

Shimadzu Packed Column for HPLC

Shim-pack

XR-ODS / C8 / Phenyl

Instruction Manual

■ Introduction

Shim-pack XR-ODS / -C8 / -Phenyl are a high performance reversed-phase column for HPLC designed for rapid separation. The packing material is composed of 2.2 μm of totally porous, high purity spherical silica particles. The surfaces of the silica particles are chemically bonded with functional groups and thoroughly endcapped.

■ Specifications

Item	Contents
Silica particles	Spherical, porous, high purity silica particles
Particle size	2.2 μm
Pore size	12 nm
Surface modification	XR-ODS: Octadecylsilyl groups (Mono-functional) XR-C8: Octyl groups (Mono-functional) XR-Phenyl: Phenylpropyl groups (Mono-functional)
Other modification	Endcapping
Carbon contents	XR-ODS: about 18.4% XR-C8: about 10.5% XR-Phenyl: about 11.0%
Type	Stainless steel packed column
Storage solvent	Please see the Column performance report.
pH range	2 - 7.5 ^{*1}
Operating temperature	80 °C ^{*1} When mixtures of water or acidic aqueous solution (pH 3 or greater) and acetonitrile are used.

*1. Refer to "■ Column Handling Precautions".

■ Certificate of Compliance

These columns come with a quality assurance certificate that refers to the physical properties, chromatographic and column performance of Shim-pack XR-ODS / -C8 / Phenyl. These items are Shown in "■ Description Items of the Certificate".

■ Description Items of the Certificate

● Physical Properties

Item	Contents
Particle Size	The particle size (μm) indicated is that of the halfway point 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.

Item	Contents
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.

● 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.

■ Lineup

Size	XR-ODS	XR-C8	XR-Phenyl
2.0 mm i.d. × 30 mm	228-41605-91	228-59901-91	228-59903-91
2.0 mm i.d. × 50 mm	228-41605-92	228-59901-92	228-59903-92
2.0 mm i.d. × 75 mm	228-41605-93	228-59901-93	228-59903-93
2.0 mm i.d. × 100 mm	228-41605-94	228-59901-94	228-59903-94
3.0 mm i.d. × 30 mm	228-41606-91	228-59902-91	228-59904-91
3.0 mm i.d. × 50 mm	228-41606-92	228-59902-92	228-59904-92
3.0 mm i.d. × 75 mm	228-41606-93	228-59902-93	228-59904-93
3.0 mm i.d. × 100 mm	228-41606-94	228-59902-94	228-59904-94
4.6 mm i.d. × 30 mm	228-41607-91	-	-
4.6 mm i.d. × 50 mm	228-41607-92	-	-
4.6 mm i.d. × 75 mm	228-41607-93	-	-
4.6 mm i.d. × 100 mm	228-41607-94	-	-

■ 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 SUS male nuts and PEEK ferrules. Ensure that the fittings are connected properly to avoid creating dead volume between the tubing and the column interface. The product name and the part numbers of the SUS male nut and the PEEK ferrule are as follows.

Item Name	P/N	Comment
Male Nut SUS	228-45717-01	2/pkg
Double Ended Ferrule PEEK	228-45717-02	10/pkg

- Tubing connections to the column must be stainless steel, 0.1 - 0.2 mm *i.d.* and 1.6 mm *o.d.* Use the shortest possible tubing connection from the injector to the column to minimize peak broadening.

● **NOTE:** The presence of air in the flow line may damage the column. Before connecting the column, be sure the flow line is completely filled with mobile phase.

■ 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.
- 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.

■ Column Handling Precautions

- Do not overtighten the male nuts to the column during installation. This may damage the fittings.
- To stable use for long time, please refer to the conditions described in the Specifications. As a rough guide about 35 MPa of the column pressure, and do not use extreme over it (pay attention to the hardware). The steep pressure change over the column may cause deterioration. Set the flow rate not to exceed the maximum operating pressure.
- 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.

$$P = 35 \times \frac{P_C}{P_L} \quad \text{Unit: MPa}$$

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.

- The optimum flow rate range is as follows. Even though the pressure is less than the maximum pressure, the very high flow rate has the possibility of making the column deterioration.

Column <i>i.d.</i>	Optimum Flow Rate
2.0 mm	0.4 - 0.5 mL/min
3.0 mm	0.9 - 1.2 mL/min
4.6 mm	2.0 - 2.5 mL/min

- Adjust the pH of mobile phase within the range described in the Specifications. Optimum lifetime is obtained at pH 2.5 - pH 7.0 and at 40 °C or less when a buffer is used.
- Filter the mobile phase and 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.
- To remove the column from the system, wait until the column temperature equals the room temperature and the pressure of the column becomes zero.
- Do not shock the column by banging it or dropping it.

■ Flushing the Column

To remove any lipid-soluble substances or ionic substances from the column which may cause unstable retention time or bad peak shapes, connect the column in reverse flow direction and flush the column as follows. 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 flushing, reconnect the column in the normal flow direction. Replace with mobile phase to remove the flushing liquid.

<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 half of the optimum flow rate Shown in "■ Column Handling Precautions".

● **NOTE:** The column cannot be regenerated if it is heavily contaminated.

■ 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 described in the Column performance report, 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.