

Care and Use Instructions

CHIRAL ART Packing Material

Coated type for normal phase mode

1. Introduction

Thank you for purchasing CHIRAL ART packing material. CHIRAL ART (coated type) is designed for separating optical isomers. The chiral selector, a polysaccharide derivative, is coated onto silica gel.

CHIRAL ART packing materials, which are manufactured under highly controlled conditions, must pass a series of strict tests before being accepted for shipment (Please refer to the inspection report). To ensure optimal performance and stability of the packing material, please follow these instructions before use.

2. Specifications

	CHIRAL ART Amylose-C CHIRAL ART Amylose-C Neo	CHIRAL ART Cellulose-C
Particle size (μm)	10, 20	
Chiral selector	Amylose tris(3,5-dimethylphenylcarbamate)	Cellulose tris(3,5-dimethylphenylcarbamate)
Type	Coated type	
Bulk density (g/cm ³)	ca. 0.53	

3. Packing instructions [for dynamic axial compression (DAC) columns]

3-1 Amount of packing material required

Calculate the amount of packing material by using the column volume and the bulk density (see section 2).

3-2 Preparation of packing slurry and packing

2-Propanol is recommended as the slurry and packing solvent. Add the solvent to obtain a slurry at a concentration of 30–40%*, and transfer the slurry to a DAC column. Packing pressure of 6–8 MPa is recommended. Take care not to exceed the maximum usable pressure of DAC column. Conditioning of CHIRAL ART Amylose-C or Amylose-C Neo with ethanol after column packing is recommended.

*slurry concentration (% w/v) = amount of packing material (kg) / total volume of slurry (L) x 100

3-3 Testing the packed column (Evaluation of column performance)

Once packing is completed, check the theoretical plate number (N) and peak shape. In the case where appropriate theoretical plate number (N) or asymmetry factor (As) is not obtained, please optimize the packing condition.

Example conditions of column performance evaluation

Eluent : *n*-hexane/2-propanol (90/10, v/v)
 Linear velocity : 180 cm/hr
 Detection : UV at 230 nm
 Sample : 1,3,5-Tri-*tert*-butylbenzene (0.5 mg/mL) [void volume marker]
 trans-Stilbene oxide (0.1 mg/mL) [peak 1, peak 2]
 Sample solvent : Eluent
 Injection : 30 x cross-sectional areas of columns (cm²) μL
 Evaluation : Theoretical plate number (N) of *trans*-Stilbene oxide (peak 2)

* Values might be influenced by column or LC system

Expected theoretical plate number (N/m) *

	10 μm	20 μm
	16,000	6,000

4. Precautions for use

CHIRAL ART (coated type) is coated with the polysaccharide derivative. Therefore trace quantities of a solvent that might potentially dissolve the polysaccharide derivative (e.g. THF, acetone, ethyl acetate, chloroform, dichloromethane, DMSO, DMF, etc.) should be eliminated. These solvents must be avoided for a mobile phase or sample solvent.

(THF: Tetrahydrofuran, DMSO: Dimethylsulfoxide, DMF: Dimethylformamide)

- Suitable solvents as mobile phase and composition (volume/volume) are shown in the table below.

alkane/2-propanol ¹	alkane/ethanol ¹	methanol/ethanol	methanol/acetonitrile ²
100/0 – 0/100	100/0 – 0/100	100/0 – 0/100	100/0 – 85/15 15/85 – 0/100

¹: Alkane commonly used is *n*-hexane or *n*-heptane. Alcohols other than the above (methanol, 1-propanol, 1-butanol, 2-butanol, etc.) can be used; however, methanol and alkane have low miscibility. When adding more than 5% methanol, the same amount of ethanol also needs to be added.

²: 100% methanol and 100% acetonitrile can be used; however, when switching between mobile phase containing methanol and acetonitrile, flush the column with ethanol (in the case of CHIRAL ART Amylose-C/Amylose-C Neo) or 2-propanol (in the case of CHIRAL ART Cellulose-C) as transition solvent.

- Make sure of miscibility between the organic solvents. When switching the mobile phase from alkane/alcohol mobile phase to polar organic solvents (methanol, acetonitrile, etc.), flush the column with ethanol (in the case of CHIRAL ART Amylose-C/Amylose-C Neo) or 2-propanol (in the case of CHIRAL ART Cellulose-C) beforehand. After that, sufficiently equilibrate the column with the mobile phase. In addition, the packing material used with polar organic solvents (such as methanol/ethanol, methanol/acetonitrile) as a mobile phase should be dedicated to this specific application.
- When a target compound is ionic, it may be necessary to use an appropriate mobile phase modifier in order to improve peak shape or separation reproducibility. High concentrations of some modifiers may shorten the life time of the packing material. When using modifiers the following guidelines are recommended:
 - Basic compounds : 0.1% (upper limit 0.5%) diethylamine (DEA), butylamine, ethanolamine, etc.
 - Acidic compounds : 0.1% (upper limit 0.5%) trifluoroacetic acid (TFA), acetic acid, formic acid, etc.
- To protect a column/packing material, a sample containing a lot of impurities should be filtered out before injection. Also it is recommended that a guard column should be used.
- Operating pressure should not exceed the packing pressure.

5. Column cleaning and storage

Cleaning procedure

- Flush the column with a solution containing a higher ratio of the stronger solvent (for example, for alkane/alcohol mobile phase, concentration of alcohol should be increased) for washing out the compounds that have a great capacity for retention in the column. When further cleaning is required, flushing with ethanol (in the case of CHIRAL ART Amylose-C/Amylose-C Neo) or 2-propanol (in the case of CHIRAL ART Cellulose-C) is effective.
- When a mobile phase containing acid or amine is used, replace the mobile phase with solvent containing neither acid nor amine (at the same ratio as the mobile phase). Then flush the column in accordance with the method described above.

Column storage

- Clean the column in accordance with the method described above, and replace the mobile phase with ethanol (in the case of CHIRAL ART Amylose-C/Amylose-C Neo) or 2-propanol (in the case of CHIRAL ART Cellulose-C). Keep away from heat and moisture. Put airtight stoppers on the both column end to prevent the packed bed from drying out.
- Avoid storing the column with a mobile phase containing modifier even if it is only for a short period.

6. Packing material storage

Unused packing material : Store the packing material in the original container, and keep away from heat and moisture.

Used packing material : At first, clean the packing material in accordance with the method described in section 5.

[Dry storage]

Flush the column with ethanol (in the case of CHIRAL ART Amylose-C/Amylose-C Neo) or 2-propanol (in the case of CHIRAL ART Cellulose-C), and then remove the packing material from the column. After drying the unpacked material at 60 °C or lower, transfer it to an appropriate container. Keep away from heat and moisture.

[Slurry storage]

Flush the column with ethanol (in the case of CHIRAL ART Amylose-C/Amylose-C Neo) or 2-propanol (in the case of CHIRAL ART Cellulose-C), and then remove the packing material from the column. Transfer the unpacked material to an appropriate container and store it in the same solvent. Please ensure that the container is tightly sealed.

NOTE - We do not guarantee the used packing material, and cannot accept any return of it.