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Preparative cylindrical thin-layer chromatography. II.

Previous papers[1,2] have described the advantages of cylindrical TLC over flat surface TLC and one simple technique for the application of a narrow sample band to an adsorbent coated test tube.

The method of sample application by end-spotting, though simple, has some disadvantages. Some samples or adsorbents do not permit the eluting of all sample from the end of the test tube and then compression into a narrow band with solvent of high eluting power. Distribution of sample about the periphery of the test tube is not perfectly uniform. Application of large amounts of solid samples tends to cause flaking of the layer. The layers on the ends of large test tubes are very fragile.

Improved techniques for layer preparation and sample application will be described here which make cylindrical TLC most versatile for preparative purposes.

Experimental

The cylindrical glass layer support for all experiments described herein is a 50 × 400 mm test tube to which is added a 24/40 standard taper ground glass female joint at the open end. It is essential that the axis of the joint be aligned with the axis of the test tube.

Thin layers are prepared by dipping test tubes into a wide-mouth jar of slurry. The slurry used in these experiments is prepared from Silica Gel GF to which K-90 PVP (polyvinylpyrrolidone supplied by the GAF Corporation and having an average molecular weight of 360,000) has been added (2% by weight). The silica gel and PVP are mixed dry before addition of chloroform–methanol (2:1), using approximately 170–180 ml of solvent for 50 g of silica gel. The slurry should not be thick. A wet layer prepared by dipping from a thick slurry sags producing an irregular surface. Thick adsorbent layers with only slight surface irregularities can easily be prepared by multiple dipping, allowing partial evaporation of slurry solvent between dips. For example, a tube dipped five times in the slurry described produced a layer *ca.* 0.75 mm thick containing 34 mg of adsorbent per square centimeter of layer.

Layers prepared by only one dipping are smooth and moderately durable; most areas on the layer permit pencil writing without removal of adsorbent. Layers prepared by two or more dips are durable at all points, and dry layers may be handled freely with little risk of damage.

The presence of PVP as binder in 2% concentration does not seem to alter the R_F values found using the same silica gel without binder. PVP does not prevent the use of fluorescent indicators in the adsorbent. PVP seems not to be eluted by developing solvent from the adsorbent; cylindrical layers developed several times remained durable. Layers containing low concentrations of PVP are not so hard, however, as to prevent the scraping off of adsorbent to isolate mixture components.

Various cellulose adsorbents such as Avicel* TG microcrystalline powder can be used to prepare durable thin layers without binder. They will not, however, satisfactorily bind other adsorbents to glass.

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The best present method of sample application to a cylindrical layer utilizes a spray technique (alternative methods such as wick streaking and injection application have not yet been investigated). The coated test tube is attached at the ground glass joint to a rotary evaporator and rotated. A Desaga* micro spray gun is rigidly clamped in a position such that the end of the barrel is 2–3 mm from the spot on the thin layer where sample would be applied (Fig. 1). Moderate care in regulating the air control trigger of the spray gun permits deposition of a sample band of width less than 2 mm (Fig. 2a). For all sample applications pictured in Fig. 2, 0.5 ml of acetone were used to dissolve 10 mg of Sudan Black B, an intensely dark solid dye. The solution is placed in a well (maximum capacity of 0.5 ml) at the top of the spray gun. A line pressure of 7.5 p.s.i. of nitrogen was used for spraying. Very slight misting of sample solution prevents the formation of a perfectly clean-edged sample band. Greater care in regulating the air control trigger will produce an even narrower sample band with less misting. This is also greatly facilitated as shown in Fig. 1 by the use of smaller volumes of sample solution. Fig. 2b pictures the result when a sample is sprayed on fairly rapidly.

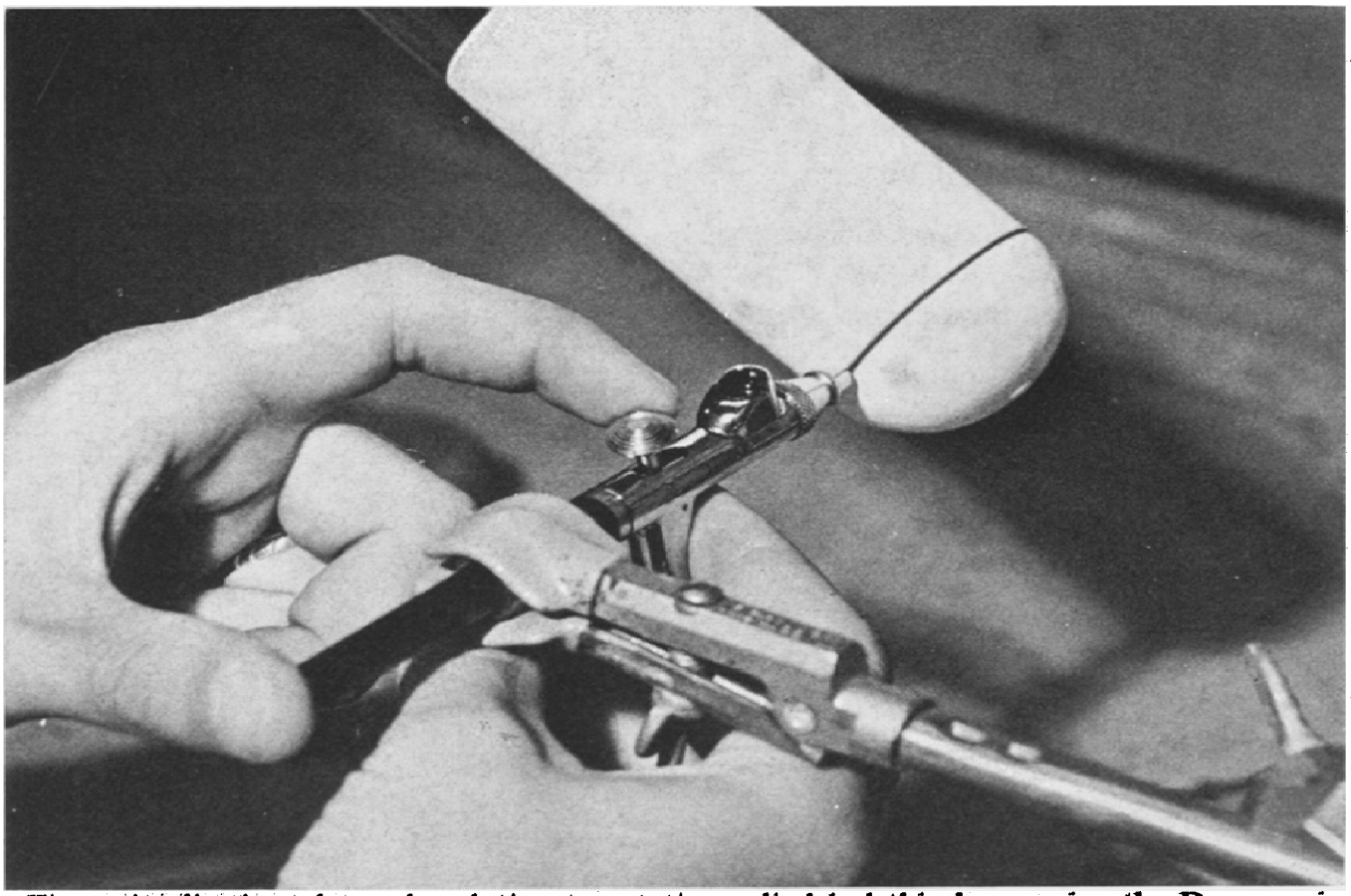

Fig. 1 Application of sample solution to rotating cylindrical thin layer using the Desaga micro spray gun.

By the use of two masking rings made from paper strips tightly taped around the test tube the diffuseness of the edges can be largely eliminated, if this is considered necessary. The masking technique works best if the thin layer is smooth. A typical

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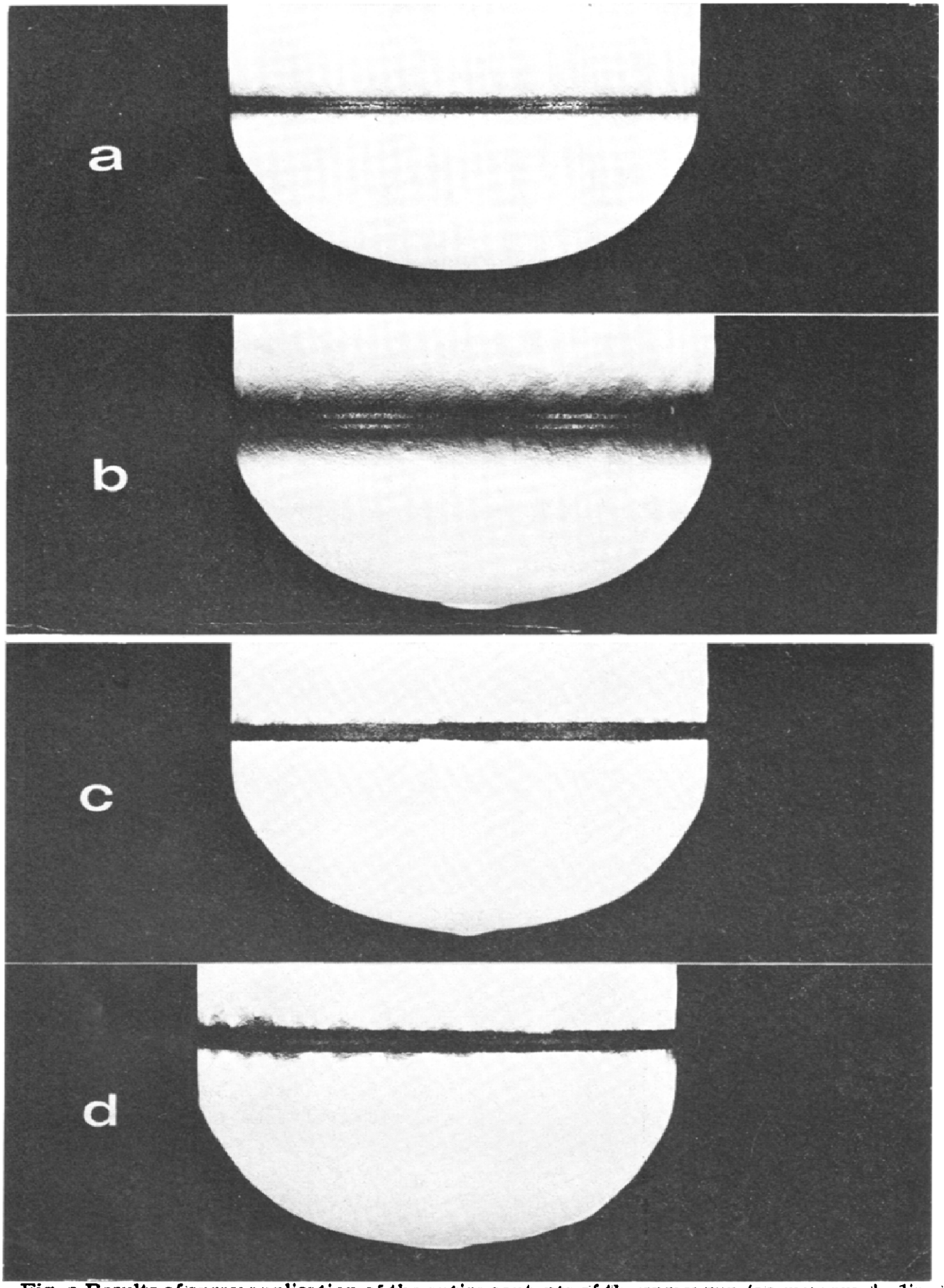

Fig. 2 Results of spray application of the entire contents of the spray gun (10-mg sample dissolved in 0.5 ml solvent) onto thin layers supported by a 50-mm-diameter test tube using: (a) a slow spraying rate; (b) fast spraying; (c) fast spraying of a masked relatively smooth layer; (d) fast spraying of a masked rough layer.

result of masking and rapidly spraying a relatively smooth thin layer is pictured in Fig. 2c. The result of spraying a relatively rough masked layer or one where the masking rings are not tight against the layer is depicted in Fig. 2d. Where quantitative application of sample to the thin layer is not necessary, masking permits fast application of sample and control of sample band width.

In the only quantitative test of cylindrical plate capacity so far, using these techniques, a 20-mg sample of Sudan Black B was deposited as a 2.5-mm wide band on a 50-mm-diameter test tube supporting a silica gel layer 170 mm long × *ca.* 0.75 thick and was completely separated into ten components by benzene development.

Discussion

The use of PVP as binder for adsorbents permits the preparation by dipping of durable thin layers of variable thickness on cylindrical surfaces. Such durability allows the spray application of sample solution in a high-speed air stream. Thick strong layers also allow the separation of relatively large samples.

Sample bands prepared by the simple rotary spraying technique compare favorably in all respects to those deposited on flat layers by a variety of elaborate and more expensive apparatus. The Desaga Micro Spray Gun, of modest cost, appears invaluable for the application of narrow sample bands without masking. With masking, other atomizer spray devices should prove satisfactory. In lieu of a rotary evaporator, a slow-speed stirring motor with a large chuck to hold a glass tube would suffice to rotate the cylindrical layer.

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