

Agilent PL Rapide Rapid Organic GPC Columns

Data Sheet

Installation

Tubing and connectors

Stainless steel tubing of 1/16 in od and 0.010 in id or 0.007 in id is recommended for column connections. Connecting tubing lengths between columns, detectors, and injection volumes should be minimized to avoid excessive dead volume which 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.

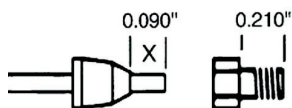


Figure 1. Compatible connectors.

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 your local Agilent office.

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, as illustrated in Figure 2.

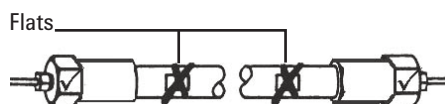


Figure 2. Don't use wrenches on the flats.

Eluent Flow Rate

For conventional GPC columns using 7.5 mm id columns, 1.0 mL/min is an optimum flow rate for most separations. With 10.0 mm id columns a flow rate of 1.8 mL/min is the optimum flow rate. When column id is increased or decreased, the volumetric flow rate should be adjusted accordingly in order to give an equivalent linear velocity through the column.

The recommended flow rates are 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 4).

Table 1. Recommended Flow Rate

Column type	Typical flow rates (mL/min)	Recommended flow rate (mL/min)
PL Rapide 7.5 mm id	0.5–1.5	1.0
PL Rapide 10.0 mm id	2.0–5.0	2.0

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). An in-line filter 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 hence spurious results will be obtained.



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

Table 2. Injection Volume Recommendations

Column type	Recommended concentration (%)	Recommended injection (μL) per column
PL Rapide 7.5 mm id	0.05–0.50	20
PL Rapide 10.0 mm id	0.05–0.50	20

Eluents

PL Rapide gel is compatible with an extensive range of organic solvents having solubility parameters in the range 7–14. Mixed organic solvent systems can also be used, but water should not be used except at concentrations less than 10% by volume in a miscible organic eluent. All eluents should be of high purity and should be filtered and degassed prior to use. PL Rapide columns are normally supplied in ethylbenzene unless otherwise stated, and can be flushed directly from ethylbenzene to THF at 0.5 mL/min. Unstabilized THF (for example, HPLC grade) is not recommended as an eluent due to the attack of peroxide on the gels.

PL Rapide columns can be transferred to other eluents with no deterioration in performance. When transferring to another eluent, miscibility and viscosity of the new eluent are of primary consideration. See Figure 3 for 7.5 mm id columns. For other id columns, apply the flow rate conversion shown in Table 3.

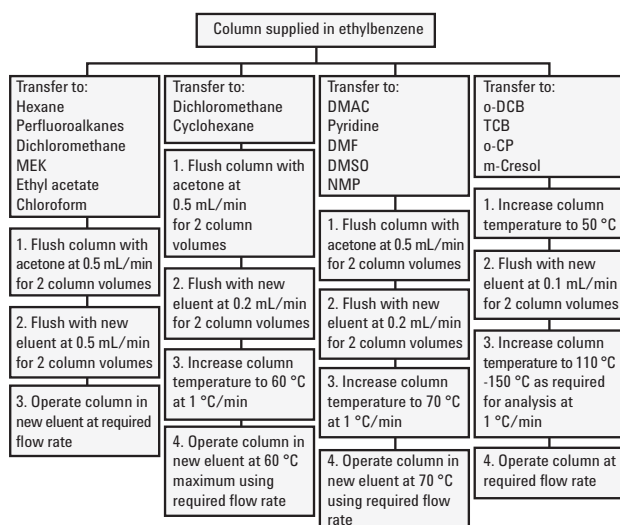


Figure 3. Eluent transfer guide for PL Rapide 7.5 mm id columns.

When heating or cooling columns in high viscosity eluents (for example, TCB, NMP, DMF), a low solvent flow rate must always be maintained. Typically 0.2 mL/min for 7.5 mm id and 0.4 mL/min for 10.0 mm id should be used prior to raising the temperature. A useful stepwise guide is provided in Figure 3.

Table 3. Flow Rate Conversions

PL Rapide 7.5 mm id	PL Rapide 10.0 mm id
0.2 mL/min	0.35 mL/min
0.5 mL/min	0.88 mL/min
1.0 mL/min	1.77 mL/min

Column Testing and Specifications

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

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

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

Symmetry = a/b

where t is the peak elution time, $W_{1/2}$ is the peak width at half peak height, $W_{5\sigma}$ 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, etc.) 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 columns. It is vital to ensure that the system dispersion is minimized in order to obtain the full potential of PL Rapide columns. Column specifications are listed in Table 4.

Table 4. Column Specifications

Specifications	PL Rapide F	PL Rapide L	PL Rapide M	PL Rapide H
Typical operating pressure psi (bar) ¹	350 (25)	225 (15)	75 (5)	75 (5)
Maximum operating pressure psi (bar)	1500 (90)	1100 (75)	1100 (75)	1100 (75)
Maximum operating temperature °C ²	150	150	220	220
Efficiency ppm	> 40,000	> 80,000	> 60,000	> 35,000

¹ Based on THF at 20 °C, PL Rapide 150 × 7.5 mm at 1.0 mL/min, and for PL Rapide 100 × 10.0 mm at 1.8 mL/min using butylated hydroxy toluene (BHT) as a test probe.

² At very high temperatures, column lifetimes are likely to be reduced.

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

The columns are covered by warranty for 90 days following delivery. For columns used at or above 170 °C, the warranty period is reduced to 30 days. Agilent cannot accept liability from improper handling or use. 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. For further technical assistance, or to use the Column Repair Service, please contact your local Agilent Technologies office.

Agilent Ordering Information

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

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