

Agilent PolarGel Organic GPC Columns

Data Sheet

Installation

Stainless steel tubing of 1/16 in od and 0.010 in id or 0.007 in id is recommended for column connections of analytical columns. Connecting tubing lengths between columns, detectors, and injection volumes should be minimized to avoid excessive dead volume that will diminish system performance. Column connections should be made using Parker compatible 1/16 in nuts and ferrules. The compatibility of column connectors is illustrated in Figure 1.



Figure 1. Compatible connectors.

The distance "x" for the standard column end fitting is 0.090 in and a minimum male nut length of 0.210 in is required. Some fittings from other manufacturers may not be compatible, for example, Waters and Rheodyne. If unsure, please contact Agilent for further advice.

Column connection

Connect the GPC column in the eluent flow direction indicated and tighten the 1/16 in nut and ferrule using wrenches on the 1/16 in nut and the actual end fitting.

It is recommended that several drops of eluent have been pumped before the column outlet is connected to another column or detector to clean out the end fitting of any particulate matter which may be present.

To avoid loosening the end fittings and causing leaks, wrenches must be used on the end fitting adjacent to the connecting nut and NOT on the column barrel or the opposite end fitting, see Figure 2.



Figure 2. Don't use wrenches on the flats.

Eluent Flow Rate

For PolarGel columns using 7.5 mm id columns, 1.0 mL/min is an optimum flow rate for most separations. The recommended flow rate range is given in Table 1, however, higher viscosity eluents should be used at reduced flow rates or elevated temperature. Flow rates should be changed progressively and pressure pulses limited.

At no time should the maximum operating pressure of the column be exceeded (see Table 3).

Table 1. Recommended Flow Rate

Column type	Typical flow rates (mL/min)	Recommended flow rate (mL/min)
PolarGel 7.5 mm id	0.5–1.5	1.0

Sample Preparation and Injection

If maximum resolution and expected column lifetime are to be achieved, care must be taken in sample preparation.

To avoid blockage of the column frits, sample filtration is recommended (0.5–2.0 μ m depending on MW). A guard column will further protect the columns with little detrimental effect on performance.

Optimum sample volumes and concentrations are best determined for each type of analysis and are dependent on sample MW. Broad distribution polymers can generally be injected at higher concentrations than lower polydispersity samples. Overloading will not damage the column, but distorted peaks and spurious results will be obtained.

Excessive injector loop volume will contribute to band broadening and reduce system performance. Agilent's injection volume recommendation is shown in Table 2.

Table 2. Injection Volume Recommendation

Column type	Recommended concentration (%)	Recommended injection (µL) per column
PolarGel 7.5 mm id	0.05–0.50	20–50



Eluents

PolarGel columns are compatible with an extensive range of organic solvents and with aqueous based eluents. Mixed organic solvent systems can also be used, assuming full miscibility of the components. Care must be taken when using solvent mixtures to ensure that a thoroughly mixed solution is employed and that the column pressure does not exceed the recommended maximum for the column (see Table 3). All eluents should be of high purity and should be filtered and degassed prior to use. PolarGel columns are normally supplied in water + 0.02% sodium azide, unless otherwise stated.

PolarGel columns can be transferred to other eluents with no deterioration in performance, as long as care is taken in the transfer procedure. When transferring to another eluent, miscibility and viscosity of the new eluent are of primary consideration. It should also be noted that if the two eluents employed in the transfer are similar in polarity, then the risk of damage to the column during transfer is minimized. Care should be taken not to transfer directly between solvents of very different polarities, or of poor miscibility.

The eluent transfer guide for PolarGel 7.5 mm id columns is shown in Figure 3. Some viscous eluents and solvent mixtures require heating to reduce the column pressure. Care must be taken when heating solvents to ensure no risk of boiling occurs, especially mixtures that may form azeotropes at certain temperatures.

When heating or cooling columns in high viscosity eluents (for example, water, DMSO, NMP, or DMF) a low solvent flow rate must always be maintained. Typically 0.2 mL/min should be used prior to raising the temperature. See Table 3.



Figure 3. Eluent transfer guide for PolarGel 7.5 mm id columns.

Column Testing and Specifications

Every PolarGel column is supplied with a test certificate indicating the test conditions and the column performance. Measurements of column performance are described below:

Efficiency (1/2 ht) (Plates/m)
$$N = 5.54 \left(\frac{t}{W_{1/2}}\right)^2/L$$

Efficiency (5 σ) (Plates/m) $N = 25 \left(\frac{t}{W5\sigma}\right)^2/L$

Symmetry = a/b

where t is the peak elution time, $W_{1/2}$ is the peak width at halfpeak height, $W5\sigma$ is the peak width at 4.4% of peak height, L is the column length in meters, and a and b are the peak widths either side of the perpendicular measured at 10% of peak height. Column efficiency is dependent on many experimental factors (system dead volume, eluent, flow rate, test probe, temperature, and so forth) and test results may differ slightly from those quoted on the column certificate due to variability in these parameters. Band broadening effects are more severe when using high efficiency or narrow bore GPC columns. It is vital to ensure that the system dispersion is minimized in order to obtain the full potential of Agilent columns.

Table 3. Column Specifications

Specifications	PolarGel	
Typical operating pressure in water psi (bar) ¹	450 (30)	
Maximum operating pressure psi (bar)	2000 (140)	
Maximum operating temperature °C	80	
Efficiency ppm ¹	> 35,000	

 1 Based on $\rm H_2O$ at 20 °C, PolarGel-M 300 \times 7.5 mm at 1.0 mL/min using propanetriol (glycerol)

Storage

On removing the column from the system, the end plugs must be replaced to prevent the column from drying out by evaporation, since subsequent shrinkage of the gel and disruption of the packing will occur. The end plugs need only be applied finger tight. All eluents mentioned previously are suitable for storage, but unstabilized THF should not be used.

Warranty

PolarGel columns are covered by warranty for 90 days following delivery. Agilent cannot accept liability from improper handling and use of columns above their maximum operation temperature and pressure (see Table 3) and will void the warranty. For a full warranty statement, please request Agilent's General Conditions of Sale.

Maintenance

Deterioration in column performance may occur as a result of damage to the packed bed or as a result of blockage in the column frits. In the case of frit blockage, the column can be reverse flushed at 1.0 mL/min for 1 minute to remove loosely retained material. If a column blockage exists despite back flushing, then the column inlet frit can be replaced by following the procedure outlined below, or alternatively, the column can be returned to Agilent Technologies.

Frit replacement procedure

- The column should be flushed into water prior to the procedure. Seal the end fitting at the outlet end of the column. Clamp the column vertically with the inlet end uppermost. Undo the end fitting on the inlet end of the column and gently remove it.
- After removing any gel from the end fitting, remove the frit, seal, and spreader with the Agilent frit removal tool, and wash all traces of gel from the end fitting. Then place a new frit, seal, and spreader in the bottom of the end fitting and ensure that they are properly seated.
- Some gel (up to 2 mm) may have expanded out of the column. Using a spatula, place a dome of PolarGel repair gel onto the top of the column and add sufficient water to bind the gel particles together.
- 4. Hold the column at an angle of 45 degrees pointing downwards and replace the end fitting complete with frit, seal, and spreader onto the column. Tighten the end fitting one flat past finger tight.
- Back flush the column with eluent at 0.5 mL/min to remove trapped air. Check for gel particles in the eluent in case the frit has not seated correctly. Return the column to normal flow conditions and check the pressure drop and column efficiency.

Agilent Ordering Information

For more information on our products, visit our web site at www.agilent.com/chem/gpcsec.

www.agilent.com/chem

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