A simple device for cleaning and coating capillary columns

A simple device is described which can be used to remove the substrate from gas chromatographic capillary columns and replace it in 15 min. The unit was constructed of readily available materials* and assembly required a minimum of skill. Several units have been described in the literature[1–3], however, the present device is simpler, faster, and more convenient.

A reservoir was constructed of 1 in. × 6 in. pipe (A), capped at one end with a pipe cap as shown in Fig. 1. The other end of the pipe was fitted with a 1 in. coupling and 1 in. to $^1/_4$ in. pipe bushing. Swagelok tube fittings were used in the assembly on all tubing. A tee (B) having two $^1/_4$ in. tube fittings and a $^1/_4$ in. pipe fitting was drilled out so that a $^1/_4$ in. tube (E) could be inserted through the pipe end extending

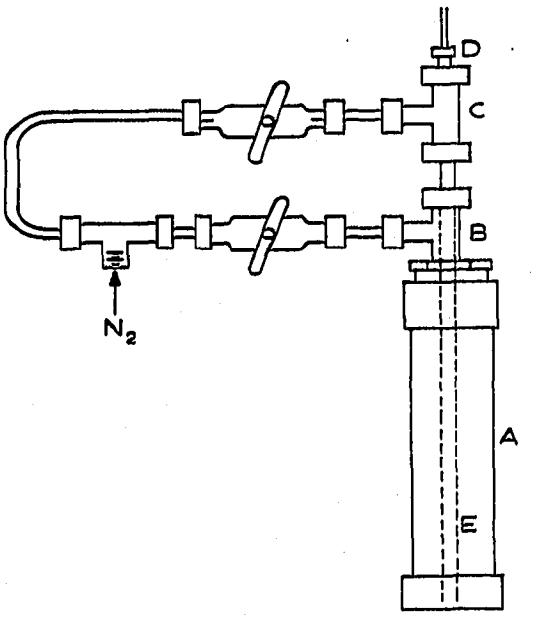

Fig. 1. Device for coating capillary columns.

to the bottom of the reservoir. The bottom of this tube was notched to allow the liquid to enter the tube. The close proximity of tube (E) to the bottom of the reservoir facilitated draining of the reservoir when cleaning. The tube was sealed by tightening the upper tube nut on tee (B). To the end of the tube was fastened another tee (C) to which ultimately the capillary column will be fastened through a $^1/_4$ in. to $^1/_{16}$ in. tube reducer (D). A shut-off valve was fitted to the center fitting of each tee and connected by tubing to a source of nitrogen gas. The entire assembly was constructed of stainless steel and all pipe threads were covered with Teflon tape prior to assembly.

New capillary columns were cleaned or the substrate removed from old columns by flushing with approximately 20 ml of solvent. The solvent was poured into the reservoir through tee (C) after removal of reducer (D). It was necessary to disconnect the nitrogen inlet fitting and open the lower valve to permit venting. The capillary

* References to specific products of commercial manufacture are for illustration and do not constitute endorsement by the U.S. Department of Agriculture.

column was attached at (D) and the solvent forced through the system with nitrogen. Nitrogen flow was continued until the entire system was dry.

Columns were coated in a similar manner except that a solution of the stationary phase was introduced instead of the pure solvent. In coating a 250-ft. column of 0.020 in. inside diameter, 50 ml of a 20 % solution of the stationary phase provided sufficient material and a nitrogen pressure of 80–100 lbs./sq. in. was adequate. The column was coated in stages to prevent substrate plugging of the capillary. The lower valve was opened for 2 min followed by opening of the upper valve facilitating distribution of the material through the column. The process was repeated until substrate appeared at the open end. Nitrogen gas was permitted to flow through the column for several minutes to evaporate the solvent. The column described has been cleaned and coated several times and each time a total of the 15 min was required for the entire procedure. If it is desired, the column can be equilibrated by allowing nitrogen to flow through it for a longer period of time.

The device was cleaned without disassembly of the reservoir. The nitrogen inlet was removed, the reservoir filled with solvent as described above and flushed using laboratory air pressure. The process was repeated at least 3 times to give satisfactory results.

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[1] G. DIJKSTRA AND J. DEGOEY, in D. H. DESTY, *Gas Chromatography*, Academic Press, New York, 1958, p. 60.
[2] S. R. LIPSKY, R. A. LANDOWNE AND J. E. LOVELOCK, *Anal. Chem.*, 31 (1959) 852.
[3] A. ZLATKIS AND J. E. LOVELOCK, *Anal. Chem.*, 31 (1959) 620.

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Apparatus for extraction of compounds from paper chromatograms

It is frequently necessary to extract the compounds separated on a paper chromatogram for further examination, for example by ultra-violet or infra-red spectrophotometry.

The apparatus described in this note (see Fig. 1) is simpler than those given by WYATT[1] and DENT[2], and can be used with volatile extracting solvents. A number may be compactly mounted in a rack for simultaneous extractions.

It is readily assembled from standard interchangeable glass joints, *i.e.*: Quickfit and Quartz Ltd. B19 test tube MF/24/2/6, a B19/B24 connecting bend (stillhead) SH1/23 and a B24 socket SRB/24 which has been drawn out and sealed as shown in the diagram.