PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY
SEPARCHROM PC01 150/900 DS

HYDRAULIC DEVICE
SEPARPRESS D30 EE 500 EX

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 part in connection with mobile phase are made of 316 (316L on the request) stainless steel according AISI. Columns are used for high performance liquid chromatography separations in instances where small rigid particles are used as column filling. Only stainless steel and ultra high molecular polyethylene (UHMWPE) are in contact with mobile phase. Columns are resisting to all common chromatographic solvents.
SEPARCHROM PC 01 150 columns with inner diameter 150 mm are designed for medium scale process chromatography and typically are working with flow rate 300 ml/min. – 1000 ml/min. depending on sorbent type and separation mode.
SEPARCHROM PC 01 150 DS column piston is pressed into the the column by a force which is generated by a hydraulic system.

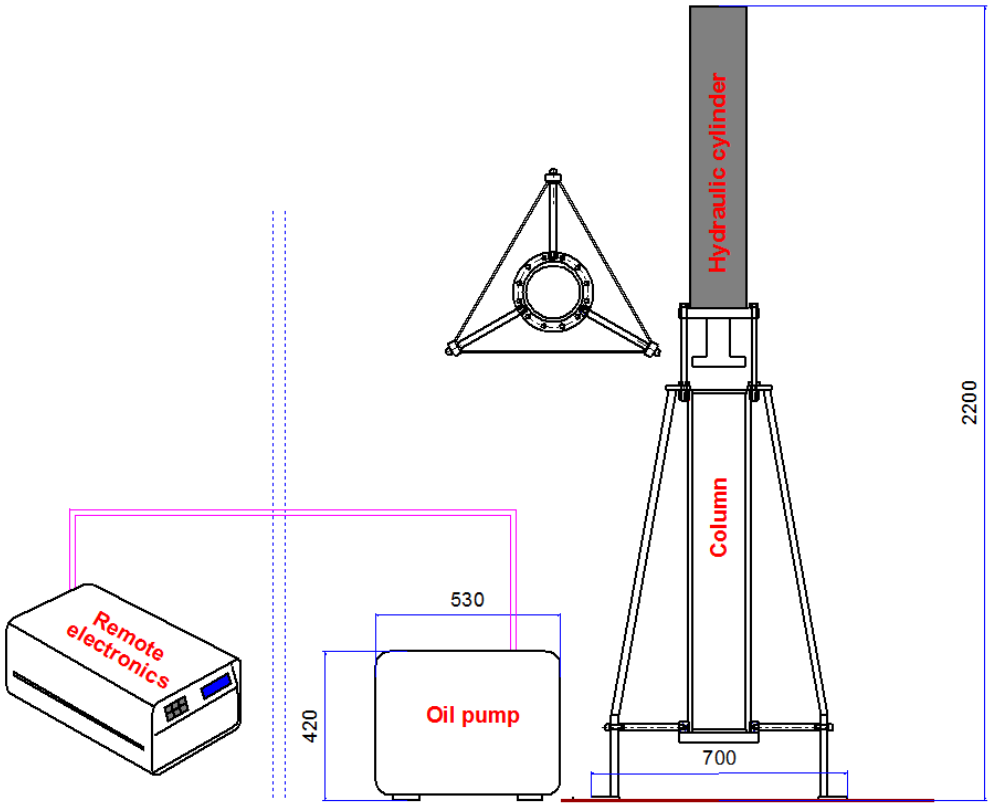

Fig. 1 PREPARATIVE COLUMN FOR LIQUID CHROMATOGRAPHY SEPARCHROM PC01 150/900 DS AND HYDRAULIC DEVICE SEPARPRESS D30 EE 500 EX

2. Column design

Typically the **PC 01 150** (see schema on Fig 2) column consists of tube, I.D. 150 mm (its internal surface is mechanically polished to attain high smoothness - Ra better than 0.3 um) which is provided by two stainless steel column flanges, each with 12 holes with M14 threads for the clamping bolts. The upper and bottom ends of the column tube are closed by identical stainless steel pistons with UHMWPE and polypropylene (PP) made seals. Each piston unit consist of five parts (see Fig. 3, 4):

- porous disc made of screen multilayer frit has 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 connecting
- own piston with liquid input (or output) and outer thread; it is screwed to a frit ring

- a set of stainless-steel nets for liquid distribution pressed from one side by a frit and from opposite side by a piston
- UHMWPE conical seal with polypropylene support ring (upper piston) or plate (bottom piston) which seals both piston against tube and piston against frit ring
- stainless steel support plate which is connected to a stainless steel connecting tube which second end is attached connecting part of hydraulic piston (on bottom side of the column is used column flange instead of a support plate).

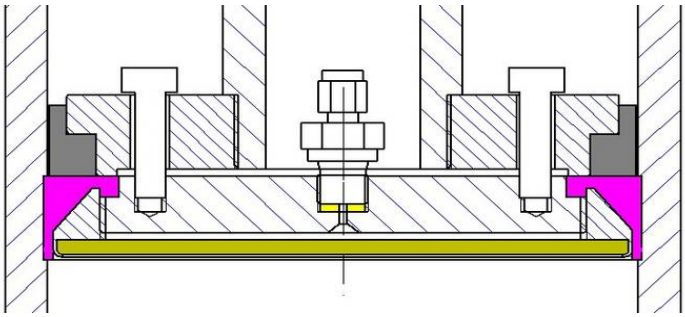

Fig. 3 Upper piston inside the column

UHMWPE piston seals is attached to the inner conical part of the piston unit (frit ring) and acts as pressure transducer. Its tightness increases when pressure is increased.
Porous disc covers nearly all tube cross section. This eliminates sorbent areas without full liquid flow. Net layers support the frit and make impossible a deformation under

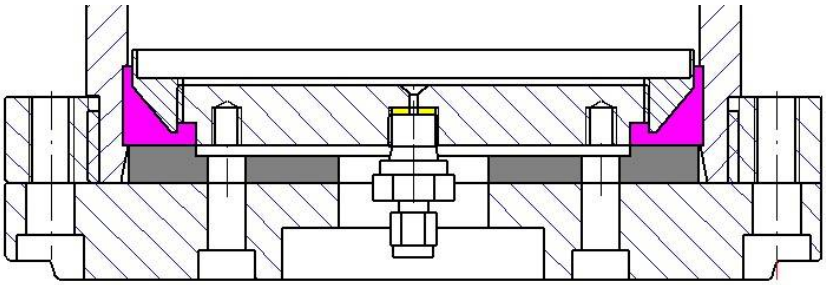

Fig. 4 Bottom piston with flange

sorbent pressure. They form perfect piston flow over whole column cross section.
The upper column flange is connected to a hydraulic cylinder by distance thread rods, long nuts M14 and M14 bolts.There are three special removable distance rods to insert the upper piston inside the column.

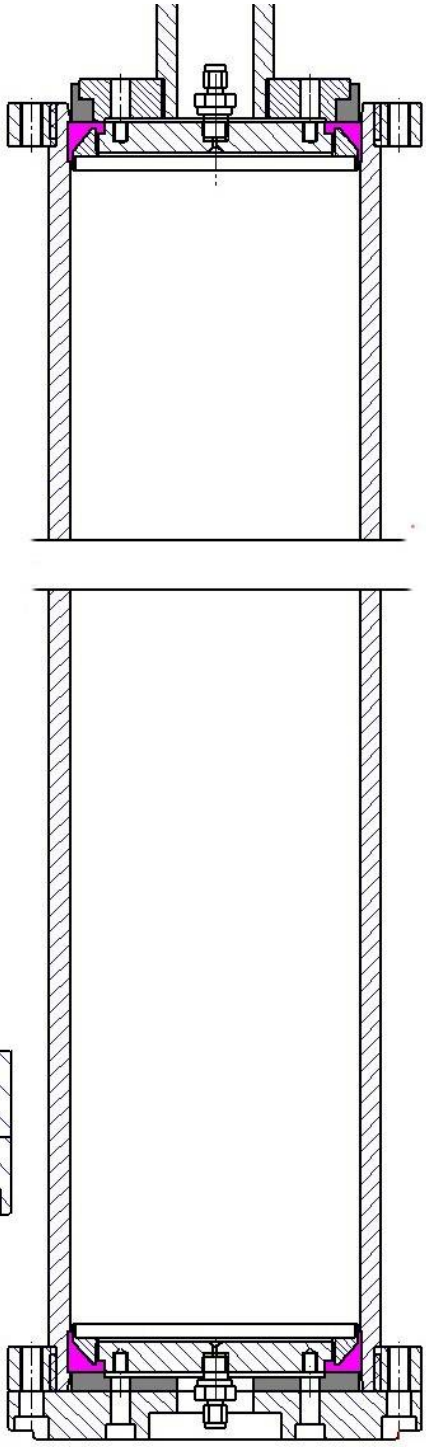

Fig. 2 Column cross secction

3. Column assembling

PC 01 150 DS 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. Instructions which are not used regulary are written in italics.

Piston assembling starts with distributor nets inserting into the frit ring. Finally the piston thread is screwed to the ring and tighten.

The piston is – when the unit is assembled – complemented with sealing ring and support plate + PP safety ring (upper piston) or with a PP plate and bottom flange (bottom piston). Both pistons have to be connected to input and output fittings using and sealed by PTFE tape. There is a central thread in upper supporting plate to connect the connecting piston tube.

Fig. 5 Frit with a ring

Fig. 6 Column piston with outer thread

Input pipeline (cca 1200 mm in length, 3,3 mm O.D.) is to be installed before. It leads through a piston tube and its side hole out of the column. It has to be fixed to the input fitting by a nut with ferrule. 8 bolts M8 has to be used than to connect support plate to the piston unit.

Output piston is connected by eight bolts to its flange as well. Both pistons are identical and can be replaced. Bottom piston with flange is now inserted to the column tube using special long bolts delivered with column. These bolts are after finishing the operation replaced by regular short bolts M14.

Column is than connected to three stand legs. Legs are connected to the upper column flange using three distance rods with M14 nuts. During this operation the column has to stay on some support, not on its legs! Column legs strengthening rods are now assembled among legs and column bottom flange (having three side holes with M12 threads for it. On the oposite side afre rods connected to each leg *via* bolts M14. Than is possible to put the column on own legs.

Fig. 7 Piston unit with seal – back view

Fig. 8 Piston unit with seal – front view

The hydraulic cylinder is picked up and positioned just against the column. Remaining distance rods (not these special ones) with distance tubes are not inserted into the space between column upper flange and cylinder flange and a space for remaining 3 rods is kept free. The assembled piston unit is inserted into the space among rods and pushed into connecting piece on the hydraulic piston.. Three bolts M8 on the connecting piece are tightened carefully to allow the piston unit to rotate.

Fig. 9 Bottom piston unit assembled to a bottom flange

Fig. 10 Bottom piston unit assembled to a bottom tube flange and side leg rod installation

Fig. 11Upper piston unit unit inserted into hydraulic piston suporting rods

4. Hydraulic installation

Fig. 12 Control box

Fig. 13 Remote unit

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 inserted into a stainless steel box, is equipped with 12 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.

Remote box and pump unit are connected by 4 cables. Their assembling is described in the Attachment 1 of this Manual.

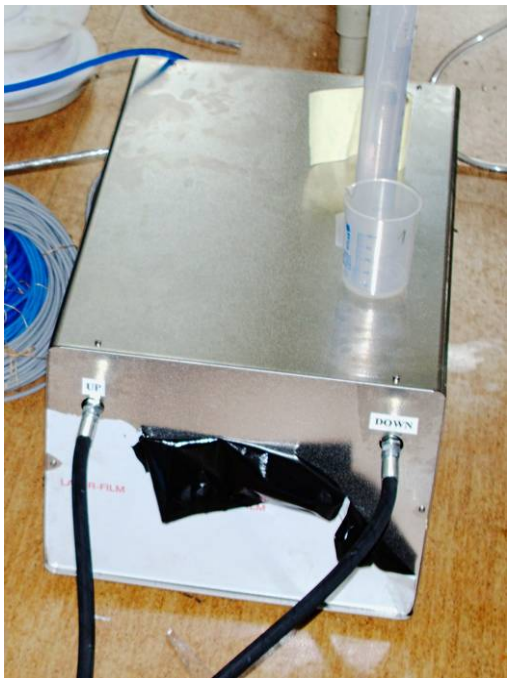

Fig. 14 Hydraulic pump unit

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.

Hydraulic hoses connecting

There are two hydraulic hoses delivered with the system. They are to be connected to the pump unit (see Fig. 14) using PTFE tape for sealing threads. Than they are connected to the hydraulic cylinder using fast connectors (see figs 15 and 16). The cylinder and the pump reservoir are filled with hydraulic oil and are ready for the use.

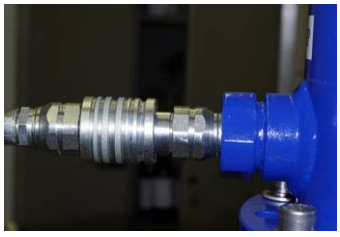

Fig. 15 Bottom piston connector

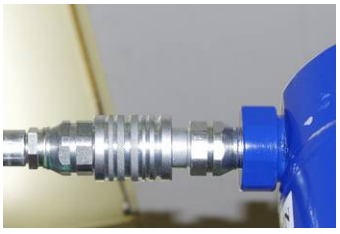

Fig. 16 Upper piston connector

5. 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 15 mm under the tube edge through a gap between the column and upper piston (see Fig. 11).
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.
Oil pressure is monitored on the remote manometer not to increase the pressure for which column and hydraulics are designed and packing instructions of the manufacturer.

Note: The pressure in the column is not equal to the pressure on the oil pump manometer. Column cross-section area is 177 cm^2 and hydraulic piston area only 154 cm^2. Thus the pressure in the column is 1,15 times lower, that the pressure of oil in the hydraulic cylinder.

In other words, having on the oil manometer 150 bar, there is 130 bar inside the column. For the column packing is recommended to use oil pressure 120 bar, but sorbent manufacturer instructions can be different.

Note: Columns having in type number letters Ex can be used in enviroment with explosion hazards, but their packing has to be done outside of such space. The upper column piston sealing has to be lubricated with a layer of powder graphite in order to decrease its electrical surface resistance. An alternative is to lubricate the surface with a viscous solution of salt in water or its mixture with organic solvents like PEG or glycerol.

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.

6. Column unpacking

The column output flange is released and sorbent is pressed out of the upper piston movement generated by hydraulic piston. An elongation rod (not part of delivery) has to be used for. The upper piston is moved to the most upper position and connecting piece bolts are released. Than is piston pressed a bit down and bottom part of elongation rod is connected. The piston is pressed into most bottom position and second part of elongation rod is screwed on the piston. Now the piston with the rod is pressed again with oil to move out all of sorbent and finally the piston itself from the column.

Fig. 17 Manual deassembling of bottom flange

In case of necessity to remove output piston and flange manually, the user can apply delivered tools. There are three special M14 bolts with small head and three stainless steel sheets 30x20 mm delivered. Standard flange bolts are removed at first. Than special bolts are screwed to the column flange from the opposite side and into the space between flanges are inserted small sheets (see Fig 16). Bolts are than tightened stepwise and the column flange is pushed out.

7. Column pressure test

Column fully assembled was tested by manufacturer on the oil pressure 200 bar (174 bar inside the column) using the delivered oil pump. To reach maximal prescribed pressure a manual oil pump was used and oil pressure was increased to the max. value 242 bar. Test protocol is enclosed in FAT documents.

8. Notes

Maximal temperature for column use is 80 °C.
Maximal temperature for hydraulic cylinder is 60 °C.
Piston sealing is to be changed when liquid leaks from the column (average frequency of the change is after 10 packing/unpacking cycles)..

9. Manufactured by

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tel 00420 242449669
e-mail: info@separlab.eu

Attachment No. 1

Connection of hydraulic pump unit and remote control box (Ex systems)

Both blocks are connected by cables K1 - K4.
K1: Cable of the motor (4 wires + shielding)
K2: Cable of valves (5 wires + hielding)
K3. Cable of pressure sensor (2 wires + hielding)
K4: Cable for small control box (8 wires + hielding)

Cables are connected in the control box. They are got throuh prepared holes on the back side of the unit (they are signed inside the box by numbers). K1 is connected directly to the frequency changer (output clamps + earth clamp, it is necessary to keep the order of phases to assure the movement of the oil pump in the right direction (see the arrow on the motor lid). K2 - K4 are connected to the terminal plates according description on wires (see schema a fig). Net cable is connected to the distribution plate 3x400V + N + PE 50Hz (independently on the order).

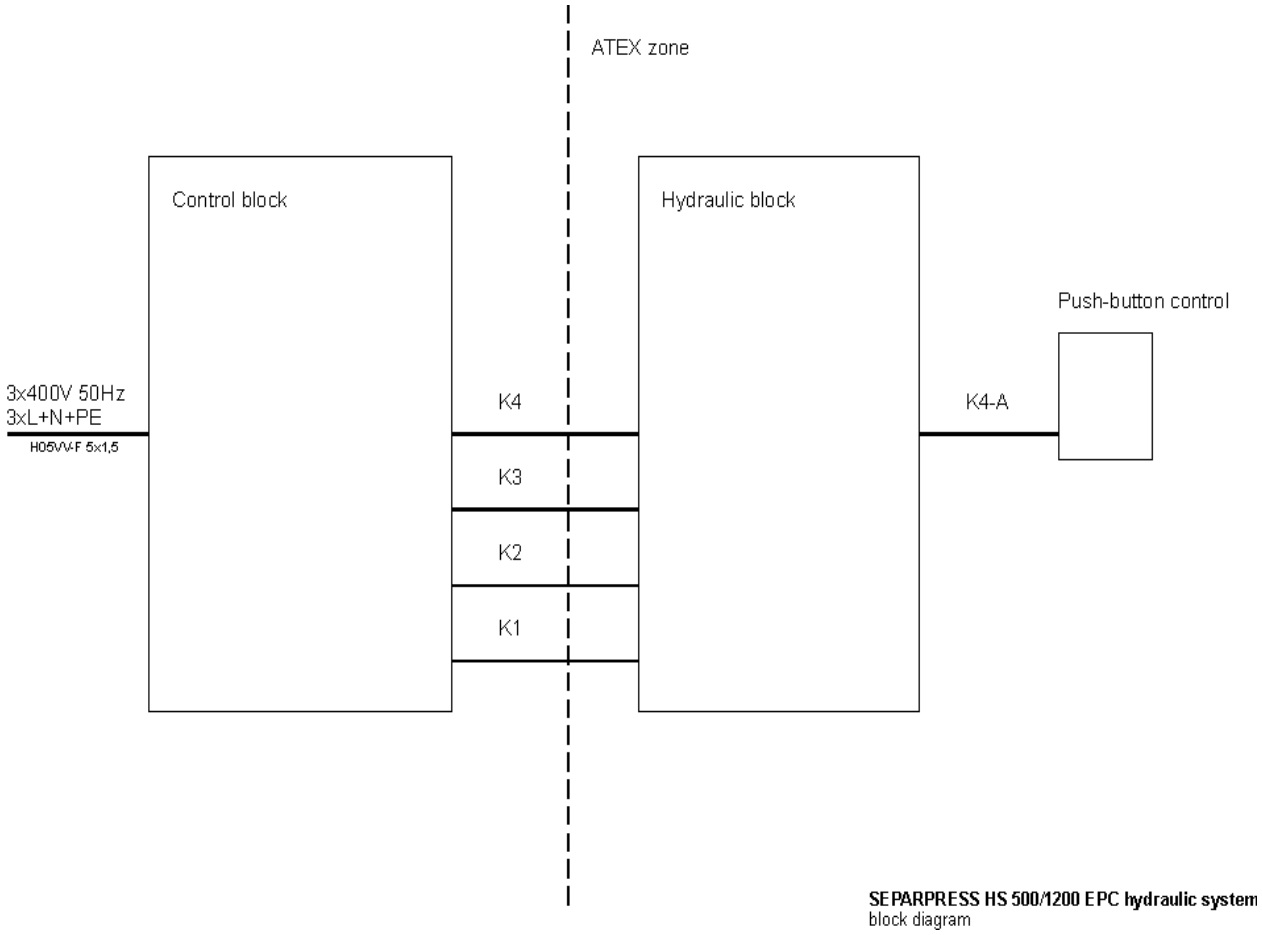


Fig. 1 General schema for connections

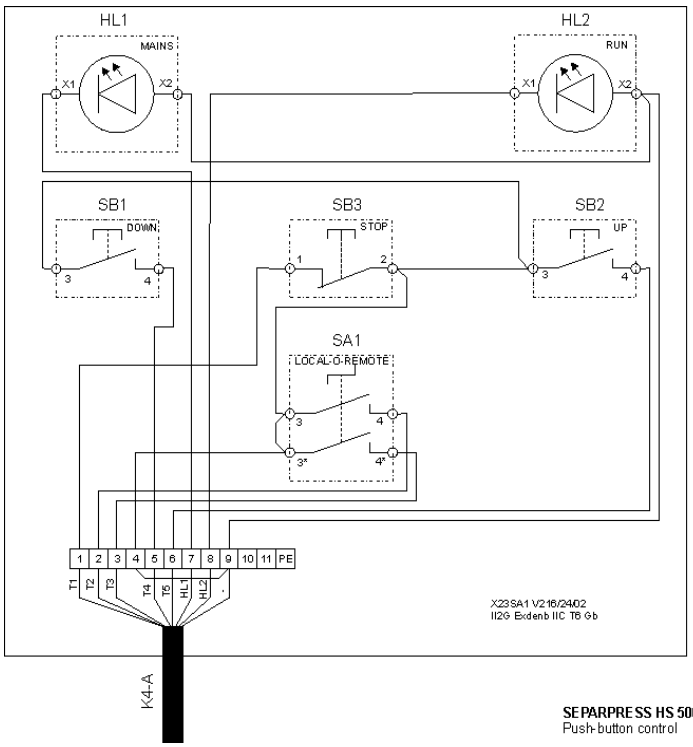


Fig. 2 Control box connecting schema 1

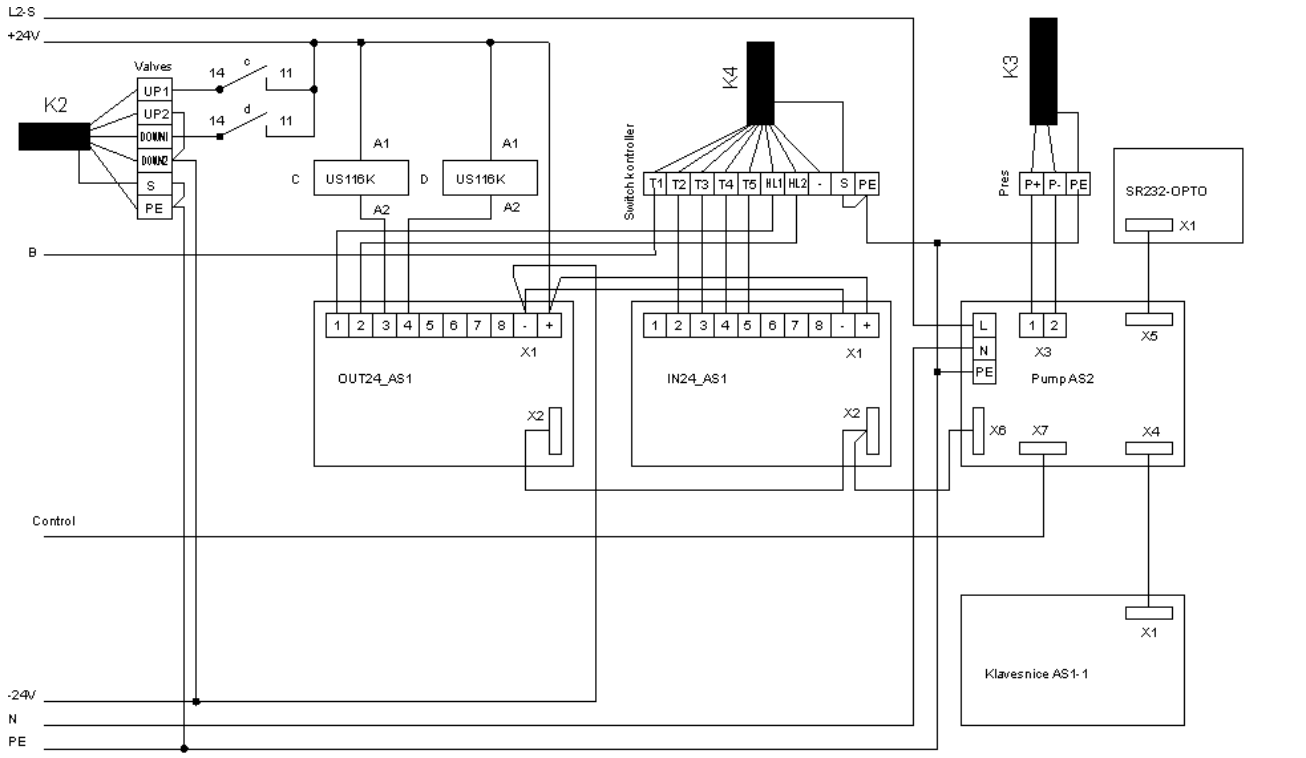
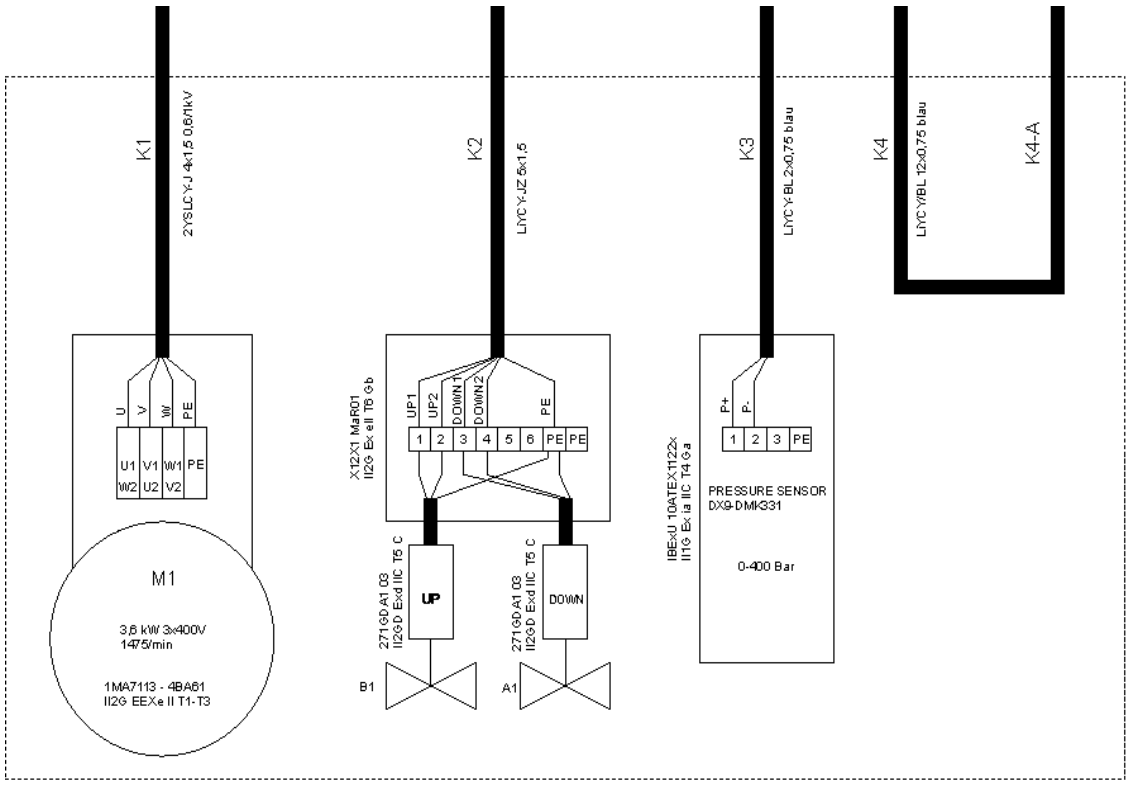
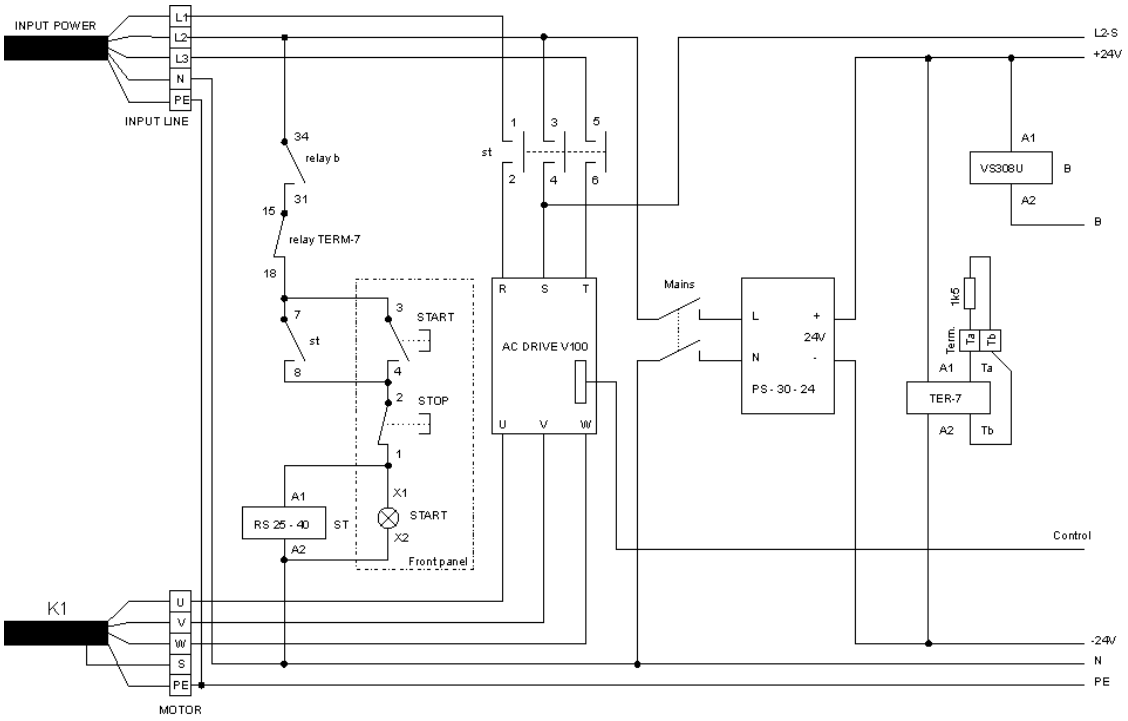


Fig. 3 Remote control unit connecting schema 1



SEPARPRESS HS 500/1200 EPC hydraulic system
hydraulic block

Fig. 4 Hydraulic pump unit connecting schema



SEPARPRESS HS 500/1200 EPC hydraulic system
Control block - power

Fig. 5 Remote control unit schema 2 - power input

Fig. 6 K2 cable connection in remote unit

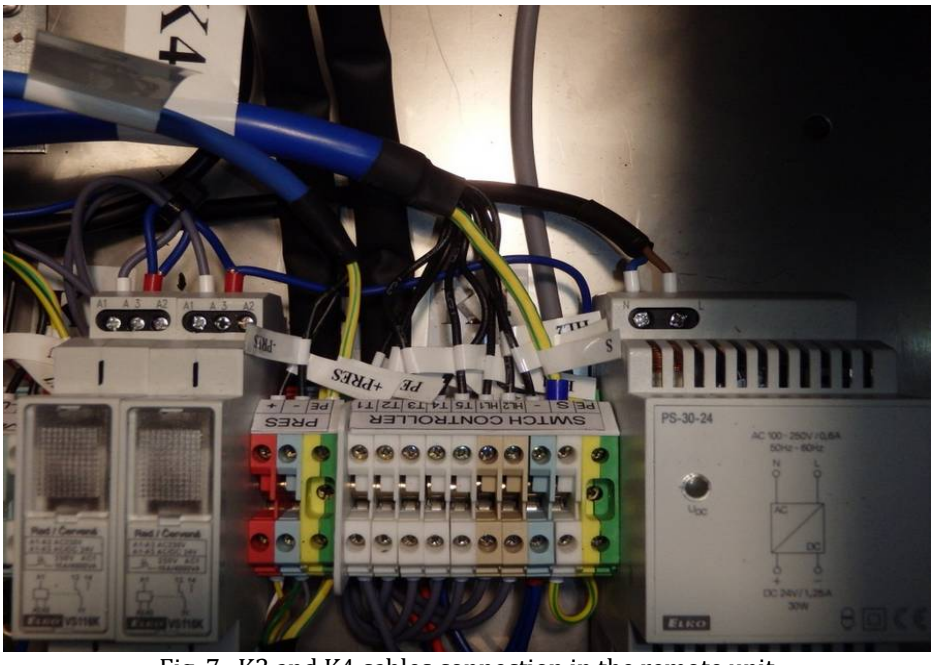


Fig. 7 K3 and K4 cables connection in the remote unit

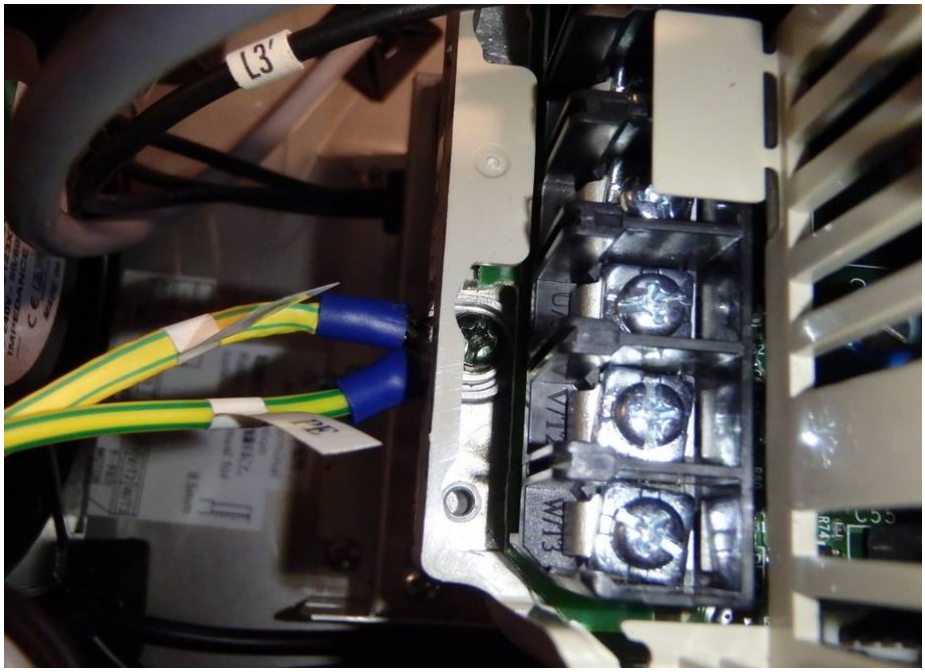


Fig. 8 Earth wires fixing in frequency changer

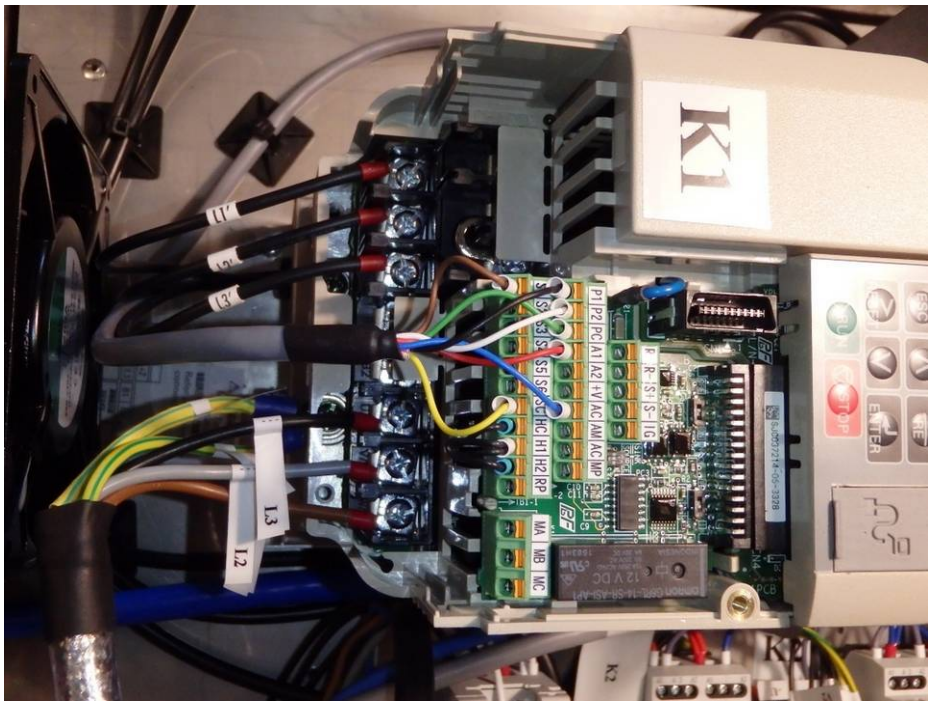


Fig. 9 K1 cable installation in frequency changer

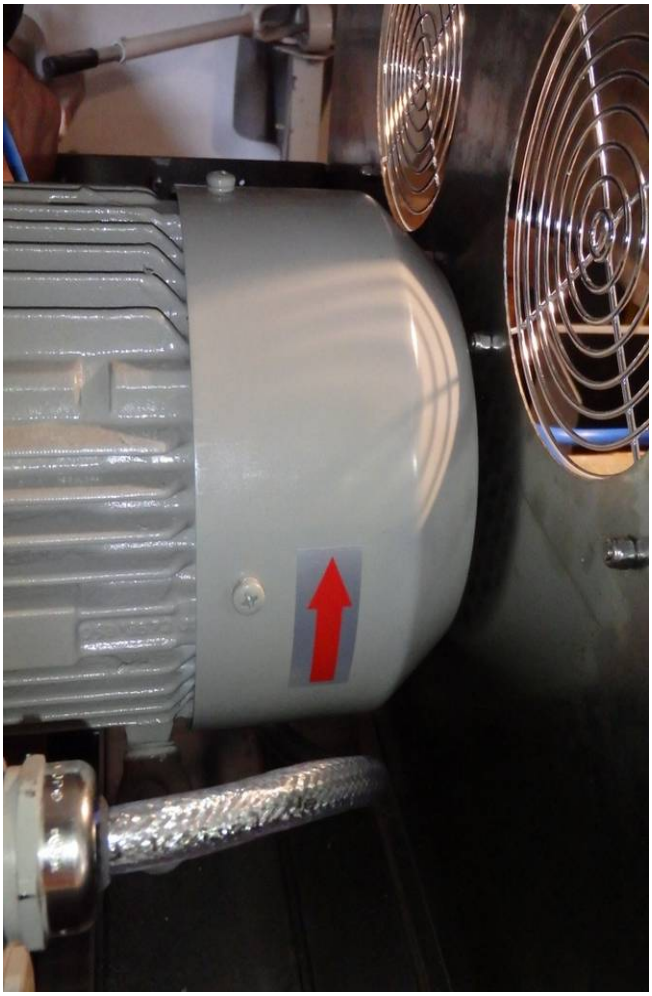


Fig. 10 Motor rotation direction arrow

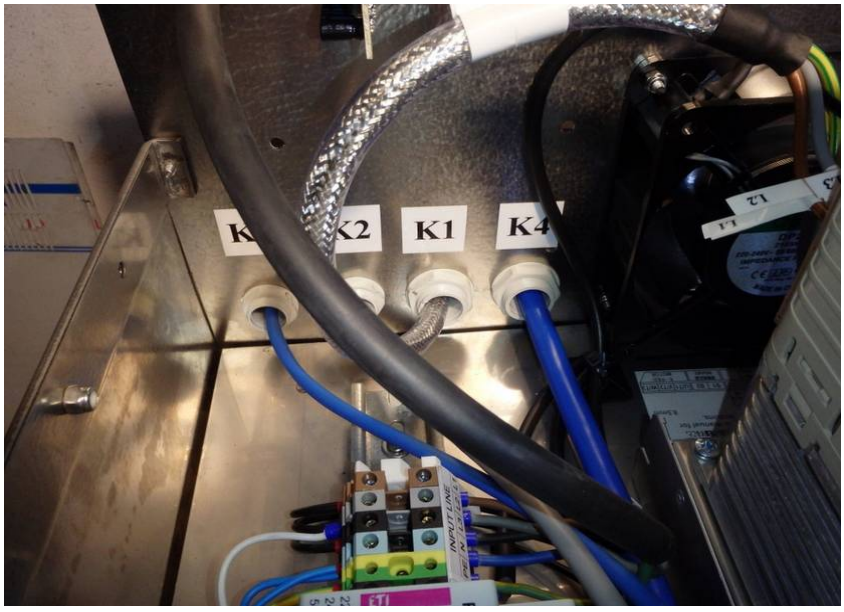


Fig. 11 Back of the remote unit with cable holes

Attachment No. 2

Connecting parts of the column

1.	Thread rod M14x200	9 pcs
2.	Distance tube l=142 mm	6 pcs
3.	Nut M14 3d	12 pcs
4.	Bolt inner hexagon M14x60	12 pcs
5.	Bolt inner hexagon M14 mod	3 pcs
6.	Distance rod 10.006	3 pcs
7.	Stand rod 30.002	3 pcs
8.	Nut M14, 0,8d	6 pcs
9.	Insert M14	22 pcs
10.	Insert M12	6 pcs
11.	Bolt M12x40	3pcs
12.	Bolt inner hexagon M8x35	12 pcs
12.	Bolt inner hexagon M8x40	12 pcs