PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY

PC 01 50/250 DS

user manual

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1. Desription 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.
PC 01 columns are used for high performance separations in instances where small rigid particles are used as column filling. Only stainless steel and polytetrafluorethylene (PTFE) are in contact with mobile phase. Columns are resisting to all common chromatographic solvents.
PC 01 50 columns with inner diameter 50 mm are designed for laboratory scale chromatography and typically are working with flow rate 30 ml/min. – 100 ml/min. depending on sorbent type and separation mode.
PC 01 50 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 50 (Fig. 1) column consists of tube, I.D. 50 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 three holes with M12 threads for the clamping screws.
The upper and bottom parts of the column are closed by stainless steel pistons with polytetrafluorethylene (PTFE) and polypropylene (PP) made seals. Each piston unit consist of five parts (see Fig. 2):

- porous disc consisting of screen multilayer have important function to distribute the liquid and form a piston flow through the column is fixed in frit ring with a large thread for piston connecting
- piston plate with liquid input or output fitting for 1/8" (3,3 mm) capillary and large outer thread which is to be screwed to a frit ring
- a stainless-steel net ring for liquid distribution inserted between a frit and a piston plate
- PTFE conical seal with which seals both outer tube and inner piston thread
- support plate which is a) made of stainless steel and connected to a stainless steel tube which is attached to the hydraulic piston on the upper side b) made of PP on bottom side and inserted between bottom flange and piston body

PTFE and PP piston seals are attached to the inner conical part of the piston unit and acts as pressure transducer. Its tightness increases when pressure is increased.
Porous disc covers nearly all tube cross section. This eliminates sorbent areas without full liquid flow. Net layer support the frit and make impossible a deformation under sorbent pressure.
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 diameter than column itself.
Bottom column flange has a hole for output fitting and three side threads for stand legs. They are hooked in 120 ° angle and equipped with plastic balls on their ends.

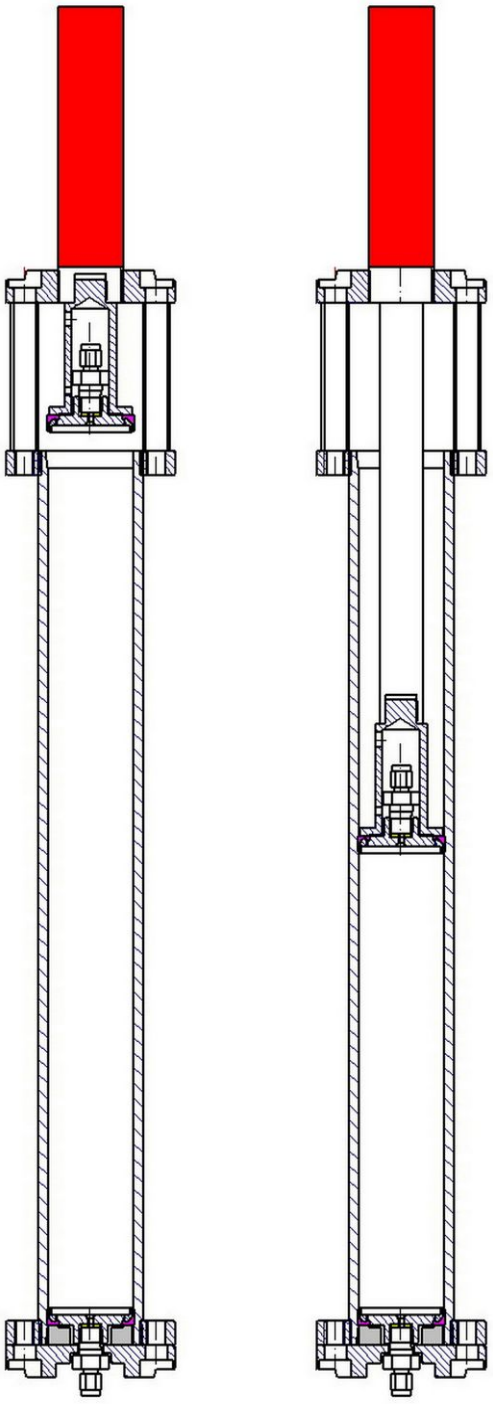

Fig. 1 Column cross section

3. Column assembling

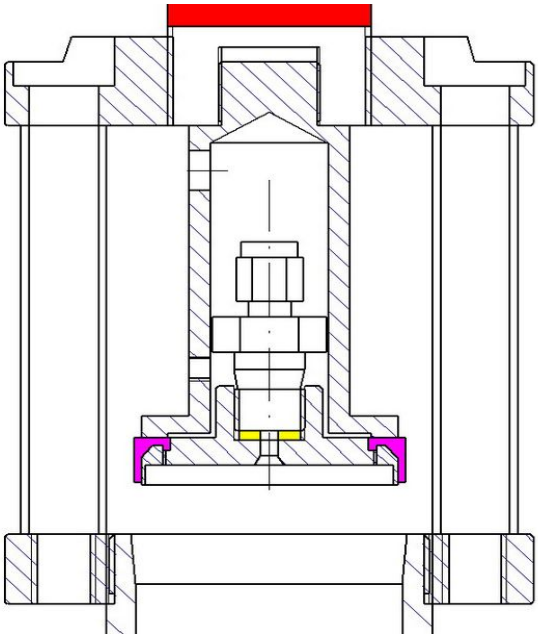

Fig. 2 Piston cross section

PC 01 50 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.

Piston assembling starts with distributor net inserting into the frit ring (see Fig. 3). The piston thread is covered by thin layer of PTFE tape before it is is screwed to the ring and tighten.

One piston is completed with sealing ring, PP support plate, bottom column flange and output fitting (Fig. 4). Then the unit is inserted into column tube and pressed inside by proper bolts (Fig. 5). Finally pressing bolts are replaced by regular bottom column flange bolts and output fitting is tightened to seal.

Upper piston has to be connected to input fitting. The unit is inserted into connecting tube, but before it an input capillary has to be installed (Fig. 6). There is a central thread on upper side of connecting tube to connect the column piston and a hydraulic piston, but before it is done, the column has to be equipped with legs and hydraulic cylinder itself.

Column is connected to stand legs which are screwed to side holes of bottom tube flange and tightened. Legs has to be properly oriented and thus there are marks on both legs and flange (Fig. 7). Legs are tightened to proper position. It is recommended to use proper adhesive to fix legs bolts in the position.

Fig. 3 Frit ring

Hydraulic cylinder is connected with upper column flange and connected *via* two long bolts with distance tube to the upper tube flange (Fig. 8). There is a hole on the place of third bolt.

Upper piston unit is connected to the connecting tube and fixed by three small bolts M6 (only slightly tighten to allow the piston move and rotate). Through a mentioned hole is piston inserted above the column tube and connected to the hydraulic cylinder piston. Input pipeline (3,3 mm O.D.) is to be installed before tis operation.

Third bolt with connection tube is added to fix upper column flange. All bolts are tightened properly. Finally hydraulic cylinder is connected to the oil pump Oil pump valve (Fig. 9) has to be closed. Column is now ready for packing.

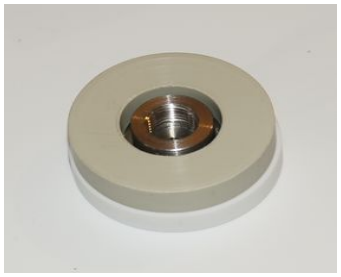

Fig. 4 Bottom piston + support

4. Column packing

Column packing procedure has to be accomplished different way. There is either dynamic slurry packing method or a sedimentation method. Both methods are working with sorbent which is mixed with proper solvent to form so called „slurry". General dynamic slurry method is described here, but each user has to follows sorbent manufacturer instruction.

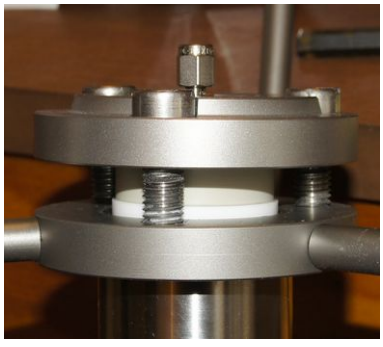

Fig. 5 Bottom piston inserting

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 inserted into a proper reservoir. 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 Fig. 11).

The hydraulic pump is use to move piston to the column. As the first part of the liquid is flowing out of the input capillary, upper cap is

Fig. 6 Capillary input

closed and bottom is opened. Now the oil pi pumped into the oil cylinder to move 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.

The pressure in the column is not equal to the pressure on the oil pump manometer. Column crossection area is 19,6 cm^2 and hydraulic piston area only 7,1 cm^2. Thus the pressure in the column is 2,8 times lower, that the pressure in the hydraulic cylinder.

In other words, having on the oil manometer 280 bar, there is 100 bar inside the column. For the column packing is recommended to use oil pressure 200 – 400 bar. When column is fully packed, the pressure start 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.

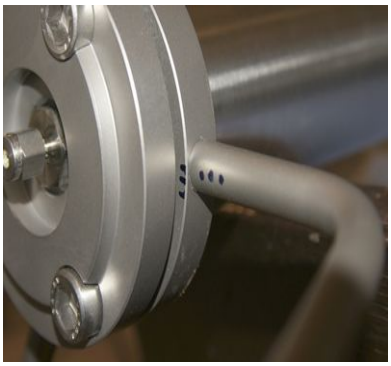

Fig. 7 Leg marks

Fig. 8 Upper piston position

5. Column unpacking

The column output flange is released and sorbent is pressed out of the column by mobile phase. Before it is done, it is necessary to move column piston to the most upper position where is still tighten. Oil pump valve has to be open before.

Fig. 9 Oil valve

The column PC 01/50 DS is equipped with one way hydraulic cylinder (back movement is done by a spring). 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.Last few mimimeters moves the piston due the hydraulic cylinder spring power.

6 . Manufacture and servicing:

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