

Operation Manual

Shodex™ GPC HK-404L

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

1. Introduction

Shodex GPC HK-404L is a rapid analysis Gel Permeation Chromatography column which is suitable for the measurement of molecular weight distribution of a polymer using an organic solvent as the eluent. It is possible to measure a wide molecular weight range due to high calibration curve linearity.

2. Handling Instructions <Important>

Caution! Please consult the MSDS for the reagents and solvents used with the columns for health concerns caused by acute exposure due to leakage from the column or adjoining tubing.

Attention! Use the column within the regular range of flow rate, pressure and temperature. The column performance may deteriorate when it is handled beyond the permissible range even for a short time.

3. Specifications

Product code	Product name	Size(mm)		Particle size (μm)	Exclusion Limit Polystyrene	Molecular range * Polystyrene	Theoretical Plate Number (Per column)
		I.D.	Length				
F6429201	HK-404L	4.6	150	3.5	1,000,000	100-1,000,000	$\geq 9,000$

* Please use it as a rough indication for the column selection.

Packing material : Styrene divinylbenzene copolymer
 Column material : SUS 316
 Screw type : Internally-threaded type No.10-32 UNF
 Shipping solvent : Tetrahydrofuran (THF)

1) It is recommended that the guard filter HK-G(Product code: F6700200) be connected to the HK-404L column inlet.

4. Usable conditions

Product Name	Flow rate (mL/min)			Max Pressure (MPa)	Temperature range ($^{\circ}\text{C}$)	
	Normal	Max	Compatible solvents		Normal	Max
HK-404L	0.3-1.0	2.0	0.3	25.0	40	60

1) In case of high molecular weight, it might indicate lower value due to shearing polymer chain. In addition, if the polymer chain is sheared, it is impossible to measure correct molecular distribution.

2) 2-4 columns can be connect directly according to separation poupose or samples, but will be very high pressure, so please check the available pressure range of the pump before use.

5. System Clean up

Clean up the LC system including the injector and the sample loop by switching the valve, and flow the eluent before column installation.

Attention! The previous eluent used for analyses in the system may damage the column if it is not compatible with the column.

Attention! When the composition of the eluent is changed greatly, there is a possibility of contamination of the column due to peeling the substances that adsorbed in the pump and tubing.

6. Column Installation

Install and use the column with the flow through the column matching the flow direction arrow on the column tag. Set the flow rate at 0.3 mL/min, and connect the column. Flow at the low rate until the column temperature increases to the setting temperature, and then increase the flow rate to the analytical condition.

Attention! The column should always be installed in the manner above, for safe and effective operation.

7. Sample Preparation

- 1) To dissolve or dilute a solid sample, the sample should be prepared with elution.
- 2) When fully dissolved, the sample solution should be filtered through a 0.45 μ m filter to remove particulates.
- 3) For HK-404L, an injection volume of 1 to 25 μ L per column is recommended. The recommended sample concentration is 0.05 to 0.5 w/v%, but the optimal concentration varies depending upon the molecular weight and viscosity of the sample. The general relationship between molecular weight and optimal concentration is shown below:

Linear range of MW	Optimal concentration (w/v %)
- 5,000	$\leq 0.5\%$
5,000 - 25,000	$\leq 0.25\%$
25,000 - 200,000	$\leq 0.15\%$
200,000 - 1,000,000	$\leq 0.05\%$

For a sample with high molecular weight, if the concentration of a sample solution is too high, the accurate molecular weight distribution may not be obtained. Therefore, decrease the sample concentration to the possible extent and then inject the sample into the column.

8. Eluent

- 1) A basic eluent is Tetrahydrofuran (THF). Chloroform and the Dimethylformamide (DMF) can also be used by solvent replacement.

Caution! THF reacts to oxygen in the air and generates the peroxide. When the peroxide is concentrated, there is explosive danger. When concentrating the fractionated components, it requires special attention.

Attention! Please avoid the use of THF that has passed time because there is a high possibility that the peroxide is generated. Please be careful that THF does not absorb the water in the atmosphere during use.

Attention! Please use THF with stabilizers(dibutylhydroxytoluene) as an eluent. It is not recommended that the using THF without stabilizers as an eluent because THF without stabilizers to generate the peroxide, a baseline doesn't become stable.

When using THF without stabilizers, please use it immediately after the opening, please do not use the THF the number of days has passed.

- 2) The eluent should be filtered through a 0.45 μ m filter to remove particulates.
- 3) Please use the fully degassed eluent. Eluent can be degassed under reduced pressure with an aspirator while using the ultrasonic generator, but it is recommended the use of dissolved gas removal device in order to obtain a stable baseline.

9. Solvent replacement

Attention! In case of solvent replacement, enough flow of intermediate solvent is applied at first, which is a 1:1 mixture of the current solvent and the final desired solvent. Next, the final desired solvent flow is applied.

Attention! When using the solvent containing salt, flow the solvent not containing salt first, then flow the solvent containing salt. When replacing the solvent from the solvent containing salt to other solvent not containing salt, please flow the same solvent not containing salt for washing out the salt, then flow the final desired solvent.

Attention! Please set the flow rate less than 0.3 mL/min when replacing the in-column eluent.

Attention! Please set the temperature of the column oven from 40°C to 60°C when replacing the in-column eluent.

Attention! Store the column as filled with the eluent used.

10. Column Cleaning

Elution characteristics of a column may change considerably after long, repeated usage, due to the accumulation of pollution components on the packing material, for example metal ions from the LC system or the sample.

Clean the guard column and analytical column separately by flowing eluent at 50% of ordinary flow rate in the opposite direction of the arrow on the column tag.

11. Storage solvent

Store the column as filled with eluent.

12. Column Inspection

Column inspection method is described in Certificate of Analysis (CoA).

Attention! Analyze by inspection condition on the Certificate of Analysis and Confirm column performance after purchasing and in each used.

13. Attention

- 1) Do not remove the end fittings of the column to prevent performance deterioration and for safety reasons.
- 2) Do not make a strong impact on the column: such as hitting or dropping on the floor.
- 3) When the column is not used for two weeks or more, remove it from the LC system, close each end with a stopper, and store it at controlled room temperature.
- 4) Contact Shodex website (<http://www.shodex.com/>) or Shodex partners regarding product and analysis applications.

14. Warranty (Ver. 3)

1) Showa Denko K. K. warrants that the Shodex™ Column, at the time of delivery to the user, will conform to the specification of the attached Certificate of Analysis, if the Shodex Column is used in accordance with the attached operating manual. The foregoing warranty is exclusive and is in lieu of all other warranties with respect to the Shodex Column, whether written, oral, implied, statutory or otherwise. No warranties by Showa Denko K. K. are implied or otherwise created, including, but not limited to, the warranty of merchantability and fitness for particular purposes.

2) Any claim of inconformity to the specification must be notified to Showa Denko K.K. within ten (10) days after delivery to the user. User's exclusive remedy and Showa Denko K.K.'s exclusive liability for such claim are limited to the replacement of the Shodex Column in question. In no event is Showa Denko K.K. liable for any indirect, incidental or consequential damage arising out of in connection with the Shodex Column, whether or not such damage is allegedly based on breach of warranty, negligence or otherwise.

3) No warranty is made in any of the following cases:

3-1 If the Shodex Column is not used in accordance with the operating manual

3-2 If the Shodex Column is remodeled by anyone other than person or firm designated by Showa Denko K.K.

3-3 If the Shodex Column is disposed of

3-4 If the Shodex Column is resold by the user without giving prior written notice to Showa Denko K.K.

3-5 If the performance of the Shodex Column is not conform to the specification of the attached Certificate of Analysis due to any of the reasons below:

a) Computer virus

b) Impurities contained in the sample, reagent, gas air or cooling water provided by the user

c) Breakdown or malfunction of equipment, apparatus or component used in combination with the Shodex Column

d) Force majeure such as fire, earthquake, flood, other natural disaster, crime, riot, act of terrorism, war or radioactive contamination

4) In no event is Showa Denko K.K. liable for

4-1 the results of analyses or preparations using the Shodex Column or any portion of the same, including, but not limited to, the reliability, accuracy, efficacy and safety of said results, and

4-2 the occupational hazard in the use of the Shodex Column, whether or not such use is made in accordance with the attached Conditions for use.

5) The Shodex Column is for laboratory use only. It must not be used for clinical diagnosis. Showa Denko K.K. is not liable for any use of the Shodex Column except laboratory use.