

PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY

PC 01 100/500 DS

*user
manual*



1. Description and use

SEPARCHROM PC 01 columns are designed for high pressure, high performance preparative liquid chromatography. They are equipped by pistons on both ends. All metal parts in connection with mobile phase are made of AISI 316 (316L) stainless steel.

PC 01 columns are used for high performance separations in instances where small rigid particles are used as column filling. Only stainless steel and UHMWPE (ultra high molecular weight polyethylene) are in contact with mobile phase. Columns are resisting to all common solvents.

PC 01 100 columns with inner diameter 100 mm are designed for both laboratory and industrial separations and typically are working with flow rate 120 ml/min. – 400 ml/min. depending on sorbent type and separation mode. Maximal pressure in these columns is 200 bar.

PC 01 100 DS columns are designed for dynamic slurry packing. They are equipped with manual high pressure hydraulic system which serves for sorbent slurry pressing in dynamic mode of packing and during the use compensates bed volume changes. .

2. Column design

Typically the **PC 01 100** (Fig. 1) column consists of tube, I.D. 100 mm. 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 10 holes with M12 threads for the clamping screws.

The upper and bottom parts of the column are closed by stainless steel pistons with UHMWPE and polypropylene (PP) made seals. Each piston consist of five parts (see Fig. 2, 3, 4, 5):

- porous disc (frit) made of Poremet 2 material consisting of 7 screen layers have important function to distribute the liquid and form a piston flow through the column; it is fixed in frit ring with a large thread for piston plate connecting
- stainless-steel net rings for liquid distribution inserted between a frit and a piston plate
- 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
- UHMWPE conical seal with which seals both outer tube and inner piston thread
- support plate which is a) made of stainless steel and PP and connected to a stainless steel tube attached to the hydraulic piston

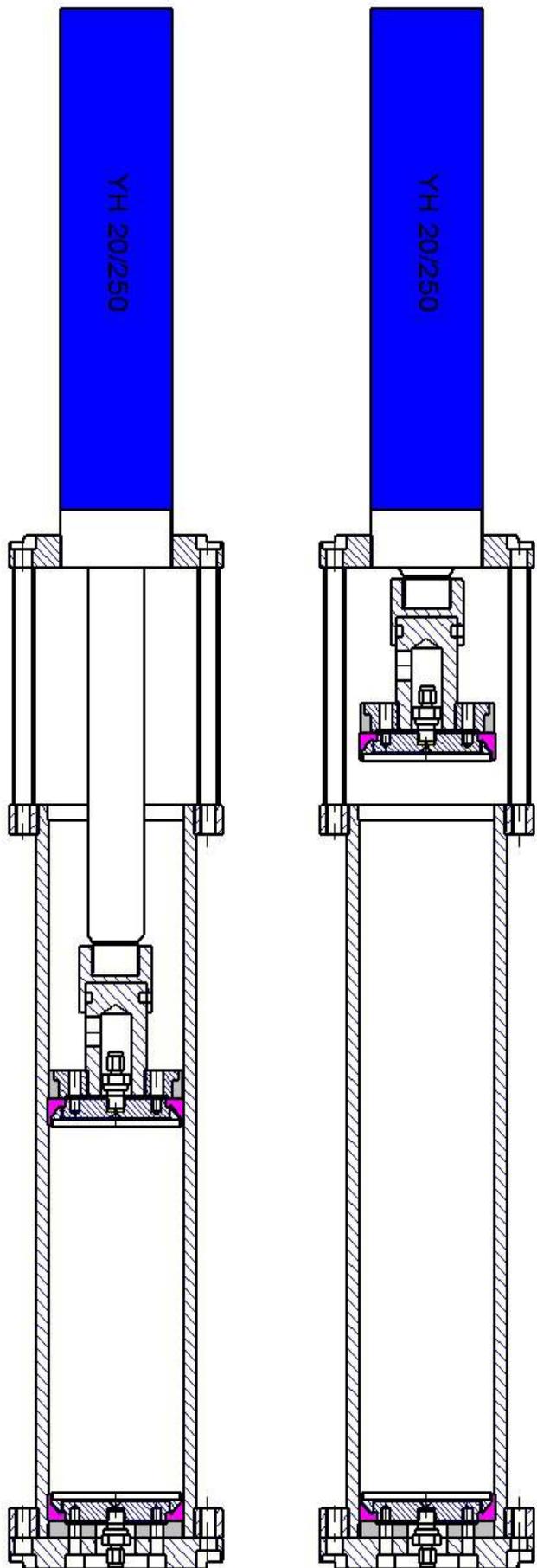


Fig. 1 Column cross section

on the upper side b) made of PP on bottom side and inserted between bottom flange and piston unit

UHMWPE and PP piston seal parts are attached to the inner conical part of the piston unit and acts as pressure transducer. The tightness thus increases when pressure is increased.

Porous frit discs cover nearly all tube cross section. This eliminates sorbent bed areas without full liquid flow. Net layers distribute liquid to the full column cross-section, support the frit and make impossible a deformation under sorbent pressure.

As written, to the column tube are fixed tube flanges. There are two column flanges too. Upper one with a central thread for a hydraulic cylinder. The cylinder is designed for higher pressure then is used inside the column (700 bar against 150 bar) and has thus smaller diameter than column itself.

Bottom column flange has a central hole for output fitting and three side threads for stand legs. Legs are made of stainless steel tubes O.D. 16 mm with a inner and outer threads on ends. Legs are hooked in 120 ° angle and equipped with a plastic (PP) plate on the bottom side. Legs are going trough and have PP rings on their ends.

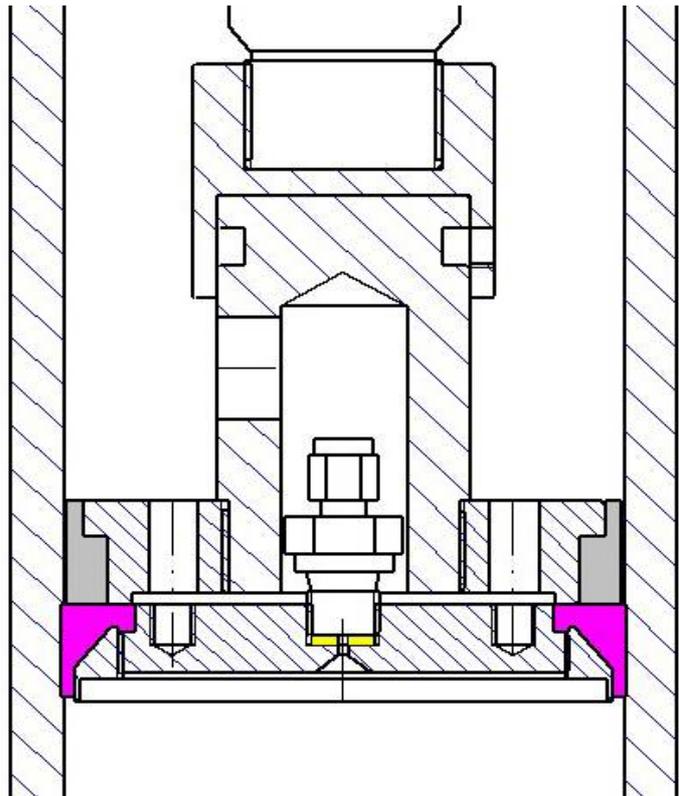


Fig. 2 Upper piston cross section

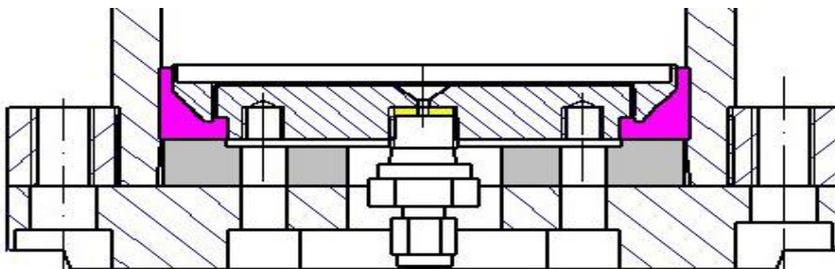


Fig. 3 Bottom piston cross section

3. Column assembling

PC 01 100 columns are usually delivered partially assembled, but here is described full assembling process to allow to the user to replace parts or repair column when necessary (in italics).

Piston assembling starts with distributor net inserting into the frit ring (see Fig. 4). The piston thread can be covered by thin layer of PTFE tape before it is screwed to the ring and tighten. Sealing ring is now fixed to the completed bottom piston followed by PP support plate by bottom column flange that is connected to the piston by 6 M8 bolts. An output fitting is assembled then.

Note: bolts M8 must not be fully tightened in the moment when piston is inserted into the tube. The unit is inserted into column tube and pressed



Fig. 4 Frit ring with distributor nets



Fig. 5 Upper piston with seal rings

install the connecting capillary. 8 bolts M8 has to be removed to have approach to the fitting. A capillary (best is for example PEEK one) is fixed on the fitting and got through a side hole on the connecting piece (the capillary has to be long enough to allow dynamic slurry packing). When the capillary is installed, M8 bolts are used to connect both pieces again. Bolts must not be fully tightened in the moment when piston is inserted into the tube. -

Column tube with bottom flange is completed by stand legs which are screwed to side holes of bottom tube flange and fix with nuts in proper position (each leg is delivered as a complete unit with fixing nut, M14 bottom bolt and a plastic ring). Stand plate which is added have three holes for legs. Leg bottom parts are equipped with M14 threads. The plate is then fixed with M14 bolts and plastic rings (see Fig. 7).

Hydraulic cylinder is carefully screwed to the upper

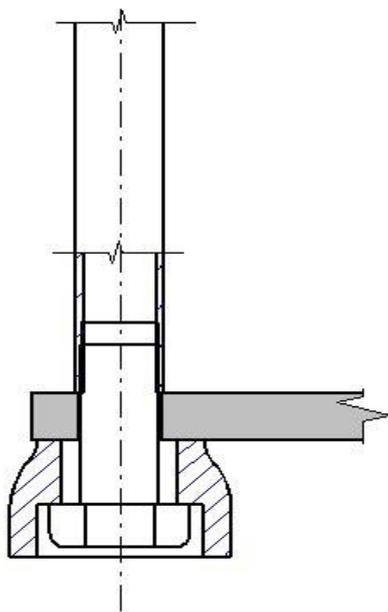


Fig. 7 Column leg on the plate (cross section)

inside by bolts of proper length. Finally pressing bolts are replaced by regular bottom column flange bolts. Piston without plastic ring can be pressed into the column first, if only regular bolts are available. Than bolts are removed, ring added and pressed once more,

Upper piston is assembled the same way like the bottom one including the connection of input fitting. Sealing UHMWPE ring is then fixed on followed by upper piston plate with PP ring (Fig. 5) which are fixed by six bolts M8. On the piston unit is screwed a connecting piece 1 (Fig. 6).

An input capillary has to be installed before the piston is fully assembled. In case the piston is delivered fully assembled, it has to be deassembled partially to

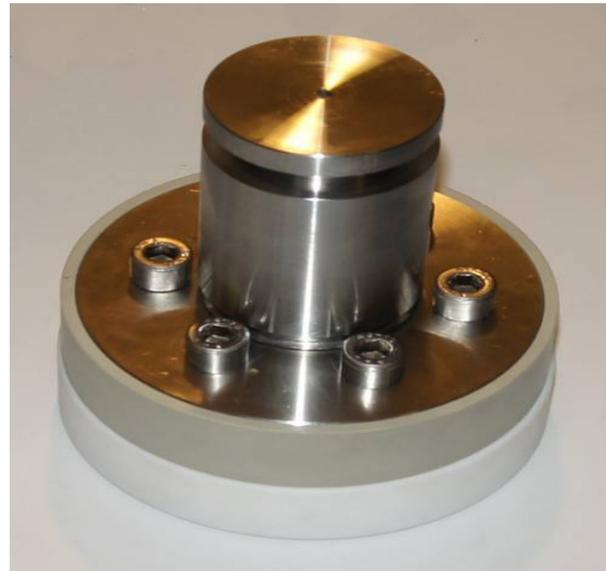


Fig. 6 Upper piston with support plate and connecting piece 1

column flange, equipped before with a connecting piece 2. This piece has a thread that fits to the hydraulic piston front flat part (delivered assembled from the factory). The hydraulic piston has to be in the most back position.

Flange unit with hydraulic cylinder is connected *via* long bolts M12 with distance tubes (3 pcs in triangle) to the upper tube flange (Fig. 8). There has to be one broader side hole remaining for the piston installation. Finally hydraulic cylinder is connected to the oil pump with connecting hoses such way, that upper cylinder input is connected to the output hole on the pump body with T piece and manometer and bottom input is connected to the second pump output. Oil pump valve (Fig. 9) is shifted to the most right position in which oil is pressed to move piston down (middle position is neutral, both ways opened, left position is to press the piston up).

Through mentioned hole is column upper piston unit inserted into the edge of the column tube. Then it is connected to the connecting piece 2 and fixed by three small side bolts M8 (only slightly tighten to allow the piston move and rotate). Piston movement is proved and its central position with respect to the tube hole is controlled.

Other bolts are added now to fix upper column flange. All bolts are tightened properly.



Fig. 8 Upper piston inserted on its position, connecting pieces fixed

4. Column packing

Column packing procedure has to be accomplished different ways. 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 follow sorbent manufacturer instruction about packing.

Dynamic slurry method needs to use part of column (about a half) for a volume of sorbent slurry and piston movement. 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. Output cap is closed. A funnel with elastic tube is used to fill the column by a slurry cca 15 mm under the tube edge through a gap between the column and upper piston (see Fig. 8).

The hydraulic pump is used to move piston to the column. As the first part of the liquid is flowing out from the input capillary, upper cap is closed and bottom is opened. Now the oil is as fast as possible pumped into the oil cylinder to move the piston into the column down.

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 cross-section area is 78,8 cm² and hydraulic piston area only 28,6 cm². Thus the pressure in the column is 2,8 times lower, than 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 – 300 bar.

When column is fully packed, the oil pressure 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.



Fig. 9 Oil pump head with switching valve

5. Column unpacking

The column output flange is released and sorbent is pressed out of the upper piston movement generated by hydraulic piston. Before it is done, it is necessary to release bolts of connecting pieces. When the piston is moved to the most bottom position, oil valve is switched and oil piston with connecting piece 2 is moved most up. An elongation rod (is not a part of standard delivery) is inserted and oil piston is again moved down. Then is again moved up and second rod is inserted. The piston is moved down again to press out all sorbent and upper piston unit as well.

6. Notes

Maximal temperature for column use is 80 °C.

Piston sealing is to be changed when liquid leaks from the column (average frequency of the change is after 10 packing/unpacking cycles)..

For manipulation with the column has to be used special hanger connected to the thread on the top of hydraulic cylinder.

7 . Manufacture and servicing:

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