SCCWRP #0622

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 endcapping 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, was observed to be weakly retained (<1 s) 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 1,3,5-tri-*tert*-butylbenzene indicated an enthalpy-driven retention process with the hexane/ isopropanol mobile phase, while an entropy-driven process with the hexane/methyl *tert*butyl ether mobile phase. The $\Delta\Delta H$ and $\Delta\Delta S$ values of chiral separation for the four probes including 1,10-bi-2-naphthol and warfarin were negative on CSP1. Nonlinear van't Hoff plots were observed for some analytes before and after the end-capping treatment. Depending on compound, end-capping 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.