



# CHIRAL CHROMATOGRAPHY

Think Chiral...Think Supelco

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Astec: Part of the Sigma-Aldrich Analytical Family

## Chiral Services

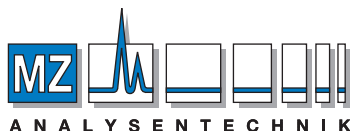
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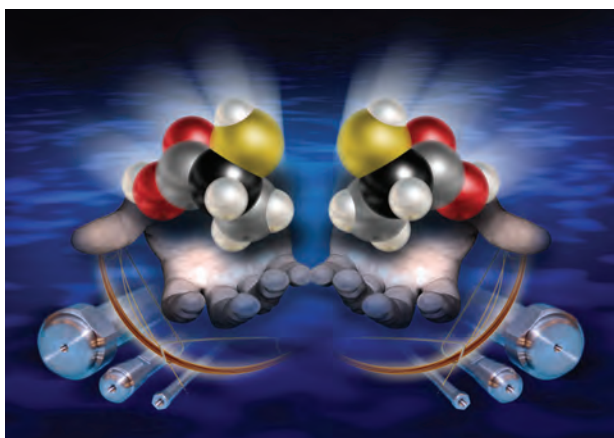
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## Think Chiral...Think Supelco

NEW PRODUCTS

## Think Chiral...Think Supelco

From a separation perspective, few types of compounds can match the challenges posed by chiral compounds. Chirality is important primarily because biological systems recognize stereochemistry. The enantiomers of chiral drug substances usually have different therapeutic efficacies, and it is not uncommon for one enantiomer to have unwanted and even toxic qualities. Eliminating the inactive enantiomer in chiral agrochemicals can reduce by half or more the amount of chemical that needs to be applied to the crop, with less waste and less environmental impact. However, chiral compounds pose a particular analytical challenge: Enantiomers have identical physical and chemical properties and differ only in their optical rotation and interactions with other chiral molecules.



## sigma-aldrich.com/chiral

Corporate web portal devoted to all things chiral

Sigma-Aldrich is a leader in chiral products and services for chemical synthesis, drug discovery, and analytical assessment. Our regularly-updated chiral web portal presents all of our products, services, seminars and technical literature for chirality in one convenient location. The site can be used to access Supelco's chiral HPLC and GC columns, our Chiral Services Laboratory, as well as Aldrich® chiral chemistry products, like privileged ligands and complexes, chiral catalysts, ligands and reagents, chiral auxiliaries and chiral building blocks.

Your resource for technical literature, bibliography, and applications for chiral chromatography

Developing a new chiral method typically involves perusing application databases, consulting the literature, screening columns, or contracting with a chiral service lab. Supelco can help with all of these approaches. We have created a centralized location of chiral resources and facile navigation. Visit and bookmark our chiral web portal to gain access to the valuable chiral resources:

- Applications: A growing list of thousands of applications on Astec and Supelco columns
- Bibliography: Up-to-date compilation of citations using our chiral columns
- Technical literature, handbooks and presentations

## Astec: Part of the Sigma-Aldrich Analytical Family



A pre-eminent innovator in chiral chromatography, Astec, its products and the expertise and dedication of its people became part of Sigma-Aldrich in 2006. Many Astec products were conceived through collaboration with Prof. Daniel Armstrong under a spirit of innovation that continues to thrive in our R&D group today.

*Chiral Columns + Chiral Reagents + Chiral Services + Expert Customer Support = Successful Enantiomer Separations*

Astec columns are backed by our world-class customer and technical support. They have become an integral part of our chiral offering, which includes:

- **HPLC & SFC columns** - Choices in stationary phase chemistry allow versatile mobile phase selection, suitable for a wide variety of analytes
- **Capillary GC columns** - Wide choice in inert, low-bleed, coated and chemically-bonded highly-enantioselective cyclodextrin derivatives
- **Reagents** - Selective chiral derivatization reagents and high-purity chiral mobile phase additives
- **Chiral Services** - Chiral column screening (HPLC and GC), method development and optimization and small-scale purification

## Chiral Services

Developing chiral methods and isolating pure enantiomers for further testing can be time consuming. To help our customers, we offer chiral column screening, method optimization and isolation of mg to gram quantities of purified enantiomer. Process-scale amounts can be purified by our SAFC facilities around the world. All work done by the expert staff of our state-of-the-art Chiral Services Laboratory is performed according to your specifications and is fully confidential. Our laboratory personnel also perform achiral separations and purifications.

## Chiral Services

|   |   |
|---|---|
| HPLC Chiral Column Screening                                    | HPLC chiral column screening protocol includes multiple mobile phase conditions run on multiple chiral stationary phases representing four separation modes (NP, RP, PIM and POM). Positive separation is verified on a separate system. Enantiomers are identified as (+) and (-)  |
| GC Chiral Column Screening                                      | GC column screening involves exploration of 4 GC chiral phases. Samples that require derivatization are verified by GC-MS.  |
| HPLC and GC Chiral Method Optimization and Development Services | Method optimization may vary, depending on the intended use of the method, which may include isolation/purification of enantiomers, resolution of metabolites, establishment of minimum detection limits, LC-MS compatible methods for clinical, stability or dissolution studies. Typical experiments in the optimization study include modifying buffer and pH, organic modifier type and strength, and column temperature. |
| Small-Scale Enantiomeric Purification                           | Milligram to gram quantities. Typical enantiomeric purity is 98% and verification is determined by analytical methods established in the screening study. Larger scale purifications are available through our SAFC offices worldwide.  |

## Chiral Services



Supelco Chiral Services Laboratory

## Chiral HPLC &amp; SFC Columns

Supelco's HPLC chiral stationary phases (CSPs) cover a broad range of chemistries, enantioselectivity, and application focus. The unique, proprietary Astec CHIROBIOTIC® and Astec CYCLOBOND® are particularly interesting from a chiral method development standpoint. Currently, the chiral HPLC and SFC columns we carry include:

- Astec CHIROBIOTIC® macrocyclic glycopeptide-based CSPs
- Astec CYCLOBOND® bonded cyclodextrin-based CSPs
- Astec Cellulose DMP polysaccharide-based CSPs
- Astec P-CAP™ and P-CAP™-DP chiral polymer-based CSPs
- Astec CLC copper ligand exchange CSPs
- CHIRALPAK® AGP, CBH, and HSA protein-based CSPs from DAICEL Corp.
- LARIHC™ and FRULIC™ cyclofructan-based CSPs from AZYP, LLC
- Kromasil® AmyCoat®, CelluCoat®, Chiral DMB, and Chiral TBB from Eka Chemicals AB (available from Sigma-Aldrich in USA, Canada, and Puerto Rico)

The development of innovative, new CSPs is an important R&D activity for us. Please call our Technical Services or visit our web site, [www.sigma-aldrich.com/chiral](http://www.sigma-aldrich.com/chiral), for information on our most current offering.

## Rapid, Efficient and Effective Chiral Method Development

Successful separations are more likely when you include Astec CHIROBIOTIC® and Astec CYCLOBOND columns in your chiral column screening protocol along with conventional cellulosic/amylosic CSPs. These two types of CSPs are highly complementary. For developing a new chiral HPLC method, we have created and use routinely in our laboratories a simple and rapid chiral column screening protocol shown below. Method development follows a simple strategy that tests polar ionic, polar organic, reversed-phase and normal phase modes.

## Method Development and Column Screening Protocol on CHIROBIOTIC® and CYCLOBOND HPLC Columns

|                     | CHIROBIOTIC® V2,<br>T, R, TAG  | CYCLOBOND I 2000,<br>HP-RSP, DMP, DNP  |  |
|---------------------|--|--|--|
| Mobile Phase System | Screening Mobile Phase   | Screening Mobile Phase   | Parameters to Optimize   |
| Polar Ionic         | (100:0.1:0.1, v/v/v)<br>CH <sub>3</sub> OH/acetic acid/<br>triethylamine |  | Change acid-base ratio, change the type of acid or base, add a volatile salt (test different ammonium salts) |
| Reversed-Phase      | (30:70) CH <sub>3</sub> CN/20 mM ammonium acetate, pH 5                  | (1) (30:70) CH <sub>3</sub> CN/20 mM ammonium acetate, pH 5<br>(2) (20:80) CH <sub>3</sub> OH/20 mM ammonium acetate, pH 5 | Change the % and type of organic modifier, adjust pH, buffer type and ionic strength                         |
| Polar Organic       | Methanol   | 95:5:0.1:0.1; CH <sub>3</sub> CN/<br>CH <sub>3</sub> OH/acetic acid/<br>triethylamine                                      | Use other polar organic solvents or blends   |
| Normal Phase        | (30:70) Ethanol/<br>heptane  | (30:70) Ethanol/heptane<br>(DMP, DNP only)   | Increase % of polar modifier, change both solvents   |

The Astec CHIROBIOTIC® and CYCLOBOND CSPs we recommend in the screening protocol are available in convenient kits. Also, you can increase the probability of success by incorporating the Astec Cellulose, Astec P-CAP, Astec CLC, LARIHC, and protein-based CSPs into your screening protocol. We would be happy to help you select the best line-up of columns for your types of analytes, detectors, and preferred mobile phase systems.



## Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

| No.     | Title                               |
|---------|-------------------------------------|
| T409107 | Chiral Method Development Wallchart |

## Preparative Chiral Separations

It is often the case that mg to gram quantities of purified enantiomer are required for safety and efficacy testing, or for further modification. Chiral HPLC is commonly used for this application. Once an enantiomer is shown to have desirable characteristics, then an asymmetrical synthesis may be developed to avoid a racemate. However, if a cost-effective synthetic approach is not readily available, then chromatography may indeed provide the best means to obtain purified enantiomer. Supelco's HPLC CSPs are amenable to preparative separations, whether in classic LC mode, SFC or in continuous preparative techniques such as SMB and multi-column processes. Supelco chiral HPLC phases offer benefits in:

- **Sample Solubility** - Choose mobile phases that maximize sample solubility, including aqueous and polar systems
- **Throughput** - Shorter retention times give higher throughput

## Supercritical Fluid Chromatography (SFC)

SFC is gaining in popularity primarily because of its speed and "green" advantages over normal phase HPLC. The CO<sub>2</sub> is readily removed from the eluate, which makes it ideal for prep. We offer several types of Astec chiral HPLC columns that are suitable for SFC separations.

- **Astec Cellulose DMP** columns are the most ideally suited for chiral SFC. They provide rapid separations with excellent enantioselectivity, long column lifetime, and low backpressure without significant column bleed.
- **Astec P-CAP** columns have recently been found to be beneficial in the separation of a complex mixture of enantiomers and achiral impurities. They provided resolution in instances where conventional polysaccharide-based CSPs failed (1).

## Chiral HPLC &amp; SFC Columns

## Supercritical Fluid Chromatography (SFC)

- A number of the Astec CYCLOBOND derivatives offer good opportunities for SFC, including CYCLOBOND I 2000 DNP, a pi acid, and the CYCLOBOND I 2000 DMP, a pi base. Astec CYCLOBOND I 2000, RSP and DM are also useful for SFC, the DM especially for fused polycyclic compounds. Since these latter CSPs have as their primary mechanism steric repulsion and hydrogen bonding, SFC's benefits of speed and efficiency are realized.
- Astec CHIROBIOTIC® CSPs are suitable for polar and non-polar neutral analytes. However, because of their ionic character, additives are required for ionized analytes to avoid lengthy analysis times. A good summary appears in Liu, et al (2).

(1) Barnhart, W. W.; Gahm, K. H.; Hua Z.; Goetzinger, W. Supercritical Fluid Chromatography Comparison of the Poly(trans-1,2-Cyclohexanediyl-bis Acrylamide) (P-CAP) Column with Several Derivatized Polysaccharide-based Stationary Phases. *J. Chromatogr. B*, **2008**, *875*, 217-229.

(2) Liu, Y.; Berthod, A.; Mitchell, C. R.; Xiao, T. L.; Zhang, B.; Armstrong, D. W. Super/Subcritical Fluid Chromatography Chiral Separations with Macroyclic Glycopeptide Stationary Phases. *J. Chrom. A*, **2002**, *978*, 185-204.

## Simulated Moving Bed Chromatography (SMB)

A continuous preparative HPLC technique, SMB or counter-current chromatography can be conceptualized as multiple columns used in series to make a single column of effectively infinite length. Supelco chiral HPLC columns and packings permit robust SMB operation. The polar organic and polar ionic (methanol or acetonitrile containing soluble ionic additives) mobile phases and larger particle sizes of Astec CHIROBIOTIC®; and Astec CYCLOBOND CSPs provide minimal back pressure, which is important in SMB to maximize through-put by allowing high flow rates. Additionally, mobile phases can be chosen to maximize sample solubility to prevent precipitation and increase the sample load. Note the special section on SMB sets in the Astec CHIROBIOTIC®; products.



## Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

No. Title  
T409105 Astec CHIROBIOTIC® Columns Sets for SMB

## Choosing a Chiral HPLC Column

When performing a chiral separation, it is usually very difficult to predict which CSP will provide adequate enantioselectivity, especially when working with new chemical entities. Even the experts use a column screening protocol. To make this process easier for you, we offer three economical and time-saving options:

- Access our extensive applications and bibliographical database by calling our Technical Services or viewing the growing applications library on our web site.
- Purchase an Astec CHIROBIOTIC® and/or Astec CYCLOBOND column screening kit. The kits contain CSPs in column geometries that have the highest success rate. They are priced below what the columns would cost if purchased separately.
- Rely on the expertise and professionalism of our Chiral Services laboratory for column screening, method optimization, and small-scale purification.

## Astec CHIROBIOTIC® Chiral HPLC Columns



## Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

No. Title  
T408131 CHIROBIOTIC® Brochure

## CHIROBIOTIC®: "Chiral by Nature"

The Astec CHIROBIOTIC® family comprises highly enantioselective chiral HPLC stationary phases based on naturally-occurring macrocyclic glycopeptides that have been bonded through multiple covalent linkages to high purity silica particles. Developed by Prof. Daniel Armstrong (1), CHIROBIOTIC® CSPs are unique in possessing ionic functional groups, which means they can be used for reversed-phase and LC-MS separation of ionizable enantiomers, as well as neutral molecules. The members of the CHIROBIOTIC® family have complementary stereoselectivity. If one CHIROBIOTIC® CSP does not give baseline resolution, testing the other CHIROBIOTIC® CSPs in the same mobile phase often results in complete resolution.

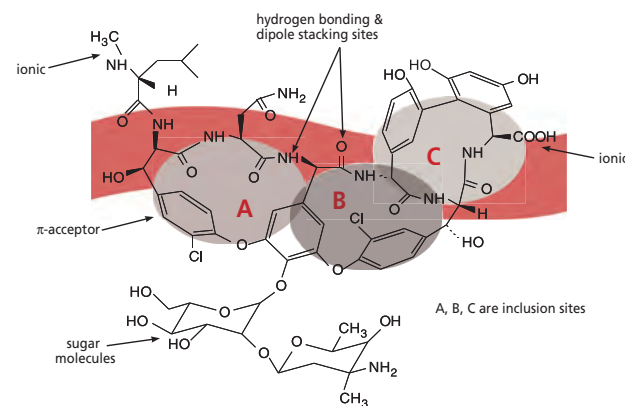
## Astec CHIROBIOTIC®; features and application areas:

- Aqueous and non-aqueous separations on the same column
- Ideal for reversed-phase and polar mobile phases for LC-MS
- No solvent or additive memory effects
- Robust columns with long lifetimes, important in bioanalysis
- Solvent choices maximize sample solubility
- Excellent preparative scalability and capacity
- Fast kinetics for speed and efficiency

(1) Armstrong, D. W.; Tang, Y.; Chen, S.; Zhou, Y.; Bagwill, C.; Chen, J. Macroyclic Antibiotics as a New Class of Chiral Selectors for Liquid Chromatography. *Anal. Chem.* **1994**, *66*, 1473-1484.

## CHIROBIOTIC® CSPs—Physical Properties

| CHIROBIOTIC® CSP      | Chiral Selector      | Chiral Centers | Sugar Groups | Inclusion Cavities | pH Range |
|-----------------------|----------------------|----------------|--------------|--------------------|----------|
| CHIROBIOTIC® V and V2 | Vancomycin           | 18             | 2            | 3                  | 3.5–7.0  |
| CHIROBIOTIC® T and T2 | Teicoplanin          | 23             | 3            | 4                  | 3.8–6.8  |
| CHIROBIOTIC® TAG      | Teicoplanin aglycone | 8              | 0            | 4                  | 3.0–6.8  |
| CHIROBIOTIC® R        | Ristocetin A         | 38             | 6            | 4                  | 3.5–6.8  |



Proposed structure of vancomycin (the chiral selector in CHIROBIOTIC® V and V2) showing different types of molecular interactions. The presence of ionic interactions is what differentiates CHIROBIOTIC® CSPs from other CSPs, and makes them valuable for polar and ionic compounds and MS detection.



## Chiral HPLC &amp; SFC Columns

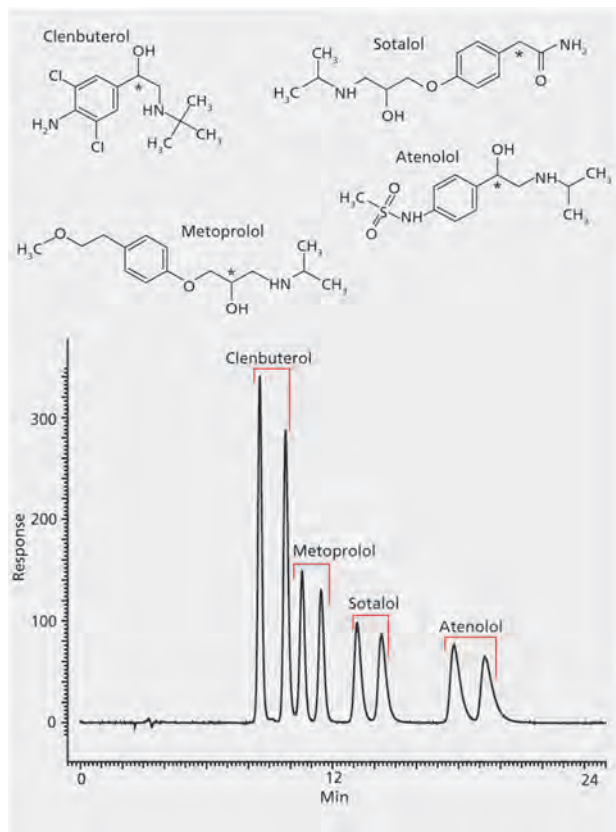
Astec CHIROBIOTIC® Chiral HPLC Columns: *Unique Multi-Modal Operation Includes Ionic Interactions***Unique Multi-Modal Operation Includes Ionic Interactions**

Astec CHIROBIOTIC® CSPs offer six different types of molecular interactions on **one** column: ionic, H-bond, pi-pi, dipole, hydrophobic and steric. They also possess multiple inclusion cavities that influence selectivity based on the molecular shape of the analyte. The optimization of enantiomer resolution is achieved by changing the mobile phase to leverage the types and relative strengths of the various interactions.

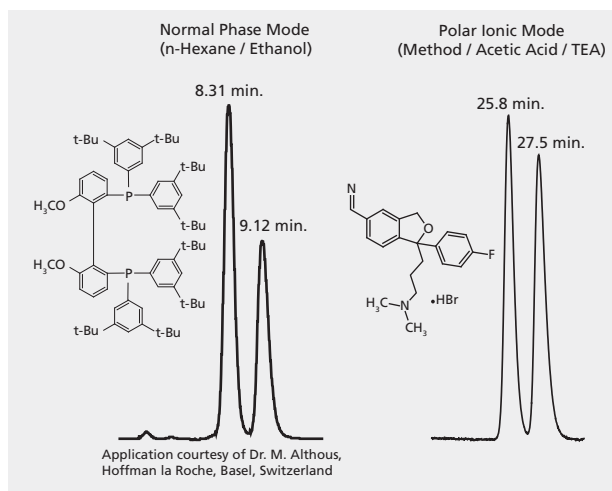
**HPLC Analysis of Beta-Receptor Agonist Enantiomers on Astec® CHIROBIOTIC® T**

## ► application for HPLC

column ..... Astec CHIROBIOTIC® T, 25 cm x 4.6 mm, 5 µm particles (12024AST)  
 mobile phase ..... 15 mM ammonium formate in methanol  
 flow rate ..... 1 mL/min  
 column temp. .... 25 °C  
 detector ..... UV at 220 nm  
 Application No. .... G004337

**The Most Versatile HPLC CSP**

Astec CHIROBIOTIC® CSPs offer the flexibility in choice of mobile phase conditions, both aqueous and non-aqueous, and are ideal for analytical and preparative separations of neutral, polar and ionic compounds. Their multiple interactions and absence of memory effects means the same CHIROBIOTIC® column can be successfully used in a variety of mobile phases, a significant benefit over CSPs that operate only in a single mode, normal or reversed-phase, for example, and must be dedicated to those mobile phase systems.



Demonstration of CHIROBIOTIC® column utility in both normal phase and polar ionic modes. The same CHIROBIOTIC® column can be used in all four modes, from aqueous to organic, without memory effects or loss of performance.  
 Column: CHIROBIOTIC® V, 25 cm x 4.6 mm, 5 µm particles (11024AST).

Left: **Normal phase mode.** 3,5-tBu-MeOBIPHEP enantiomers.  
 n-Hexane:10% ethanol in n-hexane (75:25), 1.5 mL/min.

Right: **Polar ionic mode.** Citalopram enantiomers.  
 Methanol:acetic acid:TEA (99:80:1:0.1), 0.5 mL/min.

**Astec CHIROBIOTIC® Application Areas**

Astec CHIROBIOTIC® CSPs have found utility in many areas of analytical chemistry, including:

- **Drug Discovery** - High enantioselectivity, fast screening protocols, scalability to prep, reproducibility for reliable methods, polar and non-polar analytes
- **Bioanalytical, Drug Metabolism** - High throughput, MS-compatibility, aqueous samples, short run times, rugged columns
- **Amino Acid and Peptide Analysis** - Resolves underivatized natural and synthetic chiral amino acids and peptides; different selectivity and higher preparative capacity for achiral amino acids than C18
- **Organic Synthesis** - Compatible with all HPLC solvents, including chlorinated solvents, to optimize sample solubility, fully scalable to prep

**Simplified Chiral Method Development**

Astec CHIROBIOTIC® HPLC columns enable simple method development, and are particularly useful for polar compounds due to the unique polar ionic mode. A single CHIROBIOTIC® column possesses multiple types of molecular interactions and can be run in four distinct modes. The same column can be exposed to all of the conditions outlined in the screening protocol without any change or loss of performance. This versatility is just one advantage that CHIROBIOTIC® CSPs have over other CSPs.

## Chiral HPLC & SFC Columns

Astec CHIROBIOTIC® Chiral HPLC Columns: *Complementary Selectivity to Cellulosic/Amylosic CSPs, but with Benefits*

### Complementary Selectivity to Cellulosic/Amylosic CSPs, but with Benefits

Astec CHIROBIOTIC® CSPs will perform the desired separation in nearly 75% of the cases, with a 50% overlap of the cellulosic/amylosic phases. However, the CHIROBIOTIC® CSPs often provide significant advantages, like allowing mobile phases that are better suited to the sample and detection method, or the CHIROBIOTIC® method may be faster, more efficient or more robust. A CHIROBIOTIC® method may also have advantages from a preparative standpoint in terms of solvent selection and capacity.



When performing chiral HPLC column screening, most enantiomers can be resolved on both CHIROBIOTIC® and cellulosic/amylosic CSPs. However, CHIROBIOTIC® CSPs often work better with ionic and highly polar compounds. Even in the areas of overlap, CHIROBIOTIC® CSPs often have advantages in solubility, LC/MS-compatibility and throughput.

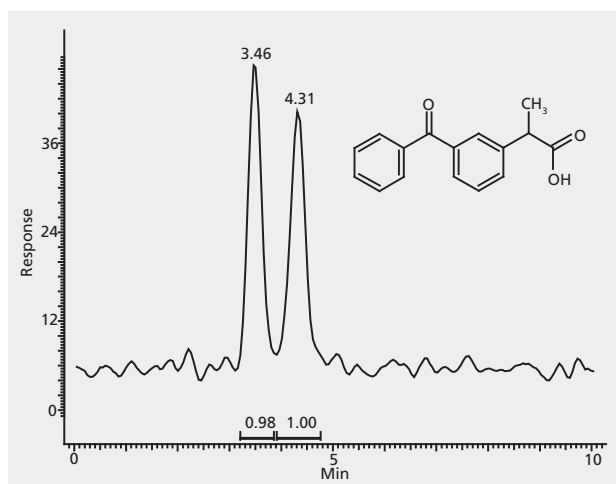
### LC-MS-friendly Chiral Separations

Each MS ionization source has an optimal set of mobile phase conditions. Outside this set, ionization may be suppressed with resulting loss in sensitivity. Astec CHIROBIOTIC® phases are uniquely able to operate across all mobile phase systems. CSPs that are limited to normal phase operation, like the majority of cellulose-based CSPs, reduce the analyst's options in detection methods. Astec CHIROBIOTIC® columns can be used in conjunction with HybridSPE-Phospholipid plates to enhance sensitivity by completely removing endogenous proteins and phospholipids, as shown in the clenbuterol from rat plasma application that follows.

### HPLC Analysis of Ketoprofen Enantiomers on Astec® CHIROBIOTIC® R (MS Detection)

#### ► application for HPLC

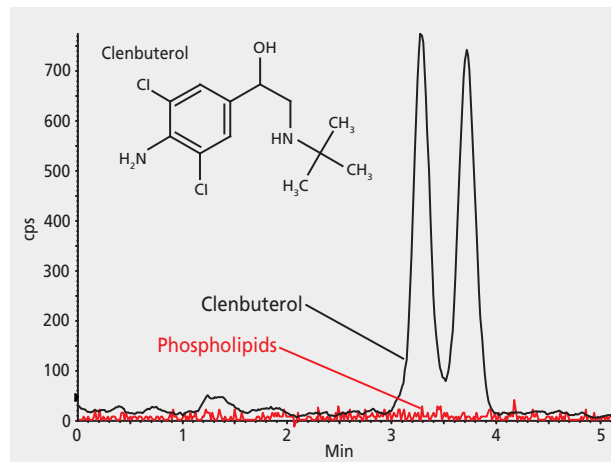
column ..... Astec CHIROBIOTIC® R, 15 cm x 2.1 mm, 5 µm particles (13019AST)  
 mobile phase ..... A: 20 mM ammonium acetate, pH 5.6 B: methanol Ratio: 70:30 (A:B)  
 flow rate ..... 0.2 mL/min  
 column temp. .... 35 °C  
 detector ..... ESI(-)  
 sample ..... ketoprofen  
 Application No. .... G004331



### HPLC Analysis of Clenbuterol Enantiomers on Astec® CHIROBIOTIC® T (ESI-MS of Plasma Extract)

#### ► application for HPLC

column ..... Astec CHIROBIOTIC® T, 10 cm x 2.1 mm I.D., 5 µm particles (12018AST)  
 mobile phase ..... 10 mM ammonium formate in methanol  
 flow rate ..... 0.3 mL/min  
 column temp. .... 30 °C  
 detector ..... ESI(+)  
 sample ..... clenbuterol, 10 ng/mL in rat plasma (phospholipids removed by extraction with HybridSPE-Phospholipid)  
 Application No. .... G004245



### Ideally Suited for Preparative Applications

Astec CHIROBIOTIC® phases offer unique opportunities for preparative purifications.

- **Excellent economics** - Especially with the polar organic and polar ionic modes. Ionic interactions play a significant role in the chiral recognition mechanism on Astec CHIROBIOTIC® CSPs. Solvents here are anhydrous and more volatile and less toxic than the typical normal phase mode.
- **No solvent limitations** - Halogenated solvents and very polar solvents are well tolerated on Astec CHIROBIOTIC® CSPs. This solvent tolerance is especially useful when optimizing for sample solubility.
- **Versatility** - The same Astec CHIROBIOTIC® column can be run in four distinctly different mobile phase types. Use of acid/base does not preclude their use in other mobile phases.
- **Stability** - Exceptional long-term stability of Astec CHIROBIOTIC® CSPs is derived from the multiple linkages used in anchoring the CSP and to the mild run conditions that are typically required.
- **Capacity** - The range of capacities is compound dependent. Significantly overlaps cellulose and amylose phases based on throughput, primarily because separations on Astec CHIROBIOTIC® CSPs are usually very fast. Capacities on Astec CHIROBIOTIC® V2/T2 phases are ~2.5 mg/gm ( $\alpha = 1.5$ ). Maximum capacity achieved was ~300 mg on column using a 250 x 21.2 mm column with  $\alpha = 2.0$ .

## Chiral HPLC &amp; SFC Columns

Astec CHIROBIOTIC® Chiral HPLC Columns: Astec CHIROBIOTIC® Column Screening Kits

## Astec CHIROBIOTIC® Column Screening Kits

The four Astec CHIROBIOTIC® CSPs we recommend in the screening protocol are available in 25 cm or 10 cm column kits. A full description of the screening procedure and instructions on how to optimize the separation are included with each kit.

Kit components:

- Astec CHIROBIOTIC®; T2
- Astec CHIROBIOTIC®; V
- Astec CHIROBIOTIC®; R
- Astec CHIROBIOTIC®; TAG
- Astec CHIROBIOTIC®; Handbook

You can further expand the screening field by incorporating Astec Cellulose DMP, Astec CYCLOBOND, Astec P-CAP, Astec CLC, LARIHC, and protein-based CSPs (sold separately) into your screening protocol.

## Astec CHIROBIOTIC® HPLC Column Screening Kit

| Description   | Cat. No. | Qty   |
|---|----------|-------|
| Astec CHIROBIOTIC® HPLC Column Screening Kit, particle size 5 µm, L 10 cm x I.D. 4.6 mm | 10300AST | 1 kit |
| Astec CHIROBIOTIC® HPLC Column Screening Kit, particle size 5 µm, L 25 cm x I.D. 4.6 mm | 10305AST | 1 kit |

## Astec CHIROBIOTIC® V and V2 (Vancomycin)

Neutral molecules, amides, acids, esters and amines show considerable enantioselectivity on these vancomycin-based CSPs. A wide variety of secondary and tertiary amines have been separated on the Astec CHIROBIOTIC® V in the polar ionic mode. Astec CHIROBIOTIC® V has demonstrated many of the separation characteristics of protein-based stationary phases, but with exceptional stability and much higher sample capacity. Some chiral analytes have been resolved that have not been reported separated on any other chiral stationary phase. Astec CHIROBIOTIC® V and V2 differ in their bonding chemistry the pore size of the support particle, giving them different selectivity and preparative capacity.

- Bonded phase: Vancomycin
- Operating pH range: 3.5 - 7.0
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 µm (other particles sizes, please inquire)
- Pore size: 100 Å (CHIROBIOTIC® V) or 200 Å (CHIROBIOTIC® V2)

For other column dimensions, particle sizes and bulk material, please inquire.

## Astec CHIROBIOTIC® V Chiral HPLC Column

| I.D. (mm)                 | L (cm) | Cat. No. | Qty  |
|---------------------------|--------|----------|------|
| <b>particle size 5 µm</b> |        |          |      |
| 2.1                       | 10     | 11018AST | 1 ea |
| 2.1                       | 15     | 11019AST | 1 ea |
| 2.1                       | 25     | 11020AST | 1 ea |
| 3.0                       | 10     | 11010AST | 1 ea |
| 4.6                       | 5      | 11021AST | 1 ea |
| 4.6                       | 10     | 11022AST | 1 ea |
| 4.6                       | 15     | 11023AST | 1 ea |
| 4.6                       | 25     | 11024AST | 1 ea |
| 10.0                      | 25     | 11034AST | 1 ea |
| 21.2                      | 25     | 11044AST | 1 ea |
| 30.0                      | 25     | 11054AST |      |

## Astec CHIROBIOTIC® V Chiral HPLC Guard

| I.D. (mm)                 | L (cm) | Cat. No. | Qty  |
|---------------------------|--------|----------|------|
| <b>particle size 5 µm</b> |        |          |      |
| 1.0                       | 2      | 11101AST | 1 ea |
| 4.0                       | 2      | 11100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

## Astec CHIROBIOTIC® V2 Chiral HPLC Column

| I.D. (mm)                  | L (cm) | Cat. No. | Qty  |
|----------------------------|--------|----------|------|
| <b>particle size 5 µm</b>  |        |          |      |
| 2.1                        | 10     | 15018AST | 1 ea |
| 2.1                        | 15     | 15019AST | 1 ea |
| 2.1                        | 25     | 15020AST | 1 ea |
| 4.6                        | 5      | 15021AST | 1 ea |
| 4.6                        | 10     | 15022AST | 1 ea |
| 4.6                        | 15     | 15023AST | 1 ea |
| 4.6                        | 25     | 15024AST | 1 ea |
| 10.0                       | 25     | 15034AST | 1 ea |
| 21.2                       | 25     | 15044AST | 1 ea |
| 30.0                       | 25     | 15054AST |      |
| <b>particle size 10 µm</b> |        |          |      |
| 4.6                        | 25     | 15124AST | 1 ea |
| <b>particle size 15 µm</b> |        |          |      |
| 4.6                        | 25     | 51041AST | 1 ea |

## Astec CHIROBIOTIC® V2 Chiral HPLC Guard

| I.D. (mm)                 | L (cm) | Cat. No. | Qty  |
|---------------------------|--------|----------|------|
| <b>particle size 5 µm</b> |        |          |      |
| 1.0                       | 2      | 15101AST | 1 ea |
| 4.0                       | 2      | 15100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

## Astec CHIROBIOTIC® T and T2 (Teicoplanin)

Astec CHIROBIOTIC® T and T2 have teicoplanin as the chiral selector. They offer unique selectivity for a number of classes of molecules, specifically underivatized  $\alpha$ ,  $\beta$ ,  $\gamma$  and cyclic amino acids, N-derivatized amino acids, hydroxy-carboxylic acids, acidic compounds including carboxylic acids and phenols, small peptides, neutral aromatic analytes and cyclic aromatic and aliphatic amines. Separations normally obtained on a chiral crown ether or ligand exchange-type CSPs are also possible on Astec CHIROBIOTIC® T and T2, but with much simpler mobile phases, such as alcohol-water. In addition, all of the known beta-blockers (amino alcohols), and dihydrocoumarins have been resolved. Astec CHIROBIOTIC® T and T2 differ in their bonding chemistry and the pore size of the support particle, giving them different selectivity and preparative capacity.

- Bonded phase: Teicoplanin
- Operating pH range: 3.8 - 6.8
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 µm (other particles sizes, please inquire)
- Pore size: 100 Å (CHIROBIOTIC® T) or 200 Å (CHIROBIOTIC® T2)
- USP Code L63

For other column dimensions, particle sizes and bulk material, please inquire.

## Chiral HPLC & SFC Columns

Astec CHIROBIOTIC® Chiral HPLC Columns: Astec CHIROBIOTIC® T and T2 (Teicoplanin)

### Astec CHIROBIOTIC® T Chiral HPLC Column

| I.D. (mm)           | L (cm) | Cat. No. | Qty  |
|---------------------|--------|----------|------|
| particle size 5 µm  |        |          |      |
| 2.1                 | 10     | 12018AST | 1 ea |
| 2.1                 | 15     | 12019AST | 1 ea |
| 2.1                 | 25     | 12020AST | 1 ea |
| 3.0                 | 10     | 12010AST | 1 ea |
| 4.6                 | 5      | 12021AST | 1 ea |
| 4.6                 | 10     | 12022AST | 1 ea |
| 4.6                 | 15     | 12023AST | 1 ea |
| 4.6                 | 25     | 12024AST | 1 ea |
| 10.0                | 25     | 12034AST | 1 ea |
| 21.2                | 25     | 12044AST | 1 ea |
| 30.0                | 25     | 12054AST |      |
| particle size 10 µm |        |          |      |
| 4.6                 | 25     | 12124AST | 1 ea |
| particle size 15 µm |        |          |      |
| 10                  | 5      | 51046AST | 1 ea |
| 4.6                 | 25     | 51047AST | 1 ea |

### Astec CHIROBIOTIC® T Chiral HPLC Guard

suitable for L63 per USP

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 1.0                | 2      | 12101AST | 1 ea |
| 4.0                | 2      | 12100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

### Astec CHIROBIOTIC® T2 Chiral HPLC Column

| I.D. (mm)           | L (cm) | Cat. No. | Qty  |
|---------------------|--------|----------|------|
| particle size 5 µm  |        |          |      |
| 2.1                 | 10     | 16018AST | 1 ea |
| 2.1                 | 15     | 16019AST | 1 ea |
| 2.1                 | 25     | 16020AST | 1 ea |
| 4.6                 | 5      | 16021AST | 1 ea |
| 4.6                 | 10     | 16022AST | 1 ea |
| 4.6                 | 15     | 16023AST | 1 ea |
| 4.6                 | 25     | 16024AST | 1 ea |
| 21.2                | 25     | 16044AST | 1 ea |
| 30.0                | 25     | 16054AST |      |
| particle size 10 µm |        |          |      |
| 4.6                 | 25     | 16124AST | 1 ea |

### Astec CHIROBIOTIC® T2 Chiral HPLC Guard

suitable for L63 per USP

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 1.0                | 2      | 16101AST | 1 ea |
| 4.0                | 2      | 16100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

### Astec CHIROBIOTIC® R (Ristocetin A)

CHIROBIOTIC®; R, based on the ristocetin A glycopeptide covalently bonded to high purity silica particles, has shown particular applicability to enantiomers of acidic compounds. Selectivity on CHIROBIOTIC®; R strongly correlates to the organic modifier, favoring the alcohol-type mobile phases by a large margin.

- Bonded phase: Ristocetin A
- Operating pH range: 3.5 - 6.8
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 µm (other particles sizes, please inquire)
- Pore size: 100 Å

For other column dimensions, particle sizes and bulk material, please inquire.

### Astec CHIROBIOTIC® R Chiral HPLC Column

| I.D. (mm)           | L (cm) | Cat. No. | Qty  |
|---------------------|--------|----------|------|
| particle size 5 µm  |        |          |      |
| 2.1                 | 10     | 13018AST | 1 ea |
| 2.1                 | 15     | 13019AST | 1 ea |
| 2.1                 | 25     | 13020AST | 1 ea |
| 3.0                 | 10     | 13010AST | 1 ea |
| 4.6                 | 5      | 13021AST | 1 ea |
| 4.6                 | 10     | 13022AST | 1 ea |
| 4.6                 | 15     | 13023AST | 1 ea |
| 4.6                 | 25     | 13024AST | 1 ea |
| 10.0                | 25     | 13034AST | 1 ea |
| 21.2                | 25     | 13044AST | 1 ea |
| 30.0                | 25     | 13054AST |      |
| particle size 10 µm |        |          |      |
| 4.6                 | 25     | 13124AST | 1 ea |

### Astec CHIROBIOTIC® R Chiral HPLC Guard

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 1.0                | 2      | 13101AST | 1 ea |
| 4.0                | 2      | 13100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

### Astec CHIROBIOTIC® TAG (Teicoplanin Aglycone)

The removal of three carbohydrate moieties gives CHIROBIOTIC®; TAG complementary selectivity to CHIROBIOTIC®; T. Resolution is enhanced toward many of the amino acids, α, β, γ and cyclic, and especially sulfur-containing methionine, histidine and cysteine. A number of neutral molecules like the oxazolidinones, hydantoin and diazepam, have shown enhanced resolution and, more remarkably, in single-solvent mobile phases, like methanol, ethanol or acetonitrile. Some acidic molecules have also shown increased selectivity.

- Bonded phase: Teicoplanin aglycone
- Operating pH range: 3.0 - 6.8
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 µm (other particles sizes, please inquire)
- Pore size: 100 Å

For other column dimensions, particle sizes and bulk material, please inquire.



## Chiral HPLC &amp; SFC Columns

Astec CHIROBIOTIC® Chiral HPLC Columns: Astec CHIROBIOTIC® TAG (Teicoplanin Aglycone)

## Astec CHIROBIOTIC® TAG Chiral HPLC Column

suitable for L63 per USP

| I.D. (mm)           | L (cm) | Cat. No. | Qty  |
|---------------------|--------|----------|------|
| particle size 5 µm  |        |          |      |
| 2.1                 | 10     | 14018AST | 1 ea |
| 2.1                 | 15     | 14019AST | 1 ea |
| 2.1                 | 25     | 14020AST | 1 ea |
| 4.6                 | 5      | 14021AST | 1 ea |
| 4.6                 | 10     | 14022AST | 1 ea |
| 4.6                 | 15     | 14023AST | 1 ea |
| 4.6                 | 25     | 14024AST | 1 ea |
| 10.0                | 15     | 14232AST | 1 ea |
| 10.0                | 25     | 14034AST | 1 ea |
| 21.2                | 25     | 14044AST | 1 ea |
| 30.0                | 25     | 14054AST | 1 ea |
| particle size 10 µm |        |          |      |
| 4.6                 | 25     | 14124AST | 1 ea |

## Astec CHIROBIOTIC® TAG Chiral HPLC Guard

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 1.0                | 2      | 14101AST | 1 ea |
| 4.0                | 2      | 14100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

## Astec CHIROBIOTIC® 8-Column Sets for SMB

## NEW PRODUCTS

SMB (simulated moving bed) is a form of preparative chromatography that utilizes multiple columns that act in concert as a single column. Throughput using SMB can be significantly higher than batch or column format. The qualities of Astec CHIROBIOTIC® CSPs that make them ideal for prep, including SMB, are excellent enantioselectivity, especially for polar and ionic compounds, mobile phase flexibility to maximize sample solubility, versatility for operation in all mobile phases without memory effects and high column efficiency for high throughput and minimal downstream processing. Especially relevant for prep by SMB, the ruggedness of Astec CHIROBIOTIC® CSPs enables long-term and reliable operation. The new Astec CHIROBIOTIC® columns for SMB feature particle size and column dimensions chosen for high flow rate and high efficiency. The 8 columns in the set have efficiencies that are matched to within 6% rsd. Single columns in 25 cm x 4.6 mm I.D. and 5 cm x 10 mm I.D. dimensions are available for method development and scale-up experiments. These sets are ideal for the Octave Chromatography System manufactured by Semba Biosciences.



Photograph of the 8-column SMB set. Each perfectly-matched column is 5 cm x 10 mm I.D.



## Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

| No.     | Title                                   |
|---------|---|
| T409105 | Astec CHIROBIOTIC® Columns Sets for SMB |

| Particle Size (µm)          | L x I.D.     | Cat. No. | Qty   |
|-----------------------------|--------------|----------|-------|
| phase Astec CHIROBIOTIC® V2 |              |          |       |
| 15                          | 5 cm x 10 mm | 51039AST | 1 set |
| phase Astec CHIROBIOTIC® V  |              |          |       |
| 15                          | 5 cm x 10 mm | 51045AST | 1 set |

## Astec CYCLOBOND® Chiral HPLC Columns



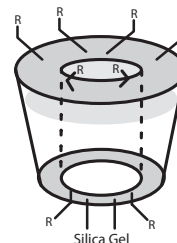
## Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

| No.     | Title                     |
|---------|---------------------------|
| T410091 | Astec CYCLOBOND® Brochure |

## Bonded Cyclodextrin Stationary Phases for Chiral HPLC Separations

Cyclodextrins are produced by the partial degradation of starch and enzymatic coupling of cleaved glucose units into crystalline, homogeneous toroidal structures of different molecular size. Three of the most widely characterized are α (alpha), β (beta) and γ (gamma) cyclodextrin. They contain 6, 7 and 8 glucose units, respectively. Since glucose is chiral, cyclodextrins are chiral. For example, β-cyclodextrin has 35 stereogenic centers. The toroidal structure has a hydrophilic surface resulting from the 2, 3 and 6 position hydroxyl groups, which makes them water soluble. The cavity is composed of the glucoside oxygens and methylene hydrogens giving it an apolar character. As a consequence, cyclodextrins can include the apolar portion of molecules of appropriate dimensions and bind them through dipole-dipole interactions, hydrogen bonding, or London dispersion forces. Therefore, the cyclodextrin structure offers unique opportunities to separate a wide variety of isomers that have the same chemical formula but differ in the spatial arrangement of their substituents.



Representation of a cyclodextrin toroid molecule attached to a silica surface. The functionalized glucose hydroxyl groups (shown as R groups in the figure) provide different enantioselectivity.

## Cyclodextrins—Physical Properties

| Cyclodextrin | Glucose Units | Stereogenic Centers | MW   | Cavity (nm) |
|--------------|---------------|---------------------|------|-------------|
| Alpha        | 6             | 30                  | 972  | 0.57        |
| Beta         | 7             | 35                  | 1135 | 0.78        |
| Gamma        | 8             | 40                  | 1297 | 0.95        |

## Chiral HPLC & SFC Columns

Astec CYCLOBOND® Chiral HPLC Columns: *Bonded Cyclodextrin Stationary Phases for Chiral HPLC Separations*

CYCLOBOND is the name given to the Astec technology for bonding cyclodextrins to high purity silica gel through a stable ether linkage. Developed in conjunction with Prof. Daniel Armstrong (1) and introduced in 1983, this patented line of chiral stationary phases retains its ability to form inclusion complexes, and allows for numerous chemical separations by selectively including into the cyclodextrin cavity a wide variety of organic molecules.

### Astec CYCLOBOND features and application areas:

- Native and derivatized  $\beta$ - and  $\gamma$ -cyclodextrins
- Covalent bonding for greater phase stability, especially in aqueous systems
- High degree of selectivity from inclusion mechanism and the unusual hydrogen bonding effects of the hydrophilic surface
- Additional interactions introduced by replacing some of the secondary hydroxyl groups with different selectors

### Astec CYCLOBOND I 2000 Series:

Based on the original CYCLOBOND I ( $\beta$ -cyclodextrin) technology, CYCLOBOND I 2000 columns are second-generation products. The CYCLOBOND I 2000 line includes native  $\beta$ -cyclodextrin and eight  $\beta$ -cyclodextrin derivatives.

### Astec CYCLOBOND II Series:

CYCLOBOND II columns are excellent chiral selectors for multi-ring structures such as those based on anthracene, chrysene or pyrene. These are  $\gamma$ -cyclodextrin bonded phases, and consist of eight glucopyranose units arranged in the same truncated cone shape.

### Astec CYCLOBOND Derivatives:

- Underivatized: CYCLOBOND I 2000, CYCLOBOND II
- Acetylated: CYCLOBOND I 2000 AC, CYCLOBOND II AC
- 2,3-di-O-Methyl: CYCLOBOND I 2000 DM
- 3,5-Dimethylphenyl carbamate: CYCLOBOND I 2000 DMP
- 2,6-Dinitro-4-trifluoromethyl phenyl ether: CYCLOBOND I 2000 DNP
- Hydroxypropyl ether (high performance): CYCLOBOND I 2000 HP-RSP
- Hydroxypropyl ether: CYCLOBOND I 2000 RSP, CYCLOBOND I 2000 SP

(1) Armstrong, D. W.; DeMond, W. Cyclodextrin bonded phases for the liquid chromatographic separation of optical, geometrical, and structural isomers. *J. Chrom. Sci.* **1984**, 22 (9), 411-415.

### Astec CYCLOBOND® Column Screening Kit

For convenience, the four Astec CYCLOBOND CSPs we recommend in the screening protocol described earlier in this section are available in a kit. A full description of the screening procedure and techniques to optimize the separation are included with each kit.

#### Kit components:

- Astec CYCLOBOND I 2000
- Astec CYCLOBOND I 2000 DMP
- Astec CYCLOBOND I 2000 HP-RSP
- Astec CYCLOBOND I 2000 DNP
- Astec CYCLOBOND Handbook

Also, you can further expand the screening field by incorporating the Astec CHIROBIOTIC®, Astec Cellulose DMP, Astec P-CAP, Astec CLC, and protein-based CSPs (all sold separately) into your screening protocol.

### Astec CYCLOBOND® HPLC Column Screening Kit

Ref: 1. Armstrong, D.W., DeMond, W. Cyclodextrin Bonded Phases for the Liquid Chromatographic Separation of Optical, Geometrical, and Structural Isomers *J. Chromatogr. Sci.* **22**, 411 (1984)

| Description  | Cat. No. | Qty   |
|--|----------|-------|
| Astec CYCLOBOND® HPLC Column Screening Kit, particle size 5 $\mu$ m, L 25 cm x I.D. 4.6 mm | 2005AST  | 1 kit |



### Related Information

Need help choosing the right chiral HPLC or GC column? Let our Chiral Services group do the work for you.

### Astec CYCLOBOND® I 2000 ( $\beta$ -Cyclodextrin)

Astec CYCLOBOND I 2000 is  $\beta$ -cyclodextrin bonded to high purity silica by a patented process to produce a stable matrix with the cyclodextrin arranged in such a way as to retain its most valuable property of forming inclusion complexes. This allows the cyclodextrin toroids to effect numerous chemical separations by selectively including into their cavities a wide variety of organic molecules. Non-inclusion type separations are also possible with the polar organic mode for a wide variety of molecule types.

- Bonded phase: Underivatized, native  $\beta$ -cyclodextrin
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10  $\mu$ m
- Pore size: 100 Å
- USP Code L45

For other column dimensions not listed, please inquire.

### Astec CYCLOBOND® I 2000 Chiral HPLC Column

| I.D. (mm)                                 | L (cm) | Cat. No. | Qty  |
|---|--------|----------|------|
| <b>particle size 5 <math>\mu</math>m</b>  |        |          |      |
| 2.1                                       | 10     | 20018AST | 1 ea |
| 2.1                                       | 15     | 20019AST | 1 ea |
| 2.1                                       | 25     | 20020AST | 1 ea |
| 4.6                                       | 5      | 20021AST | 1 ea |
| 4.6                                       | 10     | 20022AST | 1 ea |
| 4.6                                       | 15     | 20023AST | 1 ea |
| 4.6                                       | 25     | 20024AST | 1 ea |
| 10.0                                      | 25     | 20034AST | 1 ea |
| <b>particle size 10 <math>\mu</math>m</b> |        |          |      |
| 4.6                                       | 25     | 22024AST | 1 ea |

### Astec CYCLOBOND® I 2000 Chiral HPLC Guard

| I.D. (mm)                                | L (cm) | Cat. No. | Qty  |
|--|--------|----------|------|
| <b>particle size 5 <math>\mu</math>m</b> |        |          |      |
| 1.0                                      | 2      | 21010AST | 1 ea |
| 4.0                                      | 2      | 21100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

### Astec CYCLOBOND® I 2000 AC (Acetyl $\beta$ -Cyclodextrin)

Astec CYCLOBOND I 2000 AC is used primarily for aromatic alcohols or amines that are chiral on the  $\alpha$  or  $\beta$  carbon.

- Bonded phase: Acetylated  $\beta$ -cyclodextrin
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10  $\mu$ m
- Pore size: 100 Å
- USP Code L45

For other column dimensions not listed, please inquire.

## Chiral HPLC &amp; SFC Columns

Astec CYCLOBOND® Chiral HPLC Columns: *Astec CYCLOBOND® I 2000 AC (Acetyl β-Cyclodextrin)***Astec CYCLOBOND® I 2000 AC Chiral HPLC Column**

| I.D. (mm)                  | L (cm) | Cat. No. | Qty  |
|----------------------------|--------|----------|------|
| <b>particle size 5 μm</b>  |        |          |      |
| 2.1                        | 15     | 20119AST | 1 ea |
| 4.6                        | 5      | 20121AST | 1 ea |
| 4.6                        | 10     | 20122AST | 1 ea |
| 4.6                        | 15     | 20123AST | 1 ea |
| 4.6                        | 25     | 20124AST | 1 ea |
| 10.0                       | 25     | 20134AST | 1 ea |
| 30.0                       | 25     | 20154AST |      |
| <b>particle size 10 μm</b> |        |          |      |
| 4.6                        | 25     | 22124AST | 1 ea |

**Astec CYCLOBOND® I 2000 AC Chiral HPLC Guard**

| I.D. (mm)                 | L (cm) | Cat. No. | Qty  |
|---------------------------|--------|----------|------|
| <b>particle size 5 μm</b> |        |          |      |
| 1.0                       | 2      | 21011AST | 1 ea |
| 4.0                       | 2      | 21101AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

***Astec CYCLOBOND® I 2000 DM (Dimethyl β-Cyclodextrin)***

Astec CYCLOBOND I 2000 DM separates a wide variety of structural and geometric isomers and is complementary to Astec CYCLOBOND I 2000. This phase operates only in the reversed-phase mode with steric bulk as the main mechanism.

- Bonded phase: Dimethylated β-cyclodextrin
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 μm
- Pore size: 100 Å
- USP Code L45

For other column dimensions not listed, please inquire.

**Astec CYCLOBOND® I 2000 DM Chiral HPLC Column**

| I.D. (mm)                 | L (cm) | Cat. No. | Qty  |
|---------------------------|--------|----------|------|
| <b>particle size 5 μm</b> |        |          |      |
| 2.1                       | 15     | 20919AST | 1 ea |
| 4.6                       | 5      | 20921AST | 1 ea |
| 4.6                       | 10     | 20922AST | 1 ea |
| 4.6                       | 15     | 20923AST | 1 ea |
| 4.6                       | 25     | 20924AST | 1 ea |
| 30.0                      | 25     | 20954AST |      |

**Astec CYCLOBOND® I 2000 DM Chiral HPLC Guard**

| I.D. (mm)                 | L (cm) | Cat. No. | Qty  |
|---------------------------|--------|----------|------|
| <b>particle size 5 μm</b> |        |          |      |
| 1.0                       | 2      | 21019AST | 1 ea |
| 4.0                       | 2      | 21109AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

***Astec CYCLOBOND® I 2000 DMP (Dimethylphenyl β-Cyclodextrin)***

The reaction of the 3,5-dimethylphenyl isocyanate with the hydroxyl groups of β-cyclodextrin results in a pi-basic phase similar in character to the naphthylethyl carbamate phases. The selectivity is greater for the Astec CYCLOBOND I 2000 DMP when the chiral center of the analyte is part of a ring structure or is on the α carbon. This phase has been very useful for derivatized amines, like amphetamine ACQ.

- Bonded phase: 3,5-Dimethylphenyl carbamate modified β-cyclodextrin
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 μm
- Pore size: 100 Å
- USP Code L45

For other column dimensions not listed, please inquire.

**Astec CYCLOBOND® I 2000 DMP Chiral HPLC Column**

| I.D. (mm)                  | L (cm) | Cat. No. | Qty  |
|----------------------------|--------|----------|------|
| <b>particle size 5 μm</b>  |        |          |      |
| 2.1                        | 15     | 20719AST | 1 ea |
| 2.1                        | 25     | 20720AST | 1 ea |
| 4.6                        | 5      | 20721AST | 1 ea |
| 4.6                        | 10     | 20722AST | 1 ea |
| 4.6                        | 15     | 20723AST | 1 ea |
| 4.6                        | 25     | 20724AST | 1 ea |
| 10.0                       | 25     | 20734AST | 1 ea |
| 21.2                       | 25     | 20744AST | 1 ea |
| 30.0                       | 25     | 20754AST |      |
| <b>particle size 10 μm</b> |        |          |      |
| 4.6                        | 25     | 22724AST | 1 ea |

**Astec CYCLOBOND® I 2000 DMP Chiral HPLC Guard**

| I.D. (mm)                 | L (cm) | Cat. No. | Qty  |
|---------------------------|--------|----------|------|
| <b>particle size 5 μm</b> |        |          |      |
| 1.0                       | 2      | 21017AST | 1 ea |
| 4.0                       | 2      | 21107AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

***Astec CYCLOBOND® I 2000 DNP (Dinitrophenyl β-Cyclodextrin)***

This Astec CYCLOBOND derivative has dinitrophenyl functionality bonded through an ether linkage to the hydroxyl positions of the β-cyclodextrin. In this arrangement, a pi-electron sharing system is established with analytes having pi-electron systems (e.g. aromatic rings, carbonyl) in the stereogenic environment. Use of the ether linkage to anchor this pi-acidic dinitrophenyl ring results in a very stable system even under strong reversed-phase conditions. The pi-acidity of this group is further enhanced with the introduction of the trifluoromethyl group into the aromatic ring.

- Bonded phase: Dinitrophenyl modified β-cyclodextrin
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 μm
- Pore size: 100 Å
- USP Code L45

For other column dimensions not listed, please inquire.

## Chiral HPLC & SFC Columns

Astec CYCLOBOND® Chiral HPLC Columns: *Astec CYCLOBOND® I 2000 DNP (Dinitrophenyl β-Cyclodextrin)*

### Astec CYCLOBOND® I 2000 DNP Chiral HPLC Column

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 μm |        |          |      |
| 2.1                | 10     | 25018AST | 1 ea |
| 2.1                | 15     | 25019AST | 1 ea |
| 4.6                | 5      | 25021AST | 1 ea |
| 4.6                | 15     | 25023AST | 1 ea |
| 4.6                | 25     | 25024AST | 1 ea |

### Astec CYCLOBOND® I 2000 DNP Chiral HPLC Guard

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 μm |        |          |      |
| 1.0                | 2      | 25101AST | 1 ea |
| 4.0                | 2      | 25100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

### Astec CYCLOBOND® I 2000 HP-RSP (*R,S*-Hydroxypropyl β-Cyclodextrin)

In the design of this phase chemistry, it was an objective to create a very stable and reproducible phase with shorter retention times, while maintaining or improving selectivity over Astec CYCLOBOND I 2000 RSP. With that goal and more achieved, Astec CYCLOBOND I 2000 HP-RSP separates by extended H-bonding capability, and offers broad chiral selectivity for chiral screening. It is most beneficial for basic and neutral compounds.

- Bonded phase: (*R,S*)-Hydroxypropyl modified β-cyclodextrin (high performance)
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 μm
- Pore size: 100 Å
- USP Code L45

For other column dimensions not listed, please inquire.

### Astec CYCLOBOND® I 2000 HP-RSP Chiral HPLC Column

| I.D. (mm)           | L (cm) | Cat. No. | Qty  |
|---------------------|--------|----------|------|
| particle size 5 μm  |        |          |      |
| 2.1                 | 10     | 24018AST | 1 ea |
| 2.1                 | 25     | 24020AST | 1 ea |
| 4.6                 | 5      | 24021AST | 1 ea |
| 4.6                 | 10     | 24022AST | 1 ea |
| 4.6                 | 15     | 24023AST | 1 ea |
| 4.6                 | 25     | 24024AST | 1 ea |
| 30.0                | 25     | 24054AST |      |
| particle size 10 μm |        |          |      |
| 4.6                 | 25     | 24124AST | 1 ea |

### Astec CYCLOBOND® I 2000 HP-RSP Chiral HPLC Guard

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 μm |        |          |      |
| 1.0                | 2      | 24101AST | 1 ea |
| 4.0                | 2      | 24100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

### Astec CYCLOBOND® I 2000 RSP (*R,S*-Hydroxypropyl β-Cyclodextrin)

The hydroxyl groups on the surface of the β-cyclodextrin are reacted with (*R,S*)-propylene oxide to yield a general purpose chiral stationary phase. It has the added property of separating non-aromatic structures such as t-boc amino acids, for which it is a standard methodology.

- Bonded phase: (*R,S*)-Hydroxypropyl modified β-cyclodextrin
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 μm
- Pore size: 100 Å
- USP Code L45

For other column dimensions not listed, please inquire.

### Astec CYCLOBOND® I 2000 RSP Chiral HPLC Column

| I.D. (mm)           | L (cm) | Cat. No. | Qty  |
|---------------------|--------|----------|------|
| particle size 5 μm  |        |          |      |
| 2.1                 | 10     | 20318AST | 1 ea |
| 2.1                 | 15     | 20319AST | 1 ea |
| 2.1                 | 25     | 20320AST | 1 ea |
| 4.6                 | 5      | 20321AST | 1 ea |
| 4.6                 | 10     | 20322AST | 1 ea |
| 4.6                 | 15     | 20323AST | 1 ea |
| 4.6                 | 25     | 20324AST | 1 ea |
| 10.0                | 25     | 20334AST | 1 ea |
| 21.2                | 25     | 20344AST | 1 ea |
| 30.0                | 25     | 20354AST |      |
| particle size 10 μm |        |          |      |
| 4.6                 | 25     | 22324AST | 1 ea |

### Astec CYCLOBOND® I 2000 RSP Chiral HPLC Guard

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 μm |        |          |      |
| 1.0                | 2      | 21013AST | 1 ea |
| 4.0                | 2      | 21103AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

### Astec CYCLOBOND® I 2000 SP (*S*-Hydroxypropyl β-Cyclodextrin)

On Astec CYCLOBOND I 2000 SP, the hydroxyl groups on the surface of the β-cyclodextrin have been reacted with (*S*)-propylene oxide. This has the effect of extending hydrogen-bonding capabilities to accommodate analytes with chiral centers that are relatively distant from an aromatic ring structure. The (*S*)- form shows enhanced selectivity and efficiency for some separations.

- Bonded phase: (*S*)-Hydroxypropyl modified β-cyclodextrin
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10 μm
- Pore size: 100 Å
- USP Code L45

For other column dimensions not listed, please inquire.

## Chiral HPLC &amp; SFC Columns

Astec CYCLOBOND® Chiral HPLC Columns: Astec CYCLOBOND® I 2000 SP (*S*-Hydroxypropyl  $\beta$ -Cyclodextrin)

## Astec CYCLOBOND® I 2000 SP Chiral HPLC Column

| I.D. (mm)                | L (cm) | Cat. No. | Qty  |
|--------------------------|--------|----------|------|
| particle size 5 $\mu$ m  |        |          |      |
| 2.1                      | 15     | 20219AST | 1 ea |
| 4.6                      | 5      | 20221AST | 1 ea |
| 4.6                      | 25     | 20224AST | 1 ea |
| 30.0                     | 25     | 20254AST |      |
| particle size 10 $\mu$ m |        |          |      |
| 4.6                      | 25     | 22224AST | 1 ea |

## Astec CYCLOBOND® I 2000 SP Chiral HPLC Guard

| I.D. (mm)               | L (cm) | Cat. No. | Qty  |
|-------------------------|--------|----------|------|
| particle size 5 $\mu$ m |        |          |      |
| 4.0                     | 2      | 21102AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

Astec CYCLOBOND® II ( $\gamma$ -Cyclodextrin)

Consisting of eight glucopyranose units arranged in a truncated cone shape, Astec CYCLOBOND II is an excellent chiral selector for multi-ring structures. It is useful for isomeric compounds based on anthracene, chrysene and pyrene type ring structures. Astec CYCLOBOND II offers good selectivity and stability and is applicable to the polar organic mode of separation. Applications include steroids, porphyrins, Fmoc amino acids.

- Bonded phase: Underivatized, native  $\gamma$ -cyclodextrin
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10  $\mu$ m
- Pore size: 100 Å

For other column dimensions not listed, please inquire.

## Astec CYCLOBOND® II Chiral HPLC Column

| I.D. (mm)                | L (cm) | Cat. No. | Qty  |
|--------------------------|--------|----------|------|
| particle size 5 $\mu$ m  |        |          |      |
| 2.1                      | 15     | 46019AST | 1 ea |
| 4.6                      | 5      | 46021AST | 1 ea |
| 4.6                      | 10     | 40020AST | 1 ea |
| 4.6                      | 15     | 46023AST | 1 ea |
| 4.6                      | 25     | 41020AST | 1 ea |
| particle size 10 $\mu$ m |        |          |      |
| 4.6                      | 25     | 44024AST | 1 ea |

## Astec CYCLOBOND® II Chiral HPLC Guard

| I.D. (mm)               | L (cm) | Cat. No. | Qty  |
|-------------------------|--------|----------|------|
| particle size 5 $\mu$ m |        |          |      |
| 4.0                     | 2      | 42120AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

Astec CYCLOBOND® II AC (Acetyl  $\gamma$ -Cyclodextrin)

Astec CYCLOBOND II AC columns are bonded  $\gamma$ -cyclodextrin with acetylation of the 2- and 3-hydroxyl groups. As a result, the mouth of the cavity has available a hydrogen-acceptor site that can interact with a hydrogen donor, such as an amine attached to at least two or more fused rings. An example would be 1- or 2-substituted naphthylethylamine. Applications include steroids and sterols, depending on where the hydroxyl groups are positioned.

- Bonded phase: Acetylated  $\gamma$ -cyclodextrin
- Operating pH range: 3 - 7
- Particle type: High-purity, spherical silica
- Particle diameter: 5 or 10  $\mu$ m
- Pore size: 100 Å

For other column dimensions not listed, please inquire.

## Astec CYCLOBOND® II AC Chiral HPLC Column

| I.D. (mm)                | L (cm) | Cat. No. | Qty  |
|--------------------------|--------|----------|------|
| particle size 5 $\mu$ m  |        |          |      |
| 2.1                      | 10     | 47018AST | 1 ea |
| 2.1                      | 15     | 47019AST | 1 ea |
| 4.6                      | 25     | 41022AST | 1 ea |
| particle size 10 $\mu$ m |        |          |      |
| 4.6                      | 25     | 44124AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

Astec Cellulose DMP  
(Dimethylphenylcarbamate)

NEW PRODUCTS

Efficient, Rugged and Economical Columns for Chiral HPLC & SFC

Astec Cellulose DMP comprises spherical, high-purity porous silica coated with DMPC (dimethylphenyl carbamate)-derivatized cellulose packed in analytical to preparative size HPLC columns. It separates a wide range of chiral compounds under normal phase, polar organic, SFC, and reversed-phase conditions, with high efficiency, high loading capacity, and excellent column lifetime. With performance comparable to other DMPC-derivatized cellulose CSPs, but at substantially lower price, Astec Cellulose DMP is a must-have for every chiral column HPLC or SFC screening protocol. Astec Cellulose DMP is complementary to the other Astec CSPs, including CHIROBIOTIC<REFERENCE ID="3826" TYPE="trademark"/>, CYCLOBOND, and the P-CAP product lines. It should be investigated as an alternative to higher priced cellulose-DMPC columns for existing methods. The cost savings are especially dramatic when comparing preparative column dimensions.

- Phase: DMPC (dimethylphenyl carbamate)-derivatized cellulose (coated)
- Particle type: High-purity, spherical silica
- Particle diameter: 5  $\mu$ m
- Normal phase, polar organic, and SFC modes
- Scalable from analytical to preparative
- Suitable for USP Code L40



## Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

| No.     | Title                        |
|---------|------------------------------|
| T410110 | Astec Cellulose DMP Brochure |



## Chiral HPLC &amp; SFC Columns

Astec Cellulose DMP (Dimethylphenylcarbamate)

## Astec Cellulose DMP Chiral HPLC Column

| I.D. (mm)                     | L (cm) | Cat. No. | Qty  |
|-------------------------------|--------|----------|------|
| particle size 5 $\mu\text{m}$ |        |          |      |
| 2.1                           | 10     | 51112AST | 1 ea |
| 2.1                           | 15     | 51100AST | 1 ea |
| 2.1                           | 25     | 51101AST | 1 ea |
| 4.6                           | 10     | 51097AST | 1 ea |
| 4.6                           | 15     | 51098AST | 1 ea |
| 4.6                           | 25     | 51099AST | 1 ea |
| 10                            | 25     | 51102AST | 1 ea |
| 21.2                          | 25     | 51103AST | 1 ea |

## Astec Cellulose DMP Chiral HPLC Guard

| I.D. (mm)                     | L (cm) | Cat. No. | Qty   |
|-------------------------------|--------|----------|-------|
| particle size 5 $\mu\text{m}$ |        |          |       |
| 2.1                           | 2      | 51105AST | 1 kit |
| 2.1                           | 2      | 51104AST | 2 ea  |
| 4                             | 2      | 51107AST | 1 kit |
| 4                             | 2      | 51106AST | 2 ea  |
| 10                            | 1      | 51108AST | 1 ea  |
| 21.2                          | 1      | 51109AST | 1 ea  |

Guard cartridges require holders that are sold separately. The 2.1 and 4 mm I.D. cartridges use 21150AST or 59660-U (both stand-alone) or 504254 or 55205 (both integral). The 10 mm I.D. cartridges use 567499-U. The 21.2 mm I.D. cartridges use 581392-U.

## Astec P-CAP™ and P-CAP™-DP Chiral HPLC Columns

Useful for chiral HPLC and SFC separations, Astec P-CAP and P-CAP-DP are based on a unique polycyclic amine polymer that has been covalently bonded to high-purity silica particles. They offer high stability, extremely high sample loadability, easy scale-up and no memory effect. Conceptualized by Prof. Francesco Gasparrini (1) with further phase development by Prof. Daniel Armstrong (2), these CSPs are used primarily for normal phase, polar organic and SFC chiral separations. The bonding procedure offers maximum protection of the silica and excellent availability of the short-chain polymer ligand to ensure high capacity. The resulting thin, ordered layer of polymer does not alter the porous structure of the silica. The repeating chiral moiety offers both structural conformation and hydrogen bonding interactions as the driving mechanisms.

Preparative separations on Astec P-CAP and P-CAP-DP can be run in a variety of solvents, without any large impact on selectivity, to meet analyte solubility requirements. As a result of the juxtaposition of the binding sites, molecules with two or more functional groups demonstrate the best selectivity. Separations have been run in pure acetone, heptane/ethanol, dichloromethane/methanol and ethylacetate. Selectivity can be obtained in a variety of solvent choices with different efficiencies. Salt and/or acetic acid can be added to improve efficiency or enhance detection in mass spectrometry. Astec P-CAP and P-CAP-DP are available in two enantiomeric forms (R,R) and (S,S). This permits reversing the elution order, which can be very useful in preparative applications.

## Astec P-CAP and P-CAP-DP features and application areas:

- Polymeric ligand CSP for normal phase and polar organic operation
- Ideal for SFC and Sub-SFC
- No solvent limitations
- High capacity for preparative applications
- Stable, covalent chemistry
- Reversible elution order through R,R and S,S configurations
- Available in standard (Astec P-CAP) and diphenyl (Astec P-CAP-DP) chemistries

(1) Gasparrini, F.; Misiti, D.; Rompietti, R.; Villani, C. "New hybrid polymeric liquid chromatography chiral stationary phase prepared by surface-initiated polymerization" *J. Chromatogr. A* **2005**, *1064* (1), 25-38.

(2) Zhong, Q.; Han, X.; He, L.; Beesley, T. E.; Trahanovsky, W. S.; Armstrong, D. W. "Chromatographic evaluation of poly(trans-1,2-cyclohexanediyl-bisacrylamide) as a chiral stationary phase for HPLC" *J. Chromatogr. A* **2005**, *1066* (1-2), 55-70.



## Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

| No.     | Title                               |
|---------|-------------------------------------|
| T410060 | Astec P-CAP™ and P-CAP™-DP Brochure |

## Astec (R,R) and (S,S) P-CAP™

Astec P-CAP is made from a polymerized diacryloyl-trans-1,2-diphenylethylenediamine bonded to the silica surface. It utilizes hydrogen bonding and steric effects as enantiomer separation mechanisms. Astec P-CAP can be used for SFC and normal phase separations of racemic mixtures. It has high stability, high sample loading capacity (suitable for preparative scale-up), and no memory effect. The elution order of compounds can be reversed in the (R,R) versus (S,S) configuration.

- Bonded phase: Poly(trans-1,2-cyclohexanediyl-bis-acrylamide)
- Operating pH range: N/A (operated in normal phase and polar organic modes)
- Particle type: High-purity, spherical silica
- Particle diameter: 3.5, 5 or 10  $\mu\text{m}$
- Pore size: 200 Å

For other column dimensions not listed, please inquire.

## Astec (R,R) P-CAP™ Chiral HPLC Column

| I.D. (mm)                       | L (cm) | Cat. No. | Qty  |
|---------------------------------|--------|----------|------|
| particle size 3.5 $\mu\text{m}$ |        |          |      |
| 4.6                             | 15     | 30023AST | 1 ea |
| particle size 5 $\mu\text{m}$   |        |          |      |
| 2.1                             | 15     | 31019AST | 1 ea |
| 4.6                             | 5      | 31021AST | 1 ea |
| 4.6                             | 10     | 31022AST | 1 ea |
| 4.6                             | 15     | 31023AST | 1 ea |
| 4.6                             | 25     | 31024AST | 1 ea |
| particle size 10 $\mu\text{m}$  |        |          |      |
| 4.6                             | 25     | 31124AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

## Astec (S,S) P-CAP™ Chiral HPLC Column

| I.D. (mm)                       | L (cm) | Cat. No. | Qty  |
|---------------------------------|--------|----------|------|
| particle size 3.5 $\mu\text{m}$ |        |          |      |
| 4.6                             | 5      | 32021AST | 1 ea |
| 4.6                             | 15     | 32023AST | 1 ea |
| particle size 5 $\mu\text{m}$   |        |          |      |
| 2.1                             | 10     | 33018AST | 1 ea |
| 2.1                             | 15     | 33019AST | 1 ea |
| 4.6                             | 5      | 33021AST | 1 ea |
| 4.6                             | 10     | 33022AST | 1 ea |
| 4.6                             | 15     | 33023AST | 1 ea |
| 4.6                             | 25     | 33024AST | 1 ea |
| particle size 10 $\mu\text{m}$  |        |          |      |
| 4.6                             | 25     | 33124AST | 1 ea |

## Chiral HPLC &amp; SFC Columns

Astec P-CAP™ and P-CAP™-DP Chiral HPLC Columns: Astec (R,R) and (S,S) P-CAP™

## Astec (S,S) P-CAP™ Chiral HPLC Guard Column

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 4.0                | 2      | 33100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately. The 1 mm I.D. guard is sold complete and does not require a separate holder.

## Astec (R,R) and (S,S) P-CAP™-DP

The DP introduces phenyl rings to add pi-pi interactions, giving it one additional type of interaction compared to Astec P-CAP. Astec P-CAP-DP uses similar protocols as Astec P-CAP and can be optimized for either normal or polar organic mobile phases. It is less polar than Astec P-CAP, and ideal for sub- and supercritical fluid applications. The elution order of compounds can be reversed in the (R,R) versus (S,S) configuration.

- Bonded phase: Poly(diphenylethylenediamine-bis-acryloyl) or Poly-DPEDA
- Operating pH range: N/A (operated in normal phase and polar organic modes)
- Particle type: High-purity, spherical silica
- Particle diameter: 3.5 or 5 µm
- Pore size: 200 Å

For other column dimensions not listed, please inquire.

## Astec (R,R) P-CAP™-DP Chiral HPLC Column

| I.D. (mm)            | L (cm) | Cat. No. | Qty  |
|----------------------|--------|----------|------|
| particle size 3.5 µm |        |          |      |
| 4.6                  | 15     | 34023AST | 1 ea |
| particle size 5 µm   |        |          |      |
| 4.6                  | 15     | 35023AST | 1 ea |
| 4.6                  | 25     | 35024AST | 1 ea |
| 21.2                 | 25     | 35044AST | 1 ea |

## Astec (R,R) P-CAP™-DP Chiral HPLC Guard Column

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 4.0                | 2      | 35100AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately.

## Astec (S,S) P-CAP™-DP Chiral HPLC Column

| I.D. (mm)            | L (cm) | Cat. No. | Qty  |
|----------------------|--------|----------|------|
| particle size 3.5 µm |        |          |      |
| 4.6                  | 15     | 36023AST | 1 ea |
| particle size 5 µm   |        |          |      |
| 4.6                  | 15     | 37023AST | 1 ea |
| 4.6                  | 25     | 37024AST | 1 ea |

The 4 mm I.D. guard cartridge requires a holder, Cat. No. 21150AST (stand-alone) or 504254 (direct-connect), sold separately.

## Astec CLC-L and CLC-D (Copper Ligand Exchange)

Astec CLC columns use the copper ligand concept described by Davankov to effect enantiomer separation (1,2). The method uses a small, chiral bidentate ligand attached to the silica surface and a copper sulphate-containing mobile phase. The copper ions coordinate with the chiral selector on the stationary phase and carboxylic acid functional groups on the analytes to form transient diastereomeric complexes in solution. The technique also has the advantage of giving small acids with no UV chromophore a strong 254 nm signal.

Astec CLC columns are ideal for analysis of alpha-hydroxy acids, like lactic, malic, tartaric and mandelic acids, amino acids, other amines and bi-functional racemates, like amino alcohols. Two versions of the column provide elution order reversal. On the CLC-D column, the L enantiomer generally elutes before D, with the exception of tartaric acid. The reverse is true on the CLC-L column where D elutes before L. Proline and aspartic acid are particularly suited for low-level detection on the CLC column since the copper complex is detected at 254 nm UV. Both can be resolved on the CLC-D or CLC-L in 5 mM CuSO<sub>4</sub> with the usual reversal of elution order from the CLC-D to CLC-L. In theory, any analyte that can complete the coordination with the copper ion can be resolved.

## Astec CLC features and application areas:

- Separates α-hydroxy carboxylic acids, amino acids and other α-bifunctional compounds
- High selectivity with simple mobile phases
- Copper complex gives strong UV 254 nm signal
- Simple reversal of elution order, AstecCLC-L vs. AstecCLC-D
- Excellent reproducibility

## Properties of Astec CLC-L and Astec CLC-D:

- Bonded phase: Chiral bidentate ligand (L and D forms)
- Requires 5 mM CuSO<sub>4</sub> mobile phase
- Operating pH range: 3 - 6 (adjust pH of the 5 mM CuSO<sub>4</sub> mobile phase with acetic acid)
- Particle type: High-purity spherical silica
- Particle diameter: 5 µm
- Pore size: 100Å
- USP Code L32

- (1) Davankov, V. A.; Rogozhin, S. V. Ligand chromatography as a novel method for the investigation of mixed complexes: Stereoselective effects in α-amino acid copper(II) complexes. *J. Chrom. A.* **1971**, *60*, 280-283.
- (2) Davankov, V. A. Enantioselective ligand exchange in modern separation techniques. *J. Chrom. A.* **2003**, *1000*, 891-915.



## Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

| No.     | Title                                    |
|---------|--|
| T410062 | Astec CLC (Copper Ligand Exchange) Flyer |

## Astec CLC-D Chiral HPLC Column

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 4.6                | 15     | 53023AST | 1 ea |

## Astec CLC-L Chiral HPLC Column

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 4.6                | 15     | 53123AST | 1 ea |

We recommend using a Supelco ColumnSaver precolumn filter (Cat. No. 55214-U or 55215-U) to protect CLC columns.

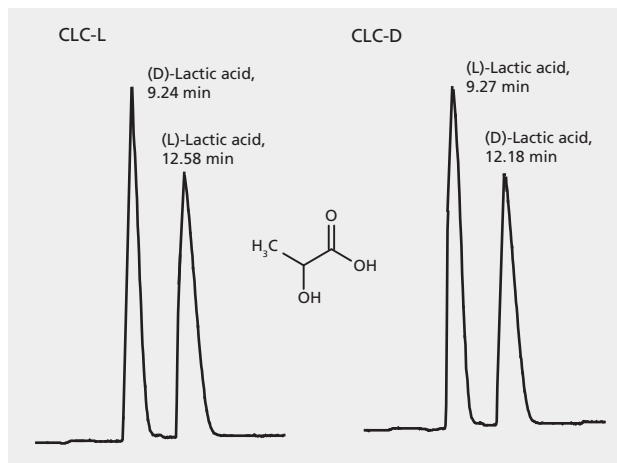
## Chiral HPLC &amp; SFC Columns

Astec CLC-L and CLC-D (Copper Ligand Exchange)

## HPLC Analysis of Lactic Acid Enantiomers on Astec® CLC-L and CLC-D

## ▶ application for HPLC

column ..... Astec CLC-L and CLC-D, 15 cm x 4.6 mm I.D., 5 µm particles (53023AST, 53123AST)  
 mobile phase ..... 5 mM CuSO<sub>4</sub>  
 flow rate ..... 1 mL/min  
 column temp. .... 25 °C  
 detector ..... UV at 254 nm  
 injection ..... 10 µL  
 sample ..... lactic acid  
 Application No. .... G004399



## Protein-based Chiral HPLC Columns

Hermansson described the use of natural proteins immobilized onto a silica support for chiral separations in 1983 (1). Proteins contain a large number of chiral centers of one configuration, and many other sites that contribute to the general retention process. We offer three CSPs with proteins as the chiral selectors, CHIRALPAK® AGP ( $\alpha_1$ -acid glycoprotein), CHIRALPAK® CBH (cellobiohydrolase) and CHIRALPAK® HSA (human serum albumin). All are manufactured by DAICEL Corporation. They are typically used in reversed-phase mode, and perform a wide variety of chiral separations. CHIRALPAK® HSA is also used for drug-binding studies. Solutes are retained by three types of interactions: ionic (for charged solutes), hydrophobic, and hydrogen bonding. The relative contribution of the different forces to solute retention depends on the nature of the analyte.

**CHIRALPAK® AGP:** Extremely broad applicability. First choice when developing methods on protein-CSPs.

**CHIRALPAK® HSA:** Analytes are typically very hydrophilic acids.

**CHIRALPAK® CBH:** Analytes are typically very hydrophilic amines and amino alcohols.

## Protein-based CSP features and application areas:

- Direct reversed-phase resolution of chiral molecules
- Stable in a variety of organic modifiers
- Available in analytical and semi-preparative sizes
- CHIRALPAK HSA is also used for drug-binding studies

(Note: These columns were previously named CHIRAL-AGP, CHIRAL-HSA, and CHIRAL-CBH prior to the acquisition of ChromTech by DAICEL Corp.)

(1) Hermansson, J. Direct liquid chromatographic resolution of racemic drugs using  $\alpha_1$ -acid glycoprotein as the chiral stationary phase. *J. Chromatogr. A*, **1983**, 269, 71-80.

CHIRALPAK® AGP ( $\alpha_1$ -Acid Glycoprotein)

CHIRALPAK® AGP has the broadest range of selectivity of all protein phases currently available. It comprises  $\alpha_1$ -acid glycoprotein (AGP) as the chiral selector immobilized onto spherical 5 µm silica particles. When bonded, AGP is very stable and tolerates pure organic solvents (up to 20%), elevated temperatures (up to 40 °C), and pH values from 4 to 7. Operated in reversed-phase mode, CHIRALPAK® AGP separates enantiomers of an extremely broad range of drug substances, such as acids, amines and neutral compounds. The mobile phases are mixtures of phosphate or acetate buffers and organic solvents such as 2-propanol or acetonitrile. The enantioselectivity and retention can easily be regulated by mobile phase pH and ionic strength, and the nature and concentration of the organic modifier. The most important tool in method development is the mobile phase pH, which affects the ionization of both solutes and the protein stationary phase. AGP has a low isoelectric point (pI) of 2.7. This means at pH 2.7 the column has a net zero charge. From pH 2.7 to 7, the net negative charge on the AGP molecule increases, providing increased retention of positively-charged analytes, like amines. These compounds are also retained by hydrophobic and hydrogen bonding interactions.

- Bonded phase:  $\alpha_1$ -Acid glycoprotein (CHIRALPAK® AGP)
- Particle type: High-purity spherical silica
- Particle diameter: 5 µm
- Operating pH range: 4 - 7
- Maximum organic percentage in mobile phase: 20%
- Maximum pressure: 2000 psi
- Maximum operating temperature: 40 °C
- Washing procedure: 10-15% isopropanol in water (do not exceed max. pressure)
- USP Code L41

## CHIRALPAK® AGP HPLC Column

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 2.0                | 5      | 58129AST | 1 ea |
| 2.0                | 10     | 58130AST | 1 ea |
| 2.0                | 15     | 58131AST | 1 ea |
| 3.0                | 5      | 58169AST | 1 ea |
| 3.0                | 10     | 58170AST | 1 ea |
| 3.0                | 15     | 58171AST | 1 ea |
| 4.0                | 5      | 58149AST | 1 ea |
| 4.0                | 10     | 58150AST | 1 ea |
| 4.0                | 15     | 58151AST | 1 ea |
| 10.0               | 10     | 58155AST | 1 ea |
| 10.0               | 15     | 58157AST | 1 ea |

## CHIRALPAK® AGP HPLC Guard Column

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 2.0                | 1      | 58178AST | 2 ea |
| 3.0                | 1      | 58158AST | 2 ea |
| 4.0                | 1      | 58188AST | 2 ea |

The 2, 3, and 4 mm I.D. guard cartridges require a guard column holder (Cat. No. 58159AST), sold separately.

## Chiral HPLC &amp; SFC Columns

Protein-based Chiral HPLC Columns: CHIRALPAK® AGP ( $\alpha_1$ -Acid Glycoprotein)

## Guard Column Holder for CHIRALPAK® AGP, HSA, and CBH

► for use with CHIRALPAK AGP, CBH, and HSA 1 cm x 2.0, 3.0, and 4.0 mm guard cartridges

stainless steel



Holder for CHIRALPAK AGP, HSA, and CBH 1 cm length guard cartridges (58159AST)



Right, holder (58159AST) and left, representative CHIRALPAK® AGP, HSA, or CBH 1 cm length guard cartridges.

58159AST

1 ea

## CHIRALPAK® CBH (Cellobiohydrolase)

Used primarily for the separation enantiomers of basic compounds, CHIRALPAK® CBH has cellobiohydrolase as the chiral selector immobilized on spherical 5  $\mu$ m silica particles. Used in reversed-phase mode, retention and enantioselectivity is regulated by changes of pH, buffer concentration and organic modifier. The mobile phases are mixtures of phosphate or acetate buffers and organic solvents such as 2-propanol or acetonitrile. The column is preferably used for the separation of enantiomers of basic drugs, particularly compounds containing one or more nitrogen atoms along with one or more hydrogen-bonding groups (alcohol, phenol, carbonyl, amide, ether, ester, etc.).

- Bonded phase: Cellobiohydrolase (CBH)
- Particle type: High-purity spherical silica
- Particle diameter: 5  $\mu$ m
- Operating pH range: 3 - 7
- Maximum organic percentage in mobile phase: 20%
- Maximum pressure: 2000 psi
- Maximum operating temperature: 40 °C
- Washing procedure: 10-15% isopropanol in water (do not exceed max. pressure)

## CHIRALPAK® CBH HPLC Column

| I.D. (mm)               | L (cm) | Cat. No. | Qty  |
|-------------------------|--------|----------|------|
| particle size 5 $\mu$ m |        |          |      |
| 2.0                     | 5      | 58529AST | 1 ea |
| 2.0                     | 10     | 58530AST | 1 ea |
| 2.0                     | 15     | 58531AST | 1 ea |
| 3.0                     | 5      | 58569AST | 1 ea |
| 3.0                     | 10     | 58570AST | 1 ea |
| 3.0                     | 15     | 58571AST | 1 ea |
| 4.0                     | 5      | 58549AST | 1 ea |
| 4.0                     | 10     | 58550AST | 1 ea |
| 4.0                     | 15     | 58551AST | 1 ea |
| 10.0                    | 10     | 58555AST | 1 ea |
| 10.0                    | 15     | 58557AST | 1 ea |

## CHIRALPAK® CBH HPLC Guard Column

| I.D. (mm)               | L (cm) | Cat. No. | Qty  |
|-------------------------|--------|----------|------|
| particle size 5 $\mu$ m |        |          |      |
| 2.0                     | 1      | 58578AST | 2 ea |
| 3.0                     | 1      | 58558AST | 2 ea |
| 4.0                     | 1      | 58588AST | 2 ea |

The 2, 3, and 4 mm I.D. guard cartridges require a guard column holder (Cat. No. 58159AST), sold separately.

## CHIRALPAK® HSA (Human Serum Albumin)

CHIRALPAK® HSA, which uses human serum albumin as the chiral selector, is highly selective for acidic racemates, preferably weak and strong acids, zwitterionic and non-protolytic (neutral) compounds. Phosphate buffers (normally 0.01-0.1M, pH 5-7) with addition of organic modifiers are used as mobile phases. Enantioselectivity and retention can be regulated by changing the mobile phase composition. However, the primary use of CHIRALPAK® HSA is for fast drug/protein binding studies (1). To calculate the % protein binding, measure the retention time of an unretained compound ( $t_0$ ) and the compound of interest ( $t_r$ ) on the CHIRALPAK® HSA column. Then use the capacity factor equation:

$$k = (t_r - t_0)/t_r$$

to calculate the % protein binding (P):

$$P = 100k/(k+1)$$

Different types of mobile phases can be used. A mobile phase consisting of 6% 2-propanol in 20 mM potassium phosphate buffer, pH 7.0 gives data in good agreement with literature data. The mobile phase conditions should be chosen to suit the drugs to be tested, i.e., for high protein binding drugs a mobile phase with higher eluting strength might be needed in order to reduce retention times.

- Bonded phase: Human serum albumin (HSA)
- Particle type: High-purity spherical silica
- Particle diameter: 5  $\mu$ m
- Operating pH range: 5 - 7
- Maximum organic percentage in mobile phase: 20%
- Maximum pressure: 2000 psi
- Maximum operating temperature: 40 °C
- Washing procedure: 10-15% isopropanol in water (do not exceed max. pressure)

(1) Goodman, A.; Gilman, A.G. *The Pharmacological Basis of Therapeutics*, 9<sup>th</sup> Edition, McGraw-Hill: New York, 1996; pp 1712-1792.

## CHIRALPAK® HSA HPLC Column

| I.D. (mm)               | L (cm) | Cat. No. | Qty  |
|-------------------------|--------|----------|------|
| particle size 5 $\mu$ m |        |          |      |
| 2.0                     | 5      | 58429AST | 1 ea |
| 2.0                     | 10     | 58430AST | 1 ea |
| 2.0                     | 15     | 58431AST | 1 ea |
| 3.0                     | 5      | 58469AST | 1 ea |
| 3.0                     | 10     | 58470AST | 1 ea |
| 3.0                     | 15     | 58471AST | 1 ea |
| 4.0                     | 5      | 58449AST | 1 ea |
| 4.0                     | 10     | 58450AST | 1 ea |
| 4.0                     | 15     | 58451AST | 1 ea |
| 10.0                    | 10     | 58455AST | 1 ea |
| 10.0                    | 15     | 58457AST | 1 ea |

## Chiral HPLC & SFC Columns

Protein-based Chiral HPLC Columns: *CHIRALPAK® HSA (Human Serum Albumin)*

### CHIRALPAK® HSA HPLC Guard Column

| I.D. (mm)          | L (cm) | Cat. No. | Qty  |
|--------------------|--------|----------|------|
| particle size 5 µm |        |          |      |
| 2.0                | 1      | 58478AST | 2 ea |
| 3.0                | 1      | 58458AST | 2 ea |
| 4.0                | 1      | 58488AST | 2 ea |

The 2, 3, and 4 mm I.D. guard cartridges require a guard column holder (Cat. No. 58159AST), sold separately.

### Kromasil® Chiral HPLC Columns

NEW PRODUCTS

We are pleased to be able to offer Kromasil products, including their high-quality chiral HPLC line, to our customers in the USA (including Puerto Rico) and Canada. Kromasil chiral stationary phases have an excellent reputation in analytical to process scale HPLC and SFC.

#### Kromasil AmyCoat® and CelluCoat®

The functionalized amylose and cellulose coated chiral selectors are coated onto a wide pore silica (>1000 Å) matrix; this silica has a low surface area, which reduces the number of achiral interaction sites and thus increases the chiral selectivity. High resolution, excellent selectivity, high-pressure stability, and stable performance when switching between compatible mobile phases are some important benefits. AmyCoat and CelluCoat columns are also available in columns that are compatible with reversed-phase operation.

- **AmyCoat:** The chiral selector is tris-(3,5-dimethylphenyl)carbamoyl amylose (USP Code L51)
- **CelluCoat:** The chiral selector is tris-(3,5-dimethylphenyl)carbamoyl cellulose (USP Code L40)

#### Kromasil DMB and TBB

Kromasil DMB and TBB bonded chiral phases separate a broad range of racemates. These 2 phases have been developed to complement each other in selectivity. The chiral monomers are polymerized with a hydrosilane to yield a network polymer, which incorporates the bifunctional C2-symmetric chiral selector and is covalently bonded onto 100 Å silica.

- **Chiral DMB:** The chiral monomer is O,O'-bis (3,5-dimethylbenzoyl)-N,N'-diallyl-L-tartar diamide
- **Chiral TBB:** The chiral monomer is O,O'-bis (4-tert-butylbenzoyl)-N,N'-diallyl-L-tartar diamide

#### Kromasil Guard Columns

The Kromasil guards are sold in packs of 3 or 5 cartridges. They require a holder and coupler that are sold separately.

- For 2.1 to 4.6 mm I.D. cartridges: Use holder **K08970954** and coupler **K08970955**.
- For 10 to 21.2 mm I.D. cartridges: Use holder **K08970956** and coupler **K08970957**.

A convenient Guard Cartridge Starter Kit for 4.6 mm I.D. Kromasil CelluCoat columns is available. It contains 5 guard cartridges, a guard cartridge holder, and a coupler. The part number is **K08971109**.



### Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

| No.     | Title                               |
|---------|-------------------------------------|
| T409214 | Kromasil® Chiral Applications Guide |

### Kromasil® AmyCoat® Chiral HPLC Column

| I.D. (mm)           | L (mm) | Cat. No.  | Qty  |
|---------------------|--------|-----------|------|
| particle size 3 µm  |        |           |      |
| 2.1                 | 50     | K08971229 | 1 ea |
| 2.1                 | 150    | K08971225 | 1 ea |
| 4.6                 | 50     | K08670344 | 1 ea |
| 4.6                 | 150    | K08670346 | 1 ea |
| particle size 5 µm  |        |           |      |
| 2.1                 | 50     | K08971230 | 1 ea |
| 2.1                 | 150    | K08971226 | 1 ea |
| 4.6                 | 50     | K08670608 | 1 ea |
| 4.6                 | 150    | K08670347 | 1 ea |
| 4.6                 | 250    | K08670348 | 1 ea |
| 10                  | 250    | K08670605 | 1 ea |
| 21.2                | 250    | K08670606 | 1 ea |
| 30                  | 250    | K08670607 | 1 ea |
| particle size 10 µm |        |           |      |
| 4.6                 | 150    | K08670603 | 1 ea |
| 4.6                 | 250    | K08670604 | 1 ea |
| 10                  | 250    | K08670600 | 1 ea |
| 21.2                | 250    | K08670601 | 1 ea |
| 30                  | 250    | K08670602 | 1 ea |

### Kromasil® AmyCoat® Chiral Reversed Phase HPLC Column

| I.D. (mm)          | L (mm) | Cat. No.  | Qty  |
|--------------------|--------|-----------|------|
| particle size 3 µm |        |           |      |
| 4.6                | 50     | K08670500 | 1 ea |
| 4.6                | 150    | K08670501 | 1 ea |

### Kromasil® AmyCoat® Chiral HPLC Guard Cartridge

| I.D. (mm)          | L (mm) | Cat. No.  | Qty  |
|--------------------|--------|-----------|------|
| particle size 5 µm |        |           |      |
| 4.6                | 10     | K08971102 | 1 ea |
| 10                 | 10     | K08971103 | 1 ea |
| 21.2               | 10     | K08971104 | 1 ea |

Kromasil guard cartridges require a holder and coupler that are sold separately. For 2.1 to 4.6 mm I.D. cartridges: Use holder **K08970954** and coupler **K08970955**. For 10 to 21.2 mm I.D. cartridges: Use holder **K08970956** and coupler **K08970957**.

### Kromasil® CelluCoat® Chiral HPLC Column

| I.D. (mm)          | L (mm) | Cat. No.  | Qty  |
|--------------------|--------|-----------|------|
| particle size 3 µm |        |           |      |
| 2.1                | 50     | K08971227 | 1 ea |
| 2.1                | 150    | K08971223 | 1 ea |
| particle size 5 µm |        |           |      |
| 2.1                | 50     | K08971228 | 1 ea |
| 2.1                | 150    | K08971224 | 1 ea |
| particle size 3 µm |        |           |      |
| 4.6                | 50     | K08670372 | 1 ea |
| 4.6                | 150    | K08670370 | 1 ea |



## Chiral HPLC &amp; SFC Columns

Kromasil® Chiral HPLC Columns

| I.D. (mm)                  | L (mm) | Cat. No.  | Qty  |
|----------------------------|--------|-----------|------|
| <b>particle size 5 µm</b>  |        |           |      |
| 4.6                        | 50     | K08670617 | 1 ea |
| 4.6                        | 150    | K08670373 | 1 ea |
| 4.6                        | 250    | K08670374 | 1 ea |
| 10                         | 250    | K08670614 | 1 ea |
| 21.2                       | 250    | K08670615 | 1 ea |
| 30                         | 250    | K08670616 | 1 ea |
| <b>particle size 10 µm</b> |        |           |      |
| 4.6                        | 150    | K08670612 | 1 ea |
| 4.6                        | 250    | K08670613 | 1 ea |
| 10                         | 250    | K08670609 | 1 ea |
| 21.2                       | 250    | K08670610 | 1 ea |
| 30                         | 250    | K08670611 | 1 ea |

## Kromasil® CelluCoat® Chiral Reversed Phase HPLC Column

| I.D. (mm)                 | L (mm) | Cat. No.  | Qty  |
|---------------------------|--------|-----------|------|
| <b>particle size 3 µm</b> |        |           |      |
| 4.6                       | 50     | K08670502 | 1 ea |
| 4.6                       | 150    | K08670503 | 1 ea |

## Kromasil® CelluCoat® Chiral HPLC Guard Cartridge

| I.D. (mm)                 | L (mm) | Cat. No.  | Qty  |
|---------------------------|--------|-----------|------|
| <b>particle size 5 µm</b> |        |           |      |
| 4.6                       | 10     | K08971106 | 1 ea |
| 10                        | 10     | K08971107 | 1 ea |
| 21.2                      | 10     | K08971108 | 1 ea |

Kromasil guard cartridges require a holder and coupler that are sold separately. For 2.1 to 4.6 mm I.D. cartridges: Use holder **K08970954** and coupler **K08970955**. For 10 to 21.2 mm I.D. cartridges: Use holder **K08970956** and coupler **K08970957**.

Kromasil guard cartridges require a holder and coupler that are sold separately. For 2.1 to 4.6 mm I.D. cartridges: Use holder **K08970954** and coupler **K08970955**. For 10 to 21.2 mm I.D. cartridges: Use holder **K08970956** and coupler **K08970957**.

## Kromasil® TBB Chiral HPLC Column

| I.D. (mm)                 | L (mm) | Cat. No.  | Qty  |
|---------------------------|--------|-----------|------|
| <b>particle size 5 µm</b> |        |           |      |
| 4.6                       | 250    | K08670376 | 1 ea |

Kromasil guard cartridges require a holder and coupler that are sold separately. For 2.1 to 4.6 mm I.D. cartridges: Use holder **K08970954** and coupler **K08970955**. For 10 to 21.2 mm I.D. cartridges: Use holder **K08970956** and coupler **K08970957**.

## Cyclofructans: LARIHC™ and FRULIC™ Chiral HPLC and HILIC Columns

## NEW PRODUCTS

Cyclofructans are cyclic oligosaccharides and the newest class of chiral stationary phases for HPLC, SFC, and HILIC. Invented by Prof. Daniel W. Armstrong (1,2) and introduced by AZYP, the LARIHC and FRULIC derivatized cyclofructan-based HPLC columns are now available world-wide through Supelco/Sigma-Aldrich.

| CF Phase       | Mode        | Description   |
|----------------|-------------|---|
| LARIHC CF6-P   | Chiral HPLC | Alkyl derivatized cyclofructan 6. Particularly useful for chiral primary amines.                      |
| LARIHC CF6-M   | Chiral HPLC | Methyl-functionalized cyclofructan 6. Complementary to LARIHC CF6-P for chiral primary amines.        |
| LARIHC CF6-RN  | Chiral HPLC | R-Naphthylethyl-functionalized cyclofructan 6. Suitable for enantiomers that are not primary amines.  |
| LARIHC CF7-DMP | Chiral HPLC | 3,5-Dimethylphenyl functionalized cyclofructan 7. Complementary enantioselectivity to LARIHC CF-6-RN. |
| FRULIC-N       | HILIC       | Neutral poly-hydroxy based stationary phase   |
| FRULIC-C       | HILIC       | Hydrophilic charged cyclofructan 6  |

All phases are available in standard HPLC column dimensions. For more information on AZYP's LARIHC and FRULIC columns, please visit our website or contact your local Sigma-Aldrich office.

## References:

- (1) Ping Sun, Chunlei Wang, Zachary S. Breitbach, Ying Zhang, and Daniel W. Armstrong. "Development of New HPLC Chiral Stationary Phases Based on Native and Derivatized Cyclofructans" *Anal. Chem.* **2009**, *81*, 10215-10226.
- (2) Ping Sun and Daniel W. Armstrong. "Effective enantiomeric separations of racemic primary amines by the isopropyl carbamate-cyclofructan6 chiral stationary phase" *J. Chromatogr. A*, **2010**, *1217*, 4904-4918.

## Protection for Chiral HPLC Columns

Within our chiral HPLC line we offer three distinct guard column formats:

- 2 cm x 1.0 mm I.D. stand-alone guard columns for all Astec CSPs (CHIROBIOTIC®, CYCLOBOND, Cellulose, P-CAP)
- 2 cm x 4.0 mm I.D. guard cartridges for all Astec CSPs (holder required)
- 1 cm x 2, 3, and 4 mm I.D. guard cartridges for the protein-based CSPs (CHIRAL-AGP, -HSA, -CBH) (holder required)

Examples are shown below. We also offer the Supelco ColumnSaver direct-connect in-line filter (55214-U or 55215-U) to remove particulate matter. The ColumnSaver can be used to protect any of our HPLC columns. This section describes the hardware needed for the various guard designs. Packed guard cartridges and columns can be found with the respective CSP they are intended to protect.



Astec 2 cm x 1 mm I.D. HPLC guard column. Does not require a holder.



Representative 2 cm length Supelguard or Astec HPLC guard cartridge. Requires a stand-alone (21150AST) or direct-connect (504254) holder.



Representative CHIRALPAK® AGP, HSA, or CBH HPLC guard cartridge, 1 cm length by 2, 3 or 4 mm I.D. Requires holder 58159AST.

## Chiral HPLC & SFC Columns

Protection for Chiral HPLC Columns: *Column Protection for Astec Chiral HPLC Columns (CHIROBIOTIC®, CYCLOBOND®, Cellulose DMP, P-CAP™, CLC)*

### Column Protection for Astec Chiral HPLC Columns (CHIROBIOTIC®, CYCLOBOND®, Cellulose DMP, P-CAP™, CLC)

Guards are available for Astec CHIROBIOTIC®, CYCLOBOND, Cellulose, and P-CAP columns in the following dimensions:

- 2 cm x 4.0 mm I.D. packed guard cartridges that use both stand-alone (21150AST) or direct-connect (504254) holders. The choice depends on user preferences. These holders accommodate standard 1/16" O.D. Valco-type nuts and ferrules, and have a freely-rotating inlet/outlet port that allows for complete rotation of tubing on one side of the holder. The direct-connect style holder attaches directly to Supelco or Astec 3.0, 4.0 and 4.6 mm I.D. columns.
- 2 cm x 1.0 mm I.D. packed guard columns for protecting 2.1 mm and lesser I.D. columns. The 1.0 mm I.D. columns do not require a holder.

Both of these guard designs use 1/16" O.D. tubing, nuts and ferrules (not included). You can couple the stand-alone holders to the analytical column using a short piece of 1/16" tubing, or use the convenient column couplers. A list of suggested hardware appears in the table below. Our complete hardware offering appears in the HPLC Accessories section of this catalog. Other guard dimensions, including **preparative guards**, are available. Please inquire.



Stand-alone HPLC guard column holder (21150AST) and representative 2 cm length Supelguard or Astec guard cartridge.



Direct-connect style holder (504254) for 2 cm length Supelguard and Astec guard cartridges. Connects to 3, 4 and 4.6 mm I.D. Supelco or Astec HPLC columns.

|   | Cat. No. | Qty   |
|---|----------|-------|
| <b>Coupler for Legacy Guard Column Holder</b>   |          |       |
| PEEK, I.D. 0.010 in. x O.D. 1/16 in. x Overall L 1 in.  | 54986    | 1 ea  |
| <b>Supelguard™ Guard Cartridge Holder</b>   |          |       |
| Stand-Alone (Swivel-type), for use with Supelguard cartridges (2 cm L. x 2 to 4.6 mm I.D.)    | 21150AST | 1 ea  |
| Direct-Connect (Swivel-type), for use with Supelguard cartridges (2 cm L. x 3 to 4.6 mm I.D.) | 504254   | 1 ea  |
| Stand-Alone, for use with Supelguard cartridges (1 cm L. x 10.0 mm I.D.)                      | 567499-U | 1 ea  |
| Stand-Alone, for use with Supelguard cartridges (1 cm L. x 21.2 mm I.D.)                      | 581392-U | 1 ea  |
| <b>Stainless Steel HPLC Fittings</b>  |          |       |
| ferrule, configured for 1/16 in. tubing   | 22988    | 10 ea |
| nut, for for 1/16 in. tubing  | 22990-U  | 10 ea |
| <b>Stainless Steel 1/16 in. Capillary Tubing</b>  |          |       |
| L 5 cm x O.D. 1/16 in. x I.D. 0.007 in.   | 56713    | 1 ea  |
| <b>HPLC Column Coupler</b>  |          |       |
| PEEK, I.D. 0.007 in. x O.D. 1/16 in. x Overall L 1 in.  | 58162AST | 1 ea  |

### Column Protection for Protein-based HPLC Columns

Guards for the CHIRALPAK® AGP, HSA, and CBH columns are supplied in 1 cm length by 2.0, 3.0 or 4.0 mm I.D. cartridge format in packs of 2. They require a holder (58159AST) that is sold separately. The holder accommodates standard 1/16" O.D. tubing. You can couple the holder to the analytical column using a short piece of 1/16" tubing, or use the convenient column couplers. A list of suggested hardware appears in the table below. Our complete hardware offering appears in the HPLC Accessories section of this catalog.



Right, holder (58159AST) and left, representative CHIRALPAK® AGP, HSA, or CBH 1 cm length guard cartridges.

|   | Cat. No. | Qty   |
|---|----------|-------|
| <b>Coupler for Legacy Guard Column Holder</b>   |          |       |
| PEEK, I.D. 0.010 in. x O.D. 1/16 in. x Overall L 1 in.                                | 54986    | 1 ea  |
| <b>Guard Column Holder for CHIRALPAK® AGP, HSA, and CBH</b>                           |          |       |
| for use with CHIRALPAK AGP, CBH, and HSA 1 cm x 2.0, 3.0, and 4.0 mm guard cartridges | 58159AST | 1 ea  |
| <b>HPLC Column Coupler</b>  |          |       |
| PEEK, I.D. 0.007 in. x O.D. 1/16 in. x Overall L 1 in.                                | 58162AST | 1 ea  |
| <b>Stainless Steel 1/16 in. Capillary Tubing</b>                                      |          |       |
| L 5 cm x O.D. 1/16 in. x I.D. 0.007 in.   | 56713    | 1 ea  |
| <b>Stainless Steel HPLC Fittings</b>  |          |       |
| ferrule, configured for 1/16 in. tubing   | 22988    | 10 ea |
| nut, for for 1/16 in. tubing  | 22990-U  | 10 ea |

### Pre-column Filters

Our applications chemists have found the Supelco ColumnSaver pre-column filter to be very good at protecting the column from particulate matter in the sample and mobile phase. This simple in-line filter comes in two frit porosities, 0.5 and 2 micron. More information on this product can be found in the HPLC Accessories section.

#### Supelco® ColumnSaver Precolumn Filter



| Description | Cat. No. | Qty   |
|-------------|----------|-------|
| 0.5 µm      | 55214-U  | 10 ea |
| 2.0 µm      | 55215-U  | 10 ea |

## Chiral HPLC &amp; SFC Columns

Chiral HPLC Column Test Mixes

## Chiral HPLC Column Test Mixes

Use these test mixes to evaluate the performance of your chiral HPLC column and make sure it is operating effectively. Consult the QA report supplied with the column, or call or email our Technical Services for mobile phase and expected performance criteria.

- 5-Methyl-5-phenylhydantoin is used to evaluate the performance of Astec CHIROBIOTIC® columns. The mobile phase is 100% methanol and detection is by UV at 254 nm. The test mix is supplied as a racemic mixture of two enantiomers.
- Trans-stilbene oxide (TSO) is used to evaluate the performance of Astec Cellulose DMP and other polysaccharide-based chiral HPLC columns. The recommended mobile phase is 10:90 IPA:hexane and detection is by UV at 220 nm. The test mix is supplied as a racemic mixture of the two TSO enantiomers and 1,3,5-tri-tert-butylbenzene is a void volume marker.

## Chiral Test Mix for Astec CHIROBIOTIC®

5-Methyl-5-phenylhydantoin  
 $C_{10}H_{10}N_2O_2$  FW 190.20

## ▶ analytical standard

5-Methyl-5-phenylhydantoin is used to evaluate the performance of Astec CHIROBIOTIC® chiral HPLC columns. The mobile phase is 100% methanol and detection is by UV at 254 nm. The test mix is supplied as a racemic mixture of two enantiomers in methanol.

## Components

5-Methyl-5-phenylhydantoin 5000 µg/mL

40095-U

1 mL

## Chiral Normal Phase Test Mix

## ▶ 30 µg/mL each component in hexane, analytical standard

Trans-stilbene oxide (TSO) is used to evaluate the performance of Astec Cellulose DMP and other polysaccharide-based chiral HPLC columns. The recommended mobile phase is 10:90 IPA:hexane and detection is by UV at 220 nm. The test mix is supplied as a racemic mixture of the two TSO enantiomers with 1,3,5-tri-tert-butylbenzene as a void volume marker. The solvent is hexane.

## Components

trans-Stilbene oxide  
 1,3,5-tri-t-Butylbenzene

40119-U

1 mL

## Chiral GC Columns

The acquisition of Astec by Sigma-Aldrich in 2006 merged two well-established lines of CSPs for enantiomer separations by capillary GC. Both Supelco DEX™ and Astec CHIRALDEX® are based on cyclodextrins and exhibit complementary selectivity. All columns are manufactured to deliver high resolution and analyte response, low bleed, and long column life.

Our chiral GC columns currently comprise:

- **Astec CHIRALDEX®** - Developed by Astec, the CHIRALDEX line of chiral capillary GC columns use specialized phase chemistries that include unique derivatives of cyclodextrins with a broad range of selectivities. The "TA" (trifluoroacetyl) derivatives possess the most popular and unique chemistry.
- **Supelco DEX™** - Developed by Supelco, DEX capillary GC columns comprise derivatized cyclodextrins that are able to perform many enantiomeric separations.



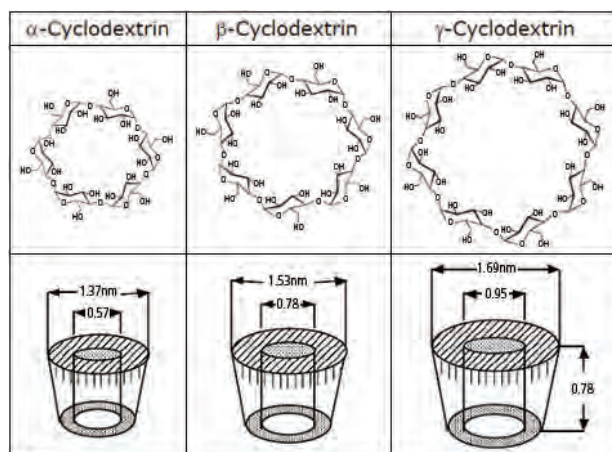
## Related Information

Request free literature by phone, fax, or visit [sigma-aldrich.com/literature](http://sigma-aldrich.com/literature).

| No.     | Title                     |
|---------|---------------------------|
| T411101 | Supelco Chiral GC Columns |

## Cyclodextrin-based GC CSPs

Cyclodextrins (CDs) are macromolecules composed of 6 or more D (+)-glucose residues bonded through  $\alpha$ -glycosidic linkages. They are classified according to the number of glucose residues they contain:  $\alpha$ -cyclodextrins (six residues),  $\beta$ -cyclodextrins (seven residues), and  $\gamma$ -cyclodextrins (eight residues). The three different sizes separate analytes over a wide range of molecular size. All hydroxyl groups, whether at the 2, 3 or 6 position, can be selectively modified with a derivative to impart unique physical properties and inclusion selectivities. Unlike LC, there is no enantioselectivity in chiral GC without derivatization of the CD.



Cyclodextrin molecules showing dimensions

## Chiral GC Columns

Cyclodextrin-based GC CSPs

## Cyclodextrins—Physical Properties

| Cyclodextrin | Glucose Units | Stereogenic Centers | MW   | Cavity (nm) |
|--------------|---------------|---------------------|------|-------------|
| Alpha        | 6             | 30                  | 972  | 0.57        |
| Beta         | 7             | 35                  | 1135 | 0.78        |
| Gamma        | 8             | 40                  | 1297 | 0.95        |

In cyclodextrin-based capillary GC CSPs, the derivatized cyclodextrin is used neat or after being doped at controlled percentages into a polysiloxane polymer matrix. Cyclodextrin GC CSPs are grouped into three general categories:

**Group 1: Surface Interactions, Complex Derivatives**

Because the predominant mechanism of retention for phases in this group is based on surface interaction,  $\gamma$ -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 2,3,6-position hydroxyl functional groups available for derivatization. High derivative concentration is beneficial for maximizing surface interactions. This group includes the highly popular CHIRALDEX® G-TA.

**Group 2: Surface/Inclusion Interactions, Simple Derivatives**

This group includes the diacetyl (Supelco DEX 225) and dimethyl (Supelco DEX 325) derivatives. The  $\beta$ -cyclodextrin has shown the greatest applicability for phases with these derivatives.

**Group 3: Inclusion Interactions**

The third group of GC CSPs relies on inclusion interactions for retention. The fact that there are three different size cyclodextrins ( $\alpha$ ,  $\beta$  and  $\gamma$ ) allows for separation of a wide variety of different size analytes. This group includes CHIRALDEX® DM and CHIRALDEX® DA, and Supelco DEX 110 and Supelco DEX 120.

## CD Derivatives in the Supelco Chiral GC Line:

| Derivative      | Phase              | Cyclodextrin Type |                |                  |
|-----------------|--------------------|-------------------|----------------|------------------|
|                 |                    | $\alpha$ (alpha)  | $\beta$ (beta) | $\gamma$ (gamma) |
| Butyryl         | Astec CHIRALDEX BP |                   |                | ✓                |
| Diacetyl        | Supelco DEX 225    | ✓                 | ✓              | ✓                |
| Dialkyl         | Astec CHIRALDEX DA | ✓                 | ✓              | ✓                |
| Dimethyl        | Astec CHIRALDEX DM |                   | ✓              | ✓                |
| Dimethyl        | Supelco DEX 325    | ✓                 | ✓              | ✓                |
| Dipropionyl     | Astec CHIRALDEX DP |                   | ✓              | ✓                |
| S-Hydroxypropyl | Astec CHIRALDEX PH |                   | ✓              |                  |
| Permethylated   | Astec CHIRALDEX PM |                   | ✓              |                  |
| Permethylated   | Supelco DEX 110    |                   | ✓              |                  |
| Permethylated   | Supelco DEX 120    | ✓                 | ✓              | ✓                |
| Propionyl       | Astec CHIRALDEX PN |                   |                | ✓                |
| Trifluoroacetyl | Astec CHIRALDEX TA | ✓                 | ✓              | ✓                |

## Choosing a Chiral GC Column

Supelco offers the most extensive line of chiral capillary GC columns in the industry. Our two premium lines of GC CSPs, Supelco DEX and Astec CHIRALDEX®, comprise a wide range of cyclodextrin derivatives with complementary selectivity. All are stable, high boiling liquids and make effective CSPs for enantiomer separations by GC. Selectivity is a function of the derivative, the degree of derivatization, the position of the derivative on the cyclodextrin, whether the derivatized cyclodextrin is used neat or doped into a polysiloxane, and if doped, at what percentage. Certain CSPs are more selective for given molecular structures and often more than one will achieve a separation. CSPs may be chosen to optimize resolution, but also elution order or analysis time. It is conventional practice to screen multiple CSPs when developing a new method. We offer **column screening kits** at very attractive prices for this purpose.

## Supelco Chiral Capillary GC Column Selection Guidelines

|  | CHIRALDEX TA | CHIRALDEX DP | CHIRALDEX PN | CHIRALDEX BP | CHIRALDEX DM | Supelco DEX 325 | Supelco DEX 225 | CHIRALDEX PM | Supelco DEX 120 | Supelco DEX 110 | CHIRALDEX DA | CHIRALDEX PH    | $\alpha$ -Cyclodextrin | $\beta$ -Cyclodextrin | $\gamma$ -Cyclodextrin |
|--|--------------|--------------|--------------|--------------|--------------|-----------------|-----------------|--------------|-----------------|-----------------|--------------|-----------------|------------------------|-----------------------|------------------------|
|  | By Chemistry |              |              |              |              |                 |                 |              |                 |                 |              | By Cyclodextrin |                        |                       |                        |
| Oxygen containing analytes in the form of alcohols, ketones, acids, aldehydes, and lactones; halogenated compounds                         | ✓            |              |              |              |              |                 |                 |              |                 |                 |              |                 |                        |                       |                        |
| Aliphatic and aromatic amines; aliphatic and some aromatic esters; polar racemates   |              | ✓            |              |              |              |                 |                 |              |                 |                 |              |                 |                        |                       |                        |
| Lactones and aromatic amines; epoxides; styrene oxide  |              |              | ✓            |              |              |                 |                 |              |                 |                 |              |                 |                        |                       |                        |
| 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, terpeneols |              |              |              |              |              |                 |                 |              |                 |                 |              |                 |                        | ✓                     |                        |
| $\alpha$ -BHC, carvone, carboxylic acids, methamphetamine  |              |              |              |              |              |                 |                 |              |                 |                 |              |                 |                        |                       | ✓                      |

## Chiral GC Columns

## Chiral GC Column Screening Kits

## Chiral GC Column Screening Kits

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



## Related Information

Need help choosing the right chiral HPLC or GC column?  
Let our Chiral Services group do the work for you.

## Astec CHIRALDEX® GC Column Screening Kit

The Astec CHIRALDEX® column kit contains three GC CSPs that cover the widest possible range of enantioselectivity: CHIRALDEX® G-TA, B-DM, and B-DA, in the popular 30 m x 0.25 mm, 0.12 µm d<sub>i</sub> dimensions. The CHIRALDEX® G-TA separates the greatest number of enantiomers, often with high enantioselectivity. The CHIRALDEX® B-DM separates the widest variety of different structural types. The CHIRALDEX® B-DA is best suited for larger multi-ring structures. Eighty-five percent of analytes that exhibit enantioselectivity on cyclodextrin based chiral stationary phases will give enantioselectivity on one of these phases. The kit provides considerable savings over the columns purchased separately.

**Kit contents:** One 30 m x 0.25 mm I.D., 0.12 µm column of each type: CHIRALDEX® G-TA, B-DM, and B-DA

## Astec CHIRALDEX® GC Column Screening Kit

| Description | Cat. No. | Qty   |
|-------------|----------|-------|
| 30 m kit    | 71030AST | 1 kit |

## Supelco DEX™ GC Column Screening Kit

These Supelco DEX kits provide the tools you need to perform most chiral separations. Confirm identities of enantiomers by monitoring elution order changes (enantioreversal) from one column to another. In combination, the columns in the two kits span the full range of Supelco DEX column enantioselectivity. Compare the savings to the columns purchased separately.

**Kit I:** One 30 m x 0.25 mm I.D., 0.25 µm column of each type: α-DEX 120, β-DEX 120 and γ-DEX 120

**Kit II:** One 30 m x 0.25 mm I.D., 0.25 µm column of each type: β-DEX 120, β-DEX 225, γ-DEX 225 and β-DEX 325

## Supelco DEX™ GC Column Screening Kit

| Description | Cat. No. | Qty   |
|-------------|----------|-------|
| kit I       | 24340    | 1 kit |
| kit II      | 24328-U  | 1 kit |

## Group 1: Surface Interactions, Complex Derivatives

Sigma-Aldrich is the only supplier of complex derivatives for chiral GC. There are four members in this important group:

- Astec CHIRALDEX® TA (Trifluoroacetyl derivatives)
- Astec CHIRALDEX® PN (Propionyl derivatives)
- Astec CHIRALDEX® DP (Dipropionyl derivatives)
- Astec CHIRALDEX® BP (Butyryl derivatives)

Because the predominant mechanism of retention for phases in this group is based on surface interaction, the gamma-cyclodextrin, with 8 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 2,3,6-position 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 chiral stationary phases. This phase does not contain a polysiloxane 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 G-TA is the **Astec CHIRALDEX® G-PN**. It functions like the G-TA but shows higher selectivity toward certain amines (amphetamine, methamphetamine). This phase is more stable to moisture than the 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 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-DP, G-PN, and G-BP often allow for major enhancements in chiral and achiral selectivity when changing from one phase to another.*

## Trifluoroacetyl (TA) Cyclodextrin Derivatives

## Astec CHIRALDEX® A-TA, B-TA, and G-TA

Trifluoroacetylation of the 3-hydroxyl group 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. Highly selective for halogenated compounds. Astec CHIRALDEX® G-TA is the most popular phase in our chiral GC line.

## Features

- Phase: 2,6-di-O-pentyl-3-trifluoroacetyl derivative of α-, β-, or γ-cyclodextrin
- Separates the widest variety and greatest number of enantiomers
- Unique retention behavior
- Extraordinary versatility and chiral selectivity
- Sensitive to moisture, but can be regenerated
- Thermal limit 180 °C (isothermal or programmed)

## Analytes

- Useful for separating 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, and sulphoxides

## Mechanism Observations

- Strong dipole-dipole interactions
- Longer alkyl chain; greater retention; increase in enantioselectivity up to C<sub>4</sub>/C<sub>5</sub>
- Halogens known to favor cavity interaction
- Dipole-dipole interactions are commonly identified in the mechanism of separation for CHIRALDEX® 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.



## Chiral GC Columns

Group 1: Surface Interactions, Complex Derivatives: *Trifluoroacetyl (TA) Cyclodextrin Derivatives*

### Size Selectivity

- The  $\gamma$ -TA (CHIRALDEX G-TA) derivative has proven to exhibit a wider chiral selectivity and usefulness than the  $\beta$  (CHIRALDEX® B-TA) 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 we have tested, the split between CHIRALDEX® G-TA and CHIRALDEX® B-TA is approximately 55/35 with only 10% of the separations accomplished on the  $\alpha$  (CHIRALDEX® A-TA).

### Astec CHIRALDEX® A-TA Capillary GC Column

Incorporates a phase consisting of a 2,6-di-O-pentyl-3-trifluoroacetyl derivative of  $\alpha$ -cyclodextrin. This phase exhibits high selectivity for oxygen-containing analytes in the form of alcohols, ketones, acids, aldehydes and lactones. It is also highly selective for halogenated compounds.

#### Temp. Limits:

- 10 °C to 180 °C, isothermal and programmed

phase ..... non-bonded; 2,6-di-O-pentyl-3-trifluoroacetyl derivative of  $\alpha$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 20         | 500        | 73002AST | 1 ea |
|           | 0.12                | 30         | 500        | 73003AST | 1 ea |
|           | 0.12                | 40         | 500        | 73004AST | 1 ea |
|           | 0.12                | 50         | 500        | 73005AST | 1 ea |

### Astec CHIRALDEX® B-TA Capillary GC Column

Incorporates a phase consisting of a 2,6-di-O-pentyl-3-trifluoroacetyl derivative of  $\beta$ -cyclodextrin. This phase exhibits high selectivity for oxygen-containing analytes in the form of alcohols, ketones, acids, aldehydes and lactones. It is also highly selective for halogenated compounds.

#### Temp. Limits:

- 10 °C to 180 °C isothermal and programmed

phase ..... non-bonded; 2,6-di-O-pentyl-3-trifluoroacetyl derivative of  $\beta$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 20         | 500        | 73022AST | 1 ea |
|           | 0.12                | 30         | 500        | 73023AST | 1 ea |
|           | 0.12                | 40         | 500        | 73024AST | 1 ea |

### Astec CHIRALDEX® G-TA Capillary GC Column

Astec CHIRALDEX G-TA is the first choice in the Group 1 CSPs (Surface Interactions, Complex Derivatives). This phase has been shown to be the most broadly-selective phase for the pharmaceutical industry, especially for 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 chiral stationary phases. G-TA has also been used to separate parent drug enantiomers and their metabolites. G-TA has its highest selectivity for oxygen-containing analytes like 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. It is also highly selective for halogenated compounds.

#### Temp. Limits:

- 10 °C to 180 °C isothermal and programmed

phase ..... non-bonded; 2,6-di-O-pentyl-3-trifluoroacetyl derivative of  $\gamma$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 10         | 500        | 73031AST | 1 ea |
|           | 0.12                | 20         | 500        | 73032AST | 1 ea |
|           | 0.12                | 30         | 500        | 73033AST | 1 ea |
|           | 0.12                | 40         | 500        | 73034AST | 1 ea |
|           | 0.12                | 50         | 500        | 73035AST | 1 ea |

### Astec CHIRALDEX® TA Series Column Regeneration Reagents

The trifluoroacetic anhydride (TFA) derivative of the cyclodextrins used in CHIRALDEX® TA phases can hydrolyze in the presence of moisture at and above room temperature. Sources of moisture include the sample, carrier gas, injection solvents and the atmosphere if stored unsealed. For long column life, ensure that the carrier gas line has an effective moisture trap, all sample extracts are moisture free, the injection solvent is anhydrous and the column is stored properly when not in use by flame-sealing the ends. Guard columns will also help protect the column from the damaging effects of moisture. This regeneration method does not restore retention due to loss of phase, it restores enantioselectivity lost by hydrolysis of the acetyl derivative. Instructions for regeneration of CHIRALDEX® TA columns are available by contacting [techservice@sial.com](mailto:techservice@sial.com).

For flame-sealing the ends of the column, we offer the Microflame Gas Torch Set (Cat. No. 22969). Details of this item can be found in the Laboratory Supplies section of this catalog.

| Description                                      | Cat. No.      | Qty       |
|--|---------------|-----------|
| Trifluoroacetic anhydride, for GC derivatization | 33164         | 25 mL     |
| Trifluoroacetic anhydride                        | 33165-U       | 10 × 1 mL |
|  | 33164         | 25 mL     |
| Methyl Red, ACS reagent, crystalline             | 250198-25G    | 25 g      |
|  | 250198-100G   | 100 g     |
| Sodium hydroxide, ACS reagent, ≥97.0%, pellets   | 221465-25G    | 25 g      |
|  | 221465-500G   | 500 g     |
|  | 221465-6X500G | 6 × 500 g |
|  | 221465-2.5KG  | 2.5 kg    |
|  | 221465-12KG   | 12 kg     |
|  | 221465-50KG   | 50 kg     |

### Propionyl (PN) Cyclodextrin Derivatives

#### Astec CHIRALDEX® G-PN

A modified version of the CHIRALDEX® G-TA, Astec CHIRALDEX® G-PN exhibits high selectivity for lactones, epoxides, and aromatic amines. Additionally, the analysis of styrene oxide can be accomplished on this phase (this analyte degrades on the TA phases). This phase is more stable to moisture than the CHIRALDEX® G-TA.

#### Features

- Phase: 2,6-di-O-pentyl-3-propionyl derivative of  $\gamma$ -cyclodextrin
- Suitable for epoxide separations
- High selectivity for lactones
- High selectivity for aromatic amines (i.e. amphetamine, methamphetamine)
- More stable to moisture than the CHIRALDEX® TA phases
- Thermal limit 200/220°C (isothermal/programmed)

#### Analytes

- Epoxides, aromatic amines (amphetamine/methamphetamine), >C6 alcohols and lactones

#### 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 CHIRALDEX® G-PN shows very little size selectivity.

## Chiral GC Columns

Group 1: Surface Interactions, Complex Derivatives: *Propionyl (PN) Cyclodextrin Derivatives*

## NEW PRODUCTS

**Astec CHIRALDEX® G-PN Capillary GC Column**

Incorporates a phase consisting of a 2,6-di-O-pentyl-3-propionyl derivative of  $\gamma$ -cyclodextrin. This phase exhibits high selectivity for lactones and aromatic amines. It is also suitable for epoxide separations. Additionally, the analysis of styrene oxide can be accomplished on this phase (this analyte degrades on the TA phases).

GC capillary column  
fused silica

**Temp. Limits:**

- 10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,6-di-O-pentyl-3-propionyl derivative of  $\gamma$ -cyclodextrin

| I.D.<br>(mm) | d <sub>f</sub> (μm) | Length<br>(m) | Beta<br>Value | Cat. No. | Qty  |
|--------------|---------------------|---------------|---------------|----------|------|
| 0.25         | 0.12                | 30            | 500           | 74033AST | 1 ea |

**Dipropionyl (DP) Cyclodextrin Derivatives****Astec CHIRALDEX® B-DP and G-DP**

This derivative demonstrates good selectivity for a wide range of analytes except alcohols and epoxides where the CHIRALDEX® G-TA remains the best choice. The CHIRALDEX® G-DP has shown very high selectivity for both aromatic and aliphatic amines and for aliphatic and some aromatic esters. Both hydrolytic and temperature stability are better than CHIRALDEX® G-TA, and for bulky fused ring structures the CHIRALDEX® G-DP is better than the CHIRALDEX® B-DP.

**Features**

- Phase: 2,3-di-O-propionyl-6-t-butyl silyl derivative of  $\beta$ - or  $\gamma$ -cyclodextrin
- Broad chiral selectivity
- Good hydrolytic stability
- High efficiency and resolution at low retention times for polar racemates
- Thermal limit 200/220 °C (isothermal/programmed)

**Mechanism Observations**

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

**Analytes**

- Excellent for aromatic and aliphatic amines
- Good for many aliphatic and some aromatic esters

**Size Selectivity**

- Speed and sample capacity point to a surface-type mechanism for very polar racemates. Large, bulky molecules still require a larger surface area than the 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. The choice between beta and gamma is compound dependent for this phase.

## NEW PRODUCTS

**Astec CHIRALDEX® B-DP Capillary GC Column**

Incorporates a phase consisting of a 2,3-di-O-propionyl-6-t-butyl silyl derivative of  $\beta$ -cyclodextrin. This phase exhibits good hydrolytic stability, broad chiral selectivity, and is excellent for aliphatic and aromatic amines. It is also good for many aliphatic and some aromatic esters as well as exhibiting high efficiency and resolution at low retention times for polar racemates.

**Temp. Limits:**

- 10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,3-di-O-propionyl-6-t-butyl silyl derivative of  $\beta$ -cyclodextrin

| I.D.<br>(mm) | d <sub>f</sub> (μm) | Length<br>(m) | Beta<br>Value | Cat. No. | Qty  |
|--------------|---------------------|---------------|---------------|----------|------|
| 0.25         | 0.12                | 30            | 500           | 78023AST | 1 ea |

**Astec CHIRALDEX® G-DP Capillary GC Column**

Incorporates a phase consisting of a 2,3-di-O-propionyl-6-t-butyl silyl derivative of  $\gamma$ -cyclodextrin. The CHIRALDEX G-DP phase was designed 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, as it exhibits high efficiency and resolution at low retention times. G-DP demonstrates better hydrolytic and thermal stability than the CHIRALDEX G-TA.

**Temp. Limits:**

- 10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,3-di-O-propionyl-6-t-butyl silyl derivative of  $\gamma$ -cyclodextrin

| I.D.<br>(mm) | d <sub>f</sub> (μm) | Length<br>(m) | Beta<br>Value | Cat. No. | Qty  |
|--------------|---------------------|---------------|---------------|----------|------|
| 0.25         | 0.12                | 30            | 500           | 78033AST | 1 ea |

**Butyryl (BP) Cyclodextrin Derivatives****Astec CHIRALDEX® G-BP**

Astec CHIRALDEX® G-BP incorporates a phase consisting of a 2,6-di-O-pentyl-3-butyryl derivative of  $\gamma$ -cyclodextrin. It is a good general-purpose column. It is also especially useful for amino acids and is, therefore, a good substitute for the bonded amino acid-type chiral GC phases.

**Features**

- Phase: 2,6-di-O-pentyl-3-butyryl derivative of  $\gamma$ -cyclodextrin
- High selectivity for amino acids, amines, and furans
- High sample capacity
- Thermal limit 200/220 °C (isothermal/programmed)

**Mechanism Observations**

- Alkyl chain on analyte contributes to chiral recognition
- High sample capacity indicates primarily surface interactions

**Analytes**

- Amino acids, amines and furans

**Size Selectivity**

- The influence of the inclusion mechanism on selectivity is much reduced and capacities are, therefore, generally higher.

## Chiral GC Columns

Group 1: Surface Interactions, Complex Derivatives: *Butyryl (BP) Cyclodextrin Derivatives*

### NEW PRODUCTS

#### Astec CHIRALDEX® G-BP Capillary GC Column

Incorporates a phase consisting of a 2,6-di-O-pentyl-3-butyryl derivative of  $\gamma$ -cyclodextrin. This phase exhibits high selectivity for amino acids, amines, and furans.

GC capillary column  
fused silica

#### Temp. Limits:

- 10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,6-di-O-pentyl-3-butyryl derivative of  $\gamma$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 30         | 500        | 75033AST | 1 ea |

## Group 2: Surface/Inclusion Interactions, Simple Derivatives

There are three different derivatives in this group:

- Supelco DEX™ 225 (Diacetyl derivatives)
- Astec CHIRALDEX® DM and Supelco DEX™ 325 (Dimethyl derivatives)
- Astec CHIRALDEX® PM, Supelco DEX™ 110, and DEX™ 120 (Permethy derivatives)

The beta-cyclodextrin has shown the greatest applicability for phases with these derivatives. 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 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  $\beta$ -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 analytes with polar groups (such as tertiary amines). The main difference between these three phases is the concentration of the permethyl-derivatized cyclodextrin that is doped into the polysiloxane carrier.

### Diacetyl (225) Cyclodextrin Derivatives

#### Supelco DEX™ 225

The Supelco DEX 225 phases are modified forms of the DEX 325 phases, employing acetyl derivatives at the 2,3-positions instead of more traditional methyl derivatives. The chiral stationary phase in DEX 225 columns contains 2,3-di-O-acetyl-6-O-TBDMS derivatized  $\alpha$ ,  $\beta$ , or  $\gamma$  cyclodextrin embedded in an intermediate polarity phase. These columns provide unique selectivity for enantiomeric separations of small molecules: alcohols, aldehydes (e.g., 2-phenylpropionaldehyde), esters (e.g., methyl malate, methyl lactate), flavor compounds and ketones.

#### Features

- Phase: 25% 2,3-di-O-acetyl-6-O-TBDMS- $\alpha$ -,  $\beta$ - or  $\gamma$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)
- Thermal limit 230 °C (isothermal or programmed)

#### Analytes

- Alcohols, aldehydes (e.g., 2-phenylpropionaldehyde), esters (e.g., methyl malate, methyl lactate), flavor compounds, ketones

#### $\alpha$ -DEX™ 225

The chiral stationary phase in  $\alpha$ -DEX 225 columns contains 2,3-di-O-acetyl-6-O-TBDMS- $\alpha$ -cyclodextrin embedded in an intermediate polarity phase.

#### Temp. Limits:

- 30 °C to 230 °C

phase ..... non-bonded; 25% 2,3-di-O-acetyl-6-O-TBDMS- $\alpha$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.25                | 30         | 250        | 24311    | 1 ea |

#### $\beta$ -DEX™ 225

The Supelco  $\beta$ -DEX 225 is a modified form of the  $\beta$ -DEX 325 phase, and employs acetyl derivatives at the 2,3-positions instead of more traditional methyl derivatives. The chiral stationary phase in  $\beta$ -DEX 225 columns contains 2,3-di-O-acetyl-6-O-TBDMS- $\beta$ -cyclodextrin embedded in an intermediate polarity phase. These columns provide unique selectivity for enantiomeric separations of small molecules: alcohols, aldehydes (e.g., 2-phenylpropionaldehyde), esters (e.g. methyl malate, methyl lactate), flavor compounds and ketones.

#### Temp. Limits:

- 30 °C to 230 °C

phase ..... non-bonded; 25% 2,3-di-O-acetyl-6-O-TBDMS- $\beta$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.25                | 30         | 250        | 24348    | 1 ea |

#### $\gamma$ -DEX™ 225

The chiral stationary phase in  $\gamma$ -DEX 225 columns contains 2,3-di-O-acetyl-6-O-TBDMS- $\gamma$ -cyclodextrin embedded in an intermediate polarity phase.

#### Temp. Limits:

- 30 °C to 230 °C

phase ..... non-bonded; 25% 2,3-di-O-acetyl-6-O-TBDMS- $\gamma$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.25                | 30         | 250        | 24312    | 1 ea |

## Dimethyl (DM, 325) Cyclodextrin Derivatives

### Astec CHIRALDEX® B-DM and G-DM

The dimethyl derivative was designed to overlap with the applications of both Astec CHIRALDEX® PM and CHIRALDEX® PH series, having similar selectivity but with shorter retention times and better resolution.

#### Features

- Phase: 2,3-di-O-methyl-6-t-butyl silyl derivative of  $\beta$ - or  $\gamma$ -cyclodextrin
- Broad chiral selectivity
- Combines selectivity of CHIRALDEX® PM (permethyl) and CHIRALDEX® PH (hydroxypropyl)
- Short retention, high resolution
- $\beta$  derivative is broadly applicable
- Thermal limit 200/220 °C (isothermal/programmed)

#### Analytes

- Resolves aliphatic, olefinic and aromatic enantiomers

#### Mechanism Observations

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

## Chiral GC Columns

Group 2: Surface/Inclusion Interactions, Simple Derivatives: *Dimethyl (DM, 325) Cyclodextrin Derivatives*

## Size Selectivity

- Size selectivity is evident, implying that the inclusion mechanism plays a role in the separation mechanism, but does not dominate as it does in the CHIRALDEX® DA series. The  $\beta$  form covers a very broad range of molecular sizes and, therefore, has the widest applicability.

## Supelco DEX™ 325

The chiral stationary phase in DEX 325 columns is the 2,3-di-O-methyl-6-O-TBDMS derivative of  $\alpha$ -,  $\beta$ -, or  $\gamma$ -cyclodextrin embedded in an intermediate polarity phase. 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.

## Features

- Phase: 25% 2,3-di-O-methyl-6-O-TBDMS- $\alpha$ -,  $\beta$ -, or  $\gamma$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)
- Thermal limit 230 °C (isothermal or programmed)

## NEW PRODUCTS

## Astec CHIRALDEX® B-DM Capillary GC Column

Through special derivatization techniques, the concentration of the cyclodextrin in the CHIRALDEX B-DM has been substantially increased in the polysiloxane carrier. This phase is very useful for a number of free acids and bases. The B-DM is able to perform most of the separations done on a beta-permethylated phase, but with higher resolution. The selectivity of the B-DM covers applications of both the B-PM and B-PH phases, although with superior performance.

## Temp. Limits:

- 10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,3-di-O-methyl-6-t-butyl silyl derivative of  $\beta$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 20         | 500        | 77022AST | 1 ea |
|           | 0.12                | 30         | 500        | 77023AST | 1 ea |
|           | 0.12                | 40         | 500        | 77024AST | 1 ea |
|           | 0.12                | 50         | 500        | 77025AST | 1 ea |

## Astec CHIRALDEX® G-DM Capillary GC Column

Incorporates a phase consisting of a 2,3-di-O-methyl-6-t-butyl silyl derivative of  $\gamma$ -cyclodextrin. This phase exhibits broad chiral selectivity, resolving aliphatic, olefinic, and aromatic enantiomers. It combines the selectivities of the PM and PH phases.

## Temp. Limits:

- 10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,3-di-O-methyl-6-t-butyl silyl derivative of  $\gamma$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 30         | 500        | 77033AST | 1 ea |

 $\alpha$ -DEX™ 325

The chiral stationary phase in  $\alpha$ -DEX 325 columns contains 2,3-di-O-methyl-6-O-TBDMS- $\alpha$ -cyclodextrin embedded in an intermediate polarity phase.

## Temp. Limits:

- 30 °C to 230 °C

phase ..... non-bonded; 25% 2,3-di-O-methyl-6-O-TBDMS- $\alpha$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.25                | 30         | 250        | 24303    | 1 ea |

 $\beta$ -DEX™ 325

The chiral stationary phase in  $\beta$ -DEX 325 columns contains 2,3-di-O-methyl-6-O-TBDMS- $\beta$ -cyclodextrin embedded in an intermediate polarity phase. The Supelco  $\beta$ -DEX 325 is similar in both chemistry and use to the CHIRALDEX B-DM phase, the main difference being the concentration of the dimethyl-derivatized cyclodextrin that is doped into the polysiloxane carrier.

## Temp. Limits:

- 30 °C to 230 °C

phase ..... non-bonded; 25% 2,3-di-O-methyl-6-O-TBDMS- $\beta$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.25                | 30         | 250        | 24308    | 1 ea |

 $\gamma$ -DEX™ 325

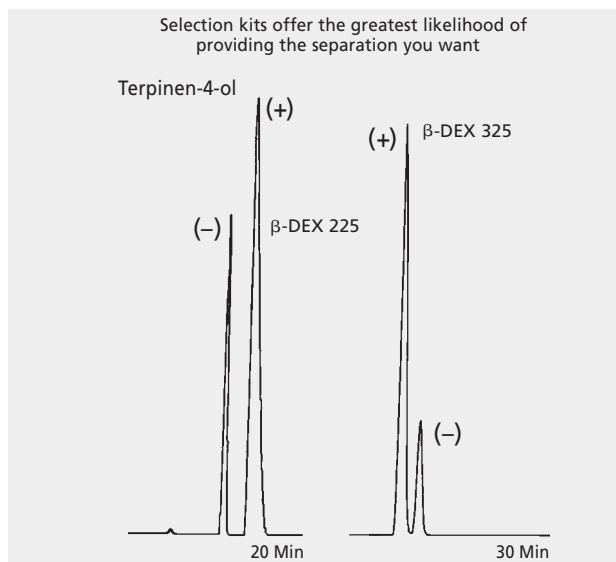
The chiral stationary phase in Supelco  $\gamma$ -DEX 325 columns contains 2,3-di-O-methyl-6-O-TBDMS- $\gamma$ -cyclodextrin embedded in an intermediate polarity phase.

## Temp. Limits:

- 30 °C to 230 °C

phase ..... non-bonded; 25% 2,3-di-O-methyl-6-O-TBDMS- $\gamma$ -cyclodextrin in SPB-20 poly(20% phenyl/80% dimethylsiloxane)

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.25                | 30         | 250        | 24306    | 1 ea |



## Chiral GC Columns

Group 2: Surface/Inclusion Interactions, Simple Derivatives: *Permethyl (PM, 110, 120) Cyclodextrin Derivatives*

### Permethyl (PM, 110, 120) Cyclodextrin Derivatives

The main difference between Astec CHIRALDEX B-PM and Supelco  $\beta$ -DEX 110 and Supelco  $\beta$ -DEX 120 is the concentration of the permethyl-derivatized cyclodextrin that is doped into the polysiloxane carrier. This confers selectivity differences between the columns.

#### Astec CHIRALDEX® B-PM

The permethylated  $\beta$ -cyclodextrin is a GC CSP with potential to separate a wide variety of racemates. The PM is especially valuable for separating certain hydrocarbons, like terpenes, some underivatized alcohols and diols, and some analytes with polar groups like tertiary amines.

#### Features

- Phase: 2,3,6-tri-O-methyl derivative of  $\beta$ -cyclodextrin
- Broad chiral selectivity
- Strong inclusion/size selectivity
- Thermal limit 200/220 °C (isothermal/programmed)

#### Analytes

- A general purpose column used for the separation of acids, alcohols, barbitals, diols, epoxides, esters, hydrocarbons, ketones, lactones, and terpenes.
- Some underivatized alcohols and diols as well as some analytes with polar groups, e.g. tertiary amines, show excellent separation.

#### Mechanism Observations

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

#### Size Selectivity

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

#### Supelco DEX™ 110 and 120

The chiral stationary phases in DEX 110 and 120 columns contain permethylated cyclodextrins embedded in an intermediate polarity stationary phase. These columns are recommended for the enantiomeric separation of a wide range of chiral compounds (ketones, esters, alkanes, alkenes, alcohols, acids, ethers, etc.). The 10% ( $\beta$ -DEX 110) and 20% ( $\beta$ -DEX 120)  $\beta$ -cyclodextrin content alters the elution order while maintaining similar enantioselectivity.

Because the elution order of a pair of enantiomers frequently reverses (enantioreversal) on a  $\gamma$ -DEX column compared to the elution order on an  $\alpha$ -DEX or  $\beta$ -DEX column, we recommend  $\gamma$ -DEX 120 columns as complements to  $\alpha$ -DEX 120 and  $\beta$ -DEX 120 columns. The  $\gamma$ -DEX is useful for enantiomeric differentiation of large analytes, e.g.  $\alpha$ -BHC, carvone, carboxylic acids, and methamphetamine.

#### Features

- Phase (110): 10% permethylated  $\beta$ -cyclodextrin in SPB-35 poly(35% diphenyl/65% dimethylsiloxane)
- Phase (120): 20% permethylated  $\alpha$ -,  $\beta$ -, or  $\gamma$ -cyclodextrin in SPB-35 poly(35% diphenyl/65% dimethylsiloxane)
- Thermal limit 230 °C (isothermal or programmed)

### NEW PRODUCTS

#### Astec CHIRALDEX® B-PM Capillary GC Column

The main difference between CHIRALDEX B-PM and the Supelco  $\beta$ -DEX 110 and Supelco  $\beta$ -DEX 120 phases is the concentration of the permethyl-derivatized cyclodextrin that is doped into the polysiloxane carrier. CHIRALDEX B-PM is a general-purpose column used for the separation of acids, alcohols, barbitals, diols, epoxides, esters, hydrocarbons, ketones, lactones and terpenes. Also, some underivatized alcohols and diols as well as some analytes with polar groups, i.e. tertiary amines, show excellent separation.

#### Temp. Limits:

- -10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,3,6-tri-O-methyl derivative of  $\beta$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 30         | 500        | 76023AST | 1 ea |
|           | 0.12                | 50         | 500        | 76025AST | 1 ea |

#### $\beta$ -DEX™ 110

The chiral stationary phase in  $\beta$ -DEX 110 columns contains permethylated  $\beta$ -cyclodextrin embedded in an intermediate polarity stationary phase. They are recommended for the enantiomeric separation of a wide range of chiral compounds (ketones, esters, alkanes, alkenes, alcohols, acids, ethers, etc.). The 10% ( $\beta$ -DEX 110) and 20% ( $\beta$ -DEX 120)  $\beta$ -cyclodextrin content alters the elution order while maintaining similar enantioselectivity.

#### Temp. Limits:

- 30 °C to 230 °C

phase ..... non-bonded; 10% permethylated  $\beta$ -cyclodextrin in SPB-35 poly(35% diphenyl/65% dimethylsiloxane)

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.25                | 30         | 250        | 24301    | 1 ea |
|           | 0.25                | 60         | 250        | 24302    | 1 ea |

#### $\alpha$ -DEX™ 120

Containing permethylated  $\alpha$ -cyclodextrin embedded in an intermediate polarity stationary phase, Supelco  $\alpha$ -DEX 120 columns provide unique selectivity for enantiomeric separations of small molecules. They are also recommended for separating positional isomers (phenols, xylenes, etc.).

#### Temp. Limits:

- 30 °C to 230 °C

phase ..... non-bonded; 20% permethylated  $\alpha$ -cyclodextrin in SPB-35 poly(35% diphenyl/65% dimethylsiloxane)

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.25                | 30         | 250        | 24310    | 1 ea |

#### $\beta$ -DEX™ 120

The chiral stationary phase in  $\beta$ -DEX 120 columns contains permethylated  $\beta$ -cyclodextrin embedded in an intermediate polarity stationary phase. They are recommended for the enantiomeric separation of a wide range of chiral compounds (ketones, esters, alkanes, alkenes, alcohols, acids, ethers, etc.). The 10% ( $\beta$ -DEX 110) and 20% ( $\beta$ -DEX 120)  $\beta$ -cyclodextrin content alters the elution order while maintaining similar enantioselectivity.

#### Temp. Limits:

- 30 °C to 230 °C

phase ..... non-bonded; 20% permethylated  $\beta$ -cyclodextrin in SPB-35 poly(35% phenyl/65% dimethylsiloxane)

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.25                | 30         | 250        | 24304    | 1 ea |
|           | 0.25                | 60         | 250        | 24305-U  | 1 ea |



## Chiral GC Columns

Group 2: Surface/Inclusion Interactions, Simple Derivatives: *Permethyl (PM, 110, 120) Cyclodextrin Derivatives***γ-DEX™ 120**

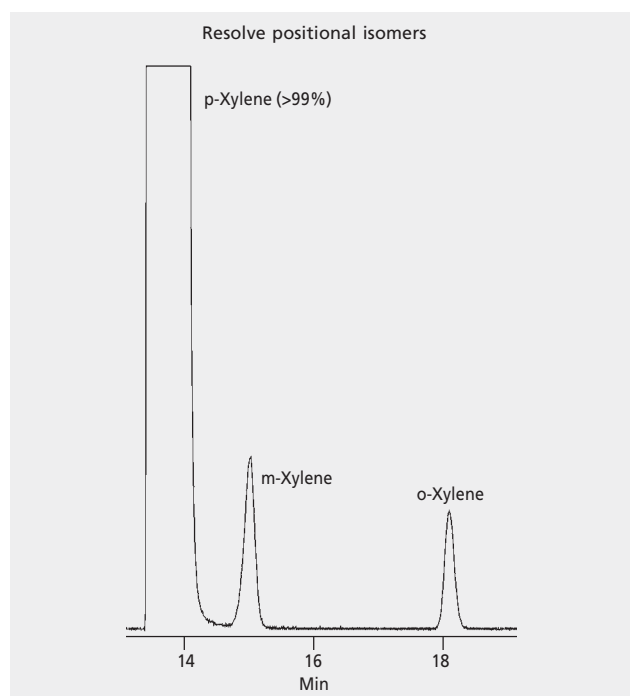
Because the elution order of the members of a chiral pair frequently reverses (enantioreversal) on a γ-DEX column compared to the elution order on an α-DEX or β-DEX column, we recommend γ-DEX 120 columns as complements to α-DEX 120 and β-DEX 120 columns. γ-DEX is useful for enantiomeric differentiation of large analytes, i.e. α-BHC, carvone, carboxylic acids and methamphetamine.

**Temp. Limits:**

- 30 °C to 230 °C

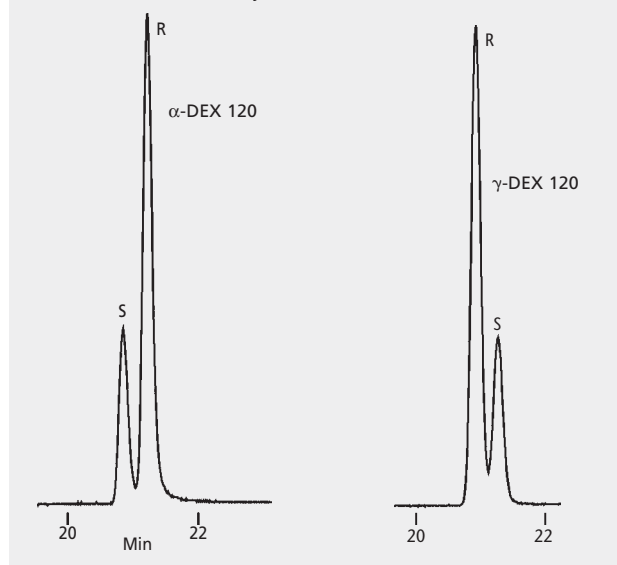
phase ..... non-bonded; 20% permethylated γ-cyclodextrin in SPB-35 poly(35% phenyl/65% dimethylsiloxane)

| I.D.<br>(mm) | d <sub>r</sub><br>(μm) | Length<br>(m) | Beta<br>Value | Cat. No. | Qty  |
|--------------|------------------------|---------------|---------------|----------|------|
| 0.25         | 0.25                   | 30            | 250           | 24307    | 1 ea |



column: α-DEX 120, 30 m × 0.25 mm I.D., 0.25 μm (24310)  
 oven: 50 °C  
 inj.: 80 °C  
 det.: FID, 300 °C  
 carrier gas: helium, 30 cm/sec  
 sample: 0.6 μL each analyte (neat), split (100:1)

Use γ-DEX to reverse elution order for many compounds  
 (methyl mandelate shown)



column: α-DEX 120 and γ-DEX 120, 30 m × 0.25 mm I.D., 0.25 μm  
 α-DEX 120 (24310), γ-DEX 120 (24307)  
 oven: 130 °C  
 inj.: 250 °C  
 det.: FID, 300 °C  
 carrier gas: helium, 35 cm/sec  
 sample: 1 μL methylene chloride (1 mg/mL each analyte), split (100:1)

**Group 3: Inclusion Interactions**

The third group relies on inclusion interactions for retention mechanism. There are two derivatives in this group:

- Astec CHIRALDEX® DA (Dialkyl derivatives)
- Astec CHIRALDEX® PH (S-Hydroxypropyl derivatives)

The fact that there are three different size cyclodextrins (α, β, and γ) 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 CHIRALDEX® DA phases most effectively separate multi-ring analytes, analysis temperatures are often higher than 150 °C. A key application area for this phase is fingerprinting 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 CHIRALDEX® B-DA phase.

## Chiral GC Columns

Group 3: Inclusion Interactions: *Dialkyl (DA) Cyclodextrin Derivatives*

### Dialkyl (DA) Cyclodextrin Derivatives

#### Astec CHIRALDEX® A-DA, B-DA, and G-DA

The dipentylated cyclodextrin derivatives show pronounced selectivity differences based on the size, shape and functionality of the analyte. Strong evidence exists for inclusion complexation as the basic driving mechanism and, therefore, resolution is affected by sample load.

The most popular member of this group is Astec CHIRALDEX B-DA. It requires minimally two ring structures, one of which is unsaturated (aromatic)  $\alpha$ ,  $\beta$  to the stereogenic center (examples include fluoxetine, methylphenidate, chlorpheniramine). Inclusion complexation or proper fit between the analyte and cyclodextrin cavity is the dominant enantioselectivity mechanism for the CHIRALDEX DA series of columns. There must be an includable group  $\alpha$  or  $\beta$  to the stereogenic center for chiral recognition. Since the Astec 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 (fluoxetine acetyl derivative).

#### Features

- Phase: 2,6-di-O-pentyl-3-methoxy derivative of  $\alpha$ -,  $\beta$ -, or  $\gamma$ -cyclodextrin
- Hydrophobic surface
- Pronounced selectivity differences based on analyte size, shape and functionality
- Different selectivity from other cyclodextrin derivatives
- Thermal limit 200/220 °C (isothermal/programmed)

#### Analytes

- Useful for separating heterocyclic amines

#### Mechanism Observations

- Stronger inclusion for CHIRALDEX DA derivatives, 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 on this phase.

**NEW PRODUCTS**

#### Astec CHIRALDEX® A-DA Capillary GC Column

Incorporates a phase consisting of a 2,6-di-O-pentyl-3-methoxy derivative of  $\alpha$ -cyclodextrin. This phase is good for separations of heterocyclic amines. It has different selectivity from other phases and often shows reversal in elution from the PH phases. MAOT = 200 °C isothermal, 220 °C programmed. GC capillary column fused silica

#### Temp. Limits:

- -10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,6-di-O-pentyl-3-methoxy derivative of  $\alpha$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 30         | 500        | 72003AST | 1 ea |

#### Astec CHIRALDEX® B-DA Capillary GC Column

CHIRALDEX B-DA requires that analytes possess a minimum of two ring structures, one of which is unsaturated (aromatic)  $\alpha$ ,  $\beta$  to the stereogenic center. Examples include fluoxetine, methylphenidate and chlorpheniramine. Inclusion complexation or proper fit between the analyte and cyclodextrin cavity is the dominant enantioselectivity mechanism for the DA series. There must be an includable group  $\alpha$  or  $\beta$  to the stereogenic center for chiral recognition. Since CHIRALDEX DA columns most effectively separate multi-ring analytes, analysis temperatures are often higher than 150°C. Enantioselectivity has been observed at temperatures >200°C (fluoxetine acetyl derivative).

#### Temp. Limits:

- -10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,6-di-O-pentyl-3-methoxy derivative of  $\beta$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 30         | 500        | 72023AST | 1 ea |

#### Astec CHIRALDEX® G-DA Capillary GC Column

Incorporates a phase consisting of a 2,6-di-O-pentyl-3-methoxy derivative of  $\gamma$ -cyclodextrin. This phase is good for separations of heterocyclic amines. It has different selectivity from other phases and often shows reversal in elution from the PH phases. MAOT = 200 °C isothermal, 220 °C programmed. GC capillary column fused silica

#### Temp. Limits:

- -10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; 2,6-di-O-pentyl-3-methoxy derivative of  $\gamma$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 30         | 500        | 72033AST | 1 ea |

### S-Hydroxypropyl (PH) Cyclodextrin Derivatives

#### Astec CHIRALDEX® 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

- Phase: (S)-2-hydroxy propyl methyl ether derivative of  $\beta$ -cyclodextrin
- Good general purpose chiral column
- Separates a wide variety of enantiomers
- Hydrophilic surface
- Resolves aliphatic, olefinic and aromatic enantiomers
- Thermal limit 200/220 °C (isothermal/programmed)

#### Analytes

- Saturated compounds with minimal functionality
- Saturated cyclics and bicyclics

#### Mechanism Observations

- Reduced influence of inclusion complexing
- Less size selectivity compared to DA derivatives
- Strong influence of temperature on selectivity

#### Size Selectivity

- Minimal size selectivity

## Chiral GC Columns

Group 3: Inclusion Interactions: *S*-Hydroxypropyl (PH) Cyclodextrin Derivatives

## NEW PRODUCTS

## Astec CHIRALDEX® B-PH Capillary GC Column

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 bicyclics. The CHIRALDEX PH series of columns shows less of a necessity for inclusion complexation for chiral recognition than the DA columns. This phase often shows a reversal of elution order (enantioreversal) compared to the B-DA phase.

## Temp. Limits:

- 10 °C to 200 °C isothermal, 220 °C programmed

phase ..... non-bonded; (S)-2-hydroxy propyl methyl ether derivative of  $\beta$ -cyclodextrin

| I.D. (mm) | d <sub>f</sub> (μm) | Length (m) | Beta Value | Cat. No. | Qty  |
|-----------|---------------------|------------|------------|----------|------|
| 0.25      | 0.12                | 30         | 500        | 71023AST | 1 ea |

## Chiral GC Column Protection

Guard columns protect your capillary column investment. They remove residual moisture, protect the column from non-volatile impurities and the high temperature of the injector and/or detector, and allow for the injection of sample volumes up to 7 μL on-column. Typically a 5-10 m long guard column is used. The guard column can be connected via a press fit or other column connector. To couple to a mass spectrometer, use a 1 m length as a transfer line. We recommend using methyl- or phenyl/methyl-deactivated guard columns to protect CHIRALDEX® and Supelco DEX™ capillary GC columns.

## Chiral GC Column Test Mixes

After you install a column in your system, use a test mix to make sure you haven't also installed some surprises (such as ferrule or tubing fragments in the column, or small leaks). Weekly tests thereafter will keep little problems from growing into big problems. Test mixes are an inexpensive aid to obtaining high quality chromatograms.

| Description                   | Concentration  | Application  | Cat. No. | Qty  |
|-------------------------------|--|--|----------|------|
| β-DEX™ 120 Column Test Mix    | 500 μg/mL each component in methylene chloride<br><i>Decane</i><br><i>3,3-Dimethyl-2-butanol</i><br><i>1-Hexanol</i> | For use with Supelco β-DEX 120.<br><i>(+)-3-Methyl-2-heptanone</i><br><i>Nonane</i><br><i>Undecane</i> | 48028    | 1 mL |
| α-DEX™ 120 Column Test Mix    | 500 μg/mL each component in methylene chloride<br><i>Decane</i><br><i>Nonane</i><br><i>1,2-Propanediol</i>           | For use with Supelco α-DEX 120.<br><i>Undecane</i><br><i>m-Xylene</i><br><i>p-Xylene</i>               | 48013    | 1 mL |
| 1-(N-TFA)-2-Methylpiperidine  | 5000 μg/mL in ethanol: isopropanol (95:5)  | For use with CHIRALDEX G-TA.   | 90002AST | 1 mL |
| 2-(N-TFA)aminoheptane         | 5000 μg/mL in ethanol: isopropanol (95:5)  | For use with CHIRALDEX B-PH, A-TA, G-DM and G-DP.  | 90003AST | 1 mL |
| 1-(N-TFA)aminoindan           | 5000 μg/mL in ethanol: isopropanol (95:5)  | For use with CHIRALDEX B-DA and G-DA.  | 90004AST | 1 mL |
| 2-(Bromomethyl)tetra-2H-pyran | 5000 μg/mL in ethanol: isopropanol (95:5)  | For use with CHIRALDEX B-TA and B-DP.  | 90005AST | 1 mL |
| 3,4-Dihydro-2-ethoxy-2H-pyran | 5000 μg/mL in ethanol: isopropanol (95:5)  | For use with CHIRALDEX A-PH and G-PH.  | 90006AST | 1 mL |
| 1-Phenyl-1-ethanol            | 5000 μg/mL in ethanol: isopropanol (95:5)  | For use with CHIRALDEX B-PM and B-DM.  | 90007AST | 1 mL |

## Guard Columns for Chiral GC Columns

| Tubing Treatment                      | Application   | Max. Temp. |
|---------------------------------------|---|------------|
| Non-polar (methyl)                    | Low polarity solvents (e.g., alkanes, carbon disulfide, ethers)             | 360 °C     |
| Intermediate polarity (phenyl/methyl) | Intermediate polarity solvents (e.g., acetone, methylene chloride, toluene) | 360 °C     |

## Non-Polar Fused Silica Tubing

max. temp. .... 360 °C

| I.D. (mm) | L (m) | Cat. No. | Qty  |
|-----------|-------|----------|------|
| 0.25      | 1     | 24025    | 3 ea |
| 0.25      | 3     | 25722    | 1 ea |
| 0.25      | 5     | 25742    | 1 ea |
| 0.25      | 15    | 25756    | 1 ea |

## Intermediate Polar Fused Silica Tubing

max. temp. .... 360 °C

| I.D. (mm) | L (m) | Cat. No. | Qty  |
|-----------|-------|----------|------|
| 0.25      | 3     | 25727    | 1 ea |
| 0.25      | 5     | 25747    | 1 ea |
| 0.25      | 15    | 25760-U  | 1 ea |

## Chiral Derivatization Reagents

## Chiral Derivatization Reagents

ChiraSelect™ is a unique, high-quality set of the most useful chiral derivatization reagents, carefully produced and rigorously analyzed in our laboratories for all your analytical applications in the chiral field. ChiraSelect reagents are specially selected to meet the requirements for derivatization reagents for enantiomeric excess determinations. To meet any analytical situation, the ChiraSelect line provides pairs of reagents with each respective enantiomer exhibiting an enantiomeric ratio of 99.5:0.5.

## ChiraSelect™ HPLC Derivatization Reagents

| CAS No.     | Compound   | Cat. No.                             | Qty                |
|-------------|--|--------------------------------------|--------------------|
| 7322-88-5   | (S)-(+)-O-Acetylmaleic acid, 99%   | 253022-5G                            | 5 g                |
| -           | (S)-5-Allyl-2-oxabicyclo[3.3.0]oct-8-ene, purum p.a., chiral derivatization reagent for HPLC, ≥97.0% (GC)  | 53835-1G<br>53835-5G                 | 1 g<br>5 g         |
| 20887-95-0  | Boc-Cys-OH, for chiral derivatization, ≥98.5%  | 15411-250MG<br>15411-1G              | 250 mg<br>1 g      |
| 19132-06-0  | L-(+)-2,3-Butanediol, for chiral derivatization, ≥97.0%  | 18967-1ML<br>18967-5ML               | 1 mL<br>5 mL       |
| 89104-48-3  | (4 <i>R</i> ,5 <i>R</i> )-2-Chloro-4,5-dimethyl-1,3,2-dioxaphospholane 2-oxide, for chiral derivatization, ≥95.0%  | 24370-500MG-F<br>24370-5G-F          | 500 mg<br>5 g      |
| 28166-41-8  | α-Cyano-4-hydroxycinnamic acid, 99%  | 476870-2G<br>476870-10G              | 2 g<br>10 g        |
| 130678-42-1 | (+)-Diisopropyl- <i>O,O'</i> -bis(trimethylsilyl)- <i>L</i> -tartrate, 99%, Flukabrand™ ChiraSelect reagent  | 420131-1G                            | 1 g                |
| 95713-52-3  | N <sub>α</sub> -(2,4-Dinitro-5-fluorophenyl)- <i>L</i> -alaninamide, for chiral derivatization, ≥99.0%   | 71478-50MG                           | 50 mg              |
| 210529-62-7 | N <sub>α</sub> -(2,4-Dinitro-5-fluorophenyl)- <i>D</i> -valinamide, for chiral derivatization, ≥98.0%  | 42100-500MG                          | 500 mg             |
| 132679-61-9 | N <sub>α</sub> -(2,4-Dinitro-5-fluorophenyl)- <i>L</i> -valinamide, for chiral derivatization, ≥98.0%  | 42102-100MG                          | 100 mg             |
| 69632-32-2  | ( <i>R</i> )-(-)-3,5-Dinitro- <i>N</i> -(1-phenylethyl)benzamide, 98%  | 296902-1G                            | 1 g                |
| 69632-31-1  | ( <i>S</i> )-(+)-3,5-Dinitro- <i>N</i> -(1-phenylethyl)benzamide, 98%  | 296910-1G                            | 1 g                |
| 154479-90-0 | (-)-1-(9-Fluorenyl)ethyl chloroformate solution, 18 mM in acetone, for chiral derivatization   | 338710-1ML                           | 1 mL               |
| 107474-79-3 | (+)-1-(9-Fluorenyl)ethyl chloroformate solution, ≥18 mM in acetone, for chiral derivatization  | 23182-10X1ML-F<br>23182-10ML-F       | 10 × 1 mL<br>10 mL |
| 124529-07-3 | <i>N</i> -Isobutyl- <i>D</i> -cysteine, for chiral derivatization, ≥97.0%  | 58689-250MG                          | 250 mg             |
| 124529-02-8 | <i>N</i> -Isobutyl- <i>L</i> -cysteine, for chiral derivatization, ≥97.0%  | 58698-250MG-F<br>58698-1G-F          | 250 mg<br>1 g      |
| 784213-51-0 | (1 <i>R</i> ,4 <i>a</i> 5,10 <i>a</i> )-7-Isopropyl-1-isothiocyanato-1,4 <i>a</i> -dimethyl-1,2,3,4,4 <i>a</i> ,9,10,10 <i>a</i> -octahydrophenanthrene, for chiral derivatization | 89394-100MG                          | 100 mg             |
| 3966-32-3   | ( <i>R</i> )-(-)-α-Methoxyphenylacetic acid, for chiral derivatization, ≥99.0%   | 65209-1G                             | 1 g                |
| 26164-26-1  | ( <i>S</i> )-(+)-α-Methoxyphenylacetic acid, for chiral derivatization, ≥99.0%   | 65208-250MG                          | 250 mg             |
| 139658-04-1 | ( <i>R</i> )-6-Methoxy-2,5,7,8-tetramethylchromane-2-carboxylic acid, for chiral derivatization, ≥99.0%  | 93509-50MG                           | 50 mg              |
| 24277-44-9  | (-)-α-Methylbenzyl isothiocyanate, for chiral derivatization, ≥99.0%   | 89568-250MG-F                        | 250 mg             |
| 24277-43-8  | ( <i>S</i> )-(+)-α-Methylbenzyl isothiocyanate, for chiral derivatization, ≥99.0%  | 75491-1G-F                           | 1 g                |
| 10420-89-0  | ( <i>S</i> )-(-)-1-(1-Naphthyl)ethylamine, ≥99%  | 237450-1G<br>237450-5G               | 1 g<br>5 g         |
| 73671-79-1  | ( <i>S</i> )-(+)-1-(1-Naphthyl)ethyl isocyanate, 99%   | 295957-250MG<br>295957-1G            | 250 mg<br>1 g      |
| 159717-68-7 | <i>N</i> -(7-Nitro-4-benzofurazanyl)- <i>L</i> -prolyl chloride, for fluorescence  | 84999-50MG-F                         | 50 mg              |
| 5978-70-1   | ( <i>R</i> )-(-)-2-Octanol, for chiral derivatization, 99%   | 147990-1G<br>147990-5G<br>147990-10G | 1 g<br>5 g<br>10 g |
| 6169-06-8   | ( <i>S</i> )-(+)-2-Octanol, 99%  | 147982-5G<br>147982-10G              | 5 g<br>10 g        |
| 7782-24-3   | ( <i>S</i> )-(+)-2-Phenylpropionic acid, 97%   | 279900-250MG<br>279900-1G            | 250 mg<br>1 g      |
| -           | Quaternary β-cyclodextrin, 100mg, neat   | 33805                                | 100 mg             |
| -           | Sulphated β-cyclodextrin, 100mg, neat  | 33806                                | 1 amp              |
| 14152-97-7  | 2,3,4,6-Tetra- <i>O</i> -acetyl-β- <i>D</i> -glucopyranosyl isothiocyanate, for HPLC derivatization  | T5783-100MG<br>T5783-1G              | 100 mg<br>1 g      |
| -           | 2,3,4,6-Tetra- <i>O</i> -(2-naphthoyl)-β- <i>D</i> -galactopyranosyl isothiocyanate, for derivatization, ~90% (HPLC)   | 04669-25MG-F<br>04669-100MG-F        | 25 mg<br>100 mg    |
| 147948-52-5 | 2,3,4,6-Tetra- <i>O</i> -pivaloyl-β- <i>D</i> -galactopyranosyl isothiocyanate, for chiral derivatization, ≥95% (HPLC, sum of enantiomers)   | 88102-100MG<br>88102-500MG           | 100 mg<br>500 mg   |
| 958300-06-6 | 2,3,4,6-Tetra- <i>O</i> -pivaloyl-β- <i>D</i> -glucopyranosyl isothiocyanate, for chiral derivatization, ≥95.0% (HPLC)   | 44891-100MG-F                        | 100 mg             |
| 62414-75-9  | 2,3,4-Tri- <i>O</i> -acetyl-α- <i>D</i> -arabinopyranosyl isothiocyanate, for chiral derivatization, ≥98.0%  | 90245-100MG                          | 100 mg             |
| 10531-50-7  | ( <i>R</i> )-(-)-α-(Trifluoromethyl)benzyl alcohol, puriss., ≥99.0% (GC, sum of enantiomers)   | 79231-1ML                            | 1 mL               |
| 340-06-7    | ( <i>S</i> )-(+)-α-(Trifluoromethyl)benzyl alcohol, 99%  | 411140-250MG<br>411140-1G            | 250 mg<br>1 g      |
| 14645-24-0  | (-)-Tröger's base, for chiral derivatization, ≥99.0%   | 40765-100MG                          | 100 mg             |
| 21451-74-1  | (+)-Tröger's base, for chiral derivatization, ≥99.0%   | 40764-100MG                          | 100 mg             |
| 135806-59-6 | ( <i>S</i> )-Trolox methyl ether, for chiral derivatization, ≥98.0%  | 93510-50MG                           | 50 mg              |

## Chiral Derivatization Reagents

ChiraSelect™ GC Derivatization Reagents

## ChiraSelect™ GC Derivatization Reagents

| CAS No.     | Compound  | Cat. No.                                | Qty                |
|-------------|---|---|--------------------|
| 36394-75-9  | (S)-(-)-2-Acetoxypropionyl chloride, for chiral derivatization, ≥99.0%                        | 00877-500MG                             | 500 mg             |
| 104530-16-7 | (1R)-(+)-Camphanic chloride, for chiral derivatization, ≥97.0%                                | 21286-250MG-F                           | 250 mg             |
| 39637-74-6  | (1S)-(-)-Camphanic chloride, for chiral derivatization, ≥98.0%                                | 21287-1G-F<br>21287-5G-F<br>21287-25G-F | 1 g<br>5 g<br>25 g |
| 3347-90-8   | (S)-2-Hydroxybutyric acid, for chiral derivatization, ≥99.0%                                  | 54918-1G-F                              | 1 g                |
| 20445-31-2  | (R)-(+)-α-Methoxy-α-trifluoromethylphenylacetic acid, for chiral derivatization, ≥99.0%       | 65361-250MG<br>65361-1G                 | 250 mg<br>1 g      |
| 17257-71-5  | (S)-(-)-α-Methoxy-α-trifluoromethylphenylacetic acid, for chiral derivatization, ≥99.0%       | 65369-250MG-F                           | 250 mg             |
| 39637-99-5  | (R)-(-)-α-Methoxy-α-(trifluoromethyl)phenylacetyl chloride, for chiral derivatization, ≥99.0% | 65363-100MG<br>65363-500MG              | 100 mg<br>500 mg   |
| 20445-33-4  | (S)-(+)-α-Methoxy-α-trifluoromethylphenylacetyl chloride, for chiral derivatization, ≥99.0%   | 65365-100MG-F<br>65365-500MG-F          | 100 mg<br>500 mg   |
| 3886-69-9   | (R)-(+)-α-Methylbenzylamine, for chiral derivatization, ≥99.0%                                | 77879-5ML<br>77879-25ML                 | 5 mL<br>25 mL      |
| 2627-86-3   | (S)-(-)-α-Methylbenzylamine, for chiral derivatization, ≥99.0%                                | 77869-5ML<br>77869-25ML                 | 5 mL<br>25 mL      |
| 33375-06-3  | (R)-(+)-α-Methylbenzyl isocyanate, for chiral derivatization, ≥99.0%                          | 77968-1ML<br>77968-5ML                  | 1 mL<br>5 mL       |
| 14649-03-7  | (S)-(-)-α-Methylbenzyl isocyanate, for chiral derivatization, ≥99.0%                          | 77970-1ML<br>77970-5ML                  | 1 mL<br>5 mL       |
| 104371-21-3 | (R)-(+)-α-Methyl-2,3,4,5,6-pentafluorobenzyl alcohol, for chiral derivatization, ≥99.0%       | 76744-1G                                | 1 g                |
| 104371-20-2 | (S)-(-)-α-Methyl-2,3,4,5,6-pentafluorobenzyl alcohol, for chiral derivatization, ≥99.0%       | 76746-1G                                | 1 g                |
| 3886-70-2   | (R)-(+)-1-(1-Naphthyl)ethylamine, for chiral derivatization, ≥99.5%                           | 70710-1ML                               | 1 mL               |
| 42340-98-7  | (R)-(-)-1-(1-Naphthyl)ethyl isocyanate, for chiral derivatization, ≥99.0%                     | 70725-1ML                               | 1 mL               |
| 1517-69-7   | (R)-(+)-1-Phenylethanol, for chiral derivatization, ≥99.0%                                    | 77848-1ML<br>77848-5ML                  | 1 mL<br>5 mL       |
| 1445-91-6   | (S)-(-)-1-Phenylethanol, for chiral derivatization, ≥99.0%                                    | 77849-1ML<br>77849-5ML                  | 1 mL<br>5 mL       |



## Chiral Mobile Phase Additives

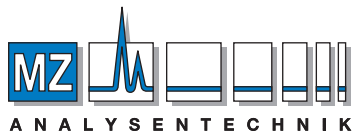
## Chiral Mobile Phase Additives

Enantiomers of the same parent compound differ in the way they interact with other chiral molecules, like chiral stationary phases. However, it is possible to affect an enantiomeric separation using conventional HPLC and CE stationary phases by adding the chiral selector to the mobile phase (1,2). These chiral selector additives generally interact via ion pair, ligand exchange or inclusion interactions with enantiomer analytes, forming mobile diastereomeric complexes that are therefore separable by conventional normal or reversed-phase columns. When free cyclodextrins are added to the mobile phase, inclusion complexes are formed and separations can approach those obtained on cyclodextrin-based CSPs. Note that this approach often leads to a reversal in the elution order obtained on a cyclodextrin CSP. Sigma-Aldrich carries a number of highly-pure cyclodextrin derivatives for this application.

(1) Armstrong, D. W. Pseudophase Liquid Chromatography: Applications to TLC. *J. Liq. Chromatogr.* **1980**, 3(6), 895-900.

(2) Ameyibor, E.; Stewart, J. T. Enantiomeric HPLC Separation of Selected Chiral Drugs Using Native and Derivatized  $\beta$ -Cyclodextrins as Chiral Mobile Phase Additives. *J. Liq. Chromatogr.* **1997**, 20(6), 855-869.

| CAS No.     | Compound  | Cat. No.  | Qty                           |
|-------------|---|---|-------------------------------|
| -           | (2-Carboxyethyl)- $\beta$ -cyclodextrin sodium salt   | 21872-1G-F<br>21872-5G-F                              | 1 g<br>5 g                    |
| -           | Carboxymethyl- $\beta$ -cyclodextrin sodium salt  | 21906-1G<br>21906-5G                                  | 1 g<br>5 g                    |
| 10016-20-3  | $\alpha$ -Cyclodextrin, purum, $\geq 98.0\%$ (HPLC)   | 28705-5G<br>28705-25G<br>28705-100G                   | 5 g<br>25 g<br>100 g          |
| 68168-23-0  | $\beta$ -Cyclodextrin hydrate, 99%  | 856088-5G<br>856088-25G<br>856088-100G                | 5 g<br>25 g<br>100 g          |
| 68168-23-0  | $\beta$ -Cyclodextrin hydrate, puriss., $\geq 99.0\%$ (HPLC)                                  | 28707-5G<br>28707-25G<br>28707-100G                   | 5 g<br>25 g<br>100 g          |
| 91464-90-3  | $\gamma$ -Cyclodextrin hydrate  | 861413-100MG<br>861413-1G                             | 100 mg<br>1 g                 |
| 699020-02-5 | $\alpha$ -Cyclodextrin, sulfated sodium salt hydrate  | 494542-5G   | 5 g                           |
| 37191-69-8  | $\beta$ -Cyclodextrin, sulfated sodium salt, extent of labeling: 7-11 mol per mol $\beta$ -CD | 389153-5G<br>389153-25G                               | 5 g<br>25 g                   |
| 51166-71-3  | Heptakis(2,6-di-O-methyl)- $\beta$ -cyclodextrin  | H0513-1G<br>H0513-5G                                  | 1 g<br>5 g                    |
| 51166-71-3  | Heptakis(2,6-di-O-methyl)- $\beta$ -cyclodextrin, $\geq 98.0\%$ (TLC)                         | 39915-1G  | 1 g                           |
| 55216-11-0  | Heptakis(2,3,6-tri-O-methyl)- $\beta$ -cyclodextrin, $\geq 90\%$                              | H4645-5G  | 5 g                           |
| 55216-11-0  | Heptakis(2,3,6-tri-O-methyl)- $\beta$ -cyclodextrin, $\geq 98.0\%$                            | 51707-1G<br>51707-5G                                  | 1 g<br>5 g                    |
| 128446-32-2 | (2-Hydroxyethyl)- $\beta$ -cyclodextrin, extent of labeling: $\sim 0.7$ mol per mol cellulose | 389137-10G  | 10 g                          |
| 128446-33-3 | (2-Hydroxypropyl)- $\alpha$ -cyclodextrin, average $M_w \sim 1,180$                           | 390690-5G<br>390690-25G                               | 5 g<br>25 g                   |
| 128446-35-5 | (2-Hydroxypropyl)- $\beta$ -cyclodextrin, average $M_w \sim 1,380$                            | 332593-5G<br>332593-25G<br>332593-100G                | 5 g<br>25 g<br>100 g          |
| 128446-35-5 | (2-Hydroxypropyl)- $\beta$ -cyclodextrin, average $M_w \sim 1,460$                            | 332607-5G<br>332607-25G<br>332607-100G<br>332607-500G | 5 g<br>25 g<br>100 g<br>500 g |
| 128446-35-5 | (2-Hydroxypropyl)- $\beta$ -cyclodextrin, average $M_w \sim 1,540$                            | 389145-5G<br>389145-25G                               | 5 g<br>25 g                   |
| 128446-35-5 | (2-Hydroxypropyl)- $\beta$ -cyclodextrin, $M_n \sim 1380$                                     | 56332   |                               |
| 128446-34-4 | (2-Hydroxypropyl)- $\gamma$ -cyclodextrin, solid  | H125-5G-I<br>H125-100G-I                              | 5 g<br>100 g                  |
| 128446-34-4 | (2-Hydroxypropyl)- $\gamma$ -cyclodextrin, extent of labeling: 0.6 molar substitution         | 390704-5G<br>390704-25G                               | 5 g<br>25 g                   |
| 128446-36-6 | Methyl- $\beta$ -cyclodextrin, average $M_n$ 1310   | 332615-5G<br>332615-25G                               | 5 g<br>25 g                   |
| -           | Succinyl- $\beta$ -cyclodextrin   | 85990-500MG<br>85990-5G                               | 500 mg<br>5 g                 |
| 23739-88-0  | Triacetyl- $\beta$ -cyclodextrin  | 332623-10G  | 10 g                          |



## AUTHORIZED DISTRIBUTOR

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