Operation Manual
Shodex™ ORpak™ CDBS-453

(Please read this manual carefully before using the column to keep its good performance and life.)

1. Introduction

The packed column of Shodex ORpak CDBS-453 is designed to be used for the separation of chiral compounds by HPLC. The column is best suited for the separation of hydrophobic optical isomers which have a cyclic structure.

2. Specifications

Column type	Column size (ID x length)	Ligand	Maximum pressure	Maximum flow rate
CDBS-453	4.6mm x 150mm	β - cyclodextrin	20.0 MPa	1.2 ml/min.
CD-G	4.6mm x 10mm	(Guard column)		

Endfitting: Internally-threaded type, No. 10 32 UNF.

In-column solvent: CH_3CN/(1%CH_3COOH + 0.2MNaCl) = 30/70

Column material: SUS316.

Packing material: Porous Silica gel bonded with β - cyclodextrin.

Usable temperature: 5 to 60°C.

Usable pH range: pH 2 to pH 7.

3. Eluent

The separation performance of CDBS-453 will be largely affected by the eluent conditions.

A buffer, an organic solvent or a mixture of buffer and organic solvent can be used as an eluent.

Commonly, a mixture is used as an eluent.

The following are buffers and organic solvents usually used.

1) Buffer

(1)Acetic buffer

(2)Phosphate buffer

Caution! ① The buffer pH range should be between 2 and 7.

② When a salt is added, the salt concentration should be lower than 1.0 M.

③ When acetic acid is added, the acid concentration should be lower than 6% (w/v).

2) Organic solvent

(1) Acetonitrile

(2) Methanol

(3) Isopropylalcohol

Caution! When an organic solvent is added, the eluent viscosity will become higher. Consequently, the column pressure also will become higher. Therefore, be careful not to exceed the maximum pressure in the specifications.

4. Preparation of eluent

1) Remove extraneous matter by passing the eluent through a 0.45um filter.

Use of the disposable filter unit is recommended.

2) Thoroughly degas the eluent, by subjecting it and ultrasonic vibration and simultaneous heating or pressure reduction with an aspirator.

Use of solvent degassing devices is recommended.

5. Installation and start-up

1) Prior to connection of the column to the liquid chromatograph, replace the solvent in the chromatograph with the solvent that is to be used as the eluent.

When a solvent containing salt is remaining in the liquid chromatograph, wash all flow line with purified water before replacement with the eluent.

2) After replacing the solvent in the chromatograph, set the flow rate at half of the flow rate to be used.

3) Connect the column to the chromatograph as that the arrow mark on the column will face downstream. Do not let air get into the column while connecting the column to the chromatograph.

Then, start pumping.

Caution! It is recommended to start from a slow flow rate. And, after confirming that the pressure does not exceed the maximum flow rate, the flow rate can be raised at the flow rate to be used.

Note: In case of the separation of optical isomers, it is recommended to use lower temperature for better separation.

6. Pre-treatment of sample

1) The sample should be dissolved in the eluent to be used.

2) Remove extraneous matter by passing the eluent through a 0.45um filter.

Use of a disposable filter unit is recommended.

7. Guard column

Install a guard column immediately upstream of the main column to protect it from contamination by the sample.

The guard column is intended to maintain the column performance as designed for a long period and not to improve its resolving power.

8. Safekeeping

1) When the column is heated, after completing analysis, keep pumping the eluent at a flow rate of 0.5 ml/min. until the column is cooled down to room temperature.

2) Stop the pump and leave the column in the chromatograph, if it is to be reused on the following day.

3) In case of 3 or more days of suspension of chromatograph in which a solvent containing salt was used as eluent, replace the eluent with purified water, setting the flow rate at 0.3 ml/min.

4) In the case of its suspension over a long period of time, take the same action as in 3) above and dismount the column from the chromatograph. Then, blank off both ends of the column.

5) Package it as delivered from the manufacturer.

6) Store it in a room that has little temperature fluctuation.

9. Trouble shooting

Table 1 below gives troubles likely to occur during use of the column and the corrective actions to take. It is not guaranteed that the corrective actions as given in the table always solve the trouble.

Therefore, after taking the actions, check the column resolution. Please note that removal of the endfittings will allow the air or other extraneous substances to enter the column, probably to deteriorate the column performance.

Trouble	Cause	Corrective action
Column pressure increase.	Plugged endfitting.	Reverse the column on chromatograph and pass the eluent through it for one hour.
	Inclusion of extraneous substances in packing.	Irreparable.
Rapid deterioration of resolution.	Void induced in the upstream end of column.	Irreparable.
	Liquid flow disturbance caused by extraneous matter clogging endfitting.	Reverse the column on chromatograph and pass the eluent through it for one hour.
No elution of sample.	Sample adsorbed.	Change the separation conditions.
	Malfunctioning detector.	Check the detector.