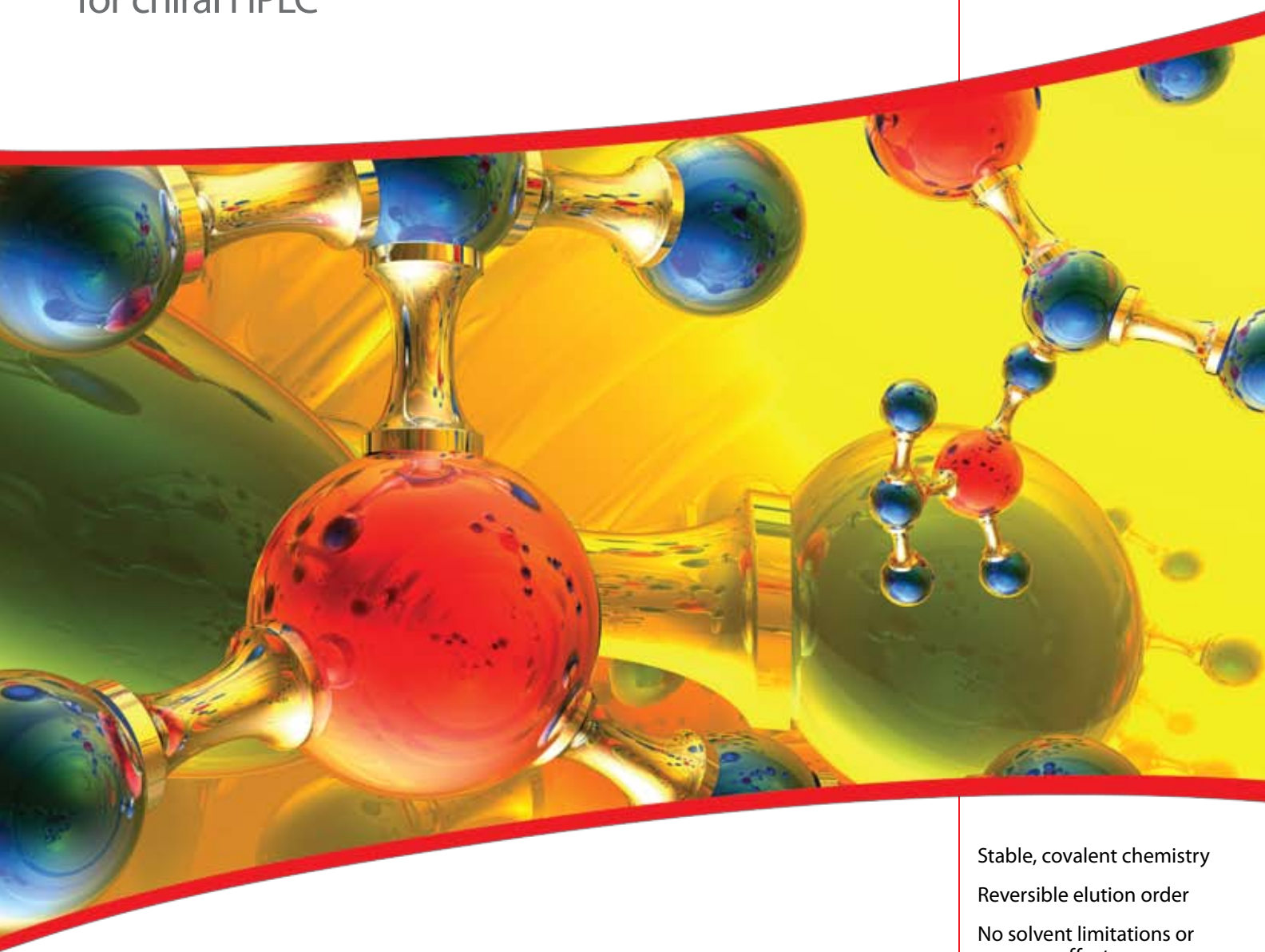
Astec P-CAP™ and P-CAP™-DP

Polycyclic amine polymer stationary phases for chiral HPLC

SUPELCO® Analytical



- Stable, covalent chemistry
- Reversible elution order
- No solvent limitations or memory effects
- High capacity for preparative applications
- MS and SFC-compatible

Chiral HPLC for Chemists: Ultimate Solvent Choice with High Capacity Using Astec's P-CAP and P-CAP-DP

Useful for chiral HPLC and SFC separations, Astec P-CAP and P-CAP-DP polymeric chiral stationary phases (CSPs) have a thin, ordered layer of chiral polymer covalently bonded to the silica surface. They offer high stability, high sample loadability, easy scale-up, and no memory effect.

Today's chiral HPLC columns too often give excellent enantioselectivity at the expense of solvent choice. Sample solubility and its link to preparative separations can mean that a compromise has to be reached between selectivity and solvent choice. Astec P-CAP and P-CAP-DP chiral HPLC columns have no solvent restrictions, so the user can select a solvent that provides optimum enantioselectivity and analyte solubility.

Astec P-CAP

- **Bonded phase: Poly(trans-1,2-cyclohexanediyl-bis-acrylamide)**
- **Invented by Prof. Francesco Gasparrini (1), P-CAP is made from a diacryloyl -trans-1,2-diphenylethylenediamine polymerization, and utilizes hydrogen bonding and steric effects as enantiomer separation mechanisms.**

Astec P-CAP-DP

- **Bonded phase: Poly(diphenylethylenediamine-bis-acryloyl) or Poly-DPEDA**
- **Invented by Prof. Daniel Armstrong (2), Astec P-CAP-DP introduces phenyl rings to add π-π interactions, giving it one additional type of interaction compared to P-CAP. P-CAP-DP is less polar than P-CAP.**

Solvent Choice and Reversal of Elution Order

The P-CAP and P-CAP-DP polyamide CSPs feature a thin, ordered polymer layer chemically bonded to 5 µm or 3.5 µm spherical silica using a patented radical polymerization. This gives the phases high permeability across the surface and, because they are synthetic, they can be identically manufactured in both R,R and S,S forms. This provides a predictable reversal of elution order in the same mobile phase (Figure 1).

The P-CAP phases have no solvent or additive memory effects, so the same column can be used in a number of different mobile phases without any detrimental effects. These phases form a new generation bridge between the traditional 'brush' type CSPs and the conventional polymeric phases.

Chiral method development is typically carried out either in normal phase (heptane/IPA or hexane/ethanol) or polar organic (acetonitrile/methanol) modes. For method optimization, a wide range of organic solvents can be used, from acetone to dichloromethane to dioxane, and many others. For acids and bases, the addition of 0.1% TFA often

Figure 1. Predictable Elution Order Reversal

columns: 25 cm x 4.6 mm I.D., 5 µm
mobile phase: 95:5, methylene chloride:methanol
flow rate: 1 mL/min.

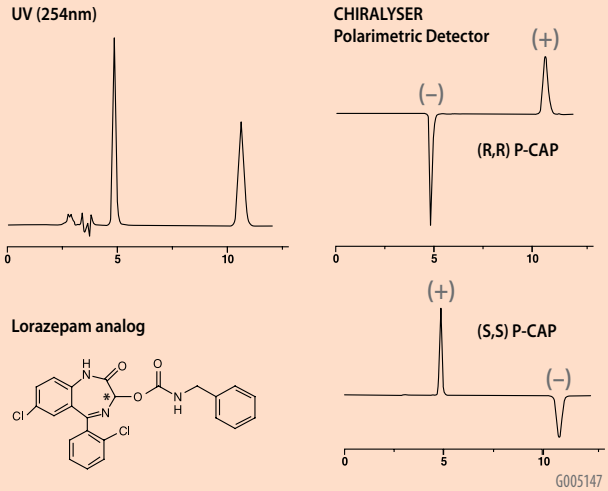
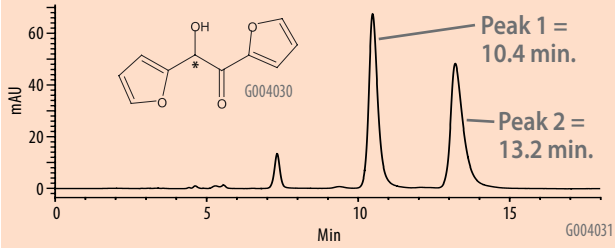


Figure 2. High-capacity Separations

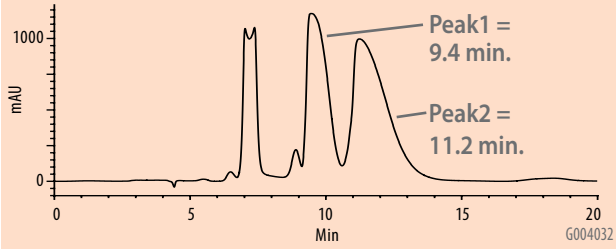
Analyte: furoin

column: (R,R) P-CAP-DP, 25 cm x 4.6 mm I.D., 5 µm particles (25024AST)
mobile phase: 80:20, hexane:ethanol
flow rate: 1.0 mL/min.
temp.: 23 °C
det.: UV at 235 nm
injection: (a) 2 µL at 4 mg/mL (8 µg total)
(b) 50 µL at 20 mg/mL (1000 µg total)

8 µg injection



1000 µg injection



increases resolution and efficiency and decreases retention times. There are no known limitations on the kind of solvents that can be used with these phases. For MS detection, volatile acids and buffers such as ammonium acetate can be added to enhance peak efficiency or to enhance ionization when needed.

High Capacity for Preparative HPLC

Solvent flexibility and high loading capacity make P-CAP and P-CAP-DP CSPs ideal for analytical, preparative and process scale separations. The separation of furoin enantiomers is shown in Figure 2 using an analytical 25 cm x 4.6 mm Astec (R,R) P-CAP-DP column and mobile phase of 80:20 hexane:ethanol. Excellent separation is demonstrated with an injection of 8 µg of the furoin racemate. Increasing the load to 1 mg on this analytical column demonstrates the phase's high loading capacity.

MS-Compatible Operation

Astec P-CAP and P-CAP-DP operate in mobile phases that are amenable to MS-detection. Salt and/or acetic acid can be added to improve efficiency or enhance ionization and detection (Figure 3).

Applications

Astec P-CAP CSPs have been used for a wide variety of molecular types and are ideal for medium to high polarity compounds. The mechanism of separation is either through hydrogen bonding for P-CAP, or through both hydrogen bonding (donor and acceptor) with additional π-π interaction for the P-CAP-DP. Both also use dipole-dipole and steric interactions. Examples of the separations completed to date are shown in Table 1.

Table 1. Examples of P-CAP and P-CAP-DP Application Areas

Hydroxycarboxylic acids	**Benzene sulfonamides**
Alcohols	**Binaphthols**
Sulfoxides	**Benzodiazepines**
Esters	**Phosphonic acids**
Amides	**Bis-Sulfones**
N-blocked amino acids	**Chromemones**

Complementary to Other Astec CSPs

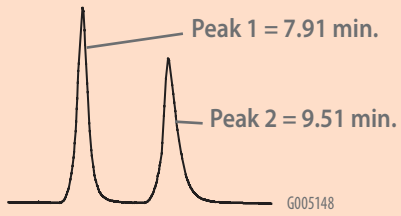
Astec P-CAP and P-CAP-DP are complementary to Astec CHIROBIOTIC, CYCLOBOND and the polysaccharide-based CSPs. We suggest you incorporate them into your chiral column screening protocol.

Figure 3. Improved Analytical Separation with MS-Compatible Mobile Phases

column: (R,R) P-CAP, 25 cm x 4.6 mm I.D., 5 µm particles (31024AST)
flow rate: 1.0 mL/min.
temp.: 25 °C
det.: UV @ 254 nm

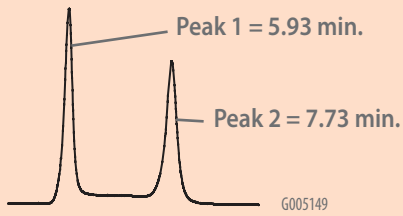
Separation of 1,1'-Bi-2-naphthol

mobile phase: 95:5:10 mM ammonium acetate:methanol:acetate



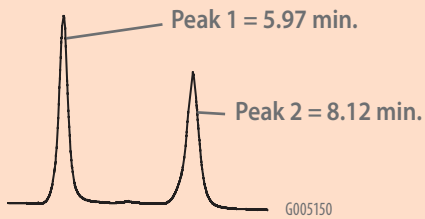
Oxazepam

mobile phase: 70:30:20 mM ammonium acetate:methanol:acetate



Lorazepam

mobile phase: 70:30:20 mM ammonium acetate:methanol:acetate



Summary

Astec P-CAP and P-CAP-DP are rugged chiral HPLC phases, experience no memory effects, and can be run in a wide variety of solvents with speed and high efficiencies. For preparative applications, a combination of wide solvent choice and high capacity make them ideal for large-scale purification.

References

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

2) Chromatographic evaluation of poly(trans-1,2-cyclohexanediyl-bisacrylamide) as a chiral stationary phase for HPLC. Zhong, Qiqing; Han, Xinxin; He, Lingfeng; Beesley, Thomas E.; Trahanovsky, Walter S.; Armstrong, Daniel W. Department of Chemistry, Iowa State University, Ames, IA, USA. Journal of Chromatography, A (2005), 1066(1-2), 55-70.

Ordering Information

Particle Size (µm)	Length (cm)	I.D. (mm)	Cat. No.
Astec (R,R) P-CAP			
3.5	5	4.6	**30021AST**
3.5	10	4.6	**30022AST**
3.5	15	4.6	**30023AST**
5	5	4.6	**31021AST**
5	10	2.1	**31018AST**
5	10	4.6	**31022AST**
5	15	2.1	**31019AST**
5	15	4.6	**31023AST**
5	25	2.1	**31020AST**
5	25	4.6	**31024AST**
5	25	10	**31034AST**
5	25	21.2	**31044AST**
10	25	4.6	**31124AST**
Guards*			
5	2	1	**31101AST**
5	2	4	**31100AST**
Astec (S,S) P-CAP			
3.5	5	4.6	**32021AST**
3.5	10	4.6	**32022AST**
3.5	15	4.6	**32023AST**
5	5	4.6	**33021AST**
5	10	2.1	**33018AST**
5	10	4.6	**33022AST**
5	15	2.1	**33019AST**
5	15	4.6	**33023AST**
5	25	2.1	**33020AST**
5	25	4.6	**33024AST**
5	25	10	**33034AST**
5	25	21.2	**33044AST**
10	25	4.6	**33124AST**
Guards*			
5	2	1	**33101AST**
5	2	4	**33100AST**

Particle Size (µm)	Length (cm)	I.D. (mm)	Cat. No.
Astec (R,R) P-CAP-DP			
3.5	15	4.6	**34023AST**
5	15	2.1	**35019AST**
5	15	4.6	**35023AST**
5	25	4.6	**35024AST**
5	25	10	**35034AST**
5	25	21.2	**35044AST**
Guards*			
5	2	4	**35100AST**
Astec (S,S) P-CAP-DP			
3.5	15	4.6	**36023AST**
5	15	2.1	**37019AST**
5	15	4.6	**37023AST**
5	25	4.6	**37024AST**
5	25	10	**37034AST**
5	25	21.2	**37044AST**
Guards*			
5	2	4	**37100AST**

*Guard Column Holders	
Guard Holders for 4 mm I.D. cartridges (holder not required for 1 mm I.D. guards)	**21150AST**

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