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Polyamide-silica gel thin-layer chromatography of red food dyes

The separation of synthetic food dyes by thin layers of cellulose¹, silica gel², aluminum oxide³, polyamide⁴, and paper chromatography have been reported, but none of these techniques gave entirely satisfactory results. The application of a mixed polyamide-silica gel thin layer in the chromatographic separation has not been reported. In this note, the separation of eleven red food dyes and two harmful red dyes (Rhodamine B and Coralline) by this mixed layer is described.

*Experimental**Preparation of polyamide-silica gel mixed layer*

Seven grams of polyamide (*ε*-polycaprolactam CM 1007S of Toyo Rayon Co., Tokyo, Japan) were dissolved in 100 ml of 75 % formic acid. After gentle warming, a homogeneous solution was obtained; then 52 g of Silica Gel G (E. Merck) was added. Of

TABLE I

CHROMATOGRAPHIC DATA

Solvent I: isopropyl alcohol-5 % NH₄Cl solution (4:1.5). Solvent II: ether-isopropyl alcohol-5 % NH₄Cl solution (0.5:1:1). Solvent III: CHCl₃-isopropyl alcohol-5 % NH₄Cl solution-glacial acetic acid (0.5:2.5:1.0:0.5). Solvent IV: *n*-butanol-ethanol-5 % sodium citrate solution (3:2:1.5). Solvent V: CHCl₃-isopropyl alcohol-5 % NaCl solution-glacial acetic acid (0.5:2.5:0.5:0.1).

No.	Substance	<i>R_F</i> value ^a				
		I	II	III	IV	V
1	Ponceau 3R	0.86 ^b	0.85 ^b	0.71 ^b	0.50 ^b	0.00
2	Amaranth	0.69	0.70	0.52	0.16	0.02
3	Erythrosine	0.44	0.43	0.56	0.22	0.40
4	Ponceau SX	0.74	0.73	0.67	0.36	0.32
5	Oil Red XO	0.67	0.81	0.97	0.94	0.88
6	Ponceau 2R	0.76	0.88	0.74	0.44	0.37 ^b
7	New Coccine	0.61	0.78	0.48	0.24	0.12
8	Eosine	0.55	0.58	0.65	0.31	0.44
9	Phloxine	0.57	0.66	0.69	0.34	0.35
10	Rose Bengale	0.37	0.45	0.53	0.26	0.25
11	Acid Red	0.64	0.64	0.78	0.48	0.42
12	Rhodamine B	0.62	0.68	0.94	0.71	0.70
13	Coralline	0.80	0.91	0.81	0.69	0.56

^a The *R_F* values are the mean of five chromatograms.

^b Tailing.

the above-mentioned solution 200 ml were poured into a dish (14.5 × 19.5 × 2.5 cm) and a glass plate (12 × 14 × 0.1 cm) was dipped into it. Both sides of the glass were covered homogeneously. The glass was hung for 2 min over the dish to let the excess solution drain back. It was then air-dried for 3 h and heated at 100° for 30 min. These layers can be stored for a long period.

Chromatographic procedure

The standard techniques of ascending thin-layer chromatography⁶ was used.

Results and discussion

R_F values obtained with five solvent systems are given in Table I. Separation is achieved by these solvent systems and the detection limit of each compound is approximately 2 μg . It has been found that small and sharp spots are obtained with the addition of salts. The time required to ascend 10 cm from origin is 3–4 h. Separation is based on the formation of hydrogen bonds between the polyamide and the sample and adsorption or partition between the silica gel and the sample. The method described gives good resolution and reproducibility. The layer did not crack or peel and can be stored easily. Both sides of the glass are independent of each other and chromatography can be performed simultaneously on both sides.

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The paper chromatography of tar polycyclic hydrocarbons

A correction of previous results

In 1957 a paper chromatographic solvent system was described which, without any previous column or other pretreatment, separated that fraction of the neutral portion of naturally occurring high-temperature tar mixtures which contains the carcinogenic polycyclic hydrocarbons¹. Then a new kind of the semi-quantitative spot evaluation *in situ* was added, *viz.* the horizontal diameter measurement calibration².

But some hydrocarbons were identified incorrectly^{1–3} due to the lack both of some authentic compounds and of reliable data concerning their fluorescence. In addition, the R_F values are higher when employing 45 × 45 cm Whatman No. 4 paper impregnated with a 10% petroleum ether–paraffin oil solution; this paper is thinner than the 46 × 57 cm sheets and therefore the separation is better. A characteristic paper chromatographic pattern was obtained and a revised identification of the hydrocarbons was made (Fig. 1). Previously a benzene–coal tar solution of suitable concentration had been applied. The spots were outlined over a U.V. transilluminator. Thus the highest analytical sensitivity was achieved. The R_F values may vary from run to run, but the sequence of the hydrocarbons is constant. The solution was applied

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