separlab

LIQUID CHROMATOGRPHY PUMP

SEPARTRIX PP03 C

INSTRUCTION MANUAL

1. Use and Function of the Product

High-pressure pump PP03 C is designed for pumping corrosive liquids, against which are resistant stainless steel AISI 316, polytetrafluoroethylene (PTFE), high density polyethylene (HDPE), polyetheretherketon (PEEK) and polycrystalline corundum ceramics. Using three parallel cylinders for pumping with overlaping of displacement periods in 120° (without any delay and maximum stroke) leads to a diminishing of pressure pulses in the hydraulic circuits and in most cases the pump can be considered as pulsless. It is therefore extremely suitable for an use in preparative high performance liquid chromatography. However, it can be used generally anywhere, where precision, high pressure and inertness are required.

2. Product Description

Pump PP03 C is a piston pump equipped with one pump head which integrates three pumping cylinders with pistons, their seals, check valves and inlet and outlet fittings . The pistons are driven by three cams¨mechanics (Fig. 1). All their moving parts are stored in the ball bearings. Cam connecting rods are fitted at the end by small ball bearings that move in grooves formed by two steel combs (not visible on the drawing). Pistons are connected to cams through especially shaped steel holders which are fixed to front surfaces of connecting rods. The U shaped holes make the installing and removing of pistons more easy. Movement of the shaft is provided by a asynchronous engine through a worm gearbox (1:5) with high efficiency. The motor speed is controlled by a frequency changer with vector control.

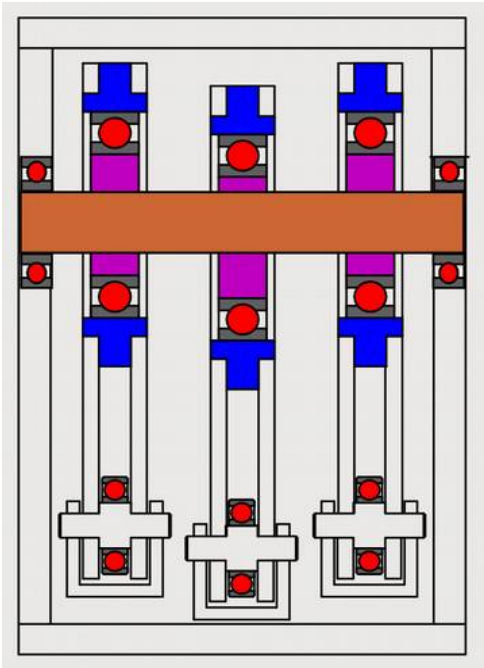


Fig. 1: Cam mechanics

Pumping head (Fig. 2) is made of stainless steel with precision drilling holes. The pistons move in a cylindrical recesses. The first part of each recess is adapted to the diameter of the piston and is used as pumping cylinder. Second with a substantially larger diameter is designed for a plastic sealing rings and compression spring made of stainless steel. The third part is provided with a thread and a screw with hole for the piston is inserted into, serving to guide the piston and to generate a power required to press seal rings. Between the through-bolt and the spring is placed secondary seal (Fig. 3). With this seal can be through-holes from the top of the head springs area washed to remove the liquid and keep unwanted products out of the piston space (additives such as buffer crystals).

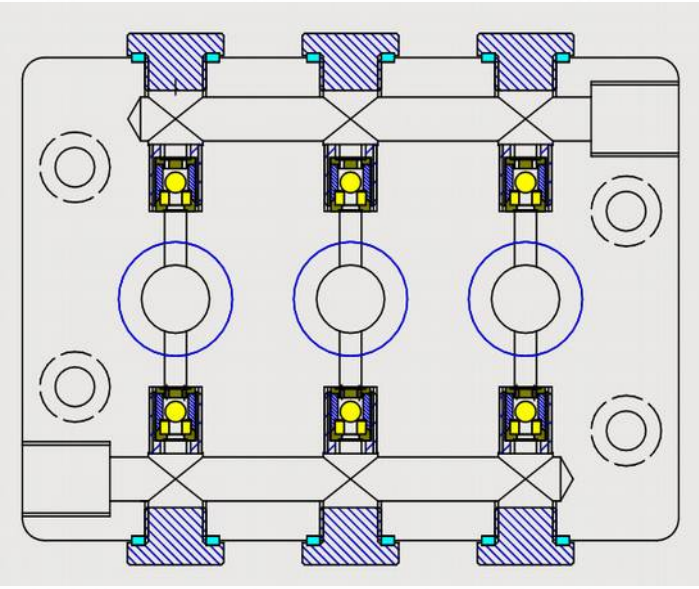


Fig. 2: Pumping head cross section

Main three seal rings are conical, made of special abrasion and chemical resistant high-molecular polyethylene. Seals are pushed to the edge of the cone shaped front part of a cylindrical sealing groove.

Pistons (Fig. 4) are made of cut and polished stainless steel cylinders of outside diameter 20 mm. They are equipped on the cam end by a rotational grove which fit to U holes in piston holders in the cam box. Piston are coated by polycrystalline carbon layer which is extremely hard.

Fig. 3: Piston with sealings, spring and through bolt

Pistons have three point guidance in order to keep precisely its way. Firs is a PEEK ring in the outer wall of the cam box, second is a PEEK ring on the back side of through-bolt and third is a PEEK made coil on the front of through-bolt.

Inlet (Fig.8) and outlet (Fig.6) valves, are made as compact cartridges and are exchanged as a whole. The outer part of steel housings are equipped with M12 thread. Inside outlet housing press PEEK and PCTFE made parts ceremic seat. The ceramic ball is inside to open and close the valve. Input valves are made simillary, but with with larger hole, provided by specially filled PEEK seats and stainless steel balls.

Valve cartridges are screwed from upper and bottom side of the pump head after removing the plugs with hexagonal heads (Fig 7). To allow screwing, cartridges are provided with hexagonal recess for key.

On both sides of pumping head are connecting standard Swagelok fittings for connecting of 3/8" (9,6 mm) O.D. tubing. Output is mase as an elastic armed PTFE tube and leads to the bypass valve block which is combined with pressure measurement gauge (Fig. 9). Cylindrical block has an input from the head in the upper part, on left side is a bypass output (1/8" (3,3 mm) O.D. Swagelok fitting) and on the right side is a 1/4" O.D. Swagelok output fitting.

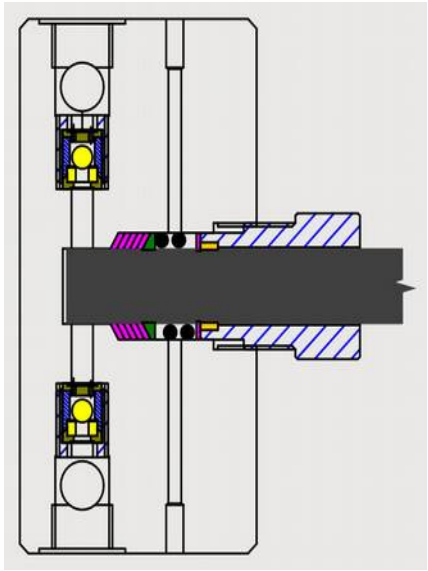


Fig. 4: One cylinder unit

Fig. 5: Pumping head parts

Pumping head protrudes from the front panel of the pump and is easily removable (four screws). From the back of the head are available through-bolts heads (Fig. 7) to adjust the pressure sealing spring forces.

When removing the head is firstly necessary to release partially through-bolts and to diassemble connecting tube on the bypass block. Then is recommended to start the pump movement for small flow rate (cca 150 ml/min.) and use a force to move the head out of the case. After removing the piston head the pump has to be stopped immediately.

To remove pistons is necessary to open pump box, to release front panel and to make entrance to front panel of the cam box. Front panel of the cam box has to be removed al well and finally pistons are removed from U shaped recesses on the cam ends.

When assembling pumping head back it is recommended to use auxiliary four longer M8 screws. Head is pressed onto pistons manualy, longer bolts are inserted and tightened to press head against pistons. Than are auxiliary bolts removed and regular bolts are inserted and tightened.

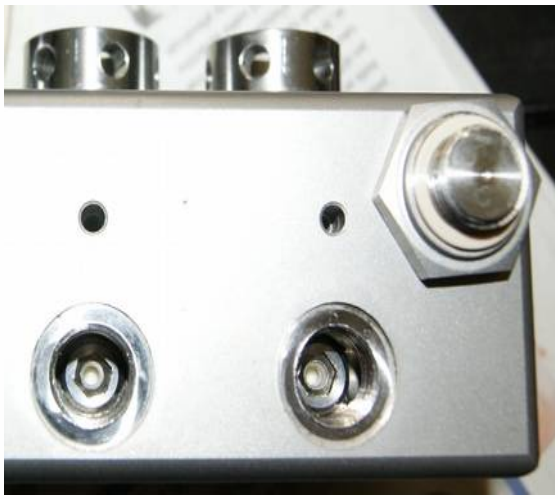


Fig. 7: Hexagonal caps and valves inside head, back side tighting screws

The pump housing is made of stainless steel. It consists of front (angled) and the rear panel and two U shaped profiles. After removing the upper U-profile (the side bolts and bolts of both panels have to be released) are accessible all parts placed inside the case. Cam mechanics, motor and frequency changer are located on the rugged construction made of of welded stainless steel profiles.

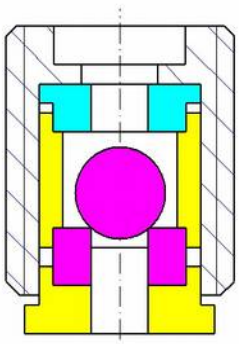


Fig. 6: Displacement check valve

On the front panel is mounted electronic display board. Frequency changer is located on the back side of the cam mechanics box. Rear panel (Fig. 10) contains the power supply cable going through. There is a power switch as well.

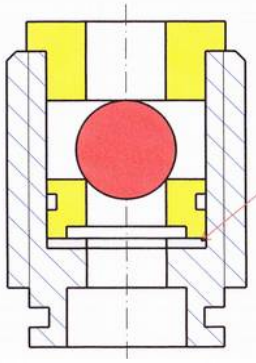


Fig. 8: Suction check valve

It is recommended to place mobile phase reservoir to the most upper place and the pump to the low place. Even all tubing in the pump system are designed to work well with maximal flow rate 3000 ml/min., such arrangement avoid problems with cavitation in pump cylinders.

Fig. 9: Bypass block

3. Basic data of the pump

Number of pistons:	3
Piston diameter:	20 mm
Piston stroke:	10 mm
Cycle frequency:	10 rpm - 330 rpm
Flow rate range:	100 ml/min. - 3000 ml/min.
Maximal pressure	70 bar
Pressure limit:	3 bar - 70 bar
Pressure hysteresis:	1 bar -15 bar
Pump input:	650 W
Dimensions (depth x width x hight):	610 mm x 385 mm x 280 mm
Weight:	28 kg

4. Pump control

The pump can be controlled independently from the keyboard on the front panel or externally via RS232 serial port. When external control is active, the keypad is locked and it is impossible to control the keyboard just to view the specified parameters. Always works but the STOP button.

Left keyboard is used for pump control, the right to control the gradient. For the transition from control to control the gradient pump is necessary to press ENTER on the keyboard and the right to go to the pump control is necessary to press ENTER on your keyboard left.

Fig. 10:Pump rear panel

Description of keyboard

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
Key START / STOP: is used for starting and stopping the pump
Note:this key is functional even if the pump is controlled from an external source.

The order of display items for controlling the pump:

- Flow
- Pressure
- Flow Settings
- Pressure Settings
- Hysteresis Settings
- Password Settings (next items are accessible only when entering a password)
- Settings Corection flow
- Zero pressure settings
- Max pressure settings

Example of operation

After switching on the pump is set to display the first item. In the upper right corner shows the status of the pump (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 start the pump 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) manual control is disabled with the command on the serial line

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

Next pressing the START / STOP stops 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 switsching, 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 presssure limit + hysteresis and starts again when pressure is going down set pressure value - hysteresis.

Settings of values - notes

Use the F1 and F2 on the left keyboard next values can be set using the "arrow up" or "arrow down" buttons. If the arrow button is held down longer time (about 0.8) values begin to grow more and more fast. Then confirm set values by pressing ENTER is the moment when old values are rewritten into the device and executed. In the upper right corner of the display for about 1s displays ENTER in such case. If not, it is probably blocked due external control of transcription. If for some reason do not want to write the changed value to the device, simply press F1 and then F2 to get the item to another screen and back again and loads the initial value is set.

External control is described in ECOMAC software manual. It is possible to set and read everything as in the internal control, in addition it is possible to block the keyboard controls.

Calibration of the pump

Performed after entering the service password on the left keyboard. Attention: in these settings change the items set important parameters pumps! The first item is the "Settings Corection flow" which can be used for validation of the instrumetn when fine correction of flow rate is necessary between of + -10%.

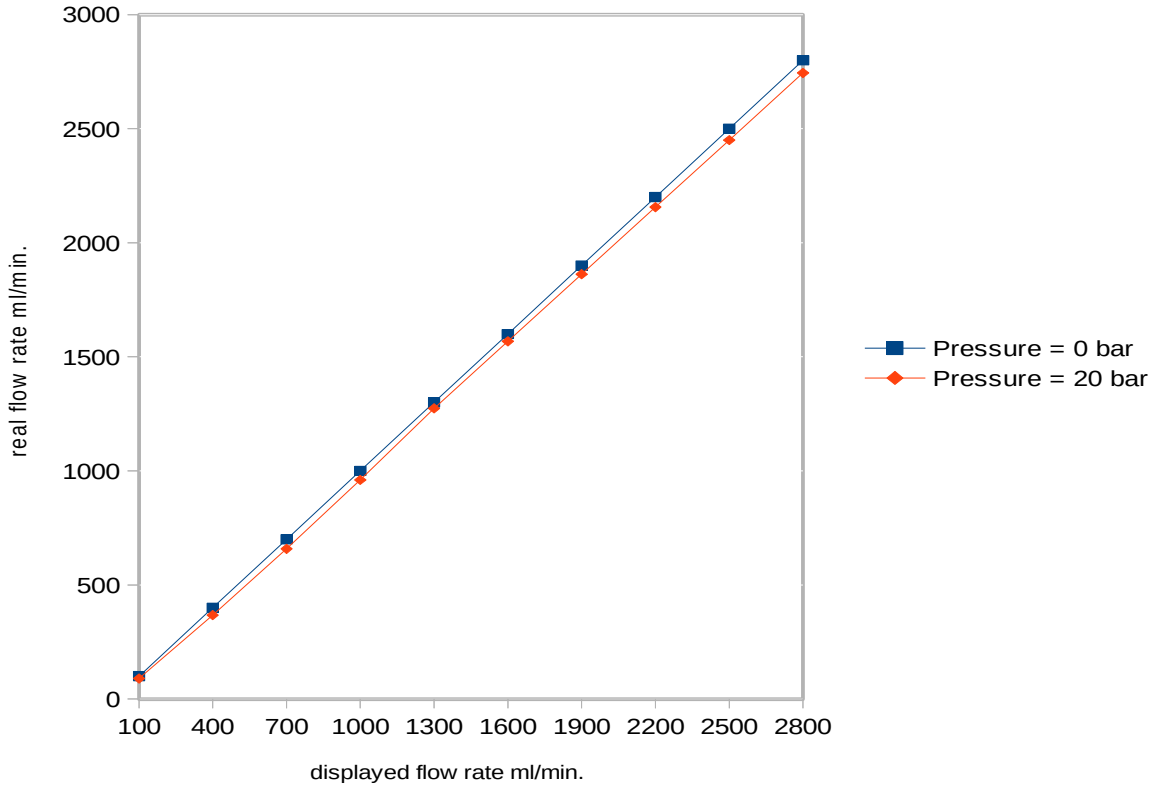
The next three items relate to the calibration gauge.The first is the "Settings Zero pressure". To executre it, the pump has to be in pressureless state. When figures on the display stabilize, press ENTER. The transducer value for pressure 0 bar is recorded. Numerical data are raw, unadjusted data A / 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.

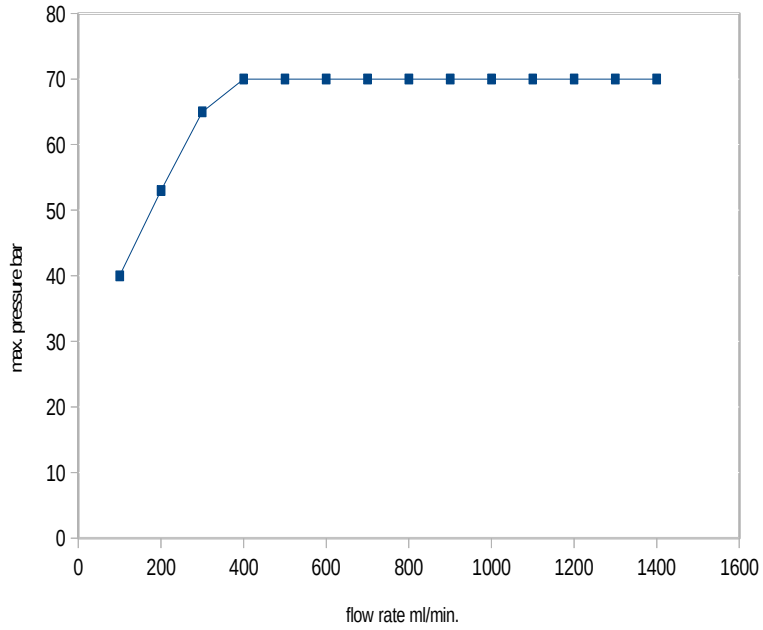
5. Pump use

Pump PP03 C can be used in all liquid chromatography modes where are its properties (flow rate range, pressure, low pulsation, inertness) advantageous. The pump has wide range of flow rates thans to the modern frequency changer used. Nevertheless it is necessary to take into the account that pumps provided with frequency changers have smaller torsion moment in low flow ranges (small speed of motor rotation). It means that under high pressure can be observed some differences between displayed and measured flow rate without and with the pressure on the output. In following graphs is possible to see both maximal pressure for the pump use and flow differences with and without the pressure.
When working with high flow rate a good feeding of the pump with liquid has to be guaranteed. It is at first a function of tubing diameter that has to be larger for large flow. Generally recommended tubing diameters and lengths are shown in following table, even they depend on liquid viscosity and reservoir position. It is recommended to set the reservoir(s) to higher position then is the pump position. At about 1 m vertical difference is a best solution.

PP03 C flow rate with pressure



PP03 C maximal pressure



Pump PP 03 C is equipped with high quality ball valves in each cylinder. Ball valves are nevertheless sensitive to liquids with high viscosity. It is thus not recommended to pump liquids with viscosity higher

than 10 cP. It is important in case of sample dosing too as some concentrated solutions can be quite viscous. It is recommended in such case to use lower flow rate for sample dosing and not to reliance on set flow rate (better is to measure injected sample volume on the column output).
Ball valves are sensitive to any solid particles or other impurities (even very small) in pumped liquid. It means that all such particles have to be eliminated by filtration or other proper method. When problems with ball valves even occur (low flow rate, pulsation, pressure changes) it is recommended to remove valves from its position, put them into ultrasonic bath, keep them inside few minutes and them by a syringe flush them with proper liquid (similar to this in use) and dry them on 60 °C two hours.

SEPARTRIX PP 03 input tubing according different flow rate

Tubing type	**Maximal flow rate ml/min.**	**Conditions**
Tubing 1/16“ (1,6 mm) O.D., 0,7 mm I.D., SS, PEEK, FEP	10 ml/min.	1 m tubing length, no overpressurising, 1 m vertical difference between pump head and (upper) reservoir level
Tubing 1/8“ (3,3 mm) O.D., 2,1 mm I.D., SS, PEEK, FEP	150 ml/min.	1 m tubing length, no overpressurising 1 m vertical difference between pump head and (upper) reservoir level
Tubing 1/4“ (6,35 mm) O.D., 4 mm I.D., SS, PEEK, FEP	600 ml/min.	1 m tubing length, no overpressurising 1 m vertical difference between pump head and (upper) reservoir level
Tubing 3/8“ (9,6 mm) O.D., 8 mm I.D., SS, PEEK, FEP	2400 ml/min.	1 m tubing length, no overpressurising 1 m vertical difference between pump head and (upper) reservoir level

6. Additonal installations

A pump PP03 C can be equipped by two holders on both sides of the front panel where either recyclation, reversation or injecting valves can be situated (not a part of standard delivery) ans is shonw on Fig. 13:

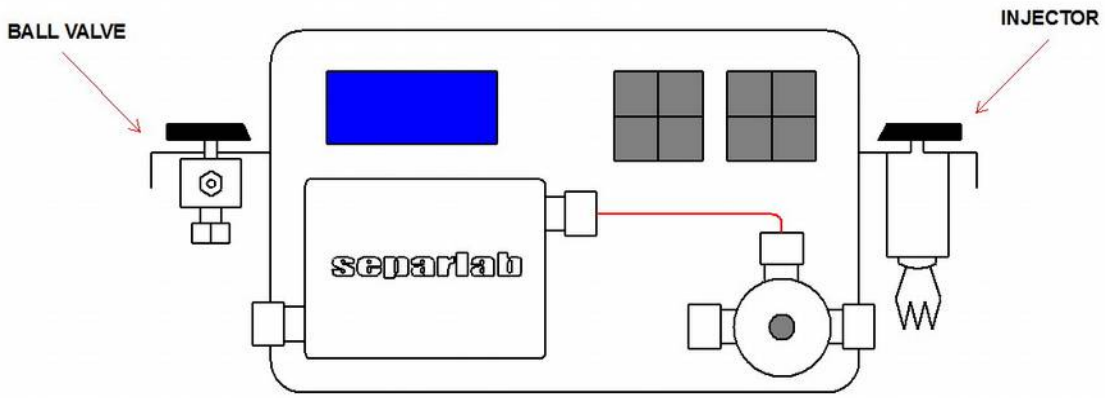


Fig. 13 Pump with holders

7. List of connections

Head input	Swagelok 3/8“, two part ferulle, nut, NPT 3/8“ thread on head side	Tubing 3/8“ (9,7 mm)
Head output	Swagelok 3/8“, special fitting + nut, NPT 3/8“ thread on head side	Flex armed tubing 3/8“ (9,7 mm)
Bypass input	Swagelok 3/8“, special fitting + nut, NPT 3/8“ thread on head side	Flex armed tubing 3/8“ (9,7 mm)
Column output	Swagelok 1/4“, two part ferulle, nut, NPT 3/8“ thread on block side	Tubing 1/4“ (6,35 mm)
Bypass output	Swagelok 1/8“, two part ferulle, nut, NPT 1/4“ thread on block side	Tubing 1/8“ (3,3 mm)

8. Manufacturer:

Separlab Ltd.
Brázdimská 214, 190 00 Praha 9, Czech Republic, Europe
tel. + 420 242449662
mail info@separlab.eu
www www.separlab.eu

Annex 1

List of spare parts for pumps PP03

Type	Specifikacation	Unit
PP03 RP01	Input fitting PP 03 **A,B,C** head - Swalegok SS-600-1-6 for PP03 C, SS-400-1-6 for PP03 A,B	pc
PP03 RP02	Output fitting PP 03 **A,B,C** head **-** Swalegok SS-600-1-6 for PP03C, SS-400-1-6 for PP03 A,B	pc
PP03 RP03	Pump head PP03 **A,B,C**	pc
PP03 RP04	Piston-PP03 **A,B,C**	pc
PP03 RP05	Piston seal spring PP03 **A,B,C**	pc
PP03 RP06	Forward spring insert PP03 **A,B,C**	pc
PP03 RP07	Bacward spring insert PP03 **A,B,C**	pc
PP03 RP08	High pressure sealing set PP03 **A,B,C**	set
PP03 RP09	Low pressure sealing ring (backflash) PP03 **A,B,C**	pc
PP03 RP10	Pressing screw hole PP03 **A,B,C**	pc
PP03 RP11	Head cap	pc
PP03 RP12	Head cap sealing	pc
PP03 RP13	Input (suction) check valve cartridge PP03 **A,B,C**	cartridge
PP03 RP14	Output (discharge) valve cartridge PP03 **A,B,C**	cartridge
PP03 RP15	Connecting tube head- bypass valve PP03 **A,B**	pc
PP03 RP16	Connecting tube head- bypass valve PP03 C	pc
PP03 RP17	Bypass valve body	pc
PP03 RP18	Screw hole bypass valve	pc
PP03 RP19	By-pass valve needle	pc
PP03 RP20	Seal of bypass valve needle axis	pc

Type	Specifikacation	Unit
PP03 RP21	High pressure seal of bypass valve	pc
PP03 RP22	Fixing ring of bypass valve needle seal	pc
PP03 RP23	Input fitting bypass vlave PP 03 **A,B** (Swagelok SS-400-1-6)	pc
PP03 RP24	Input fitting bypass valve PP03C	pc
PP03 RP25	Output fitting bypass valve PP 03 **A,B,C (**Swagelok SS-200(400,600)-1-6	pc
PP03 RP26	Bypass fitting bypass valve (Swagelok SS-200-1-4RS)	pc

By items where is A,B or C printed in BOLD is necessary to add proper letter to item type code.

Annex 2

PP03 C - communication via serial line

Basic parameters of the pump:
Flow: 100 – 3000 ml/min
Pressure Limit: 2 - 70 bar
Pressure Hysteresis: 1 - 15 Bars
Gradient: three-component, 10 steps. You enter A, B, and time. C calculates.
Time step gradient: 180 min.

Speed: MODE COM1 9600Bd: 9600, N, 8,1

Table of commands

1char	**2,3 char**	**……**	**……**	The importance of the message	**Answer**	**Note**
?	<CR>			Query the device	PUMP_P1<CR>	
P	00	<CR>		STOP command station	OK<CR>	
P	01	<CR>		START command station	OK<CR>	
P	02	<CR>		The state program	P02xy<CR>	X = 0 Pump Stop x = 1 pump RUN y = 0 the gradient is at the beginning y = 1 gradient goes y = 2 gradient standing at the end
P	03	<CR>		STOP command gradient	OK<CR>	See note below
P	04	<CR>		START command gradient	OK<CR>	
P	05	<CR>		OFF keyboard command	OK<CR>	Permission is granted to view and stop values. It is not possible to change anything.
P	06	<CR>		ON keyboard command;nothing	OK<CR>	
P	07	<CR>		OFF command mode service	OK<CR>	
P	08	<CR>		ON Command mode service	OK<CR>	
P	09	<CR>			OK<CR>	

Table of input values

1char	**2,3 char**	**……**	**……**	The importance of the message	**Answer**	**Note**
P	10	nnnn	<CR>	Enter SPEED DRAWING	OK<CR>	1…800 ml/min int- 1 ml/min
P	11	nnnn	<CR>	Entering PRESSURE LIMIT	OK<CR>	3…150 Bar int. 1 Baru
P	12	nnnn	<CR>	Entering PRESSURE HYSTERESIS	OK<CR>	1…15 Bar int. 1 Baru
P	13	xxyyzznnnn	<CR>	Entering gradient A, B, and time	OK<CR>	See bellow

Legend for P13 command:

The gradient can be entered only if the gradient is at the beginning (step 0), sometimes the data is rejected and sent back messages: ERROR-PG <CR>
xx ... 00-0A serial number of the step gradient (decimal 0-10)
yy ... 00-64 percent component A gradient (decimal 0 to 100)
zz ... 00-64 percent of component B gradient (decimal 0 to 100)
nnnn ... 0000-0708čas step gradient in tenths of minutes (dekadicky0 - 1800)
Note 1: The numbers are in hexadecimal
Note 2: In step 10, the time without meaning but must be entered, such as 0000
Note 3: The gradient tests: A + B> 100%, and> 100% and B> 100%, if one condition is met is set to A = 100% and B = 0%, or is set and the specified percent if A <B, 100% and the remainder to 100%.
note. 4: gradient runs in 6-second loop, where 1% is 0.06 s.

Table readout

1char	**2,3 char**	**……**	The importance of the message	**Answer**	**Note**
P	20	<CR>	Ask a specified rate at which	P20nnnn<CR>	
P	21	<CR>	Query to the specified pressure limits	P21nnnn<CR>	
P	22	<CR>	Query given PRESSURE HYSTERESIS	P22nnnn<CR>	
P	23xx	<CR>	Ask a gradient of A, B, and time	P23xxyyzznnnn <CR>	see up
P	30	<CR>	Ask the current SPEED	P30nnnn<CR>	
P	31	<CR>	Ask the current PRESSURE	P31nnnn<CR>	
P	33	<CR>	Ask the current gradient and B	P33xxyyzz<CR>	see up
P	34	<CR>	Query current time gradient	P34nnnn<CR>	

Table service mode

1char	**2,3 char**	**……**	**……**	The importance of the message	**Answer**	**Note**
P	80		<CR>	Calibration of gauge zero	OK<CR>	At zero pressure
P	81	nnnn	<CR>	Entering the	OK<CR>	It is reasonable to

				calibration of pressure		use the 80% range of the gauge
P	82		<CR>	Calibration at the specified pressure	OK<CR>	When the calibration of pressure
P	83	nnnn	<CR>	Entering the correct pumping speed	OK<CR>	see bellow
P	90		<CR>	Ask to zero calibration gauge	P90nnnn<CR>	
P	91		<CR>	Query to the specified pressure calibration	P91nnnn<CR>	
P	92		<CR>	Ask a calibration value at a given pressure	P92nnnn<CR>	
P	93		<CR>	Ask a specified pumping speed correction	P93nnnn<CR>	

Service mode is accessible only after permission.
Correction is in the range -10% to +10%. entered after a full percentage of the 0 (0000 hex) corresponds to -10%, 10 (hexadecimal 000A) corresponds to 0% and 20 (0014 hex) corresponds to +10%

nnnn .... A hexadecimal number in the range 0 ... FFFFh expressed in ASCII characters (decimal range = 0 ... 65535)
xx .... A hexadecimal number in the range 0 ... FFh expressed in ASCII characters (decimal range = 0 ... 255)
... yy. A hexadecimal number in the range 0 ... FFh expressed in ASCII characters (decimal range = 0 ... 255)
zz .... A hexadecimal number in the range 0 ... FFh expressed in ASCII characters (decimal range = 0 ... 255)

Example: Decimal number 15 = 000Fh = hex ASCII characters: 0,0,0, F = ASCII code: 30h, 30h, 30h, 46h.

The time is in tenths of minutes. Example: 000Fh = 1.5 min
Pressure is in bar Example: 000Fh = 15 bar
Flow rate is 1 ml / min Ex: 000Fh = 15 ml / min

If the command is received and evaluated as unknown or erroneous message is sent back: ERROR <CR>
At runtime, you can not overwrite any data!! With this pump it is just entering the gradient in a running gradient (pump can run). Back ERROR message is sent, PG <CR>

The program evaluates both large and small letters as well
Example: command P50000F <CR> is identical with or p50000f <CR> P50000f <CR>
The program sends all letters as large (small alphabet is not used!)
Example: in response to p50 is <CR> P50000F <CR>
The device is used to buffer 10 characters (this should be extended to 256 characters). The actual evaluation is performed only after the final character "<CR>" if taken more than 10 characters, so characters are transcribed from the start buffer. For this reason, it is good to wait about 25 ms for processing messages on the device and then send a new message.
After receipt of any value that is checked in the range of allowed values and possibly modified to this extent.