INSTRUCTION MANUAL FOR CHIRALCEL® OD-H COLUMNS



Please read this instruction sheet completely before using this column

Column description:

Packing composition:

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

$$R = C$$
 CH_3
 CH_3
 CH_3

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

	150 x 4.6 mm ID 250 x 4.6 mm ID Analytical columns	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 ①	~ 1ml/min Do not exceed 1.5ml/min	~ 5ml/min Do not exceed 7ml/min	~ 18ml/min Do not exceed 25ml/min	
Pressure limitation ②	Should be maintained < 50 Bar (~700 psi) for maximum column life Do not exceed 100 Bar (~1400 psi)			
Temperature	0 to 40°C			

① 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. 100 Bar).

Examples	Column 250 x 4.6mm ID Column 150 x 4.6mm ID	Column 250 x 10mm ID	Column 250 x 20mm ID	
Alkane/Alcohol mixture ~ 90:10	0.5 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	



- ② 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.
- 3 Ideal value for maximum column life, but stable up to 100 Bar.

Operating procedure

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

A - Mobile phases

	Alkane 1 / 2-propanol 2	Alkane ① / Ethanol ②	Alkane ① / MeOH ③	Alkane 	MeOH 4 + 6	CH₃CN ⑤ + ⑥ No alkane at all
CHIRALCEL® OD-H 150 x 4.6 mm ID 250 x 4.6 mm ID	100/0	100/0	100/0	100/0	0 to 100% EtOH or IPA in MeOH	0 to 100% IPA in CH₃CN
250 x 10 mm ID 250 x 20 mm ID	to 0/100	to 0/100	to 0/100	to 50/50	0-15% (Max.) CH₃CN in MeOH	0 to 15% (Max.) MeOH or EtOH 7 in CH₃CN

• 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.
- Oue 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.
- 4 Ideal starting conditions: MeOH/EtOH 50:50 (v/v) when alcohol mixtures are required
- The use of polar solvents as 100% methanol or 100% acetonitrile is possible with CHIRALCEL® OD-H 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 or acetonitrile <u>or between different polar solvents</u>, <u>it is strongly recommended to use 100% 2-propanol as a transition mobile phase</u> at a low flow rate (high viscosity of 2-propanol).

6 The column needs to be thoroughly washed with acetonitrile (~ 10 column volume) prior to the first use in this solvent as mobile phase.

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- ☐ More than 15% of alcohol other than 2-propanol, in acetonitrile may destroy the column. Compatibility of such mixtures with the chiral stationary phase cannot be guaranteed (refer to the above table).
- Other alcohols such as 1-propanol, 1-BuOH, 2-BuOH etc...can also be used, but effectiveness cannot be guaranteed. Do not use mobile phases containing more than 15% of these alcohols.



B - Modifiers

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

8	For	primary	amines	mainly
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⁹ For primary amino alcohols mainly

Basic Samples	Acidic Samples
Require	Require
Basic modifiers	Acidic modifiers
DEA	
Butyl amine ®	TFA
Ethanol amine 9	CH₃COOH
0.50/	0.50/
< 0.5%	< 0.5%
Typically 0.1%	Typically 0.1%

Column care / Maintenance

- ☐ The use of a guard cartridge 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.
- 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

- \Rightarrow STRONGLY BASIC solvent modifiers or sample solutions MUST BE AVOIDED, because they are likely to damage the silica gel used in this column.
- ⇒ This instruction sheet is not applicable to any other DAICEL columns.
- \Rightarrow If you have any questions about the use of this column, or encounter a problem, please contact <u>CHIRAL TECHNOLOGIES EUROPE</u> for assistance (<u>cte@chiral.fr</u>)

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

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TABLE OF DAICEL CHIRAL COLUMNS

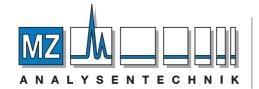


Type of Adsorbent	Column Trade Name	Phase Type		Particle Size	
			Reversed phase	5 µm	10µm
	CHIDALDAK® AD				
Amylose Carbamate	CHIRALPAK® AD	•			•
	CHIRALPAK® AD-H	•		•	
	CHIRALPAK® AD-RH		•	•	
	CHIRALPAK® AS	•			*
	CHIRALPAK® AS-H	•		•	
	CHIRALPAK® AS-RH		•	•	
	CHIRALCEL® OD	•			•
	CHIRALCEL® OD-H	•		•	
Cellulose Carbamate	CHIRALCEL® OD-R		•		•
	CHIRALCEL® OD-RH		•	•	
ochdiose carbamate	CHIRALCEL® OC	•			•
	CHIRALCEL® OF	•			•
	CHIRALCEL® OG	•			•
	CHIRALCEL® OJ	•			•
	CHIRALCEL® OJ-H	•		•	
	CHIRALCEL® OJ-RH		•	•	
	CHIRALCEL® OA	•			•
Cellulose Ester	CHIRALCEL® OB	•			•
	CHIRALCEL® OB-H	•		•	
	CHIRALCEL® OK	•			•
	CHIRALCEL® CA	•		NA	NA
Consider Falls and	CROWNPAK® CR(+)		•	•	
Crown Ether	CROWNPAK® CR(-)		•	•	
	onowiw /inco on()		•	•	
Ligand Exchange	CHIRALPAK® MA(+)		♦ 3 μι		μm
	CHIRALPAK® WH		•		•
	CHIRALPAK® OP(+)	•			•
Polymethacrylate	CHIRALPAK® OT(+)	• •			•

Columns packed with 20µm material dedicated to preparative scale applications (50 & 100mm I.D.) are also available from Chiral Technologies Europe.

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

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