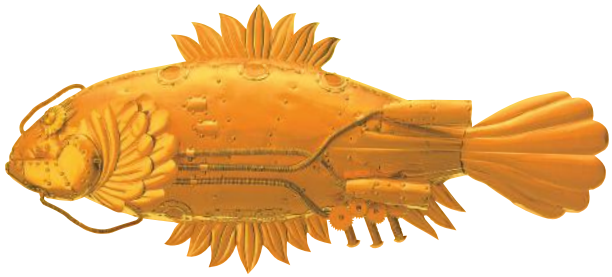
Shodex™



HPLC Columns

MANUAL

GPC KF-800 , K-800

SHOWA DENKO EUROPE

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

Shodex HPLC Columns
Europe, Middle East, Africa, Russia

For technical support please use contact details shown below:

SHOWA DENKO EUROPE GmbH
Shodex Business
Konrad-Zuse-Platz 3
81829 Munich, Germany

E-mail: support@shodex.de
Phone: +49 (0)89 93 99 62 37
www.shodex.de

Operation Manual

Shodex™ GPC KF-800, K-800

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

1. Introduction

The packed columns of the Shod ex GPC KF- 800 and K- 800 series are designed for high-speed and high-resolution performance. Having an organic solvent as the eluent and hard polystyrene gels of narrow particle size distribution as the packing, they are suited for separation of organics and oligomers by molecular size as well as measurement of the molecular weight distribution of polymers.

2. Specifications

Nomenclature	Exclusion limit[1)]	Plate number/30cm[2)]
GPC KF-801 (K-801)	1.5×10^3	>16,000
GPC KF-802 (K-802)	5×10^3	>16,000
GPC KF-802.5 (K-802.5)	2×10^4	>16,000
GPC KF-803 (K-803)	7×10^4	>16,000
GPC KF-803L (K-803L)[5)]	7×10^4	>16,000
GPC KF-804 (K-804)	4×10^5	>16,000
GPC KF-804L (K-804L)[5)]	4×10^5	>16,000
GPC KF-805 (K-805)	4×10^6	>10,000
GPC KF-805L (K-805L)[5)]	4×10^6	>10,000
GPC KF-806 (K-806)	2×10^7 (est)	>10,000
GPC KF-806L (K-806L)[5)]	2×10^7 (est)	>10,000
GPC KF-806M (K-806M)[3)]	2×10^7 (est)	>12,000
GPC KF-807 (K-807)	2×10^8 (est)	>5,000
GPC KF-807L (K-807L)[5)]	2×10^8 (est)	>5,000
GPC KF-G (K-G)[4)]	-	-

Notes: 1) Molecular weight of polystyrene as measured with tetrahydrofuran (THF) used as the eluent.

2) Eluent, THF; flow rate, 1 ml/min; sample, 20 µL of THF containing 0.2% n-propylbenzene.

3) The packing of this column is a mixture of the same packings as those used in KF- 803, -804,-805 and -806.

4) Guard column for KF- (K-) series columns.

5) Mixed gel, linear type (linear calibration curve).

Size:	ID: 8mm; length 300 mm (exclusive of K –(K-) G of 4.6 mm in ID and 10 mm in length.)
End fittings:	Internally-threaded type, No. 10-32 UNF.
Material:	SUS 316.
Packing:	Styrene-divinylbenzene gels.
In-column eluent:	THF (KF-series), Chloroform (K-series).
Working temperature:	Room temperature to 60°C.

Caution!

1) Do not use solvents, e.g., water, alcohol and hexane, that cause shrinkage styrene-divinylbenzene gels, as the eluent.
2) Do not set the flow rate above 2.0mL/min. A flow rate of 1.0 to 1.5 mL/min. is recommended.
3) In case the column pressure is likely to exceed 3,5MPa per column when the flow rate is in the above-mentioned range, decrease the flow rate or heat the column so as not to allow the column pressure to increase beyond the level. Use of a pressure gauge capable of measuring up to 15MPa is recommended to obtain a proper pressure.
4) Do not abruptly change the column pressure or the flow rate. Use a damper equipped or pulseless pump to maintain column performance at the designed level for a long period of time.
5) Do not impact or bend the column.

6) Do not remove the end fittings of the column under any circumstances; otherwise, its performance will deteriorate.
7) When separating a sample containing relatively large quantities of gels and insolubles such as rubber, tar and pitch, maximize the sensitivity of the detector and minimize the injection volume so as to prolong the column life.
8) Install guard column immediately upstream of the main column to protect it from contamination by the sample. The guard column is intended to maintain the main 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 solvent that is to be used as the eluent.

In replacing water with, for instance, chloroform, which is not soluble in water, first replace the water with acetone and then replace the acetone with chloroform.

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.

3) After replacing the solvent in the chromatograph, set the flow rate at 1 ml/min.

Note: A flow rate of 1. 0 ml/min or less is recommended for measurement of the molecular weight distribution of polymers.

In case of polymers with a molecular weight of 4,000,000 or higher, a flow rate of 1. 5 ml/min or more will produce shear force to degrade the polymer chains, thereby making it sometimes impossible to obtain the right molecular weight distribution. The lower the

flow rate, the less the effect of shear force and the better the separation of the polymers.

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

5) 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) Dissolve the sample in the same solvent that is to be used as the eluent.

Notes: i) Dissolving the sample in such solvent will make the blank peaks as small as possible.

ii) In case 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. Strong agitation or use of an ultrasonic bath for dissolution will degrade such sample.

iii) In case the sample is a polymer, its concentration in the solution and the injection volume should be 0.05 - 0.5% and 50 - 100 µl, respectively. If the concentration is higher, the retention volume of the sample will increase. The optimum concentration changes with the molecular weight and the viscosity. The following table gives the molecular weight vs. the optimum concentration.

Molecular weight	Optimum concentration (%)
Up to 5,000	<1.0
5,000 – 25,000	<0.5
25,000 – 200,000	<0.25
200,000 – 2,000,000	<0.1
Above 2,000,000	<0.05

iv) In case the sample is organic, it is desirable that the concentration be 1% maximum and the injection volume, 50µl maximum.

2) Remove extraneous matter or gels from the dissolved sample by passing it through a 0.5µm filter. Use of the disposable filter unit Shodex DT ED is recommended.

3) Dewater any sample that contains a large quantity of water.

5. Preheating of column

Preheating the column will improve its separation performance.

However, do not raise the column temperature any higher than 60°C.

6. Replacement of in-column eluent

Each column is delivered to the user, filled with THF (KF-series), or chloroform (K-series), which can be replaced with other eluents such as toluene and methylene chloride. Set the flow rate at 0.5ml/min when replacing the in-column eluent.

Avoid repeated replacement of the in-column eluent with an eluent of a different kind lest it should deteriorate column performance.

7. Safe keeping

1) Cap both ends of the column to prevent the eluent from leaking out.

2) Package it as delivered from the manufacturer.

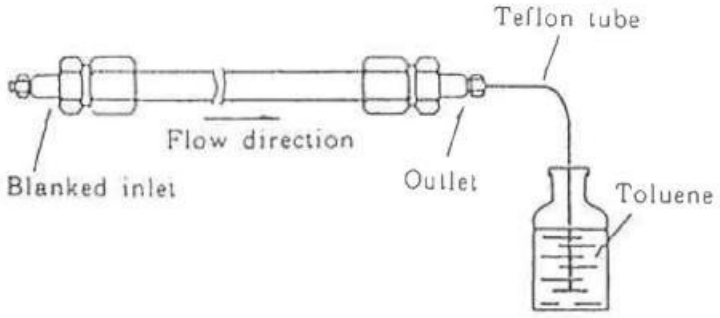
3) Store it as a place where temperature change is minimum.

4) When the 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, 0.8 mm in inside diameter and 500 mm in length to the column outlet.

5) Start pumping the eluent at a flow rate of 0. 5 ml / min and stop the pump as soon as it begins to flow out from the free end of the tube.

6) Put 80 ml of toluene into a 100-ml bottle and soak the free end in the toluene to prevent air from entering the column.

7) Dismount the column from the chromatograph, blank the column's inlet end and store it in a room that has little temperature fluctuation.



8. Calibration

Following are the conditions for calculation of the plate number.

1) Sample: Eluent containing 0.2% n-propylbenzene.

2) Injection volume: 20 µl.

3) Eluent: THF (KF-series), Chloroform (K-series).

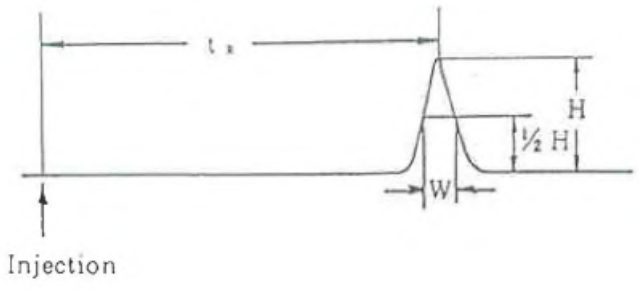
4) Flow rate: 1mL/min.

5) Detector: 254-nm UV detector

6) Chart speed: 1.5 – 2cm/min

7) Detector sensitivity: The sensitivity must be so adjusted as to obtain a peak of 10 to 15 cm in height.

8) Calculation formula : $N = 5.54 \times (t_R/W)^2$



9. Warranty

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 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 Instrument, 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:

(1) If the Shodex Column is not used in accordance with the operating manual.

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

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

(4) 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, rime, riot, act of terrorism, war or radioactive contamination

4) In no event is Showa Denko K.K. liable for (i) 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 (ii) 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 instrument 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 Instrument except laboratory use.