CHROM. 6253

An inexpensive pneumatic solvent pump for high-pressure liquid chromatography

A pump to deliver a pulseless flow of solvent at a pressure of 2–3,000 p.s.i. was needed for high-resolution liquid chromatography. The advantages and disadvantages of commercially available solvent pumps have been discussed by HENRY[1]; as suitable pumps are expensive, a pneumatic pump was designed and built in the laboratory.

A serious defect of simple pneumatic pumps is that the actuating gas dissolves in the solvent under pressure and is released as the pressure decreases along the column. BONNELYCKE[2] reviewed methods of preventing this and described a pump in which the gas was separated from the solvent by a metal bellows, which he had used at pressures up to 200 p.s.i. For solvent pressures above the normal gas cylinder pressure, however, pressure amplification is necessary and this is most easily achieved in a piston pump. The pump to be described incorporates two pistons, one under gas pressure and the other exerting pressure on the solvent, which are rigidly connected but separated by a region at atmospheric pressure. The possibility of leakage of gas into the solvent therefore decreases as the pressure increases.

Description

Fig. 1 is a sectional drawing of the pump. It consists of a brass cylinder, 1, 3¼ in. (82.5 mm) O.D. and 2½ in. (63.5 mm) I.D., and cylinder head 2. The piston assembly comprises the gas piston 3 sealed by the chevron packing 4*, the connecting rod 5 and the solvent piston 6. The solvent cylinder is a ground brass or stainless-steel sleeve 7, 2⅛ in. (54 mm) I.D., which is shrunk into 2. Solvent pressure is contained by the PTFE U-seal 8**. The spring 9 returns the piston assembly to the bottom of its stroke when pressure is released by the exhaust valve 10. The valves 11 and 12 control the flow of solvent from the reservoir and to the column. The pin 13, moving in a slot milled in the pump body, indicates the amount of solvent remaining in the cylinder when the piston assembly is approaching the top of its stroke. The slot maintains the region between the gas and solvent pistons at atmospheric pressure. The pump is pivoted in the stand 14 (to allow the solvent cylinder to be completely emptied when changing solvents) and rests against the stop 15. The gas inlet valve 16 is not essential but provides a convenient means of control and enables the pump to be kept under pressure while gas cylinders are being changed.

Operation

To fill the solvent cylinder, the piston assembly is forced to the top of its stroke with valve 11 opened and valves 10 and 12 closed. Valve 16 is then closed and valve 10 opened; solvent is drawn into the cylinder by the action of spring 9. If the pump has been disconnected from the column or solvent reservoir, the solvent cylinder is filled and emptied several times to expel trapped air, but otherwise this is unnecessary. Solvent is pumped through the column by closing valves 10 and 11 and opening valves 12 and 16. The flow-rate is controlled by adjusting the gas pressure.

* James Walker and Co. Ltd., Woking, Surrey, Great Britain, Part No. JW430LNA30.
** Crane Packing Ltd., Slough, Bucks., Great Britain.

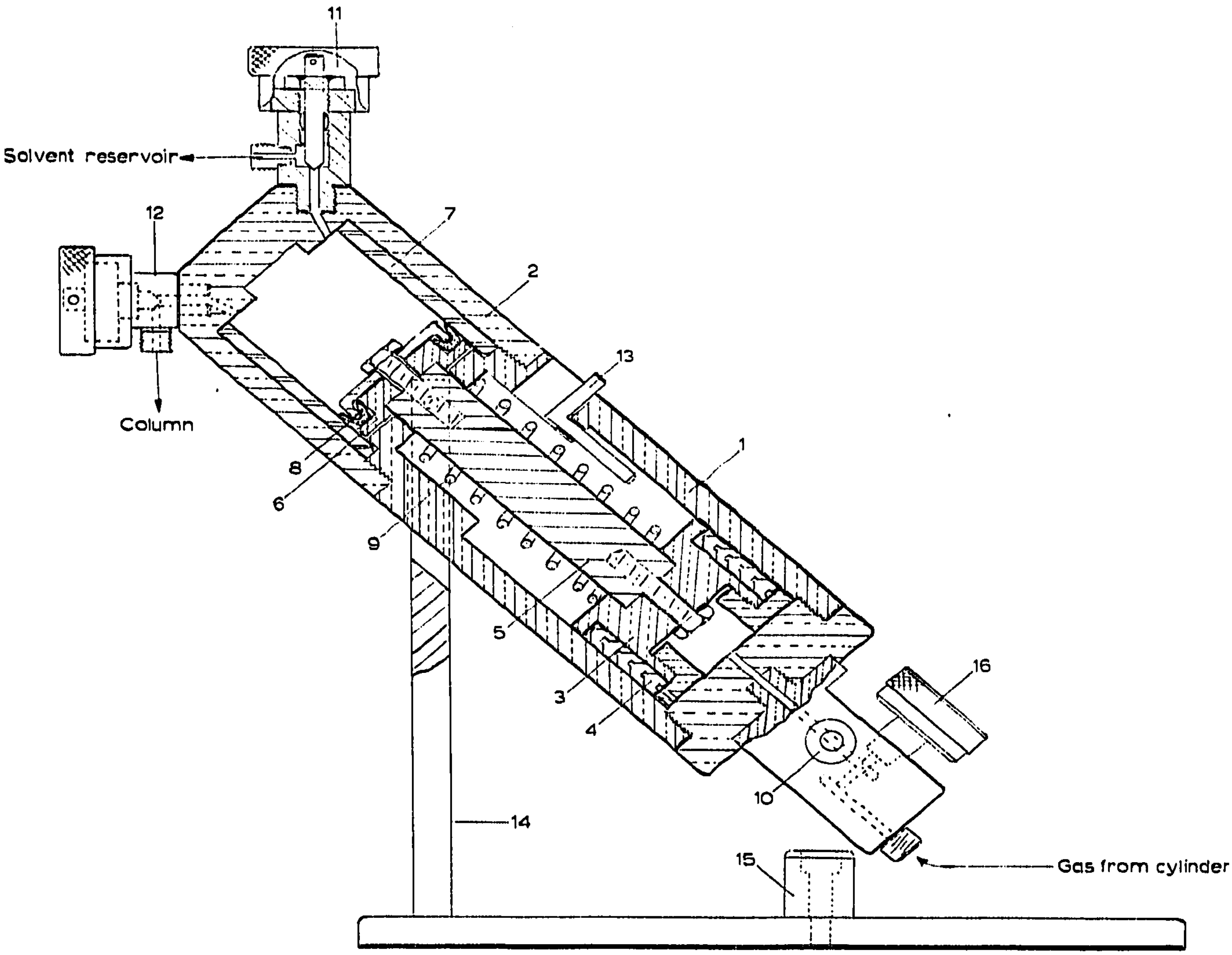

Fig. 1. Sectional drawing of pneumatic solvent pump. Scale: 3:10.

Results and discussion

The construction of the pump is straightforward, although it is important that the cylinder bores are concentric with a high surface finish. The design makes a high-pressure seal between the pump body and the cylinder head unnecessary and hence the only non-metallic components that can make contact with the solvent are the PTFE seals on the solvent piston and valve spindles, and these are not in direct contact with solvent that flows into the column.

The pump has been used continuously for several months at solvent pressures between 2,000 and 3,000 p.s.i. The only trouble during this time has been occasional leakage of solvent past the U-seal caused by small particles becoming trapped between the seal and the cylinder wall. These were easily removed but the fault could probably be cured by fitting a filter in the solvent inlet. It is, of course, important to clean the components of the pump scrupulously before assembly. Although pneumatic pumps have the disadvantage that changes in gas pressure cause changes in the solvent flow, this has caused no inconvenience in practice. Measurement of the flow-rate of the column eluate during 6 h continuous running with the gas at cylinder pressure showed a steady decrease from 0.75 to 0.70 ml/min owing to decrease in the cylinder pressure. This slow decrease would be avoided at lower pressures by incorporating a reducing valve between the gas cylinder and the pump.

Details of the design can be adapted to suit the required application; in particular, the diameters of the gas and solvent cylinders can be varied according to the solvent pressure required. (In the pump described, the solvent pressure is about 1.4 times the gas pressure.) It is feasible to construct a pump body with interchangeable heads to give a range of maximum pressures and solvent cylinder capacities. If the pump is to be used only for inorganic or alcoholic solvents, the U-seal on the solvent piston could be of a more flexible material and the pump would work satisfactorily with a less highly-finished solvent cylinder bore.

Although pumps based on broadly similar principles are available commercially[1], the simplified design of the pump described makes it much less expensive. The total cost of construction, including time and materials, was less than £100. Full details of the pump are available on request.

The authors are grateful to Mr. M. J. HARPER and Mr. F. RANDALL for the construction of the pump.

Ministry of Agriculture, Fisheries & Food,
Agricultural Development and Advisory Service,
Central Veterinary Laboratory,
New Haw, Weybridge, Surrey (Great Britain)

A. F. MACHIN
C. R. MORRIS
M. P. QUICK

1 R. A. HENRY, in J. J. KIRKLAND (Editor), *Modern Practice of Liquid Chromatography*, Wiley-Interscience, New York, 1971, pp. 59–64.
2 B. E. BONNELYCKE, *J. Chromatogr.*, 45 (1969) 135.

Received May 9th, 1972

J. Chromatogr., 72 (1972) 388–390