

# Astec® CHIRALDEX® and Supelco® DEX™ Chiral GC Columns

The Widest Variety of Derivatized Cyclodextrins



Stable, Derivatized Cyclodextrin-  
based Chiral GC Phases

Twenty-four Different Phases

Surface-interaction as well as  
Inclusion-dominant Phases

# Chiral GC Columns from Sigma-Aldrich®

Gas chromatography (GC) columns that employ a chiral stationary phase (CSP) are suitable for enantiomer separations. We offer two chiral GC column lines, Astec® CHIRALDEX® and Supelco® DEX™. Both product lines are based on the cyclodextrin molecule and exhibit complementary selectivity. All columns are manufactured to deliver high resolution and analyte response, low bleed, and long column life. The Astec line is unique in including phases with surface interaction as the sole mechanism, which result in very powerful CSPs.

## Cyclodextrins as GC CSPs

Cyclodextrins (CDs) are cyclic, chiral, toroid macromolecules composed of six or more D(+)-glucose residues bonded through  $\alpha(1-4)$  glycosidic linkages. CDs are classified by the number of glucose units they contain;  $\alpha$ -CDs contain six units (cyclohexylamylose),  $\beta$ -CDs contain seven units (cycloheptylamylose) and  $\gamma$ -CDs contain eight units (cyclooctylamylose). As shown in **Table 1**, the size and number of stereogenic centers of the cavity increases with increasing number of glucose units. **Figure 1** shows the theoretical structure and dimensions of the CD toroid. The mouth of the torus-shaped CD molecule has a larger circumference than the base and is linked to secondary hydroxyl groups of the C2 and C3 atoms of each glucose unit. The primary hydroxyl groups are located at the base of the torus, on the C6 atoms. All hydroxyl groups, whether at the 2, 3 or 6 position of each residue, can be selectively modified with a derivative to impart unique selectivities. Without derivatization, no enantiomeric selectivity is exhibited in GC.

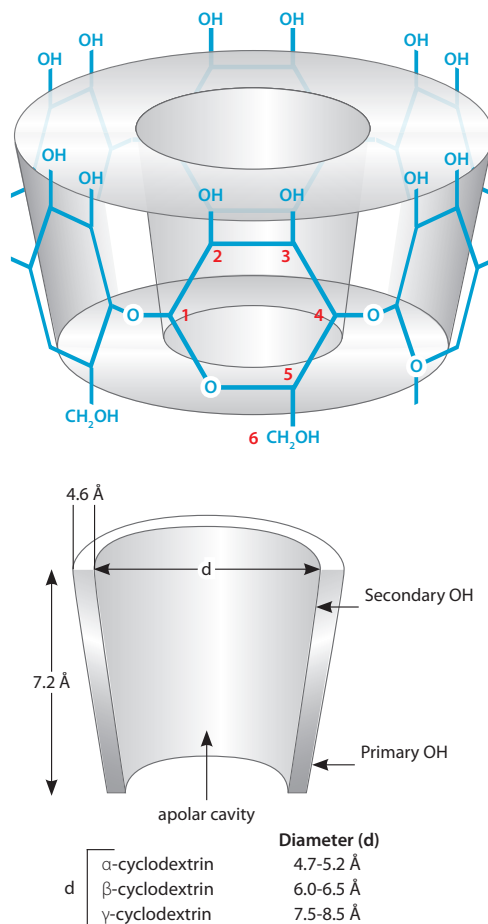
**Table 1. Properties of Cyclodextrins**

Cyclodextrin	Chemical Name	Glucose Units	Cavity Size	Stereogenic Centers
Alpha ( $\alpha$ )	Cyclohexylamylose	6	4.7 – 5.2 Å	30
Beta ( $\beta$ )	Cycloheptylamylose	7	6.0 – 6.5 Å	35
Gamma ( $\gamma$ )	Cyclooctylamylose	8	7.5 – 8.5 Å	40

Both the architecture and chemistry of cyclodextrins contribute to enantiomer separations. The toroidal cyclodextrin structure has a hydrophilic exterior surface resulting from the 2-, 3- and 6-position hydroxyl (OH) groups. The interior cyclodextrin cavity is composed of the glucoside oxygens and methylene hydrogens, which gives it a non-polar (hydrophobic) character. Chemical interactions that lead to chiral separations occur on both the exterior and interior surfaces of the cyclodextrin toroid.

Cyclodextrins have been widely applied in the separation sciences since the early 1980's.<sup>1</sup> Because derivatized CDs are stable, high boiling liquids, they make effective chiral stationary phases (CSPs) for GC.<sup>2-5</sup>

**Figure 1. Theoretical Structure of a Cyclodextrin Molecule**



## The Astec CHIRALDEX and Supelco DEX Lines

For chiral GC, Sigma-Aldrich offers two complementary lines of CD-based CSPs:

- Astec CHIRALDEX
- Supelco DEX

Combined, the two lines comprise a total of 24 complementary phase chemistries. Both conventional derivatives (e.g. permethyl) and complex derivatives (e.g. trifluoroacetyl) impart a broad range of selectivities and perform many enantiomeric separations. Selectivity is a function of the CD type ( $\alpha$ ,  $\beta$  or  $\gamma$ ), the type of derivative, the degree of derivatization, the position of the derivative on the CD, whether the derivatized CD is used neat or doped into a polysiloxane and, if doped, at what percentage. The wide range of chemistries, column dimensions and maximum temperatures for all phases that we carry are shown in the Product Listing on pages 7-8.

# How do Cyclodextrin-based CSPs Separate Enantiomers?

The most important consideration for retention and chiral recognition is proper fit of the analyte into the cyclodextrin cavity. This fit is a function of both molecular size and shape of the analyte, relative to the cyclodextrin cavity. Thus, there are two basic mechanisms at play in chiral separations on cyclodextrins; those that occur on the inside cavity surface (inclusion complexing, **Figure 2**), and those that occur on the outside surface (surface interactions, **Figure 3**) of the cyclodextrin toroid. It is important to note that derivatization of the cyclodextrin is necessary for enantiomeric selectivity in GC mode.

**Figure 2. Inclusion Interaction Schematic**



Representation of the inclusion complexing mechanism of an analyte into the cyclodextrin cavity.

**Figure 3. Surface Interaction Schematic**



Representation of the surface interaction mechanism of an analyte with the cyclodextrin.

## Group 1: Surface Interactions, Complex Derivatives

Sigma-Aldrich is the only supplier of complex derivatives for chiral GC. Because the predominant mechanism of retention for phases in this group is based on surface interaction, the  $\gamma$ -cyclodextrin, with eight glucose molecules, has been shown to be the most useful. Compared to  $\alpha$ - and  $\beta$ -cyclodextrins, the greater number of glucose molecules in a  $\gamma$ -cyclodextrin results in the greater number of hydroxyl functional groups available for derivatization. High derivative concentration is beneficial for maximizing surface interactions.

**Astec CHIRALDEX G-TA** is the first choice in this group. This phase has been shown to be the most broadly selective phase for the pharmaceutical industry, especially in the analysis of chiral intermediates and drug studies in various stages of clinical trials. Separations occur without the inclusion mechanism, and are typically faster and more efficient than most other CSPs. This phase does not contain a polysiloxane polymer carrier and, therefore, there are no deleterious effects at low temperatures. The ability of this phase to separate parent drug enantiomers and their metabolites has proven quite beneficial.

A modified version of the Astec CHIRALDEX G-TA is the **Astec CHIRALDEX G-PN**. It functions like the Astec CHIRALDEX G-TA, but shows higher selectivity toward certain amines (amphetamine, methamphetamine). This phase is more stable to moisture than the Astec CHIRALDEX G-TA.

The **Astec CHIRALDEX G-DP** phase was introduced to enhance selectivity for both aliphatic and aromatic amines in addition to aliphatic and some aromatic esters. This phase is especially useful for polar racemates. This phase demonstrates better hydrolytic and thermal stability than the Astec CHIRALDEX G-TA.

The **Astec CHIRALDEX G-BP** phase can be used as a general purpose column, but it is especially useful for amino acids.

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

## Group 2: Combined Surface and Inclusion Interactions, Simple Derivatives

$\beta$ -cyclodextrin has shown the greatest applicability for phases with these types of interactions. **Astec CHIRALDEX B-DM** is the recommended column in this category. The **Supelco  $\beta$ -DEX 325** is similar in both chemistry and use to the Astec CHIRALDEX B-DM phase, the main difference being the concentration of the dimethyl-derivatized cyclodextrin that is doped into the polysiloxane carrier.

The **Supelco  $\beta$ -DEX 225** is a modified form of the Supelco  $\beta$ -DEX 325 phase, employing acetyl derivatives at the 2,3-positions instead of more traditional methyl derivatives.

This group also includes the popular dimethyl and permethyl derivatives, and includes **Astec CHIRALDEX B-PM**, **Supelco  $\beta$ -DEX 110**, and **Supelco  $\beta$ -DEX 120** phases. They are recommended as general purpose columns for the separation of a wide variety of compounds and are especially useful for the analysis of alcohols and diols in their underivatized form, and for analytes with polar groups (such as tertiary amines). The main difference between these three phases is the concentration of the permethyl-derivatized cyclodextrin which is doped into the polysiloxane carrier.

## Group 3: Inclusion Interactions

The third group of phases relies on inclusion interactions for retention mechanism. The fact that there are three different size cyclodextrins ( $\alpha$ ,  $\beta$  and  $\gamma$ ) allows for separation of a wide variety of different size analytes. **Astec CHIRALDEX B-DA** demonstrates the strongest size selectivity. This phase requires analytes to minimally contain two-ring structures, one of which is unsaturated (aromatic). The mechanism of this phase is strongly dependent on the inclusion mechanism and is able to differentiate changes in the base structure. Because the Astec CHIRALDEX B-DA most effectively separates multi-ring analytes, analysis temperatures are often higher than 150 °C. A key application area for this phase is the fingerprinting of raw materials and identifying structural differences.

**Astec CHIRALDEX 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 saturated bicyclics. This phase often shows a reversal of elution order (enantioreversal) compared to the Astec CHIRALDEX B-DA phase.

## Recommended Chiral GC Six-Column Screening Set

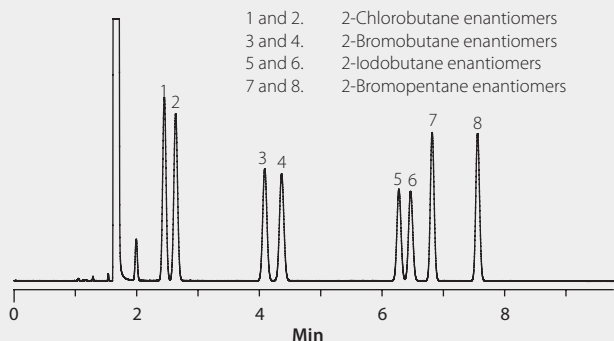
If you can't have all 24 chemistries in your collection, based on our experience with leveraging the selectivity differences among the Astec® CHIRALDEX® and Supelco® DEX™ lines, we recommend everyone performing chiral GC has the following columns. Columns 1, 3 and 5 are supplied in our Astec CHIRALDEX kit (71030AST).

### 1. Astec CHIRALDEX G-TA (Trifluoroacetyl, 2,6-di-O-pentyl-3-trifluoroacetyl)

The Astec CHIRALDEX G-TA separates the greatest number of enantiomers, often with high enantioselectivity. It has excellent 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.<sup>4</sup> Figure 4 shows the separation of halogen-containing enantiomers.

**Figure 4. 2-Halohydrocarbon Enantiomers on Astec CHIRALDEX G-TA**

column: Astec CHIRALDEX G-TA, 30 m x 0.25 mm I.D., 0.12 µm (73033AST)  
 oven: 30 °C (3 min.), 5 °C/min. to 70 °C  
 inj.: 250 °C  
 det.: FID, 250 °C  
 carrier gas: helium, 30 psi  
 injection: 1 µL, 80:1 split  
 sample: 2.5 mg/mL of each compound

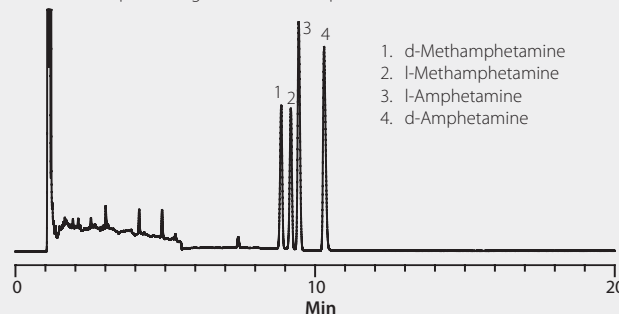


### 2. Astec CHIRALDEX G-PN (Propionyl, 2,6-di-O-pentyl-3-propionyl)

The Astec CHIRALDEX G-PN functions like the Astec CHIRALDEX G-TA but shows higher selectivity toward certain amines (amphetamine, methamphetamine), lactones, and epoxides. This column is also very selective for aromatic amines (amphetamine/methamphetamine shown in Figure 5) and >C6 alcohols.

**Figure 5. Amphetamine and Methamphetamine Enantiomers (N-Trifluoroacetyl Derivatives) on Astec CHIRALDEX G-PN**

column: Astec CHIRALDEX G-PN, 30 m x 0.25 mm I.D., 0.12 µm (74033AST)  
 oven: 130 °C  
 inj.: 250 °C  
 det.: FID, 250 °C  
 carrier gas: helium, 30 psi  
 injection: 1 µL, 80:1 split  
 sample: 3 mg/mL of each compound in methanol

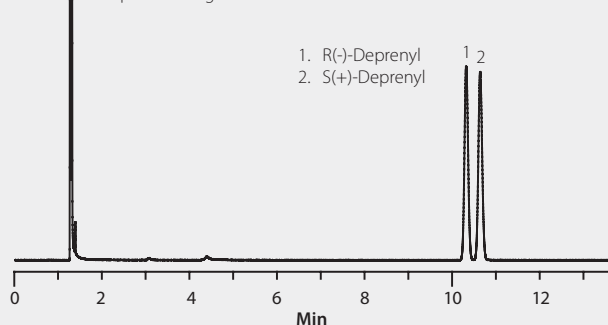


### 3. Astec CHIRALDEX B-DM (Dimethyl, 2,3-di-O-methyl-6-t-butyl silyl)

Through special derivatization techniques, the concentration of the cyclodextrin in the Astec CHIRALDEX B-DM has been substantially increased in the polysiloxane carrier relative to other dimethylated phases. This column separates the widest variety of different structural types. It is a general purpose column able to perform most of the separations done on a β-permethyl phase, but with higher resolution. 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. Figure 6 shows an example of an Astec CHIRALDEX B-DM separation.

**Figure 6. Deprenyl (Selegiline) Enantiomers on Astec CHIRALDEX B-DM**

column: Astec CHIRALDEX B-DM, 30 m x 0.25 mm I.D., 0.12 µm (77023AST)  
 oven: 130 °C  
 inj.: 250 °C  
 det.: FID, 250 °C  
 carrier gas: helium, 24 psi  
 injection: 1 µL, 80:1 split  
 sample: 2.5 mg/mL



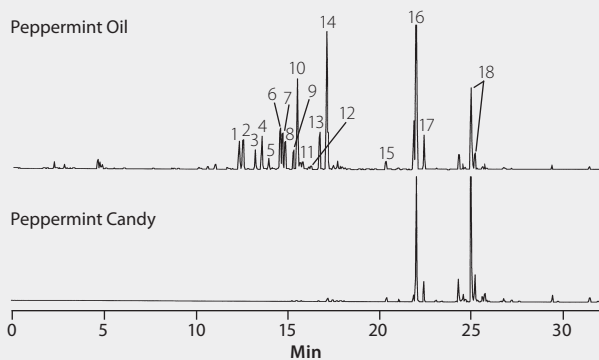
#### 4. Supelco $\beta$ -DEX 110 or 120 (Permethyl, 2,3,6-tri-O-methyl)

The Supelco  $\beta$ -DEX 110 and 120 are permethylated  $\beta$ -CD phases. They are recommended for the enantiomeric separation of a wide range of chiral compounds (ketones, esters, alkanes, alkenes, alcohols, acids, ethers, etc.), and are especially effective for saturated analytes with minimal functionality, saturated cyclics, and saturated bicyclics. The 10% ( $\beta$ -DEX 110) and 20% ( $\beta$ -DEX 120)  $\beta$ -cyclodextrin content alters the elution order while maintaining similar enantioselectivity. **Figure 7** shows an example of a Supelco  $\beta$ -DEX 120 separation.

#### Figure 7. Peppermint Oil and Peppermint Candy on $\beta$ -DEX 120 with SPME

sample: 0.5 g peppermint oil or crushed candy in 7 mL vial  
 SPME fiber: 100  $\mu$ m polydimethylsiloxane (57300-U) manual sampling  
 extraction: headspace, 30  $^{\circ}$ C, 3 min.  
 desorption: 1 min., 250  $^{\circ}$ C  
 column:  $\beta$ -DEX 120, 30 m x 0.25 mm I.D., 0.25  $\mu$ m (24304)  
 oven: 40  $^{\circ}$ C (2 min) to 220  $^{\circ}$ C at 4  $^{\circ}$ C/min  
 inj.: 250  $^{\circ}$ C  
 det.: FID, 300  $^{\circ}$ C  
 carrier gas: helium, 35 cm/sec  
 injection: 100:1 split

- |                          |                                      |                               |
|--------------------------|--------------------------------------|-------------------------------|
| 1. (-)- $\alpha$ -Pinene | 7. (-)- $\beta$ -Pinene              | 13. $\gamma$ -Terpinene       |
| 2. (+)- $\alpha$ -Pinene | 8. $\alpha$ -Terpinene               | 14. Cineole                   |
| 3. $\beta$ -Myrcene      | 9. 3-Carene                          | 15. Menthone                  |
| 4. Sabinene              | 10. (-)-Limonene                     | 16. (+)-Menthol               |
| 5. (-)-Camphene          | 11. (+)-Limonene                     | 17. (-)-Menthol               |
| 6. (+)- $\beta$ -Pinene  | 12. ( $\pm$ )- $\beta$ -Phellandrene | 18. ( $\pm$ )-Menthyl acetate |

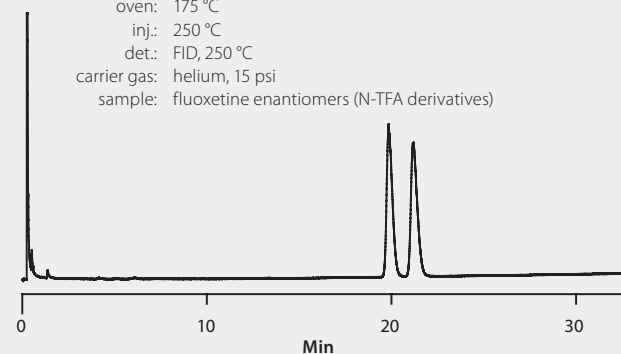


#### 5. Astec CHIRALDEX B-DA (Dialkyl, 2,6-di-O-pentyl-3-methoxy)

The Astec CHIRALDEX B-DA is best suited for larger multi-ring structures. It often shows enantioreversal of separations done on a permethylated phase. This column requires minimally two-ring structures, one of which is unsaturated (aromatic). Examples are fluoxetine, methylphenidate and chlorpheniramine. Inclusion complexation, or proper fit, between the analyte and cyclodextrin cavity is the dominant enantioselectivity mechanism for the Astec CHIRALDEX DA series of columns. The size of the includable group will dictate the choice of  $\alpha$ ,  $\beta$  or  $\gamma$  cyclodextrin. Since the Astec CHIRALDEX DA series of columns most effectively separate multi-ring analytes, analysis temperatures are often higher than 150  $^{\circ}$ C. Enantioselectivity has been observed at temperatures near 200  $^{\circ}$ C, such as with the example shown in **Figure 8**.

#### Figure 8. Fluoxetine (Prozac<sup>®</sup>) Enantiomers (TFA Derivative) on Astec CHIRALDEX B-DA

column: Astec CHIRALDEX B-DA, 10 m x 0.25 mm I.D., 0.12  $\mu$ m (72021AST)  
 oven: 175  $^{\circ}$ C  
 inj.: 250  $^{\circ}$ C  
 det.: FID, 250  $^{\circ}$ C  
 carrier gas: helium, 15 psi  
 sample: fluoxetine enantiomers (N-TFA derivatives)

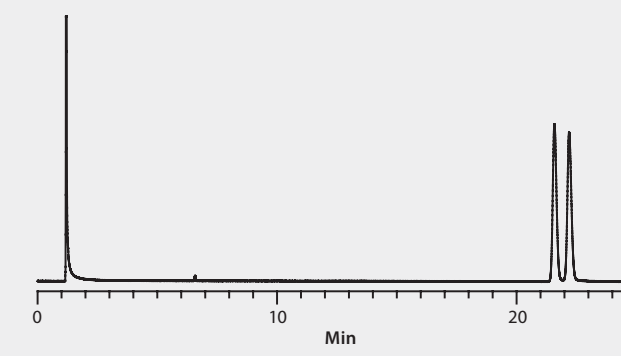


#### 6. Astec CHIRALDEX B-PH (Hydroxypropyl, (S)-2-hydroxypropyl methyl ether)

The Astec CHIRALDEX B-PH shows at least some selectivity to a great variety of analytes, but is especially effective for saturated analytes with minimal functionality (e.g. **Figure 9**), saturated cyclics and bicyclics. It often provides a reversal of elution order (enantioreversal) relative to the Astec CHIRALDEX B-DA phase. The Astec CHIRALDEX B-PH shows less of a necessity for inclusion complexation for chiral recognition than Astec CHIRALDEX DA columns.<sup>6</sup>

#### Figure 9. *trans*-1,2-Cyclohexanediol Enantiomers on Astec CHIRALDEX B-PH

column: Astec CHIRALDEX B-PH, 30 m x 0.25 mm I.D., 0.12  $\mu$ m (71023AST)  
 oven: 120  $^{\circ}$ C  
 inj.: 250  $^{\circ}$ C  
 det.: FID, 250  $^{\circ}$ C  
 carrier gas: helium, 30 psi  
 injection: 1  $\mu$ L, 80:1 split  
 sample: 2.5 mg/mL



#### References

1. Armstrong, D. W., DeMond W. *Journal of Chromatographic Science*, **1984**, 22, 411-415.
2. Armstrong, D.W., Tang, Y., Zukowski, Y. *Anal. Chem.* **1991**, 63(24), 2858-2861.
3. Armstrong, D.W., Jin, H. L., H. L. *J. Chromatogr.* **1990**, 502, 154-159.
4. Armstrong, D.W., Li, W., Pitha, J. *Anal. Chem.* **1990**, 62, 214-217.
5. Li, W., Jin, H. L., Armstrong, D.W. *J. Chromatogr.* **1990**, 509, 303-324.
6. Harada, K., *Chem. Rev.* **1998**, 98, 1803-1827.

# Chiral GC Column Selection Guidelines

Although it is difficult to firmly predict enantiomeric selectivity based on molecular structure and functionality, some generalizations can narrow down the column choice. **Table 2** provides general guidance on the phase most likely to perform a desired separation. To use

this table, find the analyte, compound class, or functionality of the enantiomer(s) in the listing above the table. Then, simply locate the correct column in the table, and follow down to identify appropriate column choice(s).

**Table 2. Chiral GC Column Suggestions by Application**

- Oxygen containing analytes in the form of alcohols, ketones, acids, aldehydes, and lactones; halogenated compounds
- Lactones and aromatic amines; epoxides; styrene oxide
- Aliphatic and aromatic amines; aliphatic and some aromatic esters; polar racemates
- Amino acids; amines; furans
- Aliphatic, olefinic, and aromatic enantiomers
- Terpenes and tertiary amines
- Heterocyclic amines
- Xylenes; menthols; cresols; substituted phenols; substituted benzenes; epoxide enantiomers
- Acids; alcohols; amines; diols; esters; ethers; halohydrocarbons; hydrocarbons; ketones; positional isomers; silanes; terpenes; terpineols
- $\alpha$ -BHC; carvone; carboxylic acids; methamphetamine

		Type of Analyte (see above)									
		1	2	3	4	5	6	7	8	9	10
<b>By Derivative</b>											
Astec® CHIRALDEX® TA	Trifluoroacetyl	●									
Astec CHIRALDEX PN	Propionyl		●								
Astec CHIRALDEX DP	Dipropionyl			●							
Astec CHIRALDEX BP	Butyryl				●						
Astec CHIRALDEX DM	Dimethyl					●					
Supelco® DEX™ 325	Dimethyl					●					
Supelco DEX 225	Diacetyl					●					
Astec CHIRALDEX PM	Permethylated						●				
Supelco DEX 110	Permethylated						●				
Supelco DEX 120	Permethylated						●				
Astec CHIRALDEX DA	Dialkyl							●			
Astec CHIRALDEX PH	S-Hydroxypropyl					●					
<b>By Cyclodextrin</b>											
$\alpha$ -Cyclodextrin									●		
$\beta$ -Cyclodextrin										●	
$\gamma$ -Cyclodextrin											●

The Astec CHIRALDEX product line includes several CSPs which are complex derivatives. No other manufacture provides these types of CSPs, so many users of chiral GC columns may be unfamiliar with

their performance characteristics. **Table 3** displays some additional guidelines specific for Astec CHIRALDEX columns.

**Table 3. Typical Compounds Separated on Astec CHIRALDEX Phases**

Astec CHIRALDEX Phase	Typical Compounds Separated
A-TA	Smaller epoxides and alcohols, amino alcohols, amino alkanes, diols.
B-TA	Broad range alkyl alcohols, halo acid esters, amino alkanes, amino acid derivatives, halocycloalkanes, certain lactones, diols, alkyl halides, furan and pyran derivatives.
G-TA	Chiral alcohols, diols, polyols, hydrocarbons, lactones, amino alcohols, halocarboxylic acid esters, homologous series, furan, pyran derivatives, epoxides, glycidyl analogs and haloepihydrins.
G-PN	Epoxides, secondary amines, higher alcohols >C4, esters, lactones, diols. Good alternate for G-TA but not as broad selectivity.
B-DP	Acid esters, amines, lactones, alcohols, some diols.
G-DP	Acid esters (long chain), fused ring/bulky amines, lactones.
G-BP	Amino acids, certain primary amines and furans.
B-DM	Aromatic alcohols, short chain alcohols ≤C6, amines, amino alcohols, epoxides, cyclic ketones, diols, aromatic acids and esters (many of these underivatized).
G-DM	Aromatic alcohols >C5, polar amines and bulky amines. Aromatic amines, halogenated aliphatic acids and derivatized long chain diols.
B-PM	Acids, alcohols, barbital, diols, epoxides, esters, hydrocarbons, ketones, lactones and terpenes.
A-DA	Smaller cyclic and aromatic amines, alcohols and epoxides.
B-DA	Heterocyclics, some multi-ring lactones, aromatic amines, sugars, certain amino acid derivatives, bicyclics, epoxides.
G-DA	Aromatic amines containing two or more rings, large cyclic diols, some heterocyclics, multi-ring compounds or compounds with bulky substituents.
B-PH	Most structural types of compounds including linear and cyclic amines and alcohols, lactones, amino alcohols, sugars, bicyclics, epoxides, haloalkanes, aromatic and cyclic hydrocarbons, and more. Very useful for hydrocarbons.

## Chiral Services

Choosing the correct CSP and/or developing a chiral method is difficult. Our Chiral Services group at our Pharmorphix (UK) site can help. The list of services offered include:

- Column screening for chiral HPLC and GC separations
- Chiral method optimization and loading studies for preparative scale
- Small scale enantiomeric purification
- Small scale asymmetric synthesis
- Large scale crystallization
- Simulated moving bed (SMB) separations
- Production of enantiomerically pure active pharmaceutical ingredients (APIs)

GC column screening involves exploration of different GC chiral phases. Samples that require derivatization are verified by GC-MS. To establish the most efficient method, an optimization study can be conducted as an outcome of the initial column screening. Method optimization may vary, depending on the intended use of the method. All information relating to the work done is kept confidential, and a nondisclosure agreement can be arranged upon request.

Contact us at [chiral@sial.com](mailto:chiral@sial.com), the regional office in your territory, or visit [sigma-aldrich.com/chiral](http://sigma-aldrich.com/chiral)

# Product Listing

## Method Development Kits

These kits provide the necessary columns to perform most chiral separations and run mechanistic studies. They are also offered at very attractive prices.

The **Astec® CHIRALDEX® Kit** provides columns for the broadest range of applications. Each kit contains 1 each of the three most effective Astec CHIRALDEX columns (G-TA, B-DM, and B-DA) in the specified dimensions. The G-TA separates the greatest number of enantiomers, often with high enantioselectivity. The B-DM separates the widest variety of different structural types. The B-DA is best suited for ring structures. Eighty-five percent of analytes which exhibit enantioselectivity on cyclodextrin based chiral stationary phases will give enantioselectivity on one of these phases. The fact that three phases offer such a wide variety of selectivities and analyzable solutes underlies the rationale behind the composition of these kits.

Description	Cat. No.
<b>Astec CHIRALDEX Kit</b>	
CHIRALDEX G-TA, B-DM, B-DA (each 30 m x 0.25 mm I.D., 0.12 µm)	<b>71030AST</b>

The **Supelco® DEX™ Kit I** is valuable for studying the effect of CD size on a separation. All three columns in the kit, α-DEX 120, β-DEX 120 and γ-DEX 120, have the same permethylated derivative, but on different size CDs. One application is to confirm enantiomer identification by monitoring elution order changes (enantioreversal) from one column to another.

Description	Cat. No.
<b>Supelco DEX Kit I</b>	
α-DEX 120, β-DEX 120, γ-DEX 120 (each 30 m x 0.25 mm I.D., 0.25 µm)	<b>24340</b>

The **Supelco DEX Kit II** contains four columns, one each of β-DEX 325, β-DEX 225, γ-DEX 225 and β-DEX 120, each 30 m x 0.25 mm I.D., 0.25 µm. Combining this kit with Supelco DEX Kit I spans the full range of Supelco DEX column enantioselectivity.

Description	Cat. No.
<b>Supelco DEX Kit II</b>	
β-DEX 325, β-DEX 225, γ-DEX 225, β-DEX 120 (each 30 m x 0.25 mm I.D., 0.25 µm)	<b>24328-U</b>

## Capillary Columns for Chiral GC\*

Phase	CD Type	Derivative	Full Description	Length (m)	I.D. (mm)	Film (µm)	Min. Temp.	Max. Temp. (Isothermal)	Max. Temp. (Progr.)	Cat. No.
<b>Surface Interactions, Complex Derivatives</b>										
Astec CHIRALDEX A-TA	α	Trifluoroacetyl	2,6-di-O-pentyl-3-trifluoroacetyl derivative of α-cyclodextrin	20	0.25	0.12	-10 °C	180 °C	180 °C	<b>73002AST</b>
				30	0.25	0.12	-10 °C	180 °C	180 °C	<b>73003AST</b>
				40	0.25	0.12	-10 °C	180 °C	180 °C	<b>73004AST</b>
				50	0.25	0.12	-10 °C	180 °C	180 °C	<b>73005AST</b>
Astec CHIRALDEX B-TA	β	Trifluoroacetyl	2,6-di-O-pentyl-3-trifluoroacetyl derivative of β-cyclodextrin	30	0.25	0.12	-10 °C	180 °C	180 °C	<b>73023AST</b>
				40	0.25	0.12	-10 °C	180 °C	180 °C	<b>73024AST</b>
Astec CHIRALDEX G-TA	γ	Trifluoroacetyl	2,6-di-O-pentyl-3-trifluoroacetyl derivative of γ-cyclodextrin	10	0.25	0.12	-10 °C	180 °C	180 °C	<b>73031AST</b>
				20	0.25	0.12	-10 °C	180 °C	180 °C	<b>73032AST</b>
				30	0.25	0.12	-10 °C	180 °C	180 °C	<b>73033AST</b>
				40	0.25	0.12	-10 °C	180 °C	180 °C	<b>73034AST</b>
				50	0.25	0.12	-10 °C	180 °C	180 °C	<b>73035AST</b>
Astec CHIRALDEX G-PN	γ	Propionyl	2,6-di-O-pentyl-3-propionyl derivative of γ-cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	<b>74033AST</b>
Astec CHIRALDEX B-DP	β	Dipropionyl	2,3-di-O-propionyl-6- <i>t</i> -butyl silyl derivative of β-cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	<b>78023AST</b>
Astec CHIRALDEX G-DP	γ	Dipropionyl	2,3-di-O-propionyl-6- <i>t</i> -butyl silyl derivative of γ-cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	<b>78033AST</b>
Astec CHIRALDEX G-BP	γ	Butyryl	2,6-di-O-pentyl-3-butyryl derivative of γ-cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	<b>75033AST</b>



Phase	CD Type	Derivative	Full Description	Length (m)	I.D. (mm)	Film ( $\mu\text{m}$ )	Min. Temp.	Max. Temp. (Isothermal)	Max. Temp. (Progr.)	Cat. No.
<b>Combined Surface and Inclusion Interactions, Simple Derivatives</b>										
Astec CHIRALDEX B-DM	$\beta$	Dimethyl	2,3-di-O-methyl-6-t-butyl silyl derivative of $\beta$ -cyclodextrin	20	0.25	0.12	-10 °C	200 °C	220 °C	77022AST
				30	0.25	0.12	-10 °C	200 °C	220 °C	77023AST
				40	0.25	0.12	-10 °C	200 °C	220 °C	77024AST
				50	0.25	0.12	-10 °C	200 °C	220 °C	77025AST
Astec CHIRALDEX G-DM	$\gamma$	Dimethyl	22,3-di-O-methyl-6-t-butyl silyl derivative of $\gamma$ -cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	77033AST
Supelco $\beta$ -DEX 325	$\beta$	Dimethyl	25% 2,3-di-O-methyl-6-O-TBDMS- $\beta$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)	30	0.25	0.25	30 °C	230 °C	230 °C	24308
Supelco $\gamma$ -DEX 325	$\gamma$	Dimethyl	25% 2,3-di-O-methyl-6-O-TBDMS- $\gamma$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)	30	0.25	0.25	30 °C	230 °C	230 °C	24306
Supelco $\alpha$ -DEX 225	$\alpha$	Diacetyl	25% 2,3-di-O-acetyl-6-O-TBDMS- $\alpha$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)	30	0.25	0.25	30 °C	230 °C	230 °C	24311
Supelco $\beta$ -DEX 225	$\beta$	Diacetyl	25% 2,3-di-O-acetyl-6-O-TBDMS- $\beta$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)	30	0.25	0.25	30 °C	230 °C	230 °C	24348
Supelco $\gamma$ -DEX 225	$\gamma$	Diacetyl	25% 2,3-di-O-acetyl-6-O-TBDMS- $\gamma$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)	30	0.25	0.25	30 °C	230 °C	230 °C	24312
Astec CHIRALDEX B-PM	$\beta$	Permethyl	2,3,6-tri-O-methyl derivative of $\beta$ -cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	76023AST
				50	0.25	0.12	-10 °C	200 °C	220 °C	76025AST
Supelco $\beta$ -DEX 110	$\beta$	Permethyl	10% permethylated $\beta$ -cyclodextrin in SPB-35 poly(35% diphenyl/65% dimethylsiloxane)	30	0.25	0.25	30 °C	230 °C	230 °C	24301
				60	0.25	0.25	30 °C	230 °C	230 °C	24302
Supelco $\alpha$ -DEX 120	$\alpha$	Permethyl	20% permethylated $\alpha$ -cyclodextrin in SPB-35 poly(35% diphenyl/65% dimethylsiloxane)	30	0.25	0.25	30 °C	230 °C	230 °C	24310
Supelco $\beta$ -DEX 120	$\beta$	Permethyl	20% permethylated $\beta$ -cyclodextrin in SPB-35 poly(35% phenyl/65% dimethylsiloxane)	30	0.25	0.25	30 °C	230 °C	230 °C	24304
				60	0.25	0.25	30 °C	230 °C	230 °C	24305-U
Supelco $\gamma$ -DEX 120	$\gamma$	Permethyl	20% permethylated $\gamma$ -cyclodextrin in SPB-35 poly(35% phenyl/65% dimethylsiloxane)	30	0.25	0.25	30 °C	230 °C	230 °C	24307
<b>Inclusion Interactions</b>										
Astec CHIRALDEX A-DA	$\alpha$	Dialkyl	2,6-di-O-pentyl-3-methoxy derivative of $\alpha$ -cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	72003AST
Astec CHIRALDEX B-DA	$\beta$	Dialkyl	2,6-di-O-pentyl-3-methoxy derivative of $\beta$ -cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	72023AST
Astec CHIRALDEX G-DA	$\gamma$	Dialkyl	2,6-di-O-pentyl-3-methoxy derivative of $\gamma$ -cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	72033AST
Astec CHIRALDEX B-PH	$\beta$	(S)-Hydroxy-propyl	(S)-2-hydroxy propyl methyl ether derivative of $\beta$ -cyclodextrin	30	0.25	0.12	-10 °C	200 °C	220 °C	71023AST

\* Custom dimensions are available, please inquire with Technical Service at [techserv@sial.com](mailto:techserv@sial.com)

## Guard Columns and Connectors

The use of a guard column/retention gap is an inexpensive technique to extend the lifetime of capillary columns. A guard column/retention gap is a short (1-5 m) piece of uncoated deactivated fused silica tubing which is placed in-line between the GC injection port and the capillary column. The guard column/retention gap is used to take the brunt of the contamination/damage from the solvent and sample. By clipping the guard column/retention gap periodically to restore performance instead of the capillary column, the capillary column remains unaltered. Therefore, chromatography (retention times and resolution) is not affected.

Column connectors are useful for attaching a guard column/retention gap to an analytical column, or for repairing a broken column. We offer two options for connecting two pieces of fused silica tubing. The butt connector is a small stainless steel fitting that makes a zero dead volume seal. The GlasSeal™ connectors offer convenience.

### Fused Silica Guard Columns



These can be used to protect analytical columns from damaging sample components. Match the deactivation of the tubing with the polarity of the injection solvent.

Deactivation	Injection Solvents	Max. Temp.
Non-Polar	Alkanes Carbon disulfide Ethers	360 °C
Intermediate Polarity	Acetone Methylene chloride Toluene	360 °C
Polar	Acetonitrile Methanol Water	260 °C

Length (m)	I.D. (mm)	Cat. No.
<b>Non-Polar Deactivation</b>		
3	0.25	25722
5	0.25	25742
3	0.32	25723
5	0.32	25743
<b>Intermediate Polarity Deactivation</b>		
3	0.25	25727
5	0.25	25747
3	0.32	25728
5	0.32	25748-U
<b>Polar Deactivation</b>		
5	0.32	25752-U

### Capillary Column Butt Connectors

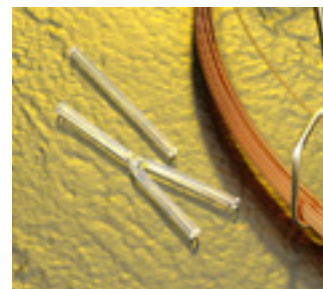


This device consists of a double-tapered ferrule and a stainless steel compression housing with a threaded cap. Small and light (2.3 cm x 0.6 cm, 4.4 g with ferrule), it provides a gas tight seal with zero dead volume. This unit maintains inertness with no change in column efficiency.

Description	Cat. No.
Capillary Column Butt Connector, body only	23804
<b>Supeltex® M-2B Ferrules, pack of two</b>	
To connect 0.20/0.25 mm I.D. to 0.20/0.25 mm I.D.	22453
To connect 0.32 mm I.D. to 0.32 mm I.D.	22454
To connect 0.53 mm I.D. to 0.53 mm I.D.	22591
To connect 0.20/0.25 mm I.D. to 0.53 mm I.D.	22455-U
To connect 0.32 mm I.D. to 0.53 mm I.D.	22586

### GlasSeal Capillary Column "Y" Connectors

Use a GlasSeal "Y" connector to split a sample to two columns for confirmatory analysis. Silanized for an inert inside surface, these can be used with our 0.10-0.53 mm I.D. tubing. To make this an extremely durable connection, use a small drop of polyimide sealing resin (cure at 200 °C, maximum temperature 350 °C).



Description	Qty.	Cat. No.
Borosilicate Glass	1 g	20480
Fused Silica	1 g	23631
Fused Silica	3 g	23632
Polyimide Sealing Resin	5 g	23817

# Common GC Accessories

## Injection Port Septa

A GC septum is located at the top of the injection port and serves two functions: 1) providing a leak-free seal to maintain carrier gas pressure inside the system, and 2) handling repeated puncturing by a syringe needle for sample introduction purposes without severe coring or leaking.

## Molded Thermogreen® LB-2 Septa



Molded Thermogreen LB-2 septa are manufactured from high quality, low bleed material using the same exclusive LB-2 rubber formulation that chromatographers are accustomed to using. The difference is that molded septa, unlike traditional die-cut septa, offer easier installation and better sealing. This is because our liquid injection molding process yields uniform septa; with all conforming shape, and having crisp, clean sides.

The useable inlet temperature range of 100-350 °C is adequate for the majority of GC applications. **Don't be fooled by other septa that advertise a maximum temperature of 400 °C (to make a septa with high thermal limits, one must also make it stiffer, resulting in septa that are harder to pierce and easier to core).** Our molded Thermogreen LB-2 septa offer the perfect combination of temperature range, low bleed, and easy puncturability.

The version with injection hole is for autosampler injections, manual injections, and/or SPME applications. The solid disc version is for manual injections.

Description	Qty.	Cat. No.
9.5 mm, with injection hole	50	28331-U
	250	28332-U
9.5 mm, solid discs	50	28670-U
	250	28671-U
10 mm, with injection hole	50	28333-U
	250	28334-U
10 mm, solid discs	50	28673-U
	250	28675-U
11 mm, with injection hole	50	28336-U
	250	28338-U
11 mm, solid discs	50	28676-U
	250	28678-U
11.5 mm, with injection hole	50	29446-U
	250	29448-U
11.5 mm, solid discs	50	29449-U
	250	29451-U
17 mm, with injection hole	50	29452-U
	250	29453-U
17 mm, solid discs	50	29456-U
	250	29457-U

## Inlet Liners

An injection port liner is used to make the connection between sample introduction and the GC column. Four primary injection techniques are used in GC; split, splitless, direct, and on-column. Inlet liners should be selected based on the injection technique being used to ensure optimal sample transfer to the column.

## FocusLiner™ Inlet Liners

The use of a wool plug in inlet liners has been used for many years to promote the rapid vaporization of the entire sample, minimize mass discrimination, and prevent



non-volatile material from entering the column. FocusLiner inlet liners incorporate a unique design that prevents shifting of the wool plug during repeated injections or sudden inlet pressure changes. Popular FocusLiner inlet liners are listed below. Other inlet liners are available, and can be viewed by referring to our catalog and/or our website [sigma-aldrich.com/gc-accessories](http://sigma-aldrich.com/gc-accessories)

Description	Qty.	Cat. No.
<b>For Agilent® 5890/6890/7890 (78.5 mm length x 6.3 mm O.D.)*</b>		
Split/splitless, 4 mm I.D.	5	2879805-U
Split/splitless, 2.3 mm I.D.	5	2879605-U
Split/splitless with single taper, 4 mm I.D.	5	2879905-U
Split/splitless with single taper, 2.3 mm I.D.	5	2879505-U
<b>For Finnigan*</b>		
Same catalog numbers as Agilent		
<b>For PerkinElmer® AutoSystem and Clarus (92 mm length x 6.3 mm O.D.)*</b>		
Split/splitless, 4 mm I.D.	5	2879205-U
Split/splitless with single taper, 4 mm I.D.	5	2879105-U
<b>For Shimadzu® 17A with SPL-17 Injector (95 mm length x 5 mm O.D.)*</b>		
Split/splitless, 3.4 mm I.D.	5	2878605-U
Split/splitless with single taper, 3.4 mm I.D.	5	2878405-U
<b>For Thermo ThermoQuest 8000/TRACE (105 mm length x 8 mm O.D.)*</b>		
Split, 5 mm I.D. (for use with 50 mm needles)	5	2877005-U
Splitless, 5 mm I.D. (for use with 70 mm needles)	5	2877205-U
Splitless with single taper, 5 mm I.D.	5	2877505-U
<b>For Varian® 1075/1077 Injectors (72 mm length x 6.3 mm O.D.)*</b>		
Split, 4 mm I.D.	5	2875405-U
Split with single taper, 4 mm I.D.	5	2874805-U
Split, 2.3 mm I.D.	5	2874705-U
<b>For Varian 1078/1079 Injectors (54 mm length x 5 mm O.D.)*</b>		
Split/splitless with single taper, 3.4 mm I.D.	5	2875705-U
Split/splitless with dual taper, 3.4 mm I.D.	5	2875505-U
<b>For Varian CP-1177 Injectors*</b>		
Same catalog numbers as Agilent		

\*All FocusLiner inlet liners are wool packed.

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