

**INSTRUCTION MANUAL FOR Analytical Columns of  
CHIRALPAK® AD-3, AS-3, AY-3, AZ-3  
CHIRALCEL® OD-3, OJ-3, OX-3, and OZ-3**

<Normal Phase>

**Please read this instruction sheet completely before using these columns**

**Column Description**

<p><b>AMYLOSE-BASED</b></p> <p><b>Coated on 3 µm silica gel</b></p>		<p><b>CELLULOSE-BASED</b></p> <p><b>Coated on 3 µm silica gel</b></p>	
<p><b>CHIRALPAK® AD-3</b></p> <p>Amylose tris(3,5-dimethylphenylcarbamate)</p>	<p><b>CHIRALPAK® AS-3</b></p> <p>Amylose tris[(S)-α-methylbenzylcarbamate]</p>	<p><b>CHIRALCEL® OD-3</b></p> <p>Cellulose tris(3,5-dimethylphenylcarbamate)</p>	<p><b>CHIRALCEL® OJ-3</b></p> <p>Cellulose tris(4-methylbenzoate)</p>
<p><b>CHIRALPAK® AY-3</b></p> <p>Amylose tris(5-chloro-2-methylphenylcarbamate)</p>	<p><b>CHIRALPAK® AZ-3</b></p> <p>Amylose tris(3-chloro-4-methylphenylcarbamate)</p>	<p><b>CHIRALCEL® OX-3</b></p> <p>Cellulose tris(4-chloro-3-methylphenylcarbamate)</p>	<p><b>CHIRALCEL® OZ-3</b></p> <p>Cellulose tris(3-chloro-4-methylphenylcarbamate)</p>

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

	50 x 2.1 mm i.d. 100 x 2.1 mm i.d. 150 x 2.1 mm i.d. 250 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
<b>Guard</b>	//	<b>10 x 4.0 mm i.d.</b> Guard Cartridge
Flow Rate Direction	As indicated on the column label	
Typical Flow Rate ①	0.1-0.5 ml/min	0.5-2.5 ml/min
Pressure Limitation②	Should be maintained < 300 Bar (4350 psi) for maximum column life <a href="#">Adapt flow rates to column size.</a>	
Temperature	0 to 40°C	
Column Fitting	Please contact <a href="#">Technical Support</a> for details	

① Flow rates in the range of 0.5-1.0 ml/min are recommended for difficult resolution of enantiomers (search of the best resolution). Flow rates higher than 1.0 ml/min, especially 3.0-5.0 ml/min, are advised for fast analyses.

② 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-3.**

	Alkane <sup>①</sup> / 2-Propanol <sup>②</sup>	Alkane <sup>①</sup> / Ethanol <sup>②</sup>	Alkane <sup>①</sup> / MeOH <sup>③</sup>	MeOH <sup>④</sup> + <sup>⑤</sup>	CH <sub>3</sub> CN <sup>⑤</sup> + <sup>⑥</sup> <u>No alkane at all</u>	Alkane <sup>①</sup> / MtBE
<b>CHIRALPAK<sup>®</sup> AD-3</b> <b>CHIRALPAK<sup>®</sup> AS-3</b> <b>CHIRALPAK<sup>®</sup> AY-3</b> <b>CHIRALPAK<sup>®</sup> AZ-3</b> <b>CHIRALCEL<sup>®</sup> OD-3</b> <b>CHIRALCEL<sup>®</sup> OJ-3</b> <b>CHIRALCEL<sup>®</sup> OX-3</b> <b>CHIRALCEL<sup>®</sup> OZ-3</b>	100/0 to 0/100	100/0 to 0/100	100/0 to 85/15	0 to 100% EtOH or CH <sub>3</sub> CN in MeOH	0 to 100% EtOH or MeOH in CH <sub>3</sub> CN	100/0 to 85/15

① 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, 2-BuOH, etc. is possible, but effectiveness is not predictable.

③ 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.

⑤ The use of polar organic solvents like 100% Methanol and/or 100% Acetonitrile is possible with CHIRALPAK<sup>®</sup> AD-3/AS-3/AY-3/AZ-3 and CHIRALCEL<sup>®</sup> OD-3/OJ-3/OX-3/OZ-3 columns. Once the column is transferred to a polar organic mode, **it is recommended the column be dedicated for this specific application.**

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

After this transition, the column needs to be thoroughly washed with Acetonitrile (~ 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 Basic additives	Acidic Samples require Acidic additives
DEA Butyl amine <sup>⑦</sup> Ethanol amine <sup>⑧</sup>	TFA CH <sub>3</sub> COOH
< 0.5% Typically 0.1%	< 0.5% 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](mailto:questions@cti.daicel.com) or call 800-6-CHIRAL

In the EU: [cte@cte.daicel.com](mailto:cte@cte.daicel.com) or call +33 (0) 3 88 79 52 00

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