

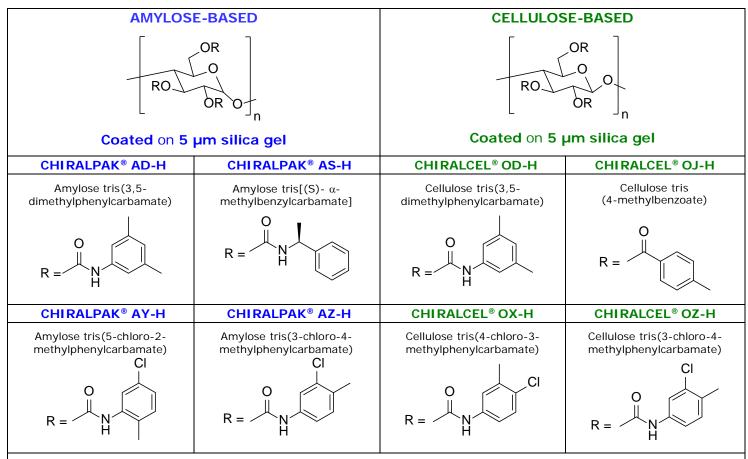


INSTRUCTION MANUAL FOR CHIRALPAK® AD-H, AS-H, AY-H, AZ-H CHIRALCEL® OD-H, OJ-H, OX-H, and OZ-H

<Normal Phase>

Please read this instruction sheet completely before using these columns

Column Description



Shipping Solvent: Hexane/Isopropanol = 90/10 (v/v)

All columns have been pre-tested before packaging. Test parameters and results, as well as the Column Lot Number, were included with the column when purchased.

THIS INSTRUCTION MANUAL IS NOT APPLICABLE TO ANY OTHER DAICEL COLUMNS

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 the column. Many of the solvents commonly used as HPLC eluents including acetone, chloroform, DMF, dimethylsulfoxide, ethyl acetate, methylene chloride, and THF, may DESTROY the chiral stationary phase if they are present, even in residual quantities, within the system.

If an auto-sampler is used, then the solvent employed to flush this unit between injections should also be changed to something compatible and the relevant solvent lines flushed.

Operating Instructions

	150 x 2.1 mm i.d. Analytical Column	50 x 4.6 mm i.d. 100 x 4.6 mm i.d. 150 x 4.6 mm i.d. 250 x 4.6 mm i.d. Analytical Columns	250 x 10 mm i.d.① 250 x 20 mm i.d.① 250 x 30 mm i.d.① 250 x 50 mm i.d.① Semi-Prep Columns		
Guard	//	10 x 4.0 mm i.d. Guard Cartridge	20 x 10 mm i.d. 50 x 21 mm i.d. 50 x 30 mm i.d. Guard Column		
Flow Rate Direction	As indicated on the column label				
Typical Flow Rate②	0.1-0.5 ml/min	0.5-2.5 ml/min	5 ml/min (10 mm i.d.) 20 ml/min (20 mm i.d.) 42 ml/min (30 mm i.d.) 118 ml/min (50 mm i.d.)		
Pressure Limitation③	Should be maintained < 300 Bar (4350 psi) for maximum column life Adapt flow rates to column size.				
Temperature	O to 40°C				
Column Fitting	Please contact Technical Support for details				

- ① When using a semi-preparative column, it is highly recommended to discard at least the first 150 ml (for 250 x 10 mm i.d) or 500 ml (for 250 x 20 mm i.d) of eluent at the beginning of each preparative work.
- (2) 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. 300 Bar).
- 3 The column pressure is the total pressure minus the system pressure. At a given temperature, the column back pressure is linearly proportional to the flow rate.

Operating Procedure

♠ Please contact Chiral Technologies for further assistance before trying any solvents not mentioned below.

A - Mobile Phases

CAUTION

Basic conditions SHOULD BE AVOIDED, both in the sample solution and the mobile phase, for CHIRALPAK® AZ-H.

	Alkane ① / 2-Propanol ②	Alkane 1/ Ethanol 2		MeOH 4 + 5	ACN 5 + 6 <u>No alkane</u> <u>at all</u>	Alkane ① / MtBE
CHIRALPAK® AD-H CHIRALPAK® AS-H CHIRALPAK® AY-H CHIRALPAK® AZ-H	100/0	100/0 to 0/100	100/0 to 85/15	0 to 100% EtOH or 2-PrOH in MeOH	0 to 100% 2-PrOH in ACN	100/0 to 85/15
CHIRALCEL® OD-H CHIRALCEL® OJ-H CHIRALCEL® OX-H CHIRALCEL® OZ-H	to 0/100			O to 15% ACN in MeOH 6	0 to 15% MeOH or EtOH in ACN 	

- Alkane: n-Hexane or iso-Hexane or n-Heptane. Some small selectivity differences have been observed when switching between these different alkanes.
- 2 The retention is generally shorter with Ethanol than with 2-Propanol, and the retention is generally shorter with higher alcohol contents. The use of other alcohols such as 1-Propanol, 1-BuOH, etc. is possible, but effectiveness is not predictable.
- Due to the limited miscibility of MeOH in Alkane, it is necessary to add an appropriate volume of EtOH, together with MeOH, to ensure a homogenous solvent mixtures. 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 organic solvents like 100% Methanol and/or 100% ACN is possible with CHIRALPAK® AD-H/AS-H/AY-H/AZ-H and CHIRALCEL® OD-H/OJ-H/OX-H/OZ-H columns. Once the column is transferred to a polar organic mode, it is recommended the column be dedicated for this specific application.
- **6** The column is particularly efficient in this range when such solvent mixtures are employed. Other alcohols such as 1-Propanol, 1-BuOH, etc. can also be used, but should not exceed 15% by volume.

To safely transfer the column from Hexane to Methanol or ACN, it is strongly recommended to use 100% EtOH or 2-PrOH as a transition mobile phase at 0.5 ml/min.

After this transition, the column needs to be thoroughly washed with ACN (~ 10 column volumes) prior to the first use in this solvent as a mobile phase.

B - Additives

For basic samples or acidic samples, it is necessary to add an additive into the mobile phase in order to achieve the chiral separation.

7 For primary amines mainly

8 For primary amino alcohols mainly

Basic Samples require	Acidic Samples require	
Basic additives	Acidic additives	
DEA Butyl amine ଡ Ethanol amine ଡ	TFA CH₃COOH	
< 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 preferably 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 / Isopropanol 90:10) when stored for more than one week.
- For columns dedicated to polar organic solvents, flush the column with the polar organic mobile phase, without the additive.
- When washing is required, use pure Ethanol at 0.5 ml/min for 1 to 3 hours. The column can also be heated to 40°C for a more efficient cleaning.

Important Notice

⇒ STRONGLY BASIC solvent additives or sample solutions MUST BE AVOIDED, because they are likely to damage the silica gel used in these columns.

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

⇒ If you have any questions about the use of these columns, or encounter a problem, contact:

In the USA: questions@cti.daicel.com or call 800-6-CHIRAL In the EU: <u>cte@cte.daicel.com</u> or call +33 (0) 3 88 79 52 00

In India: chiral@chiral.daicel.com or call +91 84 1866 0700 & 703

Locations:

North/Latin America

Chiral Technologies. Inc. 1475 Dunwoody Dr., Ste 310 West Chester, PA 19380 800 6 CHIRAL

Tel: 610-594-2100 Fax: 610-594-2325 chiral@cti.daicel.com www.chiraltech.com

Europe

Chiral Technologies Europe SAS Parc d'Innovation 160, Bd Gonthier d'Andernach CS80140 67404 Illkirch Cedex, France Tel: +33 (0) 3 88 79 52 00 Fax: +33 (0) 3 88 66 71 66

cte@cte.daicel.com www.chiraltech.com

Daicel Chiral Technologies (India) Pvt. Ltd. Survey No.542/2, IKP Knowledge Park, Turkapally, Shamirpet Mandal, Medchal-Malkajgiri District, Hyderabad-500101. Telangana, India Tel: +91 84 1866 0700 & 703

Fax: +91 84 1866 0730 chiral@chiral.daicel.com www.chiraltech.com

CHIRALCEL, CHIRALPAK, CROWNPAK and DAICEL DCpak are registered trademarks of DAICEL CORPORATION