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Thin-layer sheets with an adsorbent of a few microns thickness

A technique has been developed for preparing very thin (in general 6 μ) thin-layer (TL) sheets. The sheets are very suitable for the rapid separation of various substances.

The preparation of TLC plates with ready-made adsorbents is well known[1] as well as the usage of the factory-made precoated sheets (*e.g.* Eastman and Merck ready-for-use sheets)[2].

The preparation of a silica gel membrane of a few μ thickness is now described.

Fig. 1 shows a sketch of the method used to prepare the TL sheets. To prepare

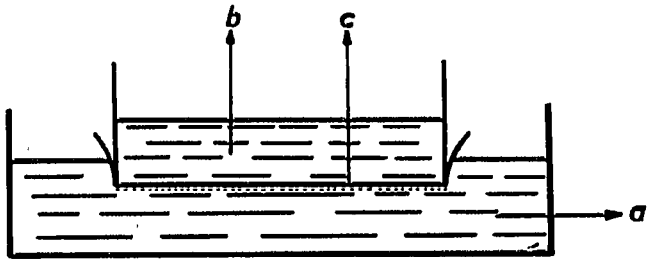

Fig. 1. Sketch of the method used to prepare thin-layer sheets. a = Sodium silicate solution, b = hydrochloric acid, c = semipermeable membrane, *viz.* cellophane.

the plates commercial potassium silicate was dissolved in an excess of sodium hydroxide and the fixed semipermeable membrane, wetted with distilled water, was dipped into the sodium silicate solution. Hydrochloric acid solution (4.5%) was then poured over the membrane for a short time (about 5–10 sec), after which a thin silica gel membrane was observable on the outer part of the cellophane. It was rinsed with distilled water and ethanol and was ready for use after cutting into suitable sizes. If necessary we can activate the sheets by heating them in a desiccator for about 50 min at 60°.

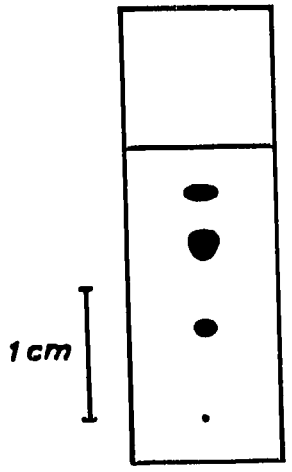

Fig. 2. Separation of (top to bottom): butter-yellow; sudan-red and indophenol.

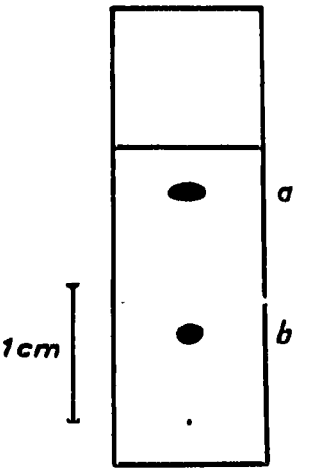

Fig. 3. Separation of two steroids: a = cholesterol; b = prednisolone.

The membranes can be hardened by gelatin or fixed on a hard white paper. Gelatin does not interfere with the adsorptive capacity of the sheets.

Development can be carried out in suitable small vessels.

Fig. 2 shows a typical separation of the dyes: butter-yellow, sudan-red and indophenol. In this case benzene was used as solvent for a separation on a 1.5 × 4 cm sheet.

Fig. 3 represents a typical separation of two steroids *viz.* cholesterol and prednisolone. It was run in a mixture of chloroform–ethanol (9:1). Detection was in U.V. light after spraying the plates by phosphoric acid–water (1:1) and heating for 5 min at 90°. In both cases the processes took only 5–10 min. The thickness of the adsorbent layer of the sheets in the cases mentioned was about 6 μ.

The prepared layers were solid enough for our purposes. We could use and activate them without damage and the layer could only be removed by strong mechanical rubbing.

The silica gel adhered to the cellophane without any subsidiary material. The layers are thin enough to separate very small quantities of substances after a short running time (0.5–2 min) because the solvents and substances only move short distances (1–3 cm).

The thickness of the layer can be slightly influenced and changed by a modification of the reaction conditions (time of the layer formation).

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