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Temperature effects on a doubly tethered diproline chiral stationary phase: Hold-up volume, enantioselectivity and robustness

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ABSTRACT

Effect of temperature on hold-up volume, enantioselectivity and robustness of a novel doubly tethered diproline chiral stationary phase (CSP1) was studied. In-column end-capping of residual silanol was utilized as a tool to exhibit *in situ* change of CSP1. The hold-up volume marker, 1,3,5-tri-*tert*butylbenzene (TTBB), was observed to be weakly retained (<1 second) on a 5 cm x 4.6 mm chiral column, and its retention time was changed with the carrier solvent and column temperature. The apparent thermodynamic parameters of TTBB indicated an enthalpy-driven retention process with the hexane/isopropanol (IPA) mobile phase, while an entropy-driven process with the hexane/methyl *tert*butyl ether (MTBE) mobile phase. The $\Delta\Delta H$ and $\Delta\Delta S$ values of chiral separation for the four probes including 1,1'-bi-2-naphthol and warfarin were negative on CSP1. Nonlinear van't Hoff plots were observed for some analytes before and after the endcapping treatment. Depending on compound, endcapping strengthened or weakened the enantioseparation. Moreover, the enantioselectivity of CSP1 was shown to be robust by testing with heating-cooling cycles and step-temperature programs.

Full Text

ftp://ftp.sccwrp.org/pub/download/DOCUMENTS/AnnualReports/2010AnnualReport/ar10_057_066.pdf