

High Performance Packed Column for HPLC

CoreFocus

Shim-pack™ GIS Series

INSTRUCTION MANUAL

■ Introduction

To ensure optimum performance of your Shim-pack GIS series column, and to maximize lifetime and maintain stability of the column, please read the following instructions carefully before use.

■ Operating Precautions

- Check if anything is missing or damaged. If there are any signs of damage, notify your local Shimadzu representative at once.
- Each Shim-pack GIS series column is individually tested and a Column Performance Report is attached.
- The report includes the column lot number, serial number, test conditions, and chromatogram of several test mix components. Please keep the report for future reference.

■ Column Performance

Shim-pack GIS series packing material is subjected to a rigorous array of QC tests, with special emphasis on reagent purity, raw material traceability, consistency in raw materials, and quality of final products.

A detailed analysis of all of the physical and chemical properties of these columns, combined with tests for chromatographic selectivity and column packing material efficiency, ensure that each lot of column is identical to all previous lots and column-to-column reproducibility is of the highest order.

The bonded phase, pore size, surface area, carbon content and end-capping treatment of Shim-pack GIS series are as follows.

Column	Bonded Phase	Pore size (nm)	Surface area (m ² /g)	Carbon content (%)	End-capping
Shim-pack GIS C18	Octadecylsilyl	10	450	15	Yes
Shim-pack GIS C18-P	Octadecylsilyl			29	No
Shim-pack GIS RP-Shield	Octadecylsilyl + polar functional groups			9	No
Shim-pack GIS CN	Cyanopropyl			14	No
Shim-pack GIS SIL	-			-	No
Shim-pack GIS HILIC	Cyanopropyl			20	No

Shim-pack GIS series columns are shipped with the solvent used for the final QC test of the column, as detailed in the Column Performance Report delivered with the column.

Type	Columns	Shipping Solvent
Reversed-phase	Shim-pack GIS C18* ¹ Shim-pack GIS C18-P* ¹ Shim-pack GIS RP-Shield* ²	Water : Acetonitrile = 35/65(v/v)
Normal-phase	Shim-pack GIS CN	n-Hexane : Ethanol = 98/2(v/v)
	Shim-pack GIS SIL	n-Hexane : Ethanol = 95/5(v/v)
HILIC-phase	Shim-pack GIS HILIC* ³	Acetonitrile = 100

*1: Shipping solvent for the column under the following conditions is Water : Acetonitrile = 50/50(v/v).

- Inner diameter 4.0 mm or less & all particle size & shorter than 50 mm.
- Inner diameter 4.6mm & particle size 5μm & shorter than 50mm.
- All inner diameter & particle size 2μm & 50mm or less (GIS C18)

*2: Shipping solvent for the column under the following conditions is Water : Acetonitrile = 50/50(v/v).

- Inner diameter 4.0 mm or less & all particle size & shorter than 50 mm.

*3: Shipping solvent for the column under the following conditions is Water : Acetonitrile = 5/95(v/v).

- All inner diameter & all particle size & 150 mm or longer.

However, the shipping solvent for the column under the following conditions is Water: Acetonitrile = 2/98(v/v).

- Inner diameter 4.6mm & particle diameter 3μm & 75mm to 100mm length.
- Inner diameter 4.0mm or less & particle diameter 3μm & 100mm length.

When switching from the current solvent to one with significantly different polarity, flush the column with at least 10 times the column volume of a solvent with intermediate polarity such as 2-propanol or dioxane (e.g. 25 mL for a 150 mm x 4.6 mm I.D. column.) Then switch the solvent to the one you want to use. Purge the column with a mutually miscible solvent such as Isopropyl alcohol at a reduced flow rate (approximately 50% lower than the normal flow rate).

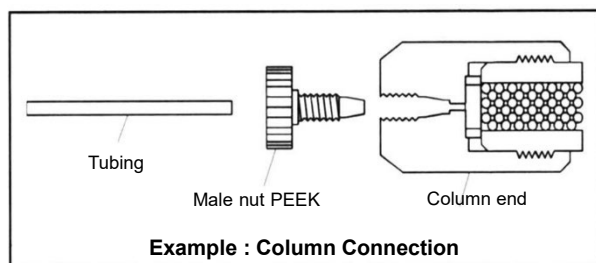
NOTE

When using the solvent for reversed-phase (e.g. water) with Shim-pack GIS CN or SIL that has the history of usage as a normal-phase column, start to use the column after enough solvent exchange. It is time consuming to flush the buffer or water in the pores of the packing and exchange solvent after using reversed-phase eluent. If a subsequent analysis is performed in the normal phase mode, and if the flush has not been thoroughly completed, the elution order and retention time may vary significantly. Therefore, this type of column should be used solely for one type of analysis.

■ Column Installation

- The flow direction of the column is shown on the column (→). When installing the column, ensure that the flow direction matches the mobile phase flow direction.
- Use PEEK tubing (SUS tubing for UHPLC) with an inner diameter of 0.25 - 0.3 mm (0.1 - 0.2 mm for UHPLC) and an outer diameter of 1.6 mm. However, never use the PEEK tubing when you use Tetrahydrofuran (THF), chloroform, hexafluoroisopropanol (HFIP), concentrated sulfuric acid, concentrated nitric acid, dichloroacetic acid, acetone, dichloromethane or dimethyl sulfoxide (DMSO) as the solvent.
- Use the shortest possible tubing connection from the injector to the column to minimize peak broadening.
- The column is connected with male nuts. Ensure that the fittings are connected properly to avoid creating dead volume between the tubing and the column interface. Male nuts can be ordered by referring to the part number below.

Item name	P/N	Remarks	Pressure
Male nut, PEEK	228-18565-84	5 pcs	20 MPa
Male nut fitting kit	228-45717-01	2 pcs	35 MPa
Male nut 1.6 MN	228-16001	1 pc.	130 MPa
Ferrule 1.6 F	228-16000-10	1 pc.	130 MPa
UHPLC Fitting 2 S	228-56867-41	1 pc.	130 MPa



NOTE

Particulates or air in the system flow line may deteriorate the column. Before connecting the column, be sure to filter all solvents and flush the solvent used as the mobile phase to flush the flow line.

- If peaks are tailing more on the early eluting compounds than later eluting compounds, there is a possibility that there is a dead volume. In such case, check that all column connections are properly connected.
- Do not overtighten column connections. This may damage the fittings or column ports and result in leaks.
- Make sure to use appropriate internal diameter and length size of tubing at the injector and detector, especially when using semi-micro size columns, to avoid system dead volumes.

■ Sample

Samples should be dissolved in an eluent or solvent weaker than the mobile phase, which helps avoid sample precipitation at column inlet/head and inconsistent retention values. In order to prevent the precipitation of salts contained in sample or solvent, check the miscibility of these with mobile phase before injection.

■ Clogging of column

The most common cause of the increase of column back pressure or split peaks is blockage of the inlet filter by sample particulates, particles created by aging pump seals, or large quantities of lipophilic compounds adsorbing to the head of the column.

- It is recommended to filter the mobile phase with a membrane filter of 0.45 μm or less before flushing the flow line for analysis.
- It is recommended to install "Ghost Trap DS"* between the pump and injector. It can efficiently remove particulates or contaminants in the mobile phase. Order guide for "Ghost Trap DS" is provided below.

Item	P/N	Description	Internal volume	Dimensions	Pressure
Ghost Trap DS	228-59921-92	Two cartridges and one holder	7.6 mmIDx 30 mm	Approx. 700 μL	35 MPa
	228-59921-94	Two cartridges and one holder	4.0 mmIDx 20 mm	Approx. 150 μL	
Ghost Trap DS-HP	228-59931-91	Packed type	30 mmL. x 2.1 mmI.D.	Approx. 60 μL	100 MPa

* Ghost Trap DS* may induce bleed noise when using a mass spectrometer as a detector. And ion pair reagents may be retained in the product. Thereby it may influence on retention times and peak shapes.

- Filtrate the sample using a syringe filter (0.2 μm - 0.45 μm) before injecting to the column.
- It is recommended to install "Guard Column" or "Guard Column for UHPLC" to prevent column clogging problems.

Baseline drift and noise can be caused by defective pumping due to air bubbles in eluent or decrease of light intensity when using a UV detector. Note that bubbles can form in the detector flow cell if the eluent is not degassed properly before introduction into the column. If the baseline drift and noise occur after flushing the column, check if the LC system and analytical condition are optimized.

■ Column Handling Precautions

- Do not drop or bump the columns, to avoid a deterioration of the column performance.
- Shim-pack GIS series columns provide optimum lifetime when used with eluents within the pH range of 2.0 to 7.5. Higher pH leads dissolution of silica gel and lower pH causes hydrolysis of bonded phase. Hence the best lifetime is obtained at intermediate pH conditions.
- The highest operating temperature of the column is 60°C (when used within the above operating pH range). However, operating at temperatures above the normal temperature between 20°C and 40°C may hasten degradation of the column depending on the mobile phase conditions.
- To maximize column life, use the columns within the pressure shown in the following table.

Description	Maximum Operating Pressure
Above 3 μm Columns	20 MPa
HP Series 3 μm Columns	50 MPa
C18 2 μm Columns with lengths below 50mm	50 MPa
C18 2 μm Columns with lengths above 75mm	80 MPa

- The column should be disconnected from the system only after the pressure shows "0 MPa."

- Avoid excessive pressure fluctuation. This increases pressure rapidly at the column inlet, which may cause premature column deterioration. When using a preparative column, a bypass from the injector is recommended.
- If there is a drift in the retention time of the compound, a baseline drift, or noise at the beginning of the analysis, the column equilibration may be inadequate. We recommend to flush the column at least five times of the column volume with the mobile phase used for analysis as a guide until the improvement is seen. If there is no improvement, check if the LC system and analytical condition are optimized.

■ Flow rate of Prep column

Please read the following instructions before using prep columns.

- The optimum performance will be gained in the range of flow rate listed below. Even so, since the column may have considerable pressure, we recommend you to use the column at pressure less than 20 MPa. However, when using a 100 mm I.D. column*, use the pressures within 10 MPa.

* 100 mm I.D. column is a special order item. When you want to purchase a 100 mm I.D. column, please contact your local Shimadzu representative.

- The flow rate will be larger than that of analytical columns. Use a 0.8 mm or 1.0 mm I.D. tubing accordingly.
- We recommend you to use an injection valve with a bypass to prevent the column from deterioration.

Inner diameter	Flow rate range
7.6 mm	2 - 4 mL/min
8.0 mm	2 - 4 mL/min
10 mm	3 - 5 mL/min
14 mm	5 - 10 mL/min
20 mm	10 - 20 mL/min
30 mm	20 - 45 mL/min
50 mm	70 - 130 mL/min

■ Precautions for the column with small-inner diameter and the UHPLC column with small-particle size columns

The extra column volume has a major effective on sample diffusion. When you use the column of the I.D. 2 mm, optimize LC system as shown below.

- 1) The tubing between injector - column - detector should be as short as possible to minimize a dead volume. The tubing I.D. should be 0.15 mm or less and optimized. No voids must be formed in the connect.
- 2) Use a low volume type of flow cell of detector, such as semi-micro or micro. Use minimum sample loop.
- 3) The response of the detector and the data sampling rates of the data processing instrument should be optimized according to the peak in order to acquire appropriate sharp peaks with short retention time.

■ Storage of Columns

- After using a column with eluent containing buffer or ion-pair reagent, flush the column with at least five times of the column volume of a salt-free eluent before storing.
- When storing the column for a long period, replace the inner solvent with 100% organic one such as methanol or the shipping solvent.
- Seal the column with the plugs provided and store it at a stable temperature and moisture.

■ Disposal Precautions

When disposing the column, comply with the processing standards determined by law, separately from general industrial waste and household garbage.

■ Technical Support

Shim-pack GIS Series columns are manufactured, inspected, packaged and shipped under strict standards of quality control. Should you find any defect in performance, please contact your local Shimadzu representative for assistance and to possibly arrange a replacement.