DAICEL
Chiral Technologies

INSTRUCTION MANUAL FOR CHIRALPAK® IA-10, IB N-10, IC-10, ID-10, IE-10, IF-10, IG-10, IH-10, IJ-10, IK-10, and IM-10

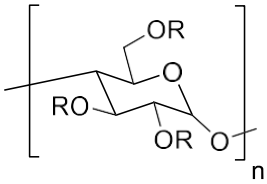
<Normal Phase>

Please read this instruction manual completely before using these columns.

These columns can also be used in reversed-phase and SFC. Please refer to the corresponding instruction manual for details.

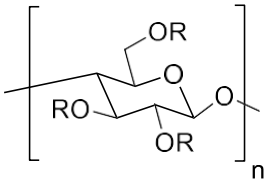
Column Description

AMYLOSE-BASED



Immobilized on **10 µm silica gel**

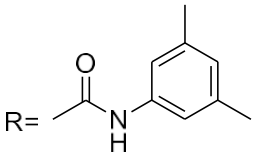
CELLULOSE-BASED



Immobilized on **10 µm silica gel**

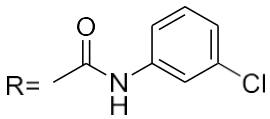
CHIRALPAK® IA-10

Amylose tris(3,5-dimethyl-phenylcarbamate)



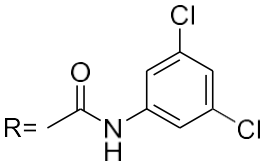
CHIRALPAK® ID-10

Amylose tris(3-chloro-phenylcarbamate)



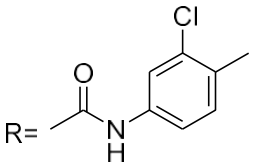
CHIRALPAK® IE-10

Amylose tris(3,5-dichlorophenylcarbamate)



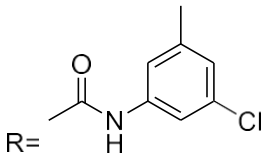
CHIRALPAK® IF-10

Amylose tris(3-chloro-4-methylphenylcarbamate)



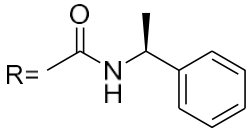
CHIRALPAK® IG-10

Amylose tris(3-chloro-5-methyl-phenylcarbamate)



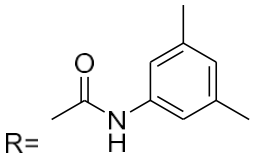
CHIRALPAK® IH-10

Amylose tris[(S)-α-methylbenzylcarbamate]



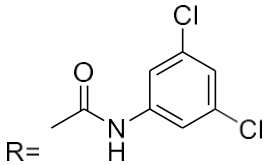
CHIRALPAK® IB N-10

Cellulose tris(3,5-dimethylphenylcarbamate)



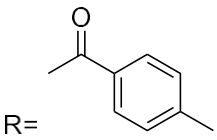
CHIRALPAK® IC-10

Cellulose tris(3,5-dichlorophenylcarbamate)



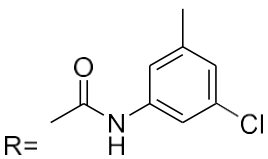
CHIRALPAK® IJ-10

Cellulose tris(4-methylbenzoate)



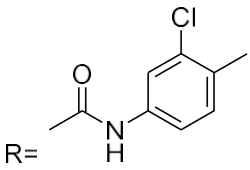
CHIRALPAK® IK-10

Cellulose tris(3-chloro-5-methylphenylcarbamate)



CHIRALPAK® IM-10

Cellulose tris(3-chloro-4-methylphenylcarbamate)



Shipping Solvent:

Hexane/IPA = 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.

Because different columns, including custom columns, can be shipped in different solvents, we recommend flushing them with 100% Ethanol or Isopropanol, at the typical flow rate listed below, before their first use to avoid any damage.

THIS INSTRUCTION MANUAL IS NOT APPLICABLE TO ANY OTHER DAICEL COLUMNS

Operating Instructions

	150 x 2.1 mm i.d. Analytical Column	**150 x 4.6 mm i.d.** **250 x 4.6 mm i.d.** Analytical Columns
Guard	**//**	**50 x 4.6 mm i.d.** Guard Column
Flow Rate Direction	As indicated on the column label	
Typical Flow Rate	0.1-0.5 ml/min	0.5-2.5 ml/min
Pressure Limitation①	Should be maintained < 300 Bar (4350 psi) for maximum column life Adapt flow rates to column size.	
Temperature	0 to 40°C	
Column Fitting	Please contact Technical Support for details	

① 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.

Method Development / Normal Phase

A - Mobile Phases

CHIRALPAK® IA-10, IB N-10, IC-10, ID-10, IE-10, IF-10, IG-10, IH-10, IJ-10, IK-10, and IM-10 can be used *with all ranges of organic miscible solvents,* progressing from the traditional mobile phases used with other DAICEL columns (mixtures of alkanes/alcohol, pure alcohol or acetonitrile (CH_3CN)) to mobile phases containing methyl *tert*-butyl ether (MtBE), tetrahydrofuran (THF), dichloromethane (DCM), chloroform ($CHCl_3$), and ethyl acetate (EtOAc) among others.

B - Method Development - Screening

When developing methods, we would recommend a screening approach.

1. The conditions described in Table 1 should be used as a Primary Screening.
2. If the compound or compound series are not soluble in any of these mobile phases, we recommend progressing directly to the Secondary Screening (Table 2).

Table 1. Immobilized Primary Screening Solvents

Primary Solvent Mixtures	Alkane❶/2-PrOH	Alkane❶/EtOH	Alkane❶/MtBE/EtOH❷	Alkane❶/THF❸	Alkane/DCM❹/EtOH
Typical Starting Conditions	80:20	80:20	0:98:2	70:30	50:50:2
Advised Optimization Range	99:1 to 50:50	99:1 to 50:50	80:20:0 to 0:40:60	95:5 to 0:100	85:15:0 to 0:80:20

❶ Alkane = n-Hexane, iso-Hexane or n-Heptane. Some small selectivity differences may sometimes be found.
❷ In absence of alkane, methanol is more efficient than ethanol when combined with MtBE.
❸ In the case of no environmental restrictions, **use of DCM is preferred to THF** in terms of better enantioselectivity that the former may induce.
❹ For excessively retained samples, addition of ethanol up to 20% in pure DCM would be helpful.

If a suitable chiral separation is not found using the Immobilized Primary Screening strategy, we recommend an Immobilized Secondary Screening to be applied using the following conditions:

Table 2. Immobilized Secondary Screening Solvents

Secondary Solvent Mixtures	EtOAc❶/Alkane❷	ACN❸/Alcohol❹
Typical Starting Conditions	50:50	100:0
Advised Optimization Range	20:80 to 100:0	100:0 to 0:100

❶ Alcohols (❹) or THF can be added into EtOAc to enhance the eluting strength for strongly retained compounds.
❷ Alkane: n-Hexane, iso-Hexane or n-Heptane. Some small selectivity differences may sometimes be found.
❸ Transfers between alkane mixtures and ACN are preferably made with a transition in alcohol in order to avoid miscibility issues.
❹ Alcohol: MeOH, EtOH and 2-PrOH.

Note: All solvent proportions indicated in this manual are by volume.

C – General Comments

⇨ Only immobilized CHIRALPAK® IA-10, IB N-10, IC-10, ID-10, IE-10, IF-10, IG-10, IH-10, IJ-10, IK-10, and IM-10 are suitable for the Secondary Screening

⇨ Additional solvent combinations such as $CHCl_3$/Alkane, 1,4-Dioxane/Alkane, Toluene/Alkane, or Acetone/Alkane can also be investigated with CHIRALPAK® IA-10, IB N-10, IC-10, ID-10, IE-10, IF-10, IG-10, IH-10, IJ-10, IK-10, and IM-10 columns.

⇨ The typical starting conditions represent the mobile phases of upper middle eluting strength. Under such conditions, most of the analytes can be eluted within a reasonable time range with a good probability of full resolution of the enantiomers.

⇨ Toluene, MtBE and chlorinated solvents can be used in their pure form as the mobile phase.

⇨ For fast eluting solvents, such as THF, we recommend adding alkane in order to modulate the retention.

⇨ Detection with a regular UV detector may become difficult depending on a combination of sample and mobile phase (e.g. EtOAc, high percentages of DCM). In these cases, an alternative detector, such as an RI detector or ELSD (Evaporative Light Scattering Detector), may be more effective than the UV detector.

D – Additives

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

❶ It has been found that certain amines, such as EDA and AE, induce much better behavior for certain basic compounds than the most commonly used DEA.
❷ For preparative purposes, it is recommended to use DEA or TEA as additives, due to their easy removal from the products by standard evaporation and drying systems.

Basic Samples require Basic additives❶❷	Acidic Samples require Acidic additives
Diethylamine (DEA) 2-Aminoethanol (AE) Ethylenediamine (EDA) Butyl amine (BA)	Trifluoroacetic acid (TFA) Acetic acid Formic acid
< 0.5% Typically 0.1%	< 0.5% Typically 0.1%

⇨ *STRONGLY BASIC solvent additives or sample solutions MUST BE AVOIDED, because they are likely to damage the silica gel used in this column*

☞ *The addition of a low percentage of an alcohol (e.g. 2% EtOH or MeOH) in the mobile phase may be helpful to ensure the miscibility of EDA and AE with the low polarity mobile phases.*

Column Care / Maintenance

- ❑ The use of a guard column is highly recommended for maximum column life.
- ❑ Samples should preferably be dissolved in the mobile phase.
- ❑ The mobile phase and the sample solution should be filtered through a membrane filter of approximately 0.5µm porosity to ensure that there is no precipitate before using.

Following extensive use of the column in multiple solvents, there may be a change in separation reproducibility. In order to ensure consistent performance, a regeneration method may be implemented to eliminate any change in chiral recognition due to the history of the column (mobile phases, additives…).

For detailed Regeneration Procedures, please click here

Column Storage

- ❑ For column storage, remove the acidic or basic additives by flushing the column with several column volumes of the same mobile phase, but without the additive.
- ❑ Columns can be stored with ends capped in the additive-free mobile phase, or the shipping solvent, at room temperature.

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: cte@cte.daicel.com 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 CS 80140
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

India
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