

PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY
PC 01 25/300 DS

User Manual



1. Description and use

PC 01 columns are designed for high pressure, high performance preparative liquid chromatography. They are equipped by moving pistons on both ends. All part in connection with mobile phase are made of 316 (316L on the request) stainless steel according AISI and of ultra high molular polzethylene.

PC 01 columns are used for high performance separations in instances where small rigid particles are used as column filling. Columns are resisting to all common chromatographic solvents.

PC 01 25 columns with inner diameter 25 mm are designed for laboratory scale chromatography and typically are working with flow rate 20 ml/min. – 40 ml/min. depending on sorbent type and separation mode.

PC 01 25 DS columns are designed for dynamic slurry packing. They are equipped with manual high pressure hydraulic system which serves for sorbent slurry packing in dynamic mode under the pressure.

2. Column design

Typically the PC 01 25 (front page, Fig. 1 - cross section) column consists of tube, I.D. 25 mm made of AISI 316 stainless steel. The internal surface of the column is mechanically polished to attain high smoothness. The tube is provided with two stainless steel flanges (tube flanges), each with four holes with M8 threads for the clamping screws.

The upper and bottom parts of the column are closed by stainless steel pistons with UHMWPE made seals. Each piston unit consist of four parts (see Fig. 2):

- porous disc consisting of screen multilayerwith function to distribute the liquid flow
- piston plate with liquid input or output fitting for 1/16" (1,6 mm) capillary
- conical seal with which seals outer tube, inner piston and the frit
- seal support plate which is made of stainless steel on upper side (on bottom side act as a plate bottom column flange itself)

Upper seal support plate is connected to a stainless steel tube which is attached to the hydraulic piston (Fig. 3). Piston seal is attached to the inner conical part of the piston unit and acts as pressure transducer. Its tightness increases when pressure is increased.

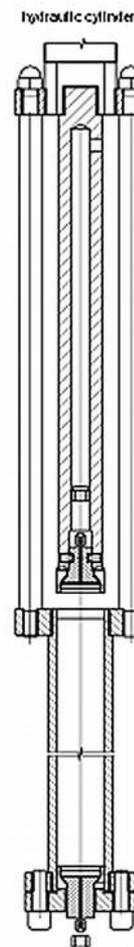


Fig. 1 Column cross section

To the column tube are connected two flanges (column flanges) with holes for connecting bolts. Upper column flange has a thread in which is connected a hydraulic cylinder. The cylinder is designed for higher pressure then is used inside the column (700 bar against 200 bar) and has thus smaller inner diameter than column itself. Bottom column flange has a hole for output fitting

. Column assembling

PC 01 25 columns are usually delivered partially assembled, but here is described full assembling process to allow to the user to replace parts are repair column when necessary.

Upper piston (Fig. 2) with seal and frit is inserted into connecting tube, but before it an input capillary has to be installed (Fig. 3). There are a side screws on bottom side of connecting tube to fix the piston and a thread on upper side for connecting tube to hydraulic piston.

Bottom piston is completed with sealing ring and frit and together with bottom column flange and pressed to column tube (Fig. 4) by connecting bolts M8.

Hydraulic cylinder is connected to upper column flange and *via four* long distance rods to the upper tube flange (Fig. 1). Before it upper piston with connecting tube is screwed to the hydraulic piston. The hydraulic piston has to be in most bottom position.

Fig. 3 Upper piston capillary connection

Input and output capillaries are connected to the sample injector capillary and detector input capillary. Hydraulic pump (Fig. 5) hose is connected to the fast connection on the hydraulic cylinder. Column is fixed in proper stand or equipped with delivered legs. Column is now ready for packing.

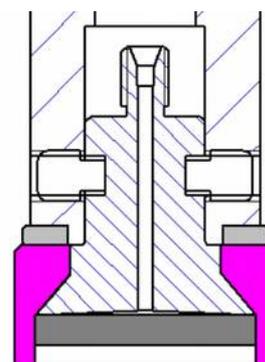


Fig. 2 Upper piston cross section

. Column packing

Column packing procedure has to be accomplished different way. There is either dynamic slurry packing method or a sedimentation method. Delivered column is designed for dynamic slurry method.

Dynamic slurry method needs to use part of column (about a half) for a volume of sorbent slurry. Assembled column has to be equipped on the input and output by a caps or valves. Output capillary has to be inserted into a proper reservoir.

On the start point output cap is closed. A funnel with elastic tube is used to fill the column by a slurry cca 10 mm under the tube edge through a gap between the column and upper piston (see Frint side Fig). The hydraulic pump is use to move piston to the column. It is necessary to take care about column upper piston at this moment – it must be directed precisely to the column tube.

As the first part of the liquid is flowing out of the input capillary, upper cap is closed and bottom is opened. Now the oil is pumped into the oil cylinder to move



Fig. 5 Manual oil pump

the piston to the column fast. Oil pressure is monitored on the manometer not to increase the pressure for which column and hydraulics are designed. Generally for DS column packing is recommended to use pressure 100 – 150, but instruction of sorbent manufacturer is a priority.

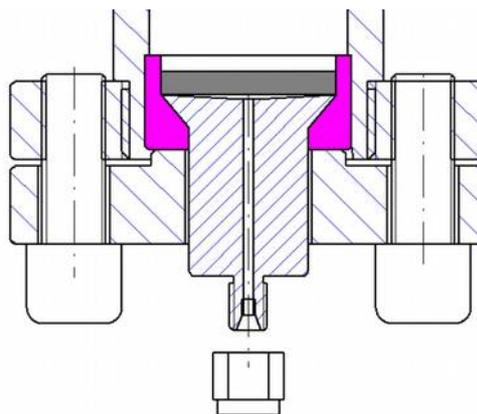


Fig. 4 Bottom piston cross section

The pressure in the column is not equal to the pressure on the oil pump manometer. Column cross section area is 4,9 cm² and hydraulic piston area 7,1 cm². Thus the pressure in the column is 1,4 times higher, than the pressure in the hydraulic cylinder.

When column is fully packed, the pressure of oil starts to increase rapidly. It is necessary to stop oil pumping at this moment. Pressure of oil is going down slowly and due this time column has to be connected to the system. Then oil pressure is increased again to reach approximate value of the working pressure of a mobile phase (after correction) and mobile phase starts to be pumped through the column.

5. Column unpacking

The column output flange is released and bottom piston followed with sorbent is pressed out of the column by mobile phase. Before it is necessary to move column piston to the most upper position where is still tighten. Oil pump valve has to be open before, because column PC 01/25 DS is equipped with one way hydraulic cylinder (back movement is done by a spring with small power). Upper column piston movement has to be done thus by mobile phase pressure from the column because the spring is not strong enough. Usually is not necessary to close column output as piston back movement needs low pressure. Process has to be carefully monitored to stop the pump just at the moment when piston is released and liquid starts to flow out the column upper edge.

6 . Manufacture and servicing:

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