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Ternary mobile phases in adsorption thin-layer chromatography

In thin-layer chromatography multi-component solvent systems are generally used as mobile phases due to their adsorptive capacity for the substances being separated. Establishing an optimal chromatography system and the separation of substances is easiest if mono- or di-component solvent systems are used. The results of investigations for both systems can be easily presented on simple diagrams, *i.e.* chromatographic spectra or curves giving the relationship R_F = f(composition of the solvent)[1-8].

The task is much more difficult with three-component solvent systems; few reports have so far dealt with this problem. They have mostly shown the relationship R_F = f(the composition of a three-component solvent system) on spatial diagrams[9]. However, diagrams of this type serve only theoretical considerations and have never been used in analytical practice. A lack of general rules does not make the choice of solvents for the system easy.

The aim of the present paper is to study the behaviour of some substances of different chemical structure during chromatography in three-component solvent systems, in addition to find an easy way of choosing optimal three-component solvent systems and presenting them on diagrams which would be available for practical use.

Experimental

Investigations were carried out by ascending TLC, using silica gel (Merck) as adsorbent. Layers of the adsorbent, 0.3 mm thick, were heated for 2 h at 135°. The following components were used as model substances: quinoline (class B according to PIMENTEL AND MCCLELLAN's classification[10]; acridine (class B); acridine orange (class B); carbazole (class AB); and fluoranthene (class N).

The spots of acridine, acridine orange and fluoranthene were detected in UV light, and those of carbazole and quinoline by spraying with H_2SO_4–$KMnO_4$ and Dragendorff's reagent, respectively[6].

The chromatograms were developed to a distance of 16 cm, using a ternary mobile phase made up of the following solvents:

1. Water (class AB)
2. Methanol (class AB)
3. Dioxane (class B)
4. Chloroform (class A)
5. Carbon tetrachloride (class N)

The organic solvents were dehydrated[8,11]. In each three-component mixture the concentration of two of the components changed steadily, the amount of the third component added being kept constant. However, the composition of the whole three-component mixture changed, as seen on the diagrams.

Measurements were made in a thermostated room at 25°, to ensure the reproducibility of the R_F values which were determined with an accuracy of ±0.02 R_F[12-14].

Results

The results are presented on diagrams as a two-coordinate system. R_F values of

the substances studied are put on the ordinates, the concentration of three-component mobile phase on the abscissa. The concentration values of this phase (volume fractions) were put on Gibbs' triangles in which the angles correspond to the single solvents. The straight line drawn across the Gibbs' triangle shows the composition of the
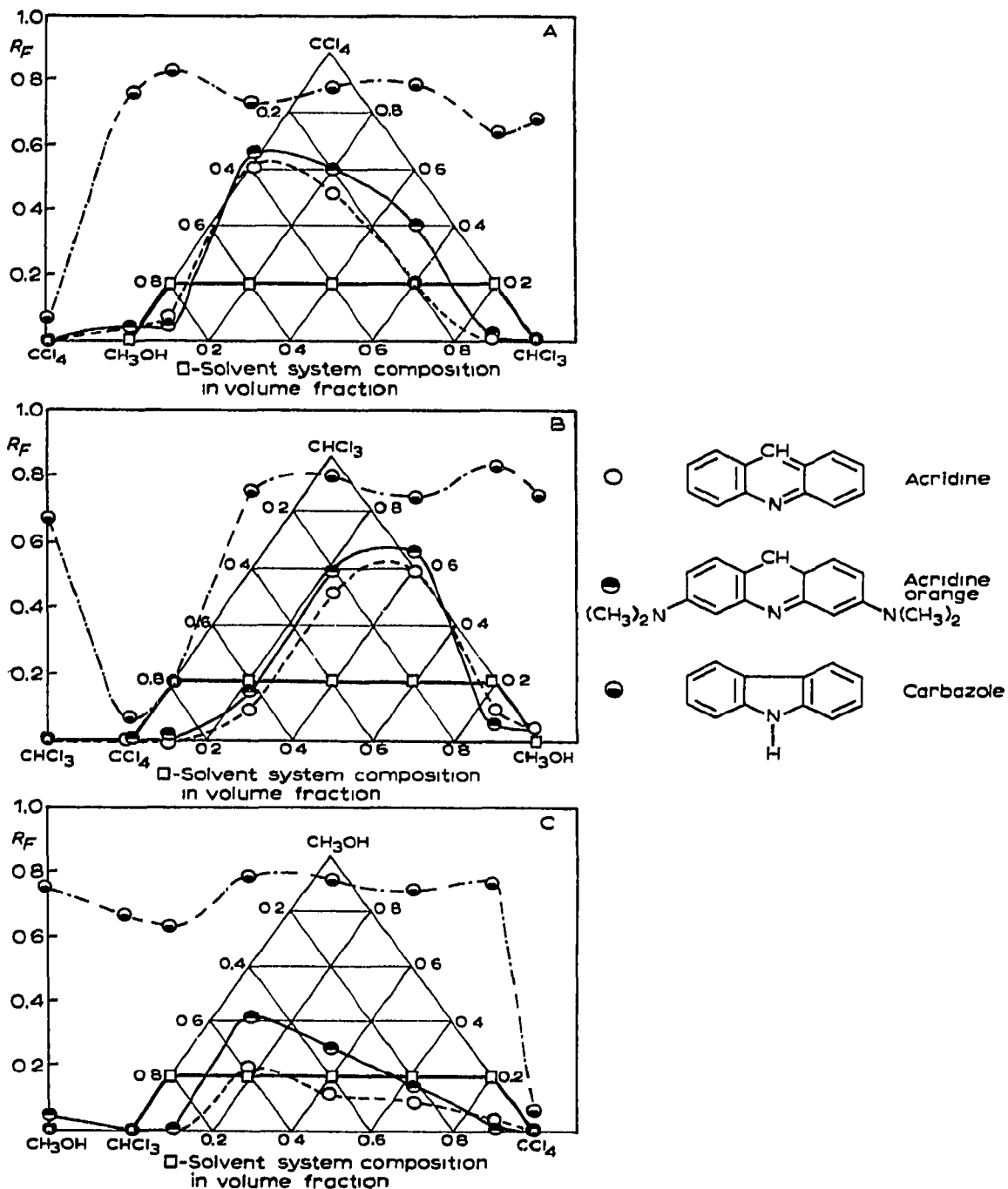

Fig. 1. R_F values of acridine, acridine orange and carbazole obtained by using methanol, chloroform and carbon tetrachloride as a mobile phase. (A) at constant amount of carbon tetrachloride in the mixture, (B) at constant amount of chloroform in the mixture, (C) at constant amount of methanol in the mixture.

three-component mobile phase. The R_F values of the substances separated were placed at points corresponding to the projection of this straight line on the ordinates.

In order to make this diagram clearer, the R_F values were put on the ordinates next to the triangle, in the case of a single solvent—on the top of the triangle.
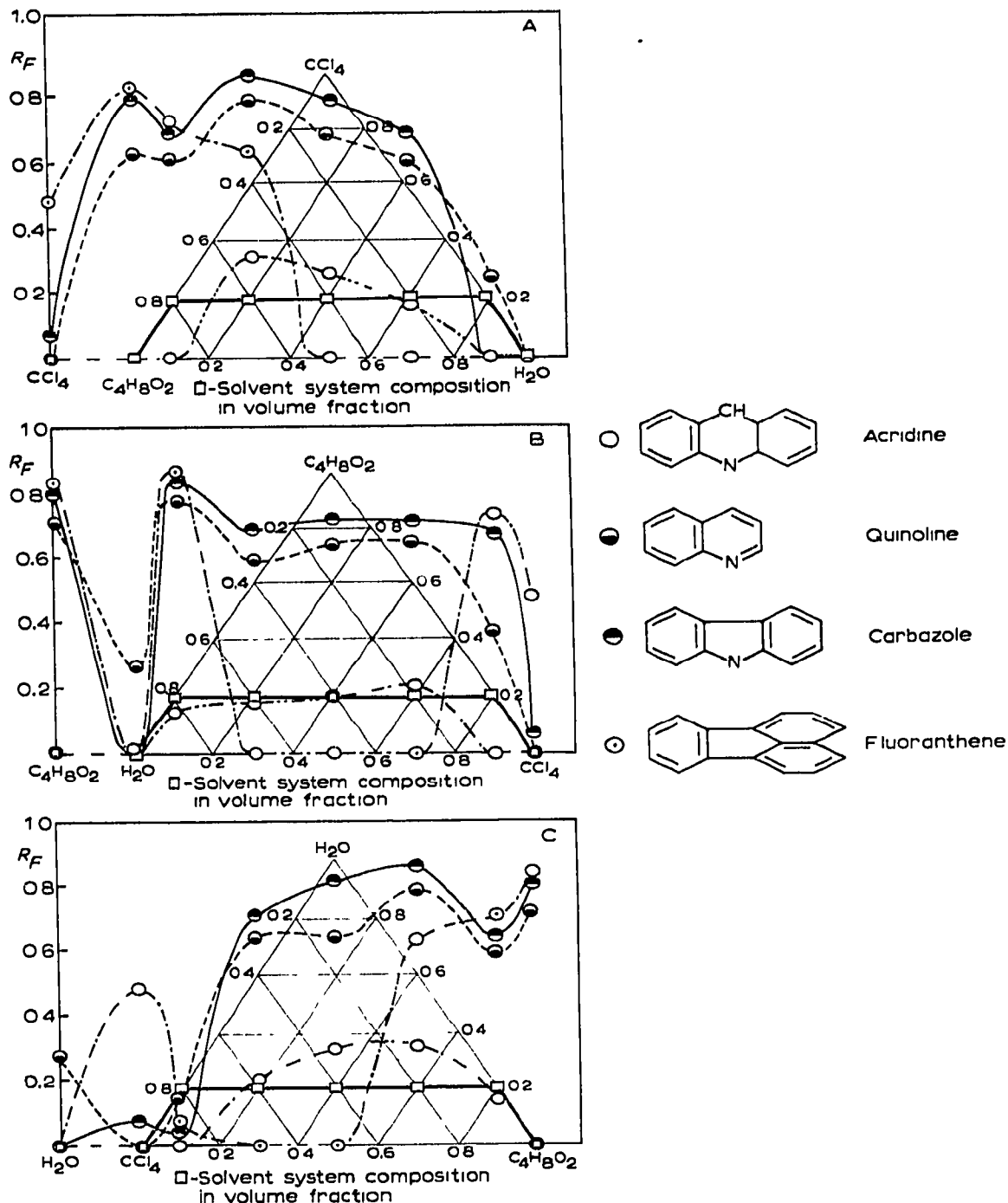

Fig. 2. R_F values of acridine, quinoline, carbazole and fluoranthene obtained by using water, ethyl acetate and carbon tetrachloride as a mobile phase (A) at constant amount of carbon tetrachloride in the mixture, (B) at constant amount of dioxane in the mixture, (C) at constant amount of water in the mixture.

This diagram permits the determination of the relationship of $R_F =$ f(composition of the three-component mobile phase). Diagrams of this type permit one to plan in advance the study of the conditions for the optimal separation of a mixture of substances by means of a three-component mobile phase. The above knowledge also allows the advance determination of the changes in the character of the electron-donor–acceptor properties of the three-component mobile phase, and thus their separation ability, which is necessary for finding an optimal chromatographic system.

Fig. 1 shows curves of function $R_F =$ f(composition of three-component mobile phase carbon tetrachloride–methanol–chloroform) obtained for acridine, acridine orange and carbazole.

The diagrams show that good separation of this mixture is obtained at definite ratio of the three components making up mobile phase: chloroform–methanol–carbon tetrachloride = 0.6:0.2:0.2 (Fig. 1A) and 0.4:0.2:0.4 (Fig. 1C).

It can be seen that the separation of the components of this mixture is hardly possible in the case of a one-component mobile phase and difficult for a two-component mobile phase[8].

Fig. 2 shows to what degree the separation of acridine, quinoline, carbazole and fluoranthene is possible if systematic studies of the separation ability of three-component mobile phase are planned in advance.

The results show that in this case, also, the best separation of all the substances examined only occurred for the following composition of mobile phase: water–carbon tetrachloride–dioxane = 0.2:0.4:0.4 (Fig. 2C).

As seen in the diagram, the separation is impossible if one- or two-component mobile phases are used.

The above considerations show an easy way of presenting the results of the chromatographic separation of mixtures of substances with a three-component mobile phase, as well as of making a choice of the three-component mobile phase if its electron-donor–acceptor properties are known in advance.

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