separlab

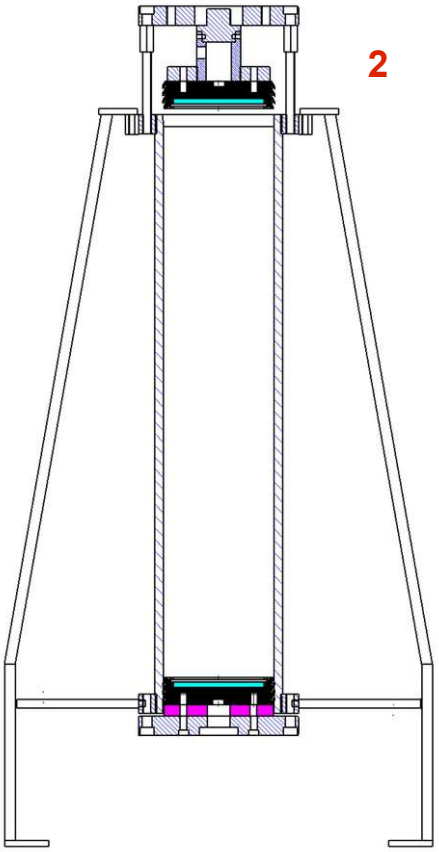
PREPARATIVE COLUMN
FOR
LIQUID CHROMATOGRAPHY

PC 02 200/1000 DS

user manual

Separlab 2020

1. Desription and use



SEPARCHROM PC 02 columns are designed for medium 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 L stainless steel.

PC 02 columns are used for high performance separations in instances where small or large 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 02 200 columns with inner diameter 200 mm are designed for industrial separations and typically are working with flow rate 200 ml/min. – 800 ml/min. depending on sorbent type and separation mode. Maximal column pressure is 20 bar.

PC 02 200 DS columns are designed for dynamic slurry packing. They are equipped with manual high pressure hydraulic system SEPARPRESS which serves for sorbent slurry pressing in dynamic mode of packing and during the use compensates bed volume changes.

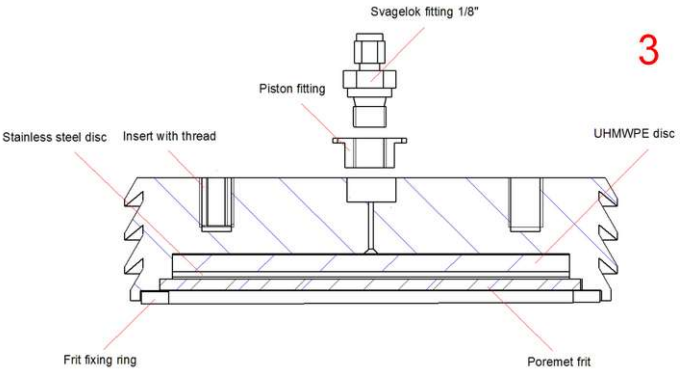
PC 02 200 DS columns are delivered in modifications depending on customer needs. Column described in this manual is additionally equipped with a system for reversation of the flow and with the cell for remote UV detector.

2. Column design

Typically the **PC 02 200/1000** (Fig. 1 on front page, Fig. 2) column consists of tube, I.D. 198 mm with the length 1080 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 12 holes with M16 threads for the clamping screws or rods. Even the column is schedulated for the pressure 20 bar, tube withstands the pressure 100 bar.

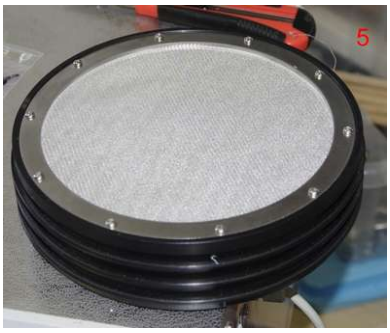
The upper and bottom parts of the column are closed by pistons made of UHMWPE (Fig 3, 4) having 3 integral sealing sleeves. Pistons are completed with:

- 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 (Fig 3, 5)
- frit fixing ring made of 3 mm thick stainless steel ring (Fig 3, 5)
- UHMWPE made disc 10 mm thick with groves on both side for precise liquid distribution (Fig 3, 6, 7)
- stainless steel sheet (1 mm thick) disc between UHMWPE disc and the frit (Fig 3, 8) complete the system of precise liquid distribution
- liquid input or output fitting for Svagelok type 1/8" (3,3 mm) capillary fitting is fixed by six bolts to the flat bottom part of the piston (Fig. 3)
- 6 stainless steel fittings on the flat bottom side of the piston for the connection to support plate or bottom flange (Fig 3)

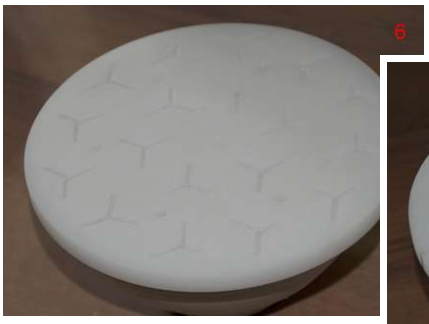


Column tube flanges are connected to two flat column flanges. Upper one with a central thread for a hydraulic cylinder is connected by 16 thread rods with long nuts. The hydraulic cylinder is designed for higher pressure then is used inside the column (250 bar against 20 bar) and has thus smaller diameter than column itself. Upper flange has three side threads M10 for column legs.

Attention!
Thread holes are in different angles (110°, 110°, 140°) in order to allow good approach to column bottom side



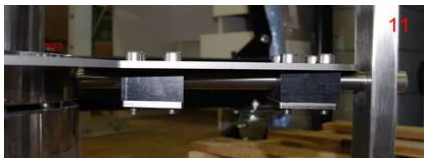
On the hydraulic cylinder piston rod output is screwed connection part 1 with a grove on the side of the column piston. On CP 1 is fixed part 2 using three bolts M8 which are screwed to the grove in PC 1. PC 2 is screwed to a support plate which is connected directly with column upper piston. In PC 2 is a side hole for capillary input (see Fig. 2, 9).



Bottom column flange has a central hole for output fitting and three side threads for stand legs supports. The piston is connected to the flange through polypropylene insert (Fig. 2) by 8 bolts M10.

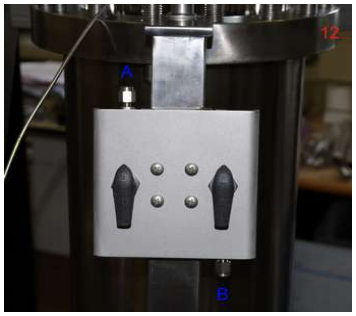
Legs (Fig. 1, 2) are made of stainless steel profiles. Legs are hooked and connected to the upper column flange (Fig. 10) Leg support rods (Fig. 11) are connected to the bottom column flange.

Support rods are completed by leg support plate made of 5 mm thick steel connected to rods by 6 sleeves On one leg are connected panel for flow reversation and UV detector remote cell (Fig 12, 13). Both panels are connected to the leg by bolts M6.



3. Column assembling

PC 02 200 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).

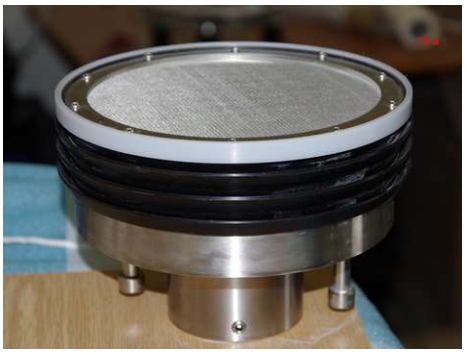


Piston assembling starts (UHMWPE disc in the piston grove and metal disc are fixed in the piston body by the manufacturer and it is not recommend to deassemble it) with the frit which is inserted above the metal disc with small holes. The frit is wrapped by PTFE tape 0,2 mm thick 12 mm in height and inserted to the grove in the piston body (Fig 14). It is covered by frit ring, fixed by bolts to the piston body and excess PTFE tape is removed by a knife (Fig. 15)

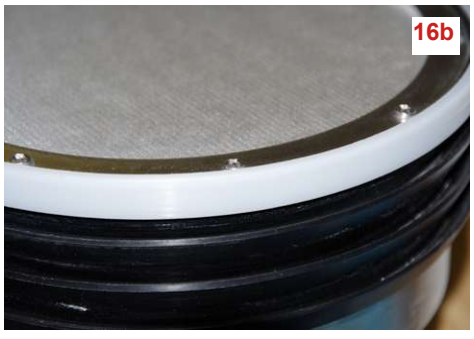
On the bottom flat side of the piston body with connection fitting fixed by six bolts is screwed Svagelog type fitting for capillary connection.

This fitting is fixed by special adhesive for bolts fixing not allow its rotation. Under the Svagelok fitting is inserted a PTFE sealing ring.
A short capillary 1/8" is connected to the upper piston having a Svagelok elbow on opposite side. An other short capillary is mounted on the bottom piston with straight connector on the opposite end.
Column tube with tube flanges which are correctly oriented for legs assembling is stand in vertical position with output flange on the top. Bottom piston is assembled with the PP insert and the bottom column flange by 8 bolts M8. UHMWPE wiper ring (delivered with the column) is pressed on the edge of the piston in order to shadow sealing sleeves of the piston (Fig 16 a,b,c,). The ring and sealing sleeves are wetted by a small amount of glycerol or low molecular weight polyethylenglycole. The piston with flange is carefully recessed into the conus on column tube edge using 4 long M16 thread rods (delivered with column). The piston is pressed inside the column symetrically by nuts tightening. It is necessary to keep the distance between flanges the same on all places. When the distance of flanges is less then 10 mm, 4 pieces of distance inserts (delivered with the column) are inserted symmetrically between both flanges (see Fig 17). Assembling bolts are replaced than with standard column bolts M16x60 mm and all bolts are tightened, to keep distance inserts on the position.

Attention!
Column PC 02 200 uses the tube and flanges from the column PC 01/200. Column PC 01 is designed for the pressure 100 bar and for this pressure the column is equipped with 16 flange bolts. For PC 02 and pressure 20 bar (testing pressure 28 bar) is enough to use 4 bolts M16 on each flange. Taking in the account that during packing procedure can be expected pressure pulses, we recommend to use 8 bolts M16 for the column PC 02/200.



Column tube with bottom flange is than elevated and completed by three stand legs which are connected by special bolts M10 to the upper column flange (Fig. 10) and their supporting rods are connected to side holes of bottom tube flange and fixed with covering nuts (Fig. 11). Before supporting rods are mounted, the suporting plate must be pull on the column tube (Fig. 1 on the front page). When rods are installed and the plate laying on them, it is connected to rods by delivered 6 sleeves (each 2 on 1 rod).
When the column with legs is not elevated, upper flange with hydraulic cylinder can be installed.



Hydraulic cylinder is carefully screwed to the upper column flange. The hydraulic piston has to be in the most back position. Upper tube flange is equipped with four distance rods with nuts (a space of four rods is kept open for operation of piston insert). All distance rods have to have the same height. Flange unit with hydraulic cylinder is elevated and connected *via* bolts M16 (delivered with column) with nuts on upper part of distance rods to the upper tube flange. Finally hydraulic cylinder is connected to the oil pump with connecting hoses.
Connection part 1 is screwed to the hydraulic cylinder piston. Connection part 2 is fixed by bolts to the CP 1 including support plate for the piston. UHMWPE wiper ring (delivered with the

column) is pressed on the edge of the piston in order to shadow sealing sleeves of the piston (Fig 16 a,b,).

Mostly upper piston is delivered with connection part 2 and support plate fixed to the piston. The installation of the capillary is not necessary. UHMWPE wiper ring is as well as pressed on the edge of the piston from the factory (in order to shadow sealing sleeves of the piston Fig 16 a,b,c,). The ring and sealing sleeves are wetted by a small amount of glycerol or low molecular weight polyethylenglycole. Connection capillary (from the upper piston to the pump or to reversation panel) curled into a ring equipped with nuts and ferulles is connected to the piston elbow. Thin metal sheet is used to cover column tube and the piston with support plate and CP 2 is moved on the sheet to its central position. Hydraulic piston is moved down carefully to reach the smallest gap between CP 11 and CP 2. Three bolts on CP 2 hate to be released during this procedure and they are slightly tightned when CP 1 is inside the grove on upper part of CP 2. The whole is moved a bit up and metal sheet is removed.
Piston is moved down (bolts connected CP 1 and CP 2 not fully tightened) and pressed inside the column carefully (most low oil flow rate is set). When first sealing sleeve is coming without problem inside the column tube colnus, connecting bolts are tightened. Piston is moved up again and once more down to dip all piston sealing sleeves inside the tube. Finally the piston is moved almost up and remaining distance rods are installed (only eight rods on Fig. 18).

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 60 mm under the tube edge through a gap between the column and upper piston (Fig. 19).
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. The proper pressure limit has to be se on the oil pump before.

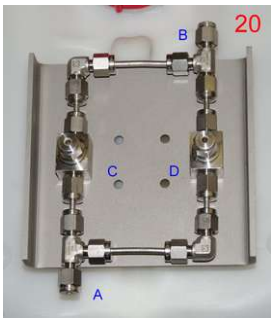
Attention!
The pressure in the column is not equal to the pressure on the oil pump display. Column cross-section area is 308 cm^2 and hydraulic piston area only 79 cm^2. Thus the pressure in the column is 3,9 times lower, that the pressure in the hydraulic cylinder. In other words, having on the oil display 78 bar, there is 20 bar inside the column. For the column packing is recommended to use oil pressure 80 bar.

When column is fully packed and upper piston is sitting on the sorbent layer, the oil pressure starts to increase rapidly. It is necessary to stop oil pumping at this moment. Limit of oil pressure is to be set now to lower value, nevertheless higher than the presure expected inside the column during separation.

5. Column unpacking

For unpacking can be used different method. Having pump for mobile phase with high output and small spherical sorbent particles, sorbent can be pushed out with mobile phase pressure. Firstly is necessary to close output from the column and to press out the piston with flange. It is necessary to use for these operations a vessel which can be put under the column (see Fig 19).
For irregular sorbent with large particles is better to press out the sorbent by the column piston and to replace mobile phase with water before. Column is washed with 19 5 l of ethanol

and than with 20 l of destilled water. Than there is no organic solvent inside the column. Proper vessel with volume 20 l or more is installed under the column bottom flange. The column output is closed and bottom flange bolts are released and the whole flange is pressed out of the column (two men must keep in hands (Fig. 19) by the pressure of water which is pumped inside the column by mobile phase pump.
When bottom piston is out (together with small amount of water and sorbent), the vessel is put outside, the piston is washed with water and sorbent is put into the store. The empty vessel is again inserted under the column and pumping of the water starts. In this time but the upper column piston is moved slowly part by part up byoil pump. When it is moved up at about 10 cm (it needs at about 8 minutes with 400 ml/min. water flow rate) pump of mobile phase is stopped and column piston is moved down with maximal oil flow rate. Sorbent is now pressed out of the column. This process is repeated until all sorbent is out of the column. The vessel with sorbent and water is put away. Sorbent is put into store. The vessel is installed again under the column and the piston is moved by oil to the most upper position. Column is washed with strong stream of the liquid (water with detergent and than pure water).

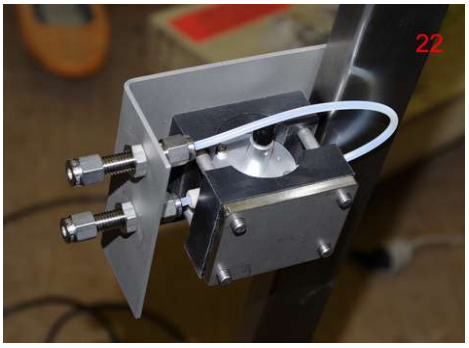


6. Reversation flow possibility installation

Reversation of the flow is usefull for cleaning of sorbent and frits. Reversation panel can be installed on the column PC 02 200/1000. The panel (Figs 12, 20, 21) is mounted on column leg. The reversation system has four inlets (A, B, C, D). Inlet A is for the capillary coming from upper piston, inlet B is for capillary from bottom piston. Inlet C is a pump input and outlet D is detector output.
On the front of the panel are available two handles for installed ball valves. In configuration on Fig. 21 valves lead the flow from top of the column to the bottom. When to be put in opposite position, the flow is going from bottom of the column to its top.

7. Detector cell installation

Column PC 02 200/1000 can work with different types of detectors, nevertheless mostly used is photometric UV detector. The liquid from the column is coming to detector box where is situated measuring cell. It can be dangerous, because strong flow of inflammable phase is coming to the box with electronics.
To avoid such danger, a remote detector cell can be installed on the column PC 02 200/1000 and signal is transferred by optical cables to detector box. Detector cell is installed on single panel and liquid input and output are situated on panel wall to avoid tension of cell itself (Fig. 13). The panel is mounted on the column leg under the reversation panel.
The are connectors for optical fibre cables on the top and on the bottom of the cell (Fig. 22). Cables must be assembled carefully to avoid a damage of quartz fibres. Cables between detector box and the cell must be carefully fixed too, not to change their position during column use.



8. Notes

Maximal temperature for column use is 60 °C.
Maximal pressure for the column is 20 bar. Testing pressure is 28 bar.
Piston sealing is to be changed when liquid leaks from the column, average frequency of the change is after 10 packing/unpacking cycles, depending on the use of scraping PTFE tape.
For manipulation with the column has to be used special hanger connected to the thread on the top of hydraulic cylinder.

9. Manufacture and servicing:

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