PTFE-GLASS PUMP. CHROMATOGRAPHIC APPLICATIONS

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The supply of solvent under pressure to chromatographic columns is usually achieved by means of gas pressure, by using a pump, or by having a solvent reservoir at a height above the column. Gas pressure has the disadvantage that dissolved gas usually bubbles out of solution near the bottom of the column, while a high reservoir is awkward to set up and the pressure obtainable is usually limited by the height of the room. These difficulties are avoided by the use of a pump, because there is no need to have gas in contact with the solvent, and high pressures can be got easily.

This note describes a pump with a glass cylinder and a polytetrafluoroethylene (PTFE) piston, which has several useful applications in chromatography. Being made

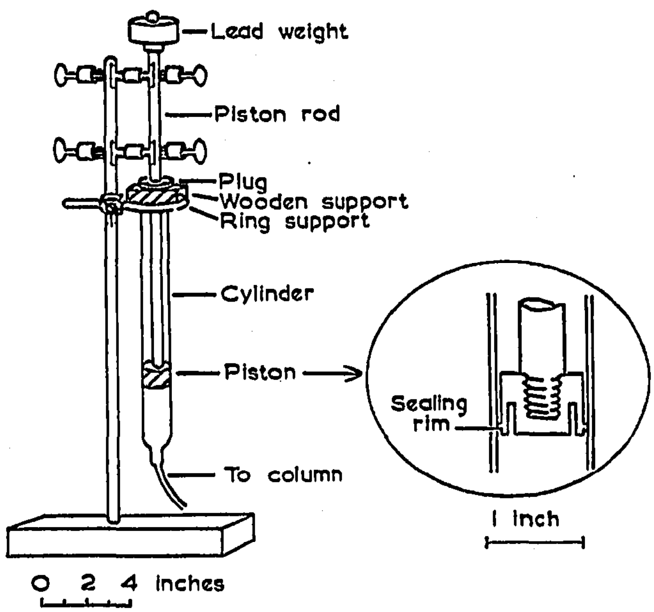

Fig. 1.

entirely of glass and PTFE it can be used with any solvent likely to be encountered. Further, a sealing rim on the piston ensures that there is no leak of solvent back past the piston, as there is with pumps of the commonly used syringe type.

The pump is shown in Fig. 1, the inset showing the construction of the piston, which is made on a lathe from PTFE rod. The sealing rim on the piston, which has an outside diameter of 2.54 cm, is turned with a tool that has a straight edge containing a semi-circular notch 0.05 cm deep.

A piston made from polyethylene instead of PTFE works just as well, but cannot be used with non-polar solvents such as hydrocarbons because these swell the polyethylene.

The cylinder is made of 2.5 cm constant-bore borosilicate glass tubing[1,*] with a heavy rim at the top resting on the wooden support. The piston rod slides through the polyethylene plug, which fits loosely into the top of the barrel.

A weight of 1.2 lb. on the piston is just sufficient to overcome its friction in the cylinder when lubricated with solvent and a weight of 9 lb. on the piston rod gives a measured pressure of 13 lb. per square inch at the outlet. Leakage of solvent back past the piston is nil with all pressures tried (0 to 15 lb.).

As well as for supplying pressure, this pump can be used for degassing solvents in the cold as follows: It is removed from the retort stand, half filled with the solvent, and all air pushed out through the outlet, which is then plugged; the piston is then pulled out a few inches to form a vacuum over the solvent causing dissolved gas to bubble out. The process is repeated until no further gas is evolved. Even under these conditions little or no air passes the sealing rim of the piston.

The flexible capillary tubing used in this work is polyethylene cannula of 1.0 mm bore. Leak-free unions with glass capillary that do not come apart under considerable pressure are made by plugging the cannula tightly into the tapered end of the capillary.

The principle described above has been used in the construction of an apparatus shown in Fig. 2, using two pumps to supply solvent for gradient elution chromatography. The piston rods P_1 and P_2 slide in brass bearings B_1 and B_2 screwed to the upright of a retort stand. W_1 and W_2 are lead weights, W_2 being heavier than W_1. B_3 is a brass arm, screwed onto P_2, that slides easily on P_1 and the upright.

In one particular application the apparatus is set up as follows: The reservoir contains 100 ml of degassed methanol and the mixer 50 ml of 60% (v/v) methanol in water. The collars C_1 and C_2 are set so that they strike B_2 when the pistons are 1.5 cm from the bottoms of their respective vessels. W_2 is 4½ lb. and W_1 is 3 lb. B_3 is set so that it just rests on the top of W_1. When the flow of solvent is started by releasing the outer two screws on B_2, P_1 and P_2 coupled by B_3 move down together under the influence of both W_1 and W_2 until C_2 strikes B_2 when P_2 stops and P_1 continues to fall alone under the influence of W_1 until C_1 strikes B_2. The composition of the solvent supplied by this apparatus is shown in Fig. 3.

This sort of apparatus has the advantages pointed out previously for the pump, *viz.* that no gas need come in contact with the solvent and a high reservoir is not needed. It has the further advantage over a gravity controlled apparatus[2] in that the relative liquid heights are not determined by their densities but can be chosen arbitrarily.

A point regarding gravity-controlled gradient elution apparatus that seems to have escaped mention in the literature is that the liquid in the connecting tube must not mix by convexion with that in the mixer if the apparatus is to function accurately;

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if such mixing does occur, one arm of the tube contains liquid that is denser than that in the other arm and the equilibrium heights of the liquids in the vessels are not simply determined by their densities. This is avoided by having the opening of the connecting tube into the mixer pointing either upwards or downwards depending on the relative densities of the two solvent components. However, the error incurred by the use of the wrong system is small if the connecting tube is not taken much above or below the level of the bottoms of the vessels. With the present apparatus (Fig. 2) these considerations do not apply, but it is necessary to prevent the contents of the vessels mixing by convexion, which is done by having the connecting tube lower than the reservoir at one point, as shown in Fig. 2.

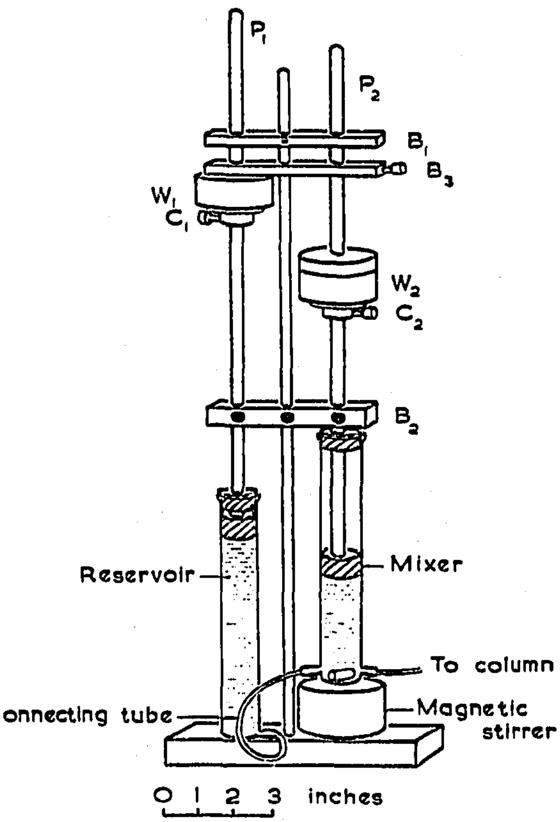

Fig. 2.

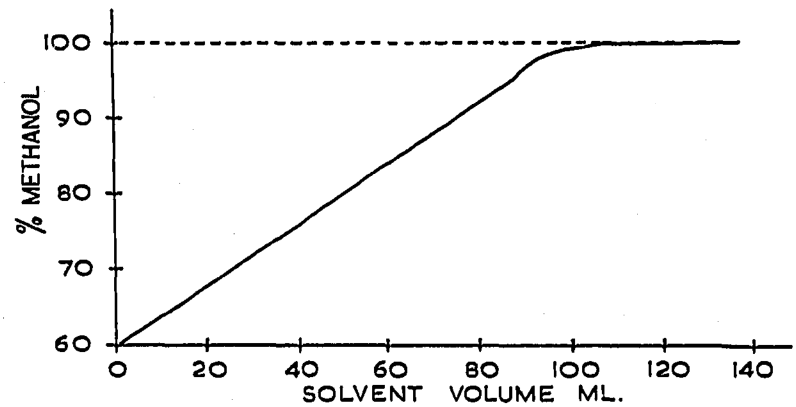

Fig. 3.

A disadvantage of the gradient elution device using pumps is that the gradient cannot be continued linearly quite up to the composition of the component in the reservoir because the stirrer index prevents the piston from going to the bottom of the mixer. In the present apparatus (Fig. 2) the reservoir continues to supply solvent after the pump in the mixer has stopped, so that the concentration of methanol in the effluent then proceeds asymptotically towards 100 % (Fig. 3).

Another application of the PTFE-glass seal that has proved useful is as an adjustable solvent connector to a chromatographic column. The piston, which is fitted tightly on the end of a thick walled glass capillary tube, is pushed down the column onto the top of the packing; solvent is fed into the top of the glass capillary.

The advantage of this system is that the piston height can be adjusted to suit the height of the packing, practically eliminating the dead solvent space that occurs when ground joints are used.

Several other chromatographic applications suggest themselves. For example, the glass-teflon pump shown in Fig. 1 and the gradient elution apparatus (Fig. 2) could be easily adapted to supply solvent at a constant rate instead of a constant pressure by attaching a slow drive to the piston rods. Such a device would have the advantage over some reciprocating syringe-type metering pumps in that the supply of solvent would be continuous instead of intermittant. The pumping principle could also be applied to apparatus for producing complex gradients[3,4]. Furthermore, the absolutely liquid-tight properties of the pump and the inertness of its materials would make it useful in a range of technical applications outside chromatography.

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SUMMARY

This paper describes a simply made pump with a liquid-tight seal between the glass cylinder and the polytetrafluoroethylene piston. Its applications to column chromatography include the supplying of solvent under pressure, gradient elution and degassing of solvents.

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