separlab

LIQUID CHROMATOGRPHY
HIGH PRESSURE GRADIENT SYSTEM

SEPARTRIX PP 03 SC BG

INSTRUCTION MANUAL

1. Use and Function of the Product

High-pressure gradient system PP03 SC BG is designed for pumping corrosive liquids, against which is resistant stainless steel AISI 316, polytetrafluoroethylene (PTFE), high molecular polyethylene (HDPE), polyetheretherketon (PEEK) and polycrystalline corundum ceramics. Using two pumps PP03 SC each with 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 to forming of smooth reproducible binary gradient. System is therefore extremely suitable for an use in preparative high performance liquid chromatography. However, due the installation of a stepping motor and broad range of flow rates it can be used in semipreparative chromatography or generally anywhere, where gradient, precision, high pressure and inertness are required.

Master pump PP03 SC GM is equipped with integrated gradient programmer (it uses pump display and a second installed keyboard). Second pump (PP03 SC GS) working as a slave has no control panel and is fully operated from the master pump. The gradient of two components can be programmed in ten linear steps with different length and slope.

2. Product Description

Pump PP03 SC (G) 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 in box of mechanics (Fig. 1). All their moving parts are stored in the ball bearings. Cams connecting rods are fitted at the end by small ball bearings that are moved 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. Movement of the shaft is provided by a stepping motor through a teeth belt. The motor speed is controlled by a own driver receiving signal from processor board.

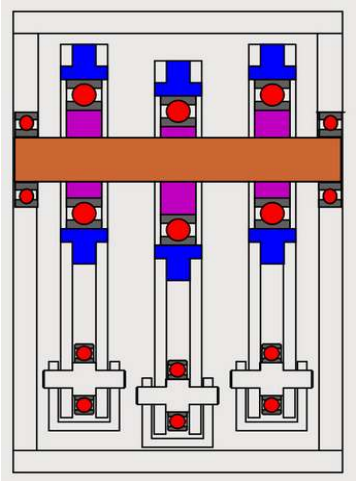

Fig. 1: Cams mechanics

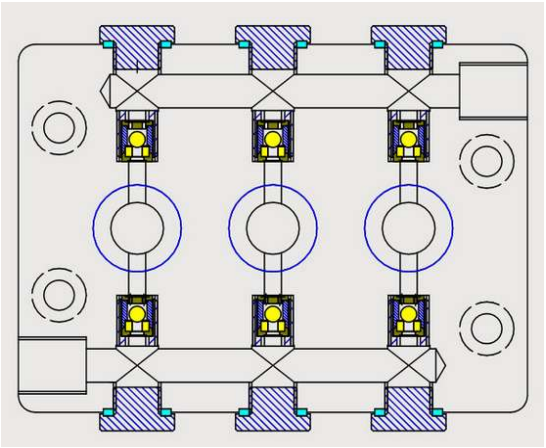

Fig. 2: Pumping head cross section

Pumping head (Fig. 2) is made of stainless steel 316 AISI 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 screw with hole 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 (additives such as buffer crystals).

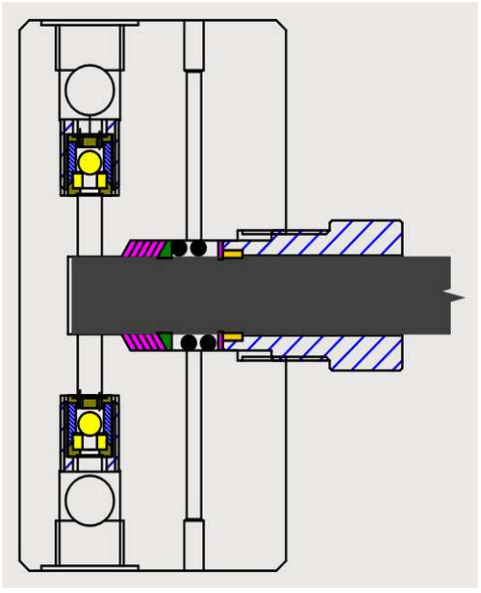

Fig. 3: Pump cylinder with pistonl

Main three seal rings are conical, made of special abrasion resistant high-molecular chemical resistant 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. 4: Piston unit

Inlet (Fig.5) and outlet (Fig.6) valves, are made as compact cartridges, exchanged as a whole. The outer portion 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 with larger seat holes and larger ceramic balls.

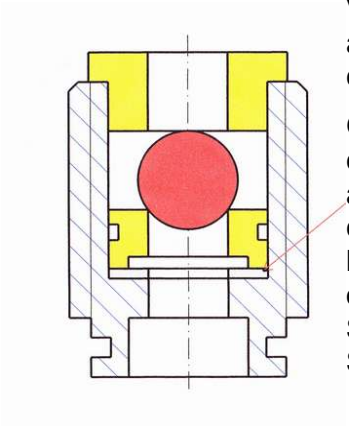

Fig. 5: Suction check valve

Valve cartridges are screwed from above and from below the pump head after removing the plug with hexagonal heads (Fig 7). To allow screwing, cartridges are provided with hexagonal recess for key type HEX.

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

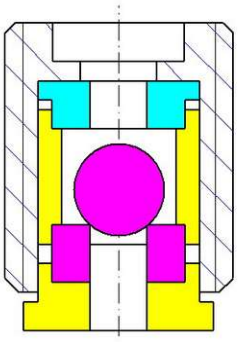

Fig. 6: Displacement check valve

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 screws with holes (Fig. 7) to adjust the pressure sealing spring forces. When removing the head it is firstly necessary to release these screws and than diassemble connecting tube on the bypass block. Then is to start the pump movement for small flow rate (cca 30 ml/min.) and use a middle force to move the head out of the case. After removing the piston head the pump has to be stopped immediately.

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 an upper panels have to be released) are accessible all parts placed inside the case. Mechanics pump, motor and its driver are located on the rugged construction of welded stainless steel profiles covered by 2 mm thick stainless steel sheet.

Fig. 8: Bypass block

On the front panel is mounted electronic display board (master pump only). Rear panel (Fig. 9) contains the power supply cable going through. There is a power switch too as well as external control connector.

System PP03 SC BG is delivered with a gradient mixer (see picture on cover page). Gradient programmer is integrated in the master pump using second installed keyboard (a right one, see Fig. 10).

Fig. 10:Pump and gradient keyboardsl

Fig. 9:Pump back panel

3. Basic data of pumps

Number of pistons:	3 (2x)
Piston diameter:	20 mm
Piston stroke:	10 mm
Cycle frequency:	4 rpm - 330 rpm
Flow rate:	2 ml/min. - 1600 ml/min. (2x)
Pressure limit:	3 Bar - 70 Bar
Pressure hysteresis:	1Bar -15 Bar
Gradient:	2 phases
Number of linear gradient steps:	10
Time of gradient step:	0,1-180 min.
Pump input:	550 W (2x)
Dimensions (depth x width x hight):	610 mm x 385 mm x 280 mm (2x)
Weight:	32 kg (2x)

4. System control

The system can be controlled from the keyboard on the front panel of master pump. Slave pump is controlled via RS232 serial port.

Left keyboard (1) is used for flow rate control (+ pressure limit, hystersis, service settings) 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.

Description of Keypad 1 drawing

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

The order of display items for controlling the pump:
Flow
Pressure
Flow Settings
Pressure Settings
Hysteresis Settings
Password Settings (the following items are accessible only after entering a password)
Settings Corection flow
Zero pressure settings
Max pressure settings
Max pressure settings

Description of Keypad 2 – gradient

F1: used to move items between displays down
F2 key: used to move items between displays up
The key "arrow down": used for deleting the setpoint
Key "arrow up": used for adding setpoint
ENTER key: used to confirm the setpoint
and switching between the keyboard and draw a gradient
Key START / STOP: is used for starting and stopping the gradient
This key is functional on any item at any time so we can start and stop the gradient, is the key functional even if the system is controlled from an external source and the keyboard is off.

The order of display items for controlling the gradient:
Status Display
A Gradient Settings (segment 0)
Time Settings (segment 0)
A Gradient Settings (Segment 1)

... .
A Gradient Settings (segment 10)
Settings of Gradient B (segment 10)

Example of operation – pump

After switching on the pump is set to display the first item. In the upper right corner shows the status of the pump (at the beggining STOP). The display shows the current flow and the current pressure.

On the bottom line is shown gradient composition. 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. Again gradient composition is shown on the last line.

Pressing the F1 key gradually check set flow rate, pressure, and hysteresis. Hysteresis means the sensitivity of the system on the pressure limit. The last offer needs Password entry, where the other items (flow correction, pressure sensor calibration) can be done by qualified serviceman.

After checking the set of valuesis possible by pressing F2 to return to the default item and we can 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) 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 rotetion 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. T

Gradient control

To switch to control the gradient on the right keyboard, press ENTER. Basic screen is displayed with BEGIN in right upper corner. By pressing F1 or F2, one can check the settings gradient. Item "Settings Gradient A" and "Time Settings" is displayed for the gradient segments up to segment 9, which is the last one (there is no longer possible to specify a time). When entering the gradient A value, system calculates the value of B so that anytime is true A + B = 100.

When enter the value of A, B is reached like 100 - A (correct value is displayed after pressing ENTER). When composition is defined, time of each linear segment is to be set. Set parameters has to be confirmed by ENTER. It is possible to stop gradient in any step, Time of the last gradient step has to be set to zero at such case.

It is necessary go to back to the initial screen when gradient is fully programmed using F2 key. Pumps are started now (ENTER on pump keyboard, STAR key, ENTER on gradient keyboard) and finally gradient is started by its STAR/STOP key. Mode in right upper corner is changer to RUN. When gradient programme is finished message END can be seen in right corner. Pumps are still working keeping end gradient composition in such case. START/STOP key goes on the programme beginning again.

Example of the gradient

General description of gradient example is given in following table (it is supposed that there is a linear change of compostition between table rows) :

Real time (min)	Composition A %	Composition B %
0	100	0
10	50	50
15	0	100

For the programming in PP 03, previous table has to be rewritten to respect that a system of segments is used in the device:

Segment No.	Segment time (min)	Composition A % on the beginning of the segment	Composition B % on the beginning of the segment
0	10	100	0
1	5	50	50
2	0	0	100

Segment are described due their time of duration and initial composition. Segments are knotted together – it means that final compostion of actual segment is the same initial compostion of the next one.

Active Button	Action Description
Main	Switch the pump on
ENTER*	Change to the gradient screen
F1	Go to segment 0, A % value setting
<>*	Set A value to 100
ENTER	Confirm A % value
F1	Go to seg. 0 time
<>	Set time value to 10
ENTER	Confirm time value
F1	Go to segment 1 A % setting
<>	Set A value to 50
ENTER	Confirm A % value
F1	Go to time setting
<>	Set time to 5 min.
ENTER	Confirm time value
FI	Go to segment 2, A setting
<>	Set value A to 0
ENTER	Confirm A % value
F1	Go to time setting
<>	Set time value to 0
ENTER	Confirm time value
F2	Press F2 so many time to come back to initial screen

*ENTER***	Change to pump screen
*START***	Start pumping

* Buttons of gradient keyboard (right)
** *Butons of pump keyboard (left)*

The gradient segment 0 is displayed, pump is running, the composition 100 % A is delivered, in the upper right corner of the keyboard shows "BEGIN". After pressing the START / STOP button on the right keyboard starts the gradient and in the upper right corner of the display will change "BEGIN" to "RUN". On the status display item then one can watch the current gradient, a given time and the running segment. In 10 min. composition is decreased lineary to 50 % A and B inreases to 50 %. After further 5 min., composition is keeping to 0 % A, B increases to 100 %. Then gradientt run stops and End is displayed in the right corner of the display. Pump is still running, keeping last composition, i.e. 0 % A and 100 % B. Next (third) pressing the START / STOP returns composition to the initial gradient compostion (gradient segment 0).

Another possibility how to stop gradient is to press the START / STOP button anytime during the run. Pump is running, delivering compostion which was on the screen at the moment of STOP action. Next (third) pressing the START / STOP returns composition to the default gradient composition (gradient segment 0).

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.

Manufacturer:
Separlab Ltd.
Radiova 1, 102 00 Praha 10, 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
PP03 RP21	High pressure seal of bypass valve	pc

Type	Specifikacation	Unit
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.