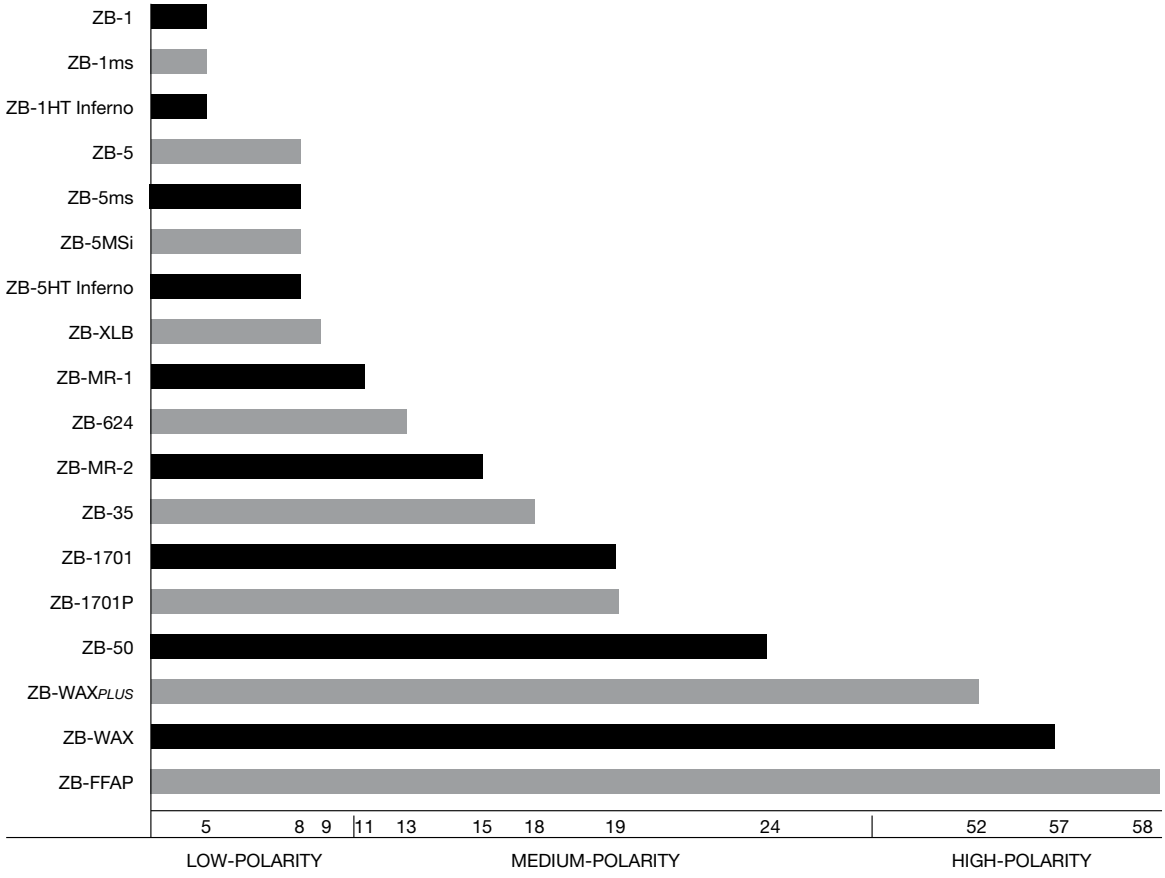
2. Only immobilized phases are solvent resistant.

3. Gas chromatography is based on volatility. The maximum operating temperature of the column should be sufficiently high to elute all components.

General Considerations for Column Selection (continued)

STARTING ADVICE: Non-Polar Phase ZB-5ms or Polar Phase ZB-WAX*PLUS*

Table 4: Phase Polarity



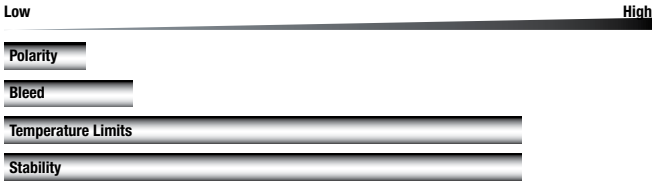
ZB-1

Alternative to any 100 % Dimethylpolysiloxane Phase:

REPLACES

DB-1, HP-1, MET-1, HP-101, HP-PONA, Rtx-1, Rtx-1 F&F, Rtx-1PONA, SPB-1, CP-Sil 5 CB, BP1, 007-1, OV-1, SE-30, AT-1, Ultra 1, DB-1 EVDX, DB-2887

Column Profile



Temperature Limits: -60 to 360/370 °C (Isothermal / TPGC)*

- Low polarity column
- Used for “fingerprinting” and routine quality control analyses (e.g., citrus oils)
- Equivalent to USP Phase G2

ZB-1 Applications:

Amines	Oxygenates and GRO	Drugs of abuse	Pesticides
Ethanol	PCBs	Essential oils	Semi-volatiles
Gases (refinery)	Simulated distillation	Hydrocarbons	Sulfur compounds (light)
MTBE	Solvent impurities	Natural gas odorants	Mercaptans

**Thicker films (≥1.0 µm df) are rated to 340/360 °C (Isothermal / TPGC)*

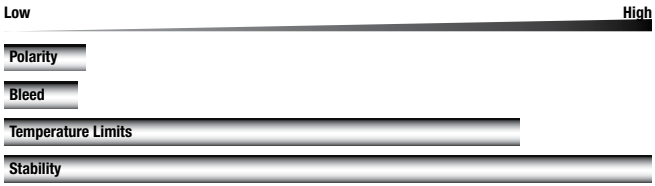
ZB-1ms

Alternative to Any MS-Certified 100 % Dimethylpolysiloxane Phase:

REPLACES

DB-1ms, HP-1ms, Rtx-1MS, VF-1ms, SolGel-1ms, CP-Sil 5 CB ms, Equity-1, MDN-1, AT-1ms

Column Profile



Temperature Limits: -60 to 360/370 °C (Isothermal / TPGC)

- Lowered bleed (MS Certified) especially suited to high sensitivity GC/MS
- Extremely inert for active compounds such as drugs or pesticides
- Improved signal-to-noise ratio for better sensitivity and mass spectral integrity
- Identical selectivity to the ZB-1
- Available with Guardian Integrated Guard Columns
- Equivalent to USP Phase G2

ZB-1ms Applications:

Amines	Polychlorinated biphenyls (EPA Method 1668)	Pesticides
Acids	Drugs of abuse	
Diesel Fuel	Flavors & fragrances	

ZB-1HT Inferno™

Alternative to Any 100 % Dimethylpolysiloxane High-Temperature Phase:

REPLACES

DB-1HT
MXT-1 SimDist
Petrocol 2887
CP-SimDist

2007 R&D 100 Award Recipient

Column Profile

Temperature Limits: -60 to 400/430 °C (Isothermal / TPGC)*

- First non-metal 100 % dimethylpolysiloxane phase stable to 430 °C
- Individually tested for low bleed, MS certified
- Rugged high temperature, polyimide coated, fused silica tubing
- Provides true boiling point separation for hydrocarbon distillation methods
- Low activity, provides good peak shape for acidic and basic samples
- Provides robust column performance for high temperature bake outs

ZB-1HT Inferno **Applications:**

High boiling petroleum products	High molecular weight waxes	Motor oils
Simulated distillation methods	Polymers/plastics	
Long-chained hydrocarbons	Diesel fuel	

* *0.53 mm ID columns are rated to 400 °C max operational temperature*

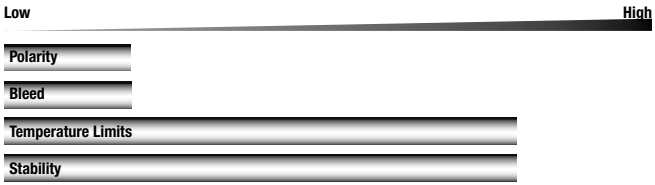
ZB-5

Alternative to Any 5 %-Phenyl-95 %-Dimethylpolysiloxane Phase:

REPLACES

DB-5, HP-5, HP-PAS-5, HP-5 Trace Analysis, Rtx-5, SPB-5, MDN-5, CP-Sil 8 CB, 007-5, OV-5, SE-54, AT-5, Equity-5, EC-5, Ultra 2, BP5, BPX5, HP-HP-101

Column Profile



Temperature Limits: -60 to 360/370 °C (Isothermal / TPGC)*

- Versatile low polarity column
- Low bleed (MS Certified) especially suited to high sensitivity work using GC/MS
- Extremely inert for active compounds such as drugs or pesticides
- Resilient to dirty samples - long column life
- Great column for unknown samples
- Equivalent to USP Phase G27

ZB-5 Applications:

Alkaloids	Residual solvents	Drugs	PCBs/aroclors
FAMEs	Essential oils/flavors	Halo-hydrocarbons	Phenols
Semi-volatiles	Pesticides/herbicides	Dioxins	

**Thicker films (≥1.0 µm df) are rated to 340/360 °C (Isothermal / TPGC)*

ZB-5ms

Alternative to Any MS-Certified 5 %-Phenyl-Arylene-95 %-Dimethylpolysiloxane Phase:

REPLACES

DB-5ms
DB-5.625
DB-5ms EVDX
VF-5ms
CP-Sil 8 CB MS

Column Profile

Temperature Limits: -60 to 325/350 °C (Isothermal / TPGC)

- Arylene Matrix Technology (AMT)
- Fully conditioned within 35 minutes
- High response for acids and bases = Very low activity
- Enhanced resolution of Polyaromantic Hydrocarbons (PAHs) and other multi-ring aromatic compounds
- The perfect choice for EPA methods 525, 610, 625, 8100, and 8270
- Equivalent to USP Phase G27

ZB-5ms Applications:

Alkaloids	Essential oils/flavors	Pesticides/herbicides	Amines
FAMEs	Semi-volatiles	PCBs/aroclors	Acids
Phenols	Drugs	Solvent impurities	Dioxins
Residual solvents	Halo-hydrocarbons	EPA methods	

ZB-5MSi

Alternative to Any 5 %-Phenyl- 95 %-Dimethylpolysiloxane Phase:

REPLACES

Rtx-5ms, MDN-5S, HP-5ms, Rtx-5Amine, HP-5msi, Rxi-5ms, DB-5

Column Profile

Temperature Limits: -60 to 360/370 °C (Isothermal / TPGC)

- Low bleed and highly inert 5% phenyl column
- Improved peak shape for acidic/basic compounds
- Maximum sensitivity from MS certified bleed levels
- Reproducible column-to-column performance insured by industry leading QC specifications
- High phase stability and high temperature limits
- Traditional bonding chemistry provides the same selectivity as the ZB-5 columns

ZB-5MSi Applications:

Drugs of abuse	FAMEs	Pesticides	Nitrosamines	Phenols	EPA methods

ZB-5HT Inferno™

Alternative to Any 5 %-Phenyl-95 %-Dimethylpolysiloxane High-Temperature Phase:

REPLACES

DB-5HT
HT-5
VF-5HT
Stx-5HT
XTI-5HT

2007 R&D 100 Award Recipient

Column Profile

Temperature Limits: -60 to 400/430 °C (Isothermal / TPGC)*

- First non-metal 5 %-phenyl 95 %-dimethylopolysiloxane phase stable to 430 °C
- Individually tested for low bleed, MS certified
- Rugged high temperature, polyimide coated, fused silica tubing
- Low activity, provides good peak shape for acidic and basic samples
- Provides robust column performance for high temperature bake outs

ZB-5HT Inferno **Applications:**

High boiling petroleum products	Polymers/plastics	Diesel fuel
Simulated distillation methods	High molecular weight waxes	Motor oils
Long-chained hydrocarbons	Triglycerides	Surfactants

* *0.53 mm ID columns are rated to 400 °C max operational temperature*

ZB-35

Alternative to Any 35 %-Phenyl-65 %-Dimethylpolysiloxane Phase:

REPLACES

DB-35ms, HP-35, Rtx-35, SPB-35, MDN-35, BPX 35, AT-35, 007-11, OV-11, Sup-Herb, SPB-608, BPX608, Rtx-35MS, DB-35, HP-35ms, EC-35

Column Profile

Temperature Limits: 50 to 340/360 °C (Isothermal / TPGC)

- Intermediate polarity column with temperature limits up to 360 °C - allows high molecular weight analysis
- Excellent inertness to minimize analyte adsorption, improve efficiency, and reproducibility
- More rugged (longer column life) than other polar phases
- Excellent for trace analysis with bleed-sensitive detectors (MS, FID, ECD, NPD)
- Equivalent to USP Phase G42

ZB-35 Applications:

Aroclors	Pesticides	Semi-volatiles	Steroids
Amines	Pharmaceuticals	Drugs of abuse	EPA methods 508, 608, 8081, 8141, 8151

ZB-50

Alternative to Any 50 %-Phenyl-50 %-Dimethylpolysiloxane Phase:

REPLACES

DB-17, HP-50+, Rtx-50, DB-17ht, SPB-50, SP-2250, CP-Sil 24 CB, BPX50, AT-50, 007-17, DB-17ms, DB-17 EVDX, SPB-17

Column Profile

Temperature Limits: 40 to 320/340 °C (Isothermal / TPGC)

- High polarity column with temperature limits up to 340 °C allows high-temperature bake-out to remove contaminants
- Excellent inertness to minimize analyte adsorption, improve efficiency, and reproducibility
- More rugged (longer column life) than other polar phases
- Excellent for trace analysis with bleed-sensitive detectors
- Great for drug screening and environmental compounds
- Equivalent to USP Phase G3

ZB-50 Applications:

Antidepressants	Pesticides/herbicides	Cholesterols
Drugs of abuse	Steroids	Triglycerides
Glycols	Aroclors	EPA methods 508, 608, 8081, 8141, 8151

ZB-624

Alternative to Any 6 %-Cyanopropylphenyl-94 %-Dimethylpolysiloxane Phase:

Temperature Limits: -20 to 260 °C

- Formulated for low bleed
- Excellent for US EPA Methods 501.3, 502.2, 503.1, 524.2, 601, 602, 8010, 8015, 8020, 8240, 8260, 8021
- Specifically designed for the separation of volatile organic compounds (VOCs)
- Increased temperature limit speeds run times and re-equilibration
- Widely used phase to separate volatile organic flavor and fragrance additives and residual solvents in industrial or pharmaceutical products (OVIs)
- Equivalent to USP Phase G43

ZB-624 Applications:

Volitile organic compounds (VOCs)	Residual solvents	EPA methods 524, 624, 8260

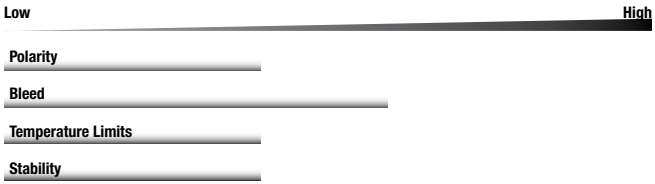
ZB-1701

Alternative to Any 14 %-Cyanopropylphenyl-86 %-Dimethylpolysiloxane Phase:

REPLACES

DB-1701, Rtx-1701, SPB-1701, CP-Sil 19 CB, OV-1701, 007-1701, AT-1701, BP10, Equity 1701, Rtx-VMS

Column Profile



Temperature Limits: -20 to 280/300 °C (Isothermal/TPGC)*

- Fast run and re-equilibration times for enhanced sample throughput and productivity
- Provides alternate selectivity to phenyl phases with similar polarity
- Equivalent to USP Phase G46

ZB-1701 Applications:

Pharmaceutical intermediates	Esters	PAHs	Solvents
Alcohols	Drugs	Steroids	
Phenols	PCBs	TMS sugars	
Tranquilizers	Aromatic hydrocarbons	Amines	

**Thicker films (≥1.0 µm df) are rated to 260/280 °C (Isothermal / TPGC)*

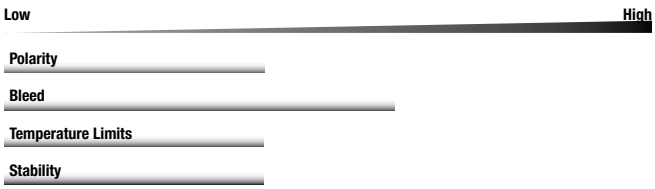
ZB-1701P

Alternative to Any 14 %-Cyanopropylphenyl-86 %-Dimethylpolysiloxane Phase:

REPLACES

DB-1701P

Column Profile



Temperature Limits: -20 to 280/300 °C (Isothermal/TPGC)*

- Specially tested to ensure response of DDT, Endrin, Endrin Aldehyde, and Endrin Ketone
- Fast run and re-equilibration times for enhanced sample throughput and productivity
- Equivalent to USP Phase G46

ZB-1701P Applications:

Organochlorine pesticides	Organophosphorous pesticides	Nitrogen containing pesticides	Aroclors

**Thicker films (≥1.0 µm df) are rated to 260/280 °C (Isothermal / TPGC)*

ZB-WAX

Alternative to Any Polyethylene Glycol Phase:

REPLACES

HP-INNOWax, Rtx-WAX, CP-Wax 57 CB, 007-CW, EC-Wax, Stabilwax-DB, SolGel-WAX, FAMEWAX, DB-WAXetr, Met-Wax, Omegawax, BP20

Column Profile

Temperature Limits: 40 to 250/260 °C (Isothermal/TPGC)

- Low bleed, (MS Certified)
- Highly stable, long lifetime
- Low activity for amines
- Bonded, solvent rinsible
- Excellent chromatography of polar complex mixtures
- Widely used for profiling and “fingerprinting”
- Close equivalent to USP Phase G16

ZB-WAX Applications:

Alcohols	OVIs	Aldehydes	Pharmaceuticals
Aromatics	Solvents	Essential oils	Styrene
Flavors/fragrances	Xylenes	Glycols	Basic compounds

ZB-WAX*PLUS*

Alternative to Any Polyethylene Glycol Phase:

REPLACES

DB-WAX, Supelcowax-10, Carbowax-20M, PEG 20M, AT-WAX, Permabond CW 20M, HP-20M, CP-WAX 52 CB, CAM, Stabilwax, AT-AquaWax BP20, BP 20

Column Profile

Temperature Limits: 20 to 250/260 °C (Isothermal/TPGC)*

- 100 % Aqueous stable
- Extremely inert for acidic compounds
- Enhanced selectivity for low boiling solvents
- High retention of alcohols and other chlorinated solvents
- Increased efficiency at 20 °C
- Bonded and solvent rinsible
- Equivalent to USP Phase G16

ZB-WAX*PLUS* Applications:

Alcoholic beverages	Glycols	Alcohols	Pharmaceuticals
OVIs	Aldehydes	Solvents	Aromatic
Styrene	Essential oils	Xylene isomers	Flavors/fragrances
Acids (free)			

**Thicker films (≥1.0 µm df) are rated to 230/240 °C (Isothermal / TPGC)*

ZB-FFAP

Alternative to Any Nitroterephthalic Acid Modified Polyethylene Glycol:

REPLACES

DB-FFAP, HP-FFAP, BP21, Nukol, CP-WAX 58 CB, 007-FFAP, Stabilwax-DA, PE-FFAP, AT-1000, CP-FFAP-CB, SPB-1000, EC-1000

Column Profile

Temperature Limits: 40 to 250/260 °C (Isothermal/TPGC)

- High polarity column
- Especially suited for organic acids, free fatty acids, and alcohols
- Excellent thermal and chemical stability
- Bonded FFAP Phase
- Replaces OV-351
- Close equivalent to USP Phase G35

ZB-FFAP Applications:

Acrylates	Ketones	Alcohols	Volatile free acids
Aldehydes	Organic acids	Free fatty acids	Phenols

ZB-XLB

Alternative to Any 5 %-Phenyl- 95 %-Dimethylpolysiloxane Phase:

Temperature Limits: 30 to 340/370 °C (Isothermal / TPGC)

- Unique low polarity si-arylene column
- Engineered specifically for use with bleed sensitive detectors such as MS
- Provides alternate selectivity to standard 5-type phases
- Often used for confirmation of pesticides, PCBs, or other environmental samples
- Good tool for sample screening to identify unknown contaminants

ZB-5msi Applications:

Polychlorinated biphenyls (PCBs)	Pesticides	Herbicides

**Thicker films (≥1.0 µm df) are rated to 260/280 °C (Isothermal / TPGC)*

Exclusive

phenomenex

ZB-MULTIRESIDUE™ (MR)-1

ESC EQUIPPED CERTIFIED MS

SIMILAR* TO

Rtx-CLPesticides

Stx-CLPesticides

DB-XLB

Rtx-XLB

Column Profile

Temperature Limits: -60 to 320/340 °C (Isothermal/TPGC)

- Proprietary phase specially designed for the separation of all types of pesticides, herbicides, and insecticides
- When used in parallel with the Zebron MR-2 column, provides baseline resolution and confirmation of all 20 chlorinated pesticides regulated under EPA Method 8081 in <10 min
- MS Certified phase provides low bleed performance for pesticide confirmation by MS
- Low activity, decreased breakdown of sensitive pesticides such as DDT
- Provides robust column performance for high temperature bake outs

ZB-MULTIRESIDUE (MR)-1 Applications:

Organochlorine pesticides	Nitrogen containing pesticides	Insecticides	Multi-pesticide residue methods
Organophosphorous pesticides	Herbicides	Aroclors/PCBs	Habacetic acids

*Not exact equivalent, selectivity might be different

Exclusive

phenomenex

ZB-MULTIRESIDUE™ (MR)-2

ESC EQUIPPED CERTIFIED MS

SIMILAR* TO

Rtx-CLPesticides 2

Stx-CLPesticides 2

Column Profile

Temperature Limits: -60 to 320/340 °C (Isothermal/TPGC)

- Unique proprietary polymer chemistry - unlike any other column on the market
- Specially designed for the separation of all types of pesticides, herbicides, and insecticides
- When used in parallel with the Zebron MR-1 column, provides baseline resolution and confirmation of all 20 chlorinated pesticides regulated under EPA Method 8081 in <10 min
- MS Certified phase provides low bleed performance for pesticide confirmation by MS
- Low activity, decreased breakdown of sensitive pesticides such as DDT
- Provides robust column performance for high temperature bake outs

ZB-MULTIRESIDUE (MR)-2 Applications:

Organochlorine pesticides	Nitrogen containing pesticides	Insecticides	Multi-pesticide Residue methods
Organophosphorous pesticides	Herbicides	Aroclors/PCBs	Habacetic acids

*Not exact equivalent, selectivity might be different

Column Installation

Please Note: Remember to wear safety glasses when near any gas system, cutting capillary tubing, and generally as you work around the lab.

Pre-Installation Check List

- Replace oxygen, moisture and hydrocarbon traps as necessary.
- Check gas cylinder pressures to ensure that an adequate supply of carrier, make-up and fuel gases is available. Carrier gases should be of the highest possible (affordable) purity. **Note**: It is critical that oxygen and water, normally present in gas cylinders, be removed from the carrier gas by the appropriate use of filters and adsorbents.
- Ensure that the injection port is clean and free of sample residues, septum or capillary debris.
- Check and replace as necessary the critical injector components such as seals, liners and septa.
- Check and replace detector seals as necessary.
- Carefully inspect your column for damage or breakage.

Installation Tools and Supplies

- Ceramic wafer or sapphire-tipped pencil
- Magnifier (10-20X)
- Ruler
- Marker (e.g., correction fluid)
- Wrenches
- Ferrules
- Vial of injection solvent
- Injection syringe
- Supply of an appropriate non-retained compound (e.g., methane)
- Appropriate column test mixture
- Gas flow meter
- Supply of replacement parts and accessories
  e.g., septa, liners, ferrules, O-rings

Detailed Column Installation Instructions

The following is a brief reminder of the general precautions required in handling and installing any organic-coated fused silica capillary column. Consult your GC manual for more details.

Fused silica capillary columns become brittle if the polyimide coating applied during manufacture is damaged. Avoid temperatures above recommended operating limits and excessive bending, twisting, and abrasion of columns, which will damage this protective coating. Remember, even if the column does not break immediately, when the protective coating is damaged the column may break spontaneously later.

The stationary phase, which coats the inside of the column, must also be protected. The ends of the column will be sealed or protected by a septum when you receive the column. Once the ends are open in preparation for installation, the column should be installed in a chromatograph as soon as practical and a flow of dry, oxygen-free carrier gas maintained until the column is removed and resealed.

All foreign material including debris from the septa or ferrules must be kept out of the column.

Figure 1: Proper and Improper Cut Capillary End

☑ **Correct** ☒ **Incorrect**

Examples of: (a) A Properly Cut Fused Silica Column End
(b) An Improperly Cut End

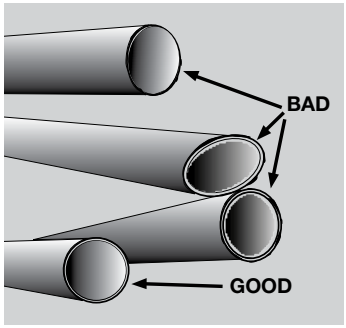
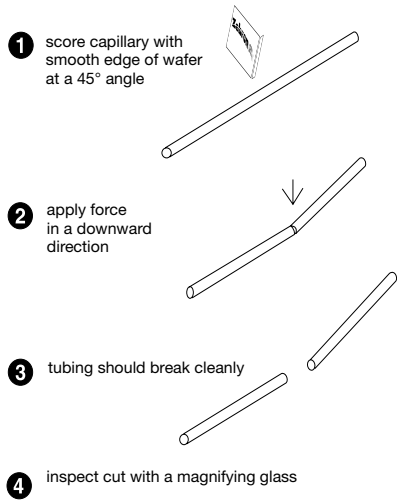


Figure 2: Cutting Fused Silica Tubing



Critical Column Installation Steps

Injector Installation:

1. Install a nut and ferrule. Cut a centimeter or two off an end of the column (Figure 2). Ensure cut is clean and square (Figure 1). Be sure the ferrule is the right size and the tapered end is toward the end.

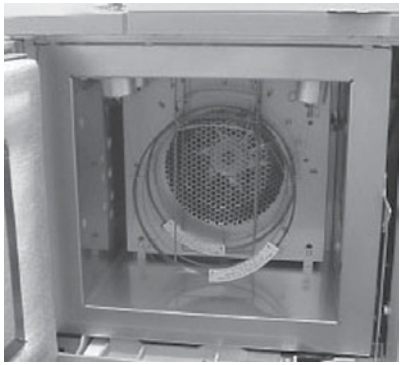
2. Mount the column in the GC oven without damaging the column coating. (Figure 3)

3. Insert the column into the injector exactly the correct distance specified in the instrument manual (Figure 4). Tighten the ferrule nut until the column resists movement. One-quarter turn past finger-tight is about right.

4. Adjust the head pressure to obtain the flow rate listed on the test chromatogram.

5. Check the inlet connections for leaks.

Figure 3: Hang column without damaging polyimide coating



Detector Installation:

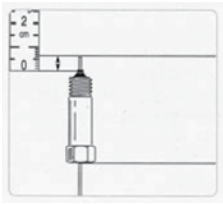
1. Install a nut and ferrule. Cut a centimeter or two off an end of the column (Figure 2). Ensure cut is clean and square (Figure 1). Be sure the ferrule is the right size and the tapered end is facing the correct direction.

2. Insert the outlet end of the column into the detector exactly the distance prescribed in the instrument manual.

3. Tighten the ferrule nut until the column resists movement. One-quarter turn past finger-tight is about right. After the column is equilibrated, inject a detectable unretained sample such as methane to determine dead volume time and linear gas velocity. Adjust gas pressure to obtain proper values for your analytical method.

4. The methane peak must have ideal peak-shape or the installation is faulty!

Figure 4: Measure proper injection port distance



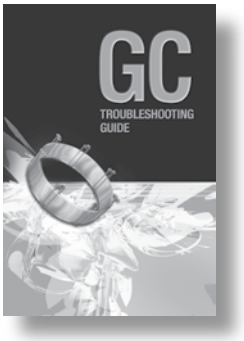
Installing the Column

A. Conditioning and Testing the Capillary Column

1. Purge the column with carrier gas for approximately 15 minutes. Further conditioning may be desirable.
2. Insert the outlet end of the column into the detector **exactly the distance prescribed** in the instrument manual. Use all purpose correction fluid to mark the exact insertion distance.
3. Set gas-flow rates to instrument specifications. **Warning! Some detectors may be damaged by heating without proper gas flow.**
4. Check the system for leaks. It is preferable to use a thermal-conductivity-type leak detector. Do not use soaps or liquid-based leak detectors with capillary columns. **Never heat the column without checking thoroughly for leaks first.**
5. Set injector and detector temperatures. Turn the detector on when steady state temperatures are achieved.
6. Increase the oven temperature to the maximum continuous operating temperature for the column. **Warning! Do not exceed the maximum operating temperature of the column**. Maintain that temperature until a flat baseline is observed. If this takes more than an hour, it could indicate a problem.
7. Inject a detectable unretained sample such as methane (see Tables 11 and 12) to determine dead volume time and linear gas velocity. Adjust gas pressure to obtain proper values for your analytical method.
8. Set oven to starting temperature. Inject another sample of a detectable unretained substance. Re set the carrier gas velocity to desired value.
9. Check the performance of the GC and the column by injecting a known sample or performance test mix. If all peaks tail, it could indicate loose fittings, improper column installation, or broken liner. See the Section on *Troubleshooting Installation Problems on page 34*.
10. Calibrate the instrument.
11. Inject a sample, ensuring that the vaporized sample volume does not exceed the inlet sleeve's buffer volume (see Tables 6 and 9).
12. For short-term standby operation of the GC instrument, continue carrier gas flow at 100-200 °C. Long-term standby conditions require that the column be removed from the instrument, flame-sealed or end-capped with septa, and stored away from light in its original box.

Troubleshooting Installation Problems

More often than not, GC column problems are traceable to something improperly done during installation. For a more complete treatment of the subject, see our **FREE** guidebook, “GC Troubleshooting”.



Hints and Tips

Protecting the Column

It is important to protect the column and instrument components from exposure to dirty samples. Non-volatile or high molecular weight components can contaminate the stationary phase, causing peak resolution and quantitative accuracy to significantly degrade. If possible, filter your samples prior to injection and use packing (glass wool or silanized fused silica) in the inlet sleeve to remove sample residue before it can enter the column. Refer to your instrument manual for specific instructions on packing the inlet sleeve.

Poor column lifetime is usually caused by non-volatile or caustic contaminants in the sample damaging the first 4 - 6 in. of the column. Cutting off the damaged portion will usually restore the column performance, but over time performance will degrade to a point where the column can no longer be used. If you are experiencing rapid degradation of column performance, there are several simple ways to help protect your column and increase lifetime.

Please contact a Phenomenex Technical Representative for steps to improve column lifetime.

Hints and Tips (continued)

Liners

The liner is the first line of defense for the column and the style chosen can make a big difference in how much contamination gets onto the column. The easiest thing to do is to add a small amount of silanized glass wool to a liner, which traps the non-volatile compounds and prevents them from entering the column. Glass wool will also help catch pieces of septa that result after repeated injections.

Caution: glass wool can also add activity for acids, bases, and pesticides. Activity is the result of free silanol (-OH) groups on the surface of the glass wool that form even after it has been deactivated. Crushing the glass wool can lead to increased activity, so it is recommended to purchase pre-packed liners, rather than try to pack your own.

Here are some liners that are available pre-packed with glass wool or provide additional column protection:

Description GC Model No.	Dimensions IDxLxOD (mm)	Material* (deactivated)	Quartz Wool (Y / N)	Mfr. No.	Part No.	Unit
Split/Splitless						
5880/5890/6890	4 x 78.5 x 6.3	B (y)	Y	092002	AG0-7515	5/pk
				092219	AG0-7582	25/pk
Split/Splitless, Recessed Gooseneck Liner						
5880/5890/6890	1.5 x 78.5 x 6.3	B (y)	N	5183-4691	AG0-4661	5/pk
				5183-4692	AG0-4662	25/pk
Cup Splitter/Split Liner						
5880/5890/6890	4 x 78.5 x 6.3	B (n)	N	5183-4699	AG0-4647	5/pk
				5183-4700	AG0-4648	25/pk
Cup Splitter/Split Liner						
Autosystem	3.5 x 100 x 5	B (n)	N	0330-5181	AG0-4663	5/pk
Split/Splitless Single Taper/Gooseneck Liner						
5880/5890/6890	4 x 78.5 x 6.3	B (y)	Y	5183-4693	AG0-4657	5/pk
				5183-4694	AG0-4658	25/pk

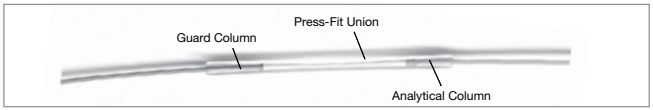
** B = Borosilicate; Deactivated = Yes (y) or No (n)*

Guard Columns – Standard Guards

Z-Guard columns are 5 or 10 m pieces of deactivated tubing that are connected to an analytical column using a glass press-fit connector (Figure 5). The tubing acts like a trap for non-volatile residues that would otherwise damage the stationary phase of your analytical column. Since there is no stationary phase in the Z-Guard, cutting the column does not significantly affect retention times or chromatographic separation. Additional Z-Guards may be attached as necessary, as long as the analytical column is still providing good separation.

Since guard columns can also be a source of activity, each Z-Guard is individually QC tested to ensure it is completely deactivated. This added level of quality ensures that you will get the best performance possible out of your column.

Figure 5: Z-Guard connection using press-fit union

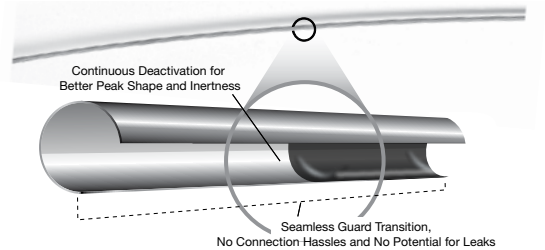


Guardian™ Integrated Guard Columns

Guardian columns have the 5 m or 10 m guard built directly into the analytical column in one continuous length of tubing (Figure 6). Unlike traditional guard columns, there is no mechanical connection between the guard and the analytical column. Each Guardian column undergoes a special deactivation and QC-testing procedure that ensures good performance for acids, bases, and other sensitive compounds. **The result: all the benefits of a guard column without the possibility of leaks or activity resulting from a faulty connection.**

The Guardian system is the ideal solution to all the problems associated with traditional guard columns!

Figure 6. Representation of a Guardian column



Column Bake Out

The easiest way to reduce column contamination is to add a short, high temperature bake out at the end of the standard GC method. This bake out helps remove high boiling contaminants that would otherwise remain in the column and cause damage. If adding a bake out significantly increases the method run time, a separate high temperature cleaning program can be run after every 10 injections or so.

The temperature used for bake out has a direct impact on the amount of contaminants removed. Using columns with higher upper temperature limits will increase the ability to remove unwanted contaminants. For example, the Zebron ZB-5 30 meter x 0.25 mm x 0.25 µm column can sustain temperature programs up to 370 °C, which is 20 °C higher than the ZB-5ms column of similar dimension. Check the upper temperature limit of the column currently used. If a high temperature bake out could improve column life, consider using a phase with increased thermal stability.

Apply this thought process to the column phase chosen when developing a method. For example, many pesticide applications are done on polar phases such as a Zebron ZB-1701 (14 %-cyanopropylphenyl-86 %-dimethylpolysiloxane), which has an upper temperature limit of 300 °C. In some cases, these same methods can be done on alternate phases such as the Zebron ZB-35 (35 %-phenyl–65 %-dimethylpolysiloxane), which has an upper temperature limit of 360 °C.

Considerations when Optimizing a GC Method: Phase Ratio (β)

When changing column dimensions, it is important to consider the affect it will have on the retention characteristics of the column. The Distribution Constant (K) describes the concentration of compound A in the stationary vs. the carrier gas mobile phase (Equation 1). Since a compound is only moving when it has entered the carrier gas, changes in this ratio shift the equilibrium and can affect column retention and selectivity if conditions do not change.

Equation 1: $K = \frac{[A_m]}{[A_s]} = k\beta$

A_m = Concentration of the solute in the Mobile Phase
A_s = Concentration of the solute in the Stationary Phase
k = capacity factor
β = Phase Ratio

When looking to optimize column dimensions, it is important to consider phase ratio (β) to ensure that selectivity will remain the same. Phase ratio for a given column is calculated using Equation 2; smaller β values result in greater retention. Chromatographically this means that when using columns of the same ID, the column with a thicker film will have greater retention for a given analyte. Table 5 lists the β values for common IDs and film thicknesses.

Equation 2: $\beta = \frac{ID}{4 \times d_f}$

ID = Internal Diameter (µm)
d_f = Film Thickness (µm)

Table 5: Phase ratio (β) value for common columns

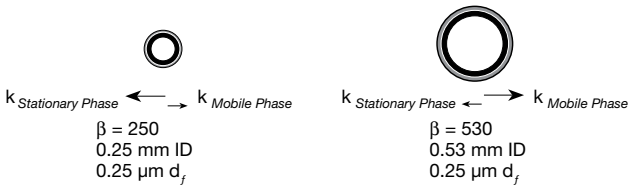
Film Thickness d_f (µm)	Column Diameter (mm)					
	0.10	**0.18**	**0.20**	**0.25**	**0.32**	**0.53**
0.10	250	450	500	625	800	1325
0.18	139	250	278	347	444	736
0.25	100	180	200	250	320	530
0.33	—	—	151	—	—	—
0.50	—	90	100	125	160	265
1.00	—	—	50	63	80	133
1.50	—	—	—	42	53	88
3.00	—	—	—	21	27	44
5.00	—	—	—	13	16	27

Increasing Retention

Increasing Retention

When using columns of two different IDs, the same film thickness does not translate to the same retention characteristics. Figure 5 demonstrates the phase ratio of a 0.25 µm film thickness on a 0.53 mm and a 0.25 mm ID column.

Figure 7: Phase ratio of a 0.25 µm film thickness on columns of differing internal diameters



Using Phase Ratio to our Advantage:

The optimum phase ratio depends on the goal of the separation. If analyte retention is low, a column with a low β can be used to increase retention. If column provides good retention, β can be reduced to increase column efficiency and decrease run time.

Let's use the separation of light hydrocarbon impurities found in butane as an example. On a column with a high β such as the Zebron ZB-1 60 meter x 0.32 mm x 0.25 µm (β = 360) the isomers co-elute due to the lack of interaction with the stationary phase (Figure 8). By using a column with a lower Beta such as the Zebron ZB-1 60 meter x 0.32 mm x 3.00 µm (β = 27), we are able to achieve separation (Figure 9).

Figure 8: Butane Isomer separation on 60 meter x 0.32 mm x 0.25 µm (β = 360)

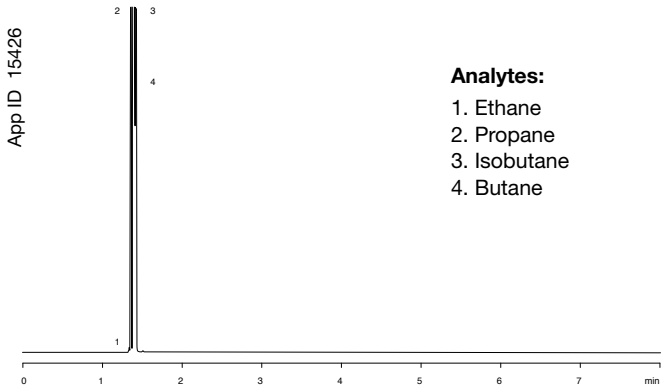
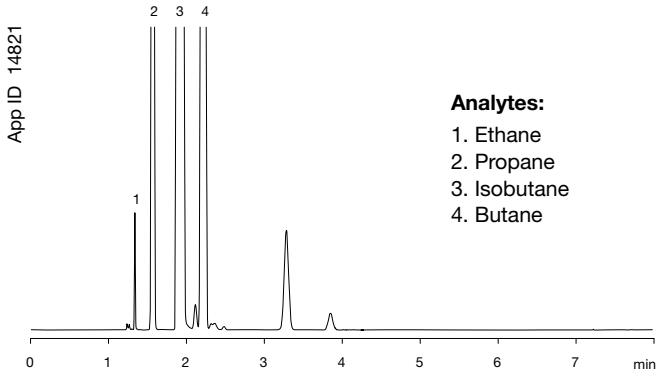


Figure 9: Butane Isomer separation on 60 meter x 0.32 mm x 3.00 µm (β = 27)



Shortening Run Times:

The increase in efficiency offered by narrow bore GC columns often improves separation enough to allow the same separation to be done in much less time. Figure 10 shows the separation of 17 priority Polyaromatic Hydrocarbon contaminants using a standard 30 meter x 0.25 mm x 0.25 µm column (β = 250). By choosing a column with similar phase ratio, but smaller ID the method can be shortened by over 50 % while still meeting resolution requirements for key analytes (Figures 11 & 12).

Hints and Tips (continued)

Figure 10: Separation of PAHs on ZB-5ms
30 meter x 0.25 mm x 0.25 µm (β = 250)

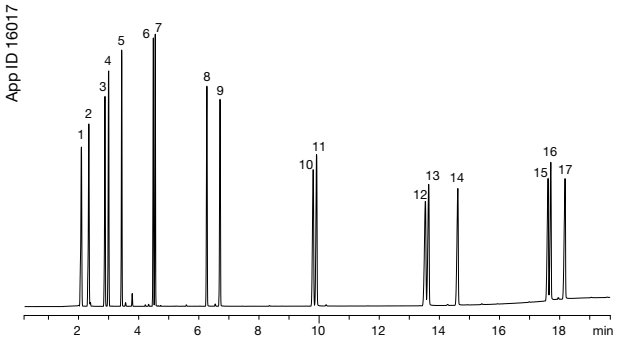


Figure 11: Separation of PAHs on ZB-5ms
20 meter x 0.18mm x 0.18 µm (β = 250)

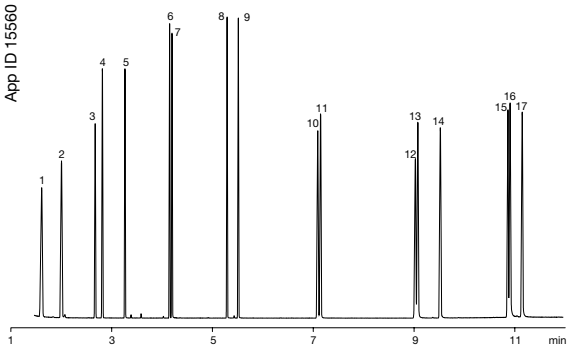
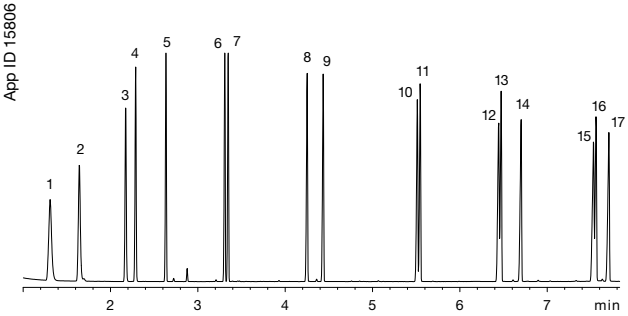


Figure 12: Separation of PAHs on ZB-5ms
10 meter x 0.10 mm x 0.10 µm (β = 250)



Analytes: (for figures 8-10)

1. Naphthalene
2. 2-Methylnaphthalene
3. Acenaphthalene
4. Acenaphthene
5. Fluorene
6. Phenanthrene
7. Anthracene
8. Fluoranthene
9. Pyrene
10. Benz[a]anthracene
11. Chrysene
12. Benzo[b]fluoranthene
13. Benzo[k]fluoranthene
14. Benzo[a]pyrene
15. Indeno[1,2,3-cd]pyrene
16. Dibenz[a,h]anthracene
17. Benzo[g,h,i]perylene

Phase ratio is a critical step in optimizing GC separation. If you would like more information on how it can be used to improve your chromatography, please contact your Phenomenex Technical Consultant.

Carrier Gas Selection and Flow Optimization

It is advisable to use the highest purity gas possible. Ultra high purity (99.99 %), ultra pure carrier (99.995 %), or even research grade (99.9999) is preferred to minimize critical impurities, instrument downtime and troubleshooting. Air, moisture and organic traps should be used, but it is better to start with the highest purity gas and reduce the load on gas purifiers as much as possible.

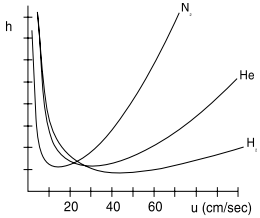
Helium should be used for capillary GC whenever possible; nitrogen shows inferior performance due to slow optimum linear velocity and steep van Deemter profile.

Three types of gas are commonly used as a carrier gas:

1. Hydrogen (H_2): Hydrogen will yield maximal number of theoretical plates for thin film columns and the high efficiency is largely retained at velocities higher than u_{opt}. Hydrogen is not generally recommended due to its hazardous nature.
2. Helium (He): When hydrogen is not used, helium is the best alternative for speed and sensitivity.
3. Nitrogen (N_2): Nitrogen is the last choice for thin film columns. For thick film columns, nitrogen yields the highest number of theoretical plates. However, the optimal velocity is fairly low (long analysis times), and the loss in efficiency at higher velocities is high. If resolution is sufficient, hydrogen or helium are good alternatives.

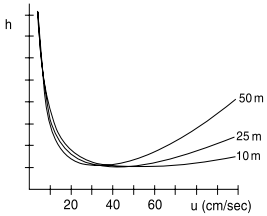
Figure 13: Carrier Gas Selection and Velocity Optimization Plots

Carrier Gas



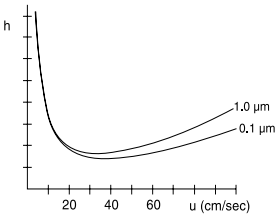
H (u) curves for different gases

Length



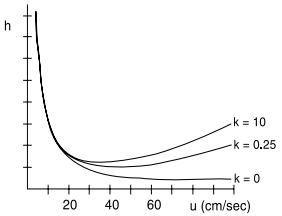
H(u) curves for different column lengths

Film Thickness



H(u) curves for different film thicknesses

Retention Factor

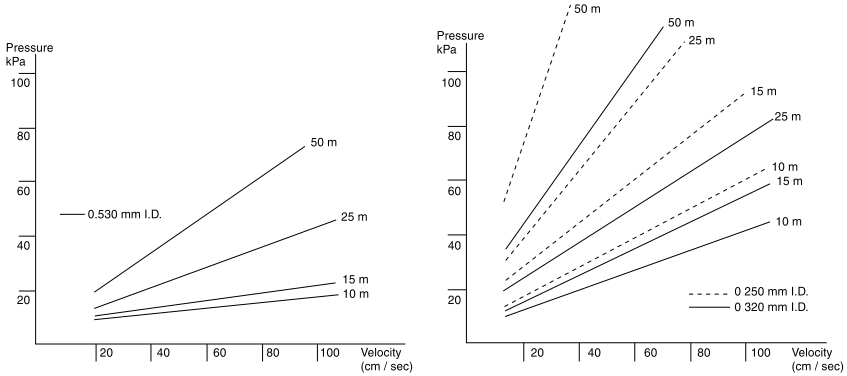


H(u) curves for different k values

Optimal velocities for low d_f values:	u_{opt} (cm/sec)	Optimal velocities for high d_f values:	u_{opt} (cm/sec)
H_2:	40	H_2:	25
He:	25	He:	15
N_2:	10	N_2:	7

Figure 14: Inlet Pressure vs. Velocity

The velocity diagrams below show the relation between inlet pressure and velocity for different internal diameters and lengths, measured for hydrogen at 120 °C isothermal and splitless injection (approximate values).



Temperature Programming

There are no strict rules to determine the optimal oven temperature of your analysis. Suggestions can be found in literature data and in this guidebook. Experience and trial and error are usually the most valuable tools.

General considerations:

1. Isothermal Temperature: for separation of components with slightly differing boiling points.
2. Temperature programming (single or multistep): for separation of samples with wide boiling point range.

Checking for Leaks

Use a thermoconductivity detector to check for leaks. It is highly sensitive to H_2, He and it won't contaminate the instrument or column. Liquid leak indicators are not recommended for capillary columns because there is always the risk of drawing the liquid into the column or column fittings and contaminating the system.

Note: If graphitized Vespel ferrules are being used, leakage can occur after the initial heating phase due to ferrule shrinkage and/or deformation. Be sure that the fitting is re-tightened after this initial heating phase then carefully checked for leaks. Better yet, also pre-condition vespel-containing ferrules in an oven at 250 °C for at least 4 hours prior to use.

Injection Techniques

Table 6: Injection Modes and Selected Specification

Parameter	Split	Splitless	Direct	On-Column
Column ID (mm)	0.10-0.53	0.18-0.53	0.53	0.18-0.53*
Injection Temp (°C)	High 250-300	Moderate 200-250	Moderate 200-250	Low (15 °C below bp of solvent)
Vent or Purge	Continuous	After initial time	None	None
Typical Sample Size (µL)	1-2	2-4	2-4	1-4
Concentration Range (ppm)	10-1000	0.01-100	0.1-100	0.01-100

** Special needles required*

Table 7: Gases used with Common Detectors

Detector	Carrier Gas	Fuel Gas	Make-up Gas
ECD	N_2, Ar / 5 %CH_4	None	N_2, Ar / 5 % CH_4
ECD	H_2	None	Ar / 5 % CH_4
ELCD, Hall	He, H_2	H_2	None
FID	He, H_2, N_2	Air + H_2	N_2, He, H_2
FPD	N_2, He	Air + H_2	Same as carrier
HID	He	None	He
NPD	He, N_2, H_2,	Air + H_2	He
PID	He, H_2, N_2	None	N_2, He
TCD	He, H_2	None	Same as carrier

Gas Flow Settings

Table 8: Typical Headpressures (for Helium)

Column Length (m)	ID (mm)	Column Head Pressure (psig)
15	0.25	6
30	0.25	12
60	0.25	24

Calculating Split Ratio and Column Flow Rate:

Split Ratio = split vent flow (mL/min) + column flow (mL/min) / column flow (mL/min)

Flow (mL/min) = (π) column radius (cm)^2 column length (cm) / dead volume time (min)

Average Linear Velocity $\bar{u}$ (cm/sec) = column length (cm) / retention time of non-retained peak (sec)

Table 9: Expansion Volumes of Common Solvents

Injection Volume (µL)	H_2O (µL)	CS_2 (µL)	CH_2Cl_2 (µL)	Hexane (µL)
0.1	142	42	40	20
0.5	710	212	200	98
1.0	1420	423	401	195
2.0	2840	846	802	390
3.0	4260	1270	1200	585
4.0	5680	1690	1600	780
5.0	7100	2120	2000	975

Sample and solvent expansion volume = nRT / P

Where:
- **n** = number of moles of solvent and sample, calculated as: volume (mL) x density (g/mL) / MW (g/mole)
- **T** = absolute temperature of the injector (°K)
- **P** = column head pressure (atm) + 1 atm
- **R** = Universal gas constant

Volume of inlet sleeve = $\pi r^2 L$

Where:
- **r** = radius (cm)
- **L** = length (cm)

Table 10: Splitless Hold Times

Column ID (mm)	Hold Time (min)	Column Flow Rate (mL/min)		Sample Transfer Time (min)	
		He	H_2	He	H_2
0.25	1.0	0.7	1.4	1.2	0.6
0.32	0.75	1.2	2.4	0.7	0.4
0.53	0.50	2.6	5.2	0.3	0.2

Column Dead Times and Markers

Table 11: Recommended Dead Volume Time Markers

Detector Type	Marker Compound
ECD	Methylene chloride[2,3], Dichlorodifluoromethane
FID	Methane, Butane[1]
NPD	Acetonitrile[2,4]
PID ELCD	Vinyl chloride
TCD, MS	Methane, Butane[1], air

1. From a disposable lighter
2. Place 1-2 drops in an autosampler vial and tightly cap. Shake and inject 1-2 µL from the headspace of the vial. Do not inject any liquid.
3. Use a column temperature above 55 ºC.
4. Use a column temperature above 95 ºC.

Methane with FID / TCD.
Calculate average linear velocity by injecting 25-100 µL of 1 % methane in N_2 gas blend. Measure the retention time of the methane peak and calculate the following:

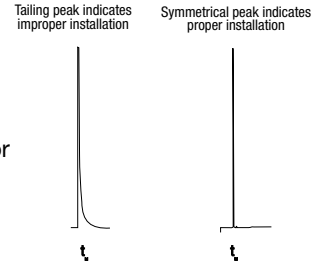
Average Linear Velocity ($\bar{u}$) = L/t_o

Table 12: Recommended Methane Retention Times

Length (m)	H_2 (sec)	He (sec)	N_2 (sec)
15	38	75	150
30	75	150	300
60	150	300	600

Examine the shape of the **Dead Volume Peak or Solvent Peak**. A correct column installation will yield a sharp narrow symmetrical peak. Any tailing or distortion in peak shape indicates a faulty installation.

Figure 15: Dead Volume Peak Shape Test



If the peak is broad and / or tailing, check the following potential problem areas:

- Improper column positioning / insertion into inlet or detector
- Gross contamination of the splitter sleeve
- Chipped or cracked splitter sleever
- Improper sweeping of column end by makeup gas
- Damaged or crushed column end
- Obstructed column

Sample Capacity

Table 13: Typical Sample Capacity (max for single component)

ID (mm)	d_f (µm)	Capacity (ng)
0.18	0.18	20 - 75
0.25	0.25	50 - 125
0.32	0.50	100 - 250
0.53	1.0	500 - 1,000

Note: Capacity represents maximum loading per component. Sample capacity increases with film thickness.

Solvent Rinsing of Zebron Capillary Columns

Select the rinse solvent from the Table below.

Table 14: Phase Compatibility with Rinsing Solvents

Phase	Water	Methanol	CH_2Cl_2	$CHCl_3$	Acetone	Hexane
ZB-1	OK	OK	OK	OK	OK	OK
ZB-1ms	OK	OK	OK	OK	OK	OK
ZB-1HT$_{Inferno}$	OK	OK	OK	OK	OK	OK
ZB-5	OK	OK	OK	OK	OK	OK
ZB-5ms	OK	OK	OK	OK	OK	OK
ZB-5HT$_{Inferno}$	OK	OK	OK	OK	OK	OK
ZB-624	OK	OK	OK	OK	OK	OK
ZB-35	OK	OK	OK	OK	OK	OK
ZB-1701	OK	OK	OK	OK	OK	OK
ZB-1701P	OK	OK	OK	OK	OK	OK
ZB-50	OK	OK	OK	OK	OK	OK
ZB-WAX*plus*	AVOID	OK	OK	OK	OK	OK
ZB-WAX	AVOID	OK	OK	OK	OK	OK
ZB-FFAP	AVOID	OK	OK	OK	OK	OK
MR-1	OK	OK	OK	OK	OK	OK
MR-2	OK	OK	OK	OK	OK	OK

Table 15: Rinsing Conditions

Column ID (mm)	Rinse Solvent Volume (mL)	Pressure (psig)
0.25	5	40
0.32	5	40
0.53	10	20

Important: Rinse from the back to the front of the column to avoid pushing inlet contaminants further into the column.

Chemical Compatibility

Important! Water and organic solvents such as those listed in the table above will not damage Zebron column stationary phases. However, inorganic acids and bases should be completely avoided or rapid degradation and permanent damage to the stationary phase will result. In the event chemical damage is incurred, the removal of 0.5-2 m of capillary off the front end will often restore column performance.

"Baking Out" the Column

Column contamination and degradation in analytical performance can occur if the highest boilers are not eluted with every run. The final oven temperature needs to be set high enough to ensure elution of these compounds, but not so high as to cause thermal damage. This can be done either isothermally, or more commonly, via a gradient or ballistic increase until the last components elute from the column.

NEVER exceed the upper temperature limits of the column. Severe degradation and loss of stationary phase, as well as permanent damage to the tubing surface, may result. Chromatographically this may manifest itself in excessive column bleed, peak tailing, decreased resolution, shortened run times, reduced column lifetimes or even column failure. To prevent accidental overheating and thermal damage to the column, set the oven's maximum temperature at or slightly below the column's upper temperature limit.

Extracted samples often cause a buildup of contaminants that require a more concerted "baking out", although this technique should be used carefully and sparingly. ***DO NOT exceed more than 15 minutes at the upper isothermal temperature limit specified for the column.*** Baking out a contaminated column may cause some sample residues to be converted to insoluble materials that can no longer be removed, even by solvent rinsing. The column may be irreversibly damaged as a result. The best way to guard against column contamination and degraded analytical performance is to introduce only samples that have first been thoroughly and carefully extracted and filtered. Before baking out to remove contaminants, try solvent rinsing the column first.

Column Storage

Important! The column may be left in the instrument for short-term storage. Ensure a flow of carrier gas through the column at 100-200 °C. For long-term storage, disconnect from the GC and cap or seal the capillary ends. Oxygen and moisture can degrade or irreversibly damage the column, especially cyanopropyl-based phases. Wax (polyethylene glycol) and cyanopropyl-based phases are also susceptible to UV-induced degradation and should be shielded from light (fluorescent or sunlight). Store the column in the original box. Upon reinstallation, cut column ends to ensure that septum fragments or other debris have not been left in the column.

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☑ **Top Recommendations**

Glossary

Adsorption mode: Chromatography in which the stationary phase is a solid. Separation occurs through mechanisms of adsorption and resorption.

Band broadening: Extra column band broadening is due to a loss in efficiency through the chromatographic system. All parts of a chromatographic system can cause band broadening.

Baseline: Detection of the mobile phase (i.e. carrier gas).

Baseline: Any signal not resulting form analytes.

Baseline noise: Low level, high frequency signals superimposed on the baseline signal, due to column bleed, impurities in injector and detector, etc.

Bleed: Decomposition and vaporization of the stationary phase. Most apparent when using the column near its maximal allowable operating temperature.

Capacity: See Sample capacity.

Capacity factor: A number which indicates the relative time a solute spends in the liquid phase with regard to the time it spends in the mobile phase.

Capillary columns: Narrow (from 0.050 to 0.530 mm internal diameter), long (from 10 to 100 m) chromatographic columns, offering high efficiencies.

CGC: Capillary gas chromatography

Chemical bonded: Term which indicates a chemical reaction of the stationary phase with the capillary wall, rendering the column solvent-resistant. See “Immobilized”.

Christmas tree effect: A Christmas tree-like shape of a solute peak due to uneven temperature distribution in the GC system.

Coating efficiency (CE): Expresses the percentage of the measured efficiency over the efficiency theoretically possible.

Coelution: The simultaneous elution of two components.

Crosslinked: Interlinking of the stationary phase to obtain a stable and solvent-resistant film. See “Immobilized”.

Dead volume: The volume of a capillary column. The retention time of an unretained solute (t_M) is used to convert carrier gas flow into carrier gas velocity.

Distribution constant (K_D): K_D describes the equilibrium between the concentration of a solute in the stationary phase and in the mobile phase.

ECD: Electron Capture Detection.

Effective plate number: The number of effective plates.

Efficiency: A term indicating the ability of the column to elute a component in a narrow chromatographic peak. Column efficiency is described by ‘N’ and ‘H’; see equations.

Elution: The transfer of a component through the column.

FID: Flame Ionization Detection.

Film thickness: The thickness of the stationary phase coated onto the column wall.

FSOT: Fused Silica Open Tubular.

FTIR: Fourier Transform Infra-Red spectroscopic detection.

Fused silica: Synthetic polymer of silicon dioxide. Due to its high purity, fused silica has replaced glass as material for capillary columns.

Golay equation: Chromatographic equation, describing the column efficiency in capillary gas chromatography.

Height equivalent to a theoretical plate (H): The length of the column occupied by one theoretical plate. The smaller H, the higher the plate number for a given length. See equation.

Immobilized: The immobilization of the phase due to cross-linking and/or chemical bonding.

Inertness: The absence of active groups in the column which would react with polar solutes, resulting in tailing peaks.

Liquid phase: The stationery phase, coated onto a column which is in a liquid state at operating temperatures.

MAOT: Maximal Allowable Operating Temperature of the column.

McReynolds constants: A classifying system for the polarity and selectivity of stationary phases.

Mobile phase: The medium used to carry the solutes through the column. In capillary GC three gases are applied as mobile phase: hydrogen, helium and nitrogen.

Non-polar: The absence of polar groups in the stationary phase.

On column injection: Injection technique in capillary gas chromatography. The sample is injected directly into the column.

Glossary

Overloading: Exceeding the sample capacity for a solute on a given column, resulting in asymmetrical peak shapes and variable retention times.

Phase ratio: Number which indicates the ratio between the mobile phase (column radius) and the stationary phase (film thickness).

PLOT: Porous Layer Open Tubular: An adsorbent such as aluminium oxide is deposited on the inner column wall, increasing the contact surface.

Polar: The presence of polar groups in the stationary phase.

Polarity: Stationary phases are often classified according to their degree of polarity, indicating the ways of interaction in a chromatographic separation.

Polyimide: The outer polymer coating of fused silica tubing giving it strength and flexibility.

Resolution: A measure of the ability of a column to separate two components. See Equation.

Retention: Interaction between stationary phase and solute will retain the solute in the column. The degree of the interactions will determine the strength of the retention.

Retention time: The time needed for a solute to elute from the column, starting from the injection. Total retention time is the sum of the time the solute spends in the mobile phase and the time it spends in the liquid phase (t_o = adjusted retention time).

Sample capacity: Maximum sample size of a compound which can be introduced onto a capillary column coated with a particular stationary phase without disturbing the chromatographic performance of the column. See equation.

SCOT: Support Coated Open Tubular columns: Capillary columns where the liquid stationary phase is coated onto a fine solid support.

Selectivity: The degree by which the stationary phase differentiates solutes. Separation in CGC is based on different selectivities for components present in a sample.

Selectivity factor: A number describing the selectivity of the stationary phase for two chemicals, relative to each other. A selectivity factor greater than one is required to achieve separation. See equation.

Solid State Injection: Injection technique in CGC where the solvent is vaporized prior to injection into the column.

Solutes: The components present in a sample.

Split-splitless injection: Injection technique in CGC where the sample is injected into a heated chamber, vaporized and then carried by the mobile phase into the column. After vaporization, the sample can be partially vented (split mode) or not (splitless mode).

Stabilized: An uncrosslinkable phase can be dissolved from the column when rinsed with a solvent. To avoid breakdown of the film, a stabilizing agent is added.

Stationary phase: Polymer which is coated onto the inner column wall to provide the selectivity for separation.

Tailing: The tail-like shape of a chromatographic peak, caused by active sites in the column.

TCD: Thermal Conductivity Detection.

Theoretical plate: The representation of a unit of efficiency of a column.

TPGC: Temperature Programmed Gas Chromatography.

Trennzahl: The resolution between two consecutive members of the e-paraffin homologous series. See equation.

van Deemter equation: The basic equation describing the rate of band broadening of a column in gas chromatography. This equation was adapted to capillary GC by Golay (see “Golay equation”).

WCOT: Wall Coated Open Tubular columns: Capillary columns where the stationary phase is coated onto the column wall as a smooth uniform film.

Equations

1. Resolution (R)

- **Graphically:**

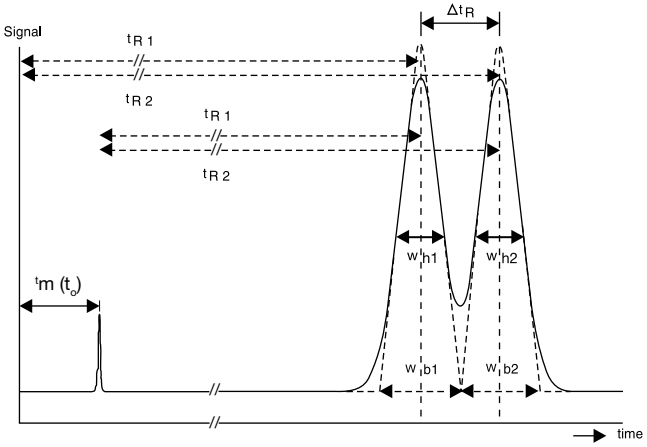
$$R = \frac{2\Delta t_R}{W_{b1}+W_{b2}}$$

- **Chromatographically:**

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

- **Trennzahl (TZ):**

$$TZ = \frac{\Delta t_R}{W_{h1}+W_{h2}} - 1$$



2. Efficiency (N and H)

- **N = number of theoretical plates**

$$N = 5.54\left(\frac{t_R}{W_h}\right)^2 = 16\left(\frac{t_R}{W_b}\right)^2 \qquad N = \frac{L}{H}$$

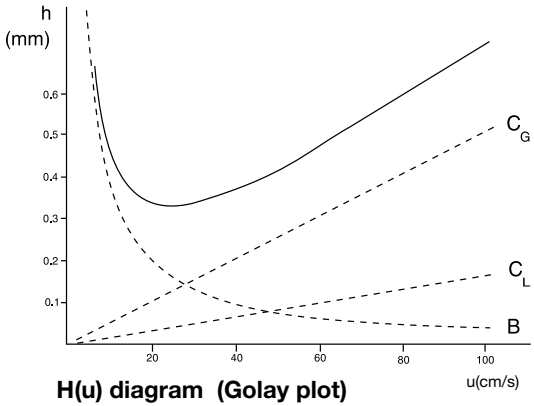
- **H = height equivalent to a theoretical plate**

Golay equation:

$$H = B/u + C_G u + D_L u$$

$$\bar{u} = L / t_O$$

$$H = \frac{2D_G}{u} + \frac{(1 + 6k + 11K)^2 r^2}{24 (1 + k)^2 D_G} u + \frac{2k d_f^2}{3 (1 + k)^2 D_L} u$$



H(u) diagram (Golay plot)

Equations (continued)

3. Effective plate number

$$N_{eff} = 16 \left(\frac{t_R'}{W_b} \right)^2$$

4. Coating efficiency

$$CE\% = \left(\frac{N_{exp}}{N_{theor}} \right) 100$$

5. Selectivity factor (α)

$$\alpha = \frac{t_{R\,2}'}{t_{R\,1}'} \qquad t_R' = t_R - t_O \qquad t_{R2} > t_{R1}$$

6. Capacity factor (Retention Factor)

$$k = \frac{t_R - t_O}{t_O} = \frac{t_R'}{t_O}$$

7. Distribution constant

$$K_D = k \bullet \beta$$

8. Phase ratio

$$\beta = \frac{r}{2d_f}$$

9. Sample capacity
for a two component mixture

$$SC \sim r^2 (1 + k) \sqrt{LH}$$

$$\sim r^2 \left(1 + \frac{2d_f K_d}{r}\right) \sqrt{LH}$$

10. Minimal analysis time

$$t_{Ne} = 16 \left(\frac{\alpha}{(\alpha - 1)} \right)^2 \left(\frac{(1 + k)^3}{k^2} \right) \left(R^2 \frac{H}{\bar{u}} \right)$$

11. Split Ratio = split vent flow + column flow / column flow (mL/min)

12. Flow (mL/min) = (π)(column radius (cm)^2 (column length (cm) / dead volume time (min)

13. Average Linear Velocity $\bar{u}$ (cm/sec) = column length (cm) / retention time of non-retained peak (sec)

14. Sample and solvent expansion volume = nRT/P

Where: **n** = number of moles of solvent and sample, calculated as:
volume(mL) x density (g/mL) / MW (g/mole)
T = absolute temperature of the injector (°K)
P = column head pressure (atm) + 1 atm
R = Universal gas constant

15. Volume of inlet sleeve = $\pi r^2 L$

Where: **r** = radius (cm)
L = length (cm)

16. Average Linear Velocity ($\bar{u}$) = L/t_o

Equations (continued)

List of symbols and abbreviations

α = Selectivity factor

B = Longitudinal diffusion term

β = Phase ratio

CE = Coating efficiency

C_G = Resistance to mass transfer in the gas phase

C_L = Resistance to mass transfer in the liquid phase

d_f = Film thickness

D_G = Diffusion coefficient in the gas phase

D_L = Diffusion coefficient in the liquid phase

H = Height equivalent to a theoretical plate

k = Capacity factor

K_D = Distribution constant

L = Column length

$\bar{u}$ = Average linear gas velocity

u = Linear gas velocity

N = Number of theoretical plates

$\acute{N}$ = Effective plate number

R = Resolution

r = Column radius

SC = Sample capacity

t_O = Retention time of unretained solute

t_R = Retention time of the solute

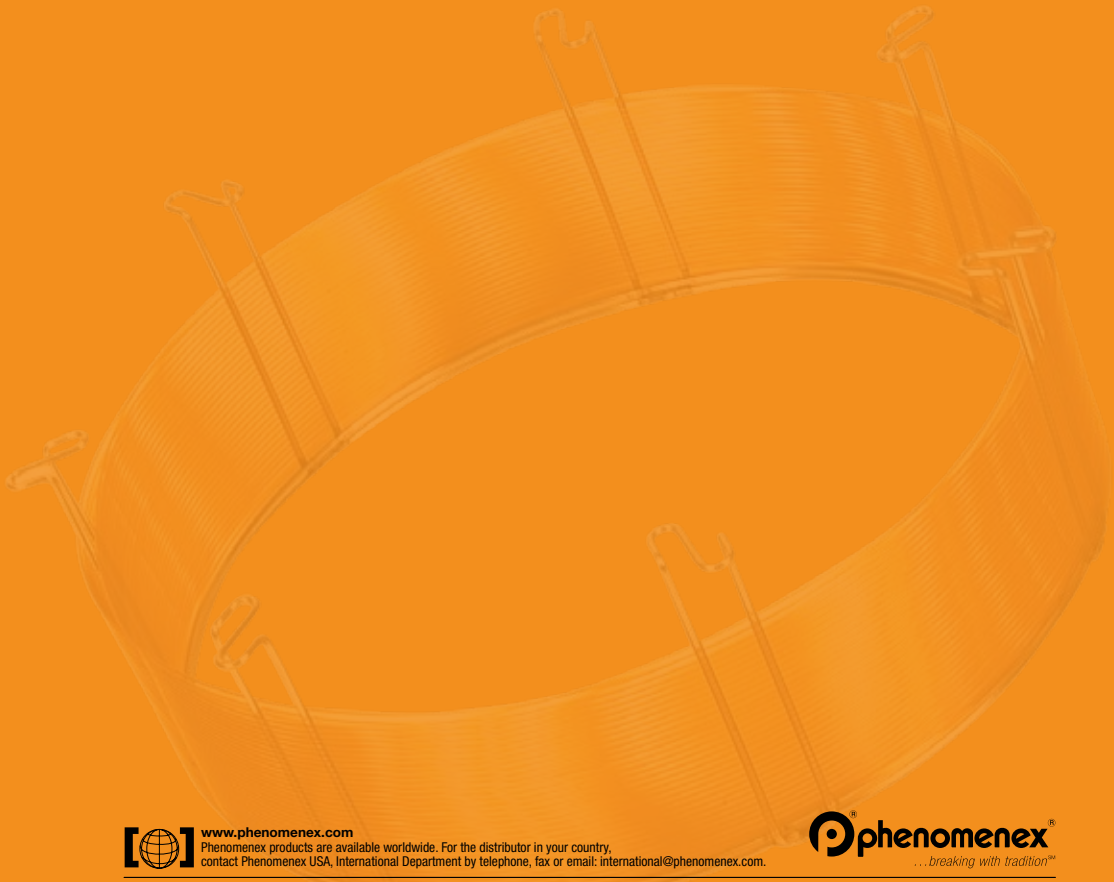
t_R' = Adjusted retention time of the solute

w_b = Peak width at base

w_h = Peak width at half height

NOTE:

While every attempt has been made to ensure
the accuracy of the information
contained in this guide,
Phenomenex assumes no responsibility for its use.
We welcome any additions or corrections
for incorporation into future editions.



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