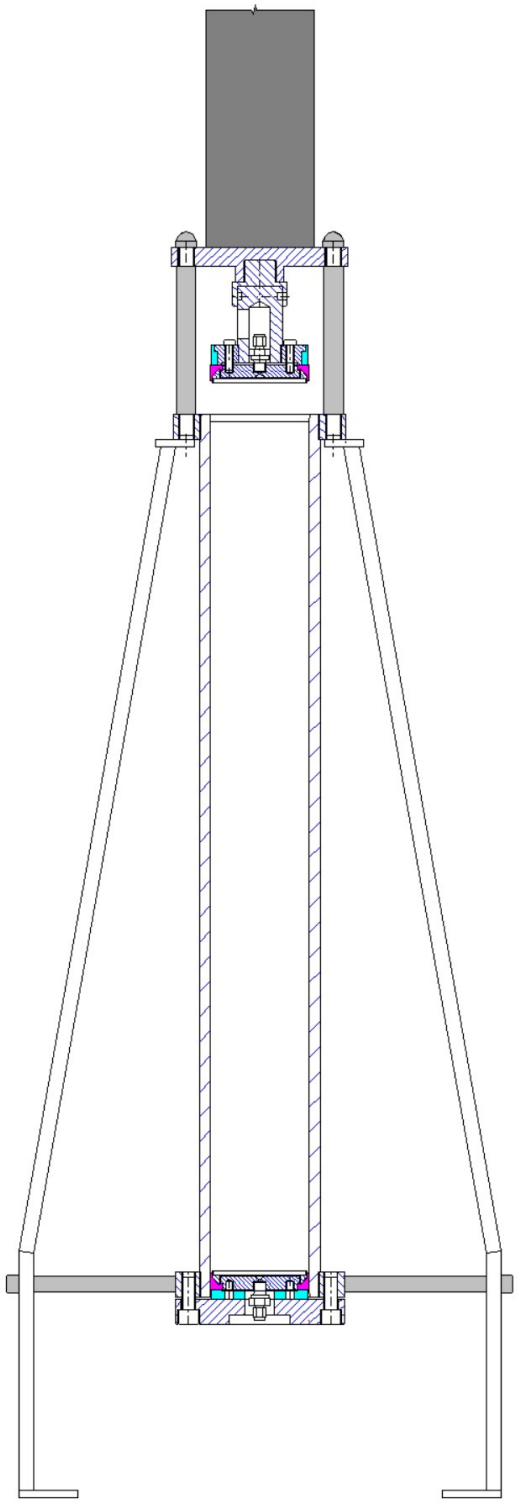
separlab

PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY

PC 01 100/800 DS

user manual



Separlab 2020

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 (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 SEPARPRESS D20 MM 250 (20 tons, double action, stroke 250 mm) 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 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 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 on the upper side b) made of PP on bottom side and inserted between bottom flange and piston unit

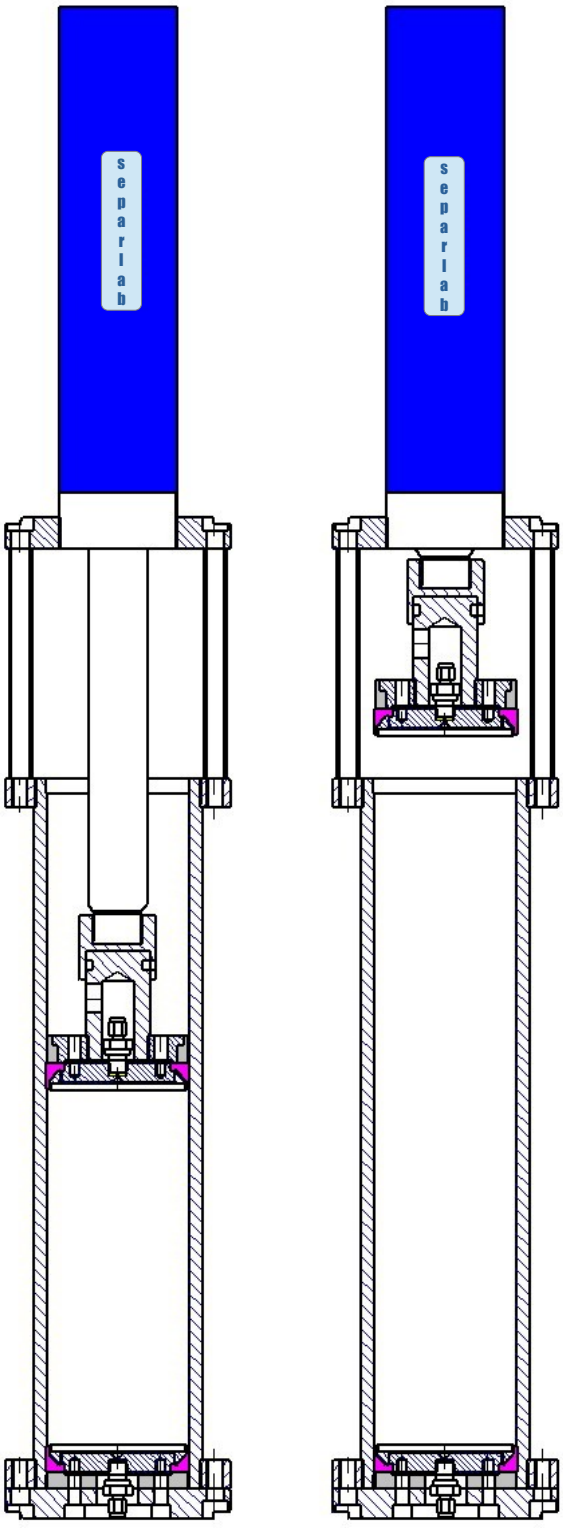


Fig. 1 Column cross section

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 is only one bottom column flange. Insted of upper flange is used the hydraulic cylinder one. The cylinder is designed for higher pressure then is used

inside the column (250 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 supports (Fig. 4). Legs are made of stainless steel profiles and are fixed from bottom side of upper tube flange (Fig. 5).

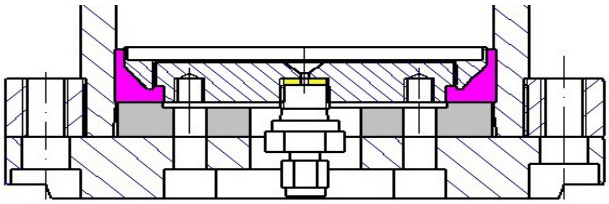


Fig. 3 Bottom piston cross section

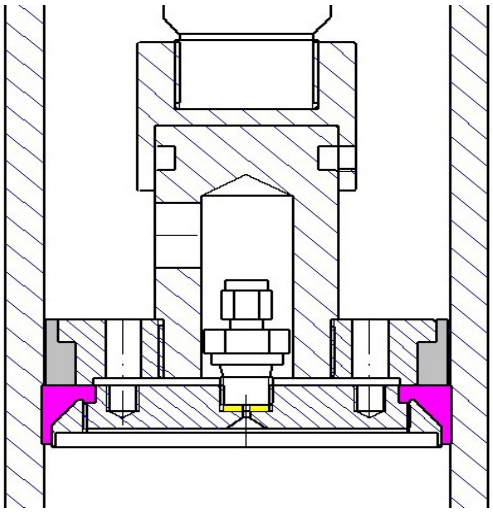


Fig. 2 Upper piston cross section

Fig. 4 Leg support

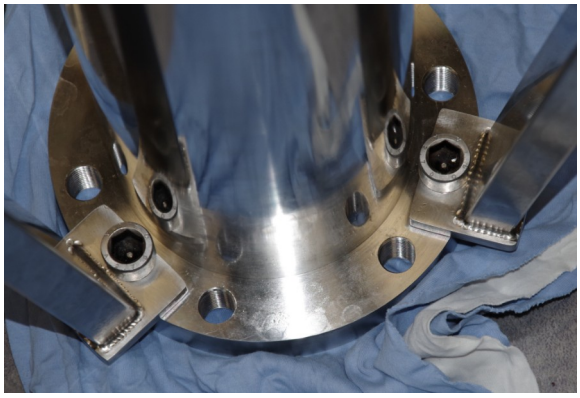


Fig. 5 Legs connected to the flange

Column assembling

PC 01 100 column is 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 (Fig. 6) inserting into the frit ring. The piston is screwed to the ring and tighten. Sealing ring is now fixed to the completed piston followed by PP support plate and all is fixed by 6 bolts M8 to the bottom column flange (bottom piston) or to upper piston support (upper piston, see Fig. 7)). Input fitting is assembling to the piston before connecting to the support incluting a piece of 1/8" capillary and an elbow 1/8". An output fitting can be assembled later.
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 inside by bolts of proper length. Finally pressing bolts are replaced by regular bottom column flange bolts.
Usually the bottom piston with flange is delivered installed to the column tube.

Fig. 6 Distributor nets

An input capillary has to be installed before use. In case the piston is delivered fully assembled, the capillary is connected to the 1/8" elbow on piston connection part 2. Note: for 100 mm column is connection part 1 is delivered with the hydraulic cylinder. It has to be removed and screwed to the hydraulic piston before use.

Fig. 7 Upper piston with seal rings and support

Column tube with bottom flange is to be equipped by three stand legs. Their support rods are screwed to side holes of bottom tube flange. Upper leg parts are connected to the bottom tube flange by M 14 bolts and fixed by support rods (Fig 7) with nuts in proper position.

When legs are delivered mounted to the tube, hydraulic cylinder installation can start immediately. Distance rods with thread are screwed to the upper tube flange (Fig. 8). Distance tubes are added (including inserts) and hydraulic cylinder is carefully positoned to the column and fixed with next inserts and cover nuts (Fig. 9). One distance rod is not installed to allow installation of the upper piston.

Hydraulic cylinder is to be connected to Separpress unit now using delivered hoses with fast connectors. The unit is started (see the Manual for it) and hydraulic piston is moved down and up. During all following operations hydraulic piston has to be in the most back position.

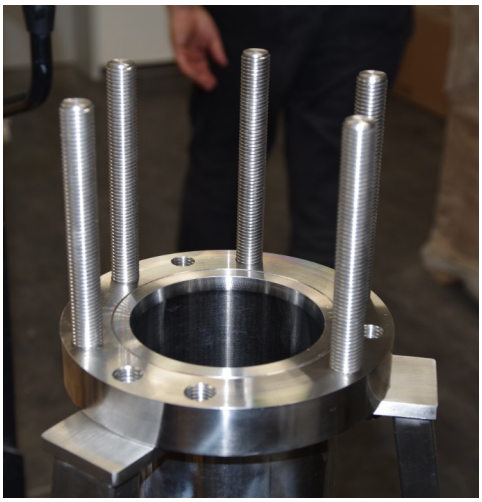


Fig. 8 Upper tube flange with distance rods

Upper piston unit (Fig. 7) is inserted among distance rods and fixed by three small bolts M8 to the connection piece 1 on hydraulic piston (see Fig. 10). Do not tighten these bolts to much now to allow the piston move and rotate.

Input capillary has to be installed (connected to the installed elbow) on upper piston before it is connected to the hydraulic piston. It is recommended to coil the capillary over the piston support.

Piston movement to the column tubeis proved and its central position with respect to the tube hole is controlled. Missing distance rod is added now to fix upper flange. All cover nuts are tightened properly.

Fig. 9 Column with distance tubes and hydraulic cyloinder

Upper piston is now moved inside the column stepwise and when is fully deeped, smal bolts M 8 on connection piece 2 are tightened properly. Movement from the tube and to the tube is checked once more. Than is piston parked in most upper position. Column is reafdy for packing and use.

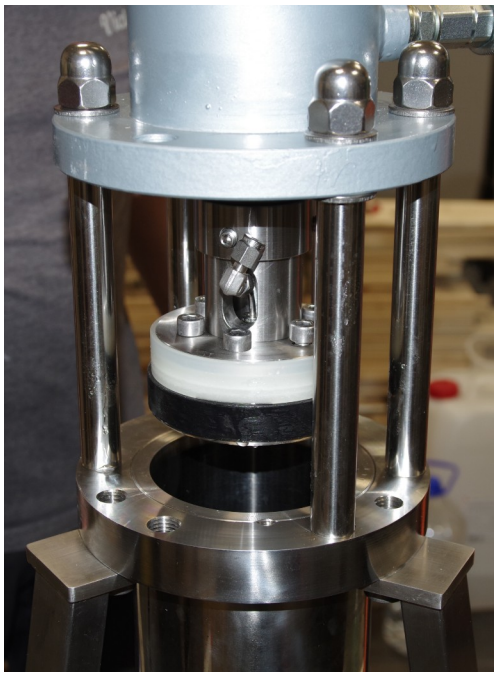


Fig. 10 Upper piston installed

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 'sufficiently big) reservoir. Output cap is closed. Proper oil flow rate is set on the oil pump as well as the pressure limit (see note bellow).
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 tube and upper piston (see Fig. 10). 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 is the oil is as fast as possible pumped into the oil cylinder to move the piston into the column down.

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 63,6 cm^2. Thus the pressure in the column is 1,2 times lower, that the pressure in the hydraulic cylinder.

In other words, having on the oil pressure 190 bar, there is 150 bar inside the column. For the column packing is recommended to use oil pressure limit on 120 – 130 bar.
When column is fully packed, the oil pressure starts to increase rapidly and the pump stops on the limit. After few minutes pressure limit is changed, to be a bit higher that expected pressure of mopbile phase during column use. Pressure of oil is going down slowly and due the time and column can be connected to the system. Liquid starts to be pumped through the column.

5. Column unpacking

The column output flange is released and bottom piston is pressed out due the upper piston movement generated by hydraulic piston. Sufficiently big tank is inserted under the column tube now. The upper piston is moved up than and liquid is pumped into the column simultaneously. When piston is on the edge of column tube (still inside) the pump is stopped and piston is moved down with maximal oil flow rate. Sorbent is pushed out of the column.
The upper piston is moved up again, column tube is cleaned by a liquid and bottom section (piston + flange) is installed again. Column is ready for next packing.

6. Notes

Maximal temperature for column use is 70 °C.
Piston sealing is to be changed when liquid leaks from the column (average frequency of the change is after 10 packing/unpacking cycles)..

6. Manufacture and servicing:

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