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Note

Improved preparative dry-column chromatography*

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"Dry-column" chromatography (DCC) for separation of mixtures and purification of compounds has received much attention recently[1–3] and its advantages over liquid-filled column chromatography have been adequately enumerated. In particular, use of inexpensive, UV-transparent, and disposable nylon columns has contributed much to the success of DCC. Separated components are readily isolated by sectioning the adsorbent-packed nylon column with a knife, much like slicing a sausage.

A drawback of commercial nylon tubing is that it is flat-pressed. Imparted creases on each side must be removed as much as possible for satisfactory columns[1], otherwise channeling may occur. Furthermore, nylon creases are more easily smoothed out by the denser alumina which is often favored over silica gel in DCC. Accordingly, a better method was sought for packing crease- and channel-free columns with either adsorbent.

This note describes an improved routine for preparative DCC employing a slotted metal sheath to support the nylon column (Fig. 1), a tamper to pack adsorbent and concomitantly press out creases, and a puncture-sampling technique[4] for analysis of column resolution by thin-layer chromatography (TLC).

PROCEDURE

The column was prepared as follows. A suitable size rubber or cork stopper (1) was placed in the bottom of the nylon tubing (2) previously inserted in the metal sheath (3). The tubing-stopper junction was taped and the sheath was positioned vertically allowing it to butt against the exposed end of the stopper. A piece of cotton (4) was pushed with the tamper (5) to the bottom of the column followed by a layer of sand (6) (optional). Careful tamping of incrementally added adsorbent gave a tightly packed and essentially crease-free column. The top of the column was covered with about 2 cm of sand (7) and one or two 18-gauge syringe needles were inserted into the cotton to allow for escape of displaced air.

Chromatography, carried out in the usual manner, was complete when the frontal zone of the developing liquid descended to 2–3 cm of the bottom of the

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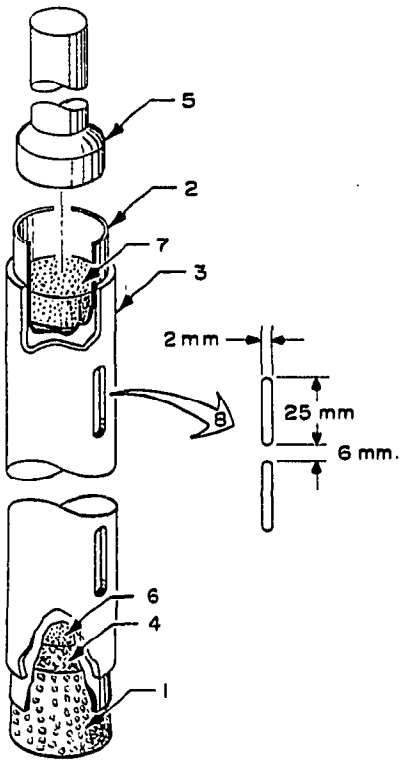

Fig. 1. Apparatus for dry-column chromatography. For explanation, see text.

column (less than 30 min were needed to pack a 125-g 100–200 mesh silica gel column (about 400×25 mm) and development was complete within 1 h). The top of the nylon column was then twist-sealed and taped shut. The sheath was laid horizontally for removal of core samples and subsequent TLC analysis. Disposable open-end melting point capillary tubes were used instead of pipets for sampling[4].

Radioactive components were easily located by transfer of core samples to scintillation vials followed by conventional scintillation counting techniques.

Based on these analytical results, the supported column was marked through slots (8) for sectioning, removed from the sheath, and sectioned accordingly.

The metal sheath (selected from commercial copper, aluminum, and steel tubing) had a diameter slightly larger (by *ca.* 0.5–1 mm) than that of the nylon tubing used*. Length and overall dimensions of the column required for a preparative separation can be predicted[1]. Advantageously, the sheath was made longer than generally needed and the nylon column was filled only to the height necessary.

Slots, machined along one side of the sheath, allowed viewing of column packing, removing of creases (by aligning a crease of the nylon tubing with the slots), monitoring progress of the chromatogram, and sampling of the limp column.

Each slot was numbered consecutively with a number punch. A core sample taken through each slot was transferred to a vial held in a coincidingly numbered

* See footnote 1 of ref. 1 for three suppliers of nylon tubing. Two additional suppliers are J. T. Baker, Phillipsburgh, N.J., U.S.A., and M and Q Plastic Products, Freehold, N.J., U.S.A.

vial holder*. This procedure minimized labeling, especially when many samples were extracted.

The head of the tamper was machined such that its diameter was slightly smaller (by *ca.* 0.5–1 mm) than that of the nylon tubing and that its edges, as well as those of the sheath, were polished smooth to prevent puncturing or cutting of the tubing. The beveled edge allowed adsorbent to slide into the column as the tamper was withdrawn.

ACKNOWLEDGEMENT

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REFERENCES

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* Vial holders were made from a block of wood with holes drilled out and numbered consecutively with a number punch. One-half dram vials (Owens-Illinois Glass Co., Vineland, N.J., U.S.A., Kimble Article No. 60910-L) were ideal for TLC.