Thin-layer chromatography of some straight-chain saturated and unsaturated alcohols

Simple saturated alcohols are usually separated by thin-layer chromatography of their 3,5-dinitrobenzoates[1]. This method, however, does not give satisfactory results for some C_6–C_{12} unsaturated alcohols. Recently, SINGH AND GERSHBEIN[2] reported the direct separation of higher alcohols by thin-layer chromatography.

In the present study, the chromatographic behaviour of some C_6–C_{12} saturated and unsaturated alcohols is reported. A well stirred mixture of 25 g silica gel (Kieselgel G, Merck) and 50 ml water was applied to smooth glass plates (20 × 20 cm) at a thickness of about 0.25 mm with a thin-layer applicator. The coated plates were activated by heating for 1 h at 110°. The saturated alcohols were commercial products. The unsaturated alcohols were prepared by condensing the corresponding aldehyde with malonic acid, then reducing the products with lithium aluminum hydride. According to this method[3], the compounds obtained possess the *trans* configuration concerning the double bonds. The samples of alcohols were dissolved in chloroform and the appropriate amounts containing 1–10 μl of the samples were spotted at a distance of 2.0 cm from the lower end of the plate and dried by an air blower. Ascending development, at 25°, was applied in a glass chamber equilibrated with the solvent.

After evaporation of the developing solvent the plates were sprayed with 0.05 % alcoholic Rhodamine 6 G. The spots were detected under short-wave ultraviolet light. Saturated and monoenolic alcohols give yellowish orange colorations against a yellowish background, dienolic alcohols give violet-blue colorations. So they are mutually distinguishable. Otherwise, the spots can be more sensitively detected by

TABLE I

R_F VALUES OF ALCOHOLS

Solvent systems: (I) acetone–water (60:40, v/v); (II) dioxane–water (60:40, v/v).

Compound	*R_F values*	
	I	*II*
Saturated alcohols		
Hexanol	0.90	0.75
Heptanol	0.78	0.67
Octanol	0.63	0.62
Nonanol	0.41	0.55
Decanol	0.30	0.45
Undecanol	0.24	0.40
Dodecanol	0.19	0.37
trans-2-Monoenols		
Hexenol	0.93	0.78
Octenol	0.72	0.69
Decenol	0.40	0.52
trans-2,4-Dienols		
Hexadienol	0.95	0.83
Octadienol	0.86	0.73
Decadienol	0.55	0.60
Dodecadienol	0.38	0.35

sprayimg with concemtrated sulfuric acid containing 0.5 % potassium dichromate. Mamy kimds of solvemt systems were tested as developing solvent, the solvent consisting of acetome–watter ((60:40„ v//v)) was the most useful. Chromatographic data for various kimds of alcohol are summarized im Table I. R_F values represent the average of three experimments. Om plottimg the R_F values as a function of the chain length for the components of various homologous series„ smooth curves are obtained (Fig. 1). The R_F values of 2„4-alkadiemols are higher tham those of the corresponding 2-alkenols, those of alkamols are the lowest of them.

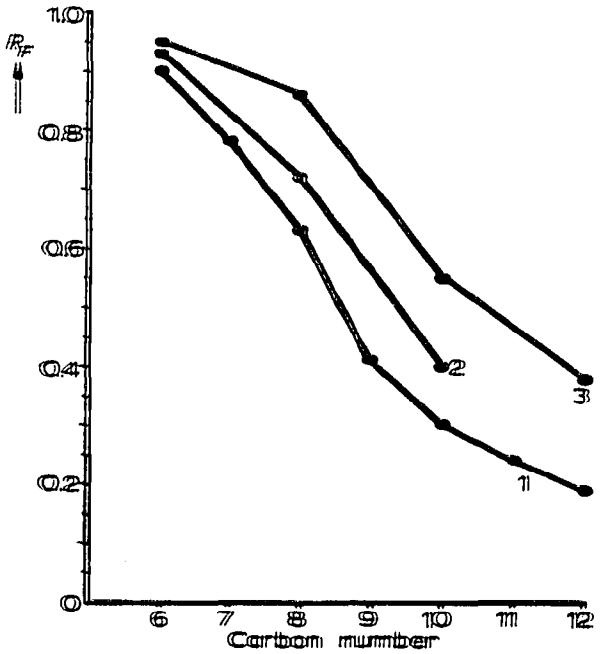

Fig. 1. Migratiom of variours alcohols. Developing solvent: acetone–water, 60:40 v/v. 1 = Saturated alcohols;; 2 = *trams*-2-allkemols;; 3 = *trams*-2-*trams*-4-allkadiemols.

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