CHIRAL TECHNOLOGIES INC
DAICEL GROUP

CHIRALPAK® IC™ – An Immobilized Polysaccharide Chiral Stationary Phase with a Unique Chiral Selector

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A new, immobilized chiral stationary phase (CSP) based on a novel chiral selector for HPLC and SFC is described. Its unique selectivity as a complement to Daicel's other commercial immobilized polysaccharide CSPs is demonstrated.

The development of immobilization technologies in combination with the popular polysaccharide-based chiral stationary phases has led to the possibility to use new selectors which have hitherto been inaccessible. One such chiral selector is cellulose tris(3,5-dichlorophenylcarbamate). This was identified as being too soluble to make successful chiral stationary phases by Okamoto (1) some years ago. More recently, using photochemical and thermal immobilization techniques, Francotte (2) has demonstrated the wide selectivity range and capability of this selector. A new commercial material, CHIRALPAK IC^T, is now available, based upon cellulose tris(3,5-dichlorophenylcarbamate) polymer immobilized on silica using proprietary techniques.

Experimental

Columns 4.6 x 250 mm (Daicel Chemical Industries, Ltd) were used throughout. Flow rates of 1 ml/min (Tropic acid & Prilocaine) or 1.5 ml/min (Devrinol) were used. Agilent and Shimadzu HPLC systems fitted with autosamplers and diode array detectors set at appropriate wavelengths for the compounds concerned were employed.

Results.

The figures show three examples of separations which, while difficult to achieve using pre-existing immobilized stationary phases (CHIRALPAK IA and IB), are easy to perform with the new selectivity of CHIRALPAK IC. It is interesting to note that two of the separations were performed with MTBE-based mobile phases with CHIRALPAK IC. These solvent mixtures may only be used with immobilized polysaccharide-based CSPs.

Conclusions

CHIRALPAK IC demonstrates the expected unique selectivity from its chiral selector. In many cases its selectivity is complementary to the other immobilized polysaccharide-based CSPs, leading to a high degree of success in screening for chiral method development using a combination of all three columns.

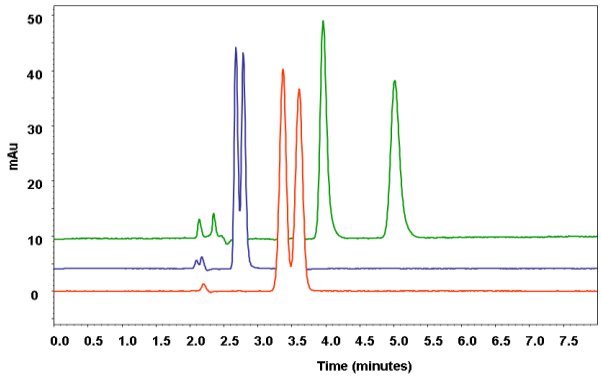

Figure 1. Separation of Devrinol enantiomers.
-- *CHIRALPAK IA* -- *CHIRALPAK IB* -- *CHIRALPAK IC*
Mobile phase : 95:5 MTBE:EtOH; 1.5 ml/min

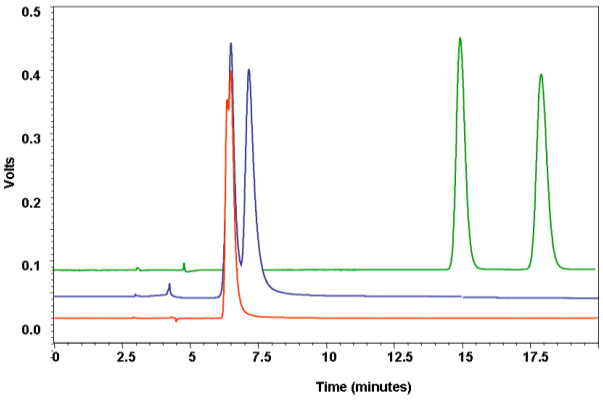

Figure 2. Separation of Prilocaine enantiomers.
-- *CHIRALPAK IA* -- *CHIRALPAK IB* -- *CHIRALPAK IC*
Mobile phase : 90:10:0.1 Hexane:IPA:DEA; 1.0 ml/min

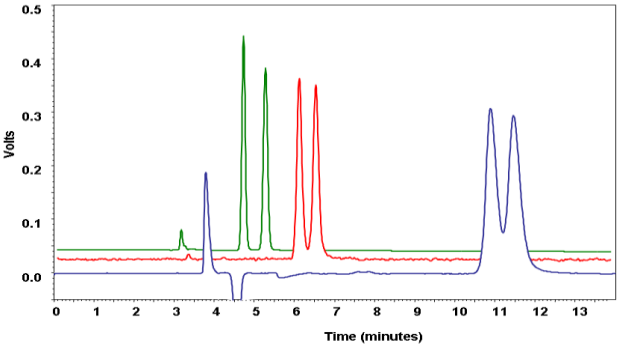

Figure 3. Separation of Tropic Acid enantiomers
-- *CHIRALPAK IA : 65:35:0.1 Hexane :THF :TFA*
-- *CHIRALPAK IB : 90:10:0.1 Hexane:IPA:TFA*
-- *CHIRALPAK IC : 98:2:0.1 MTBE:MeOH:TFA*

(1) Y Okamoto, M Kawashima and K Hatada, *J Chromatogr*., **363,** 173 (1986).
(2)E Francotte and D Huynh, *J Pharm Biomed Anal*., **27,** 421 (2002).