

DNPS-indole derivatives in most solvents. After chromatography of this treated mixture in solvent D the R_F values for the four DNPS-derivatives were found identical to standard compounds either alone or in mixture.

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Polyamide-silica gel thin-layer chromatography of food preservatives

The chromatography of food preservatives has been studied by numerous investigators. The separation of these preservatives on thin layers of cellulose acetate-polyamide¹, cellulose² and silica gel³ has been reported, but there is no report on separation by polyamide-silica-gel layers. In a previous report⁴, better separation of red food dyes was obtained with polyamide-silica gel layers; therefore, this method was further applied to separate ten preservatives. For comparison, the thin-layer chromatography of only polyamide and of only silica gel is also described.

Experimental

Preparation of polyamide-silica gel mixed layer. Ten grams of polyamide (ϵ -polycaprolactam CM 1007S of Toyo Rayon Co., Tokyo, Japan) were dissolved in 80 ml of 90% formic acid; then 20 ml of distilled water were added. After gentle warming (below 40°) and stirring, a homogeneous solution was obtained. It was then cooled to room temperature, and 52 g of Silica Gel G (E. Merck) were added. Of the previous solution 200 ml were poured into a dish (14.5 × 19.5 × 2.5 cm) into which a glass plate (12 × 14 × 0.1 cm) was dipped. Both sides of the glass were covered homogeneously.

J. Chromatog., 44 (1969) 201-203

The glass was hung for 2 min over the dish to let the excess solution drain off. It was then air dried for 3 h and heated at 100° for 30 min. These layers can be stored for a long period.

Preparation of polyamide layer. Instead of 10, 20 g of polyamide were dissolved before proceeding as described in the previous method, but without adding Silica Gel G.

Preparation of silica-gel layer. Dilute slurries of Silica Gel G (45 g to 100 ml of water) were sprayed at 2 kg/cm³ pressure from a distance of 20 cm onto 8 horizontal glass plates (12 × 14 cm) which were then dried at 100° for 30 min. The thickness of the layers was about 250 μ.

Chromatographic procedure. A 0.2% alcoholic solution of samples was applied to the starting line 1.5 cm from the bottom of the layer, and the plate was developed by ascending techniques. The chamber had been equilibrated with the respective solvent for 30 min before use.

Visualization. The layers were sprayed with a 0.07% alcoholic solution of Rhodamine B, and deep-violet spots could be observed under UV light at 366 mμ.

Results and discussion

R_F values obtained with two solvent systems are given in Table I. It is interesting to note that the R_F values of the *p*-hydroxybenzoic acid ester in the two solvent systems are reversed. In the nonaqueous system (solvent I), the R_F values increase with an increase in the molecular weight of the esters, *i.e.* the order of R_F values is the same as that obtained by previous workers¹⁻³. However, in the aqueous system (solvent II), the R_F values decrease with an increase in the molecular weight.

In both solvent systems, when using polyamide-silica gel mixed layers the R_F values are lower and separation is better than when polyamide and silica gel layers are employed. A 10-cm ascent from the origin is more rapid using the mixed layers

TABLE I

CHROMATOGRAPHIC DATA

Solvent I: *n*-hexane-benzene-glacial acetic acid (1:1:1); solvent II: water-28% ammonia solution (20:5). a, R_F value obtained on polyamide-silica gel layer; b, silica gel layer; c, polyamide layer.

No. Substance	Solvent I			II		
	a	b	c	a	b	c
1 Methyl <i>p</i> -hydroxybenzoate	0.30	0.73	0.61	0.55	0.91	0.78
2 Ethyl <i>p</i> -hydroxybenzoate	0.35	0.77	0.69	0.46	0.88	0.70
3 Propyl <i>p</i> -hydroxybenzoate	0.41	0.78	0.74	0.34	0.79	0.59
4 Isopropyl <i>p</i> -hydroxybenzoate	0.44	0.78	0.74	0.32	0.77	0.60
5 Butyl <i>p</i> -hydroxybenzoate	0.49	0.79	0.78	0.24	0.69	0.49
6 Isobutyl <i>p</i> -hydroxylbenzoate	0.53	0.80	0.81	0.21	0.69	0.50
7 Sorbic acid	0.88	0.87	0.98	0.73	0.95	0.94
8 Benzoic acid	0.84	0.89	0.98	0.64	0.96	0.88
9 Salicylic acid	0.66	0.84	0.62	0.49	0.96	0.74
10 Dehydroacetic acid	0.74	0.96	0.80	0.62	0.95	0.85
Time required (min) ^a	90	25	240	130	15	180

^a Time required to ascend 10 cm from origin.

can using polyamide layers. The content (16%) of polyamide in this polyamide-silica mixed layer is higher than that in the previous report (12%)⁴ for getting a more able layer.

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J. Chromatog., 44 (1969) 201-203

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Polyamide-kieselguhr thin-layer chromatography of yellow food dyes

The thin-layer chromatography of food dyes has been studied by numerous investigators. The separation of synthetic food dyes by thin layers of cellulose¹, starch², silica gel³, aluminum oxide⁴ and polyamide⁵ has been reported. Recently, a better separation of red food dyes was obtained by CHIANG⁶ with a mixed polyamide-silica gel thin layer. Therefore, further modification of this method was tried. In this note, the separation of five yellow food dyes and three harmful yellow dyes (auramine, metanil yellow and picric acid) by mixed polyamide-kieselguhr thin-layer chromatography is described. For comparison, the thin-layer chromatography on only polyamide and on only kieselguhr is also described.

Experimental

Preparation of polyamide-kieselguhr mixed layer. Ten grams of polyamide chip (Nylon 6, type 1022B of UBE Industrial Ltd., Osaka, Japan) were dissolved in 80 ml of 90% formic acid; then 20 ml of distilled water were added. After warming (below 60°) and stirring, a homogeneous solution was obtained. It was then cooled to room temperature, and 40 grams of Kieselguhr G (E. Merck) were added. Of the previous solution 200 ml were poured into a dish (14.5 × 19.5 × 2.5 cm) into which a glass plate (12 × 14 × 0.1 cm) was dipped. Both sides of the glass were covered homogeneously. The glass was hung for 2 min over the dish to let the excess solution drain off. It was then air dried for 3 h and heated at 100° for 30 min.

Preparation of polyamide layer. Instead of 10 g, 20 g of polyamide were dissolved before proceeding as described in the previous method, but without adding Kieselguhr G.

Preparation of kieselguhr layer. Dilute slurries of Kieselguhr G (45 g in 100 ml of water) were sprayed at 2 kg/cm³ pressure from a distance of 20 cm onto 8 horizontal glass plates (12 × 14 cm) which were then dried at 100° for 30 min. The thickness of the layers was about 250 μ.

J. Chromatog., 44 (1969) 203-204