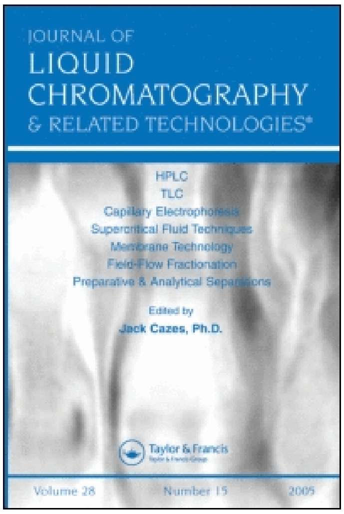

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VACUUM PLANAR CHROMATOGRAPHY (VPC): A NEW VERSATILE TECHNIQUE OF FORCED FLOW PLANAR CHROMATOGRAPHY

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ABSTRACT

Taking the disadvantage of capillary development and vapor phase effects into account, new methods of forced flow development have appeared.

The new process of chromatographic development described is part of forced flow system. It is the vacuum (Pressure lower than atmospheric pressure) which is implemented in order to achieve the chromatographic development by inhaling of the mobile phase through the stationary phase, unlike the other forced flow methods which press the solvent.

A complete development system was studied ; It consists of a plate positionning chamber and a membrane of polyethylene. In order to compartmentalize the useful surface of the chromatographic plate and to produce a soft pressure on the system without compression of the polymer.

This material is a " closed cell " polymer (Recticel, Clichy, France) which makes it waterproof for the eluting solvent. The only path for the solvent during analysis is inside the adsorbant layer.

Above this is a " Vacuum chamber " connected to a suction pump.This system creates a homogeneous depression at the level of the development chamber, then into the adsorbant layer and triggers a narrow application of the polymer on the chromatographic support.

So, the depression thus obtained has the following effects : inhaling the eluent when the bottom of the set is dipping into a tank containing the solvent (forced flow system by inhaling), maintening the polymer membrane strongly applied on the adsorbant layer (micro chamber on depression) and suppressing the vapor phase the analysis progresses (vapor and dissolved gases contained in the layer) on the not chromatographic part of the plate. The plate remains dry and gas - free during the whole analysis.

The advantages of this new system reside in the fact of not having to prepare the plate as in the classical method of thin - layer chromatography and contrarily to the other forced flow methods. On the other hand the first results obtained show that the development is like the vaporless phase development as for the forced flow system.

INTRODUCTION

Although TLC is at first sight a relatively simple technique, the behavior of the systems employed can combine to make it more difficult to apply reproducibly than *e.g.*, HPLC : the complicated nature of the forces governing the chromatographic process can be further aggravated by the variability of the sorbents.

In adsorption chromatography, such as generally occurs on silica gel, alumina, and other inorganic sorbents, the choice of eluents is based on the same criteria as are valid for column adsorption chromatography : as is true for the latter technique, when multicomponent solvent systems are employed their composition changes during the course of development and a mobile phase gradient is formed in the layer, detracting from the reproducibility of the technique. As a consequence, eluents comprising one or two solvents only should be used whenever possible.

In partition chromatography, the basis of separations employing cellulose, kieselguhr, reversed phase TLC, and occasionally silica gel, in which the sorbent serves merely as a support for the stationary phase, it is possible to select the eluent by analogy with HPLC. The presence of the gas phase does, however, complicate matters : development in most TLC chambers is a three-phase process and it has been shown that the participation of the gas can play an undesired rôle in the chromatography [1].

The analogy with HPLC is further disrupted by the mechanism by which the solvent moves through the layer : when plates are developed solely by capillary action efficient separations can be achieved only with short development distances, especially with HPTLC layers.

Several forced flow development methods have been introduced in an attempt to overcome the problems of capillary development and eliminate the influence of the gaseous phase. Initially these used the pressurized ultra-micro chamber (PUM, [2]) ; this was followed by overpressured (thin) layer chromatography (OPLC) and pressurized chambers have recently been developed for circular and anticircular planar chromatography [3], ultra rotation planar chromatography (URPC, [4]), and overpressured multi layer chromatography (OPMLC, [5]).

Despite these advances the presence of the gaseous phase at the solvent front is an unwanted complication : to eliminate this we have developed a new forced flow system in which solvent transport is effected by the application of a vacuum, rather than by pressure.

EXPERIMENTAL

All chemicals were of analytical reagent grade and were supplied by Prolabo (Paris, France). Chromatography was performed on 20 x 20 cm precoated silica gel 60 F^{254} plates (Merck, Darmstadt, FRG) which were used as received.

The sorbent layer was covered with a polymer membrane cut slighly smaller (180 x 200 x 3 mm) than the glass plate supporting the sorbent ; both were installed (Figure 1) in a special development chamber which was then clamped together. The chamber was immersed in a small trough containing the mobile phase and connected to a suction pump (Figure 2). Reduction of the pressure within the chamber and the sorbent layer causes the polymer membrane to be pressed gently on to the surface of the stationary phase.

The polymeric material used for the membrane was a “closed cell” polyethylene (Recticel, Clichy, France).

RESULTS AND DISCUSSION

The properties of the polymer (Table 1) were such as to ensure good, hydrophobic, contact between the membrane and the stationary phase. As the solvent could not

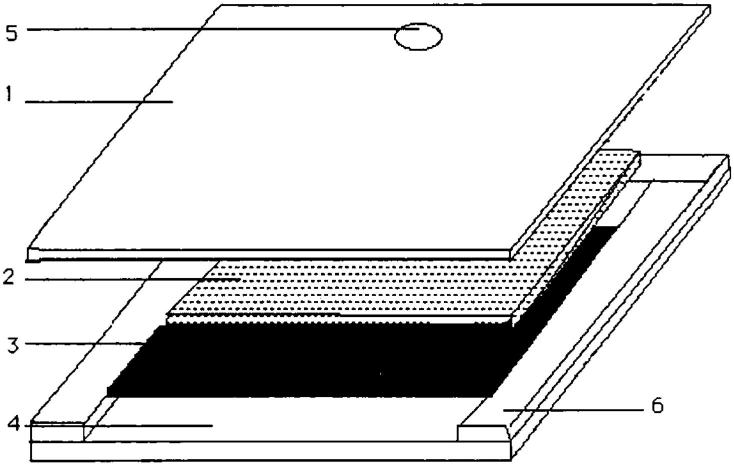

FIGURE 1 Vacuum Planar Chromatographic Chamber
1 and 4: upper and lower part of the instrument
2: polyethylene membrane
3: absorbent layer
5: vacuum
6: stirrup

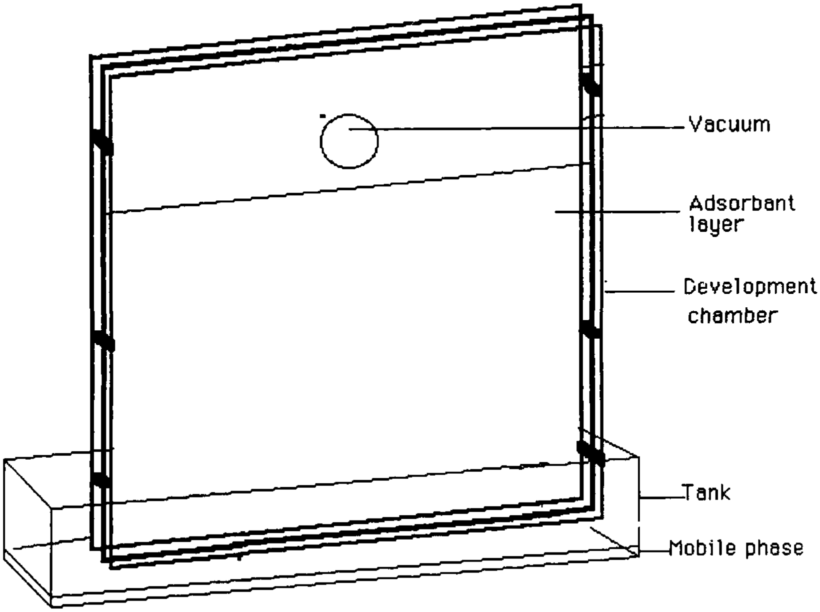

FIGURE 2 Vacuum Planar Chromatographic Apparatus.

TABLE 1

Physicochemical Properties of the Polymer

Water absorption capacity	0%
Ignitability	inflammable
Volumic masse	30 kg/m^3
Stability (72 h)	
high temperature (110°C)	no change
low temperature (- 18°C)	no change
UV light (λ = 350 nm, 32°C)	no change

TABLE 2

Effect on Polymer of Solvents and Reagents commonly used in TLC

Solvent	Dilatation coefficient (%)
water	< 5
methanol	14
ethanol	16
n-butanol	16
acetone	16
heptane	15
toluene	32
chloroform	29
ethyl acetate	16
diethyl ether	13
hexane	16
acetonitrile	16
methyl ethyl ketone	16
hydrochloric acid	20
acetic acid	16
formic acid	< 5
ammonia	17
pyridine	16

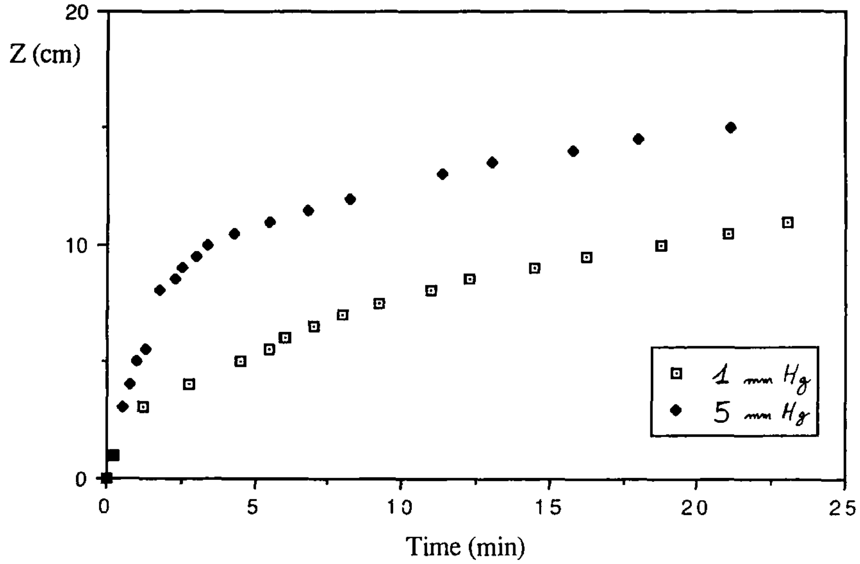

FIGURE 3 Distance (z) Travelled by the Solvent (Methanol) on Silica Gel 60 Plate (HPTLC) Versus Time.

travel through the closed pores of the polymer its only path was through the sorbent layer.

The stability of the polymer towards several solvents commonly used in mobile phases has been investigated ; the results, listed in Table 2, are indicative of the suitability of the material. No changes were observed in the thickness of the material (dilatation occuring only lengthwise) and no physical deterioration was apparent in any solvent.

The effect of the pressure reduction on solvent transport was investigated by measuring the distance travelled by methanol over 25 min. at two different pressures. The results are plotted in Figure 3. It is apparent that the velocity of the solvent front is a function of the applied pressure and that the relationship is not linear. Only by continuous variation of the pressure is it possible to generate a linear relationship ($\Delta l = kt$) ; only under such conditions is solvent transport by capillary action completely eliminated.

CONCLUSION

A new forced flow planar chromatographic technique, vacuum planar chromatography has been developed in order to eliminate problems arising in conventional TLC as a result of : (i) the limitations of solvent transport by capillary action (solvent is drawn through the stationary phase by the application of vacuum) and (ii) the effect of the gaseous phase on the chromatography (the plate remains gas-free throughout the analysis).

Another advantage of the new technique resides in its being applicable to plates prepared in the classical manner : other forced flow techniques can be used only with plates prepared on flexible (*e.g.* polyethene, aluminum) supports. The technique is as simple to apply as classical ascending development, and multi-layer development is also possible [5].

If conditions can be arranged such that $\Delta l = kt$, the results obtained are equivalent to those generated by other forced flow techniques.

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