Journal of Chromatography, 132 (1977) 128–129

CHROM. 9658

Note

A simple pulse damper for liquid chromatography*

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(Received August 30th, 1976)

Simple piston-type reciprocating pumps are often used in liquid chromatographs in spite of the recent development of advanced pulse-free pumping systems. The reason is their low price, reliability and, in comparison with the positive displacement syringe-type pumps, also their ability to deliver large volumes of liquids continuously. The main disadvantage of reciprocating pumps is the necessity to use a pulse damper.

A typical pulse damper consists of a hydraulic resistor (*e.g.*, a capillary) and a hydraulic capacitor. A common problem with the capacitors is the choice of an elastic diaphragm separating the (minimally compressible) liquid from the elastic damping part, such as compressed gas or a metal spring. Appropriately shaped devices made of metals or plastics, such as Bourdon tubes and bellows, are most commonly used for this purpose[1–3]. It is also possible to use mercury[4] or other liquids that are immiscible with the eluent, but these "liquid membranes" may contaminate the eluent. The capacitors usually have limited application: it is often necessary to change the parameters of either the membrane or the elastic part for each pressure region.

These problems can be overcome by using the capacitor shown in Fig. 1. It consists of a column containing a bed of small particles of filling material. Part of the column is filled with the eluent while the remainder contains the gas bubble. The "membrane" is formed by the liquid in the bed of the filling material. When the valve V is closed, the gas in the column is periodically compressed and released when the pressure of the eluent is changed. The filling material prevents the gas bubble from moving across the column. The liquid at the interface is saturated with the gas; however, the diffusion and mixing rate of the liquid in the capacitor is very low because of presence of particles of filling material. For any pressure produced by the pump, a rapid "equilibrium" gradient of concentration of the gas dissolved in the liquid is achieved.

The conditions for efficient damping based on the described principle are as follows:

(a) The hydraulic resistance of the liquid chromatographic columns (plus hydraulic resistor) must be higher than the resistance of the bed of filling material in the capacitor.

* Patent pending.

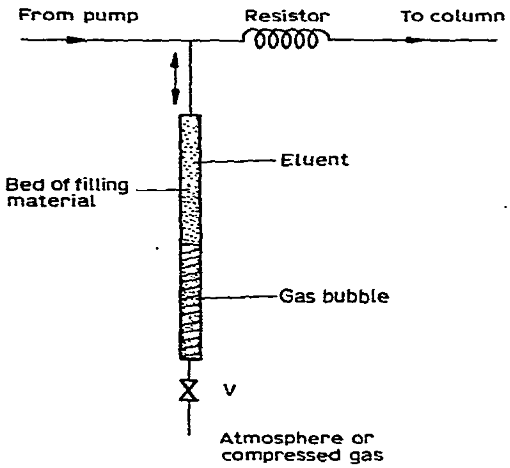

Fig. 1. Schematic diagram of pulse damper.

(b) The liquid layer and the gas bubble in the capacitor must be long enough. The appropiate sizes can easily be found experimentally. For most applications, a simple liquid chromatographic column with a volume comparable to that of the columns used for separation is sufficient. The size of the gas bubble can be changed simply by operating the valve V.

(c) The appropriate filling material must be used. Non-compressible inorganic porous liquid chromatographic carriers were found to be most efficient. In this instance the gas in the pores of the filling material also takes part in damping. The average diameter of the particles depends on the operating pressure, and ranges from several to several hundred micrometres.

The device was applied successfuly in liquid chromatographs at pressures of 0.5–5 MPa (about 5–50 atm). The upper pressure limit seems to be constrained only by the size of the capacitor. No adjustments were necessary during several months of continuous use at various pressures. The pulses of a single-piston reciprocating pumps were damped efficiently for use of a differential refractometer with a sensitivity of 10^{-7} refractive index units. We observed no baseline drift produced by contamination of the degassed eluent by the liquid from the pulse damper which might be saturated with damping gas (air in this work).

The advantages of the pulse damper lie in its simplicity, reliability and universality. On the other hand, its relatively large dead volume may complicate recycling and gradient elution. The same principle can also be applied to pulse damping in other hydraulic systems.

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