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Note

An easy-to-make sample applicator for column chromatography

T. K. KORPELA
Department of Biochemistry, University of Turku, 20500 Turku 50 (Finland)
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There are two basic injection systems in liquid chromatography, syringe and valve[1]. The main advantage of valve injection is reproducibility, but it is less flexible than the syringe. Fixed-volume (four-port) and external loop are the most common valve types of injectors[2]. I have constructed a simple modification of the fixed-volume valve, containing three discs (A–C in Fig. 1a, b) mounted on a shaft. The middle part (B) is capable of rotation. Two lines (I, II in Fig. 1a) bore through all the discs. The disc B has sampling channels drilled on the same radius as the lines I and II. The sample is injected or pulled into the sampling channel through line I and then the dial B is rotated to line II for elution. The dial B may have bores of either the same or different diameters (*e.g.*, 0.5–5 mm for the applicator sizes of Fig. 1). The device has the following advantages, *viz*: (a) it does not contain high precision joints or any other part difficult to prepare and thus it can be made in laboratory workshops, (b) several samples (*e.g.* 12) can be loaded into the applicator at the same time, and (c) sample volumes can be varied without changing any part. "Disc stack" construction can also be modified to produce complex valves.

TECHNICAL DESCRIPTION

The device was made of a transparent acrylic (Perspex) rod apart from the central shaft which was made of stainless steel. Because the plastic does not resist many organic solvents, it is convenient to use a material such as PTFE or Kel-F. A transparent sampling dial is recommended so that possible air bubbles can be observed. If good pressure capability is demanded, the insides of dials A and C ought to have a smooth finish, and the inlays of the O-rings (of dial B) must be made carefully. About 2/3 of the O-rings were sinked. If the applicator is used to load several samples, the lines I and II must be near each other (*e.g.* 60°) to avoid contamination which may arise if the sample goes over the elution line. The by-pass line (see Fig. 1a) is not always necessary.

OPERATION

Turn the by-pass vents to "by-pass". Loosen the hand screw (10, in Fig. 1a) 1/2 turn and revolve the first sampling channel to line I. Rinse the line and pull the

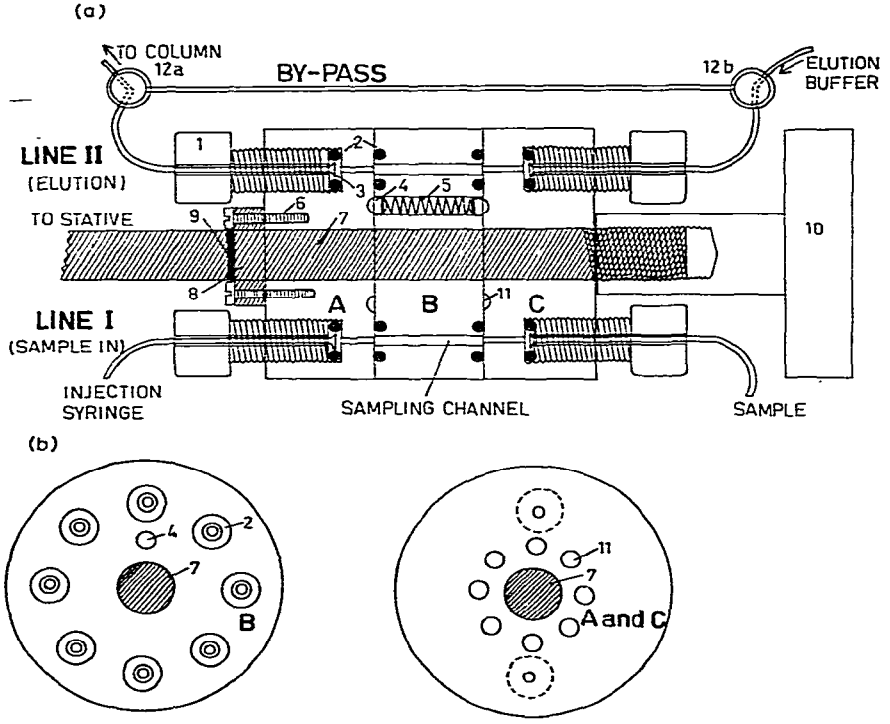

Fig. 1. (a) Sampling applicator for column chromatography. A, C, dials containing tube connectors; B, sampling dial (A–C each 50 mm diameter); 1, connector screw; 2, silicone rubber O-rings; 3, extension of tube; 4, 5, steel ball and spring to facilitate settings of dial B; 6, anchoring screw for dial A; 7, stainless steel shaft (10 mm); 8, 9, bushing soldered onto the shaft; 10, hand screw for tightening joints of dials A–C; 11, cavities for ball (4); 12, by-pass vents of the applicator. (b) End views of dials A–C from (a). Dials (A) and (C) are identical.

sample into the channel. Repeat the process until the first loaded sample is on the elution line. Tighten the hand screw and turn the by-pass vents to "elution".

CALIBRATION OF SAMPLE VOLUMES

Measurement of the accurate sample volumes were made spectrophotometrically by the above prccedure using $K_2Cr_2O_7$ in 0.1 *M* H_2SO_4 as a "sample". The solution was rinsed through the elution line to graduated bottles and the colour intensity was measured. Six separate channels (20 mm long) made with a 2 mm drill gave mean volumes of 91–94 mm^3. Sampling with one channel gave S.D. of 0.2 mm^3 ($n = 6$).

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