

Performance Comparison: ZirChrom[®]-Chiral to Silica CSPs

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High-performance liquid chromatography is the dominant method for both analytical and preparative separation of chiral pharmaceuticals. However, no currently available commercial chiral stationary phase uses zirconia as a substrate. A promising new route to preparing chiral stationary phases using a zirconia substrate particle has been developed (See ZirChrom Technical Bulletin #313). The following demonstrates the performance of these new zirconia-based chiral HPLC columns compared to that of analogous conventional silica phases. The zirconia chiral selector attachment chemistry is extremely stable under routine use conditions with the added potential feature of selector regeneration or substitution, for chiral screening, by use of a high pH wash.

Introduction

The zirconia-based chiral phases used in this work were synthesized using a two-step approach to provide a more robust and flexible platform for chiral stationary phase (CSP) design when compared to silica gel. Columns produced in this manner were compared to silica columns having analogous chiral selectors and found to have similar resolving power for the selected probe enantiomers. Most importantly, the Lewis acid-base bonded chiral selectors on zirconia were found to be stable enough for extended routine use; however, they could be completely removed by washing with a high pH (>pH 12) aqueous solution and could be then easily regenerated. Results shown in Figures 1 & 2 for the zirconia-based CSP columns are comparable to the performance of silica-based columns having an analogous chiral selector. This new approach, the synthesis of chiral stationary phases that are regenerable on-column, promises to significantly extend the useful lifetime of both analytical and preparative enatiomer separation media.

Experimental

Brush-type CSPs were selected for initial experiments due to their ease of synthesis, wide scope of applicability, and large body of available silica-based separations data for comparison. The separation conditions were as follows:

 Column:
 See Figures, All 5 mm x 4.6 mm columns

 Mobile Phase:
 See Figures

 Temperature:
 30 °C with Metalox™ 200-C column heater

 Flow Rate:
 1.0 ml/min.

 Injection Vol.:
 0.5 µl

 Pressure Drop:
 195 bar

 Detection:
 UV at 254 nm

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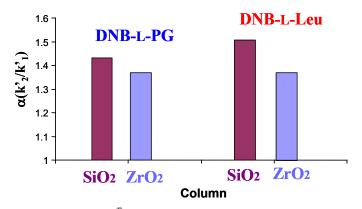


Figure 1: ZirChrom[®]-Chiral (S)LEU CSP column (part # ZRC01-0546) & ZirChrom[®]-Chiral (S)PG CSP column (part # ZRC04-0546) Selectivity Comparisons. Conditions: 99/1 hexane/isopropanol. Compound: trifluoroanthryl ethanol.

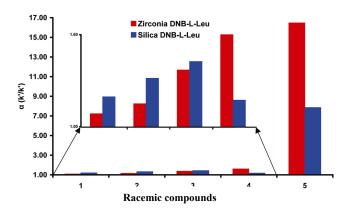


Figure 2: Comparison of ZirChrom[®]-Chiral (S)LEU CSP column (part # ZRC01-0546) and Silica CSP Selectivity. Conditions: 99/1Hexane/isopropanol, 1. trans-stilbene oxide, 2. 1,1'-bi-2-naphthol, 3. trifluoranthyl ethanol, 4. napropamide, 5. 1-naphthyl leucine ester.

Please contact ZirChrom technical support at 1-866-STABLE-1 or <u>support@zirchrom.com</u> for more information regarding this exciting new technology.

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