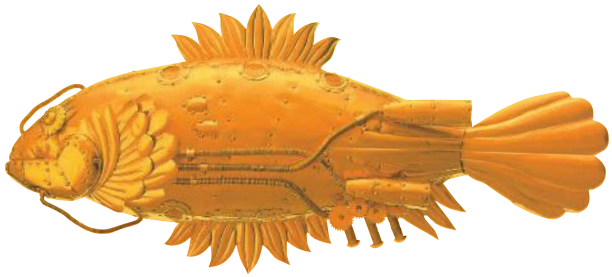
Shodex™



HPLC Columns

MANUAL

GPC AT series

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

SHOWA DENKO EUROPE

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

Operation Manual
Shodex™ GPC AT-800S

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

1. Introduction

The Shodex GPCAT-800S series comprises high-performance GPC packed columns of different exclusion limits, of which in-column eluent is toluene.

The columns are specially developed for high-temperature GPC analysis.

2. Specifications

Nomenclature	Exclusion limit	Theoretical plates/25cm	Solvent packed
Shodex GPC AT-803S	7×10^4	6,000 minimum	Toluene
Shodex GPC AT-804S	4×10^5	6,000 minimum	Toluene
Shodex GPC AT-805S	5×10^6	6,000 minimum	Toluene
Shodex GPC AT-806S	(5×10^7)	6,000 minimum	Toluene
Shodex GPC AT-806MS	(5×10^7)	6,000 minimum	Toluene
Shodex GPC AT-807S	(2×10^8)	3,300 minimum	Toluene
Shodex GPC AT-G①	Guard column	Guard column	Toluene

Note: Exclusion limits are the molecular weight of polystyrene.

Size:	8 mm I.D. x 250 mm length (main column).
	8 mm I.D. x 50 mm length (guard column).
Endfitting:	Internally-threaded type, No. 10-32 UNF.
Column material:	SUS 316.
Packing material:	Styrene-divinylbenzene gels.
Max. usable flow rate:	1.5 ml/min.
Max. usable temperature:	150°C.
Max. usable pressure:	2.0MPa/column.
Usable eluent:	o-dichlorobenzene or 1, 2, 4-tri-chlorobenzene.

Caution!

1) Use a high-purity eluent, such as GPC grade for replacement.

2) When the in-column eluent is replaced with 1, 2, 4-trichlorobenzene, keep the column temperature at 20°C minimum when the column is not in use, because the eluent congeals at 17° C.

3) When it is required to lower the column temperature after suspension of the operation, reduce the flow rate to 0.2 or 0.3ml/min. and gradually lower the temperature.

4) Keep the in-column pressure at 2.0MPa maximum, regardless of the flow rate.

Even if the flow rate is maintained at 1.5ml/min. maximum, the danger remains of the pressure exceeding the limit, depending on the column temperature or the kind of eluent used. In such case, therefore, lower the flow rate to the extent necessary to keep the pressure at the specified level.

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) Do not impact or bend the column.

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

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 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 to be used as the eluent.

2) Thoroughly degas the solvent to be used as the eluent, by subjecting it and ultrasonic vibration and simultaneous heating or pressure reduction with an aspirator.

3) Connect the column to the chromatograph as that the arrow mark on the column will face downstream.

4) Heat the column to 60°C and flow 30ml of the replacing eluent through it at a flow rate of 0.2 or 0.3 ml/min.

Note: In the event the number of columns is increased by a factor of, for instance, 3, the amount of replacing eluent must also be increased by the same factor, i.e. 90ml.

5) Heat the column to the range of 140°C to 150°C and raise the flow rate to 1.0ml/min. in about 15 minutes.

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

4. Pre-treatment of sample

1) Dewater any sample that contains water.

2) Dissolve the 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 50 to 100µl, respectively.

5) Remove extraneous matter or gels from the dissolved sample by passing it through a 0.45um filter.

5. Safekeeping

1) Even after completion of an analysis, keep pumping the eluent at a flow rate of 0.1 to 0.2ml/min. until the column is cooled down to room temperature.

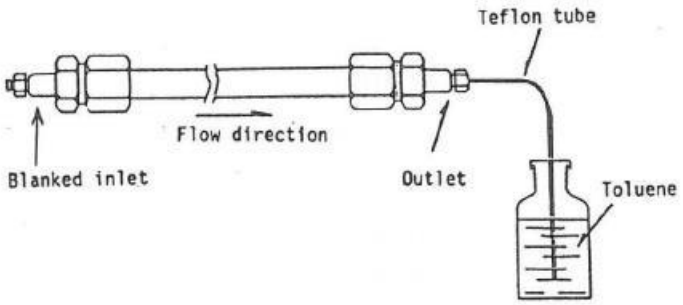
2) Cap both ends of the column to prevent the eluent from leaking out.

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 0.5 ml/min. and stop the pump as soon as it begins to flow out from the free end of the tube. Put 80ml of toluene into a 100-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 little temperature fluctuation.



6. Calibration

The column is calibrated by ensuring that the specified plate number is maintained. Following are the conditions for calculation of the plate number:

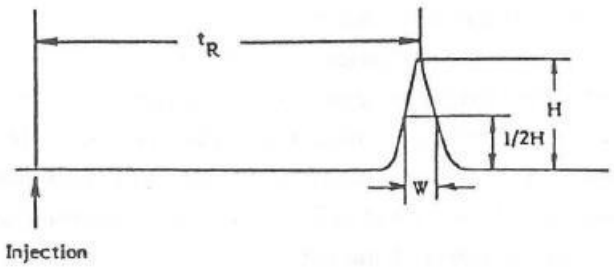
1) Sample: 1% acetone in toluene.
2) Injection volume: 20µl.
3) Eluent: Toluene.
4) Flow rate: 1.0 ml/min.
5) Detector: Shodex RI.
6) Chart speed: 1.5 to 2.0 cm/min.
7) Detector sensitivity: The sensitivity must be so adjusted as to obtain a peak of 10 to 15 cm in height.
8) Temperature: Room temperature.

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

Where N: Theoretical plate number

t_R: Retention time

W: Peak half width



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.