Large divided columns

PILLAY *et al.*[1] have stated well the problems encountered in liquid–solid adsorption column chromatography, and have presented the technique of divided columns for improving the sharpness of separations. To isolate minor constituents from some oils obtained in aroma investigations, hundreds of grams of material may need to be chromatographed. Obviously kilograms of adsorbents must be used, and irregular zone fronts and time of completion of separation become major problems.

A combination of CLAESSON'S recommendation[2] to use a series of columns with diminishing diameters and PILLAY'S technique of dividing the column of adsorbent into shorter sections can be utilized to good effect in large column-work. This combination can be accomplished by assembling the column in sections, which can be varied in size from 8 cm diameter and 40 cm high to 4 cm diameter and 20 cm high. The number of sections used can also be changed according to the amount of adsorbent necessary. The Kontes O-ring connector*, size 40, is a convenient means for connecting the sections (Fig. 1). Grease contamination, as in the case of ball-socket joints, is eliminated with this type of connector, since neoprene or silicone rubber O-rings are used for seals. For tall columns, more tension than that exerted by the conventional No. 65 ball-socket clamp is necessary. The tension needed to stop leakage is obtained by cutting off the hinge and by applying tension at three points with three bolts and nuts (Fig. 2).

The irregular zone front is sharpened as the material passes from one section to another through a zone of solvent above the adsorbent. The "coning" effect is compensated for by the funnel at the bottom of each section, where a plug of glass wool prevents the solid adsorbent from following into the next section. Also, if the column is built in short sections, the weight of the adsorbent does not continue to add and cause the adsorbent to pack so tightly that the resistance to eluant flow becomes great. If 100–200 mesh adsorbent is used, flows of 2 to 4 l per h through 3 to 4 kg of adsorbent in 4 large sections can be easily achieved.

With large quantities and fast separations, the heat of adsorption of strongly adsorbed materials can cause difficulties. But such difficulties can be diminished by proper deactivation of the adsorbent and careful gradation from solvent to solvent.

Since large volumes of eluants are used, a solvent feeding system must be used that provides a constant-head pressure and some insurance against going dry. Fig. 3 shows a simple system which has proved convenient. The lower "S" section from the reservoir to the column provides vertical freedom in assembly. The small reservoir provides a constant-head pressure, and the "goose-neck" curve in the tubing from the reservoir stops any mixing of the liquid as the polarity (therefore, usually the density) of the eluant in the feeders is increased. The two inverted 2 l round bottom flasks with standard-taper joints and offset feeder arms can be used to maintain the level in the reservoir. These feeder flasks can be filled easily by removing the

* Reference to a company or product name does not imply approval or recommendation of the product by the Department of Agriculture to the exclusion of others that may be suitable.

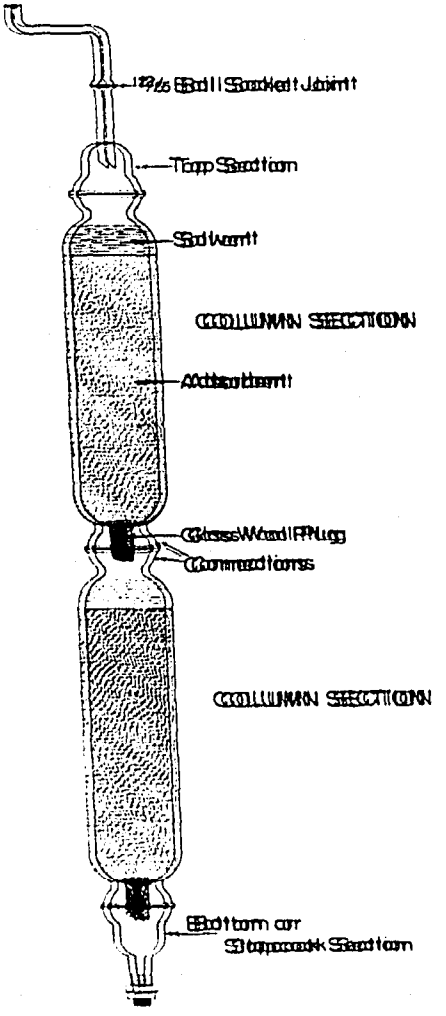

Fig. 1. Column assembly.

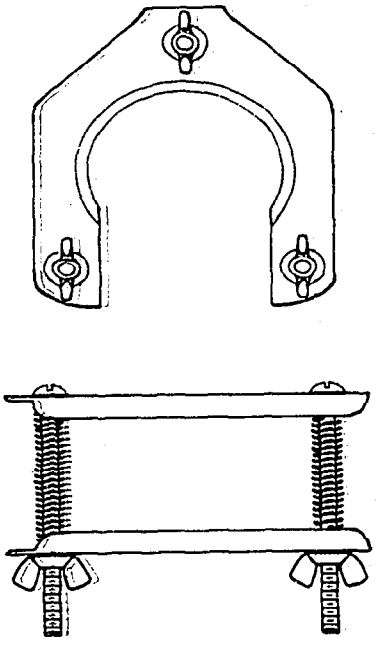

Fig. 2. Modified clamp.

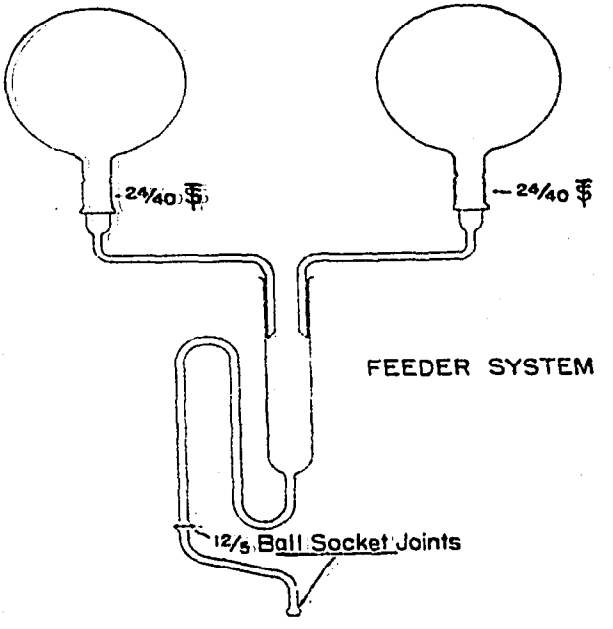

Fig. 3. Solvent feeder assembly.

feeder arms. Since the pressures in the feeder system are small, the ball-socket and standard-taper joints can be assembled without grease.

With the described arrangement, chromatographic separation of several hundred grams of material can be completed in a few days.

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[1] P. P. PILLAY, D. S. RAO, C. P. N. NAIR AND E. T. VARKEY, *Chem. Ind. (London)*, (1958) 258.
[2] S. CLAESSON, *Arkiv Kemi Mineral. Geol.*, 16 (1957) 24A.

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A spraying method for the preparation of thin-layer chromatoplates*

Thin-layer chromatography on microslides has been found particularly useful for the rapid checking of samples obtained at different stages in the preparation or purification of organic compounds.

In our laboratory the technique has also been used for finding a suitable solvent system for use in column chromatography of steroids. A rapid method has been devised for applying a thin layer of silica gel or alumina on microscope slides. The method involves spraying a slurry of silica gel (10 g in 30 ml of water) with a conventional glass sprayer used for spraying reagents. The process is illustrated in Fig. 1. The precaution

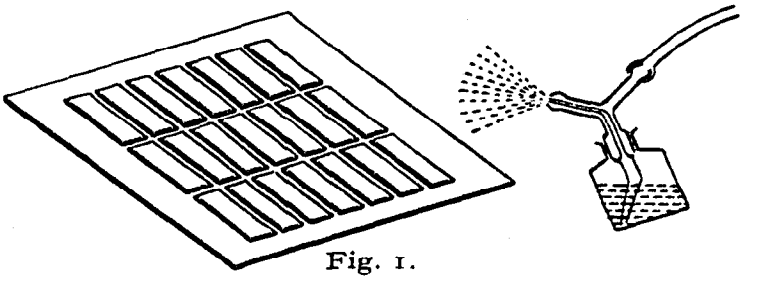

Fig. 1.

should be taken to keep the microslides on a flat surface to secure a uniform coating. The plates can be dried at room temperature or at an elevated temperature (100°, 30 min). The thickness of the coating prepared by this technique ranges around 200 μ–300 μ and 10 g of commercially available Silica Gel G (Merck, Germany) is sufficient to coat 20 microslides (75 × 25 mm).

The technique is quickly learned and has the advantage that the necessary equipment is available in every laboratory.

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* The method was devised by one of us (K.M.) when he worked at the National Institutes of Health, Bethesda 14, Md., U.S.A.