

High Performance Packed Column  
for High Performance Liquid Chromatograph

Shim-pack PREP Series  
PREPARATIVE COLUMN

INSTRUCTION MANUAL

1. Introductions

The Shimadzu Shim-pack "PREP" Series of columns are 20 mmID X 25 cm long stainless steel tubes packed with totally porous, irregular-shaped silica particles (15 μm particle diameter, 100Å pore diameter) with chemically modified surfaces. This series has been designed exclusively for preparative separations.

The silica packings are fully end-capped to suppress residual silanol-group influences.

The PREP series consists of seven types of stationary phase groups as shown in the following table.

2. Operating Care

- Mobile phase flow in the direction indicated on the column label only.
- Maximum usable pressure is 250 kg/cm<sup>2</sup>; lower pressures (≤ 150 kg/cm<sup>2</sup>) result in longer lifetime at high efficiency.
- To protect the chemically modified silica surface do not use solutions of lower than pH 2 or higher than pH7.5.
- Do not subject to shock such as from dropping, etc.
- Dust particles in mobile phase or sample solution lead to plugging at the column inlet filter or at the top of column bed. Filter the mobile phase and sample solution prior to use, if necessary.

3. Selection of Mobile Phase

<PREP-ODS, -C<sub>8</sub>, -TMS, -Phenyl>

- A polar organic solvent (e.g., methanol, acetonitrile, etc.) and an aqueous buffer are commonly used as a mobile phase.

For the separation of ionic substances, appropriate adjustment of pH and/or addition of salts and/or counter ions may result in improved elution times and peak shapes, Recommended are

Salt: Potassium biphosphate

Counter ion (acidic samples) : tetrabutylammonium ions

Counter ion (basic samples) : pentanesulphonic ions

Note: Make sure mobile phase pH is within the specified usable range.

- To elute strongly adsorbed substances more rapidly, employ a stronger non-polar solvent of either an isocratic or binary composition (e.g., addition of tetrahydrofuran to methanol/water brings earlier elution).

<PREP-CN, -NH<sub>2</sub>>

Reversed Phase Applications

- Same as in ODS, etc.

Normal Phase Applications

- n-Hexane, chloroform, etc. are commonly used as mobile phase solvents. To speed up elution, add an increased proportion of iso-propanol or ethanol to mobile phase.
- Before use in normal phase, wash the column with isopropanol as the column is shipped with an aqueous medium prefill. (On washing, maintain the flow rate at less than 2 mL/min to avoid high pressure across the column.

column description	part number	stationary phase	separation mode	guaranteed minimum theoretical plates	shipping and storage solvent prefill
Shim-pack PREP-SIL	228-00814-91	silica	adsorption	4,000	n-Hexane 90/Ethanol 10
Shim-pack PREP-ODS	228-00815-91	octadecyl group	reversed phase	5,000	85% Methanol
Shim-pack PREP-C <sub>8</sub>	228-00816-91	octyl group	reversed phase	5,000	70% Methanol
Shim-pack PREP-TMS	228-00817-91	trimethyl group	reversed phase	5,000	60% Methanol
Shim-pack PREP-CN	228-00818-91	cyanopropyl group	reversed phase (normal phase)	5,000	60% Methanol
Shim-pack PREP-Phenyl	228-00819-91	phenyl group	reversed phase	5,000	60% Methanol
Shim-pack PREP-NH <sub>2</sub>	228-17879-91	aminopropyl group	reversed phase (normal phase) (ion exchange)	4,000	65% Acetonitrile

Also available: The PREP(H) series with 5μm packings.

Ion Exchange Applications [PREP-NH<sub>2</sub>]

- A mixture of phosphate buffer and organic solvents, such as methanol or acetonitrile, is commonly used.

<PREP-SIL>

- The column is employed in normal phase separation.
- n-Hexane, chloroform, etc. are commonly used as mobile phase solvents. To speed up elution, add an increased proportion of isopropanol or ethanol to the mobile phase.

4. Maintenance of Column

<General Precautions>

- For long term storage wash the column and fill it with appropriate solvent followed by plugs to seal the column.
- If an abnormally high pressure is observed disconnect the column and observe pressure drop indicating the column is at fault. In many cases, plugging occurs at the inlet filter (Filters of inlet and outlet are 2μm). Wash the filters in a ultrasonic bath after disassembly of the column ends.
- For washing, set flow rate at 2 mL/min or less to avoid over pressure.

<PREP-ODS, -C<sub>8</sub>, -TMS, -Phenyl>

- After use, wash with methanol then store with plugged ends to prevent evaporation.
- If salts were added to the mobile phase, first wash with water sufficiently, then store as described above.
- However, if amines, such as trimethylamine or tetrabutyl ammonium, were added to the mobile phase, wash with a mixture of methanol 50/0.05% phosphoric acid 50, then with methanol. Do not pump with water only.
- For removing adsorbed matter from the column top, use a wash solvent having strong dissolving power such as tetrahydrofuran or chloroform, etc.

<PREP-CN, -NH<sub>2</sub>>

In the Case of Reversed Phase

- Same as in the case of PREP-ODS, etc.

In the Case of Normal Phase

- Store filled with the ordinal normal phase solvent.
- For removing adsorbed polar matter from the column top, wash with isopropanol, etc.

- In case of sugar analysis by PREP-NH<sub>2</sub>, wash with a mixture of acetonitrile 50/water 50 and store with a mixture of acetonitrile 70/water 30.

In the Case of Anion Exchange

- Wash out the salt from the mobile phase by a water/ acetonitrile mixture (same composition as mobile phase, but no salt), then store with a mixture of water 30/acetonitrile 70.

<PREP-SIL>

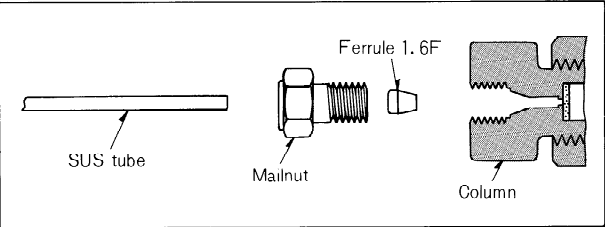
- Store with the same solvent as the mobile phase.
- For removing adsorbed polar matter from the column top, wash with isopropanol, etc.

5. Column Connection

- For installing the column with stainless steel piping, the following connection parts are required.

description	part number	pieces (required)
Mailnut 1.6MN	228-16001	2
Ferrule 1.6F	228-16000	2

- Connect as in the Figure.



Do not tighten the joints excessively!

6. Column Performance

Column test report and chromatogram are included. Guaranteed efficiency in terms of theoretical plate number is shown in the table.

The contents of this catalog are subject to change without notice.