Polyamide-Kieselguhr Thin-layer Chromatography of Antihistamines and Tranquilizers

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Separation of tranquilizers on thin-layer chromatography of silicagel [1,2] and aluminum oxide [3] have been reported. Polyamide-kieselguhr mixed layer had good success in separation of food dyes, [4] antipyretics, [5] and antioxidant [6]; therefore, this method was further applied to separate nine antihistamines and tranquilizers. For comparison, thin layer chromatography using only polyamide and only silica gel is also described. Separation on polyamide-kieselguhr mixed layer was found to be preferable.

EXPERIMENTAL

Preparation of polyamide-kieselguhr mixed layer

Ten grams of polyamide chip (Nylon 6, type 1022 B of UBE Industrial Ltd., Osaka, Japan) were dissolved in 100 ml of 90% formic acid. After warming (below 40°) and stirring, a homogeneous solution was obtained; after cooling it to room temperature, 50 grams of silica gel G (E Merck) were added 200 ml of the above-mentioned solution were poured into a dish and a glass plate (12 x 14 x o.1 cm) was dipped into it. Both sides of the glass were covered homogeneously. The glass were placed over the dish for several minutes to let the excess solution drain. It was then air-dried for 3 hours and heated at 100° C for 30 minutes.

Preparation of polyamide layer

Twenty grams of polyamide chip were dissolved in 90 ml of 90% formic acid. After completely dissolving, added 10 ml of distillated water and stirred, a homogeneous solution was obtained; then the procedure as described in the previous method.

Preparation of kieselguhr G layer

Slurries of kieselguhr G (45 grams in 100 ml of water) were sprayed at 2 kg/cm² pressure from a distance of 20 cm onto 8 sheets of glass plates (12 x 14 cm) in a horizontal position, then dried at 100° C for 30 minutes. The thickness of the layer was about 250μ.

Chromatographic Procedure

All sample solutions were applied to start line 2 cm from the bottom of the plate. The chamber has been saturated with the respective solvent for 30 minutes before ascending.

Visualization

The layer was sprayed with 0.07% rhodamine B alcoholic solution and spots can be recognized under UV light at 254 mμ.

Result and Discussion

Rf values of polyamide-kieselguhr mixed layer, polyamide layer and kieselguhr layer with three solvent systems are given in Table I.

It has been found that the result obtaining using the mixed layer (polyamide-kieselguhr) show better separation and sharper spots. The mixed layers did not crack or peel and could be stored easily. So the method is suitable for the identification of various compounds.

Table I

Samples	Solvent I			Solvent II			Solvent III		
	P*K	K	P	P*K	K	P	P*K	K	P
Tripelenamine	0.82	0.87	0.67	0.73	0.89	0.71	0.85	0.95	0.72
Chlorpheniramine	0.43	0.00	0.00	0.63	0.00	0.00	0.53	0.00	0.00
Chlorpromazine HCl	0.39	0.75	0.23	0.27	0.72	0.34	0.45	0.86	0.34
Pyrilamine	0.85	0.86	0.70	0.78	0.80	0.70	0.87	0.87	0.72
Diphenhydramine	0.72	0.87	0.59	0.72	0.91	0.63	0.74	0.88	0.64
Reserpine	0.18	0.74	0.11	0.12	0.92	0.22	0.28	0.93	0.17
Chlordiazepoxide	0.33	0.69	0.14	0.22	0.91	0.15	0.43	0.93	0.12
Pyrabital	0.92	0.00	0.79	0.86	0.96	0.81	0.80	0.00	0.82
Thioridiazine	0.28	0.79	0.16	0.18	0.83	0.03	0.37	0.95	0.08
Time required (min)	90	30	480	110	35	510	110	40	540

Solvent I = methanol : acetone : ammonia chloride = 10:8:42

Solvent II = DMF : methanol : aceton : NaCl = 1:10:5:40

Solvent III = DMF : aceton : isopropylalcohol : NH_4Cl : NaCl = 1:10:5:20:20

P*K = polyamide - kieselguhr layer

P = polyamide layer

K = kieselguhr layer

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〔中 文 摘 要〕

抗組織胺劑與鎮定劑之多醯胺和矽藻土混合薄層分析

抗組織胺劑和鎮定劑曾經用矽藥土和氧化鉛土來分離過，而矽藻土和多醯胺之混合薄層亦曾經很有效地被使用於食用色素，退熱劑和抗氧化劑等，現用矽藻土和多醯胺混合薄層來分離之，同時和矽藻土和多醯胺之各別單一薄層作比較。