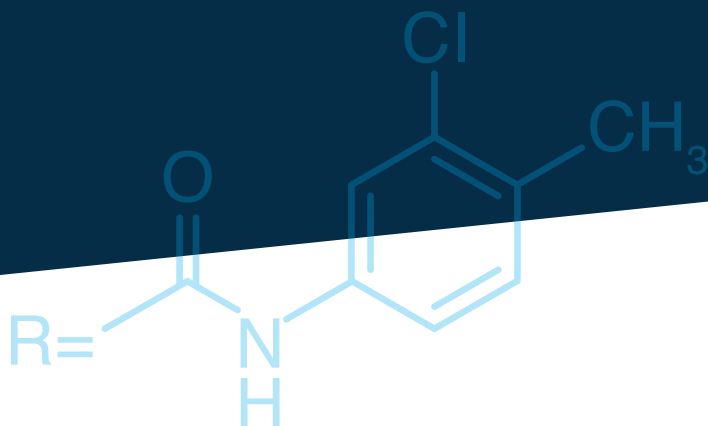


The best separations demand the best columns

INTRODUCING DAICEL'S NEW CHIRALPAK® IM



CHIRALPAK® IM is the immobilized equivalent of CHIRALCEL® OZ-H

Designed for HPLC and SFC to improve methods for challenging separations

5 micron available – other particle sizes to follow

DAICEL
Chiral Technologies

Item Code	Description
92311	CHIRALPAK IM, Guard Cartridge (3)
92324	CHIRALPAK IM, 4.6x150mm, 5µm
92325	CHIRALPAK IM, 4.6x250mm, 5µm
92335	CHIRALPAK IM, 10x250mm, 5µm
92337	CHIRALPAK IM, 10x20mm, 5µm Guard
92342	CHIRALPAK IM, 20x50mm, 5µm Guard
92345	CHIRALPAK IM, 20x250mm, 5µm
92355	CHIRALPAK IM, 50x250mm, 5µm
92372	CHIRALPAK IM, 30x50mm, 5µm Guard
92375	CHIRALPAK IM, 30x250mm, 5µm
92394	CHIRALPAK IM, 2.1x150mm, 5µm
92422	CHIRALPAK IM/SFC, 4.6x50mm, 5µm
92423	CHIRALPAK IM/SFC, 4.6x100mm, 5µm
92432	CHIRALPAK IM/SFC, 10x50mm, 5µm Guard
92435	CHIRALPAK IM/SFC, 10x250mm, 5µm
92442	CHIRALPAK IM/SFC, 21x50mm, 5µm Guard
92445	CHIRALPAK IM/SFC, 21x250mm, 5µm
92455	CHIRALPAK IM/SFC, 50x250mm, 5µm
92472	CHIRALPAK IM/SFC, 30x50mm, 5µm Guard
92475	CHIRALPAK IM/SFC, 30x250mm, 5µm

Improved Chiral Separation of Mephenesin on CHIRALPAK® IM

Dr. Weston J. Umstead, John M. Ferraro

INTRODUCTION

Mephenesin (Figure 1) is a cresol glyceryl ether that has been used for several indications including as a muscle relaxant, an antidote for strychnine poisoning, and as a treatment for Parkinson's disease and Multiple Sclerosis.

The chiral separation of Mephenesin was previously reported on CHIRALCEL® OZ-H, which is a coated tris (3-chloro-4-methylphenylcarbamate) cellulose-based chiral stationary phase (CSP). The separation with a normal phase alkane/alcohol mobile phase afforded just baseline resolution of the two enantiomers.

Daicel's newest immobilized CSP, CHIRALPAK® IM, is the immobilized equivalent of CHIRALCEL® OZ-H. The added robustness coming from the immobilization process increases this selector's utility because of expanded solvent compatibility. This resulted in a significant improvement of the enantiomeric separation with the additional of ethyl acetate to the mobile phase.

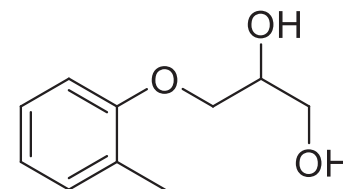


Figure 1: Mephenesin

Chromatographic Conditions for the Separation of Mephenesin

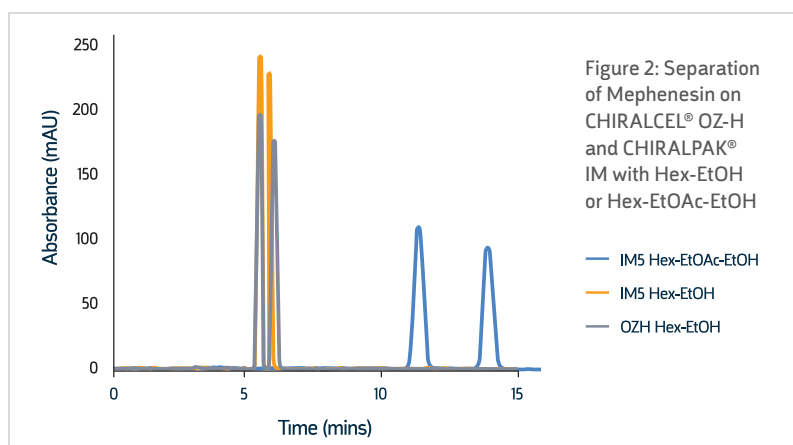
Column	CHIRALCEL® OZ-H (250 mm x 4.6 mm i.d.) Part #: 42325	CHIRALPAK® IM (250 mm x 4.6 mm i.d.) Part #: 92325
Mobile Phase	Hex-EtOH = 80-20 (v/v)	Hex-EtOH = 80-20 (v/v) Hex-EtOAc-EtOH = 70-30-0.1 (v/v/v)
Flow Rate	1.0 ml/min	
Detection	UV 280 nm ref. 450 nm	
Temperature	25°C	
Sample	1.9 mg/ml in EtOH	
Injection Volume	5 µl	

EXPERIMENTAL

Mephenesin was purchased from Sigma Aldrich and used as-is. The solvents used were all purchased from Pharmco, were HPLC-grade or higher, and were used as-is. Specifically the hexanes (Hex) contained 95% n-hexane. The ethanol (EtOH) was reagent alcohol, which contains 90% EtOH, 5% methanol, and 5% isopropanol (v/v/v). Initial separation check and optimization were performed on an Agilent 1200 equipped with a quaternary mixing pump and utilized a DAD.

DISCUSSION

Mephenesin was prepared as a 1.9 mg/ml solution in EtOH, and the separation from Daicel's application database (Hex-EtOH = 80-20 (v/v)) checked on both CHIRALCEL® OZ-H and CHIRALPAK® IM (Figure 2). This demonstrated almost baseline resolution on both columns. The separation on IM was optimized by the addition of EtOAc and removal of most EtOH (as indicated in the table above). This yielded a significant improvement in the chiral resolution, producing a greater-than-baseline resolution (Figure 2).



CONCLUSIONS

The increased robustness of immobilized chiral selector technology is critical for finding new chiral separations, and improving existing separations as demonstrated with the separation of Mephenesin. Under traditional normal phase conditions, the resolution was just baseline, and provided for minimal room for further optimization. The addition of EtOAc greatly influenced the analyte interactions with the CSP, and produced a greatly improved resolution on CHIRALPAK® IM.