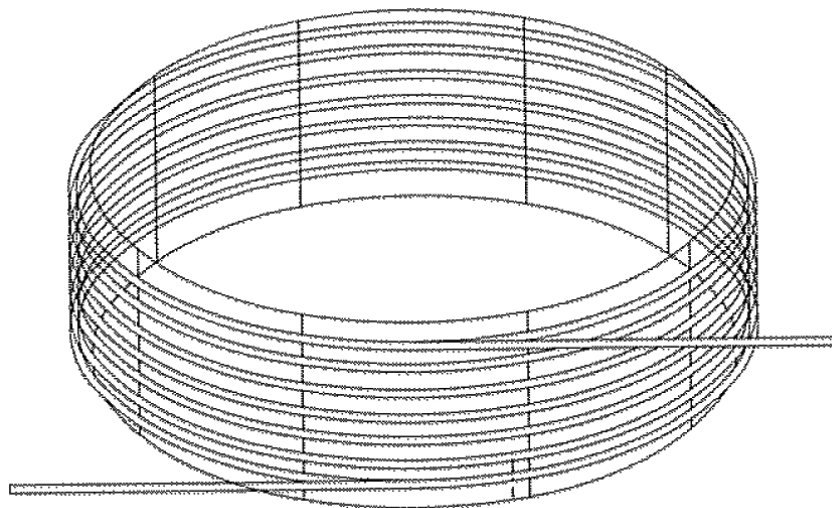


Astec

— **CHIRALDEX**[™] —

GC Columns

A Guide to Using Cyclodextrin Bonded Phases for
Chiral Separations by Capillary Gas Chromatography



CHIRALDEX™ GC Columns

Derivatized Cyclodextrin Phases for Capillary Gas Chromatography

Introduction

Cyclodextrins have been widely applied in the separation sciences since the early 1980's. The evolution of cyclodextrins and cyclodextrin derivatives for the separation of enantiomers by gas and liquid chromatography has been intense over the last few years. In 1983, Astec introduced the first cyclodextrin based column for the separation of enantiomers with a covalently bonded cyclodextrin for reversed phase HPLC tradenamed CYCLOBOND. As a result of a greater understanding of the chiral recognition mechanism, an ever increasing number of chiral analytes can be separated by both GC and LC.

In 1991, Astec received two global patents for the use of two specific cyclodextrin derivatives for the separation of enantiomers by capillary GC. This technology is tradenamed CHIRALDEX. The ability to resolve small non-aromatic chiral compounds has been a major growth factor for capillary GC especially to those involved in asymmetric synthesis. Two publications investigating the purity of 192 chiral catalysts, auxiliaries and synthons utilized 5 CHIRALDEX phases described in this Handbook. These five phases resolved the enantiomers of over 60% of these essential building blocks. The CHIRALDEX G-TA alone separated over 40% with the CHIRALDEX B-DM covering the next largest group. For more details of this important work for synthetic chemists, see publications 31 and 33 in the bibliography. Increased thermal stability, high resolution and large peak capacity of current gas capillary columns makes these tools ideal for the analysis of complex mixtures commonly encountered in samples originating from biological sources. A significant number of plasma samples have been analyzed for chiral drugs as well as their metabolites using CHIRALDEX phases.

With the technological advances in chiral stationary phases, there are often multiple technique choices for a particular analyte, and hence, multiple opportunities for enantioseparation. This Handbook will center on understanding structural relationships and the underlying mechanistic theories as well as supplying a large number of actual chiral separations to assist in making the best column choice. We will also highlight CHIRALDEX (three column) Kit choices to give the broadest possible opportunities for successful separations with the most economical investment.

The three cyclodextrins currently commercially available are α , β and γ -cyclodextrin. These differ in the number of glucose units (6, 7 and 8 respectively) in the structure and thus the diameter of the cyclodextrin cavity and the available surface. Many of these derivatives function without inclusion complexation and, therefore, the gamma-cyclodextrin is preferred for both small and large analytes.

For a more detailed description of the cyclodextrins and the inclusion mechanism, see the CYCLOBOND HANDBOOK.

General Characteristics

Eight phase types will be covered in this manual detailing their unique selectivity and application areas. They will be referred to as:

TA*	Trifluoroacetyl (2,6-di-O-pentyl-3-trifluoroacetyl)
DM	Dimethyl (2,3-di-O-methyl-6-t-butyl silyl)
DP	Dipropionyl (2,3-di-O-propionyl-6-t-butyl silyl)
DA	Dialkyl (2,6-di-O-pentyl-3-methoxy)
PN	Propionyl (2,6-di-O-pentyl-3-propionyl)
BP	Butyryl (2,6-di-O-pentyl-3-butyryl)
PH*	S-Hydroxypropyl (S)-2-hydroxy propyl methy ether)
PM	Permethyl (2,3,6-tri-O-methyl)

Using the prefix A, B and G will describe the cyclodextrins, alpha, beta and gamma, respectively, i.e., CHIRALDEX G-TA will refer to the Gamma Trifluoroacetylated phase. A CHIRALDEX B-PM is the Beta Permethylated phase.

Specifications

Fused Silica Capillary Tubing	250 μ m \pm 12 x 350 \pm 15
Lengths	10,20,30,40,50m \pm 1%
Film Thickness	0.125 μ m \pm 10%
Operating T°C	See MAOT chart page 4
Split Ratio	>100/1
Carrier Gases	Nitrogen Helium Hydrogen

LC vs. GC

Presently, there is considerable interest in enantiomerically resolving diverse classes of compounds. Many analytes can be separated only by GC or only by HPLC, but there exists a considerable overlap in which compounds can be analyzed by both techniques, and this overlap in applicability continually grows larger as technology improves. Table 1 generalizes analyte structural key points which may indicate a favorable separation potential for GC, HPLC or both techniques.

Table 1. Structural key points which would indicate separation technique

GC	Analyte Property	HPLC
√	Non-aromatic	
√	Analytes with BP < 260°C	
√	Derivatizable Group (OH, NH ₂ , COOH)	√
√	Halogen, alcohol, amine off chiral center	√
√	Halogen off of aromatic ring which is α or β from chiral center	√
	Thermally Labile	√
	Racemize with temperature	√
	BP > 260°C	√

Key points GC

Enantioselectivity is rare at temperatures π 200°C.

The lower the elution temperature, the greater the opportunity for enantioselectivity. Achiral derivatization can be used to lower the required elution temperature (see page 17).

If an analyte to be enantioresolved has a boiling point under 260°C (elution temperatures are related to analyte vapor pressure, not boiling point) or can be derivatized to such a point, it is a candidate for separation by GC. Although enantioselectivity can occur at elution temperatures greater than 200°C, it is more the exception. In general, the lower the temperature at which the compound can be eluted, the greater the opportunity for chiral separation. Temperature plays a significant role in selectivity and resolution for both GC and HPLC.

Key points LC

Aromatic functionality.

Multiple polar groups suggest the use of the polar organic mode (POM).

π - π interactions suggest the use of the normal phase mode (NP).

Includable group near the stereogenic center suggests the use of the reversed phase mode (RP).

HPLC is the separation technique of choice if the analyte has a high boiling point, decomposes or racemizes at elevated temperatures. To take advantage of the solute/stationary phase inclusion phenomenon in the RP mode, the analyte must contain a non-polar segment (i.e., aromatic). If this aromatic portion of the molecule is close to the stereogenic center (α or β for a native cyclodextrin or greater for derivatized cyclodextrins), an enantioselective interaction can occur, otherwise the interaction will be purely retentive. The aromatic portion of the analyte will more strongly include into the cyclodextrin cavity if it has a halogen ($I > Br > Cl > F$), nitro, phospho or sulfo substitution in the following positions ($p > o > m$). As a general rule, it is necessary for a solute to have an aromatic ring if it is to be easily and routinely resolved on a HPLC CSP. The main cyclodextrin based stationary phase which does not follow this rule is the CYCLOBOND I 2000 RSP (hydroxypropyl derivatized). This phase does resolve a number of analytes, i.e. t-boc amino acids (1) that do not contain an aromatic ring. Based upon this work, it has been noted that other non-aromatic molecules can be separated using a small amount of MTBE in the mobile phase (2). Interestingly, the analogous hydroxypropyl derivatized GC stationary phase (PH) also shows a high degree of selectivity towards aliphatic analytes (3).

Table 2 compares some mechanistic relationships between GC and HPLC. As noted earlier, depending upon the composition of the mobile phase (RP, NP or POM), the enantioselective mechanisms centers around either inclusion complexation (RP) or external surface interaction (NP and POM). The same

phenomenon occurs in GC (4) although driven more by the type of cyclodextrin derivative. Many derivatized cyclodextrin GC stationary phases are classified under inclusion dominant selectivity mechanisms, but surface interactions can play a role in selectivity as with TA, DP, PN and BP series. Presently, each analyte has to be investigated for the chiral recognition mechanism. This can be accomplished using molecular modeling, NMR, GC and MS.

Table 2. Comparison of mechanistic relationships between GC and HPLC

Mechanisms	GC	HPLC
Inclusion Dominant ¹ H-Bonding β -CD most selective	DA/DM/PH/PM ✓	RP ² ✓ ✓
Surface Interaction ¹ H-Bonding π - π Interactions γ -CD most selective	BP/PN/TA/DP ✓	POM/NP POM ³ NP ⁴
1	Inclusion dominated chiral separations generally have lower analyte capacity due to fewer interaction sites as compared to a surface interaction dominated mechanism. Size selectivity is generally more important in inclusion dominated separations.	
2	Reversed phase HPLC separations utilize an aqueous/organic mobile phase. In this mode, organic molecules include in the cyclodextrin cavity.	
3	The polar organic mode is composed mainly of acetonitrile, with minimal amounts of methanol, glacial acetic acid and triethylamine to control retention and selectivity. Acetonitrile would compete with the analyte to include into cavity. Interaction with CD hydroxyl groups or an attached functional group is the dominant chiral resolving mechanism.	
4	The normal phase (Hex/IPA), in combination with naphthylethyl carbamate derivatized CD (π -acid) or π -base/ π - π interactions. High organic mobile phase composition would compete with and overwhelm the analyte for including into the cavity.	

Mechanism and Analyte Capacity

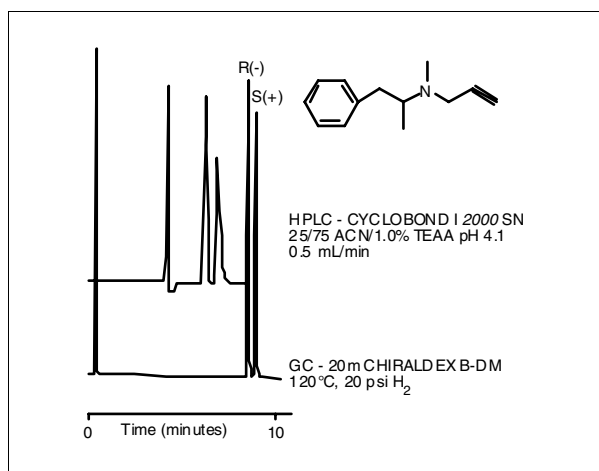
Thermodynamic relationships and sample loading studies can be used to indicate the dominant chiral recognition mechanism (4). Enantiomers that have large $-H^\circ$ and large S° values (> 1.8 kcal/mol) also show relatively low sample loading capacity (sample capacity is determined by injecting progressively more sample on the column and monitoring efficiency). This suggests that inclusion plays a role in the enantioselective mechanism. Enantiomers that have smaller $-H^\circ$ and

S° values show relatively higher sample loading capacity. This implies that inclusion plays a less dominant or negligible role in the enantioselective mechanism, and some sort of external association between the analyte and cyclodextrin has a greater influence on chiral selectivity. This is an idealized situation, and many mechanistic variations probably exist.

The formation of an inclusion complex is relatively slow (slow mass transfer, hence lower efficiency) as compared to an external surface interaction. Thus, stationary phases which have an external surface interaction as the dominant chiral recognition mechanism (BP, PN, DP and TA) should provide better chiral efficiency than inclusion dominant stationary phases (DA, DM, PH and PM). Another point of interest is the cyclodextrin (α , β or γ) which shows the overall best applicability. For inclusion type separations (GC and HPLC), the beta form is the most selective, while for the external adsorption dominant stationary phases, the gamma form has the greatest applicability (GC only). The inclusion mechanism has excellent selectivity for structural isomeric differences.

Figure 1 shows the separation of Deprenyl. Deprenyl is an Anti-parkinson drug in which the R(-) form is active. Both GC and HPLC methods are dilute and inject with retention times under 10 minutes.

Figure 1. GC and HPLC enantiomeric separation of Selegiline (Deprenyl)



Choosing the Right Column

Certain phases are more selective for given molecular structures. The extensive list of resolved analytes will be useful in demonstrating the unique separation properties of each phase. While many compounds can be enantiomerically resolved on multiple CHIRALDEX phases, often an advantage can be found in selectivity, elution order and/or analysis speed from one phase to another.

The first consideration in choosing the right CHIRALDEX column is the elution temperature of the analyte. Enantioselectivity is rare at temperatures greater than 200°C. All CHIRALDEX columns are temperature rated which may be a limiting factor in the choice of the column. Table 3 gives the maximum allowable operating temperature (MAOT) both for isothermal and temperature programmed conditions.

Table 3. Maximum allowable operating temperatures

Phase	MAOT °C Isothermal	MAOT °C Programmed
BP	200	220
DA	200	220
DM	200	220
DP	200	220
PH	200	220
PM	200	220
PN	200	220
TA	180	180

The next consideration is the molecular structure. The three most effective GC chiral columns are G-TA, B-DM, B-DA with the G-TA separating the greatest number of enantiomers, often with high enantioselectivity, B-DM separating the widest variety of different structural types, and the B-DA best suited for larger multi-ring structures.

Eighty-five percent of analytes which exhibit enantioselectivity on cyclodextrin based chiral stationary phases will give enantioselectivity on one of three main CHIRALDEX phases: CHIRALDEX G-TA, CHIRALDEX B-DM and CHIRALDEX B-DA. The fact that three phases offer such a wide variety of selectivities and analyzable solutes, underlies the rationale behind the CHIRALDEX Kit. The CHIRALDEX Kit is special pricing on any three column types of the same length and inner diameter. The reason other CHIRALDEX derivatives exist is to resolve “the other 15%”, reverse enantiomeric elution order or reduce analysis time.

Following is a breakdown of the general classes of compounds separated on the phases which have the broadest separation capabilities.

G-TA (Gamma-cyclodextrin, Trifluoroacetyl)

G-TA has its highest selectivity for alcohols, diols and polyols as the free alcohol and as an acyl derivative; amines as acyl derivatives; amino alcohols, halogens (Cl>Br>F), amino acids, hydroxy acids, lactones, furans and pyrans. See pages 17-19 for chiral derivatization effects and procedures.

B-DM (Beta-cyclodextrin, Dimethyl)

B-DM is a general purpose column. The selectivity of this phase covers applications of both the B-PH and B-PM. From the tests run to date, the B-DM has shown superior performance over the B-PM and B-PH. It is the column of choice when elution temperature exceeds 200°C. This column is also very useful for a number of free acids and bases and is the only chiral GC phase that can do this type of polar separation.

B-DP (Beta-cyclodextrin, Dipropionyl)

This column demonstrates good selectivity for a wide range of analytes except alcohols and epoxides where the G-TA remains the best. The G-DP has shown very high selectivity (Rs 2-16) for both aromatic and aliphatic amines and for aliphatic and some aromatic esters (Rs 2-21). Both hydrolytic and temperature stability are better than G-TA and for bulky fused ring structures the G-DP is better than the B-DP.

B-DA (Beta-cyclodextrin, Dialkyl)

B-DA requires minimally two ring structures, one of which is unsaturated (aromatic) α, β to the stereogenic center (examples-Prozac, methylphenidate, chloropheniramine). Inclusion complexation or proper fit between the analyte and cyclodextrin cavity is the dominant enantioselectivity mechanism for the DA series of columns. There must be an includable group α or β to the stereogenic center for chiral recognition. The size of the includable group will dictate the choice of alpha, beta or gamma cyclodextrin. Since the CHIRALDEX DA series of columns most effectively separate multi-ring analytes, analysis temperatures are often higher than 150°C. Enantioselectivity has been observed at temperatures >200°C (Prozac acetyl derivative).

G-PN (Gamma-cyclodextrin, Propionyl)

G-PN has its highest selectivity for epoxides, aromatic amines (amphetamine/methamphetamine), >C6 alcohols and lactones.

G-BP (Gamma-cyclodextrin, Butyryl)

G-BP can be used as a general column but it is especially useful for amino acids and is, therefore, a good substitute for other bonded amino phases.

B-PH (Beta-cyclodextrin, Hydroxypropyl)

B-PH shows at least some selectivity to a great variety of analytes but is especially effective for saturated analytes with minimal functionality, saturated cyclics and

bicyclics. The CHIRALDEX PH series of columns shows less of a necessity for inclusion complexation for chiral recognition than the DA columns.

B-PM (Beta-cyclodextrin, Permethylated)

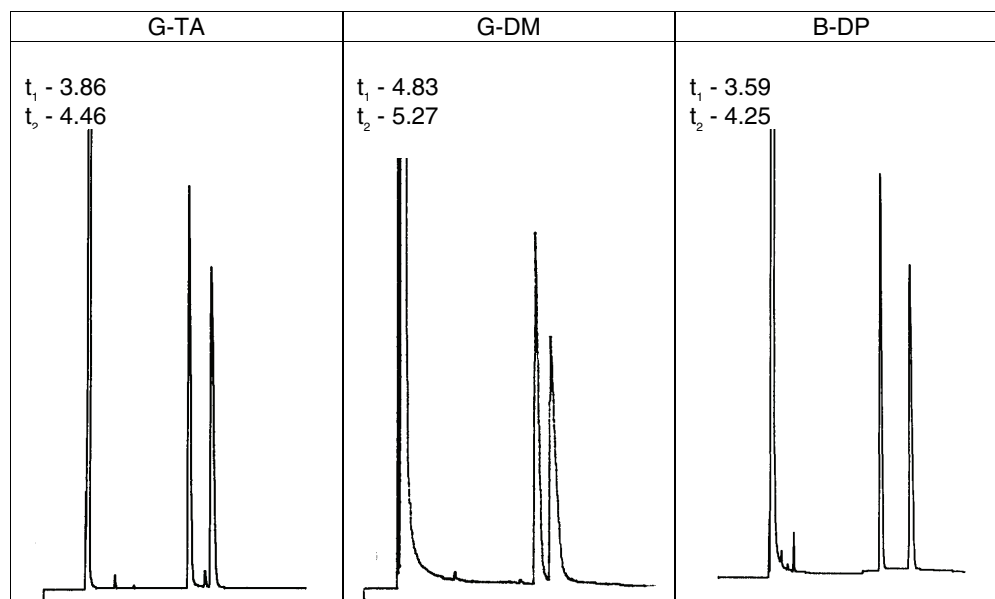
The B-PM can be used as a general purpose column for the separation of acids, alcohols, barbitals, diols, epoxides, esters, hydrocarbons, ketones, lactones and terpenes. Some alcohols and diols can be better separated underivatized on this phase as well as some analytes with polar groups, i.e. tertiary amines. Often greater selectivity and efficiency are achieved on other CHIRALDEX phases.

Note: The subtle differences in functional groups between G-TA, G-PN, G-BP and G-DP often allow for minor enhancements in chiral and achiral selectivity when changing from one derivative to another.

Comparison G-TA/B-DM/G-DM/B-DP/G-DP for Some Polar Racemates TA vs DM vs DP

Compound	R-	Column Phase	Column Length	T(°C)	k'	α	Rs	N _i	Elution Order
Amphetamine	-COCF ₃	G-DP	20m	140	9.2	1.092	4.28	55,000	D,L
	-COCF ₃	B-DM	30m	110	9.2	1.038	2.28	79,000	L,D
	-COCF ₃	G-TA	30m	110	14.1	1.023	1.18	62,000	
2-Aminoheptane	-COCF ₃	B-DP	30m	110	4.9	1.150	6.60	63,000	
	-COCF ₃	G-DM	30m	75	21.0	1.047	1.77	34,000	
	-COCF ₃	G-TA	30m	90	9.1	1.124	5.12	40,000	
β -Butyrolactone		B-DP	30m	140	2.0	1.275	5.98	28,000	
		G-DM	30m	70	4.2	1.113	1.55	8,900	
		G-TA	30m	100	2.2	1.224	3.95	16,000	
Mandelic Acid, Methyl Ester		B-DP	30m	140	5.0	1.088	3.74	63,000	R,S
		B-DM	30m	110	14.6	1.0			
		G-TA	30m	110	8.1	1.121	4.46	56,000	S,R

Chromatographic Comparison for β -Butyrolactone



Large increases in selectivity, efficiency and resolution were obtained on the G-DP at surprisingly lower retention times for a large number of volatile polar racemates.

CHIRALDEX Trifluoroacetyl Derivatives A-TA, B-TA, G-TA*

Trifluoroacetylation of the 3 position hydroxyl groups after pentylation of the 2,6 hydroxyl groups creates a phase with high selectivity for oxygen containing analytes in the form of alcohols, ketones, acids, aldehydes, lactones. This phase is also highly selective for halogenated compounds. For the first time, the gamma-cyclodextrin* form demonstrates the broadest selectivity.

Features

- Separates the widest variety of enantiomers
- Separates the greatest number of enantiomers
- Gamma more selective than beta-cyclodextrin
- Unique retention behavior
- Extraordinary versatility and chiral selectivity
- Useful for homologous series of:
 - Amino acids (primary, secondary, aromatic and aliphatic)
 - Amines (primary, secondary, cyclic, aromatic and halogenated)
 - Amino alcohols
 - Alkanes, hydrogenated alkanes

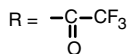
- Alcohols (aliphatic and aromatic)
- Acids (halogenated and hydroxy)
- Esters (aromatic, aliphatic, hydroxy, di-ester)
- Diols
- Lactones
- Ketones
- Phthalides
- Sulphoxides

Mechanism Observations

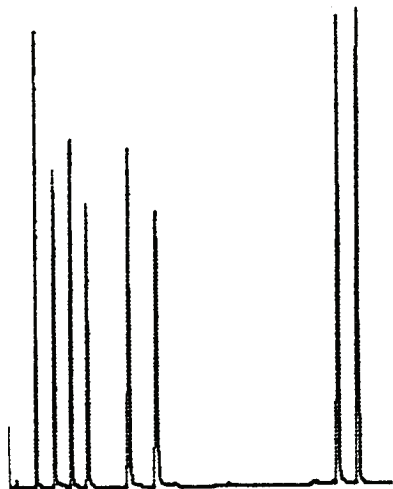
- Strong dipole-dipole interactions
- Longer alkyl chain; greater retention; increase in enantioselectivity up to C₄/C₅
- Halogens known to favor cavity interaction

Dipole-dipole interactions are commonly identified in the mechanism of separation for TA phases. In a homologous series of alkane enantiomers, identical alpha values are observed regardless of chain length or branching indicating only 1 or 2 carbons may be contributing to chiral recognition. Alpha values are greatly affected by size and polarity of the head group. Functional groups like epoxides, amino alcohols and alcohols can dictate the cyclodextrin selection. Aldehydes, carboxylic acids and epoxides separate better on the gamma while alcohols, alcohol amines and other linear molecules separate better on the beta derivative.

Enantiomeric Resolution of 1,2-Alkyl diols



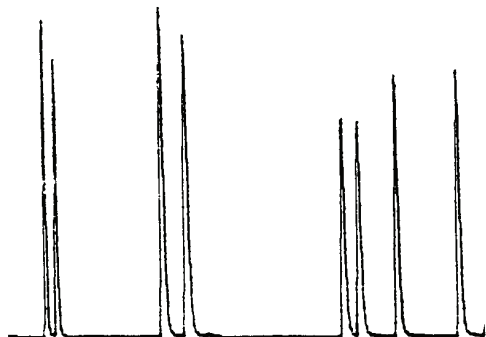
t₁ - 1.96, t₂ - 2.44 Propanediol
t₃ - 2.87, t₄ - 3.27 Pentanediol
t₅ - 4.33, t₆ - 5.03 Hexanediol
t₇ - 9.68, t₈ - 10.18 Octanediol



CHIRALDEX G-TA (40m)
80°C:5 min.
80-130°C @ 5°C/min.
Hydrogen @ 25 psi
Split Ratio - 100/1

Enantiomeric Resolution of 2-Halohydrocarbons

t₁ - 2.02, t₂ - 2.19 2-Chlorobutane
t₃ - 3.86, t₄ - 4.23 2-Bromobutane
t₅ - 6.78, t₆ - 6.92 2-Iodobutane
t₇ - 7.52, t₈ - 8.49 2-Bromopentane



CHIRALDEX G-TA (40m)
30°C:3 min.
30-70°C @ 5°C/min.
Hydrogen @ 25 psi
Split Ratio - 100/1

Separation of Propylene Oxide



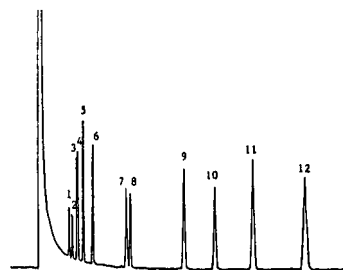
t₁ - 3.32
t₂ - 3.51



CHIRALDEX A-TA (20m)
25°C
Nitrogen @ 7 psi
Split Ratio - 50/1

Separation of D,L-Alkyl Amines

Peaks
1,2 - 2-Aminobutane
3,4 - 2-Amino-3,3-dimethylbutane
5,6 - 2-Aminopentane
7,8 - 3-Aminoheptane
9,10 - 2-Aminoheptane
11,12 - 2-Amino-6-Methylheptane



CHIRALDEX B-TA (10m)
100°C
Nitrogen @ 2 psi
Split Ratio - 100/1

Size Selectivity

The gamma TA derivative has proven to exhibit a wider chiral selectivity and usefulness than the beta analog. The influence of the inclusion mechanism for chiral recognition is very much reduced and capacities are generally higher indicating more surface interaction. Of all the compounds tested (see Appendix), the split between gamma TA and beta TA is approximately 55/35 with only 10% of the separations accomplished on the alpha TA.

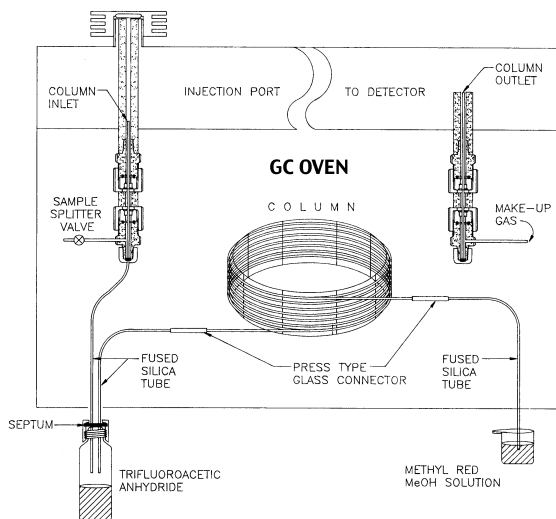
Important Notice About CHIRALDEX TA Columns

The TFA derivative of the cyclodextrins used in the CHIRALDEX TA series will hydrolyze in the presence of moisture at room temperature or above. Sources of moisture include injected samples, the carrier gas or air during unsealed storage.

To properly seal a CHIRALDEX TA column for storage, the column must be heated to 160°C under normal chromatographic conditions for one hour and flame sealed. If sealed under vacuum, it will then store indefinitely. To ensure long column life, be sure all samples are free of moisture before injecting, that the sample solvent, i.e. ethyl ether, methylene chloride, etc. is anhydrous and that the carrier gas has an efficient and functional moisture trap. Maximum operating temperature for this column is 180°C.

Regeneration Procedure for CHIRALDEX TA (Trifluoroacetyl) Columns

Figure 2. Connection for column regeneration



Procedure courtesy of Tokyo Kasei Kogyo Co., Ltd., Tokyo, Japan.

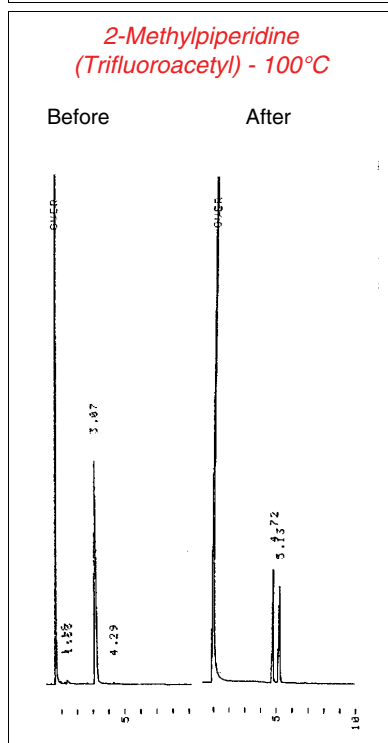
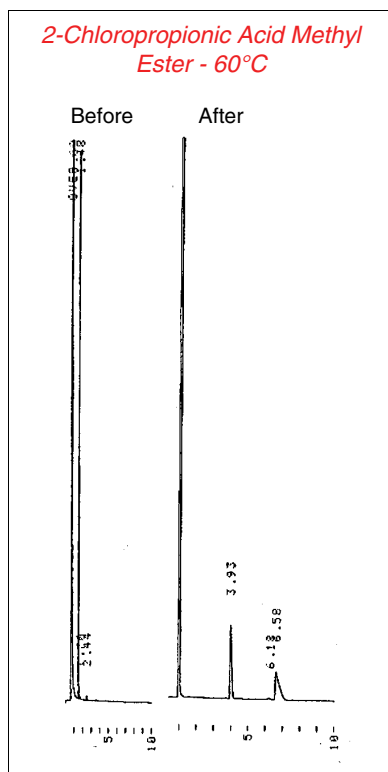
This procedure is effective for regenerating columns that have lost selectivity due to the hydrolysis of the trifluoroacetyl group on the stationary phase. If the column has lost its efficiency, the coating itself has been compromised and the regeneration procedure will be ineffective. It is a good idea to test the column with the test mixture provided with the column prior to regeneration to ensure that selectivity has been lost.

The following steps should be followed for regeneration:

1. Under normal carrier gas flow chromatographic conditions, heat the column to 150°C and remain at this temperature for 30-60 minutes. Cool to room temperature.
2. Following Figure 2, assemble the apparatus for the regeneration. Please note the column is inside the oven while the vial of trifluoroacetic anhydride (TFA) and the beaker of methyl red solution are outside the oven. Fused silica tubing is quite flexible and with care, can be drawn outside the GC oven and the door closed without capillary breakage.
3. Fused silica tubing (deactivated or non-deactivated) is connected from the injection port to the head space of a vial of TFA. Use a 2 meter length of capillary tubing. The vial for the TFA should have a septum type cap.
4. Another piece of fused silica tubing is placed from the head space of the vial of TFA and connected to the CHIRALDEX TA column. A variety of connectors can be used (glass type press fit, butt connectors or Teflon tubing).
5. Use another piece of fused silica capillary and another connector to link the CHIRALDEX TA column to a beaker of methyl red indicator. An alcoholic solution of methyl red will indicate when trifluoroacetic acid is exiting the system, turning from a yellowish color to red in the presence of the exiting acid fumes.
6. Close the oven door and introduce carrier gas into the system at the pressure of 1-2 psi per 10 meter of CHIRALDEX column. At this point, make sure there is flow through the system (bubbles are forming in the methyl red solution).
7. When the indicator solution starts to turn red (20-30 minutes after the carrier gas is introduced into the system), put the outlet end into base (0.05-1M NaOH) to neutralize the outgoing acid.
8. At this point, initiate a temperature program from 40°C to 150°C at 5°C/minute. Hold the 150°C for 30-60 minutes. Cool to room temperature.
9. Remove the capillary from the vial of TFA, and cap vial. Remove the capillary tubing from the injection port and replace it with the fused silica tubing connecting to the CHIRALDEX TA column.
10. Re-initiate the temperature program from 40°C to 150°C at 5°C/minute. Hold the 150°C for 30-60 minutes. Cool to room temperature.
11. The CHIRALDEX TA column can now be disconnected from the attached fused silica tubing. Cut 2-5cm of CHIRALDEX TA column from the end that was connected to the TFA vial.

12. The column can now be flame sealed for storage (see page 22) or connected to the GC for chromatographic separations.

Evaluation Test



CHIRALDEX Dimethyl Derivative B-DM*, G-DM

This particular dimethyl phase has been devised to overlap with the applications of both the PM and PH series of columns. To date, it has shown similar selectivities with shorter retention times and greater resolution. It also has exhibited thermal stability equivalent to the PM and is, therefore, the column of choice when elution temperature of the analyte exceeds 200°C. It is considered to be one of the essential columns in a kit purchase replacing the PH series and further extending the kit potential.

Features

- Broad chiral selectivity
- Combines selectivity of PM and PH
- Thermal limit to 200/220°C
- Short retention, high resolution
- Beta derivative broadly applicable
- Resolves aliphatic, olefinic and aromatic enantiomers

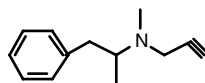
Mechanism Observations

- Size selectivity present but not dominant as in DA
- Fewer structural requirements
- Characteristic temperature selectivity

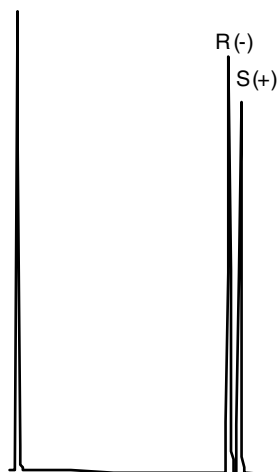
Size Selectivity

Size selectivity is evident, therefore, the inclusion mechanism is playing a role in the separation mechanism but does not dominate as it does in the DA series. The beta form as in the PH series covers a very broad range of molecular sizes and, therefore, has the greatest applicability.

Separation of Selegiline (Deprenyl)

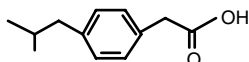


t₁ - 8.38 R(-)
t₂ - 8.77 S(+)

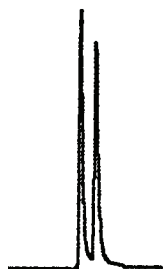


CHIRALDEX B-DM (20m)
120°C
Hydrogen @ 20 psi
Split Ratio - 100/1

Separation of Ibuprofen



t₁ - 11.30
t₂ - 12.31



CHIRALDEX B-DM (20m)
165°C
Helium @ 50 psi
Split Ratio - 100/1

CHIRALDEX Dipropionyl Derivatives B-DP*, G-DP

Features

- Broad chiral selectivity
- Good hydrolytic stability
- Thermal limit 200/220
- Excellent for aromatic and aliphatic amines
- Good for many aliphatic and some aromatic esters
- High efficiency and resolution at low retention times for polar racemates

Mechanism Observations

- Mostly surface interactions
- Fused ring structures better selectivity on gamma
- Acids have better selectivity as methyl rather than ethyl esters

Size Selectivity

Both speed and sample capacity indicate a surface type mechanism for very polar racemates. Large bulky molecules still require a larger surface area than beta provides, therefore, an increase in selectivity is seen on the gamma derivative for fused ring structures. The smaller alpha cavity offered no selectivity while the beta covered the largest range of molecular sizes but the choice between beta and gamma is compound dependent for this phase.

CHIRALDEX

Dialkyl Derivatives

A-DA, B-DA*, G-DA

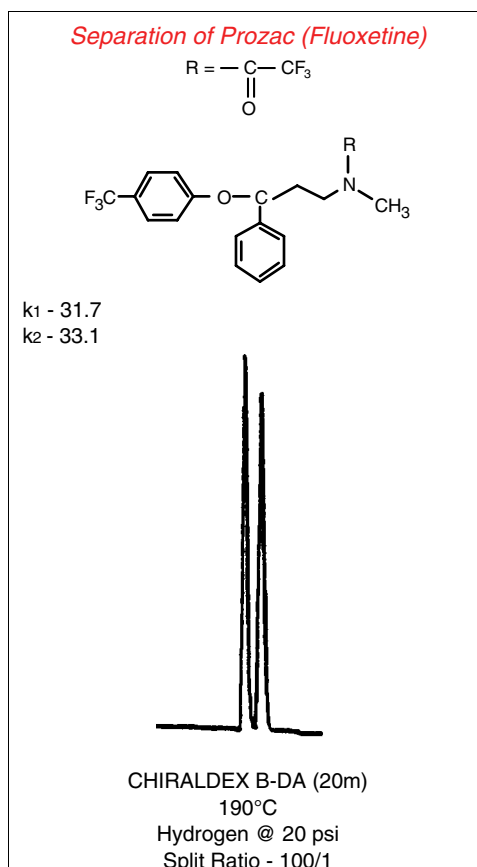
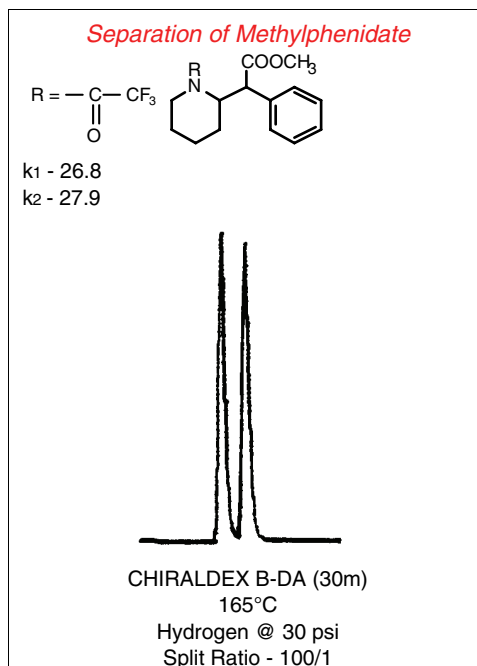
The dipentylated derivatives of alpha, beta and gamma cyclodextrin show pronounced selectivity differences based on the size, shape and functionality of the analyte. Strong evidence exists for inclusion complexation which is the basic driving mechanism and, therefore, resolution is effected by sample load. The beta-cyclodextrin* form has the broadest applicability. Sample volumes under 1 microliter at concentrations of 5mg/ml are typical with the 100/1 split ratio.

Features

- Hydrophobic surface
- Pronounced selectivity differences based on analyte size, shape and functionality
- Separates heterocyclic amines
- Different selectivity from other cyclodextrin derivatives
- Often shows reversal in elution order from PH series
- Selectivity priorities:
 - Alpha Simple epoxides, cyclic ethers, linear substituted alkanes
 - Beta Heterocyclic amines
 - Gamma Naphthyl analogs

Mechanism Observations

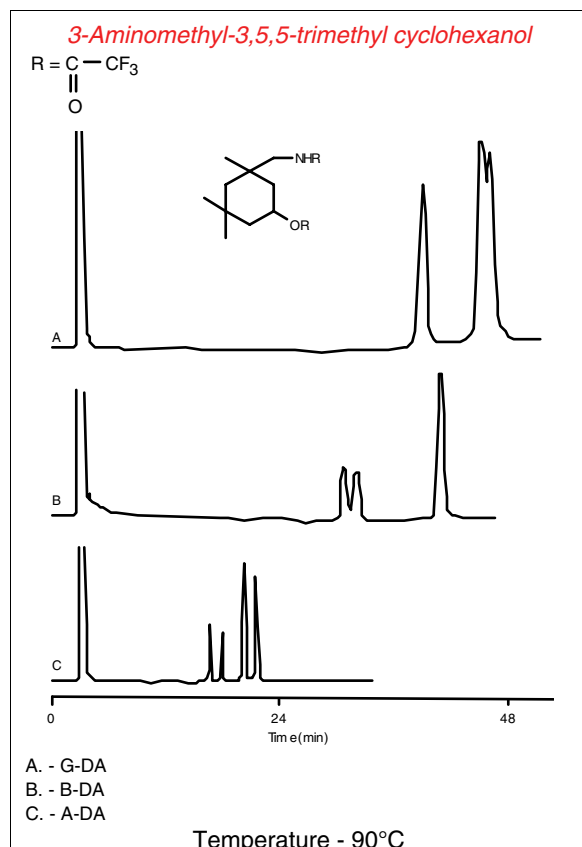
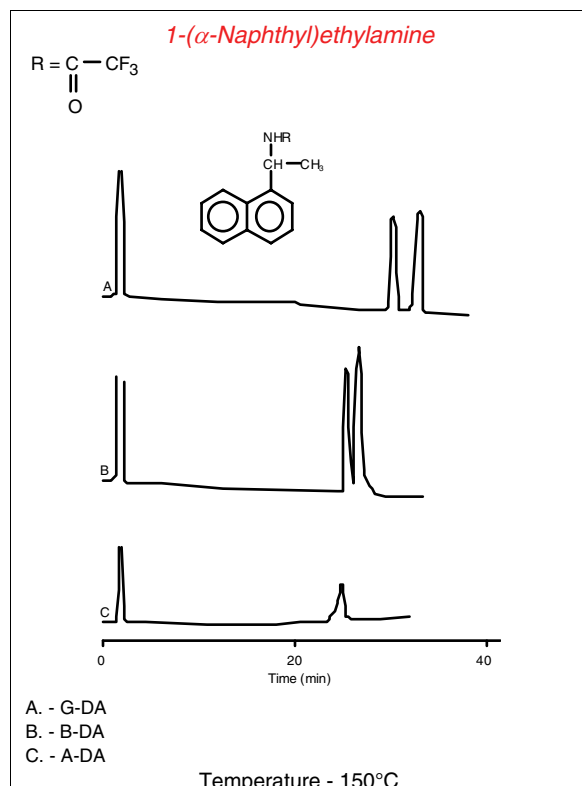
- Stronger inclusion for DA and, therefore, size selectivity is important.
- Critical temperature dependence for enantioselectivity
- Above this temperature no separation occurs.



Size Selectivity

Unlike LC, the size selectivity and chiral recognition applies to both aromatic and nonaromatic enantiomers for this phase. Alpha substituted naphthyl structures separate well on the gamma derivative.

Examples of Size Selectivity on the DA Series



CHIRALDEX Propionyl Derivative G-PN

This phase was designed to extend the scope of the CHIRALDEX G-TA. Instead of trifluoroacetylation of the position 3 hydroxyl, a propyl ester is formed. This has demonstrated two benefits: enhanced selectivity for chiral aromatic amines and increased selectivity for lactones. In addition, it has been noted that styrene oxide can be degraded catalytically by the trifluoroacetyl group. This degradation leads to phenylaldehyde as a by-product and interferes with the accurate determination of the enantiomeric ratio on CHIRALDEX TA columns. The CHIRALDEX G-PN resolves the enantiomers of styrene oxide without catalyzing any conversion. It also allows for simultaneous analysis of phenylaldehyde which may result from thermal degradation in the injector, usually less than 1%. It is projected other epoxides would demonstrate similar effects.

Features

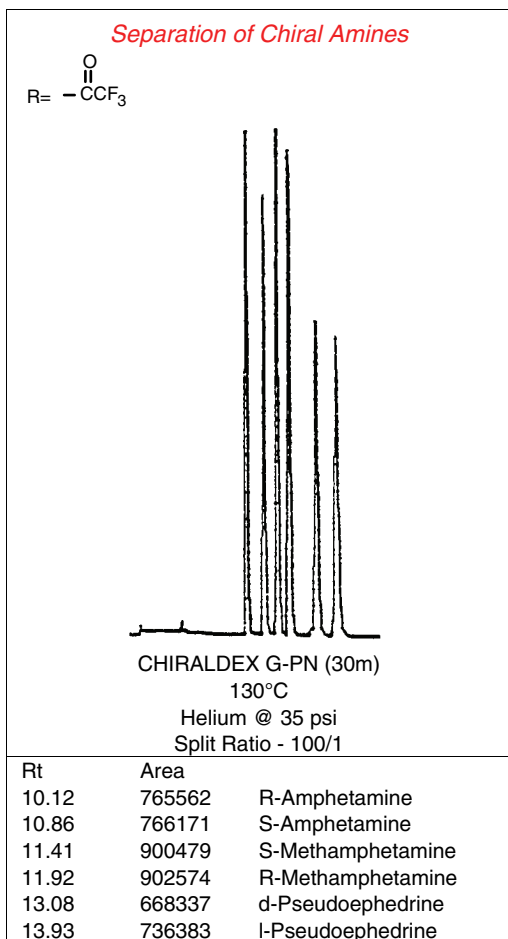
- Suitable for epoxide separations
- High selectivity for lactones
- High selectivity for aromatic amines

Mechanism Observations

There is little evidence of inclusion formation. Retention increases with increased chain length of analyte. This allows for efficient separation of a series of homologs.

Size Selectivity

The gamma cyclodextrin shows a higher degree of selectivity over the beta for this phase and very little selectivity based on cavity size.



CHIRALDEX Butyryl Derivative G-BP

This phase has shown its best selectivity for amino acids, certain amines and furan structures. It is a nonpolar phase and has the temperature stability of the alkyl type phases. It is considered a good alternative to the bonded amino phases with much broader separation capabilities.

Features

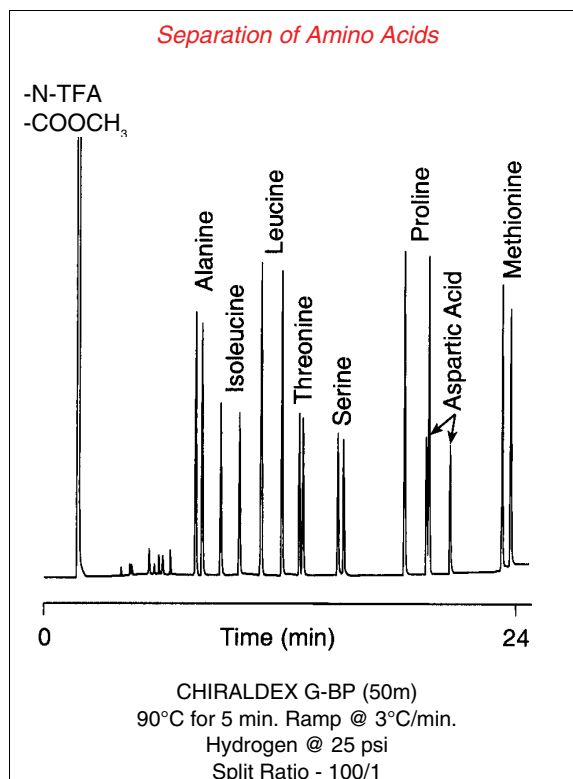
- Gamma most versatile
- High selectivity for amino acids, amines, furans
- Thermally stable

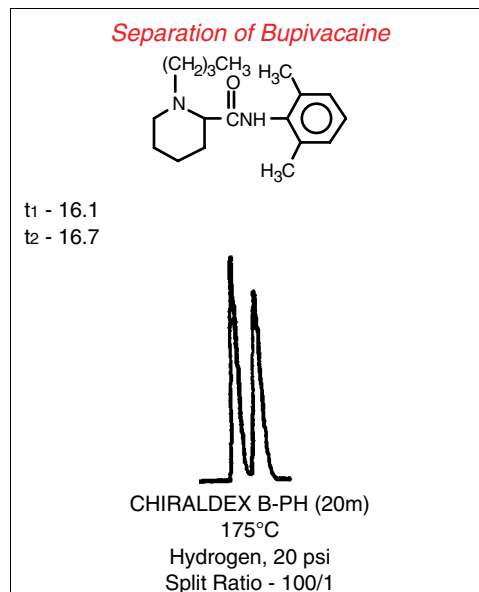
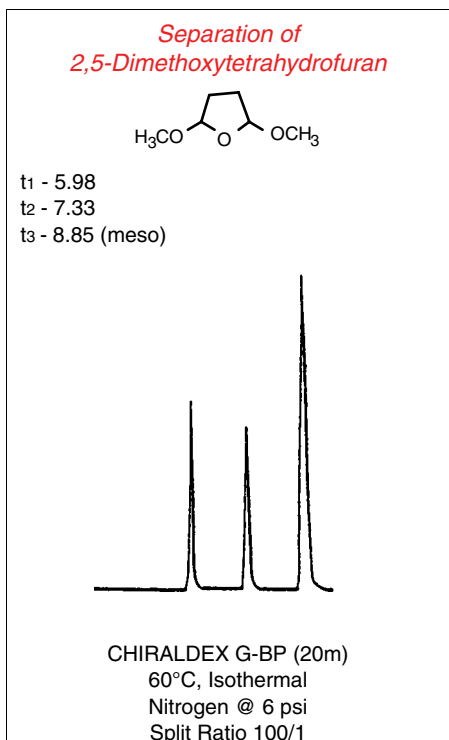
Mechanism Observations

- Alkyl chain on analyte contributes to chiral recognition
- High sample capacity, therefore, primary surface interactions

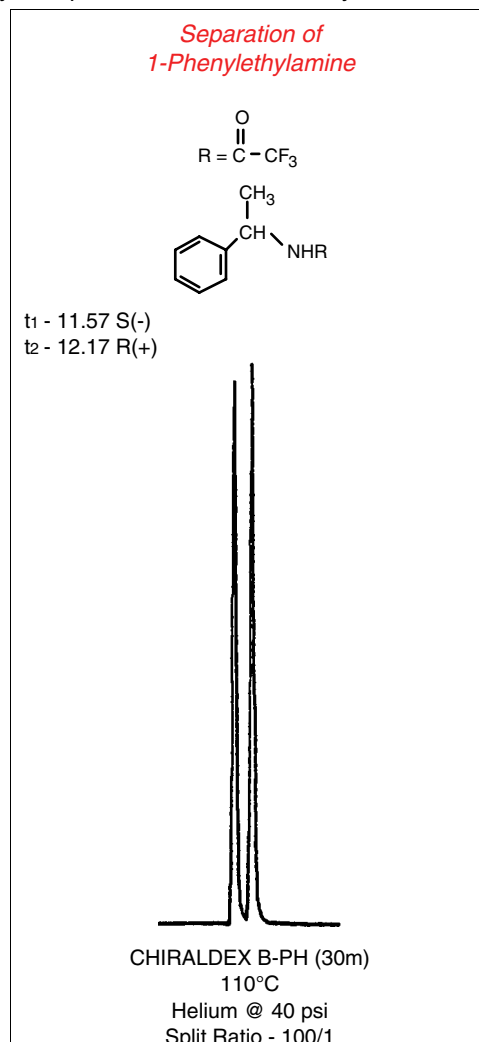
Size Selectivity

For this derivative, gamma-cyclodextrin exhibits a much broader selectivity than beta-cyclodextrin. The influence of the inclusion mechanism on selectivity is much reduced and capacities are, therefore, generally higher.





The most striking property of the PH phases are their ability to separate molecules with very little functionality.



CHIRALDEX S-Hydroxypropyl Derivatives A-PH, B-PH*

The first general purpose derivative involved substitution of the cyclodextrin hydroxyl groups with pure "S" hydroxypropyl followed by permethylation. The surface is hydrophilic in character and the influence of size and shape selectivity is greatly reduced but not absent. The beta-cyclodextrin* form has the broadest applicability.

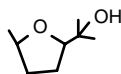
Features

- chiral stationary phase
- Hydrophilic surface
- Beta is more selective than alpha
- Separates a wide variety of enantiomers
- Resolves aliphatic, olefinic and aromatic enantiomers
- Good general purpose chiral column

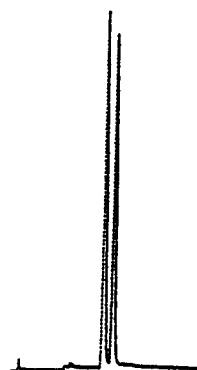
Mechanism Observations

- Reduced influence of inclusion complexing
- Less size selectivity for PH as opposed to DA
- Temperature range for enantiomeric selectivity: Isothermal to 200°C; temperature program to 220°C
- Characteristic temperature selectivity on a given column for a given class of compounds
- Fewer functional requirements for analyte than LC

Separation of 2-(1-Hydroxy-1-methylethyl)-5-methyltetrahydrofuran



t₁ - 16.38
t₂ - 17.86

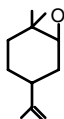


CHIRALDEX B-PH (30m)
80°C
Nitrogen @ 22 psi
Split Ratio - 100/1

Size Selectivity

For the PH series a minimal effect is seen based on size selectivity. The slightly higher temperature indicates stronger binding forces (retentive) for alpha which are not contributing to enantioselectivity differences. This is a general characteristic of PH phases.

Application: Limonene Oxide



Type	Length (m)	T°C	Alpha
A-PH	10	90	1.06

CHIRALDEX Permethylated Derivative B-PM

The permethylated beta-cyclodextrin is a stationary phase that has indicated a potential to separate a wide variety of racemates. Many of those separations are better resolved on other CHIRALDEX phases based on lower retention, better selectivity and greater efficiency. What the PM does especially well are some hydrocarbons like terpenes, certain underivatized alcohols and diols and some analytes with polar groups like tertiary amines.

Features

- Broad chiral selectivity
- Thermal limit to 230/250°C
- Strong inclusion/size selectivity
- Beta most selective of cyclodextrins

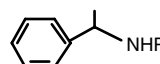
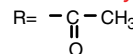
Mechanism Observations

- Inclusion a dominant mechanism
- Highest temperature stability of CHIRALDEX phases along with the DM

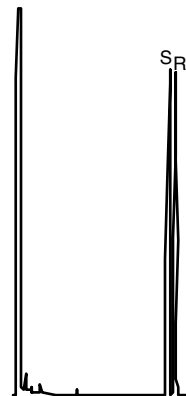
Size Selectivity

Some size selectivity is evident with the permethylated phase. The beta form will cover a broad range of molecule sizes.

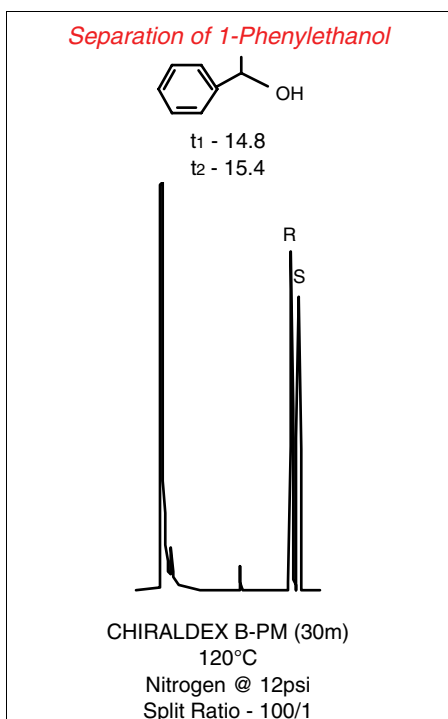
Separation of 1-Phenylethylamine



t₁ - 29.18
t₂ - 30.38



CHIRALDEX B-PM (30m)
130°C
Helium @ 34 psi
Split Ratio - 100/1



General Operating Parameters

Retention Behavior

Two types of retention behavior have been defined on these phases. All racemic separations can be placed in one of these two groups.

Group 1: Large $-\Delta(\Delta H)$'s indicate inclusion dominated enantioselective mechanism. Compounds in this group show large drops in NTP when the injection concentration of the analyte is increased. This is found predominantly in the CHIRALDEX DA series.

Group 2: Smaller $-\Delta(\Delta H)$'s of transfer indicate little or no complexation but analyte concentrations can be increased 10 to 50 times more than Group 1 solutes with no loss in NTP. This concentration experiment will help establish operating requirements and analyte group behavior.

Conditioning

All CHIRALDEX phases are conditioned after manufacture @ 170°C overnight. It may be necessary to condition the column prior to use. See MAOT chart on page 4 for column temperature maximums.

Conditioning times vary from 1 to 4 hours and is determined by the time it takes for baseline stabilization.

Both moisture and oxygen affect the selectivity and stability of these phases, therefore, good drying and oxygen traps are essential for continued optimum performance.

Split Ratio and Sample Solvent Choices

CHIRALDEX stationary phases are not bonded and can only be run in a split mode unless a retention gap is used. Maintain at least a 50/1 ratio on the 0.25mm columns. The phase coating can be affected by on-column injection as well as solvent choices. Choose a solvent which volatilizes at least 40°C below elution temperature for the first component of interest and keep the injection port temperature substantially higher than the column temperature (usually 200-250°C). It is possible to inject up to 5 microliters of solvent onto these columns with the use of the retention gap. Care must be taken, however, to properly volatilize the solvent so as not to affect film integrity. The retention gap technique described by Grob (5,6) is the safest way to deal with large sample volumes (see Page 22). Separation performance is sometimes affected by the amount of sample injected so it is best to routinely work with minimum sample and highest instrument sensitivity.

Sample Preparation

The capacity of chiral capillary columns is very low so samples should be sufficiently pure to protect the stationary phase. For the TA series it is especially important to have the sample free from moisture.

Methylene chloride extracts of aqueous samples contain >100ppm water, sufficient to cause the hydrolysis of the TA. Evaporation to near dryness in the presence of dimethoxypropane or passing the methylene chloride solution through a bed of anhydrous sodium sulfate will adequately dry the sample.

Solvents routinely used to dissolve or dilute samples are: ethyl ether, hexane, methylene chloride, methanol and ethanol. The alcohols are not to be used for the TA CHIRALDEX columns or for TFA derivatized alcohols.

Derivatization Effects

Achiral Derivatization

- ✓ Generally better efficiency (sensitivity). Diminishes interactions between analyte and CD hydroxyl and fused silica silanols.
- ✓ Generally increases volatility
- ✓ Lower analysis temperatures - maximize energy differences in diastereomeric association complexes
- ✓ Faster analyses - more throughput
- ✓ Validation procedures similar
- ✓ Changes interactions with stationary phase. In some cases, changing the derivative will enhance selectivity. Sometimes reversal of elution orders occur.

It is sometimes desirable to derivatize chiral analytes using achiral reagents. Some of the problems associated with derivatizing with chiral reagents are avoided because there is no need to worry about the optical purity of the reagent or chiral discrimination in enantiomeric reactivity. Derivatization may also enhance stereoselective interactions with the chiral stationary

phase (4). The effect of the derivatizing reagent on enantioselectivity is demonstrated in Figures 3-5. Common achiral derivatizing reagents applicable to selected compound classes are listed in Table 4. The procedures for preparing some common derivatives are listed in Table 5.

The first consideration when contemplating derivatization is that the derivatization reaction does not cause racemization. Second, the possibility that the derivatization reaction may produce by-products that interfere with the analysis must be taken into account. Greater care must be taken when the analyte has more than one functionality which is capable of reacting with the derivatizing agent (i.e., amino alcohols). Repeating the derivatization procedure on the sample helps ensure that the reaction goes to completion.

- Derivatization influences enantioselectivity
- One derivative type not necessarily applicable to all phases
- Derivatization affects volatility, polarity, hydrogen bonding and steric factors

*Figure 3 - Separation of different acyl derivatives of racemic 1-Methoxy 2-Aminopropane
Temperature - 130°C*

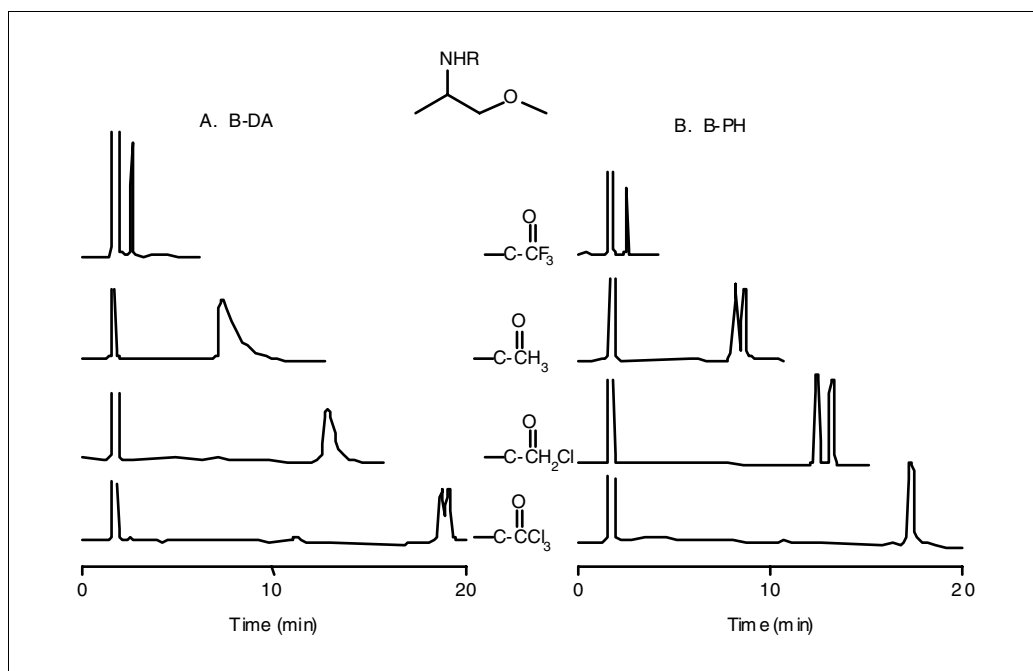
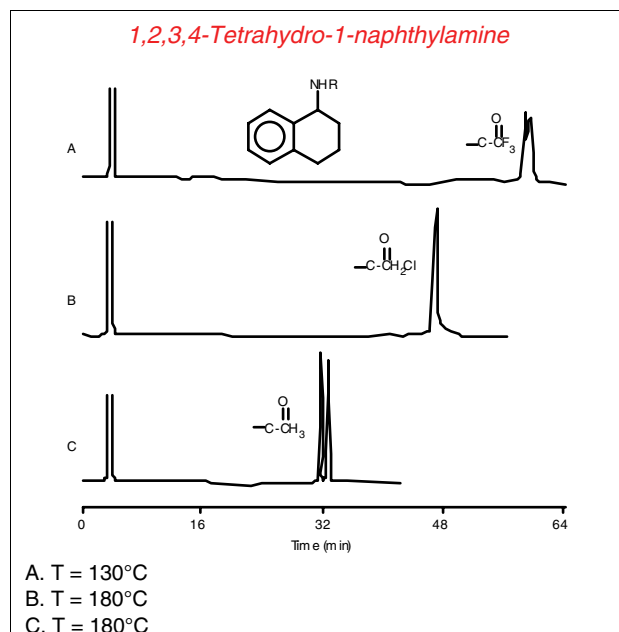


Figure 4 - Acetylation effect - bulky analyte



In Figure 5 chromatograms compare the separation of underivatized 2-butanol with different acyl derivatives. The same conditions were used for all separations. Note the reversal of elution order with the changing acyl derivative. Also, through derivatization, increased selectivity and efficiency is achieved. The bulky halogenated acyl derivative has the R,S elution order while the less bulky acetyl or no derivative has the opposite elution order (S,R).

Figure 5 - Effect of acylating reagent on selectivity and elution order of 2-butanol

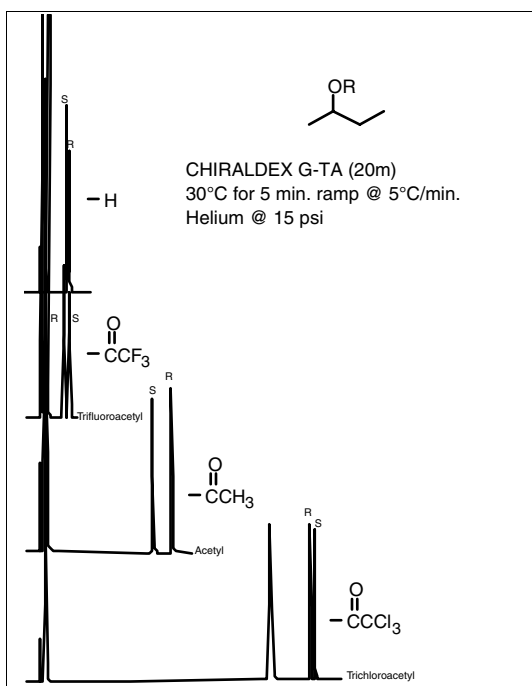


Table 4. Derivatizing reagents

Amines and Alcohols	Acids
Acetic Anhydride Trifluoroacetic Anhydride* Chloroacetic Anhydride Trichloroacetic Anhydride	Methyl Esters Trimethyl Silyl Esters

*Good for >85% cases studied.

Table 5. Derivatization procedures

Methyl Ester Method for Carboxylic Acids
<p>Method A. Methanolic HCl (2M)</p> <p>Materials:</p> <p>Refrigerated stock solution of 2M methanolic HCl. Three milliliter screw cap vials with silicone rubber inserts.</p> <p>Method:</p> <ol style="list-style-type: none"> 1) To approximately one milligram of analyte in a 3ml screw top vial add 1ml of 2M methanolic HCl. Place in boiling water bath (100°C) for 30 minutes with the screw-cap fastened. (Use only silicone rubber seals). 2) After 30 minutes, remove vial cap and allow solution to evaporate to dryness. Addition of small amounts of methanol or dimethoxypropane near end will help remove last bit of moisture from the sample. This is important to the stability of the CHIRALDEX TA series of columns. Care must also be exercised in not allowing sample to set too long in hot water to prevent evaporation of the ester. 3) Residue is then dissolved in 0.5ml of methylene chloride for injection. Other solvent choices are diethyl ether, hexane, methanol or ethanol.
<p>Method B. BF₃/MeOH</p> <p>Materials:</p> <p>BF₃/methanol</p> <p>Method:</p> <ol style="list-style-type: none"> 1) To 5mg organic acid in a 3ml vial, add 0.5ml BF₃/methanol solution. 2) Heat to 60°C for 10 minutes. Cool and transfer to a separatory funnel along with 3ml hexane. 3) Wash 2 times with saturated NaCl solution, dry organic layer over anhydrous Na₂SO₄ and evaporate solvent to near dryness. Dilute in solvent of choice.

Trifluoroacetylation Method for Amines and Alcohols

Materials:

Methylene chloride
Trifluoroacetic anhydride (TFA)
Three milliliter screw-cap vials with silicone rubber inserts.

Method:

- 1) Add to approximately 5mg analyte 2ml methylene chloride.
- 2) Add 0.2ml TFA, cap vial and heat @ 60°C for 20 minutes. For polyols heat to 100°C. Some polyols may require re-reaction.
- 3) Remove cap and evaporate to near dryness using dry nitrogen. Care should be taken when evaporating solvent not to vaporize sample. For highly volatile samples, evaporation to near dryness may result in loss of analyte. In cases such as this, a better sample work-up procedure would be:
 - A. Using dry nitrogen in a fume hood, evaporate any remaining trifluoroacetic anhydride from sample (methylene chloride and trifluoroacetic acid will still be present).
 - B. Extract sample twice with a 5% wt/v sodium bicarbonate solution.
 - C. Pass methylene chloride layer through a bed of anhydrous sodium sulfate (packed in Pasteur pipet) to remove residual water.
 - D. Sample is ready for injection.
- 4) Dissolve residue in 1ml methylene chloride. No further clean-up is necessary.

Trimethylsilyl Ester Method for Carboxylic Acids

Materials:

BSTFA - N,O-bis (trimethylsilyl) trifluoroacetamide
TMCS - Trimethylchlorosilane
Three milliliter screw-cap vials with silicone rubber inserts.

Method:

- 1) To approximately 1mg of analyte in a 3ml screw-cap vial add 0.1ml BSTFA containing 1% TMCS (v/v).
- 2) Add 0.1ml pyridine, cap vial and mix well.
- 3) Heat for 5 minutes at 45 °C, cool to room temperature and inject.

Temperature Effects

Enantioselectivity decreases with increasing temperature. For separations below 130°C a temperature gradient of 1-5°C/minute is used. Over 130°C a gradient of 5-10°C/minutes can be used. To effect faster elution or sharper peaks at temperatures below 130°C increase linear gas flow velocity. Never heat or cool the column at more than 15°C/min.

Temperature Settings

Column injector and detector port temperatures should be set at 200-250°C. A one meter section of deactivated tubing can be used between the column and injector and column and detector to protect it from high heat.

Column Lengths, Carrier Gas, Linear Velocity

Methods development on chiral stationary phases by gas chromatography differ from traditional (achiral) methods in achieving and optimizing the selectivity. In achiral gas chromatography, optimum to slightly higher than optimum linear velocities of carrier gas are used to control retention. In chiral (gas and liquid) chromatography, the highest enantiomeric selectivity is achieved by maximizing the energy differences in the diastereomeric association complexes formed between each enantiomer and chiral stationary phase. These energy differences become smaller with increasing temperature. **Therefore, to optimize a chiral separation, lower elution temperatures in conjunction with relatively higher linear velocities of carrier gas are generally best.**

Once this principle is known, carrier gas choice and other operational parameters must be investigated. The three carrier gases most often used are nitrogen, helium and hydrogen, and all three have distinct advantages. The highest purity gases, along with oxygen and moisture traps should be used. The advantages of nitrogen are low cost and highest efficiency. The disadvantage of nitrogen is the optimum linear velocity is 12cm/sec, and efficiency is rapidly lost with increasing linear velocity (the slope of the line for HETP vs linear velocity is steep). Helium is more expensive to use, but the optimum linear velocity is 20cm/sec, and the slope of the line for HETP vs linear velocity is flatter. Hydrogen has an optimum linear velocity of 40cm/sec, and has the flattest slope for HETP vs linear velocity and the lowest viscosity. Hydrogen will form explosive mixtures with air, and it must be used with care. The following table lists the optimum and maximum linear velocities along with relative viscosities for each carrier gas.

Table 6

Carrier Gas	μ_{opt} cm/sec	μ_{max} cm/sec	Relative Viscosity (η)
Nitrogen	12	30	1.00
Helium	20	60	1.22
Hydrogen	40	120	0.497

Helium is a good general purpose carrier gas. It can be used at high linear velocities and is safe to work with. As a starting point, 30-40cm/sec linear velocity is recommended. This will give a good mix of column efficiencies and analysis speed. Flow rates for helium or hydrogen are 2 to 4 times nitrogen values. As differences in energy between the diastereomeric complexes need to be exploited through lower temperatures and higher linear velocities, the use of hydrogen produces more efficient peaks. For highly volatile analytes (propylene oxide), low temperature and low linear velocities are necessary to achieve sufficient analyte stationary phase interaction for chiral recognition. In cases like these, nitrogen is the best carrier gas choice.

Often, it is desirable to switch between carrier gases. To calculate the head pressure of carrier gas needed for a certain linear velocity on a 0.25mm inner diameter column or the linear velocity at a certain column head pressure, use the following table. These values are approximate.

Table 7

Column Length (m)	cm/sec/psi			
	50°C	100°C	150°C	200°C
20	2.78	2.51	2.29	2.12
30	1.82	1.64	1.50	1.39
50	1.29	1.16	1.06	0.98

Example 1: Calculating linear velocity

To measure average linear velocity, use the equation

$$v_{ave} = \frac{L}{t_0}$$

where L is the length of the column in cm, and t_0 is the length of time (seconds) necessary for an unretained component to pass from the injector to the detector. A good unretained analyte is methane or the hydrocarbon gas from a disposable lighter.

A 20mx0.25mm column is being operated at 50°C. Its carrier gas is hydrogen at 15 psi. An injection of methane gives a retention time of 0.401 minutes. The actual average linear velocity is:

$$v_{ave} = \frac{2000 \text{ cm}}{(0.401 \text{ min}) \left(\frac{60 \text{ sec}}{\text{min}} \right)} = 83 \text{ cm/sec}$$

Example 2: Calculating linear velocity from nitrogen head pressure

A 30m x 0.25mm column is operated at 12 psi nitrogen at 100°C. The (calculated) average linear velocity (μ_{av}) would be:

$$\mu_{ave} = \frac{(p_i)(\mu_n)}{\eta}$$

Where:

μ_{av} is the average linear velocity in cm/sec

p_i is the column head pressure in psi

μ_n is the linear velocity per psi using nitrogen as the carrier gas from Table 7

η is the relative carrier gas viscosity from Table 6

$$\mu_{ave} = \frac{(12 \text{ psi}) \left(\frac{1.64 \text{ cm}}{\text{sec}} \right) (\text{psi})}{1.00} = 19.7 \text{ cm/sec}$$

Example 3: Calculating linear velocity from hydrogen head pressure

If, under these same exact conditions, hydrogen is used as the carrier gas, then the average linear velocity would be:

Relative viscosity for hydrogen from Table 6 = 0.497

$$\mu_{ave} = \frac{(12 \text{ psi}) \left(\frac{1.64 \text{ cm}}{\text{sec}} \right) (\text{psi})}{0.497} = 39.6 \text{ cm/sec}$$

Example 4: Calculating head pressure from linear velocity

A 50mx0.25mm column is to operate at a helium linear velocity of 40 cm/sec at 150°C. The necessary column head pressure would be:

$$p_i = \frac{(\mu_{ave})(\eta)}{\mu_n}$$

η from Table 6 = 1.22

μ_n from Table 7 = 1.06

$$p_i = \frac{\left(\frac{40 \text{ cm}}{\text{sec}} \right) (1.22)}{\left(\frac{1.06 \text{ cm}}{\text{sec}} \right) (\text{psi})} = 46 \text{ psi}$$

Detection

All capillary columns have extremely low capacities. To obtain the highest column efficiency and chiral selectivity, set the detector at the highest usable sensitivity and inject the lowest amount of sample (e.g. 1.0 μ l of 5 mg/ml solution with a split ratio of 100:1). All detector types including MS have been used with these phases.

For Mass Spectrometer Coupling: Use a 1 meter length of methyl/phenyl deactivated tubing for the transfer line. This can be connected via a press fit or other column coupler. For storage, leave the connecting tubing in place and vacuum seal at this terminus.

Reversing Elution Order

One of the most interesting and useful phenomena observed with the CHIRALDEX phases is the ability to reverse elution order. The significance of this reversal is the reliable quantitation of trace enantiomers. It is always desirable to have the trace component elute first to avoid interference from tailing of the larger component. This reversal has been accomplished in four ways. In order of probability they are:

Type:

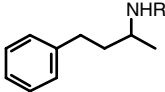
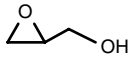
1. One cyclodextrin type to another (e.g. G-TA to B-TA)
2. One phase type to another (e.g. B-PH to B-DA)
3. Change of derivative (e.g. trifluoroacetyl to acetyl)
4. Operating below ambient temperatures

Current data indicate that molecules with limited inclusion complexing strength can reverse their elution order by any one of the first three methods. Aromatic structures have a fixed position in the cyclodextrin cavity and, as a result, have predictable elution orders for a given cyclodextrin derivative (Table 8). Saturated and heterocyclic rings as well as linear molecules have a more random interaction and, therefore, reversal can occur by restricting the cyclodextrin cavity (Table 8) (example - gamma to beta cyclodextrin or beta to alpha cyclodextrin). The reversal at sub-ambient conditions is only possible for molecules eluted below 100°C under normal operating conditions. The peaks usually exhibit low NTP.

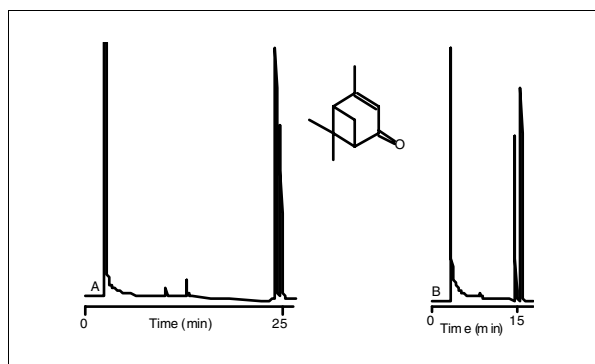
Examples :

Type 1 Verbenone
Type 2 Weiland Miescher Ketone
Type 3 Proline

Table 8. Reversal of elution order, aromatic vs. aliphatic

Compound/Structure	Phase	L(m)	T(°C)	k'	α	1st Isomer
4-Phenyl-2-butylamine 	B-TA	30	150	7.21	1.02	R
	G-TA	30	150	13.42	1.02	R
Glycidol 	A-TA	30	65	5.30	1.07	R
	B-TA	20	65	11.92	1.04	S
	G-TA	30	70	2.72	1.04	R
	A-PH	30	60	7.97	1.02	R
	B-PH	30	90	2.05	1.0	-

Type 1 reversal of elution order for verbenone (d>l)



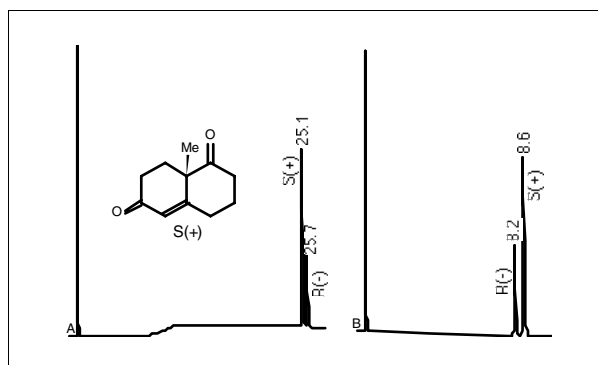
Conditions:

Chromatogram A : CHIRALDEX G-TA, 30m x 0.25mm
130°C, Isothermal, Nitrogen

Chromatogram B : CHIRALDEX B-TA, 30m x 0.25mm
130°C, Isothermal, Nitrogen

Note that even though the G-TA column does give adequate selectivity, analysis times could be reduced by 10 minutes by using the B-TA column.

Type 2 reversal of elution order for Weiland Miescher ketone



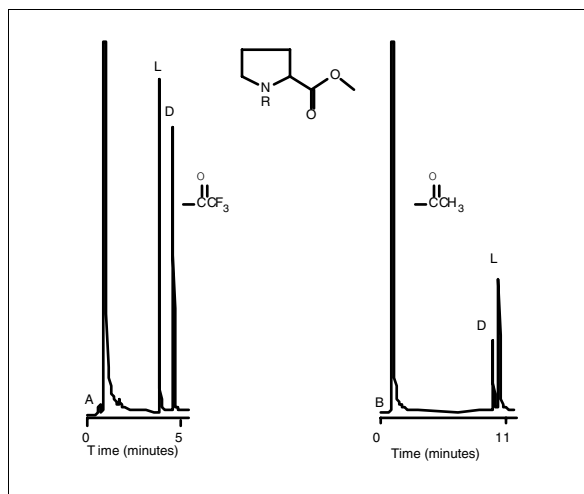
Conditions:

Chromatogram A : CHIRALDEX G-TA, 30m x 0.25mm
120°C for 5 min, ramp @ 5°C/min,
hold @ 150°C Helium @ 35 psi

Chromatogram B : CHIRALDEX G-BP, 10m x 0.25mm
155°C, Isothermal Helium @ 10 psi

Note that although the G-TA column does resolve the two isomers, the G-BP gives a better separation in one third of the analysis time.

Type 3 reversal of elution order, same stationary phase, different acyl derivative. Analyte is proline methyl ester.



Conditions:

Chromatogram A: trifluoroacetyl derivative
CHIRALDEX G-TA, 30m x 0.25mm
140°C, Helium @ 34 psi

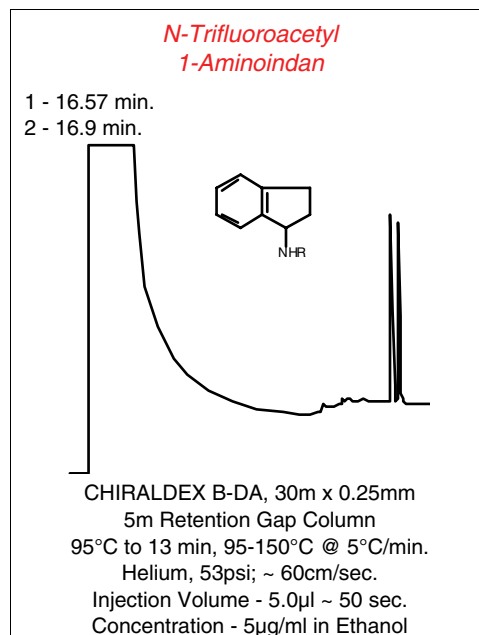
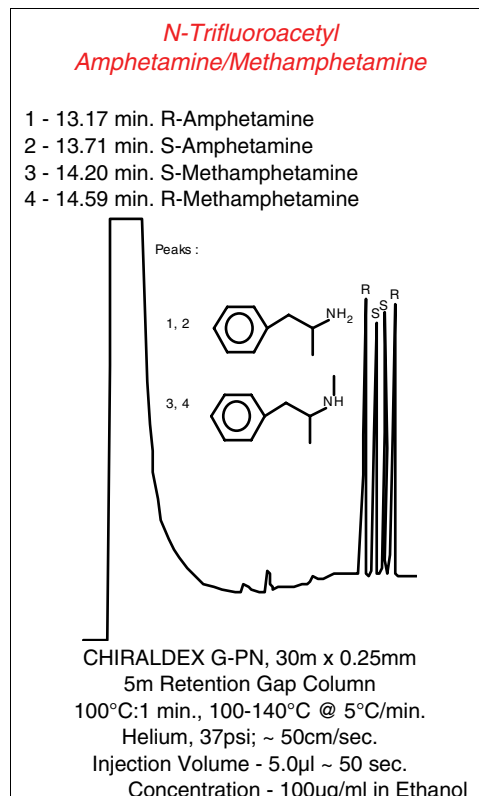
Chromatogram B: acetyl derivative
CHIRALDEX G-TA, 30m x 0.25mm
150°C, Helium @ 34 psi

Methyl Phenyl Deactivated Tubing as Retention Gap Guard Column

Precolumns for CHIRALDEX GC phases
Useful as transfer lines or guard columns
Inertness tested

A methyl phenyl deactivated fused silica precolumn can be used to protect capillary GC columns from non-volatile sample impurities as well as to allow for the injection of sample volumes up to 7µl "on column". A 5 or 10 meter column is generally used. The large volume (1-7µl) is slowly injected (1-2 minutes) at a temperature above the boiling point of the solvent and below the vaporization temperature of the analyte. For this to work, an elution temperature difference of at least 40°C is necessary between solvent and analyte. After completion of the injection, the temperature is programmed @ 5°/min to the normal operating temperature. Minimal loss in efficiency (~3%) is involved and generally lower limits of detection are obtained from plasma or serum extracts (see references 5 and 6).

Examples - Direct Injection Method



Storage

When storing these columns heat activate at 170°C under normal chromatographic conditions: cool in oven with carrier gas flow. Remove when cool, flame seal one end, pull vacuum for five minutes and flame seal at vacuum end. Sealing ends with rubber septum is not effective for keeping oxygen and water vapor out of column and should not be used.

Column Assessment Parameters and Quality Assurance

Operating and installation instructions are included with each column along with a column assessment sheet specific for that column and test sample for selectivity. We recommend the column be evaluated with this test mixture before proceeding with your own compounds and to evaluate the column from time to time especially prior to method development.

CHIRALDEX GC Test Mixtures

It is important to validate both the instrument and column before developing a new assay especially after prolonged storage of the column. The following standards have been prepared for that purpose.

Test mixtures are available in 1ml size with a concentration of 5mg/ml. See Ordering Information section for catalog numbers.

Description	CHIRALDEX GC Column
Tetrahydro-2-(2-propynyloxy)-2H-pyran	A-DA
2-Methylpiperidine (N-TFA)	G-BP G-TA
2-Aminoheptane (N-TFA)	G-PN A-TA B-PH G-DP G-DM
1-Aminoindan (N-TFA)	B-DA G-DA
2-(Bromomethyl)tetrahydro-2H-pyran	B-TA B-DP
3,4-Dihydro-2-ethoxy-2H-pyran	A-PH
1-Phenyl-1-ethanol	B-PM B-DM

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Applications

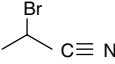


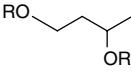

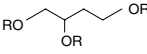

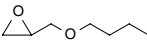
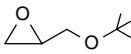
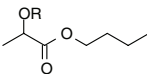

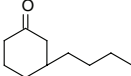
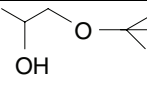


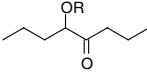

The recommended CSPs and operating conditions for the compounds listed below were chosen from a 16 year accumulated database. During the development and application of these CSPs based on derivatized cyclodextrins, new understanding evolved that led to the development of a number of new and better performing CSPs for certain classes of analytes. As a result, many compounds appeared in previous Handbooks with multiple choices for the separation. It is the intention of this current list to provide the analyst with the conditions for the highest resolution with the shortest retention at the lowest temperature. When a reversal of elution order was identified between two CSPs, we included both sets of conditions so the best elution order could be chosen in order to have the residue enantiomer elute first.

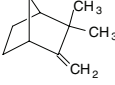

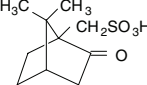


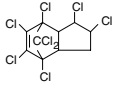
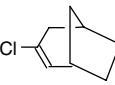
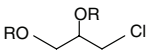




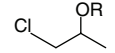

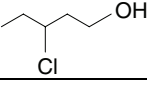


Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
Acetoine			B-DM	30	50	2.5	1.388	12.43
2-Acetoxypropanal			B-TA	30	80	3.36	2.69	
α-Acetyl-α-methyl-γ-butyrolactone			B-TA	10	120	6.28	1.59	
Endo and exo-2-Acetyl-5-norbornene			G-TA	10	100	4.30 6.32	1.11 1.09	
N-Acetylhomocysteine thiolactone			G-PN	10	175	3.0	1.15	
endo and exo-2-Acetylnorbornane			A-DA	10	40	3.09	1.05 1.0	
Alanine		-CH ₃ -COCF ₃	B-TA	40	110	1.76	1.22	
Allyl glycidyl ether			B-PH	20	45	10.1	1.05	
3-Amino-1,2-propanediol		-COCF ₃	B-TA	10	140	6.14	1.12	
2-Amino-1-butanol		-COCF ₃	G-TA	10	100	7.04	1.17	
3-Amino-1-hexanol		-COCF ₃	B-TA	10	120	4.0	1.08	
2-Amino-1-hexanol		-COCF ₃	G-TA	10	100	10.4	1.1	
2-Amino-1-methoxypropane		-COCF ₃	G-TA	10	100	1.71	1.08	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
2-Amino-1-pentanol		-COCF ₃	B-TA	10	120	2.6	1.11	
2-Amino-1-propanol		-COCF ₃	G-TA	10	100	6.53	1.99	
1-Amino-2-propanol		-COCF ₃	G-TA	10	100	5.71	1.20	
2-Amino-3-methyl-1-butanol		-COCF ₃	G-TA	10	100	6.43	1.19	
2-Amino-4-methylpentane		-COCF ₃ -COCH ₃ -COCH ₂ Cl	B-TA	10 10 10	100 120 100	1.83 2.50 7.33	1.25 1.09 1.02	
2-Amino-6-methylheptane		-COCF ₃ -COCH ₃	B-TA	10 10	100 120	7.90 8.13	1.22 1.36	
2-Aminobutyric acid		-CH ₃ -COCF ₃	G-TA	30	100	3.12	1.61	
3-Aminobutyric acid, methyl ester		-COCF ₃	B-DP	30	110	13.9	1.089	4.62
2-Aminoheptane		-COCF ₃ -COCH ₃ -COCH ₂ Cl -COCHCl ₂	B-TA	10 10 10 10	100 120 120 140	5.22 6.13 6.61 4.81	1.23 1.25 1.08 1.03	
3-Aminoheptane		-COCF ₃	G-DP	20	110	6.7	1.113	5.04
1-Aminoindan		-COCF ₃	G-DP	20	150	12.0	1.345	16.5
3-Aminomethyl-3,5,5-trimethylcyclohexanol		-COCF ₃	A-DA	10	90	10.8	1.20 1.19	
endo-2-Aminonorbornane		-COCF ₃	B-TA	10	100	11.17	1.07	
exo-2-Aminonorbornane		-COCF ₃	B-TA	10	100	13.3	1.10	
2-Aminopentane		-COCF ₃ -COCH ₃ -COCH ₂ Cl	B-TA	10 10 10	100 120 120	1.57 2.13 2.52	1.24 1.23 1.07	
Amphetamine		-COCF ₃	G-PN	20	120	18.82	1.09	
Aspartic acid		-CH ₃ -COCF ₃	G-TA	30	120	7.08	1.37	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
D,L-Aspartic acid, dimethyl ester		-COCF ₃	G-BP	20	140	8.6	1.06	
<i>endo</i> - and <i>exo</i> -2-Benzoyl-5-norbornene			G-TA	10	120	34.6 41.8	1.0 1.04	
4-Benzyl-2-oxazolidinone			B-DA	20	180	87.5	1.02	
<i>endo</i> and <i>exo</i> -2-Benzylbornane			A-DA	10	70	3.61	1.0 1.02	
2-Benzylpyrrolidine		-COCF ₃	B-TA	10	140	10.2	1.06	
3,7-Bicyclo-3-methyl-4-ene heptalactone			G-TA	30	130	10.5	1.18	
1,d-Borneol			B-DM	30	100	14.1	1.084	4.12
<i>endo</i> -Brevicomine			G-BP	20	75	10.72	1.18	
<i>exo</i> -Brevicomine			G-BP	20	75	7.81	1.12	
α-Bromo-γ-butyrolactone			B-DP	30	180	3.0	1.559	17.5
α-Bromo-γ-butyrolactone			B-DM	30	100	33.9	1.154	4.32
α-Bromo-γ-valerolactone			B-DP	30	160	3.5 4.1	1.898 1.433	25.35 17.11
2-Bromo-1-chloropropane			G-TA	10	60	22.0	1.02	
2-Bromo-1-phenylpropane			G-TA	10	100	6.39	1.06	
3-Bromo-2-butene			B-TA	10	50	4.89	1.31	
2-Bromo-3-methylbutyric acid		-CH ₃	G-TA	10	90	5.66	1.09	
2-Bromobutane			G-TA	40	30	2.98	1.16	


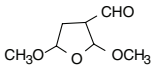
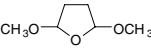
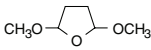
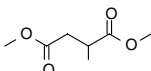
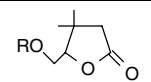


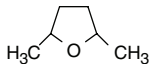
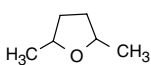


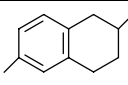
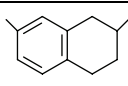
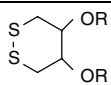
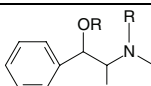
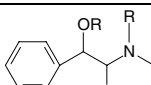
Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
2-Bromobutyric acid <i>sec</i> -butyl ester			G-TA	10	80	10.0 10.6	1.22 1.07	
2-Bromobutyric acid, methyl ester			B-DP	30	80	4.7	1.084	3.55
2-Bromoheptane			G-PN	10	60	7.41	1.04	
2-(Bromomethyl)tetra-hydro-2H-pyran			B-TA	10	90	3.72	1.16	
<i>exo</i> -2-Bromonorbornane			A-DA	10	30	4.43	1.05	
2-Bromooctanoic acid, methyl ester			G-DP	20	110	9.8	1.076	2.82
2-Bromopentane			B-DM	30	45	5.9	1.107	2.70
Bromopheniramine			B-DA	30	160- 180	75.6	1.02	
2-Bromopropionic acid <i>sec</i> -butyl ester			G-TA	10	80	4.89 5.39	1.29 1.02	
2-Bromopropionic acid <i>sec</i> -heptyl ester			G-TA	10	90	15.1 15.4	1.16 1.04	
2-Bromopropionic acid hexyl ester			G-TA	10	80	30.9	1.04	
2-Bromopropionic acid <i>sec</i> -hexyl ester			G-TA	10	80	14.7 15.1	1.25 1.07	
2-Bromopropionic acid methyl ester			G-TA	10	80	2.44	1.47	
2-Bromopropionic acid <i>sec</i> -octyl ester			G-TA	10	90	29.3 29.6	1.16 1.03	
2-Bromopropionic acid <i>sec</i> -pentyl ester			G-TA	10	80	8.0 8.37	1.25 1.05	
2-Bromopropionic acid ethyl ester			G-TA	10	80	3.50	1.16	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
2-Bromopropionitrile			G-TA	10	70	4.13	1.06	
1,3-Butadiene diepoxide			G-TA	10	80	3.93	1.11	
1,2-Butadienediepenide			B-DP	30	90	16.7	1.059	2.16
1,3-Butanediol		-COCF ₃	G-TA	10	70	4.79	1.21	
2,3-Butanediol		-COCF ₃	G-TA	10	70	1.43	1.58	
1,2,4-Butanetriol		-COCF ₃	G-PN	10	100	7.25	1.06	
2-Butanol		-COCF ₃	B-TA	10	40	0.69	1.22	
<i>n</i> -Butyl glycidyl ether			G-TA	10	45	23.2	1.04	
<i>t</i> -Butyl glycidyl ether			A-DA	10	30	4.32	1.06	
Butyl lactate		-COCF ₃	B-PH	10	60	9.3	1.04	
2-Butylamine		-COCF ₃	G-TA	10	80	4.3	1.04	
3-Butylcyclohexanone			B-TA	40	120	8.2	1.06	
1- <i>t</i> -Butyloxy-2-propanol			B-DM	30	60	5.4	1.04	8.29
3-Butylphthalid			G-TA	30	170	5.73	1.09	
3-Butyn-2-ol			B-DM	30	30	1.7	1.25	5.90
Butyrolin		-COCF ₃	G-PN	10	80	6.34	1.11	
β-Butyrolactone			B-TA	10	100	6.19	1.30	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
l,d-Camphene			B-DM	30	60	7.7	1.049	2.03
Camphor			G-DP	20	100	13.9	1.125	4.35
Camphorsulfonic acid			G-TA	30	160	20.25	1.06	
Carvone			B-TA	10	110	7.05	1.09	
Cashmeran			G-TA	30	120	18.8	1.08	
cis-Chlordane			G-BP	20	180	28.54	1.03	
3-Chloro(3,2,1)bicyclo-2-octene			B-TA	10	70	6.8	1.02	
3-Chloro-1,2-propanediol		-COCF ₃	B-TA	10	90	3.52	1.09	
3-Chloro-1-butene			B-TA	10	50	1.01	1.06	
1-Chloro-2-bromopropane			B-TA	10	30	4.72	1.29	
3-Chloro-2-butanone			G-TA	10	60	2.43	1.59	
endo- and exo-3-Chloro-2-norbomanone			G-TA	10	120	6.57 10.2	1.06 1.04	
1-Chloro-2-propanol		-COCF ₃	G-TA	10	70	5.21	1.02	
2-Chlorobutane			G-TA	40	30	1.03	1.17	
2-Chlorobutanol			B-TA	30	70	15.55	1.06	
2-Chlorocyclohexanone			G-TA	10	100	4.26	1.10	
2-Chlorocyclopentanone			G-TA	10	110	2.47	1.33	

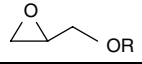
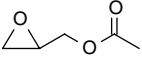
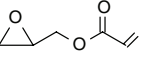
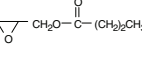
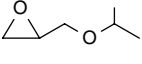
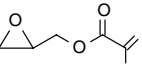
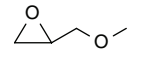
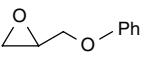
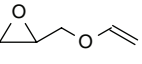
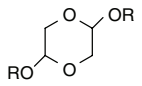
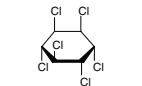
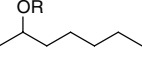
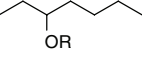
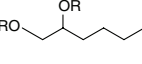
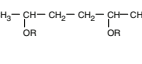
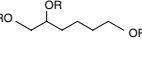
Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
2-(Chloromethyl)tetra-hydro-2H-pyran			B-TA	10	90	1.89	1.29	
5-Chloromethyl-2-oxazolidinone			B-PM	30	175	10.49	1.04	
exo-2-Chloronorbornane			A-DA	10	30	2.87	1.08	
1-(p-Chlorophenyl)ethanol		-COCF ₃	B-TA	30	120	2.3	1.09	
1-(p-Chlorophenyl)ethanol			B-DM	30	130	8.9	1.067	2.43
p-Chlorophenylalanine		-CH ₃ -COCF ₃	G-TA	10	140	13.2	1.09	
R,S-2-Chloropropionic acid, methyl ester			B-DP	30	70	5.3	1.849	21.29
S,R-2-Chloropropionic acid, methyl ester			B-DM	30	70	1.5	1.152	5.32
2-Chloropropionitrile			G-TA	10	70	1.65	1.06	
Chlorpheniramine			B-DA	30	160-180	46.79	1.02	
Ciprofibrate			B-DM	30	155	14.0	1.046	2.37
Ciprofibrate, methyl ester			G-TA	30	140	69.5	1.02	
trans-1,2-Cycloheptanediol			B-DM	30	120	11.1	1.079	1.38
trans-1,2-Cyclohexanediol			B-DM	30	120	5.2	1.095	1.84
trans-1,2-Cyclohexanediol		-COCF ₃	G-DM	20	80	4.5	1.198	5.02
1-Cyclohexyl-2,2-dimethylpropanol			G-TA	40	75	33.14	1.04	
S,R-1-Cyclohexylethylamine		-COCF ₃	B-DP	30	130	7.4	1.126	5.61

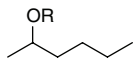
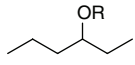
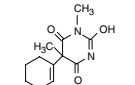
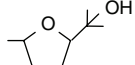
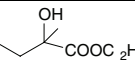
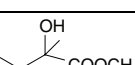
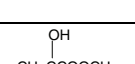
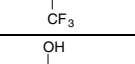
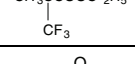
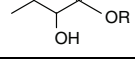
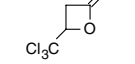
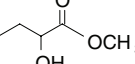
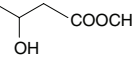
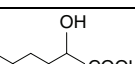
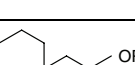
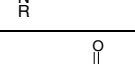
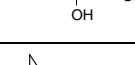
Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
<i>trans</i> -1,2-Cyclooctanediol		-COCF ₃	G-DM	20	90	10.9	1.038	1.28
γ-Decalactone			B-DP	30	160	8.0	1.031	1.61
γ-Decalactone			B-DM	30	130	14.9	1.225	3.08
2-Decanol		-COCF ₃	G-DM	20	60	54.3	1.028	1.41
δ-Decanolactone			G-TA	10	140	11.8	1.02	
γ-Decanolactone			G-TA	20	170	14.3	1.03	
γ-Decyl-γ-butyrolactone			G-TA	20	170	14.29	1.04	
Dehydronorketamine		-COCF ₃	G-DM	20	150	7.0	1.200	1.88
RR,SS- <i>trans</i> -1,2-Diaminocyclohexane		-COCF ₃	B-DM	30	150	3.7	1.071	2.93
SS,RR- <i>trans</i> -1,2-Diaminocyclohexane		-COCF ₃	G-DM	20	150	5.7	1.135	4.22
1,2-Dibromo-3-chloropropane			G-TA	10	*	13.7	1.04	
1,3-Dibromobutane			B-TA	10	70	7.56	1.05	
2,3-Dichlorobutane			B-TA	10	70	1.72	1.45	
1,2-Dichloropropane			B-TA	10	70	1.11	1.16	
<i>N'</i> -(Difluoroethyl) nornicotine			B-DA	30	160	5.3	1.02	
3,4-Dihydro-2-ethoxy-2H-pyran			B-DM	30	70	3.4	1.025	1.74
3,4-Dihydro-2-methoxy-2H-pyran			B-TA	10	60	1.83	1.44	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
Dilactone			G-PN	10	150	2.59	1.44	
2,5-Dimethoxy-3-tetrahydrofuran carboxaldehyde			B-PH	10	100	5.0	1.05 1.10 1.09 1.02	
2,5-Dimethoxytetrahydrofuran			B-DM	30	60	3.8	1.293	10.12
<i>trans</i> -2,5-Dimethoxytetrahydro-furan			G-TA	10	60	1.46	1.92	
Dimethyl methylsuccinate			G-TA	30	110	1.50	1.03	
β,β-Dimethyl-γ-(hydroxymethyl)-γ-butyrolactone		-COCF ₃	B-TA	10	120	12.3	1.10	
3,3-Dimethyl-2-butylamine		-COCF ₃	B-DP	30	100	4.0	1.174	6.57
3,3-Dimethyl-2-butylamine		-COCF ₃	G-DM	20	40	31.8	1.148	1.71
<i>trans</i> -2,5-Dimethyltetrahydro-furan			G-TA	10	45	8.57	1.06	
<i>trans</i> -2,5-Dimethyltetrahydro-furan			G-BP	20	80	0.9	1.15	
1,4-Dimethyltetralin			B-PH	10	80	21.5	1.06	
1,8-Dimethyltetralin			B-PH	10	110	6.9	1.03	
2,6-Dimethyltetralin			B-PH	10	70	35.2	1.03	
2,7-Dimethyltetralin			B-PH	10	170	38.4	1.02	
<i>trans</i> -1,2-Dithiane-4,5-diol		-COCF ₃	A-DA	10	70	8.68	1.12	
Ψ-Ephedrine		-COCF ₃	B-DA	20	120	13.3	1.04	
Ephedrine		-COCF ₃	G-PN	10	130	5.86	1.15	

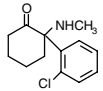
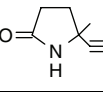
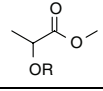
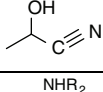
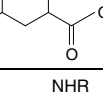
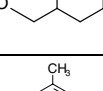
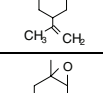
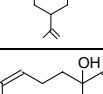
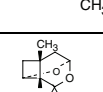
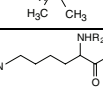
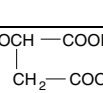
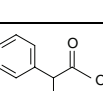
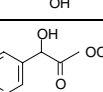
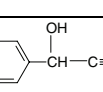
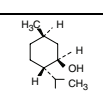
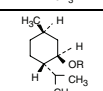
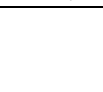
Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
Epibromohydrin			G-BP	10	45	12.37	1.15	
Epichlorhydrin			G-TA	10	40	7.5	1.234	
Epichlorohydrin			G-DM	20	40	4.8	1.142	1.35
Epifluorohydrin			G-TA	10	30	2.50	1.02	
Epifluorohydrin			A-TA	20	40	1.73	1.13	
1,2-Epoxy-3-butene			B-TA	30	22	2.5	1.19	
1,2-Epoxy-5-hexene			G-DP	20	30	10.1	1.056	1.2
1,2-Epoxy-7-octene			A-DA	10	40	4.62	1.05	
<i>trans</i> -2,3-Epoxybutane			B-TA	10	40	2.1	1.03	
1,2-Epoxybutane	$\text{CH}_3\text{-CH}_2\text{-CH-O-CH}_2$ 		G-TA	20	30	1.54	1.12	
1,2-Epoxydecane			A-TA	10	90	9.88	1.02	
1,2-Epoxydodecane			A-TA	10	90	108.0	1.02	
1,2-Epoxyhexane			G-TA	10	40	5.36	1.10	
1,2-Epoxyoctane			A-TA	10	50	22.3	1.04	
1,2-Epoxytetradecane			A-TA	10	100	125.0	1.02	
2-Epoxytetrahydrofuran			B-DM	30	60	2.2	1.177	4.20
2-Ethoxytetrahydrofuran			B-DM	30	60	2.2	1.179	4.88

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
Ethyl 2-bromovalerate			G-PN	20	70	15.62	1.14	
Ethyl 2-methylbutyrate			G-TA	30	60	1.8	1.06	
Ethyl 3-hydroxy- butyrate		-COCF ₃	G-PN	10	80	2.44	1.15	
Ethyl 3-phenylglycidate			G-TA	10	120	16.6 23.8	1.05 1.04	
Ethyl lactate			B-TA	30	70	4.22	1.25	
L,D-Ethyl lactate			B-DM	30	50	7.5	1.295	2.09
Ethyl mandelate			G-TA	20	120	15.23	1.07	
Ethyl nipecotate		-COCF ₃	G-PN	10	130	6.76	1.04	
Ethyl pipecolinate		-COCF ₃	G-PN	10	130	3.65	1.13	
3-Ethylheptanoic acid		-CH ₃	G-TA	30	70	13.43	1.03	
1-Ethylindan			A-PH	10	90	15.0	1.05	
2-Ethyltetralin			B-PH	10	70	40.5	1.01	
Fenfluramine		-COCF ₃	G-TA	40	120	15.8	1.06	
3-Formyltetrahydro-2H-Pyran			G-TA	20	100	2.94	1.23	
Frontalin			B-TA	30	95	1.3	1.07	
Glutamic Acid		-CH ₃ -COCF ₃	G-TA	30	140	3.66	1.06	
Glyceric acid methyl ester		-COCF ₃	G-PN	20	90	19.0	1.03	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
Glycidol		-COCF ₃	G-TA	10	60	4.29	1.06	
Glycidyl acetate			G-TA	40	70	11.4	1.25	
Glycidyl acrylate			G-TA	10	80	10.1	1.14	
Glycidyl butyrate			G-TA	30	80	25.69	1.05	
Glycidyl isopropyl ether			A-DA	10	30	1.08	1.06	
Glycidyl methacrylate			B-TA	10	80	6.39	1.04	
Glycidyl methyl ether			G-TA	10	45	5.89	1.16	
Glycidyl phenyl ether			G-TA	40	100	17.3	1.02	
Glycidyl vinyl ether			A-DA	10	30	1.88	1.04	
Glycolaldehyde		-COCF ₃	G-PN	10	90	7.33 8.61	1.06 1.07	
Halothane	CIBrCHCF ₃		B-TA	40	21	1.8	1.06	
α-HCH			G-DP	20	170	16.8	1.163	6.78
2-Heptanol		-COCF ₃	B-TA	10	60	3.11	1.14	
3-Heptanol		-COCF ₃	G-TA	10	40	8.39	1.26	
1,2-Hexanediol		-COCF ₃	G-TA	40	80	3.09	1.22	
(SS), (RR)-2,5-Hexanediol		-COCF ₃	G-PN	20	60	13.56	1.13	
1,2,6-Hexanetriol		-COCF ₃	B-TA	10	110	23.3	1.07	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
2-Hexanol		-COCF ₃	G-TA	10	40	3.75	1.31	
3-Hexanol		-COCF ₃	G-DP	20	40	4.0	1.144	3.07
Hexobarbital			B-DA	10	180	24.4	1.02	
2-(1-Hydroxy-1-methylethyl)-5-methyltetrahydrofuran			B-PH	30	80	3.3	1.08	
2-Hydroxy-2-methylbutyric acid, ethyl ester			G-DM	20	45	20.7	1.149	4.90
2-Hydroxy-2-methylbutyric acid, methyl ester			B-DP	30	70	4.0	1.091	3.16
2-Hydroxy-2-trifluoromethylpropionic acid, methyl ester			B-DP	30	70	2.9	2.035	18.01
2-Hydroxy-2-trifluoromethylpropionic acid, ethyl ester			B-DM	30	35	8.3	1.101	3.5
2-Hydroxy-3-methylbutyric acid		-CH ₃	G-TA	30	90	2.62	1.14	
3-Hydroxy-4,4,4-trichlorobutyric-β-lactone			G-TA	10	100	15.6	1.19	
2-Hydroxybutyric acid, methyl ester			B-DM	30	70	3.3	1.217	7.17
3-Hydroxybutyric acid, methyl ester			B-DP	30	70	10.4	1.127	5.51
2-Hydroxycaproic acid			B-DM	30	100	5.3	1.105	3.85
2-(2-Hydroxyethyl)piperidine		-COCF ₃	G-PN	10	130	7.05	1.09	
2-Hydroxyhexanoic acid		-CH ₃	G-TA	30	110	1.85	1.12	
exo-2-Hydroxymethyl-norbornane			A-DA	10	40	17.71	1.07	
2-Hydroxyoctanoic acid		-CH ₃	B-TA	40	110	6.28	1.11	

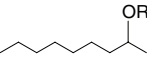
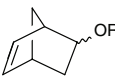
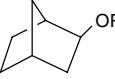
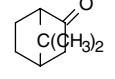
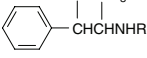
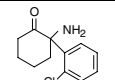
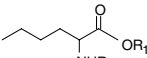
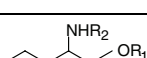
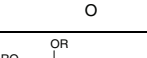
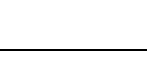
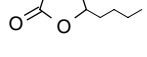
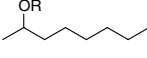
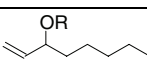
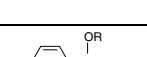
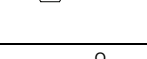
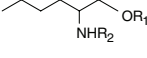
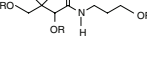
Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
2-(4-Hydroxyphenyl) propionic acid		-CH ₃ -COCF ₃	G-TA	30	130	7.15	1.06	
4-Hydroxyproline		-CH ₃ -COCF ₃	B-TA	40	130	12.73	1.05	
2-Hydroxytetrahydrofuran		-COCF ₃	B-TA	10	100	3.53	1.12	
3-Hydroxytetrahydro-pyran		-COCF ₃	G-TA	10	50	7.14	1.18	
2-Hydroxyvaleric acid, methyl ester			B-DM	30	100	1.8	1.156	6.63
Ibuprofen			B-DM	30	170	14.2	1.092	2.55
Ifosfamide			G-TA	20	180	31.3	1.12	
Ifosfamide metabolite (3-DCE-IFF)			G-TA	20	180	41.5	1.06	
Ifosfamide metabolite (2-DCE-IFF)			G-TA	20	180	32.27	1.08	
1-Indanol		-COCF ₃	B-PH	20	110	3.7	1.05	
2-Iodobutane			G-TA	40	40	4.36	1.08	
α-Ionone			B-DM	30	120	10.7	1.072	2.47
Isoborneol		-COCF ₃	G-TA	10	70	8.68	1.05	
1-(p-Isobutylphenyl)chloro-ethane			G-TA	30	100	24.24	1.02	
Isoleucine		-C ₂ H ₅ -COCF ₃	G-TA	30	110	3.39	1.13	
Isopinocampheol		-COCF ₃	B-PH	10	90	3.0	1.10	
1-Isopropylindan			A-PH	10	90	19.4	1.02	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
Ketamine (free base)			G-TA	20	155	17.8	1.05	
Lactame			G-TA	20	120	16.1	1.04	
Lactic acid, methyl ester		-COCF ₃	G-TA	10	50	6.79	1.47	
Lactonitrile			B-TA	10	100	5.0	1.19	
Leucine		-C ₂ H ₅ -COCF ₃	G-TA	30	110	3.24	1.20	
Leucinol		-COCF ₃	B-TA	10	110	4.33	1.14	
S,R-Limonene			B-DM	30	70	5.5	1.107	1.64
Limonene Oxide			B-TA	10	90	5.39 5.52	1.02 1.04	
Linalool			B-DM	30	75	14.5	1.061	1.97
Lineatin			B-TA	30	95	7.4	1.05	
Lysine		-CH ₃ -COCF ₃	G-TA	30	150	29.4	1.12	
Malic acid		-CH ₃ -COCF ₃	G-BP	20	110	2.62	1.14	
R,S-Mandelic acid			B-DM	30	70	5.6	1.260	4.74
S,R-Mandelic acid, ethyl ester			B-DM	30	130	11.2	1.028	1.27
Mandelonitrile			G-PN	10	105	4.83	1.06	
Menthol			B-DP	30	90	16.7	1.046	1.75
Menthol menthyl acetate		-H -COCF ₃	G-TA	40	85	14.28 15.65	1.04 1.03	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
Mephobarbital			B-DA	10	180	24.5	1.02	
Methionine		-CH ₃ -COCF ₃	G-TA	30	130	6.92	1.11	
Methioninol	$\text{CH}_3\text{SCH}_2\text{CH}(\text{NHR})\text{CH}_2\text{OR}$	-COCF ₃	G-PN	10	135	9.40	1.04	
1-Methoxy-1-phenylethanol			B-DM	30	100	13.6	1.040	1.32
1-Methoxy-1-phenylethanol			B-DP	30	110	9.8	1.032	1.59
1-Methoxy-2-methylpropylene oxide			G-PN	10	35	2.19	1.11	
1-Methoxypropylamine		-COCH ₃	A-DA	10	40	20.5	1.03	
2-Methoxytetrahydrofuran			G-DM	20	30	8.2	1.055	1.10
Methyl phenidate		-COCF ₃	B-DA	30	165	26.8	1.04	
Methyl phenyl sulfoxide		-COCF ₃	G-BP	10	145	8.27	1.31	
<i>trans/cis</i> -β-Methyl styrene oxide			G-PN	30	80	7.90 8.32	1.37 1.20	
α-Methyl-γ-butyrolactone			A-TA	10	110	3.03	1.29	
β-Methyl-γ-valerolactone			B-TA	40	110	11.78	1.06	
4a-Methyl-1,2,3,4,4a,9,10,10a-octahydrophenanthrene			B-PH	10	105	34.3 48.9	1.01 1.03	
4-Methyl-2(3H)-naphaldione			G-BP	10	155	22.5	1.05	
1-Methyl-2-cyclohexen-1-ol			B-PH	30	80	3.2	1.03	
3-Methyl-2-cyclohexen-1-ol			B-PH	30	80	7.9	1.09	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
6-Methyl-2-heptylamine		-COCF ₃	A-PH	10	90	24.0	1.04	
4-Methyl-2-pentanol		-COCF ₃ -H	B-TA	10 10	40 40	1.72 4.97	1.34 1.05	
4-Methyl-2-pentylamine		-COCF ₃	G-DP	20	90	15.1	1.270	8.83
4-Methyl-2-pentylamine		-COCF ₃	B-DM	30	60	6.3	1.061	2.40
3-(3-Methyl-2-pyrrolidinyl) pyridine		-COCF ₃	A-DA	20	140	9.43	1.06 1.07	
2-Methyl-3-pentylamine		-COCF ₃	G-PN	20	55	13.0	1.03	
(2S,3S), (2R,3R)-2-Methyl-3-phenylglycidol		-COCF ₃	G-TA	10	100	13.6	1.06	
2-Methyl-6-methylene-7-octen-4-ol			B-PH	30	80	9.4	1.02	
2-Methylbutyric acid		-CH ₃	G-TA	30	70	0.75	1.09	
2-Methylcyclohexanol		-COCF ₃	B-TA	10	60	3.83 4.61	1.02 1.02	
3-Methylcyclohexanol		-COCF ₃	B-TA	10	65	3.29 4.33	1.05 1.09	
2-Methylcyclohexanone			G-TA	10	80	5.29	1.08	
3-Methylcyclohexanone			B-TA	20	80	4.50	1.15	
4-Methylcyclohexene			B-TA	10	30	1.0	1.03	
2-Methylenevinyl cyclopentane			G-TA	30	30	3.15	3.13	
4-Methylnornicotine		-COCF ₃	B-DA	10	170	13.4	1.05	
2-Methyloctanol	$\text{CH}_3(\text{CH}_2)_5\text{CH}(\text{CH}_3)\text{CH}_2\text{OR}$	-COCF ₃	B-TA	40	70	7.89	1.03	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
2-Methylpentanol			B-DM	30	50	13.4	1.066	2.98
3-Methylpiperidine		-COCF ₃	G-TA	10	100	5.54	1.13	
2-Methylpiperidine		-COCF ₃	G-DP	20	110	8.1	1.162	6.95
α-Methyl-p-tyrosine methyl ester		-COCF ₃	B-DA	30	170-200	10.6	1.02	
2-Methyltetrahydrofuran-3-one			G-PN	10	40	9.01	1.05	
2-Methylvaleric acid		-CH ₃	G-TA	20	50	5.63	1.12	
2-Methylvaleric acid			B-DM	30	80	10.8	1.131	1.48
Mevalonic lactone			B-DM	30	140	9.7	1.057	3.31
1-(α-Naphthyl)ethylamine		-COCF ₃	G-DA	10	150	8.1	1.08	
1-(1-Naphthylethylamine		-COCF ₃	G-PN	20	140	39	1.07	
Naproxen, methyl ester			B-DM	30	140	81.4	1.022	1.15
cis-2-(2-Nitroethyl)-cyclohexanol			B-TA	40	150	10.8	1.06	
trans-2-(2-Nitroethyl)-cyclohexanol		-COCF ₃	G-TA	40	130	11.8	1.07	
1-(p-Nitrophenyl)-2-butanol		-COCF ₃	B-TA	30	150	24.6	1.03	
1-(p-Nitrophenyl)-3-methyl-2-hexanol		-COCF ₃	B-TA	30	150	49.2 51.6	1.07 1.07	
α-(p-Nitrophenyl)ethanol		-COCF ₃ -H	B-TA	30 30	150 155	8.3 15.3	1.11 1.03	
γ-Nonanoic lactone			B-DM	30	130	8.3	1.065	3.16

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
2-Nonanol		-COCF ₃	G-DM	20	60	21.7	1.024	1.30
<i>endo</i> and <i>exo</i> -5-Norbornen-2-ol		-COCF ₃	B-PH	10	60	4.3	1.00 1.10	
<i>exo</i> -Norbornenol		-COCF ₃	B-TA	10	60	13.0	1.02	
Norcamphor			B-DP	30	100	5.9	1.129	5.41
Norephedrine		-COCF ₃	B-DM	30	110	7.9	1.027	1.33
Norketamine (free base)			G-TA	20	155	19.8	1.06	
Norleucine		-C ₂ H ₅ -COCF ₃	G-TA	30	110	4.87	1.19	
Norvaline		-CH ₃ -COCF ₃	G-TA	30	110	2.28	1.60	
1,2-Octanediol		-COCF ₃	G-TA	40	100	3.42	1.08	
γ-Octanoic lactone			B-DP	30	140	7.7	1.057	2.57
2-Octanol		-COCF ₃	G-TA	10	40	20.7	1.15	
1-Octen-3-ol		-COCF ₃	G-TA	30	60	5.4	1.13	
Octopamine		-COCF ₃	B-DA	30	140- 200	19.3	1.01	
Ornithine		-CH ₃ -COCF ₃	B-TA	20	160	17.95	1.08	
Panthenol		-COCF ₃	G-TA	30	140	16.3	1.07	
Pantolactone, methyl ether			B-TA	10	100	9.39	1.15	
1,4-Pentanediol		-COCF ₃	B-TA	10	90	5.23	1.06	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
1,2-Pentanediol		-COCF ₃	G-TA	40	80	1.71	1.22	
2-Pentanol		-COCF ₃	B-TA	10	40	1.56	1.67	
1-Penten-3-ol		-COCH ₃	G-PN	10	60	2.10	1.11	
2-Pentylamine		-COCH ₃	B-DP	30	110	9.1	1.047	2.19
Phenoxy-α-cyano mandelic acid		-CH ₃	G-TA	20	170	24.5	1.07	
Phenyl benzyl sulfoxide			G-TA	20	160	33.3	1.11	
γ-Phenyl-γ-butyrolactone			B-DM	30	150	12.4	1.154	7.96
4-Phenyl-1,3-dioxane			G-TA	10	120	7.36	1.04	
1-Phenyl-2-propanol			A-DA	10	60	18.57	1.02	
S,R-1-Phenyl-3-butylamine		-COCF ₃	B-DP	30	150	14.3	1.149	7.67
R,S-1-Phenyl-3-butylamine		-COCF ₃	B-DM	30	125	10.8	1.040	1.81
endo-trans-5-Phenyl-6-methylbicyclo(2,2,2) oct-2-ene			G-TA	20	100	36.0	1.03	
D,L-Phenylalanine, methyl ester			B-DM	30	130	7.3	1.071	2.32
R,S-1-Phenylethanol			B-DM	30	120	3.1	1.061	1.84
sec-Phenylethyl butyrate			G-TA	20	120	2.27	1.06	
l,d-1-Phenylethylamine		-COCF ₃	G-DP	20	130	12.1	1.155	7.86
d,l-1-Phenylethylamine		-COCF ₃	B-DM	30	110	5.7	1.044	1.72

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
l,d-1-Phenylethylamine		-COCF ₃	G-DM	20	90	18.1	1.051	1.90
Phenylmercuric lactate		-COCF ₃	G-TA	10	**	12.4	1.02	
2-Phenylpropanal			B-DM	30	70	23.9	1.032	1.40
2-Phenylpropionaldehyde			B-TA	10	100	3.92	1.01	
1-Phenylpropylene oxide			G-TA	30	130	2.30	1.10	
2-Phenylpyrrolidine		-COCF ₃	B-DA	10	140	16.4	1.03	
β-Phenylvalerolactone			B-TA	20	160	7.48	1.05	
d,l-α-Pinene			G-DP	20	50	6.7	1.048	1.62
l,d-β-Pinene			G-DP	20	70	4.5	1.094	3.33
l,d-α-Pinene			B-DM	30	35	10.2	1.085	3.51
d,l-β-Pinene			B-DM	30	50	15.1	1.072	3.08
Proline, methyl ester		-COCF ₃	G-DP	20	140	5.6	1.112	4.92
L,D-Proline, methyl ester			B-DM	30	90	3.0	1.069	2.7
1,2-Propanediol		-COCF ₃	G-TA	40	80	0.86	1.52	
1,2-Propylene glycol t-butyl ether		-COCF ₃	B-TA	40	40	9.1	1.08	
1,2-Propylene glycol t-butyl ether			B-TA	40	40	9.74	1.03	
Propylene oxide			A-TA	20	25	0.59	1.15	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
1-Propylindan			A-PH	10	90	19.4	1.02	
2-(2-Propynyloxy)tetra-hydrofuran			B-TA	10	70	7.44	1.06	
Prozac (Fluoxetine)		-COCF ₃	B-DA	20	190	31.7	1.04	
Pseudoephedrine		-COCF ₃	G-PN	10	130	8.79	1.07	
N-α-(3-Pyridyl)ethylethylamine		-COCF ₃	A-DA	20	160	3.04	1.07	
2-(2-Pyridyl)pyrrolidine		-COCF ₃	B-DA	10	140	14.5	1.02	
2-(4-Pyridyl)pyrrolidine		-COCF ₃	B-DA	10	170	9.7	1.03	
2-Pyridyl-3-methylpyrrolidine		-COCF ₃	B-PH	20	140-180	10.8 1.00	1.01 1.00	
Serine, methyl ester		-COCF ₃	B-DP	30	110	14.8	1.126	6.05
Silvex, methyl ester			G-TA	10	145	20.1	1.02	
Solketal		-COCF ₃	G-TA	10	60	2.43	1.59	
trans-Stilbene oxide			G-TA	10	140	20.0	1.02	
Styrene Oxide			B-DM	30	120	11.2	1.040	2.21
Tartaric acid		-C ₂ H ₅	G-TA	30	150	3.61	1.14	
Tartaric acid, diisopropyl ester		-COCF ₃	B-TA	10	90	8.57	1.07	
D,L-Tartaric acid, dimethyl ester			B-DM G-DM	30 20	140 120	3.7 9.6	1.268 1.093	6.35 1.83
Terpinen-4-ol			A-DA	10	40	18.14	1.05	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
1,2,3,4-Tetrahydro-1-naphthol		-COCH ₃	A-DA	20	120	2.04	1.07	
1,2,3,4-Tetrahydro-1-naphthylamine		-COCF ₃	G-PN	20	120	32	1.03	
1,2,3,4-Tetrahydro-1-naphthylamine		-COCF ₃	B-DM	30	120	3.9	1.057	2.89
Tetrahydro-2-(2-propynyloxy)-2H-pyran			B-DM	30	80	7.7	1.098	4.32
Tetrahydro-2-furoic acid, methyl ester			B-DM	30	90	3.5	1.129	4.52
Tetrahydro-3-furoic acid, methyl ester			B-DP	30	80	9.0	1.082	4.04
Threonine		-C ₂ H ₅ -COCF ₃	G-TA	30	100	4.21	1.10	
Threonine, methyl ester		-COCF ₃	G-DP	20	120	8.3	1.061	1.82
Tiazina			B-DM	30	150	10.8	1.068	3.10
α-(Trichloromethyl)benzyl acetate			B-DA	30	150	10.5	1.04	
N-(Trifluoromethyl) normocotine			B-DA	20	140	5.2	1.01	
α-(Trifluoromethyl)benzyl alcohol			G-DM	20	100	16.1	1.024	0.9
1,1,3-Trimethyl-2-(3-methyloctyl) cyclohexane			B-PH	10	85	40.5	Ref.8	
2,6,10-Trimethyl-7-(3-methylbutyl)dodecane			A-PH	10	100	70.4	1.01	
1,5,8-Trimethyltetralin			B-PH	10	120	10.3	1.05	
m-Tyrosine		-C ₂ H ₅ -COCF ₃	B-TA	20	160	20.9	1.03	
α-Tyrosine		-CH ₃ -COCF ₃	B-TA	40	150	17.0	1.03	

Compound	Structure	R-	Column	L (m)	T°C	k' ₁	α	Rs
<i>p</i> -Tyrosine		-C ₂ H ₅ -COCF ₃	B-TA	20	145	23.6	1.04	
Undecanoic-γ-butyrolactone			B-DP	30	160	12.7	1.032	1.78
β-Undecanolactone			B-TA	20	145	6.16	1.15	
D,L-Valine, methyl ester		-COCF ₃	G-BP	20	100	3.4	1.15	
Verbanone			B-TA	30	130	4.24	1.08	
(1)- <i>cis</i> -Verbenol			B-PH	30	105	5.3	1.02	
Vigabatrin		-COCF ₃	B-TA	30	140	8.8	1.03	

*30°→140° @ 3°C/min.

**50°→90°C @ 1°C/min.

AVAILABILITY

CHIRALDEX GC Columns

Phase Type	0.25mm				
	10m	20m	30m	40m	50m
CHIRALDEX A-PH	71001AST	71002AST	71003AST	71004AST	71005AST
CHIRALDEX B-PH	71021AST	71022AST	71023AST	71024AST	71025AST
CHIRALDEX A-DA	72001AST	72002AST	72003AST	72004AST	72005AST
CHIRALDEX B-DA	72021AST	72022AST	72023AST	72024AST	72025AST
CHIRALDEX G-DA	72031AST	72032AST	72033AST	72034AST	72035AST
CHIRALDEX A-TA	73001AST	73002AST	73003AST	73004AST	73005AST
CHIRALDEX B-TA	73021AST	73022AST	73023AST	73024AST	73025AST
CHIRALDEX G-TA	73031AST	73032AST	73033AST	73034AST	73035AST
CHIRALDEX G-PN	74031AST	74032AST	74033AST	74034AST	74035AST
CHIRALDEX G-BP	75031AST	75032AST	75033AST	75034AST	75035AST
CHIRALDEX B-DM	77021AST	77022AST	77023AST	77024AST	77025AST
CHIRALDEX G-DM	77031AST	77032AST	77033AST	77034AST	77035AST
CHIRALDEX B-DP	78021AST	78022AST	78023AST	78024AST	78025AST
CHIRALDEX G-DP	78031AST	78032AST	78033AST	78034AST	78035AST

CHIRALDEX B-PM

Phase Type	0.25mm	
	30m	50m
CHIRALDEX B-PM	76023AST	76025AST

CHIRALDEX GC Kits

CHIRALDEX 3 column kits include 1 each of three columns of the same ID and length at an economical price. We recommend as a starter kit for the broadest range of applications a CHIRALDEX-30 Kit including 1 each of B-DA, B-DM, G-TA.

	Length	0.25mm
CHIRALDEX-10 Kit	10m	71010AST
CHIRALDEX-20 Kit	20m	71020AST
CHIRALDEX-30 Kit	30m	71030AST
CHIRALDEX-40 Kit	40m	71040AST
CHIRALDEX-50 Kit	50m	71050AST

CHIRALDEX GC Test Mixtures

Cat. No.	Description	Size	Conc.	CHIRALDEX Column
90001AST	Tetrahydro-2-(2-propynyloxy)-2H-pyran	1 ml	5mg/ml	A-DA
90002AST	2-Methyl piperidine (<i>N</i> -TFA)	1 ml	5mg/ml	G-BP,G-TA
90003AST	2-Aminoheptane (<i>N</i> -TFA)	1 ml	5mg/ml	B-PH,G-PN,A-TA,G-DP,G-DM
90004AST	1-Aminoindan (<i>N</i> -TFA)	1 ml	5mg/ml	B-DA,G-DA
90005AST	2-(Bromomethyl) tetrahydro-2H-pyran	1 ml	5mg/ml	B-TA,B-DP
90006AST	3,4-Dihydro-2-ethoxy-2H-pyran	1 ml	5mg/ml	A-PH
90007AST	1-Phenyl-1-ethanol	1 ml	5mg/ml	B-DM,B-PM

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