separlab

PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY
SEPARCHROM PC02 500/1200 DS

HYDRAULIC DEVICE
SEPARPRESS D40 EE 1000/500 PC02

user manual

Stainless steel made liquid chromatography column SEPARCHROM PC 02 500/1200 HDC is designed for medium pressure preparative chromatography. It is used together with hydraulic axial compression device SEPARPRESS D40 EE 1000/500 PC02, which press column piston to the sorbent bed.

1. Description

Column design is apparent from Fig. 1. Column consists of a stainless steel made tube with inner diameter 492 mm and wall thickness 10 mm. Column has on both sides flanges with 12 holes each. In the column are from both sides inserted identical pistons made of circular stainles steel plates (36 mm thickness). Pistons are provided with UHMWPE/PP sealings. There is a flow distributor made of three layers of stainless steel nets with channels on front sides of each piston, covered by Poremet 2 frits (10 layers, 5 mm thickness) with pore size 2 um. The whole is fixed to the piston plate by a stainless steel/UHMWPE rings fixed by smal bolts M6.

Column output piston is connected to outlet tube by end fitting designed for a tube 1/2" (12,7 mm) O.D.. On the input piston is screwed an armed PTFE tube equipped on the end with a fitting for a tube 1/2" (12,7 mm) O.D.. Bottom piston is inserted to the column tube and fixed to bottom stainless steel flange. Between the piston and the flange is an additional polypropylene ring inserted. Its role is to press seals. To the piston body are screwed bolts which are going through a bottom flange. Their tightening increases a sealing power. Bottom flange can be equipped with four stainless steel legs

Upper piston design is more complicated (see Figure). Its side being in contact with a liquid is the same as in the case of bottom piston.

Hydraulic dual action cylinder with maximal oil pressure 200 bar is situated in upper flange of the unit. Hydraulic piston is provided by a circular stopper and it is connected to a piston tube (8 bolts M10x30) which is screwed into a stainless steel tube connected to the piston support plate. The hydraulic flange is connected to the upper part of the column by 12 long bolts M22 with distance tubes.

Hydraulic is connected by two high pressure rubber tubes to the electric motor driven oil pump. Tubes have fast connection parts on the side of the cylinder. The oil pump with an electronic manometer has 30 l reservoir and a solenoid switching valves for movement of hydraulic piston up and down.

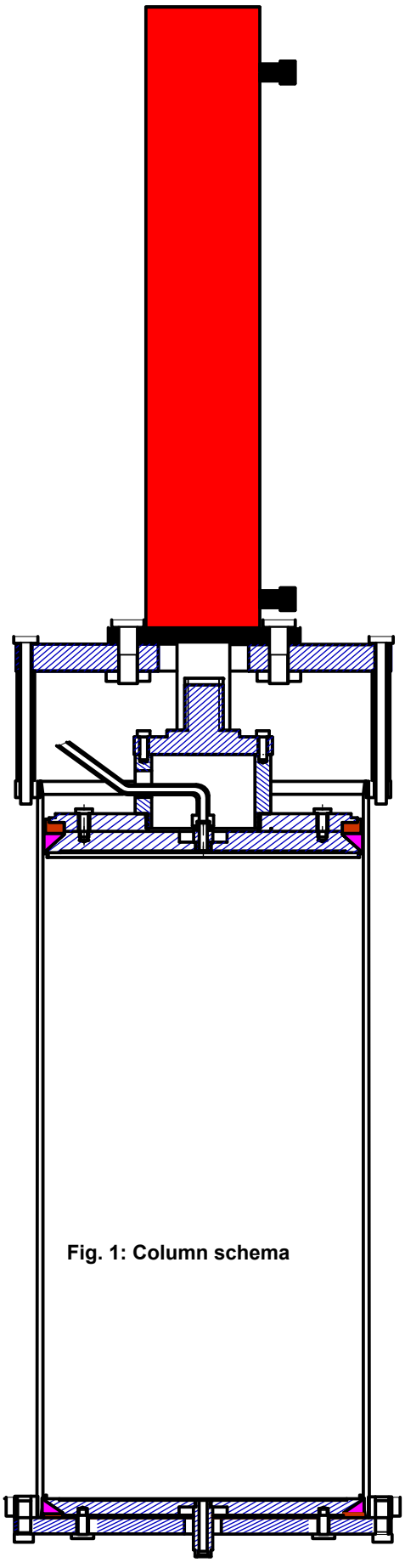

Fig. 1: Column schema

2. Hydraulic installation

Hydraulic instalation is composed of hydraulic cylinder, pump unit, control box with buttons and remote control box. Hydraulic cylinder is filled with hydraulic oil and equipped with fast connector for armed oil tube. The pump unit is inserted into a stainless steel box, equipped with 30 l oil tank, system of solenoid valves and pressure gauge. The pump unit is connected by cables to the control box situated outside. The control box is equipped with frequency changer for oil pump motor, PLC unit for system control and by a display with keyboard.

The small control box with buttons is used to operate with the system on the place (move the hydraulic piston up and down and star automatic regime) as well to switch the system by emergency button. The remote box is used for setting parameters.

Description of Keypad 1 drawing

F1:	used to move items between displays down
F2	key: used to move items between displays up
Key "arrow down":	used for deleting the setpoint
Key "arrow up":	used for adding setpoint
ENTER key:	used to confirm the setpoint a
Key START / STOP:	is used for starting and stopping the oil pump

Note: The last key is functional on any item at any time even if the system is controlled from an external source and the whole keyboard is off.

The order of display items:
Flow (%)
Pressure (bar)
Flow Settings
Pressure Settings
Hysteresis Settings
Password Settings (the following items are accessible only after entering a password)
Zero pressure settings
Max pressure settings

Example of operation

After switching on the unit is set to display the first item. In the upper right corner shows the status (at this moment, STOP). The display shows the current flow and the current pressure. After pressing the F1 key to get to the second operating item display, where it is displayed as the current primary pressure and secondary current flow.

Pressing the F1 key gradually check set flow rate, pressure, and hysteresis and end up in the Password entry, where the other items we get to the password.

After checking the set of values is possible by pressing F2 to return to the default item and we can start the unit by pressing the START / STOP. After pressing the change in the upper right corner is visible (to RUN) and the pump starts to pump. If not, it is possible that the pump is blocked by one of the following reasons.

a) pressure exceeded the set limit (in the bottom row shows the actual pressure)

b) autonomic control is disabled with the command on the serial line

c) drive motor is not ready or is in an error state, then RUN flashes for a while and just starts STOP.

Then is possible pressing the START / STOP again to stop the pump. The pump motor starts stops rotation stepwise during approx. 4 s.

Pressure limit control function stops and starts the pump depending on the current pressure which was set. To avoid fast on and off switching, an interval in which pump stops and starts again is to be set. This interval is called hysteresis and can be set between 1 and 15 bar. It is recommended to set hysteresis between 5 and 10 bar. Pump stops when the real pressure excess set pressure limit + hysteresis and starts again when pressure is going down set pressure value - hysteresis. T

Calibration of the pressure

Performed after entering the service password on the left keyboard. Attention: in these settings change the items set important parameters pumps!

The three items relate to the calibration gauge. The first is the "Settings Zero pressure". To execute it, the pump has to be in pressure less state. When figures on the display stabilize, press ENTER. The transducer value for pressure 0 bar is recorded. Numerical data are raw, unadjusted data A / D converter, thus they are constantly changing a bit. The second is "Max pressure Settings" Here enter the value of the pressure at which is to calibrate the gauge. It is recommended to use at least half of the maximum pump pressure. The third alows to set "Max pressure". Here pressurized to a pressure pump from the previous item and after stabilization figure press ENTER. A value of converter for a given pressure is recorded. Once calibrated repeatedly press the F2 key to leave the screen of calibration.

Control box near the column

The control box situated on the flexible cable near to the column and is used to operate hydraulic system. On the bottom side of the box is a switch allowing to set local or remote (on the electronics box) control. When local is chosen a down or up movement of hydraulic piston can be selected or an automatic function of pressure hold can be selected (RUN, STOP). This function is mainly used for column packing and working with.

3. Column assembling

Column tube is symmetrical. Column piston plates are identical, as well as UHMWPE seals, but upper and bottom PP piston supports differ. Column assembling starts just with piston units. First of all is

assembled bottom circular piston plate with end fitting parts. Then are pistons are equipped with distributors and frits according following figures:

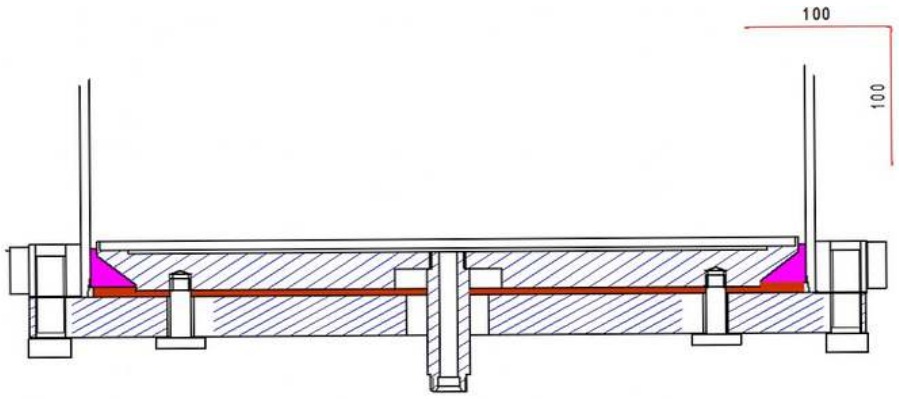

Fig. 2: Output bottom piston

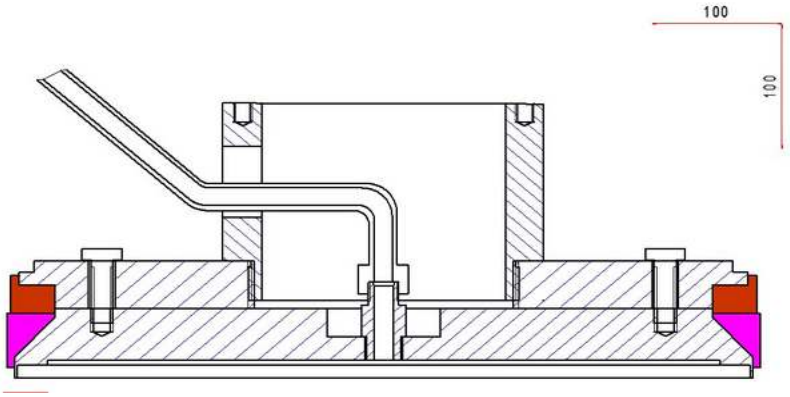

Fig. 3: Input upper piston

Frits are covered by UHMWPE fixing rings and stainless steel fixing. Finally M6 bolts are added to fix the whole and tightened. Bottom piston is than assembled with sealing ring and connected to the bottom flange (12 bolts M16x70) with a PP support plate in between. Upper piston is equipped with a seal and a support plate with PP support ring. Support plate are to be connected by 12 bolts M16x50. before connecting, the input flexible armed tube is attached to the piston plate and a connecting tube is screwed to the support plate. Support plate is put on the piston and flexible tube is got through a hole in connecting tube out of the piston whole. Only then are to plates connected by bolts.
Bottom column flange with piston is connected to column by 12 bolts M22x110 with insers and nuts. The piston is pressed to the column by tightening flange bolts. Then column is equipped by column legs and reversed. Legs are fixed to the floor.
Upper column flange is connected to columm by four thread rods with distance tubes. Each two of these rods have to be situated near to each other to allow the piston to move inside the column tube.Then the hydraulic cylinder and assembled to the top of column flange by 12 bolts M24x110. A lid of connecting tube is than screwed on the hydraulic piston.

Fig. 4: A piston with frit

Fig. 5: A piston with frit and frit sealing ring

Fig. 6: A piston with frit and frit, sealing ring and fixing ring

Fig. 7: Bottom piston with a cap

Fig. 8: Bottom piston with sealing ring

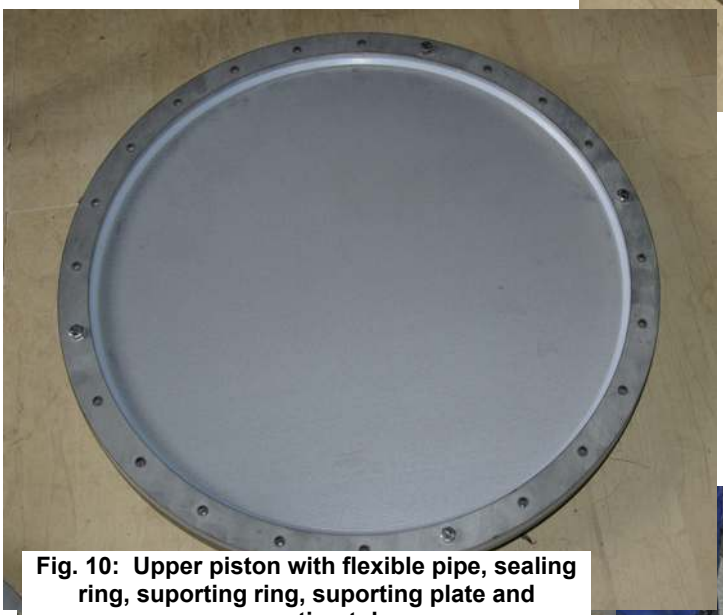

Fig. 10: Upper piston with flexible pipe, sealing ring, suporting ring, suporting plate and connecting tube

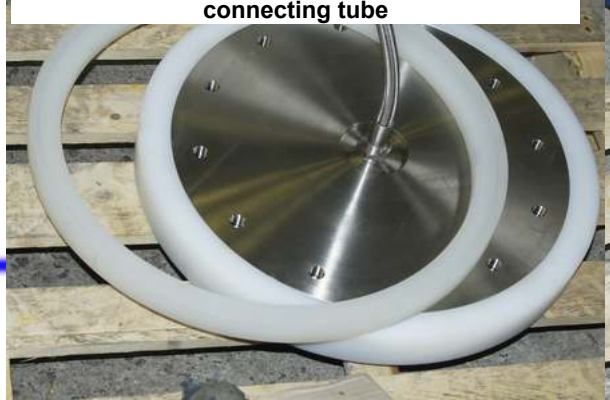

Fig. 11: Bottom flange pressed to the column tube

Fig. 12: Hydraulic cylinder on the upper flange

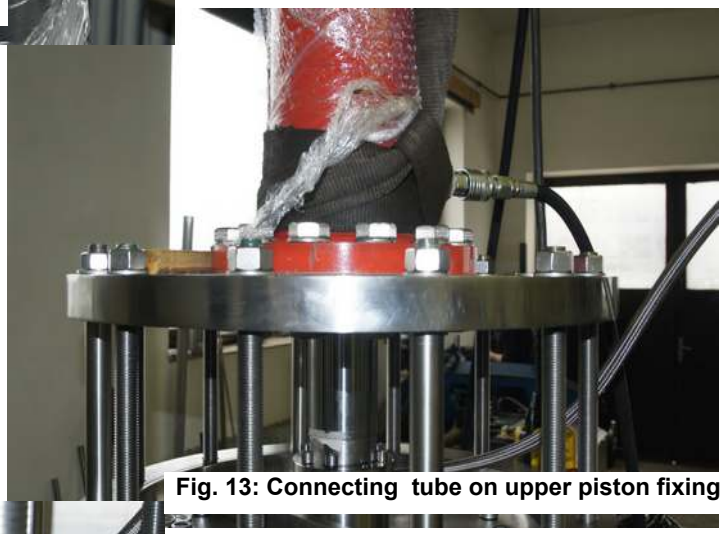

Fig. 13: Connecting tube on upper piston fixing

Caution:
For next operations is necessary to complete automatic hydraulic system and connected it to the electrical net.

A thin sheet of metal (3 mm thick) is laying than on the column flange and the piston by aid of a crane is put on and moved inside rods under the hydraulic piston. The hydraulic piston is moved slightly down to the connecting tube. The column piston is moved such way to have connecting tube under its lid and holes for bolts against each other. Lid of connecting rod is connected to the tube by 8 bolts M10x30 than. The piston is moved up and a metal sheet is released. Column is ready for packing.

4. Column packing and unpacking

Column is now filled with proper sorbent according manufacturer prescription. It is mixed in proper vessel with a solvent and then transferred to the column (output tube closed). It is necessary to be assured that sorbent suspension upper layer is laying cca 10 cm under upper column edge and column walls are clean us dry sorbent. The upper piston unit is now moved against the column immediately. As the liquid starts flow out of the imput tube, the movement is stopped for a moment, imput is closed and output opened. The piston movement starts again and it stops only on preset pressure. The column is kept in hold position few hours and the pressure is increased if recommended. Now the column is ready for use.

When unpacked, upper flange is released and removed (with support plate). A proper support is installed under the output piston and upper piston as well as sorbent bed is pressed down. Bottom piston is moved out of the column. It is removed and an reservoir for sorbent in inserted under the column. Sorbent is stepwise pressed out of the column. In most bottom position is upper piston cleaned (it is still a bit inside the column) and moved up again. The column is washed by a flow of liquid from upper side then. Finally a bottom piston is again assembled with the flange, inserted under the column, connected by bolts and pressed inside the column.

5. Column pressure test

Column fully assembled was tested by manufacturer on the oil pressure 200 bar (21 bar inside the column) and without hydraulic system on the pressure 28 bar, in both cases filled by water. Test protocol is enclosed.

Maximal allowed operating pressure is 20 bars!
The ratio between oil pressure and pressure inside the column is 10.

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. Manufacturing by

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