

O p e r a t i o n M a n u a l
f o r
S h o d e x P A K
S i l i c a s e r i e s

For the longest possible service life of the column,
Please carefully observe the instruction in this manual.

Shodex Silica series

1. Introduction

Shodex Silica series are high resolution type columns packed with totally porous spherical type silica gels for adsorption and partition chromatography.

[C 1 8 M]

The columns are packed with octadecyl-bonded(monomeric type) silica gels for reverse-phase chromatography. These columns are used for general purpose.

[C 1 8 P]

The columns are packed with octadecyl-bonded(polymeric type) silica gel for reverse-phase chromatography. These columns are used for general purpose.

[5 C 8]

The columns are packed with octyl-bonded silica gel for reverse-phase chromatography. These columns are used for general purpose.

[5 C 4]

The columns are packed with butyl-bonded silica gel for reverse-phase chromatography. These columns are used for general purpose.

[5 TMS]

The columns are packed with trimethyl-bonded silica gel for reverse-phase chromatography. These columns are used for general purpose.

[5 CN]

The columns are packed with cyanopropyl-bonded silica gel.

[5 NPE]

The columns are packed with nitrophenylethyl-bonded silica gel.

[5 PYE]

The columns are packed with 2-(1-pyrenyl)ethyl-bonded silica gel.

[5 NH]

The columns are packed with aminopropyl-bonded silica gel. The column can be used for three kinds of separation modes, normal-phase, reverse-phase and weak anion exchange chromatography.

The column is used for separation of substances such as sugars and vitamins.

[5 S I L]

The columns are packed with silica gel. The columns are used for the separation of substances such as synthesized medicines and extract from natural materials.

2. Specifications

Nomenclature	Column size (I. D. x length)	Plate cont (min.)	Gel type	Gel size	Solvent Packed
C18M 4D	4.6mm x 150mm	10,000	(a)	5 μm	70%CH ₃ OH/H ₂ O
C18M 4E	4.6mm x 250mm	16,000	(a)	5 μm	70%CH ₃ OH/H ₂ O
C18M 2D	2.0mm x 150mm	9,000	(a)	5 μm	60%CH ₃ CN/H ₂ O
C18M 6D	6.0mm x 150mm	9,000	(a)	5 μm	70%CH ₃ OH/H ₂ O
C18M 6E	6.0mm x 250mm	16,000	(a)	5 μm	70%CH ₃ OH/H ₂ O
C18M 10E	10 mm x 250mm	16,000	(a)	5 μm	80%CH ₃ OH/H ₂ O
C18M 20E	20 mm x 250mm	16,000	(a)	5 μm	80%CH ₃ OH/H ₂ O
C18P 4D	4.6mm x 150mm	10,000	(b)	5 μm	70%CH ₃ OH/H ₂ O
C18P 4E	4.6mm x 250mm	16,000	(b)	5 μm	70%CH ₃ OH/H ₂ O
C18P 2D	2.0mm x 150mm	9,000	(b)	5 μm	60%CH ₃ CN/H ₂ O
C18P 6D	6.0mm x 150mm	9,000	(b)	5 μm	70%CH ₃ OH/H ₂ O
C18P 6E	6.0mm x 250mm	16,000	(b)	5 μm	70%CH ₃ OH/H ₂ O
C18P 10E	10 mm x 250mm	16,000	(b)	5 μm	80%CH ₃ OH/H ₂ O
C18P 20E	20 mm x 250mm	16,000	(b)	5 μm	80%CH ₃ OH/H ₂ O
5C8 4D	4.6mm x 150mm	9,000	(c)	5 μm	66%CH ₃ OH/H ₂ O
5C8 4E	4.6mm x 250mm	15,000	(c)	5 μm	66%CH ₃ OH/H ₂ O
5C8 10E	10 mm x 250mm	15,000	(c)	5 μm	66%CH ₃ OH/H ₂ O
5C8 20E	20 mm x 250mm	15,000	(c)	5 μm	66%CH ₃ OH/H ₂ O
5C4 4D	4.6mm x 150mm	9,000	(d)	5 μm	55%CH ₃ OH/H ₂ O
5C4 4E	4.6mm x 250mm	15,000	(d)	5 μm	55%CH ₃ OH/H ₂ O
5C4 10E	10 mm x 250mm	15,000	(d)	5 μm	55%CH ₃ OH/H ₂ O
5C4 20E	20 mm x 250mm	15,000	(d)	5 μm	55%CH ₃ OH/H ₂ O
5TMS 4D	4.6mm x 150mm	7,000	(e)	5 μm	50%CH ₃ OH/H ₂ O
5TMS 4E	4.6mm x 250mm	12,000	(e)	5 μm	50%CH ₃ OH/H ₂ O
5TMS 10E	10 mm x 250mm	12,000	(e)	5 μm	50%CH ₃ OH/H ₂ O
5TMS 20E	20 mm x 250mm	12,000	(e)	5 μm	50%CH ₃ OH/H ₂ O
5CN 4D	4.6mm x 150mm	7,000	(f)	5 μm	40%CH ₃ OH/H ₂ O
5CN 4E	4.6mm x 250mm	12,000	(f)	5 μm	40%CH ₃ OH/H ₂ O
5CN 10E	10 mm x 250mm	12,000	(f)	5 μm	40%CH ₃ OH/H ₂ O
5CN 20E	20 mm x 250mm	12,000	(f)	5 μm	40%CH ₃ OH/H ₂ O
5NPE 4D	4.6mm x 150mm	8,000	(g)	5 μm	55%CH ₃ OH/H ₂ O
5PYE 4D	4.6mm x 150mm	7,000	(h)	5 μm	70%CH ₃ OH/H ₂ O
5NH 4D	4.6mm x 150mm	5,000	(i)	5 μm	95%CH ₃ CN/H ₂ O
5NH 4E	4.6mm x 250mm	8,000	(i)	5 μm	95%CH ₃ CN/H ₂ O
5NH 10E	10 mm x 250mm	8,000	(i)	5 μm	95%CH ₃ OH/H ₂ O
5NH 20E	20 mm x 250mm	8,000	(i)	5 μm	95%CH ₃ OH/H ₂ O

5SIL 4D	4.6mm x 150mm	9,000	(j)	5 μ m	95% C_6H_{14} / C_2H_5OH
5SIL 4E	4.6mm x 250mm	15,000	(j)	5 μ m	95% C_6H_{14} / C_2H_5OH
5SIL 10E	10 mm x 250mm	15,000	(j)	5 μ m	95% C_6H_{14} / C_2H_5OH
5SIL 20E	20 mm x 250mm	15,000	(j)	5 μ m	95% C_6H_{14} / C_2H_5OH

- (a) Octadecyl-bonded totally porous silica gel (monomeric type)
- (b) Octadecyl-bonded totally porous silica gel (polymeric type)
- (c) Octyl-bonded totally porous silica gel (monomeric type)
- (d) Butyl-bonded totally porous silica gel (monomeric type)
- (e) Trimethyl-bonded totally porous silica gel (monomeric type)
- (f) Cyanopropyl-bonded totally porous silica gel (monomeric type)
- (g) Nitrophenylethyl-bonded totally porous silica gel
- (h) 2-(1-Pyrenyl)ethyl-bonded totally porous silica gel
- (i) Aminopropyl-bonded totally porous silica gel (monomeric type)
- (j) Totally porous silica gel

Endfitting: Internally-threaded type, No.10 32 UNF
 Column material: SUS-316
 Max. pressure: 20 MPa
 Usable pH range: 2 ~ 7.5

Caution

- 1) 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.
- 2) Do not impact or bend the column.
- 3) Do not remove the endfittings of the column under any circumstances; otherwise, its performance will deteriorate.
- 4) Highly pure reagents should be used as the eluent.
- 5) Hydrochloric acid should not be used as the eluent because it is corrosive to column or piping of the instrument.
- 6) The pressure should not exceed the max. usable pressure shown in the specifications. When the gradient method is used, be careful that steep gradient sometimes causes high pressure.

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.
CAUTION ① In replacing the solvent which is insoluble in the eluent to be used, first replace the solvent with an intermediate solvent and then replace it with the eluent.
② When the solvent containing salts is replaced with the eluent containing organic solvents, or when the solvent containing organic solvents is replaced with the eluent containing salt, purified water should be used as the intermediate solvent.
③ 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 solvent that is to be used as the eluent, by subjecting it and ultrasonic vibration and simultaneous heating or pressure reduction with an aspirator.
Use of solvent degassing devices will facilitate the degassing work.
 - 3) After replacing the solvent in the chromatograph, set the flow rate at 1.0ml/min.
 - 4) Connect the column to the chromatograph as that the arrow mark on the column will face downstream.
 - 5) 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) 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.
 - 2) Remove extraneous matter or gels from the dissolved sample by passing it through a 0.45 μ m filter.
Use of the disposable filter unit Shodex DT is recommended.
 - 3) The pH, salt concentration and polar organic solvent concentration of sample solution should be as same as those of the eluent.

5. Safekeeping

- 1) After completing analysis, keep pumping the eluent at a flow rate of 0.3 to 0.5ml/min. until the column is cooled down to room temperature if the column is used at an elevated temperature.
- 2) Replace the in-column eluent with the following solvents and disconnect it from the chromatograph and cap both ends of the column to prevent the eluent from leaking out.
 - C18M, C18P, 5C8, 5C4, 5TMS, 5NPE, 5PYE: 50~100% Methanol/H₂O
 - 5CN: Acetonitrile
 - 5SIL: n-Hexane or n-Heptane
- 3) Package it as delivered from the manufacturer and store it in a room that has little temperature fluctuation.