



ZirChrom®

# Performance of ZirChrom®-Chiral Zirconia-based CSPs

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High-performance liquid chromatography has become the dominant method for the analytical and preparative separation of chiral pharmaceuticals. However, no current chiral stationary phase uses zirconia or inorganic oxides other than silica as a substrate. A promising new route to preparing chiral stationary phases using a zirconia substrate has been developed (See ZirChrom Technical Bulletin #313). The following demonstrates the performance of these new versatile, durable, and efficient zirconia-based chiral HPLC columns is comparable to that of conventional silica phases.

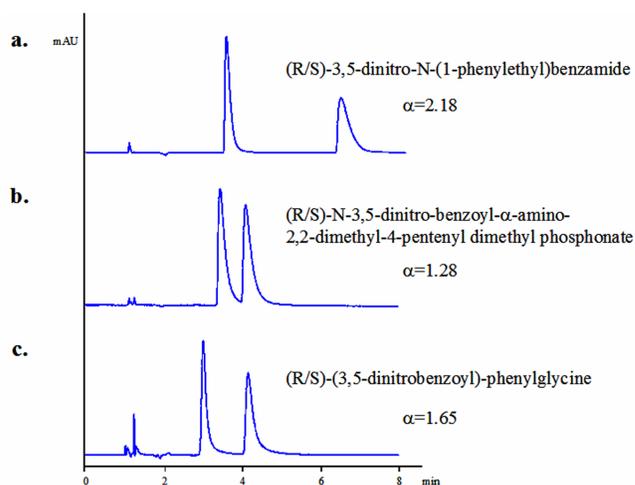
### Introduction

The zirconia-based chiral phases employed in this study 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 chemisorbed chiral selectors on zirconia were found to be stable enough for extended routine use; however, they were completely removed by washing with a high pH (>pH 12) aqueous solution and could be easily regenerated. Results shown in Figures 1–3 for the zirconia-based CSP columns are comparable to the performance of silica-based columns having a similar chiral selector.

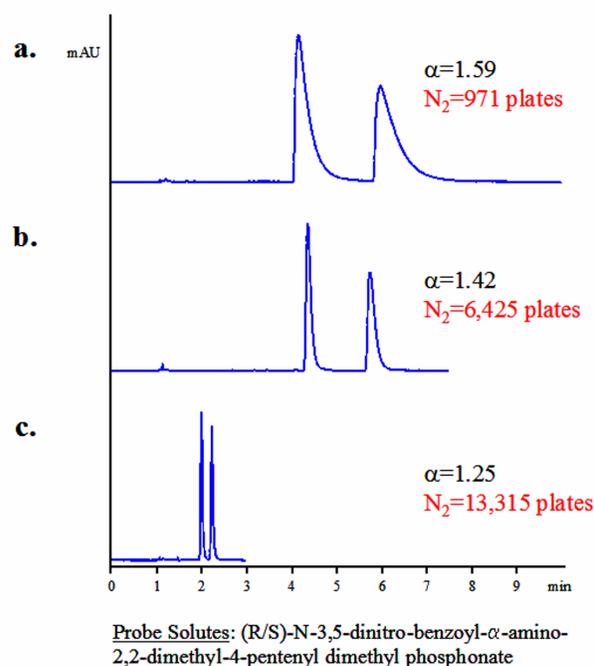
### Experimental

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:

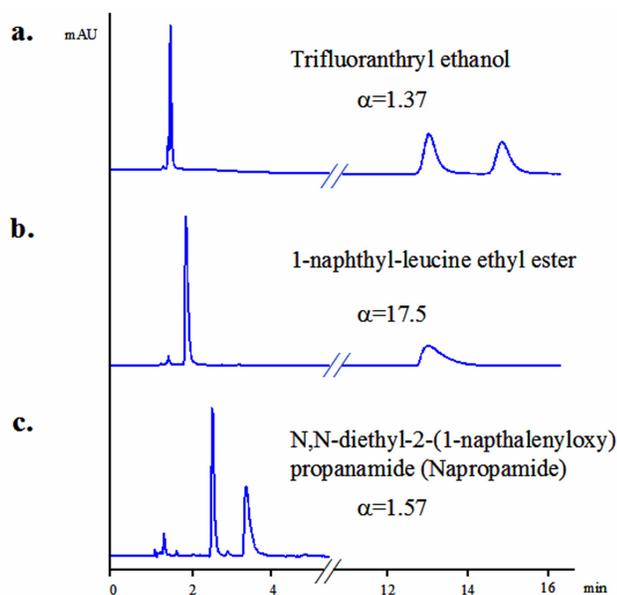
Column: See Figures, All 100 mm x 4.6 mm columns  
Mobile Phase: See Figures  
Temperature: 30 °C with Metalox™ 200-C column heater  
Flow Rate: 1.0 ml/min.  
Injection Vol.: 0.5 µl  
Pressure Drop: 195 bar  
Detection: UV at 254 nm



**Figure 1:** ZirChrom®-Chiral(R)NESA CSP column (part # ZRC02-1046) Selectivity Examples. Conditions: a) and b) 88.9/11/0.1 hexane/isopropanol/TFA (trifluoroacetic acid), c) 75/15/9/1 hexane/methanol/isopropanol/TFA.



**Figure 2:** Effect of mobile phase changes on ZirChrom®-Chiral(R)NESA CSP column (part # ZRC02-1046). Conditions: a) 89/11 hexane/isopropanol, b) 95/2/3 hexane/methanol/isopropanol, c) 87/10/3 hexane/methanol/isopropanol.



**Figure 3:** ZirChrom<sup>®</sup>-Chiral(S)LEU CSP column (part # ZRC01-1046) Selectivity Examples. Conditions: 99/1 hexane/isopropanol (all runs).

ZirChrom<sup>®</sup>-Chiral stationary phases are available in packed columns with a growing variety of chiral selectors. Currently, ZirChrom offers five different chiral columns in the ZirChrom<sup>®</sup>-Chiral line:

- ZirChrom<sup>®</sup>-Chiral(S)LEU; Chiral Selector(CS): (S)-3,5-dinitrobenzoyl-leucine, Part# ZRC01
- ZirChrom<sup>®</sup>-Chiral(R)NESA; CS: (R)-N-[1-(1-naphthyl)ethyl]succinamic acid, Part# ZRC02
- ZirChrom<sup>®</sup>-Chiral(S)NESA; CS: (S)-N-[1-(1-naphthyl)ethyl]succinamic acid, Part# ZRC03
- ZirChrom<sup>®</sup>-Chiral(S)PG; CS: (S)-3,5-dinitrobenzoyl-phenylglycine, Part# ZRC04
- ZirChrom<sup>®</sup>-Chiral(R)PG; CS: (R)-3,5-dinitrobenzoyl-phenylglycine, Part# ZRC05

Product development is underway for a single, highly stable zirconia column plus a kit of pure CSP coating reagents that will allow users to easily remove and replace chiral selectors by reproducible and simple methods. Chiral selectors with multiple chiral centers, featuring both  $\pi$ -donor and  $\pi$ -acceptor groups, are also under development, as are chiral selectors based on polysaccharides.

Please contact ZirChrom technical support at 1-866-STABLE-1 or [support@zirchrom.com](mailto:support@zirchrom.com) for more information regarding this exciting new technology.

## References

(1) American Laboratory, 37, No. 21, pp 22-4 (October 2005).

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