

Shimadzu Packed Column for HPLC

Shim-pack XR-ODSⅢ

Instruction Manual

■ Introduction

Shim-pack XR-ODSⅢ is a high performance reversed-phase column for HPLC designed for rapid separation. The packing material is composed of 2.2 μm or 1.6 μm of totally porous, high purity spherical silica particles. The surfaces of the silica particles are chemically bonded with octadecylsilyl (ODS) groups and thoroughly endcapped.

■ Specifications

● Packing

Item	Contents	
Silica particles	Spherical, porous, high purity silica particles	
Particle size	2.2 μm	1.6 μm
Pore size	8.0 nm	7.5 nm
surface modification	Octadecylsilyl groups (Mono-functional)	
Other modification	Endcapping	
Carbon loading	about 20 %	about 21 %

● Column

Item	Contents
Type	Stainless steel packed column
Storage solvent	Please see the Column performance report.
Maximum operating pressure	100 MPa *1
pH range	2 ~ 7.5
Maximum operating temperature	80 °C (When mixtures of water or acidic aqueous solution (pH 3 or greater) and acetonitrile are used.)

*1. Refer to the paragraph of "■ Column Handling Precautions".

■ Lineup

Size	P/N	Particles
2.0 mm i.d. \times 50 mm	228-59922-91	1.6 μm
2.0 mm i.d. \times 75 mm	228-59922-92	
2.0 mm i.d. \times 150 mm	228-59910-91	2.2 μm
2.0 mm i.d. \times 200 mm	228-59910-92	

■ Certificate of Compliance

This column comes with a quality assurance certificate that refers to the physical properties, chromatographic and column performance of Shim-pack XR-ODSⅢ. These items are listed in the following tables.

● Physical Properties

Item	Contents
Particle Size	The particle size (μm) indicated is the median value of the particle size distribution.
Pore Size	The average pore size (nm) is determined by the nitrogen adsorption method.
Pore Volume	The pore volume (mL/g) is determined by the nitrogen adsorption method.
Specific Surface Area	The specific surface area (m^2/g) is determined by the nitrogen adsorption method.
Trace Metal Contents	The total and individual trace metal content (ppm) of the silica is determined for six different metals.
Carbon Loading	The carbon loading (%) of the octadecyl and methyl groups in the packing determined by CHN measurement.
C18 Surface Coverage	The even distribution of octadecyl groups per unit of packing surface area ($\mu\text{mol}/\text{m}^2$) is certificated.

● Chromatographic Performance

Item	Contents
Hydrophobic Interaction	The relative retention (α) of amylbenzene and butylbenzene is calculated to determine how hydrophobic the stationary is.
Basic Compound	The tailing factor (symmetry factor, T_f) and relative retention (α) of <i>N</i> -acetylprocainamide against phenol are examined to determine the elution characteristics of basic compounds.
Acidic Compound	The tailing factor (symmetry factor, T_f) and relative retention (α) of salicylic acid against phenol are examined to determine the elution characteristics of acidic compounds.
Chelating Compound	The interaction of chelating compounds and the bonded phase is measured by examining the theoretical plate number (N) and relative retention (α) for 8-quinolinol against toluene.

● Column Performance

項目	内容
Retention Time	The retention time of naphthalene (t_R) is used to determine whether the column meets hydrophobic level requirements.
Plate Number	The number of theoretical plates (N) is calculated for naphthalene to ensure that the column is packed properly. The following formula is used to calculate the number. $N = 5.54 \times (t_R / W_{1/2})^2$ t_R : retention time $W_{1/2}$: peak width at 1/2 height
Tailing Factor	The tailing factor (symmetry factor, T_f) of naphthalene is used to determine that the column is uniformly packed. The following formula is used to calculate the factor. $T_f = W_{0.05} / 2f$ $W_{0.05}$: peak width at 5 % height f : width from peak upslope to peak apex at 5 % height
Pressure	The column head pressure (MPa) is measured to ensure that the column is packed properly.

■ Column Installation

- The flow direction of the column is shown on the column tag. When installing the column, ensure that the flow direction arrow matches the mobile phase flow direction.
- The column is connected with UHPLC Fitting. Ensure that the fittings are connected properly to avoid creating dead volume between the tubing and the column interface.

Item Name	P/N	Comments
UHPLC Fitting	228-53204	1/pkg

- Tubing connections to the column must be stainless steel, 0.1 - 0.2 mm *i.d.* Use the shortest possible tubing connection from the injector to the column to minimize peak broadening.

NOTE:

The stain or air in the flow line may deteriorate the column. Before connecting the column, be sure to flow the mobile phase to flush the flow line.

■ Mobile Phase Solvent

- Generally, the mobile phase consists of a mixture of water and acetonitrile.
- When analyzing ionic substances, the separation characteristics of the compounds are kept uniform by the addition of acids, such as acetic acid or formic acid, or pH modifiers, such as phosphate buffer. However, the pH must be carefully monitored to ensure that it is within an acceptable range for stationary phase stability.

NOTE:

Filter the mobile phase through a 0.22 µm membrane filter, or an equivalent, before use. Suspended particles will lead to column clogging, which will increase the system pressure. Use a fresh mobile phase every analysis, and do not add mobile phase to the bottle.

■ Column Handling Precautions

- Do not overtighten the male nuts to the column during installation. This may damage the fittings.
- Observe the pressure and temperature limits given in "■ Specifications". The steep pressure change over the column may cause deterioration.
- When you develop a new method by this column, to prevent pressure over issue that occurs when the column while changed, you keep the maximum operating pressure below the value estimated by the following equation. In addition, to keep the initial performance, the maximum operating pressure is as well as possible low.

$$P = 100 \times \frac{P_C}{P_L} \quad \text{Unit: MPa}$$

1 00: Maximum operating pressure

P_L : Maximum pressure of criteria in the column performance report.

P_C : Pressure of result in the column performance report.

P : Maximum operating pressure in a new method.

- Set the flow rate not to exceed the maximum operating pressure. The following table shows a rough guide for the

optimum and maximum flow rate range. Even the pressure does not exceed the maximum pressure, do not set the flow rate over the maximum flow rate.

Particle size	Optimum Flow Rate	Maximum Flow Rate
2.2 µm	0.4 ~ 0.5 mL/min	1.2 mL/min
1.6 µm	0.4 ~ 0.8 mL/min	1.2 mL/min

- Adjust the pH of mobile phase within the range described in "■ Specifications". Optimum lifetime is obtained at pH 2.5 - pH 7.0 and at 40 °C or less when a buffer is used.
- To remove the column from the system, be sure to confirm the temperature of the column becomes the room temperature and the pressure of the column becomes zero.
- Do not shock the column by banging it or dropping it.
- The solute retention can be also controlled by the addition of an ion-pair reagent, such as a tetrabutylammonium salt or 1- octanesulfonate salt. Determine conditions such that the solute retention remains constant, even if the ion-pair concentration fluctuates.

NOTE:

Filter the sample solutions through a 0.22 µm membrane filter, or an equivalent, before use. Suspended particles will lead to column clogging, which will increase the system pressure.

■ Flushing the Column

To remove the lipid-soluble substances or ionic substances from the column which may cause unstable retention time or bad shape of the peak, connect the column in reverse flow direction and flush the column as followings. To remove salt (phosphate and so on) insoluble to the organic solvent from the column, flush the salt by pure water at the rate mentioned below. After the flushing, reconnect the column in the normal flow direction. Replace with mobile phase not to remain the flushing liquid. The column cannot be regenerated if it is heavily contaminated.

<Flushing Procedure>

Firstly, flush with 0.1% TFA in acetonitrile or methanol for 1 hour and, secondly, flush with acetonitrile or methanol for 10 min at the optimum flow rate Shown in "■ Column Handling Precautions".

■ Column Storage

When removing the column from the system, cap both ends of the column so that the solvent cannot evaporate. For long-term storage, first flush the column, replace the mobile phase with water/acetonitrile (1/1, v/v), then cap both ends of the column before storage. Remember to flush with water first if buffers were used as the mobile phase.

■ Technical Support

It is the customer's responsibility to develop and validate analytical conditions for a particular application. However, Shimadzu offers technical support by e-mail and phone for customers who need help. Write specific questions to analytic@group.shimadzu.co.jp or call your local representative.

※ The contents of this instruction sheet are subject to change without notice.