Two novel polysaccharide-based chiral stationary phases: CHIRALPAK® AY-H and CHIRALCEL® OZ-H

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Polysaccharide-based chiral stationary phases (CSP) are successfully employed to achieve the majority of analytical and preparative enantioselective separations. However, there are a number of separations where significant modification or enhancement of selectivity is necessary. Two new columns, CHIRALPAK AY-H and CHIRALCEL OZ-H, have been introduced to meet these needs.

Chiral stationary phases based on methylchlorophenyl carbamates of cellulose (CHIRALCEL OZ) and amylose (CHIRALPAK AY and AZ), coated on 20-micron silica, have been available for some years for preparative separations. These CSPs have proven to be valuable in Chiral Technologies, Inc's custom separation service operations, generating results superior (~ 10% of the separations) to our more established CSPs. To exploit the enhanced selectivity of these complementary CSPs, 5-micron versions have been developed for analytical and semi-preparative use.

Structure

The new 5-micron phases are based upon the same wide-pore silica as is used for the other Daicel media. CHIRALPAK AY-H is the tris-(5-chloro-2-methylphenylcarbamate) of amylose whereas CHIRALCEL OZ-H is tris-(3-chloro-4-methylphenylcarbamate) of cellulose. Scale-up from these 5-micron columns, if needed, to the 20-micron preparative phases can easily be accomplished.

Stability

Pressure stability for HPLC columns is increasingly critical as chromatographers develop faster applications. Pressure testing was carried out by running the columns at pressure drops up to 250 bar over a period of 3 weeks. No change in performance was observed during these tests.

Selectivity

The chromatograms show various separations where the selectivity of the new columns is seen to be different from that of other phases. Figure 1 shows the separation of the enantiomers of methyl 1-benzyl-5-oxo-3 pyrrolidinecarboxylate using CHIRALCEL OZ-H (red trace) and CHIRALCEL OD-H (blue trace). Figures 2 and 3 show the separation of diastereoisomers of the vasodilator cyclandelate (Fig 2) and the herbicide metolachlor (Fig 3) in comparison with separations on CHIRALPAK AD-H and CHIRALCEL OD-3, respectively.

Conclusions

Novel CSPs based on 5-and 20-micron silica with methyl chlorophenylcarbamates have been developed. These new CSPs clearly demonstrate alternative selectivity to other polysaccharide-based chiral phases for both analytical and preparative applications.

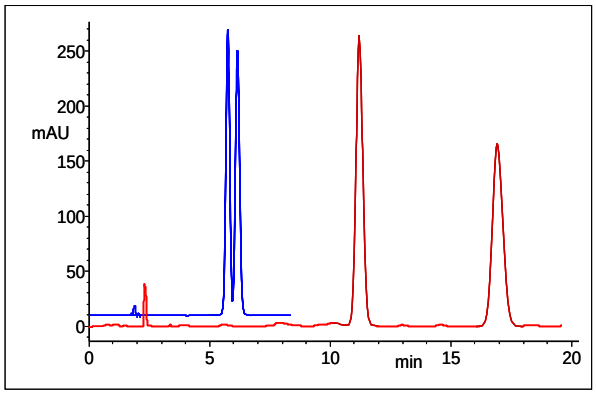

Figure 1. Separation of methyl 1-benzyl-5-oxo-3-pyrrolidinecarboxylate enantiomers. Red trace: CHIRALCEL OZ-H. Blue trace: CHIRALCEL OD-H; both columns 250 x 4.6mm; 80:20 hexane ethanol, 1.5 ml/min, 25°C.

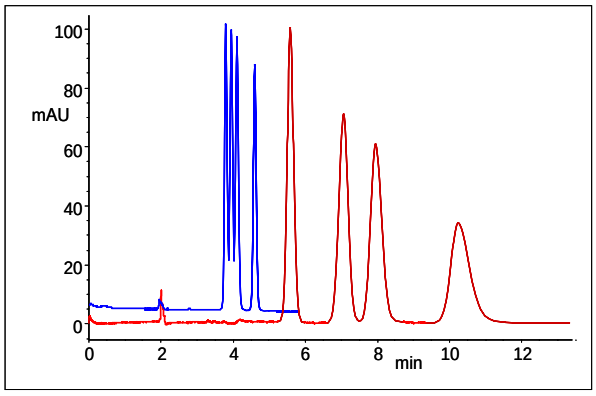

Figure 2. Separation of cyclandelate diastereomers. 90:10 hexane ethanol, 1.5 ml/min, 25°C. Red trace: CHIRALPAK AY-H. Blue trace: CHIRALPAK AD-H; both columns 250 x 4.6mm.

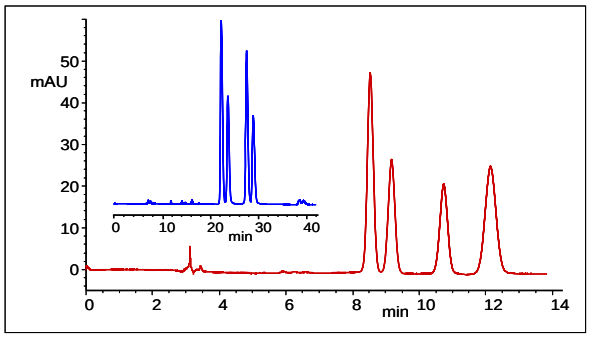

Figure 3. Separation of metolachlor diastereomers.. Red trace: CHIRALPAK AY-H; 250 x 4.6 mm; 95:5 hexane: ethanol, 1 ml/min; 25°C. Blue trace: CHIRALPAK OD-3 2 x 150 x 4.6mm; 97.5:2.5 hexane: IPA, 0.5 ml/min, 25°C.

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