Agilent Prep-Sil Columns

Datasheet

General Description

Agilent Prep-Sil columns are packed with an ultrapure (Type B) silica with high specific surface area (400 m^2/gram) and 100Å pore size in two particle sizes – 5-microns and 10-microns. The Agilent Prep-Sil columns have high sample loadability and give symmetrical peaks even for strongly basic compounds. The columns are stable at pH 0.8-8. Agilent Prep-Sil is especially useful for the preparative purification of acidic, basic, and other highly polar compounds by normal-phase liquid chromatography. Columns are loaded to a stable, uniform bed density using a proprietary, high-pressure slurry-loading technique to give maximum column efficiency and maintain column bed stability.

Column Characteristics

The Agilent Prep-Sil columns have two formats: cartridge format for 21.2 mm ID columns and conventional fixed end-fitting formats for 30 and 50 mm ID columns. For the cartridge columns, a special fitting kit is needed to connect the columns with an instrument. The kit can be ordered from Agilent and the Agilent P/N is 820400-901. The nominal average particle size of the packings used for Agilent Prep-Sil columns is either 5-microns or 10-microns with each column length and particle size combination chosen to produce columns with high separation performance and low operation pressures. In general, in order to obtain equivalent separation on a preparative column, relative to those obtained using analytical column of the same packing, the mobile phase flow rate must be adjusted proportional to the square of the ratio of the column internal diameters (see Table 1).

All larger-diameter columns (20 mm or greater) are susceptible to cross-sectional thermal gradients where the interior core of the column becomes warmer during use compared to the area of the column near the column wall. This thermal gradient, while small during room-temperature operation, can cause observable band-broadening when the column is operated under non-overload sample conditions. When operating the column using typical preparative sample-overload conditions, these temperature effects are seldom important and can be ignored in most cases. The cross-sectional temperature variations are caused by the frictional heating of the mobile phase as it is forced under pressure through the packed bed of the column and the small-diameter tubing of the instrumentation. The heat near the column walls is more easily dissipated through the heat-conductive steel walls of the column while the heat in the center of the column is insulated by the relatively non-heat-conductive silica packing material. This thermal band-broadening increases if the column is being operated at temperatures higher than ambient. To avoid thermal effects, it is recommended that large-diameter columns, especially the higher mass 30 and 50 mm ID x 250 mm long columns, be heated in a water bath at 30°C or higher, if desired. It is important to also maintain the mobile phase at the same temperature as the column to avoid thermal mis-matches which may result in distorted peaks.

The packings used in the Agilent Prep-Sil columns are produced using the same technology for 5µm and 10 µm particles. The same thorough quality control procedures are used to monitor all Agilent Prep-Sil products, including the measurement of surface area, pore size, and particle size of the base silica packing. Sensitive chromatographic tests are also performed on all packings to confirm lot-to-lot reproducibility. This technology permits the direct scale-up of separations from analytical to preparative proportions with little or no modifications required in methodology.

Table 1
Typical Sample Capacities

Column ID	Normalized Flow Rate	Separation Type (small molecules)	
		Easy (alpha > 1.5)	Difficult (alpha=1.2-1.5)
4.6 mm	1.0 mL/min	3-15mg	0.5-3 mg
21.2 mm	20 mL/min	70-400 mg	20-70 mg
30 mm	40 mL/min	140-800 mg	40-140 mg
50 mm	100 mL/min	400-2000 mg	100-400 mg

Safety Considerations

The following points with respect to the safe use of preparative columns should be considered:

- Because of the larger volumes of mobile phase used with preparative columns, special awareness of solvent toxicity and flammability hazards is recommended.
- Maximum operating pressure limit for Preparative Columns is 340 bar (5000psi). Since liquid chromatographic columns are totally hydraulic in nature, little stored energy is present in these columns during use. Should a column be over-pressurized and a tubing or fitting failure occur, the major result will be a large flow leak of mobile phase. Special caution is required in this regard for flammable or toxic solvents.

Operational Guidelines

- The direction of flow is marked on the column.
- While not harmful to the column, reversing flow should be avoided except to attempt removal of inlet pluggage.
- Agilent Prep-Sil columns are shipped containing hexane. Care should be taken not to pass any mobile phase through the column that might cause a precipitate.
- Agilent Prep-Sil is compatible with water and all common organic solvents.
- The use of a guard column is recommended to protect the Agilent Prep-Sil column and extend its useful lifetime. Avoid use of this column below pH 0.8 or above pH 8. Maximum operating pressure is 340 bar.
- Maximum operating temperature of unbonded silica columns is typically limited only by the temperature limits of the mobile phase.

Preparative Strategies

A detailed discussion of how to conduct preparative liquid chromatography is beyond the scope of this data sheet. However, a few helpful guidelines can be given.

- Prior to initial start-up of the preparative column or for start-up after prolonged storage (e.g., greater than 5 days), it is recommended that the column be pre-flushed with 10 column volumes of 100% organic solvent (e.g. Methanol or Acetonitrile) followed by at least 10 column volumes of mobile phase before use to elute potential contaminations.
- Use larger sample volumes of dilute solutions to avoid column overload at the inlet. However, sample volume generally should not exceed one-third the volume of the earliest eluting peak of interest.
- Method development is best accomplished by employing analytical-scale HPLC techniques. Once the optimum mobile phase/stationary phase system has been established using these approaches, the separation can be scaled up to the preparative system with only minor adjustments.
- To prevent the deposition of strongly retained sample components on the preparative column, precautions such as sample filtration and pre-fractionation of the sample using gravity-feed chromatography columns, re-crystallization, distillation, etc., should be taken to maximize column life and sample throughput. Use of a guard column is highly recommended.
- The interested reader is referred to the book "Preparative Chromatography" B.A. Bidlingmeyer, ed., Elsevier Publishing (Volume 38 in the "Journal of Chromatography Library Science Series") for a good compendium on strategies for successful preparative separations.

Storage Recommendations

To avoid potential metal corrosion, long-term storage of any HPLC column in halogenated solvents (e.g., butyl chloride, methylene chloride, etc.) should be avoided. If the column has been used with a buffered mobile phase, the column should be purged with 10-20 column volumes of a mixture of acetonitrile and water followed by 20-30 column volumes of the pure organic solvent. Storage of unbonded silica columns in most other liquids is typically acceptable. Before storing the column, the end-fittings should be tightly capped with the original caps or end-plugs used for shipping the columns to prevent contamination or damage to the threaded column ends.

Agilent Ordering Information

For more information or to order our products, visit our Agilent Technologies home page on the World Wide Web at: www.Agilent.com/chem/supplies
For Technical Support in the US and Canada, call 1-800-227-9770 or call your local Agilent sales office.

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