



INSTRUCTION MANUAL FOR CHIRALPAK® AD

Please read this instruction sheet completely before using this column

Column Description

CHIRALPAK® AD

Amylose tris(3,5-dimethylphenylcarbamate) coated on 10µm silica-gel.

$$R = C$$

$$Silica-gel$$

$$R = C$$

$$CH_{2}$$

$$CH_{3}$$

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 Conditions

	250 x 4.6 mm i.d. Analytical column	250 x 10 mm i.d. Semi-Prep. column	250 x 20 mm i.d. Semi-Prep. column	
Flow rate direction	As indicated on the column label			
Typical Flow rate ①	~ 1ml/min	~ 5ml/min	~ 18ml/min	
Pressure limitation ②	Should be maintained < 150 Bar (2175 psi) for maximum column life Adapt flow rates to column size.			
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. 150 Bar).
- 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.

Operating Procedure

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

A - Mobile Phases

	Alkane 	Alkane ① / Ethanol ②	Alkane ① / MeOH ⑤	MeOH ❹+⑤	CH₃CN ⑤+⑥ <u>No alkane at all</u>
CHIRALPAK [®] AD	100/0	100/0 to 85/15	100/0	0 to 100% EtOH or IPA in MeOH	0 to 100% IPA in CH₃CN
	to and 0/100 40/60 to 0/100	to 85/15	0-15% (Max.) CH ₃ CN in MeOH	0 to 15% (Max.) MeOH or EtOH ② in CH₃CN	

- Alkane: n-hexane or iso-hexane or n-heptane. Some small selectivity differences may sometimes be found.
- ₱ The range Hexane/EtOH 85/15 ~ 40/60 may adversely affect baseline stability due to polymer leakage.
- To safely transfer the column from 15%EtOH to 60%EtOH, it is strongly recommended to use 100% 2-propanol as a transition mobile phase.
 - ☐ The retention is generally shorter with Ethanol than with 2-propanol.
 - ☐ The retention is generally shorter with higher alcohol contents.
 - ☐ The use of other alcohols such as 1-propanol, 1-BuOH, 2-BuOH etc...is possible, but effectiveness cannot be guaranteed. Do not use mobile phases containing more than 15% of these alcohols.
- 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.
- 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 CHIRALPAK® AD columns. Nevertheless once the column is transferred to a polar mode <u>it should be dedicated to this specific application</u>.

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>.

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

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:

- S For primary amines mainly
- For primary amino alcohols mainly

Basic Samples	Acidic Samples		
Require	Require		
Basic modifiers	Acidic modifiers		
DEA Butyl amine Ethanol amine	TFA CH₃COOH HCOOH		
< 0.5%	< 0.5%		
Typically 0.1%	Typically 0.1%		

Column Care / Maintenance

- ☐ The use of a guard column is highly recommended for maximum column life.
- \square 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 additive.
- When washing is required, flush pure Ethanol at for 3 hours. (Columns used with alkane/alcohol mobile phase only).
- Before flushing with 100% Ethanol use 100% 2-propanol as a transition mobile phase.

Important Notice

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

Operating this column 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:

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