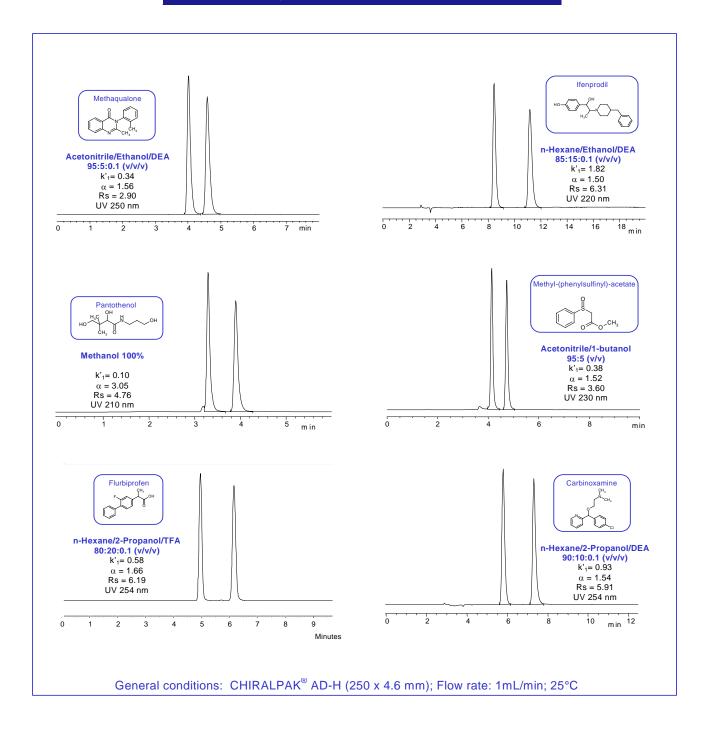
CHIRALPAK® AD-H **Analytical HPLC applications**



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INSTRUCTION MANUAL FOR CHIRALPAK® AD-H COLUMNS



Please read this instruction sheet completely before using this column

Column description

CHIRALPAK® AD-H

Amylose tris-(3,5-dimethylphenylcarbamate) coated on 5µm 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 conditions

	2.1 x 150 mm 2.1 x 250 mm Analytical columns	4.6 x 150 mm 4.6 x 250 mm Analytical columns	10 x 250 mm Semi-prep. columns	20 x 250 mm Semi-prep. columns
Flow rate direction	As indicated on the column label			
Typical Flow rate ①	~ 0.1 - 0.2 ml/min	~ 1 ml/min	~ 5 ml/min	~ 18 ml/min
Pressure limitation	Should be maintained < 300 Bar (4350 psi) for maximum column life Adapt flow rates to column size.			
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. 300 Bar).

Operating procedure

Please contact DAICEL CORPORATION 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 ⑤	MeOH 4 + 6	CH₃CN ⑤ + ⑥ <u>No Alkane at all</u>
CHIRALPAK®AD-H	100/0 100/0 to to 0/100 0/100		100/0	0 to 100% EtOH or IPA in MeOH	0 to 100% IPA in CH₃CN
		to 85/15	0-15% <mark>(Max.)</mark> CH₃CN in MeOH	0 to 15% (Max.) MeOH or EtOH ② in CH ₃ CN	

• 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 higher alcohol contents.
- ☐ The use of other alcohols such as 1-propanol, 1-BuOH, 2-BuOH etc...is possible, but effectiveness cannot be guaranteed.
- 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 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 solvents as 100% methanol or 100% acetonitrile is possible with CHIRALPAK® AD-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</u> solvents,

it is strongly recommended to use 100% 2-propanol as a transition mobile phase.

- 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 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:

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0	For	primary	amino	alcohols	mainl	У
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Basic Samples	Acidic Samples		
Require	Require		
Basic modifiers	Acidic modifiers		
DEA	TFA		
Butyl amine ®	CH₃COOH		
Ethanol amine ®	HCOOH		
< 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 additive.
- When washing is required, flush pure Ethanol for 3 hours. (Columns used with alkane/alcohol mobile phase only).
- $lue{lue{b}}$ Before flushing with 100% Ethanol <u>use 100% 2-propanol as a transition mobile phase</u>.

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.
- ⇒ If you have any questions about the use of these columns, or encounter a problem, please contact <u>DAICEL CORPORATION</u> for assistance (<u>chiral@ip.daicel.com</u>).

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

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