

Detection of Chromatogram by Swelling of Adsorbent

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In the field of adsorption chromatography the column chromatography was devised for the first time by Tswett¹⁾ about fifty years ago. Afterwards, in 1950, the chromatostrip as the modifier was introduced by Kirchner et al.²⁾ and more recently the larger strip called chromatoplate was used by Reitsema³⁾. In these

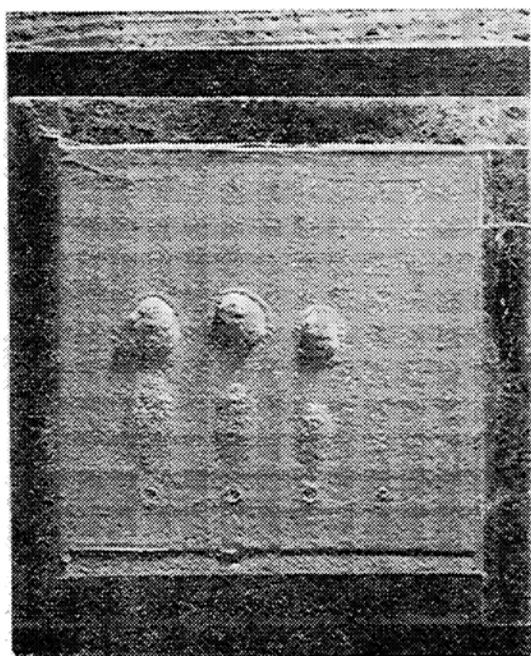


Fig. 1. Swelling of adsorbent by water vapor.

Developer: 10% ethylacetate in *n*-hexane

Adsorbent: Silica gel (about 200 mesh)

Sample: Benzaldehyde

The amount of sample is 15, 10, 5 and 1 drops from left to right, respectively.

methods the adsorbent was sealed with a glass wall or hardened with a binder.

Simplifying the operation of the above-mentioned chromatoplate, the authors used the adsorbent alone sprinkled on a glass plate without a binder. As an adsorbent was used silica gel of about 200 mesh. It was sprinkled on the glass plate in constant thickness (2 mm.) and the surface was smoothed with a glass rod. The commercially available pure benzaldehyde was used as a sample. It was developed by 10% ethylacetate in hexane and dried in air to remove the developer.

The present authors happened to discover that the adsorbent of the part of the spot made by benzaldehyde was upheaved by swelling when the plate prepared by the above-described process was left in an enclosed case of high humidity. In order to confirm this phenomenon, the authors left another plate prepared in quite the same way in the sealed case of 100% relative humidity and over a long time observed the change expected to be brought about on it. After 2 to 3 hr. an umbrella-shaped swelling of the adsorbent began to occur at the place of

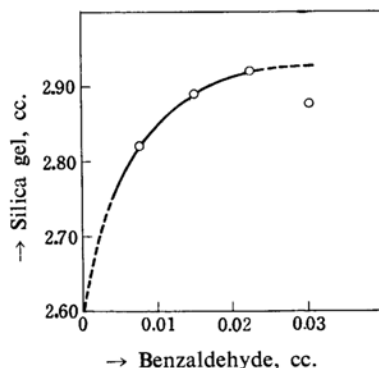


Fig. 2. The apparent volume of silica gel as a function of the amount of benzaldehyde added to 1 g. of silica gel.

1) M. Tswett, *Ber. deut. botan. Ges.*, 24, 384 (1906).

2) J. G. Kirchner and G. J. Keller, *J. Am. Chem. Soc.*, 72, 1867 (1950).

3) R. H. Reitsema, *Anal. Chem.*, 26, 900 (1954).

R_f about 0.5. After one day that of oval shape began to arise at R_f about 0.22 and after three days the rising of ring shape was found around the first one. The second swelling and the third one seem to be caused by derivatives of benzaldehyde or its oxidation products. In either case it is considered that polar groups in these compounds act on polar groups of silica gel and consequently the swelling by water vapor takes place. The condition of the plate after four days is shown in Fig. 1. The amount of the sample dropped on silica gel was 15, 10, 5 and 1 drops from left to right, respectively. In a drip of one drop hardly any upheaval is observed.

Fig. 2 was obtained by plotting the apparent

volume of silica gel as a function of the amount of benzaldehyde added to 1 g of silica gel. As is seen in the figure the apparent volume of silica gel increased with the increasing amount of benzaldehyde until it came to 0.025 cc.

The swelling phenomenon was scarcely noted when the relative humidity in the enclosed case was decreased to below 40 or 50%.

So far the expression of the spot of chromatography has been two dimensional or superficial. But the use of the swelling phenomenon above described will make the three dimensional observation possible.

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