NOTES

between theoretical and found values for the succinate and the UCON-oil columns (marked with q) could be explained by the support material: with these columns, Chromosorb W or similar material is necessary in order to obtain relatively symmetrical peaks. It is, however, not understood why the actual w_b/w_h value for these two columns is smaller than 1.70; in case of tailing, one would rather expect larger values

The most striking difference can be observed in the case of the two polyester columns. The butanediol succinate (BDS) column was made of the relatively inert HMDS-treated Chromosorb W and as a result, the peaks are practically completely symmetrical. On the other hand, the support material for the diethylene glycol succinate column is the standard diatomaceous earth type material without any treatment which has a considerable adsorption effect on such polar samples as the fatty acid methyl esters. Also, the large deviation of the w_b/w_h ratio from the theoretical value for the adsorption columns can easily be related to the fact that the peaks on adsorption columns usually show some tailing. As mentioned above, most of the other values can also be interpreted in this direction. Thus, the results seem to demonstrate the applicability of this calculation for peak symmetry control.

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A new apparatus for thin-layer chromatography

Various chromatographic techniques have been developed in the last few years with the purpose of reducing time-consuming separations and facilitating the reproducibility of the results.

Thin-layer chromatography on silicic acid, alumina or cellulose, was developed by STAHL who stratified these materials on glass plates¹⁻³. This method allows the chromatographic separation of mixtures of substances difficult to separate, such as oil esters, polyterpenes, tars, steroids, bile salts, amino acids, etc.

The two main advantages of STAHL's method for chromatographic separation are the shorter run of the solvents and the uniform chromatographic support obtained by using the standardized gel preparation.

To prepare "thin layers", small devices, manually operated, are now available⁴. These stratifiers do not give good results because the layer presents an uneven thickness which disturbs the chromatographic separation.

This difficulty prompted us to develop an apparatus which would improve the deposition of the adsorbing material on the glass plates.

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After a number of attempts to solve this problem we devised an apparatus in which the plates progress at a constant speed ensured by supporting rollers. The plate must pass through an adjustable slit under a reservoir containing the chromatographic support. Details of the apparatus are shown in Figs. 1a and b.





Fig. 1. (a) View of the apparatus from above. (b) Side view.

- 1. Corrugated board
- 2. Rubberized rollers
- 3. Sprocket wheels
- 4. Supporting sprocket
- 5. Bulkheads
- 6. Adjustable bulkheads
- 7. Adjustable screw
- 8. Spring

- 9. Spring screw
- 10. Pulley
- 11. Motor
- 12. Current point
- 13. Switch
- 14. Casing of the apparatus
- 15. Outlet for wash water
- 16. Glass plate

The apparatus consists of a grooved slab; on this plane are two rollers, belted with rubber rings. These rollers are set in motion by a motor which ensures that the glass plates move along at a constant speed; the motion of the roller is such that it displaces the plate as shown in the layout, from right to left. In this way the plate passes under two bulkheads forming a small room in which the adsorbent is placed. The bulkhead (No. 6) can be regulated by two microscrews and allows stratification at various thicknesses.

With the help of a switch the stratifying process is begun, and a new plate is added every time the preceding one has passed under the reservoir. It is possible in this manner to stratify an indefinite number of plates with the same thickness and a perfect distribution of the adsorbent in all parts of the plate.

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With this apparatus^{*} we have obtained good results for the separation of a great variety of substances, in particular amino acids and bile salts.

For special purposes, and in particular for stratifying mixtures of silica gel and silver nitrate^{5,6}, the apparatus has been constructed with the reservoir in plexiglas.

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A method for the detection of β -phenylethylamines and β -phenylethylamino acids

A convenient method for the detection of β -phenylethylamines on chromatographic paper has not been available. In the course of a study in which it was necessary to treat chromatograms with Ehrlich's reagent after they had been treated with ninhydrin, it was noted that each of a number of substituted β -phenylethylamines appeared as a pink spot. Since these compounds do not react with Ehrlich's reagent alone it was felt that this combination of color reagents might be useful in the detection of β -phenylethylamines. Accordingly, a systematic examination of a series of substituted β -phenylethylamines and related compounds was undertaken.

Procedure and results

The compounds studied were dissolved in methanol-water for application to paper chromatograms. 5 μ g of material were routinely chromatographed on Whatman No. 1 paper. The chromatograms were developed in butanol-acetic acid-water (4:1:1). Other solvent systems were also employed, and it was found that if the chromatograms were adequately air dried, the choice of solvent system did not significantly affect the results.

The dried chromatograms were then dipped in ninhydrin-pyridine reagent (0.2 % ninhydrin in acetone-pyridine 9:1), air dried, and heated for 1 min at 105°. After notation was made of the color, the chromatograms were dipped in a modified Ehrlich's reagent (2 % p-dimethylaminobenzaldehyde in acetone-conc. HCl 9:1). After the ninhydrin color had completely faded, certain compounds evidenced a pink color which slowly changed to orange. The pink color was maximum approximately 30 min after the strips were dipped in Ehrlich's reagent and the colors were therefore