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USE OF LIQUID CRYSTALS IN LIQUID CHROMATOGRAPHY

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SUMMARY

Results of a study of the liquid chromatographic retention behaviour of benzene derivatives on two types of liquid crystals deposited on silica gel using hexane as the eluent are presented. An increase in the capacity factor for isomers of benzene derivatives as well as an improvement in their separation in the phase transition interval with increasing temperature is observed.

INTRODUCTION

Due to their universal properties, liquid crystals are used in different spheres of science and technology, in particular in chromatography. They are employed as sorbents in gas chromatography for separation of organic isomers^{1,2}, polynuclear aromatic hydrocarbons³ and optical isomers⁴. In liquid chromatography they have been used much less intensively. Cholesteric liquid crystals with transition interval 20–100°C chemically bonded to silica gel have been studied for steroid separation⁵. It was found that the sorbents exhibited properties of liquid crystals and the capacity ratio of the steroids increased with increasing column temperature.

In the present paper we describe the use of liquid crystals of nematic and cholesteric types as sorbents in liquid chromatography with hexane as the eluent.

EXPERIMENTAL

Liquid crystals of 4-ethoxybenzylidene-4'-*n*-butylaniline (EBBA) from a solution in chloroform or of cholesteryl oleate from an alcoholic solution were deposited on silica gel Silasorb-600 (mean particle size 5 μm) to an amount of 20% and 40%, respectively. EBBA has a phase transition at 35.5–77.5°C, cholesteryl oleate at 20–33°C.

Measurements were made with a Tsvet-304 liquid chromatograph equipped with an UV detector (254 nm). The sample size was 1–2 μl . Averages of three determinations were taken.

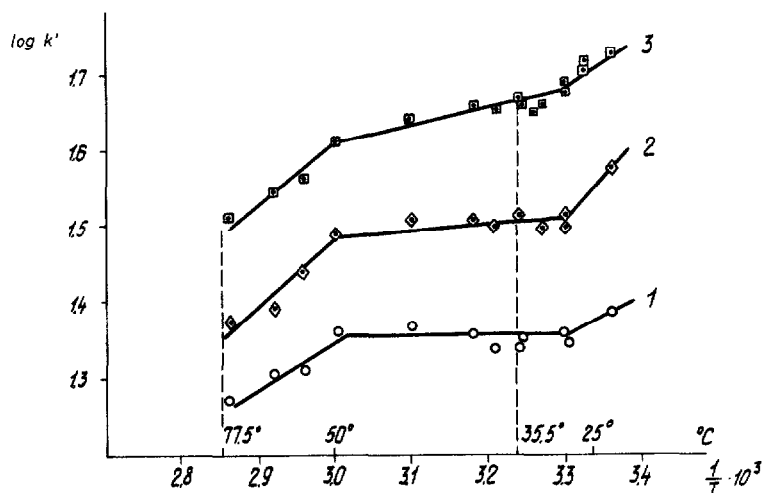


Fig. 1. Dependence of $\log k'$ on reciprocal temperature for dinitrobenzene isomers (1 = *para*; 2 = *meta*; 3 = *ortho*). Column: 20% EBBA on Silasorb-600, 10 cm \times 0.4 cm, 5 μ m. Eluent: hexane; flow-rate 3 cm³/min.

RESULTS AND DISCUSSION

The retention times, capacity factors (k') and selectivities of *o*-, *m*- and *p*-isomers of dinitrobenzene, nitrotoluene and bromonitrobenzene were measured within the phase transition temperature intervals.

Fig. 1 shows the dependence of $\log k'$ on the reciprocal temperature for dinitrobenzene isomers; the adsorbent phase transition is clearly seen, but is shifted to lower temperatures in comparison with pure EBBA.

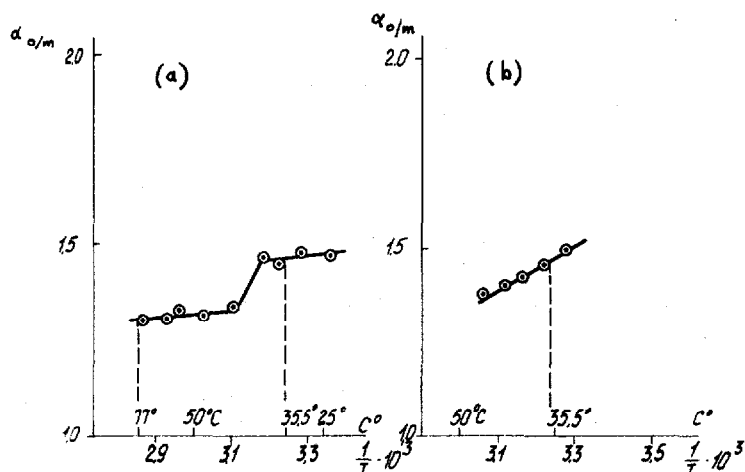


Fig. 2. Dependence of the selectivity coefficient, $\alpha_{p/m}$, for *o*- and *m*-isomers of dinitrobenzene on the reciprocal temperature. Columns: (a) 20% EBBA on Silasorb-600, 10 cm \times 0.4 cm, 5 μ m; (b) Silasorb-600, 5 μ m, 10 cm \times 0.4 cm. Eluent: hexane, flow-rate 3 cm³/min.

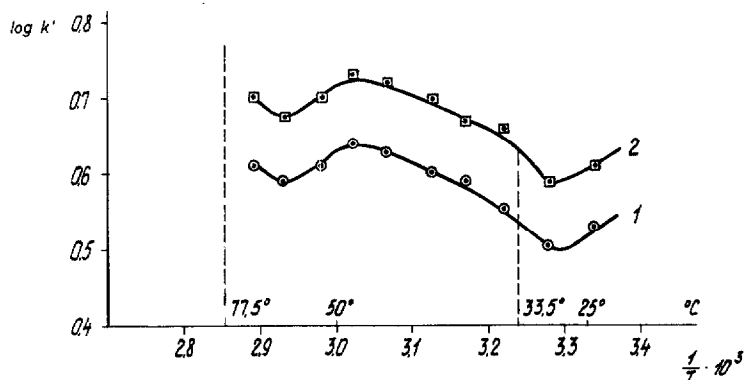


Fig. 3. Dependence of $\log k'$ on reciprocal temperature for *o*-nitrotoluene (1) and *p*-nitrotoluene (2). Column: 20% EBBA on Silasorb-600, 10 cm \times 0.4 cm. Eluent: hexane, flow-rate 1 cm³/min.

The dependence of the selectivity coefficient, α , for *o*- and *m*-isomers of dinitrobenzene on the reciprocal temperature was compared for Silasorb-600 with 20% EBBA and for pure Silasorb-600 under identical conditions (Fig. 2). In the phase transition interval ($\approx 36^\circ\text{C}$), the decrease in α on Silasorb-600 with 20% EBBA may correspond to the point of transition into the mesophase. It is connected with the effect on retention of liquid crystal deposited on Silasorb-600, and is not observed on pure Silasorb-600 where the selectivity decreases regularly with temperature.

A phase transition interval (30–60°C) is also observed for nitrotoluene (Fig. 3), the logarithm of the capacity factor increasing with increasing column temperature. Here again the phase transition is shifted to lower temperatures, which is probably due to the adsorption on the silica gel surface.

The dependence of $\log k'$ on reciprocal temperature for nitrotoluene isomers was determined on a liquid crystal of the cholesteric type on which in the mesophase an increase in retention also occurs. This increase occurs in the liquid phase at a temperature higher than that of the phase transition (33°C) (Fig. 4). Such behaviour

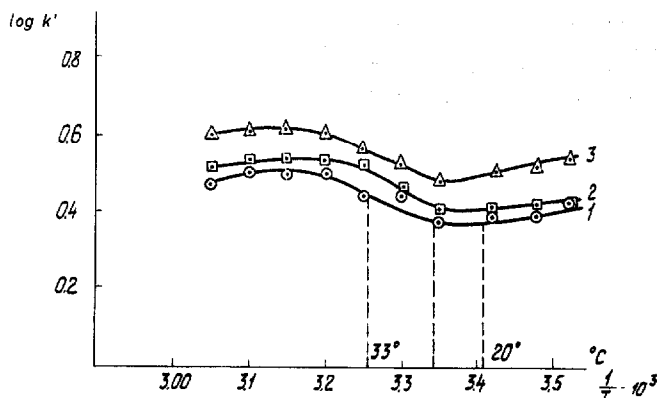


Fig. 4. Dependence of $\log k'$ on reciprocal temperature for nitrotoluene isomers (1 = *ortho*; 2 = *meta*; 3 = *para*). Column: 40% cholesteryl oleate on Silasorb-600, 10 cm \times 0.4 cm, 5 μm . Eluent: hexane; flow-rate 1.5 cm³/min.

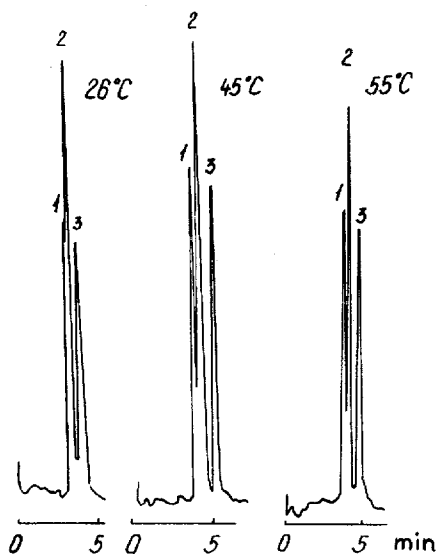


Fig. 5. Chromatograms of nitrotoluene isomers (1 = *ortho*; 2 = *meta*; 3 = *para*) at different temperatures. Conditions as in Fig. 4.

of isomers on the surface of liquid crystals deposited on silica gel by adsorption provides an improvement in separation (Fig. 5) at high temperatures.

Liquid chromatography may be used to identify the intervals of phase transition of liquid crystals into the mesophase. Sorbents with liquid crystals deposited on silica gel may be used selectively to separate isomers of otherwise poorly separated compounds.

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