PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY

PC 01 200/400 DS

user manual

1. Desription 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 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 01 200 columns with inner diameter 200 mm are designed for industrial separations and typically are working with flow rate 300 ml/min. – 1200 ml/min. depending on sorbent type and separation mode. Maximal column pressure is 120 bar.

PC 01 200 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.

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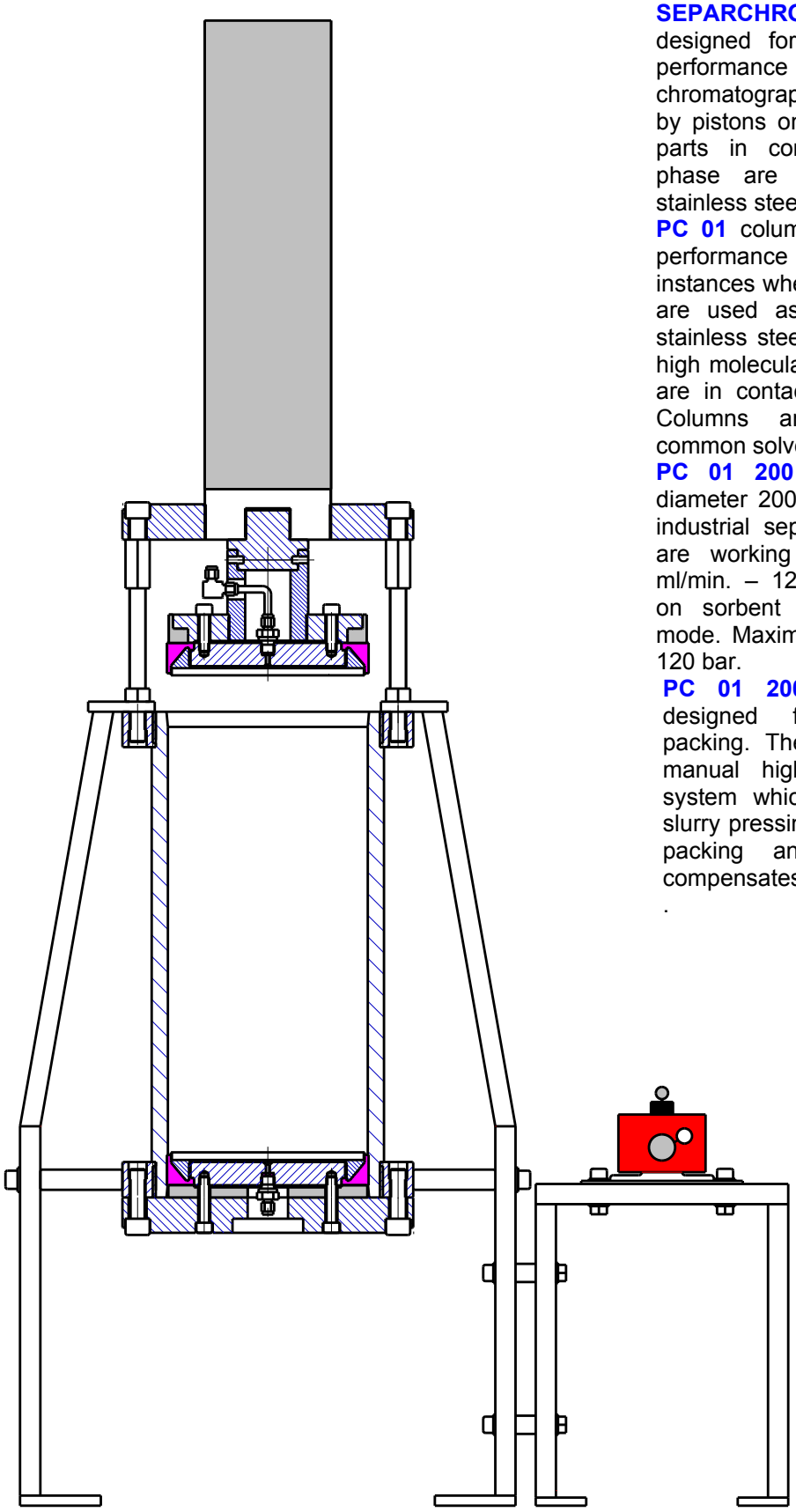

Fig. 1 Column cross section

2. Column design

Typically the **PC 01 200/400** (Fig. 1) column consists of tube, I.D. 200 mm with the length 450 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 16 holes with M16 threads for the clamping bolts or rods.
The upper and bottom parts of the column are closed by stainless steel pistons with UHMWPE made seals and stainless steel/polypropylene (PP) support. Each piston unit consist of five parts (see Fig. 2, 3, 4, 5):

- ➔ porous disc (frit) made of Poremet 2 material consisting of 10 screen layers (5 mm thickness) 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 1/4" (6,4 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 on the upper side b) made of PP plate on bottom side and inserted between bottom flange and piston body

UHMWPE piston seal part is 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. 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 120 bar) and has thus smaller diameter than column itself.

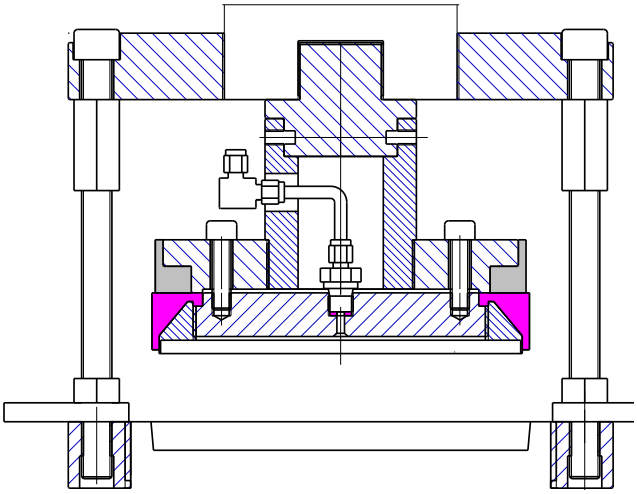

Fig. 2 Upper piston cross section

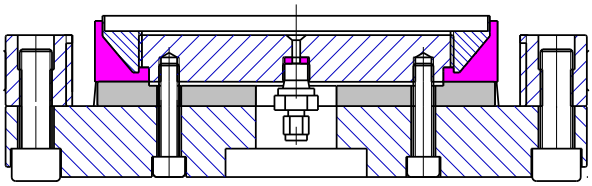

Fig. 3 Bottom piston cross section

Bottom column flange has a central hole for output fitting and four side threads for stand legs. Legs are made of stainless steel profiles 20 x 40 mm. Legs are hooked and connected either to the upper column flange and to bottom column flange (via short rods with threads). There are two special suppors for oil pump. The front one is connected to the column leg.

3. Column assembling

PC 01 200 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 (in italics).
Piston assembling starts with distributor net inserting into the frit ring (Fig 4). Sealing ring is than 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.
Note: An output fitting is already assembled from the factory and fixed on its position in piston plate by special adhesive.

*No*te: 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 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, iIf only regular bolts are available. Than bolts are removed, ring added and pressed once more,

Upper piston is to be assembled the same way like the bottom one. Sealing UHMWPE ring is then fixed and followed by upper piston support plate with PP ring (Fig. 5) which are fixed by eight bolts M10. On the piston unit is screwed a connecting piece 1 (Fig. 6).

Note: input fitting is fixed on piston plate by special adhesive and as well as is fixed output tube in the fitting. A connection to the system is done on the elbow fitting which is situated out of the side hole of the connecting piece 1 (see Fig. 5). Flexible armed PTFE tubing 1 m in length to be connected to the elbow is the part of delivery.

Note: If necessary piston can be fully deassembled (instead of input fitting and input tube).

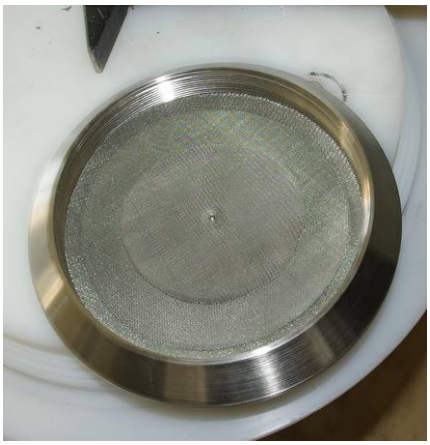

Fig. 4 Frit ring with distributor nets

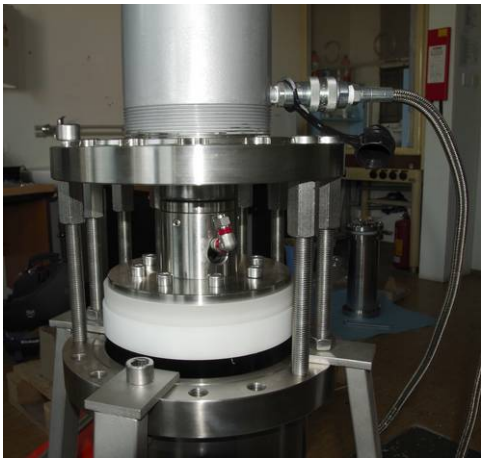

Fig. 5 Upper piston installed in the column

Column tube with bottom flange is completed by four stand legs. Legs are connected to the upper column flange (see Fig. 5). Three legs are connected by distance rods (three rods are equipped with long and short nuts, remaining have only long nuts. Side rods (see Fig 6)re screwed to side holes of bottom tube flange and fix with bolts in proper position on the each leg. i

Hydraulic cylinder is carefully screwed to the upper 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.

*T*he oil pump is connected to the front and back U shaped supports (see Fig 8 and 9). From pump support with side holes is connected to the column leg having the same holes (see Fig. 7).

Upper column tube flange is equipped with 11 distance rods (Fig, 10) and a space of five rods is kept open.

Fig. 6 Leg with side rod

Flange unit with hydraulic cylinder is connected *via* bolts M16 to distance rods of the upper tube flange. 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 (Front Fig.) is shifted to the most left 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). Oil pump rotation valve has to be closed.

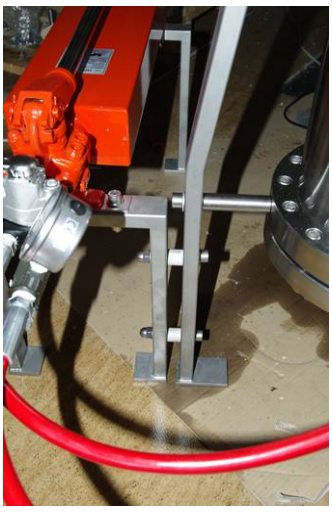

Fig. 7 Oil pump stand connection to column leg

The upper piston unit is inserted into the gate in distance tube and connecting piece 1 and 2 are fixed together by three small bolts M8 on side of piece 1 (Fig. 11). If necessary the piece is moved down by the pump. 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. Armed PTFE input capillary 1/4” is connected to the elbow on the piston.

Four distance rods are added now to fix upper column flange (see Fig. 12). All bolts are tightened properly.

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 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 20 mm under the tube edge through a gap between the column and upper piston..
The hydraulic pump is use 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.

Fig. 8 Oil pump fixed on the back support

Fig. 9 Oil pump fixed on the front support

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 314 cm^2 and hydraulic piston area only 71 cm^2. Thus the pressure in the column is 4,4 times lower, that the pressure in the hydraulic cylinder.

In other words, having on the oil manometer 440 bar, there is 100 bar inside the column. For the column packing is recommended to use oil pressure 400 – 500 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.10 Column input flange with distance rods

5. Column unpacking

Fig.11 Upper piston installation

The column output flange is released and sorbent is pressed out of the upper piston movement generated by hydraulic piston. When the piston is moved to the most bottom position, oil valve is switched and oil piston with column piston is moved most up.

Fig. 12 Fully assembled column head

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 hangers connected to the side holes of hydraulic cylinder.

Manufacture and servicing:

Separlab Ltd. Radiova 1, 102 00 Praha 10, Czech Republic
tel +420 242449669
e-mail: info@separlab.eu