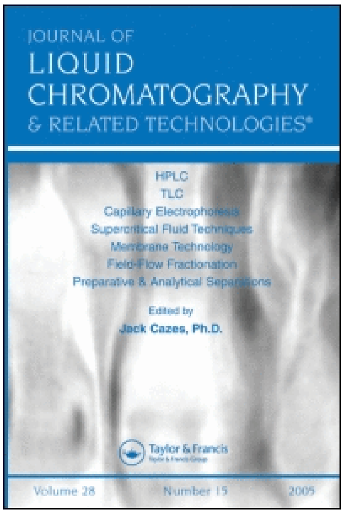

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MECHANICALLY BLENDED SILICA-BASED BONDED PHASES. APPLICATION OF THE SIMPLE MODULATION OF THE POLARITY OF THE STATIONARY PHASE TO T. L. C.

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ABSTRACT

A mixture of polycyclic aromatic hydrocarbons and aza-arenes was used to test C_{18}/cyano mixed phases prepared by mechanical mixing of silica-based bonded phases for one-dimensional T.L.C. It was shown that for a normal phase polarity mechanism, the modulation in polarity of the stationary phase, obtained by simple variation of the percentage of the cyano phase, contributed to an improvement in the resolution of the mixture.

INTRODUCTION

In order to achieve an improvement in resolution, R, on a T.L.C. plate, a solvent is introduced or exchanged in the mobile phase based on Snyder's[1] triangle of selectivity, α ; the retention factor, R_f may also be optimized.[2] If double behavior phases are used such as the cyanopropyl phase,[3] then by changing the mechanism α and thus R can then be strongly modified. However, a certain rigidity limits this type of phase since on the one hand the ratio [cyano group] / [methylene group] is fixed, and on the other hand the total quantity of cyano group is approximately constant depending on the reproducibilities of the phase and layer preparations.

When these constraints are removed by use of mechanically blended mixed-mode phases,[4,5] we have shown that an improvement in R can be obtained by introducing a new parameter. The variable retention force of mixed C_{18}/cyano stationary phases has thus been successfully tested on a mixture of polycyclic aromatic hydrocarbons (P.A.H.) and aza-arenes.

MATERIALS

The P.A.H. and aza-arenes used were pyrene (Fluka), benzo[b]fluoranthene (Aldrich), acridine (Sigma), benzo[h]quinoline (Fluka) and phenanthridine (Fluka) of purity : > 97 %, 99 %, > 95 %, > 99 %, and >98 % respectively.

The characteristics of the pure commercial phases used : C_{18}-bonded silica and CN-bonded silica were as described previously.[4]

METHODS

The preparations of the blended phases and the plates, together with the development and detection techniques, have been described previously.[4] All the results were confirmed three times.

RESULTS AND DISCUSSION

The mixture tested contained two P.A.H. not resolvable on the unmixed C_{18} phase[6] : pyrene and benzo[b]fluoranthene, both hydrophobic and polarizable, and three isomeric aza-arenes : benzo[h]quinoline, acridine and phenanthridine, all hydrophobic, basic and dipolar.

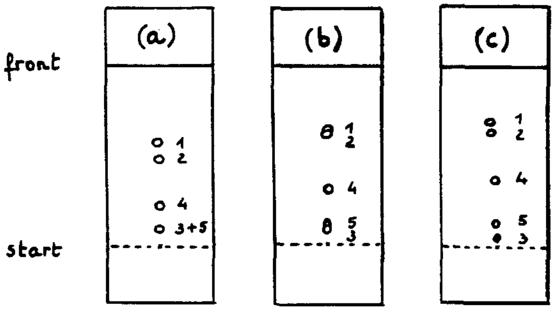

Figure 1. Effects of variations of the polarities of the mobile phase and the stationary phase on the resolution. Compounds : 1 = pyrene, 2 = benzo[b]fluoranthene, 3 = acridine, 4 = benzo[h]quinoline, 5 = phenanthridine. Stationary phases : (a) C_{18}/cyano 50/50, (b) C_{18}/cyano 50/50, (c) C_{18}/cyano 30/70,. Mobile phases : (a) hexane, (b) hexane / chloroform 85/15, (c) hexane / chloroform 80/20.

According to Smith and Cooper's triangle of selectivity, relating to bonded hydrophilic phases, the cyanopropyl phase is essentially characterized by the dipole orientation.[7] Combinations of such a phase with a C_{18} phase should thus be complementary with respect to the mixture being studied. Although a 50/50 mass percent C_{18}/cyano combination, combined with an eluent such as hexane, has proved to be satisfactory for the separation of certain P.A.H.,[4] when it was applied to aza-arenes it caused the co-elution of acridine and phenanthridine (Fig. 1a), which are strongly retained and in addition have the same basicity (pK_a = 5.60). In order to move these two compounds into an R_f zone more favorable to R,[2] the polarity of the eluent was increased. By addition of chloroform, essentially a proton donor[1] with respect to the basic aza-arenes, or by addition of nitroethane, essentially a creator of dipole-dipole interactions[1] with respect to the P.A.H. and the aza-arenes, it was only possible to achieve a weak increase in R for the two aza-arenes. This was at the expense of the P.A.H. whatever the percentage of modifier in the eluent. The best result obtained is shown (Fig. 1b).

On the other hand, an increase of the dipole-dipole type stationary phase-solute interaction forces by accentuation of the cyano phase percentage, combined with a slight increase in the eluent polarity, led to resolution of the mixture (Fig. 1c). We have shown with the C_{18}/cyano combinations that in the 50 - 100 % cyano range, the mechanism is normal phase polarity, as long as the polarity P' of the eluent does not reach the value 3.5.[4] With an invariant mechanism, the modulation of the polar phase percentage in a mixed phase is thus easy to achieve and very flexible, constituting an additional variable which would seem to be of some interest. Studies are continuing with a view to defining this and to suggest predictive models.

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