CHIRAL TECHNOLOGIES EUROPE
SUBSIDIARY OF DAICEL CHEMICAL INDUSTRIES, LTD.

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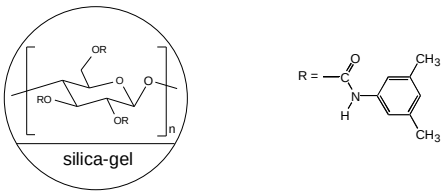
INSTRUCTION MANUAL FOR CHIRALCEL® OD COLUMNS

Please read this instruction sheet completely before using this column

Column description:

Packing composition: **Cellulose tris (3,5-dimethylphenylcarbamate) coated on 10µm silica-gel.**



Shipping solvent: **n-Hexane / 2-propanol solvent mixture (90:10 v/v)**

All columns have been pre-tested before packaging. Test parameters and results, as well as the Column Lot Number, are included on a separate (enclosed) page.

CAUTION

The entire HPLC system including the injector and the injection loop must be flushed with a solvent compatible with the column and its storage solvent prior to connecting. Many of the solvents commonly used in HPLC eluents such as acetone, chloroform, DMF, dimethylsulfoxide, ethyl acetate, methylene chloride and THF may DESTROY the chiral stationary phase if they are present, even in residual quantities, in the system.
If an auto-sampler is used, then the solvent employed to flush this unit between injections should also be changed and the relevant solvent lines flushed.

Operating restrictions

	250 x 4.6 mm ID Analytical column	250 x 10 mm ID Semi-Prep. column	250 x 20 mm ID Semi-Prep. column
Flow rate direction	As indicated on the column label		
Typical Flow rate •	~ 1ml/min Do not exceed 1.5ml/min	~ 5ml/min Do not exceed 7ml/min	~ 18ml/min Do not exceed 25ml/min
Pressure limitation ,	Should be maintained < 30 Bar (~430 psi) ƒ for maximum column life Do not exceed 50 Bar (~700 psi)		
Temperature	0 to 40°C		

- **The maximum flow rate depends on the mobile phase viscosity (mobile phase composition), and should be adjusted in accordance with the pressure upper's limit (i.e. 50 Bar).**

Examples	Column 250 x 4.6mm ID	Column 250 x 10mm ID	Column 250 x 20mm ID
Alkane / Alcohol mixture ~ 90:10	1.0 to 1.5 ml/min	5 to 7 ml/min	18 to 25 ml/min
100% EtOH	~ 0.5 ml/min	~ 2 to 3 ml/min	~ 5 to 8 ml/min
100% 2-propanol	~ 0.2-0.3 ml/min	~ 1 ml/min	~ 3 to 5 ml/min

, The back pressure value that should be taken into account is the one generated by the column itself. This value is measured by calculating the difference between the pressure of [LC system + column] and the pressure of the LC system free of the column.

f Ideal value for maximum column life, but stable up to 50 Bar.

Operating procedure

M ***Please contact CHIRAL TECHNOLOGIES EUROPE for further assistance before trying any solvents not mentioned below.***

A - Mobile phases

	Alkane Œ/ 2-propanol •	Alkane Œ/ Ethanol •	Alkane Œ/ Methyl-*tert*-butyl ether (MTBE)	Alkane Œ/ MeOH Ž	MeOH • + •
CHIRALCEL® OD 250 x 4.6 mm ID 250 x 10 mm ID 250 x 20 mm ID	100/0 to 0/100	100/0 to 0/100	100/0 to 50/50	100/0 to 0/100	0 to 100% EtOH or IPA in MeOH

Œ Alkane: n-hexane or iso-hexane or n-heptane. Some small selectivity differences may sometimes be found.

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- The retention is generally shorter with Ethanol than with 2-propanol.
- The retention is generally shorter with a higher alcohol content.
- The use of other alcohols such as 1-propanol, 1-BuOH, 2-BuOH etc...is possible, but effectiveness cannot be guaranteed.

Ž Due to limited miscibility of MeOH in Alkane, it is necessary to add an appropriate volume of EtOH together with MeOH in order to obtain an homogenous solvent mixture.
A maximum of 5% MeOH in n-hexane only may be used without adding EtOH.

• Ideal starting conditions: MeOH/EtOH 50:50 (v/v) when alcohol mixtures are required

• The use of polar solvents as 100% methanol is possible with CHIRALCEL® OD columns. Nevertheless once the column is transferred to a polar mode it should be dedicated to this specific application.

To safely transfer the column from hexane to methanol it is strongly recommended to use 100% 2-propanol as a transition mobile phase at a low flow rate (high viscosity of 2-propanol).

B – Additives

For basic samples or acidic samples, it is necessary to add a modifier into the mobile phase in order to achieve the chiral separation:

Basic Samples Require Basic modifiers	Acidic Samples Require Acidic modifiers
DEA Butyl amine ‘ Ethanol amine ’	TFA CH_3COOH
< 0.5% Typically 0.1%	< 0.5% Typically 0.1%

‘ For primary amines mainly
’ For primary amino alcohols mainly

Column care / Maintenance

- The use of a guard column is highly recommended for maximum column life.
- Samples should be dissolved in the mobile phase and should be filtered through a membrane filter of approximately 0.5µm porosity.
- For alkane containing mobile phases, flush the column with Storage Solvent (Hexane / 2-propanol 9:1) when stored for more than one week.
- For columns dedicated to polar solvents, flush the column with the regular mobile phase without the modifier.

F When washing is required, use pure Ethanol at an appropriate flow rate for 3 hours. (Columns used with alkane/alcohol mobile phase only).

Important Notice

ð STRONGLY BASIC solvent modifiers or sample solutions MUST BE AVOIDED, because they are likely to damage the silica gel used in this column.

ð <u>This instruction sheet is not applicable to any other DAICEL columns.</u>

ð If you have any questions about the use of this column, or encounter a problem, please contact <u>CHIRAL TECHNOLOGIES EUROPE</u> for assistance (cte@chiral.fr)

Operating this column in accordance with the guidelines outlined here will result in a long column life.

For more detailed information, refer to our catalogue also available on our website http://www.chiral.fr or contact Chiral Technologies Europe.

Columns packed with 20µm material dedicated to preparative scale applications are also available from Chiral Technologies Europe.

TABLE OF DAICEL CHIRAL COLUMNS

Type of Adsorbent	Column Trade Name	Phase Type		Particle Size	
		Normal Phase	Reversed phase	5 µm	10µm
Immobilized Amylose Carbamate	CHIRALPAK® IA	W	W	W	
Amylose Carbamates	CHIRALPAK® AD	W			W
	CHIRALPAK® AD-H	W		W	
	CHIRALPAK® AD-RH		W	W	
	CHIRALPAK® AS	W			W
	CHIRALPAK® AS-H	W		W	
	CHIRALPAK® AS-RH		W	W	
Immobilized Cellulose Carbamate	CHIRALPAK® IB	W	W	W	
Cellulose Carbamates	CHIRALCEL® OD	W			W
	CHIRALCEL® OD-H	W		W	
	CHIRALCEL® OD-R		W		W
	CHIRALCEL® OD-RH		W	W	
	CHIRALCEL® OC	W			W
	CHIRALCEL® OF	W			W
	CHIRALCEL® OG	W			W
Cellulose Esters	CHIRALCEL® OJ	W			W
	CHIRALCEL® OJ-H	W		W	
	CHIRALCEL® OJ-RH		W	W	
	CHIRALCEL® OA	W			W
	CHIRALCEL® OB	W			W
	CHIRALCEL® OB-H	W		W	
	CHIRALCEL® OK	W			W
	CHIRALCEL® CA	W		NA	NA
Immobilized Quinine & Quinidine Carbamates	CHIRALPAK® QD-AX	W	W	W	
	CHIRALPAK® QN-AX	W	W	W	
Crown Ether	CROWNPAK® CR(+)		W	W	
	CROWNPAK® CR(-)		W	W	
Ligand Exchange	CHIRALPAK® MA(+)		W	3 µm	
	CHIRALPAK® WH		W		W
Polymethacrylate	CHIRALPAK® OP(+)	W			W
	CHIRALPAK® OT(+)	W			W

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