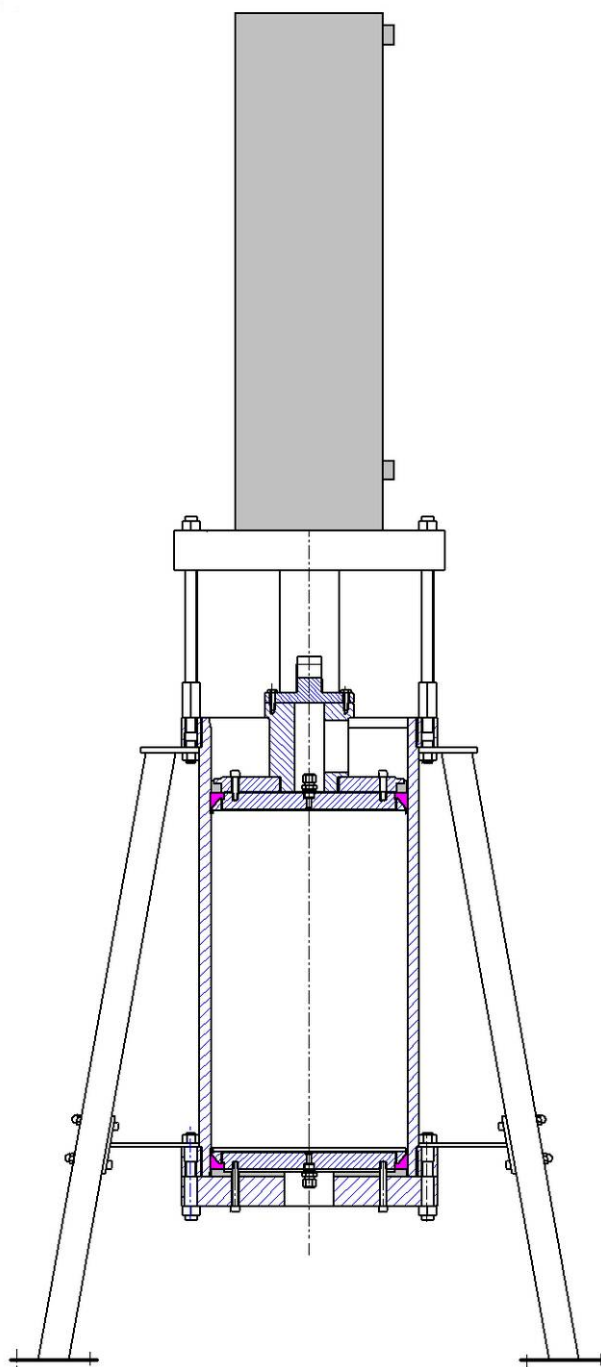


**PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY
SEPARCHROM PC01 400/800 DS**

**HYDRAULIC DEVICE
SEPARPRESS D100 EE 800**

**user
manual**



Stainless steel made liquid chromatography column SEPARCHROM PC 01 400/800 DC is designed for medium pressure preparative chromatography. It is used together with hydraulic axial compression device SEPARPRESS D100 EE 800, which press column piston to the sorbent bed and allow column packing by dynamic slurry method.

1. Description

Column design is apparent from Fig. 1 on front page. Column consists of a stainless steel made tube with inner diameter 399 mm and wall thickness 23 mm. Column has on both sides flanges with 20 holes each with M24 threads. In the column are from both sides inserted identical pistons made of circular stainless steel plates (36 mm thickness) which are separated on two parts – own piston and piston ring with pressed frit. Pistons are provided with UHMWPE/PP sealings. There is a flow distributor made of six layers of stainless steel nets. There are Poremet 2 frits (10 layers, 5 mm thickness) with pore size 2 µm as well. 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 a 1/2" flexed stainless steel tube, the elbow piece and PTFE tube 1/2" (12,7 mm).

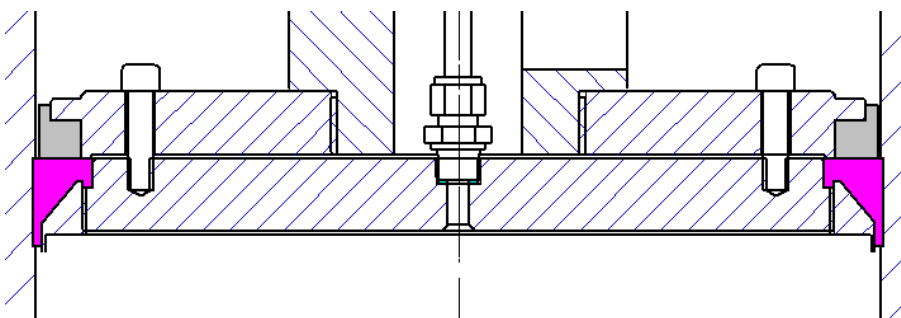


Fig. 2: Column upper piston cross section

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 support 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.

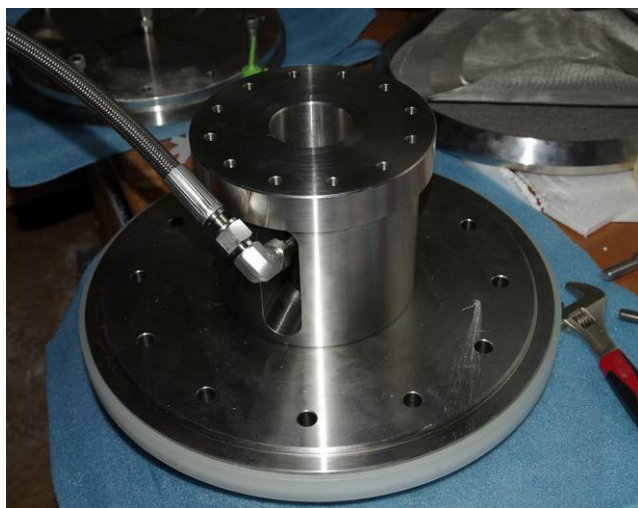


Fig. 3: Column upper piston with support, piston tube and connecting tubing

Upper piston design is more complicated (see Fig. 2). Its side being in contact with a liquid is the same as in the case of bottom piston. The sealing ring is again pressed by a PP made ring a the whole piston is attached to the piston support with connecting tube and connecting flange which is connected to the hydraulic piston.

Hydraulic dual action cylinder with maximal oil pressure 200 bar is situated beyond the column. Cylinder flange has the same shape and number of holes like column flange. Hydraulic piston is provided by a circular flange and it is connected to a piston tube (12 bolts M12x30). Own flange is screwed into hydraulic piston. The hydraulic cylinder flange is connected to the column flange by 20 thread rods M24. Some of them are provided

by with distance tubes (see Fig. 3), eight of them is composed from two parts as are used for leg connecting (see the chapter about assembling).

Hydraulic cylinder is connected by two high pressure armed 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.

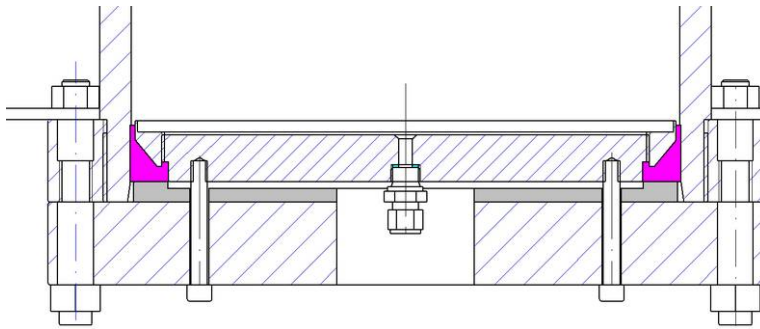


Fig. 4: Bottom piston with flange cross section

First of all is assembled frit ring with the frit to the piston plate with end fitting. Net distributors has to be placed to the piston plate before according following figures:

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 flexed tube (without elbow) its nut on piston side is tightened. Then is added a seal and a support plate with PP support ring and connecting tube is screwed to the support plate (see pictures). Support plate is to be connected by 12 bolts M12x45 to the piston.

Bottom column flange with piston is connected to column by 16 bolts M24x140 with insers and four threaded rods with thread M24 and M20 (see Fig. 4) for legs connection. The piston is pressed to the column by tightening flange bolts.

The column tube with bottom piston and output flange is lifted (lifting lugs are delivered) and placed to the backplate which height is more than 500 mm and which has sufficient breaking strength. Column upper flange is than equipped with eight special threaded rods (the length is 190 mm and there are two threads – M24 and M20 on them). They are ordered as four twins symmetrically with four (respectively two empty holes in between (see Fig. 14). Each twin has to be situated in front of the special bolt with M20 thread which is mounted on to the bottom column flange. The M20 threads on twin rods have to be situated on column tube side (bottom). Threaded rods has to protrude on upper side of column flange not more than 25 mm.

Four legs are connected to the upper column flange using mentioned M20 threads, inserts and proper nuts (see Fig. 8 and 10). Delivered distance tools are then inserted in between each leg and bottom flange. The are connected to the leg by two M12 bolts with nuts and to the bottom column flange by one M20 nut and insert (see Figs. 9, 11, 12). Column is now lifted down. Legs are not fixed to the floor yet.

The hydraulic cylinder is lifted up on the place where the column will be used using delivered tools (lugs, girdles and metal ring to fix girdless on upper side of the cylinder) and the column with legs is moved under the cylinder carefully. Delivered long nuts M24 are screwed to threaded rods which protrude from column flange.

The cylinder has to in position cca 140 mm beyond the column upper flange just in the axis of the column. The shorter connecting rods (8 pcs) are now pushed through holes in cylinder flange and screwed to twins of long nuts on column flange. A normal M24 nut is screwed to each rod after the pushing through as described. A second M24 nut is screwed to one rod (which is on the place where the column piston will be inserted finally) under the flange of hydraulic piston. Four long threaded rods are now pushed through hydraulic cylinder flange holes among twins of short rods. Each

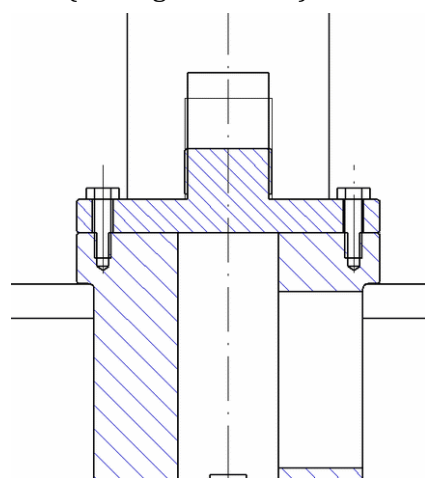


Fig. 6: Upper piston tube with hydraulic piston flange cross section

2. Column assembling

Column is delivered partially assembled, nevertheless here we described all steps which are necessary for assembling. These which are already done are written by italics.

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.

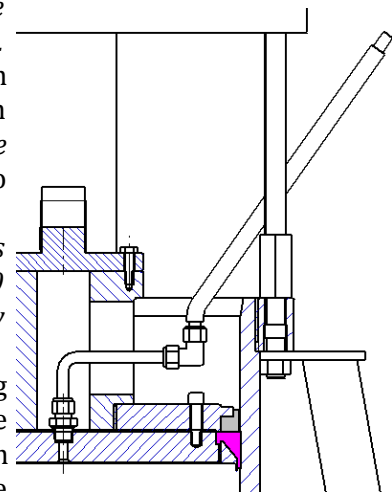


Fig. 5: Upper piston with output tubing, cross section



Fig. 7: Column leg assembled

longrod is equipped with M24 nut which has to be between column and cylinder flanges. Nuts are very near to column flange. Long rods are screwed into threads in column flange. Together there is 12 rods for cylinder elevation.

The system is now ready for final assembling, but column legs have to be fixed on the floor before using two holes in each bottom part of the leg (see Fig. 13), because now legs will keep the whole weight of the system.

Proper metal plate with sufficient breaking strength is placed on to the column tube hole to be able to keep hydraulic piston which will be now used for final elevation of the cylinder. The hydraulic hoses are to be connected to the cylinder and to the pump. The pump is to be connected to the electric net and a device allowing the movement of the piston up and down on the column place has to be installed.

The oil pump is started and oil piston is pushed out to be leaned to the inserted plate. Next movement of the piston causes the movement of the cylinder in upper direction. It is still necessary to secure the cylinder balance from upper side if possible by a crane or a rope. As the cylinder is moved up, the normal nuts on four long rods rods are screwed up to fix in each moment the position of the cylinder.

When final height of the hydraulic cylinder is reached (cca 305 mm between column and cylinder flange, next 8 long threaded rods are used, pushed through cylinder flange, but delivered distance tubes are inserted between flanges according schema on Fig. 14 (red rings). Threaded rods with distance tubes are equipped with M24 nuts from bottom side. The cylinder is slowly released to move down and to sit on distance tubes. Nuts on rods are tightened to fix cylinder on the place. Remaining nuts M24 are adeed and tightened too. One M24 nut on the short rod is screwed to most upper position and tightened too.

Distance between flanges is controlled to be the same on all sides. The cylinder is released from security holders and hydraulic piston is moved a bit inside the cylinder. The whole ssystem is controlled to stabil and safe. Now seven rods are removed again on the side where the column piston will be inserted (see Fig. 16) – three short rods from legs and four long rods staying between legs. Three long nuts M24 are released as well. The inlet is used to insert column piston.

The hydraulic piston flange is now screwed on (already with bolts) to the hydraulic piston which is lifted to the proper position. The piston is then moved to its highest position. A sheet of plastic (25 mm thick) is placed than on the column flange and the column piston unit (piston, support, piston tube, flexed tube with the elbow) is moved inside rods under the hydraulic piston flange. The hydraulic piston is moved carefully slightly down to the connecting tube. The column

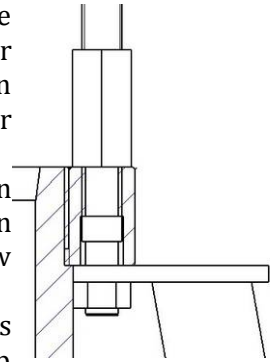


Fig. 8: Column leg - upper connecting section

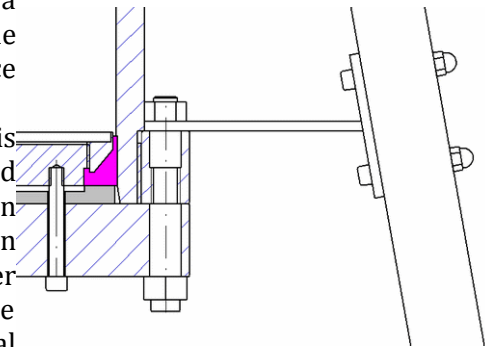


Fig. 9: Column leg – bottom connecting schema



Fig. 10: Column leg - upper connecting - detail



Fig. 11: Column leg – bottom connecting detail 1



Fig. 12: Column leg – bottom connecting detail 2

piston is positioned such way to have connecting tube under hydraulic piston flange and bolts on the hydraulic piston flange against holes on connecting tube. There has to be only a short slit between these two parts. The flange is connected to the connecting tube by 12 bolts M12x40 than. The hydraulic piston is moved up and a metal sheet is released. The hydraulic piston is very carefully slowly down and the central position of column piston against column tube is controlled. The piston is slightly moved down and down and controlled by three pairs of eyes from all sides to fit just to the hole in the column tube.

If the position is wrong (this is not common), hydraulic piston have to be lifted up, column piston removed, proper plate inserted under the column piston and using oil pressure the hydraulic cylinder has to be lifted a bit up and thin inserts have to be placed between distance tubes and flanges stepwise to change the position of the column piston.

When ready, the proof is repeated and then flexible tubing is connecting to the elbow on column input. Remaining distance rods are installed including distance tubes. All nuts are tightened. Column is ready for packing now.



Fig. 13: Column leg – bottom base

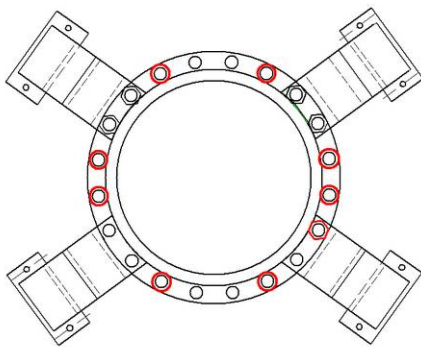


Fig. 14: Column with distance tubes – bird's view final arrangement

is laying cca 10 cm under upper column edge and column walls are clean without dry sorbent. The upper piston is now moved against the column ASAP. As the liquid starts flow out of the input tube, the movement is stopped for a moment, input is closed and output opened.

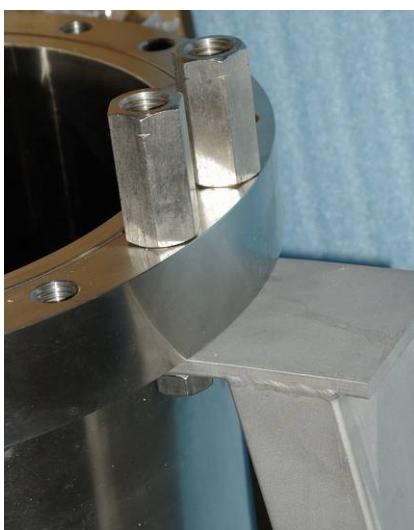


Fig. 16: Column leg with long nuts for distance rods

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 (column output tube closed) using a flexinle tube which is inserted into the slit between column flange and column piston.

It is necessary to be assured that sorbent suspension upper layer

is moved against the column ASAP. As the liquid starts flow out of the input tube, the movement is stopped for a moment, input is closed and output opened. The piston movement starts again with high output of oil pump and it stops only on preset pressure – when column is full, the oil pressure starts to grow fast. 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, a bottom flange is released and removed (with support plate). A proper support is installed under the output piston and the piston is pressed out by the power of oil on upper piston. An reservoir for sorbent in inserted under the column. Sorbent is stepwise pressed out of the column by oil piston. In most bottom

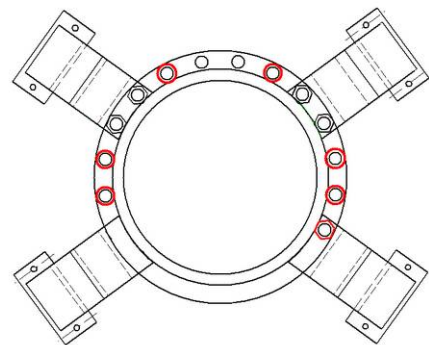


Fig. 15: Column with rods and distance tubes – bird's view for piston installation

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 than. Finally a bottom piston is again assembled with the flange, the whole inserted under the column, lifted up, connected by bolts and pressed inside the column.

5. Column pressure test

Column fully assembled was tested by manufacturer on the pressure 98 bar filled by water. Test protocol is enclosed.

Maximal allowed operating pressure in the column is 70 bar
Maximal operating pressure of oil is 200 bar
The ratio between oil pressure and pressure inside the column is 2,76

6. Hydraulic system connection

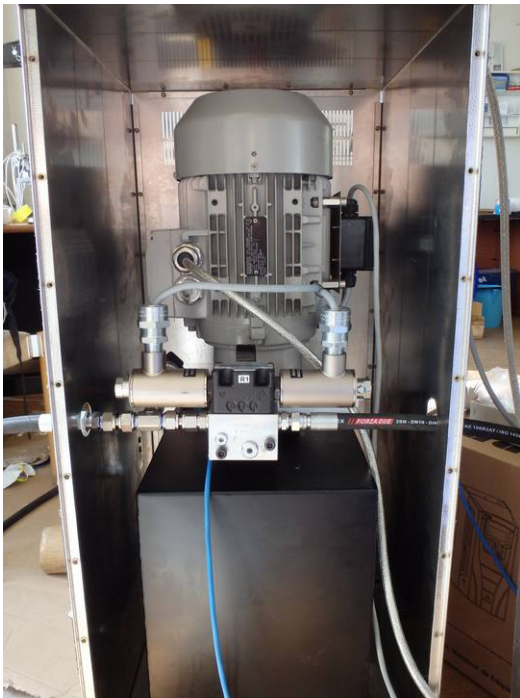


Fig. 17: Box with hydraulic pump

to remove upper front steel panel from the box for this procedure and open the input cap. Hydraulic hoses are to be connected to the pump outlets which are on sides of stainless steel box (see picture) as well. Opposite ends of hydraulic hoses have to be connected to the hydraulic cylinder.

Hydraulic oil pump is delivered as a block where own pump, oil reservoir, valve system and pressure sensor are integrated. The pump is driven by a 3 phase motor with output 3,6 kW. The motor has to be connected to the frequency changer, which is connected to the electric net 3x400V. Switching valves are driven by 24 V (DC) and have output 9,5 W. Installed pressure sensor (0-250 bar) has output 4-20 mA.

Hydraulic system has to be connected to electricity systems. Delivered frequency changer has to be connected to the net 3x400V. There is necessary to install cables between the changer and the motor according schema (see pict. 19). There has to be installed a logical member with pressure measurement and pressure limit setting and valves control and power supply. It is recommended to install a small box with bottoms near to the column having following functions: movement (hydraulic piston) up, movement down, pressure hold and emergency switch off.

Before starting the system it is necessary to fill the oil reservoir with oil which is delivered with the column. It is necessary



Fig. 18: Hydraulic hoses connection

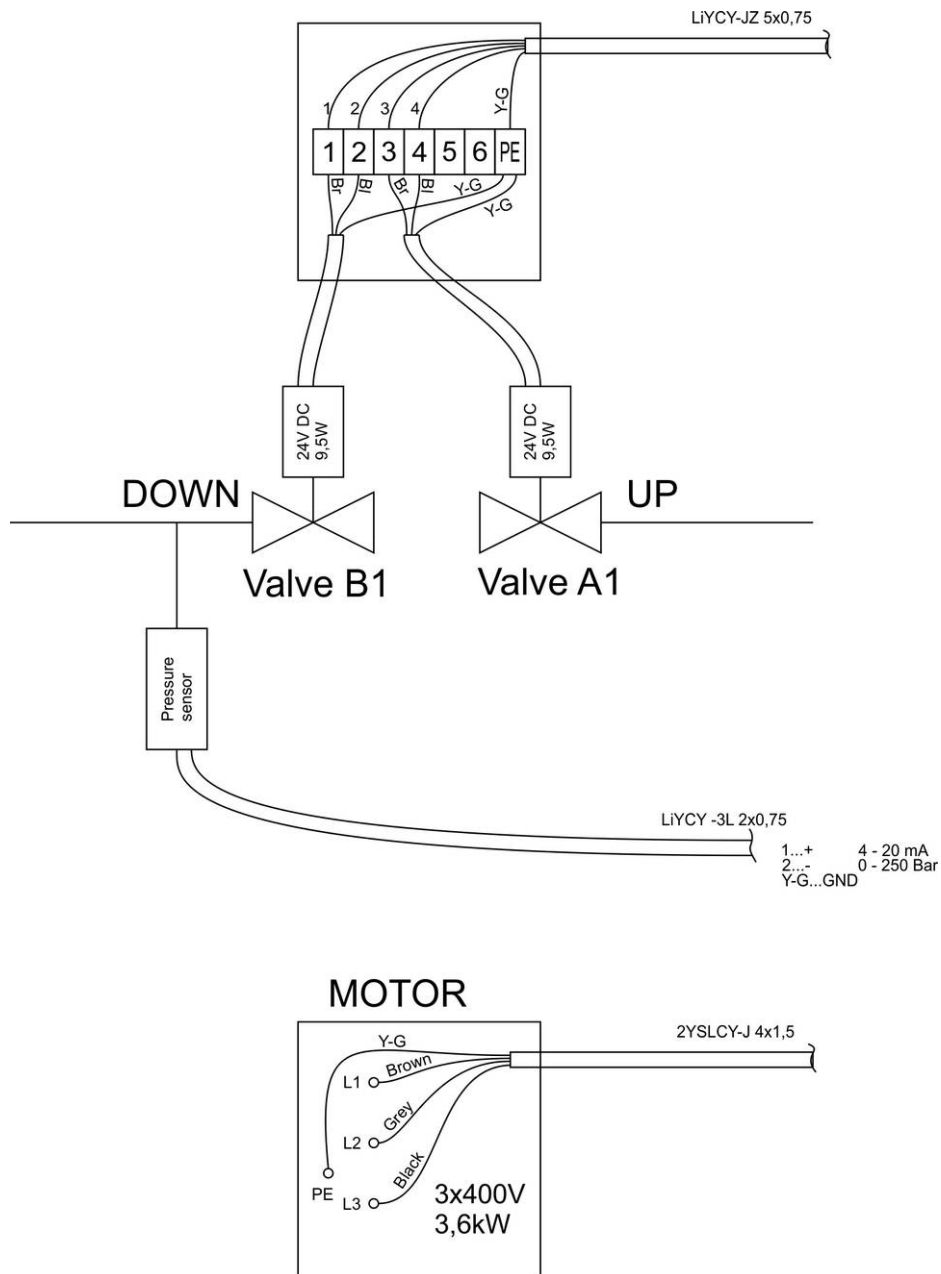


Fig. 19: Schema of hydraulic system connection

7. 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)..

8. Manufacturing by

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