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Characterization of column hold-up volume with static and dynamic methods on an immobilized polysaccharide-based chiral stationary phase

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ABSTRACT

Polysaccharide-based chiral stationary phases (CSPs) are efficient for enantioseparation of many chiral compounds. Immobilized polysaccharide CSP, as used in the Chiralpak IA column, is a new configuration that was recently introduced for application in chiral separation. As shown in several previous studies, the characteristics of Chiralpak IA columns cannot be simply extrapolated from the coated version. In this study, hold-up volume of a Chiralpak IA column was evaluated by static and dynamic methods. The static pyconometry method gave similar hold-up volumes either as an average value from a range of solvents or a direct measurement from the carbon tetrachloride-isopropanol (IPA) solvent pair. The dynamic method with 1,3,5-tri-tert-butylbenzene (TTBB) was influenced by the ratio of n-hexane and 2-propanol in the mobile phase but not by the dissolving solvent of TTBB. The two methods resulted in the same hold-up volume of *3.0 mL. TTBB showed weaker retention on the IA column after correction of isobaric thermal expansion of the mobile phase. During temperature variations in the range of 15–50 °C, the hold-up volume of TTBB was highly reproducible. Results of this study improve our understanding of the chromatographic features of the immobilized polysaccharide IA column.

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