# PlusPore Organic GPC Columns



### **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 of analytical columns and 0.020 in. ID for 25 mm preparative columns. Connecting tubing lengths between columns, detectors and injection volumes should be minimised 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 below.

### Figure 1





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, eg Waters and Rheodyne. If unsure, please contact Polymer Laboratories.

### **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

### Information Installation Maintenance

In order to obtain the longest possible service life of the column, please observe the instructions in this user guide.

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 below.

Figure 2



### **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. 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 3).

### Table 1

Column Type	Typical flow rates (ml/min)	Recommended flow rate (ml/min)
PlusPore 4.6 mm ID	0.2 - 0.5	0.3
PlusPore 7.5 mm ID	0.5 - 1.5	1.0
PlusPore 25 mm ID	5.0 - 20.0	10.0

### SAMPLE PREPARATION AND INJECTION

If maximum resolution and expected column lifetime are to be

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achieved care must be taken in sample preparation.

To avoid blockage of the column frits, sample filtration is recommended (0.5-2.0 $\mu$ m depending on MW). A guard column will further protect the columns with little detrimental effect on performance, a list of those available is provided at the end of this guide.

Optimum sample volumes and concentrations are best determined for each type of analysis and is 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.

Excessive injector loop volume will contribute to band broadening and reduce system performance, particularly with high efficiency or narrow bore columns. PL's injection volume recommendations are shown in Table 2.

Та	b	le	2

Column Type	Recommended concentration (%)	Recommended injection (µl) per column
PolyPore & OligoPore 7.5 mm ID	0.05 - 0.50	20 - 50
PolyPore & OligoPore 4.6 mm ID	0.01 - 0.20	1 - 20
MesoPore & ResiPore 7.5 mm ID	0.05 - 0.50	10 - 20
MesoPore & ResiPore 4.6 mm ID	0.01 - 0.20	1 - 10
Preparative 25 mm ID	0.5 - 5.0	500 - 2000

### ELUENTS

PlusPore gels are 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. PlusPore 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 (eg HPLC grade) is not recommended as an eluent due to the attack of peroxide on the gels.

PlusPore columns can be transferred to other eluents. When transferring to another eluent, miscibility and viscosity of the new eluent are of primary consideration. The following eluent transfer guide is for 7.5 mm ID columns; for other ID columns apply the flow rate conversion shown overleaf.

When heating or cooling columns in high viscosity eluents (eg TCB, NMP, DMF) a low solvent flow rate must always be

maintained. Typically 0.2 ml/min for 7.5 mm ID and 0.1 ml/min for 4.6 mm ID should be used prior to raising the temperature, a useful stepwise guide is provided below.

## ELUENT TRANSFER GUIDE FOR PlusPore 7.5 MM ID COLUMNS



### Flow Rate Conversion

PlusPore 4.6 mm ID	PlusPore 7.5 mm ID	PlusPore 25 mm ID
0.08 ml/min	0.2 ml/min	2.0 ml/min
0.18 ml/min	0.5 ml/min	5.0 ml/min
0.38 ml/min	1.0 ml/min	10.0 ml/min
0.76 ml/min	2.0 ml/min	20.0 ml/min

### COLUMN TESTING AND SPECIFICATIONS

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

L

L

Efficiency (1/2ht) (Plates/m)	N = 5.54	$\left( \begin{array}{c} t \\ W_{1/2} \end{array} \right)^2 /$
Efficiency (5 <del>0</del> ) (Plates/m)	N = 25	$\left(\frac{t}{W5\sigma}\right)^{2}$

Symmetry = a/b

where t is the peak elution time, W1/2 is the peak width at half peak height,  $W5\sigma$  is the peak width at 4.4% of peak height, L is the column length in metres 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 and/ or narrow bore GPC columns. It is vital to ensure that the system dispersion is minimised in order to obtain the full potential of PL columns.

#### Table 3

Specifications	PolyPore 5µm	ResiPore 3µm	MesoPore 3µm	OligoPore 6µm
Typical Operating Pressure psi (bar)1	450 (30)	750 (50)	750 (50)	450 (30)
Maximum Operating Pressure psi (bar)	2200 (150)	2700 (180)	2700 (180)	1500 (100)
Maximum Operating Temperature °C <sup>2</sup>	160	120	120	120
Efficiency <sup>1, 3, 4</sup>	>60,000	>80,000	>80,000	>55,000

<sup>1</sup>Based on THF at 20 °C, PlusPore 300 x 7.5 mm at 1.0 ml/min, PlusPore 250 x 4.6 mm at 0.3 ml/min and PlusPore 300x25 mm at 10 ml/min. <sup>2</sup>At very high temperatures, column lifetimes are likely to be reduced. <sup>3</sup>Using butylated hydroxy toluene (BHT) as a test probe.

<sup>4</sup>PlusPore 250 x 4.6 mm columns may be typically 10,000ppm lower in efficiency due to system dispersion.

### STORAGE

On removing the column from the system the end plugs must be replaced to prevent the column 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 unstabilised THF should not be used.

### WARRANTY

The columns are covered by warranty for 90 days following delivery. Polymer Laboratories, now a part of Varian, Inc., cannot accept liability from improper handling and use of columns above their maximum operation temperature and pressure (Table 3) will void the warranty. For a full warranty statement, please request Polymer Laboratories' 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 the column returned to Polymer Laboratories.

### Frit Replacement Procedure

1. 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.

2. After removing any gel from the end fitting, remove the frit, seal and spreader with the PL 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.

3. Some gel (up to 2 mm) may have expanded out of the column. Using a spatula place a dome of PlusPore repair gel onto the top of the column and add sufficient ethylbenzene to bind the gel particles together.

4. Hold the column at an angle of 45° 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.

5. 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.

It is recommended that preparative columns are returned to Polymer Laboratories for fitment of replacement frits or repair, however, a similar procedure to the above can be applied if necessary. For further technical assistance, or use of the Column Repair Service, please contact Polymer Laboratories, now a part of Varian, Inc.

### COLUMN PARTS AND ACCESSORIES

Parts and Accessories	Part No.
Frit Removal Tool – threaded columns only	PL1310-0001
End Fitting for threaded columns, 7.5 mm ID	PL1310-0004
Frit (2 $\mu$ m) Kit (Pk of 5) for threaded columns, 7.5 mm ID	PL1310-0002
End Fitting for threaded columns, 4.6 mm ID	PL1310-0034
Frit (2µm) Kit (Pk of 5) for 4.6 mm ID	PL1310-0041
Replacement Frit and End Fitting, 25 mm ID	PL1310-0011
Column Connecting Nuts (Pk of 5), 1/16 in. tube	PL1310-0007
Tubing Ferrules (Pk of 5), 1/16 in. tube	PL1310-0008
Column End Plugs (Pk of 10), 1/16 in.	PL1310-0003
LDV Intercolumn SS Connector	PL1310-0005
OligoPore Column Repair Gel	PL1413-0320
MesoPore Column Repair Gel	PL1413-0325
ResiPore Column Repair Gel	PL1413-0300
PolyPore Column Repair Gel	PL1413-0500

# PlusPore Organic GPC Columns

Guard Columns	Part No.
PolyPore Guard, 50 x 7.5 mm	PL1113-1500
PolyPore Guard, 50 x 4.6 mm	PL1513-1500
ResiPore Guard, 50 x 7.5 mm	PL1113-1300
ResiPore Guard, 50 x 4.6 mm	PL1513-1300
MesoPore Guard, 50 x 7.5 mm	PL1113-1325
MesoPore Guard, 50 x 4.6 mm	PL1513-1325
OligoPore Guard, 50 x 7.5 mm	PL1113-1320
OligoPore Guard, 50 x 4.6 mm	PL1513-1320





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