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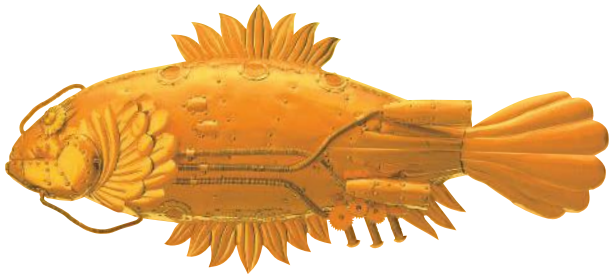

HPLC Columns

MANUAL

GPC HFIP-600

SHOWA DENKO EUROPE

Columns manufactured by Showa Denko K.K Japan
Made in Japan

Shodex HPLC Columns
Europe, Middle East, Africa, Russia

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Operation Manual

Shodex™ GPC HFIP-600 series

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

1. Introduction

Shodex GPC HFIP-600 series are small volume type high-performance HPLC columns for GPC , which use HFIP (hexafluoroisopro-panol) as the eluent.

Engineering plastics such as polyamides, polyesters and polyacetates are soluble in HFIP at room temperature though they are insoluble in THF and DMF at the temperature. Polypeptides, polyvinylalcohol, ethylenevinyl-alcohol copolymers, styrenemaleamide copolymer, polyacrylonitrile, poly-methylmethacrylate are also soluble in HFIP.

HFIP-600 columns are specially developed for the size-exclusion analysis of such oligomers and polymers at room temperature.

2. Specifications

Nomenclature	Exclusion limit	Theoretical plates/column	Solvent packed
Shodex GPC HFIP-603	7×10^4	14000 minimum	HFIP
Shodex GPC HFIP-604	4×10^5	13000 minimum	HFIP
Shodex GPC HFIP-605	4×10^6	5000 minimum	HFIP
Shodex GPC HFIP-606	(2×10^7)	5000 minimum	HFIP
Shodex GPC HFIP-606M	(2×10^7)	6000 minimum	HFIP
Shodex GCP HFIP-607	(2×10^8)	3000 minimum	HFIP
Shodex GPC HFIP-G		Precolumn	HFIP

Notes: Exclusion limits are the molecular weight of polystyrene.

Size: 6.0 mm ID x 150 mm length (main column)
4.6 mm ID x 10 mm length (precolumn)

End fittings:	Internally-threaded type, No. 10-32 UNF.	
Column material:	SUS 316.	
Packing material:	Styrene-divinylbenzene gels.	
Max. usable temperature:	50°C.	
Max. usable pressure:	(603, 604)	6.0MPa/column
	(605 – 607)	1.0MPa/column

Caution!

1) Use HFIP of high purity as the eluent.

2) HFIP is the solvent of highly moisture adsorption. Be careful not to use HFIP under the open air because sample sometimes cannot be solved in such HFIP solvent which contain much water.

3) HFIP is toxic and vaporable. Be careful not to touch it with skin. And use it under the ventilation.

4) Do not set the flow rate above (603, 604 are) 0.3ml/min (605 - 607 are) 0.6ml/min.

5) Do not abruptly change the column pressure or the flow rate while the liquid chromatograph is in operation.

Use a damper-equipped or pulseless pump to maintain the performance of the column at the designed level for a long period of time.

6) It is recommended to use the column in a constant temperature chamber at the range of 30°C to 40°C.

7) Do not impact the column.

8) Do not remove the endfittings of the column under any circumstances; otherwise, its performance will deteriorate.

9) Install precolumn immediately upstream of the main column to protect it from contamination by the sample. The precolumn is intended to maintain the column performance as designed for a long period of time and not to improve its resolving power.

3. Installation and start-up

1) Prior to connection of the column to the liquid chromatograph, replace the solvent in the chromatograph with the HFIP solvent.

If the liquid chromatograph is equipped with a device in which complete replacement of the solvent is not possible, e. g., a Bourdon pressure gauge, disassemble the device and wash it with the solvent that is to be used as the eluent.

2) Thoroughly degas the eluent by subjecting it to ultrasonic vibration and simultaneous heating or pressure reduction with an aspirator. Use of solvent degassing devices of Shodex DEGAS KT series will facilitate the degassing work.

Do not use as the eluent THF that has long been exposed to air or any solvent that has a high water content, as the eluent.

3) After replacing the solvent in the chromatograph, set the flow rate at 0.05 to 1.0 ml/min. using a stopwatch and 10ml measuring cylinder.

4) Connect the column to the chromatograph so that the arrow on the column will fase downstream.

5) Set the column in a constant temperature chamber and raise the temperature in it to 30°C to 40°C.

6) Upon completion of the connection, start the pump, watching for any sudden change in the column pressure or the flow rate.

4. Pretreatment of sample

1) Dewater any sample that contains water.

2) Dissolve the completely dried sample in the same solvent that is to be used as the eluent.

To make the blank peaks as small as possible when a detector such as a differential refractometer is used, it is recommended that the sample be dissolved in solvent obtained from the reservoir of the chromatograph for the solvent with which the sample is to be separated.

3) In case of the sample has a molecular weight of 1, 000,000 minimum, soak it in such solvent for 12 to 24 hours to let it swell.

After the swelling, agitate the solvent gently for dissolution.

4) In case of the sample is a polymer, its concentration in the solution and the injection volume should be 0.05 to 0.5% and 1 to 20 ul, respectively.

5) Remove extraneous matter or gels from the dissolved sample by passing it through a 0.45um filter. Use of the disposable filter unit Shodex DT ED-03CR, 13CR or 25CR is recommended.

5. Safekeeping

1) Even after completion of an analysis, keep pumping the eluent at a flow rate of 0. 1ml/min. until the column is cooled down to room temperature.

2) Cap both end of the column to prevent the eluent from leaking out.

3) Package it as delivered from the manufacturer.

4) Store it at a place where temperature change is minimum.

5) When a column is not expected to be used for a long period of time detach the detector inlet line from the column and connect a Teflon tube of 1/16 inch in outside diameter, 0.8mm in inside diameter and 500mm in length to the column outlet.

Start pumping the eluent at a flow rate of 0. 1 ml/min. and stop the pump as soon as it begins to flow out from the free end of the tube. Put 30ml of toluene into a 50-ml bottle and soak the free end of the toluene to prevent air from entering the column. Dismount the column from the chromatograph, blank the column's inlet end and store it in a room that has Iittle temperature fluctuation.

6. Standard sample

For the GPC analysis using THF eluent, polystyrene standards are used to get the calibration curve. In case of GPC analysis using HFIP eluent, polymethylmethacrylate standards are used to get the calibration curve. Please refer to the following paper for the details: T. Provder et a I., Advan. Chem. Ser. No. 125, 117-137 (1973).

7. Calibration

The column is calibrated by ensuring that the specified plate number is maintained.

Following are the conditions for calculation of the plate number:

1) Sample: 5% acetone in HFIP solvent

2) Injection volume:	2ul	
3) Eluent:	HFIP solvent	
4) Flow rate:	(603, 604)0.25ml/min	(605- 607)0.5ml/min.
5) Detector:	254-nm UV detector	
6) Calculation formula:	$N = 5.54 \times (t_R/W)^2$	