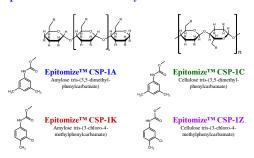


Abstract

Polysaccharide-based chiral stationary phases are well-known for their broad applicability in the separation of optical isomers and are available for both analytical and preparative applications. The introduction of UHPLC offers chromatographers increased productivity and efficiency by dramatically decreasing the turnaround times required for analyzing samples and by lowering solvent costs.

Orochem Technologies has combined the high efficiency of 1.7 micron silica gel particles with the broad applicability of polysaccharide-based chiral stationary phases to produce the Epitomize M 1.7 Micron class of chiral stationary phases. These new columns permit the very rapid separation of all the racemates separable on their 3 micron and larger analogs. Additionally, they are particularly useful for LC-MS and inline process monitoring. Mobile phase usage and turnaround times are typically cut to less than 10% of those observed for their 5 micron particle size analogs that use larger column dimensions. As for all Epitomize CSPs, the 1.7 micron series is available in four different chiral phases, which can be run in normal, polar, or reversed phases.

EpitomizeTM Chiral Stationary Phases



UHPLC Conditions

Instrumentation

Pump: ECOM Alpha 5

Injector: Rheodyne 7520 with a 0.5 µL internal loop

Detector: Shimadzu SPD-20A equipped with a semi-micro flow cell

Conditions

Columns: 1.7 Micron Epitomize™ CSP-1A

1.7 Micron Epitomize™ CSP-1C

Column Dimensions: 2.1 mm I.D. x 50 mm long

Injection Volume: 0.5 uL

Sample Concentration: Adjusted for detector response

Mobile Phases: Blends of 2-propanol and heptane, methanol, or acetonitrile

Mobile Phase Modifiers: Diethylamine or ethanolamine at 0.1%

Flow Rate: 0.15 mL/min Detection: UV at 254 nm Temperature: Ambient

EpitomizeTM 1.7 Micron Chiral Stationary Phases

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Table 1. Selection Guide using examples of racemates separated using the Epitomize CSPs. The three columns under "Conditions" describe the mobile phase and whether or not it has been modified using basic (e.g., ethanolamine, diethylamine, or triethylamine) or acidic (trifluoroacetic acid or acetic acid) modifiers. The two columns under "Phase" note if a normal or polar mobile phase was used. An asterisk indicates the chromatogram is shown.

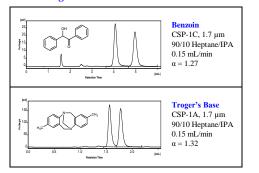
Racemate		CSP Type			Conditions			Phase	
		1C	1K	1Z	Neutral	Basic	Acidic	Normal	Pola
* Benzoin	X	Х	П		X			X	
Benzyl (2S,3qR,7qS)-octahydroindole-2-carboxylate	\top	Х	П	Х	Х			Х	
Bi-2-naphthol, 1,1'-	X	Х			X			X	X
Butanediol-dibenzoate, 1,3-	\top	Х	П		X			X	
Citalopram	Т	Х				X		X	X
* Flavanone	X	Г	Х		X			X	X
lbuprofen	\top	Х	П				X	X	
Indanol, 1-	Т	Х			X			X	
Methyl phenyl sulfoxide	┰	Х	П		X			X	
Methyl-2-naphthalenemethanol, alpha	\top	Х	П	Х	X			X	
* Mianserin	\top	Х	Х		Х	X		X	X
N,N'-bis-(benzyloxy)-piperazic acid-t-butylester	┰	Х	П	Х	X			X	
Naphthyl)ethanol, 1-(1-	\top	Х	П		X			X	X
* Phenoxy-2-propanol, 1-	\top	Х	П		Х			Х	
Phenyl-1-cyclohexanol, trans-2-	X	г	П	П	Х				X
Phenylethanediol, 1-	\top	Г	Х		X			X	
Phenylethanol, 1-	\top	Х	П		Х			X	
* Pindolol	┰	Х	П			X			X
* Propranolol	Х	Х	П			X		X	Х
* Stilbene Oxide, trans-	X	Х	П	Х	Х			X	
Tetrahydro-1-naphthol, 1,2,3,4-	┰	г	П	Х	X			X	
* Troger's Base	Х	Г	П		X			X	Х
Warfarin	\top	Х					Х		Х

Table 2 summarizes a comparison of the 1.7 micron Epitomize CSP-1C with its 5 micron analog. The test racemate was *trans*-stilbene oxide. The 1.7 and the 5 micron CSPs used column hardware typical for their particle size. The mobile phase and the flow rate were kept constant. The 1.7 micron column required only 0.8 minutes and 0.4 mL of mobile phase to complete the separation, whereas the 5 micron column required 12 minutes and 4.6 mL of mobile phase to complete a similarly efficient separation.

Column, mm	4.6 x 250	2.1 x 50		\wedge
Mobile Phase	10% IPA-hept	10% IPA-hept		
Flow Rate, mL/min	0.50	0.50	U	
Backpressure, psi	330	3620		
Time of Sep, min	12	0.8		
Mobile Phase, mL	Phase, mL 4.6			
- 1 (Milya)	And the State of t	20	1 2	- 1 (34) to (80) or (80) (
5 µm		1.7	μm	

1-Phenoxy-2propanol CSP-1C, 1.7 µm 90/10 Heptane/IPA 0.15 mL/min $\alpha = 1.84$ Mianserin CSP-1A, 1.7 um 90/10/0.1 Heptane/IPA/DEA 0.15 mL/min $\alpha = 1.42$ CSP-1A, 1.7 um Methanol 0.15 mL/min $\alpha = 2.11$ Propranolol CSP-1C, 1.7 um 80/20/0.1 Heptane/IPA/EA 0.15 mL/min $\alpha = 1.53$ **Pindolol** CSP-1C, 1.7 µm 100/0.1Acetonitrile/EA 0.15 mL/min $\alpha = 1.23$

Chromatograms



Summar

The Epitomize 1.7 Micron chiral stationary phases are specifically designed for use with UHPLC and offer the chromatographer fast throughput, low solvent consumption, quick column equilibration, and inline process monitoring. Their high efficiency and low mobile phase requirements make them particularly useful for LC-MS applications. They can separate any of the racemates separable on larger particle sizes and under similar chromatographic conditions. As with other Epitomize CSPs, the 1.7 micron series can be used in normal, polar, and reversed phases. Reversed phase conditions can alter the characteristics of polysaccharide-based CSPs; therefore, it is recommended that a separate set of CSPs be dedicated for reversed phase use.

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