HIGH PRESSURE PREPARATIVE COLUMN

MAGic 5/150H

OPERATING MANUAL

1. Desription and use

MAGic 5 columns are designed for high-pressure, high-performance preparative chromatography. Their parts in connection with mobile phase are made of 316 stainless steel (AISI). ***MAGic 5H*** columns are destinated for the use in hydraulic column stands. They can be changed to regular columns when adding selected parts.
All columns can be used for high - efficient separation in instances where rigid sorbents capable of withstanding pressure of several MPa (several thousand p.s.i.) are used. The column is made of materials resisting all common chromatographic solvents and, accordingly, imposes no restrictions on mobile phase selection.

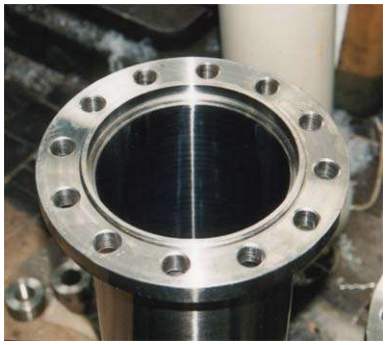

Fig. 1 Column output

The ***MAGic 5/150*** column consists of tube, I.D. 145 mm, which is provided with two flanges, each with twelve holes for the clamping screws M16. The internal surface of the column is mechanically polished to attain high smoothness (better than 0.3 um). The bottom part of the column consists of cover attached to outlet flange by means of 6 bolts M8.
The internal side of output cover is provided with a groove for seal made of a PTFE ring. Output stainless steel frit (porosity 2 um, thickness 2,9 mm, 316L stainless steel) is inserted directly to the column and the distributor consists of several layers of a fluoroplast fabric (mesh size 60 μm) with gradually decreasing diameter. Distributor is inserted in a conical recess machined in the inner surface of the cover. The assembling process is shown in figures 1 – 3.

Fig. 2 Output with frit

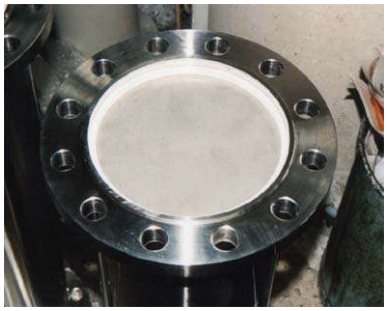

Fig. 3 Column with sealing

Fig. 4 Piston parts

The upper part of the column is closed by piston with PTFE seal, and attached by means of M8 bolts to supporting plate. A recess houses the same mobile phase

Fig. 5 Piston with frit sealing, distributor

distributor and stainless-steel frit as on the output. The sintered disk is sealed by means of fixing stainless steel ring and PTFE sealing tape (Flangiflon 1,5 x 3,5 mm). The piston assembly is forced into the column proper by distance tube which lid is connected to the hydraulic stand frame. PTFE piston sealing with inner conical part of the piston acts as pressure transducer and the sealness is better when pressure is higher.

Fig. 6 Assembled piston

2. Column packing

The outlet cover is attached to the outlet flange from the factory. It is only necessary to tighten strongly fixing bolts M16. The packing adapter (consist of a polypropylene tube with flange of the same dimension as the column flange) has then to be connected to the inlet side of the column by means of 6 bolts M16 using Flangiflon sealing (14x5 mm). Bolts are to be tightened only slightly to prevent deformation of polypropylene flange (see Fig. 7).

A suspension of the sorbent in acetone is poured into the adapter - column assembly and allowed to flow through the outlet opening to a container. A suitable (not air tight) cover is placed on the upper opening. Sedimentation is allowed to proceed for 12 hours (overnight). The remaining acetone, if any, is then removed, the packing adapter is screwed off and an upper sorbent layer (some 25 mm thick) is scraped off. The piston unit including the seal is assembled, additional sealing protects the PTFE sealing is inserted on the front part of the piston ring (Flangiflon 7x3 mm) and pressed with cylindrical tool such way not to oversize in diameter the diameter of PTFE sealing. The column walls are wiped clean and column is inserted into the hydraulic stand.

About 150 ml of a thick sorbent slurry in acetone is poured into the column and the piston assembly is inserted as described in column stand manual. Once the liquid begins to flow out from the inlet, it is closed and outlet is opened. The piston is pushed in auto mode with pressure limit which is set to the lowest value. When is impossible to push the piston inside more, inlet is connected to the chromatograph (by means of stainless steel capillaries, O.D. 6.4 mm (1/4")).

The column is then washed with acetone (about 20 column volumes) in the recycling mode and tightness of the whole system is checked. If the pressure in the mobile phase is higher then oil pressure, the oil pressure limit is to be set to higher value to be in any case a bit higher then mobile phase pressure.

The flowrate is increased gradually until at least 1.5 times the nominal pressure is reached. The column nuts are checked and then tightened if necessary. The process is repeated with methanol (for reverse-phase chromatography) or another suitable solvent similar to that subsequently used as the mobile phase. The oil pressure limit is again set to such value which is little bit higher than mobile phase pressure. Note: prior this recommendation the sorbent manufacturer instruction is followed during packing process.

Fig. 7 Column with packing adaptor

3. Manufacture and servicing:

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