Astec P-CAP™ and P-CAP-DP Series HPLC Column Operating Instructions

Description

Astec P-CAP: Poly(trans-1,2-cyclohexanediamine-bis-acrylamide) bonded to high-purity silica gel. Invented by Prof. Francesco Gasparrini (1), P-CAP is made from a diacryloyl derivative of trans-1,2-diamino-cyclohexane polymerization, and utilizes hydrogen bonding and steric effects as enantiomer separation mechanisms.

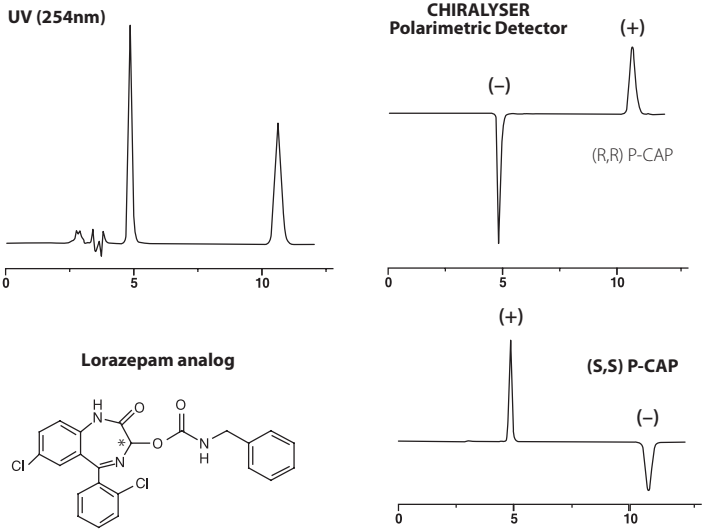
Astec P-CAP-DP: Poly(diphenylethylenediamine-bis-acryloyl) or Poly-DPEDA bonded to high-purity silica gel. Developed by Prof. Daniel Armstrong (2), Astec P-CAP-DP introduces phenyl rings to add p-p interactions, giving it one additional type of interaction compared to P-CAP. P-CAP-DP is less polar than P-CAP.

Introduction

Astec 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 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).

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.



Application Areas

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.

Mobile Phases

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.

Method Development & Optimization

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 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.

Polar Organic Mode (POM): 70:30:0.1 wt%, acetonitrile:methanol:ammonium acetate. Change the ratio of acetonitrile to methanol in the mobile phase.

Normal Phase Mode (NP): ethanol:heptane (50:50). Change ratio. Try the following systems: IPA:heptane (50:50); dichloromethane:methanol (97:3); ethyl acetate:methanol (97:3); heptane:THF (50:50).

Considerations for MS Detection: Astec P-CAP and P-CAP-DP operate in mobile phases that are amenable to MS-detection. Volatile salts and/or acetic acid can be added to improve efficiency or enhance ionization and detection.

Flow Rate: 0.5 - 2 mL/min.

Maximum Pressure: 3000 psi

Temperature Range: 5 to 45 °C

Column Washing: 50:50:0.1 w%, acetonitrile:methanol:ammonium acetate, then pure organic solvent, like methanol or ethanol.

Storage: Typical normal phase system, e.g. ethanol:heptane (50:50), or 100% acetonitrile, methanol, or ethanol are ideal for long and short-term storage.

References

1. New hybrid polymeric liquid chromatography chiral stationary phase prepared by surface initiated polymerization. Gaparrini, 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. Journal of Chromatography, A (2005), 1066(1-2), 55-70.