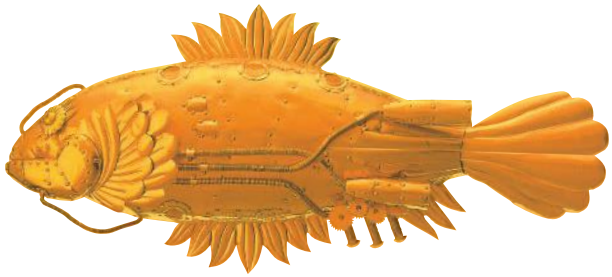
Shodex™



HPLC Columns

MANUAL

GPC KD-800/AD-800S series

Shodex HPLC Columns
Europe, Middle East, Africa, Russia

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

Operation Manual

Shodex™ GPC KD-800, AD-800S

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

1. Introduction

The Shodex GPC AD-800/S and KD-800 series comprises high-performance GPC pavked columns of different exclusion limits, which use dimethylformamide (DMF) exclusively as the eluent and sort out polar compounds, such as melamine resin, phenolic resin, polyamide, polyurethane and polyvinyl pyrrolidone, according to size. Careful observance of the instructions in this operation manual will enable the packed columns to function as designed over the longest possible of time.

2. Specifications

2.1 GPC AD-800/S series

Nomenclature	Exclusion limit[1)]	Plate number/25cm[2)]
GPC AD-802/S	5×10^3	7000 minimum
GPC AD-802.5/S	2×10^4	7000 minimum
GPC AD-803/S	7×10^3	7000 minimum
GPC AD-804/S	4×10^5	7000 minimum
GPC AD-805/S	5×10^6	7000 minimum
GPC AD-806/S	2×10^7 (est)	7000 minimum
GPC AD-806MS	2×10^7 (est)	7000 minimum
GPC AD-807/S	2×10^8 (est)	4000 minimum
GPC AD-G	-	-

Size: ID = 8mm; Length = 250mm (exclusive of AD-G of 6mm in ID and 50mm in length).

End fitting: Internally-threaded type, No. 10-32 UNF.

Material: SUS 316

Packing material: Styrene-divinylbenzene gels

In-column eluent: DMF

2.2 GPC KD-800 series

Nomenclature	Exclusion limit[1)]	Plate number/30cm[2)]
GPC KD-801	2.5 x 10^3	17000 minimum
GPC KD-802	5 x 10^4	17000 minimum
GPC KD-802.5	2 x 10^3	17000 minimum
GPC KD-803	7 x 10^5	17000 minimum
GPC KD-804	4 x 10^6	17000 minimum
GPC KD-805	5 x 10^6	11000 minimum
GPC KD-806	2 x 10^7 (est)	11000 minimum
GPC KD-806M	2 x 10^7 (est)	13000 minimum
GPC KD-807[3)]	2 x 10^8 (est)	6000 minimum
GPC KD-G	-	-

Size: 10 = 8mm; length = 300mm (exclusive of KD-G of 4.6mm in ID and 10mm in length).

End fittings: Internally-threaded type, No. 10-32 UNF.

Material: SUS 316.

Packing: Styrene-divinylbenzene gels.

In-column eluent: DMF

NOTES:

1) Molecular weight of polyethylene glycol.
2) Eluent, DMF; flow rate, 1.0ml/min; column temperature, 40°C; sample 20µl of DMF containing 0.5% acetone.
3) The packing of this column is a mixture of the same packings as those used in -803, -804, -805, and -806.
4) Precolumn for AD-series columns.
5) Precolumn for KD-series columns.

Caution!

1) Do not replace the DMF contained in the packed columns of this series with any other eluent, because they are designed to use DMF exclusively as an eluent.

2) Use a DMF that is classified as a guaranteed reagent. Since it may deteriorate when exposed to air, use DMF that has been obtained direct from the bottle or whatever else its container immediately after opening the container.
3) In the case of aromatic hydrocarbons, e.g., benzene and toluene, or of standard polystyrenes, molecular adsorption causes elution to be somewhat slower than when THF is the eluent.

   It is therefore, recommended that either polythylene glycol or polypropy glucol be used to obtain a standard chromatogram.
4) The exclusion limit of standard polystyrene is higher than when THF is used as the eluent in a column containing the same packing material as that of the packed columns of this series.
5) In analyzing specimens of some materials by gel permeation chromatography, using a polar eluent like DMF, a salt, e.g., lithium bromide, has to be added in small quantities to the eluent to reduce their adsorptivities and to keep the ionic strength of the eluent constant.
6) Install precolumn Shodex immediately upstream of the main column to protect it from contamination by the specimen.
7) Heat both the main column and the precolumn to temperature of 40-60° C and the flow rate must not exceed at 1.5ml/min.

   Note: Since DMF is too viscous at normal temperatures to enable the column to attain its maximum performance, its viscosity must be lowered by heating the column to disperse the molecules extensively and thereby improve the resolution of the column.
8) Keep the column pressure at 1.0 MPa (AD-800 series), 3.0 MPa (KD-800 series) maximum per column. Use of a pressure gauge capable of measring up to 10.0 MPa is recommended for accurate measurement of the pressure.
9) Do not abruptly change the column pressure or the flow rate. Use a damper to prolong the service life of the column.

3. Installation

1) Prior to connection of the column to a liquid chromatograph, replace the eluent in the liquid chromatograph with DMF.

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.

3) Set the flow rate at 1 ml/min.

4) Connect the column to the liquid chromatograph so that the arrow marked on the column will face downstream.

5) Heat the column and the pre-column to a temperature of 40-60° C range and start the pump.

4. Pre-treatment of specimen

1) In case the sample contains water, dehydrate it by any method deemed appropriate.

Note: Water in the sample will cause a high minus peak in the chromatogram, shrinh the gel and impair the performance of the packed column.

2) Dissolve the sample in the same DMF that is used as an eluent.

Note: In case the specimen is a macromolecular compound, its concentration in the solution and the amount in which it is injected should be 0.05-0.5% and 100-500µl respectively and in case it is an organic, the maximums should be 1% and 1 µl repectively.

3) Remove extraneous matter or gels from the dissolved sample by passing through a 0.45µm filter. Use of the disposable filter unit Shodex DT is recommended.

5. Dismounting and safekeeping

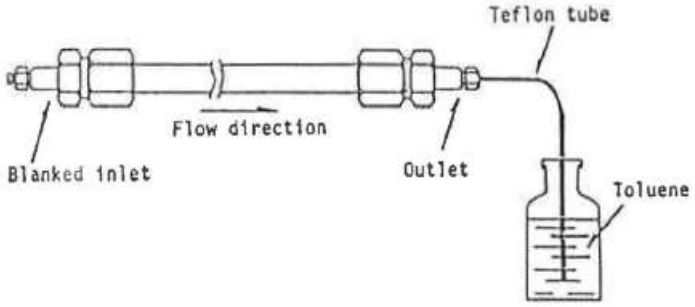
1) Even after completion of an analysis, keep pumping the eluent at a flow rate of 0.5ml/min until the column is cooled down to room temperature.

2) 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, 0.8mm in inside diameter and 500mm in length to the column outlet.

3) Start pumping the mobile phase at a flow rate of 0.5ml/min and stop the pump as soon as it begins to flow out from the free end of the tube.

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

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



6. Calibration

Following are the conditions for calculation of the plate number.

1) Sample: Eluent containing 0.5% acetone

2) Injection volume: 20µl

3) Mobile: DMF

4) Flow rate: 1 ml/min

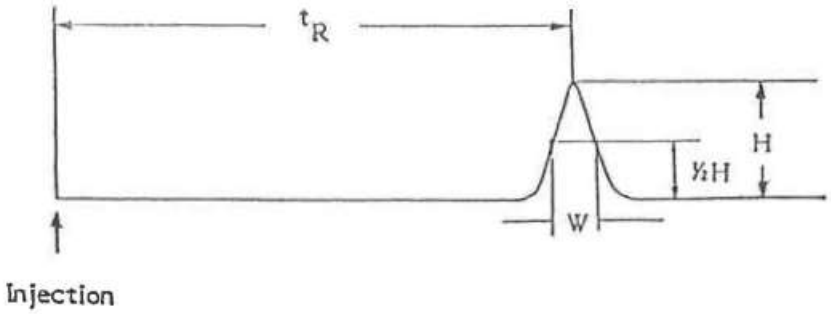
5) Column temperature: 40° C

6) Detector: Shodex RI detector

7) Calculation method

The number of the theoretical plates is calculated as per the following formula:

$$N = 5.54\ (t_R/W)^2$$



7. 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.