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Analysis of Chromatographic Parameters in the Systems with Ternary Mobile Phases. II. Two Polar Solvents in Mobile Phase

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**ANALYSIS OF CHROMATOGRAPHIC
PARAMETERS IN THE SYSTEMS
WITH TERNARY MOBILE PHASES.
II. TWO POLAR SOLVENTS IN
MOBILE PHASE**

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ABSTRACT

The experimental data are presented on Gibbs triangles in the form of isolines. The effect of change of the type of two polar mobile phase components on the chromatographic parameters of model substances has been investigated. The presence of alcohol in ternary mobile phase leads to formation double or "closed" R_F isolines on Gibbs triangles. Such presentation of the experimental data allowed to complex analysis of the chromatographic process in systems with ternary mobile phase. The measurements were made by adsorption thin layer chromatography on silica gel at temperature 293 K.

INTRODUCTION

The composition of the mobile phase is one among basic factors influencing the course of the chromatographic process (1). At present the multi-component mobile phases are more and more frequently used. In many cases this permits to choose the optimal conditions for the chromatographic separation of the components of a given mixture. For this reason the examination of effect of the nature and concentration of individual components of multicomponent mobile phase on the properties of investigated chromatographic systems appears to be very interesting. In previous paper (2) the values of chromatographic parameters of adsorption liquid chromatography have been considered. The effect of change of the type (or class according to Pimentel and McClellan classification (3)) of one mobile phase component on chromatographic parameters of model substances has been investigated. Two solvents which were not changed belonged to N class (carbon tetrachloride and benzene). Third component belonged successively to A (trichloroethylene), B (acetone) or AB (n-propanol) classes (2). In this way three types of ternary mobile phases characterized by increasing elution power of third component have been obtained. Experi-

mental data (R_F) determined for such chromatographic systems were marked on Gibbs triangle, forming the systems of the isolines connecting the points corresponding to equal values of investigated parameters (2). Such presentation of the experimental data allowed to the analysis of the chromatographic process in systems with ternary mobile phase.

The change of the type of only one mobile phase component limits the range of chromatographic parameters changes of the chromatographed substances. For this reason it was decided to investigate the changes in the course of isolines of chromatographic parameters R_F on Gibbs triangle with the changes of the type of two components of mobile phase. In this way the extended picture, comparing to previous investigations (2) of the effect of mobile phase composition on the course of the chromatographic process can be received.

EXPERIMENTAL

The measurements were carried out by thin layer chromatography method using saturated Stahl chambers at 293 K. Silica gel 60H (Merck, Darmstadt, FRG) was used as adsorbent. The chromatograms were developed at the distance of 16 cm; the test substances were

visualized in iodine vapours. Six model substances i.e. naphthalene (N), fluorenone (B), o-nitrotoluene (B), o-nitrobromobenzene (B), o-nitroaniline (AB) and o-nitrophenol (AB⁶) were used. Following solvents and their mixtures were used as mobile phases:

- pure solvents: benzene, trichloroethylene, acetone and n-propanol,
- binary mixtures of above solvents containing 0.1, 0.3, 0.5, 0.7 and 0.9 molar fraction of more polar solvent,
- ternary mixtures of above solvents.

Binary mixtures: acetone + n-propanol, trichloroethylene + n-propanol and trichloroethylene + acetone were used as basic solutions to ternary mobile phases preparation. In these mixtures the ratios of molar fractions of n-propanol to acetone, trichloroethylene to acetone and thichloroethylene to n-propanol were 1:3, 1:1 and 3:1. To basic solutions a third non-polar component, benzene, was added in such amounts that its concentrations were equal to 0.1, 0.3, 0.5, 0.7 and 0.9 molar fraction.

RESULTS AND DISCUSSION

Three systems of ternary mobile phases have been prepared. One component belonged to N class (benzene)

was always the same, and two others have belonged to A, B or AB classes. The compositions of these systems were as follows:

- benzene + trichloroethylene + acetone (N+A+B)
- benzene + trichloroethylene + n-propanol (N+A+AB)
- benzene + acetone + n-propanol (N+B+AB)

The results obtained in this paper together with our earlier investigations (2) permit to complex study of adsorption liquid chromatography process in the systems with ternary mobile phases.

The results of chromatographic measurements for model substances were presented on Gibbs triangle forming the systems of isolines i.e. lines connecting the points corresponding to the same R_F values. The examples of such procedure are illustrated in Figures 1-9 in which the experimental data obtained for three chromatographed substances (naphthalene, o-nitrotoluene, o-nitroaniline) are given. Analysis of the results and the conclusions resulting from this analysis are based, however, on complete experimental material. The experimental data obtained for three ternary mobile phases containing one polar component were utilized as basic data (Figs 1,2,3 first triangles on left). The Figures 1,2,3 (second and third triangles) show the changes of R_F values isolines due to introduction of second polar component to ternary mobile

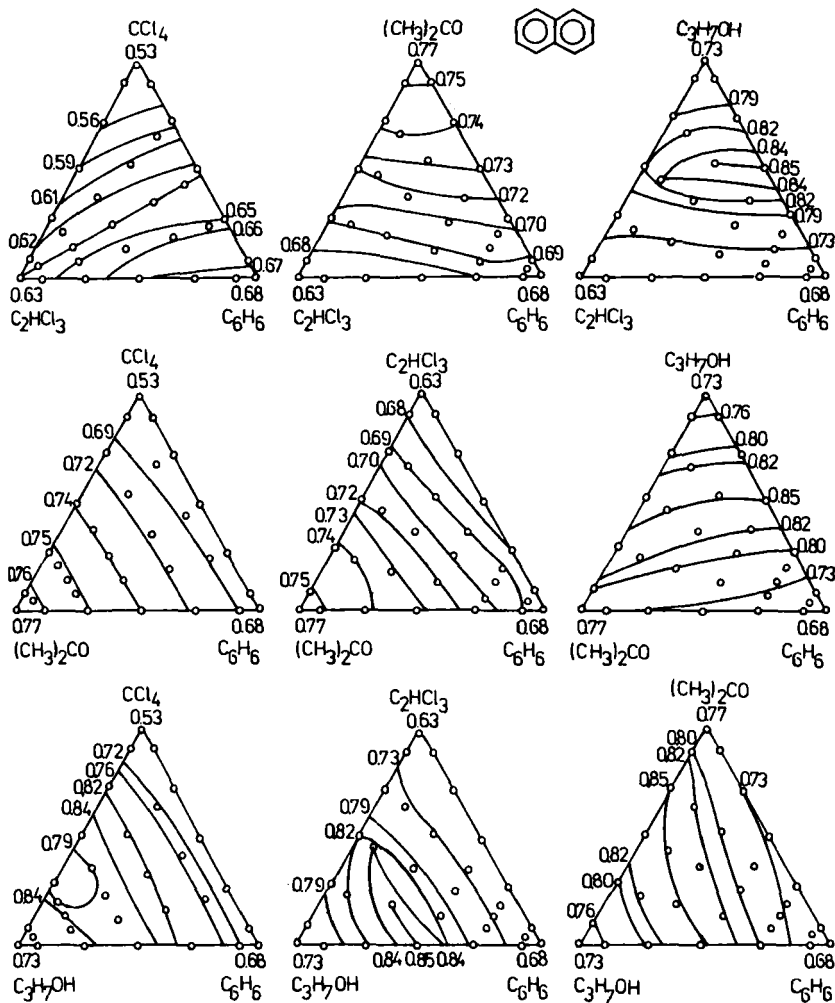


Fig.1 The R_F values isolines systems plotted for naphthalene in the ternary mobile phases containing one or two polar solvents

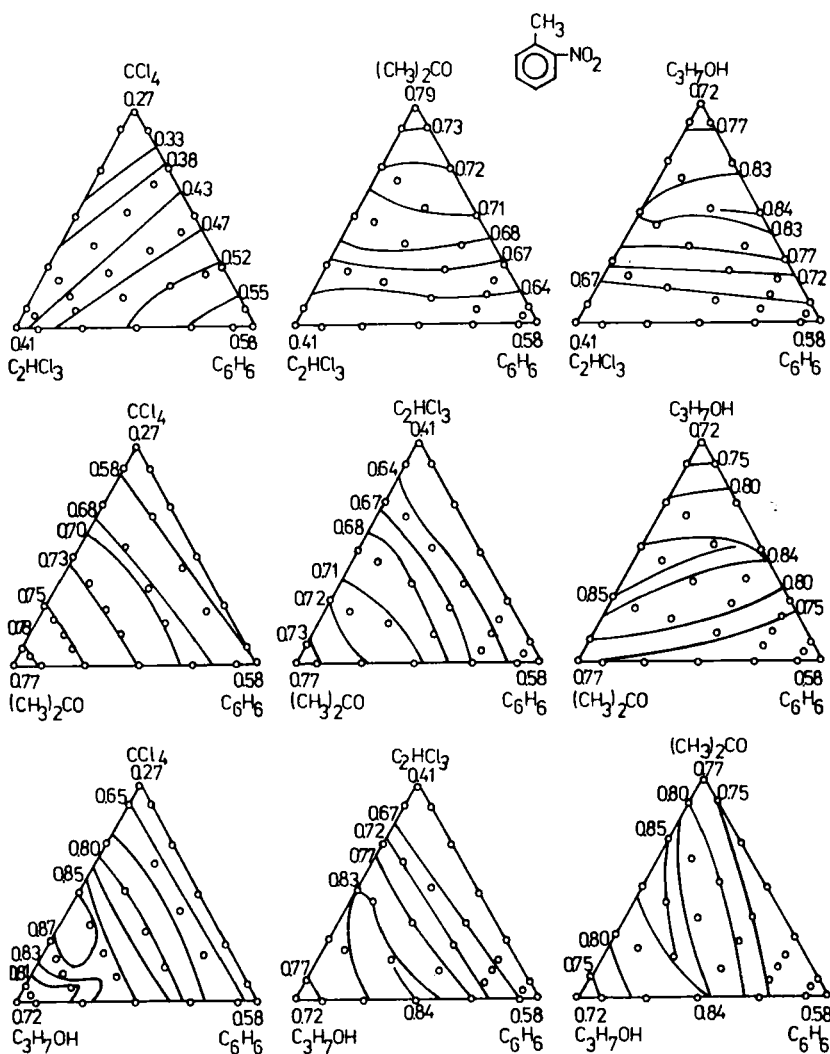


Fig.2 The R_F values isolones systems plotted for o-nitrotoluene in the ternary mobile phases containing one or two polar solvents

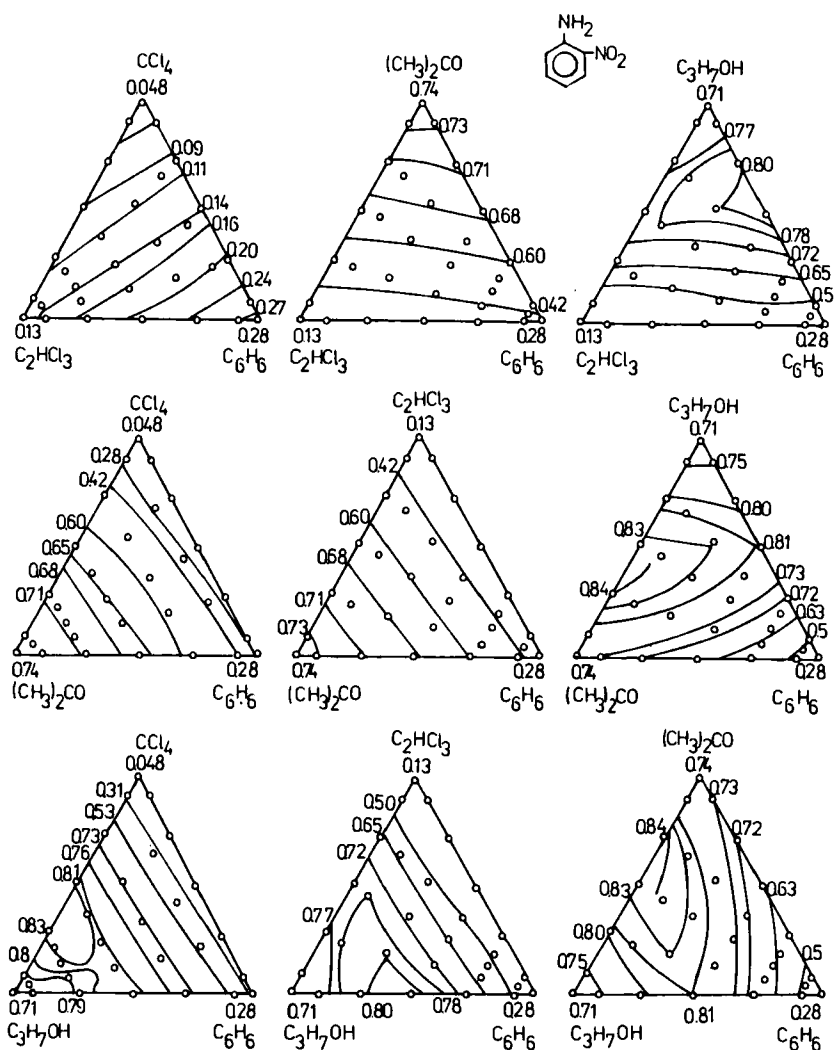


Fig.3 The R_F values isolines systems plotted for o-nitroaniline in the ternary mobile phases containing one or two polar solvents

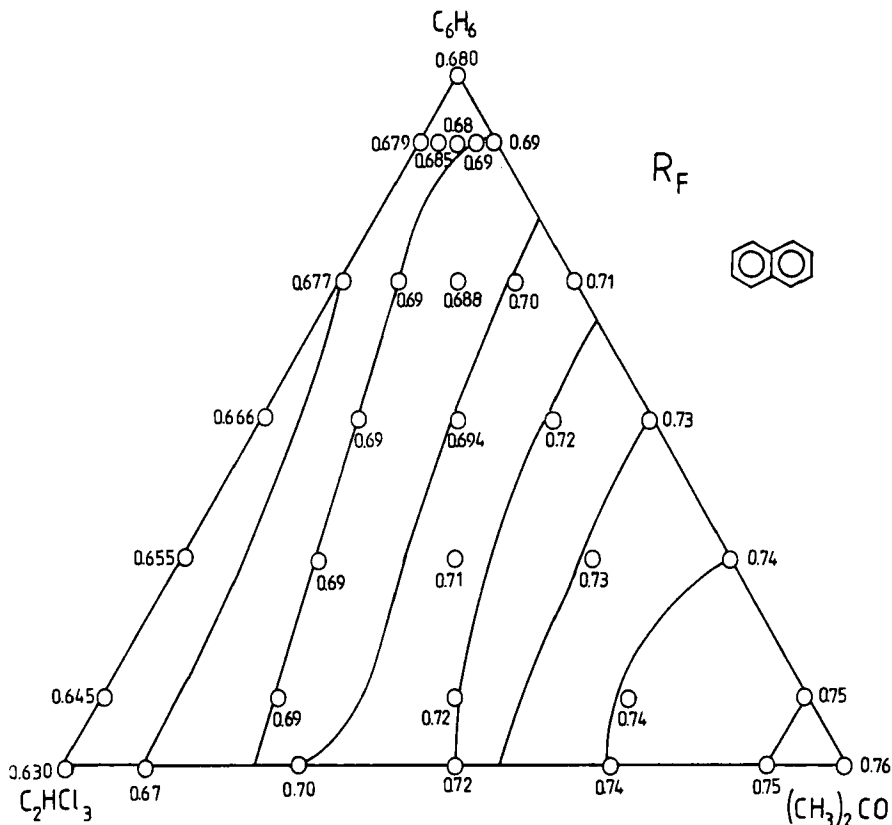


Fig.4 Isolines of R_F values for naphthalene in trichloroethylene + benzene + acetone system

phase. Trichloroethylene, acetone or n-propanol were added to these phase instead of carbon tetrachloride. For initial systems the localization of R_F values isolines of the chromatographed substances depends on the power of mobile phase polar component. For all systems the isolines are less or more parallel to

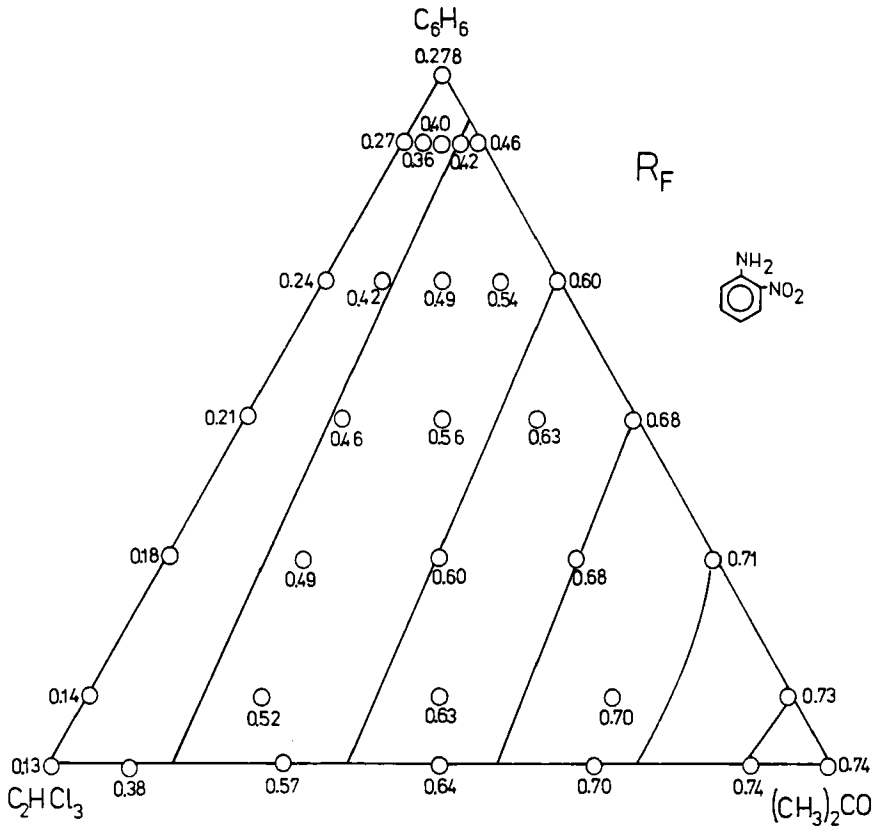


Fig.5 Isolines of R_F values for o-nitroaniline in trichloroethylene + benzene + acetone system

this side of Gibbs triangle which lies opposite to vertex corresponding to R_F values of the substance chromatographed in pure polar solvent (Figs 4,5). If all three mobile phase components have similar elution powers the isolines systems prefer no solvents (Figs 1,2,3 the first triangle on the left side).

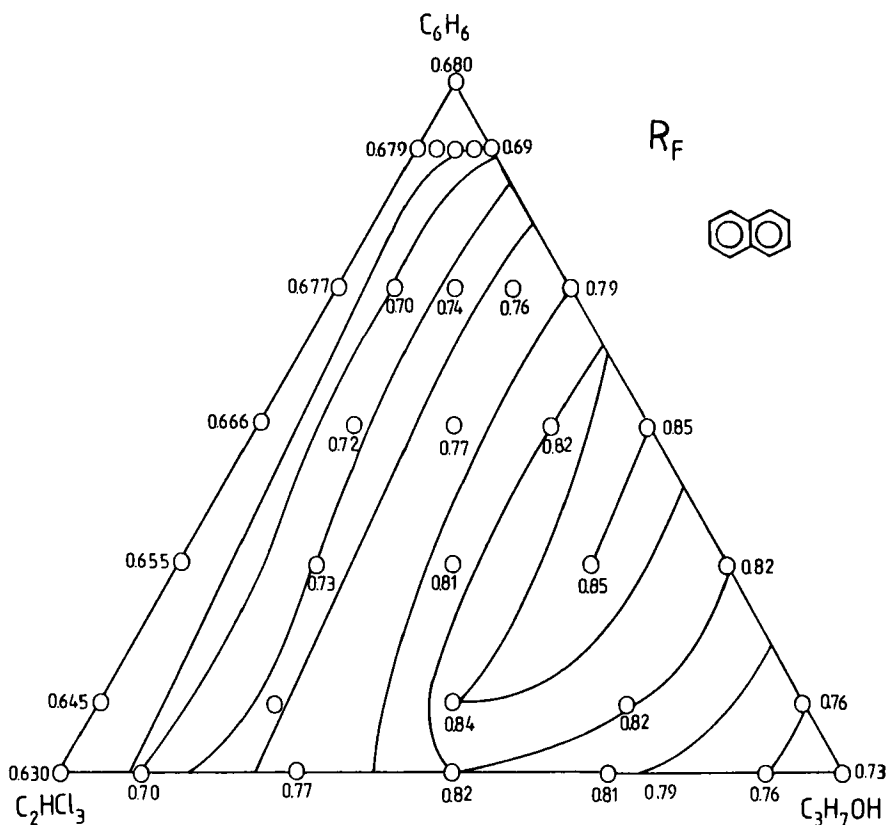


Fig.6 Isolines of R_F values for naphthalene in tri-chloroethylene + benzene + n-propanol system

The introduction of second polar component to mobile phase can change not only R_F values but also the localization of the isolines on the Gibbs triangle. The change in the course of R_F isolines takes place when elution power of introduced solvent is higher than elution power of polar component of initial mo-

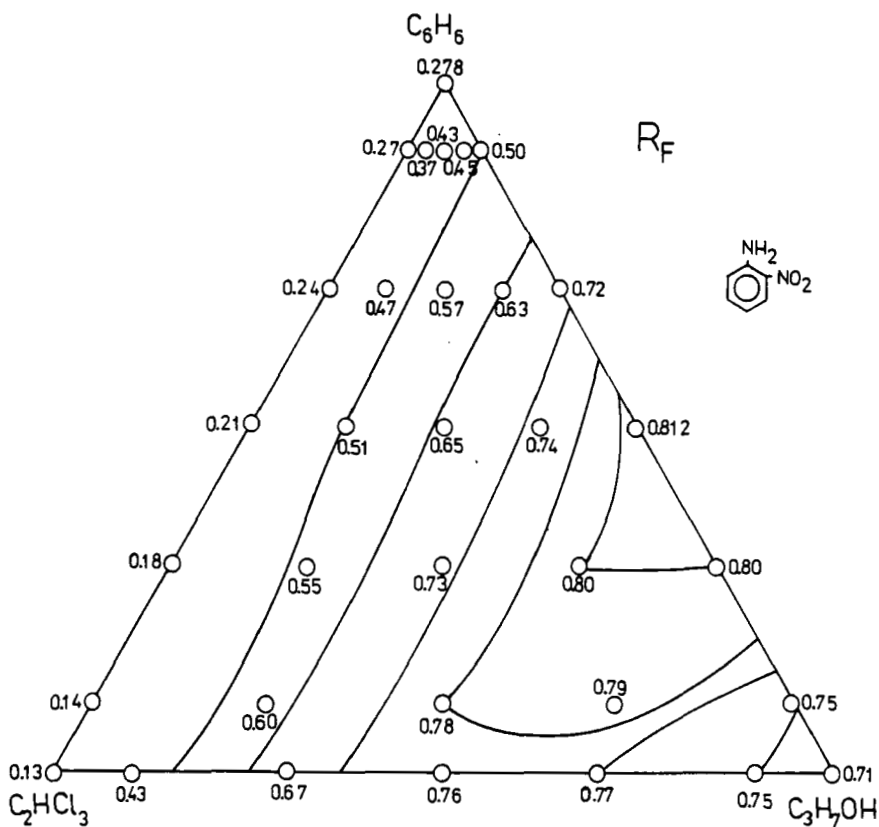


Fig.7 Isolines of R_F values for o-nitroaniline in trichloroethylene + benzene + n-propanol system

bile phase (Fig. 1,2,3). The changes in the R_F isolines localization will become evident after addition of acetone or n-propanol to the system containing trichloroethylene or after addition of n-propanol to the system containing acetone. The presence of alcohol in ternary mobile phase leads to formation double

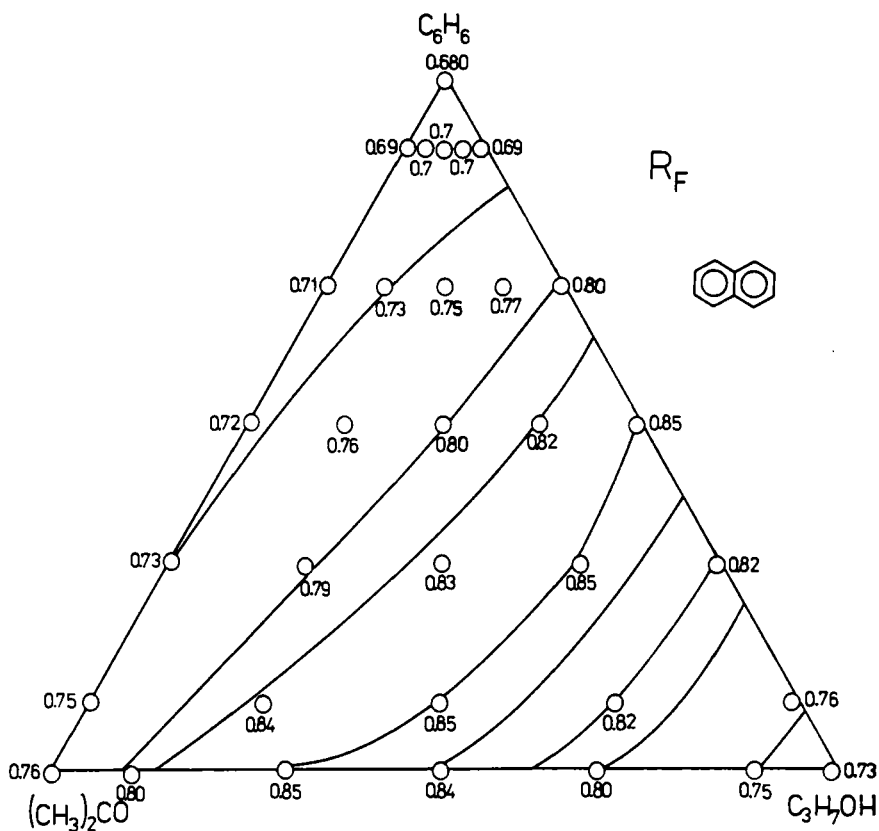


fig.8 Isolines of R_F values for naphthalene in acetone + benzene + n-propanol system

or "closed" R_F isolines on Gibbs triangle (Figs 6-9). This is strictly connected with the presence of the maxima on the graph of $R_F=f(\text{mobile phase composition})$ function. Localization of R_F isolines on Gibbs triangle depends in little part on the nature (class) of test substances. Thus it can be concluded that in the case

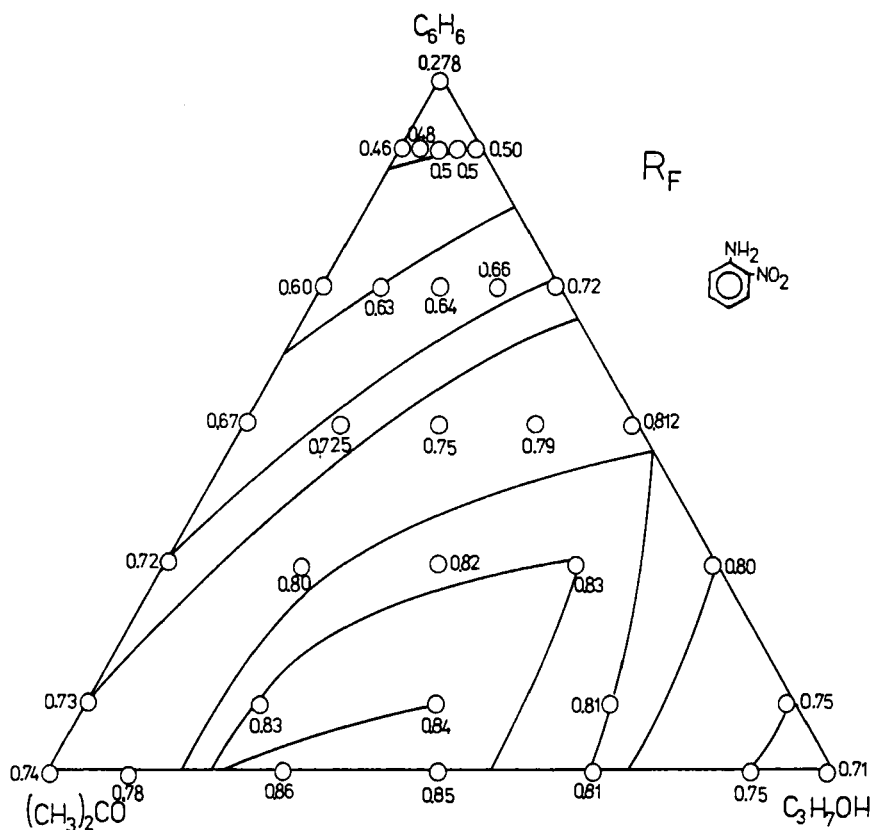


Fig.9 Isolines of R_F values for o-nitroaniline in acetone + benzene + n-propanol system

of considered systems the most important effects influencing on adsorption liquid chromatography process, for a given adsorbent, are the nature and composition of mobile phase. This fact betokes to a predominant role of molecular, mainly specific, interactions in the mobile phase on this process. Analysis

of the results presented in this and previous paper (2) lead to the following conclusions:

- presentation of R_F , also R_M or k' , values of the chromatographed substances in the form of isolines plotted on Gibbs triangle makes possible a complex analysis of the mobile phase composition effect on chromatographic process,
- the course of R_F isolines depends mainly on the mobile phase composition and in little part on the nature of the test substance. In this case the specific molecular interactions between mobile phase components play an important role,
- the presence of the extreme in the course of $R_F = f(\text{mobile phase composition})$ function determines the existence of two isolines characterized by the same R_F values. Sometimes a single isoline characterized by extremal R_F value is also observed.
- differences in polarities (in elution power) of individual mobile phase components determine a shape of the R_F values isolines,
- analysis of the course of R_{F1}/R_{F2} values isolines (2) permits to the choice of such mobile phase composition which assures optimal separation of of test mixture.

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