separlab

PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY

# PC 01 100/250 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 pistons on both ends. All part in connection with mobile phase are made of AISI 316 (316L on the request) 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 polytetrafluorethylene (PTFE) are in contact with mobile phase. Columns are resisting to all common chromatographic solvents.
**PC 01 100** columns with inner diameter 100 mm are designed for semiindustrials separations and typically are working with flow rate 120 ml/min. – 400 ml/min. depending on sorbent type and separation mode.
**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.

## 2. Column design

Typically the **PC 01 100** (Fig. 1) column consists of tube, I.D. 100 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 12 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, 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 connecting
- ➔ a stainless-steel nets ring 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
- ➔ PTFE conical seal with which seals both outer tube and inner piston thread
- ➔ support plate which is a) made of stainless steel and polypropylene and connected to a stainless steel tube attached to the hydraulic piston on the upper side b) made of PP on bottom side and inserted between bottom flange and piston unit

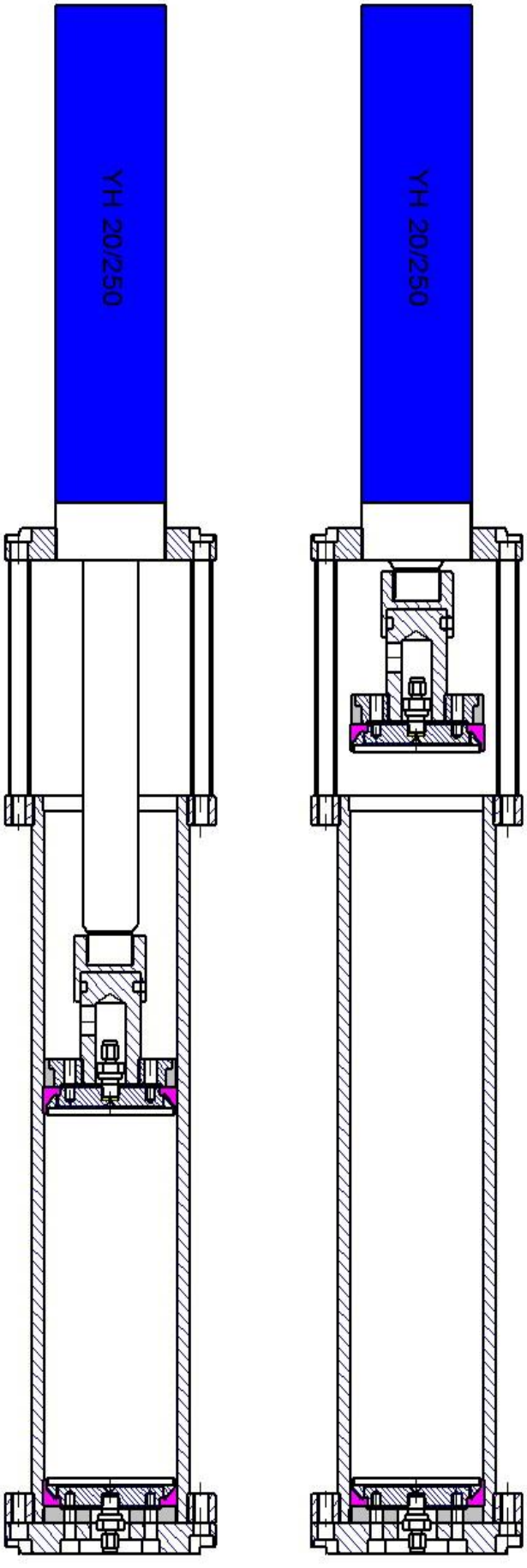


***Fig. 1 Column cross section***

PTFE and PP piston seal parts are attached to the inner conical part of the piston unit and acts as pressure transducer. The tightness 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.
To the column tube are connected two flanges (tube flanges) having thread holes for connecting bolts. There are two column flanges too. Upper column flange has a central thread in which is connected 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 hooked in 120 ° angle and equipped with a plastic (PP) plate on the bottom side. Legs are going trough and have balls on their ends.

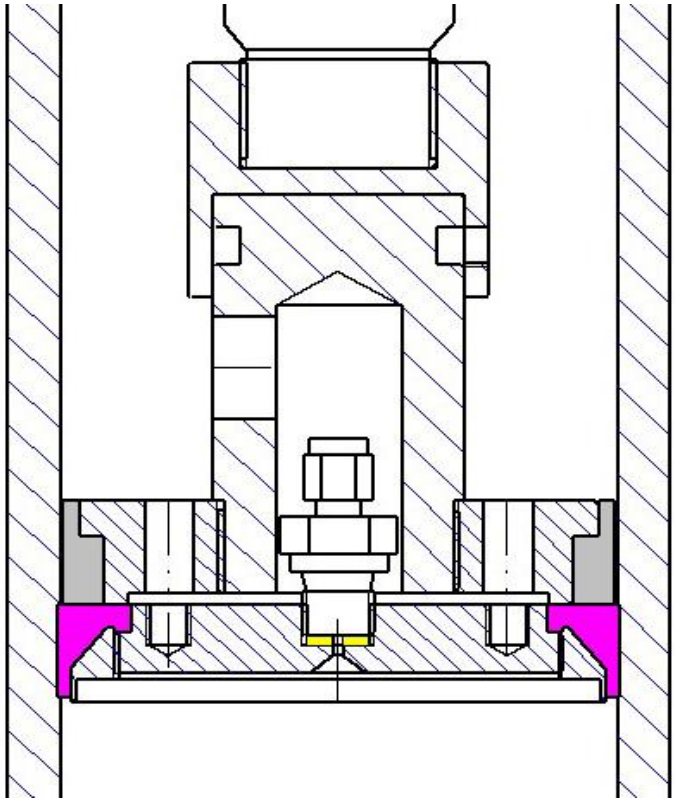

*Fig. 2 Upper piston cross section*

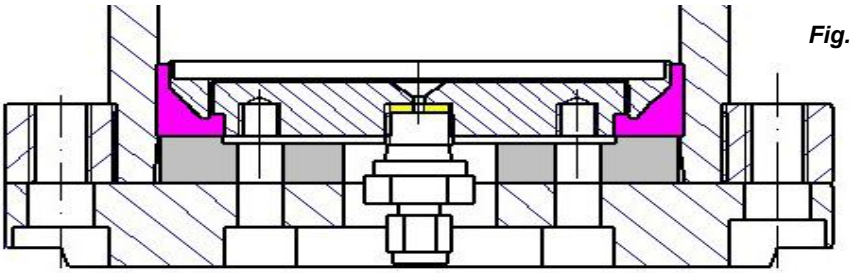

*Fig. 3 Bottom piston cross section*

## 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 are repair column when necessary.
Piston assembling starts with distributor net inserting into the frit ring (see Fig. 4). The piston thread is to be covered by thin layer of PTFE tape before it is screwed to the ring and tighten.
PTFE 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. The unit is inserted into column tube and pressed inside by proper bolts. Finally pressing bolts are replaced by regular bottom column flange bolts. Upper piston has to be connected to input fitting firstly. Sealing PTFE ring is then fixed on followed by upper piston plate with PP ring (Fig. 5) which are fixed by six bolts M8.

*Fig. 4 Frit ring with distributor nets*

*Fig. 5 Upper piston with seal rings*

Through mentioned hole is piston unit inserted above 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).
Other bolts are added now to fix upper column flange. All bolts are tightened properly. Finally hydraulic cylinder is connected to the oil pump such way, that upper cylinder input is connected to the upper output hole on the pump body and bottom input is connected to the bottom pump output. Oil pump valve (Fig. 8) is shifted to the most forward position in which oil is pressed to move piston down (middle position is neutral, both ways opened, back position is to press the piston up). Piston movement is proved and its central position with respect to the tube hole is controlled.

*Fig. 7 Upper piston inserted on its position, connecting pieces fixed*

The piston unit is screwed to a connecting piece 1 (Fig. 6), but before it an input capillary has to be installed.
Column with bottom flange completd by stand legs which are screwed to side holes of bottom tube flange and slighly tightened on positions. Stand plate which is added have three holes for legs. Leg bottom parts are equipped with M12 threads on which fixing ball are finally screved.
Hydraulic cylinder is connected to the upper column flange, equipped with a connecting piece 2. The piece has a thread that fits to the hydraulic piston end part.
Flange unit with hydraulic cylinder is connected *via* long bolts with distance tubes (4 pcs) to the upper tube flange (Fig. 7). There has to be a side hole remaining for the piston installation.

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

## 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 follows sorbent manufacturer instruction.
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. 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. 7).
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 closed and bottom is opened. Now the oil is fastly 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 $cm^2$ and hydraulic piston area only 28,6 $cm^2$. 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 – 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. 8 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, iol 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 . Manufacture and servicing:**

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