Online Regeneration of ZirChrom®-Chiral Zirconia-based CSP

Richard Henry, Ph.D.*, Bingwen Yan, Ph.D.*, Clayton McNeff, Ph.D.* and Thomas Hoye, Ph.D.**
*ZirChrom Separations, Inc.
**University of Minnesota

Technical Bulletin # 317

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. The following details the reaction scheme and reproducibility of the online regeneration of these new versatile, durable, and efficient ZirChrom®-Chiral zirconia-based chiral stationary phases (CSP).

Introduction

In this application note we present a method for the online regeneration of zirconia-based chiral columns. The phase is created using a simple single-step reaction scheme. The reproducibility of column regeneration is demonstrated by measuring the separating capability of the phase is examined before and after the stripping procedure as well as after regeneration. This new technology, targeted for future commercialization, will allow users to rapidly evaluate the suitability of different chiral selectors for resolving enantiomers with a single, highly stable zirconia column plus a kit of pure CSP coating reagents.

Experimental

The separation conditions were as follows:

Columns:	ZirChrom-PHASE, 100 mm x 4.6 mm i.d. (Part Number: ZR02-1046)
Mobile Phase:	See Figures
Temperature:	30 ºC 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 illustrates the single-step reaction scheme that enables chiral selectors to be easily removed and replaced by simple, low-temperature reactions.

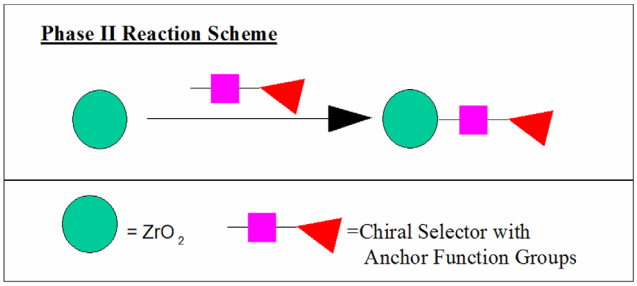

Figure 1: A chiral reagent containing an integral tethering group is attached to bare zirconia in a simple, one-step process.

A separation on a zirconia-based chiral column prepared with N-(4-nitrobenzoyl)-L-glutamic acid using a single-step reaction process is shown in *Figure 2*; the column was stripped and regenerated to demonstrate reproducibility of the simple, one-step loading process. After stripping, the bare zirconia column did not exhibit any retention or selectivity for the chiral analytes but resumed its initial performance after regeneration.

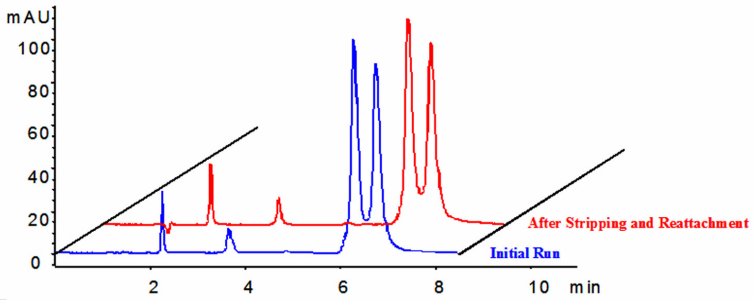

Figure 2: Comparison between the separation of (R/S)-2,2,2-trifluoro-1-(9-anthryl)ethanol before and after stripping and reattaching an N-(4-nitrobenzoyl)-L-glutamic acid (CSP) reagent according to the scheme shown in *Figure 1*. Mobile phase conditions: 99/1 hexane/isopropanol.

ZirChrom now offers a full line of chiral phases. ZirChrom chiral phases utilize a fully renewable platform and offer unique selectivity, high efficiency, and excellent stability. Please contact ZirChrom technical support at 1-866-STABLE-1 or support@zirchrom.com for more information regarding this exciting new technology.

References

(1) American Laboratory, 37, No. 21, pp 22-4 (October 2005).
(2) Phase II SBIR Grant (NIH R44 HL070334-02A2).

ZirChrom Separations, Inc.
617 Pierce Street, Anoka, MN 55303
1-866-STABLE-1
support@zirchrom.com

Visit www.zirchrom.com for more application notes using ultra-stable, high efficiency ZirChrom columns.