臺灣藥學雜誌 JOURNAL OF THE TAIWAN PHARMACEUTICAL ASSOCIATION
第二十三卷 第一期 Vol. 23 No. 1 (1971)

Polyamide Mixed Layer Chromatography of Sulfa Drugs

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(Received Jan. 10, 1971)

Thin-layer chromatography of sulfa drugs has been studied by numerous investigators. The separation of these drugs on silica gel (1-4), kieselguhr (5), alumina (6) and polyamide (7) has been reported. Recently, polyamide mixed layers have been successfully used, e.g. polyamide-silica gel mixed layer for water-soluble vitamins (8) and polyamide-kieselguhr mixed layer for antipyretics (9).

Therefore, these durable polyamide mixed layers were further applied to separate ten sulfonamides.

For comparison, the thin-layer chromatography of only polyamide, only silica gel and only kieselguhr is also described. Separation on polyamide mixed layer was found to be preferable.

EXPERIMENTAL

Materials—The polyamide chip was Nylon 6[1], type 1022B of UBE Industrial Ltd. (Osaka, Japan). The solvents were reagent grade of WAKO Pure Chemical Industries, Ltd. (Osaka, Japan).

Preparation of Polyamide-Silica Gel Mixed Layer or Polyamide-Kieselguhr Mixed Layer — Ten g of polyamide were dissolved in 100ml of 90% formic acid. After standing for overnight, a homogeneous solution was obtained; then 50g of silica gel G (E. Merck) or kieselguhr G (E. Merck) were added and mixed well. Of the previous solution 300ml were poured into a dish (15×20×3cm) into which a glass plate (12×16×0.1cm) was dipped. Both sides of glass were covered homogeneously. The glass plate was hung for two min. over the dish to let the excess solution drained off. It was then dried in air for 3 hr. and heated at 100°C for 30 min.

Preparation of Polyamide Layer — Dissolve 20g of polyamide in 90ml of 90% formic acid, then 10ml of distilled water were added. After stirring, a homogeneous solution was obtained; then the procedure as described in the previous method, but without adding silica gel G or kieselguhr G, was followed.

Preparation of Silica Gel or Kieselguhr Layer — Dilute slurries of silica gel G or kieselguhr G (45g to 120ml of water) were sprayed at 1.5kg/cm^3 pressure from a distance of 20cm onto 8 sheets of glass plate (12×16cm) in a horizontal position, then dried at 100°C for 30 min. The thickness of layer are about 250μ.

Chromatographic Procedure — A ammonia solution (28%) of 0.4% sulfa drugs was applied to the start line 1.5cm from the bottom of the layer. The plates were developed in the dark by ascending techniques for 10cm. The chambers had been equilibrated with the respective solvent system for 30 min. before use.

Visualization — The layers were sprayed with Ehrlich reagent (1 g of p-dimethyl amino-

1 U. S. Supplier: American Enka Corp.

benzaldehyde in 1ml of concentrated HCl and 98 ml, 95% ethanol) and yellow color would be produced.

RESULTS AND DISCUSSION

Rf values of polyamide–silica gel layers, silica gel layers, kieselguhr layers and polyamide layers with two solvent systems are given in Table I. Also those of polyamide–kieselguhr layers, silica gel layers, kieselguhr layers and polyamide layers are given in Table II. The Rf values are the mean of five chromatograms.

It has been found that the better results is obtained on the polyamide–silica gel layer with the solvent system I and on polyamide–kieselguhr layer with the solvent system III.

The spots on silica gel layers and kieselguhr layers are rather diffused and larger.

In the mixed layer, polyamide also serves as a strong binder and makes the layer durable and easy to handle. Also the layer did not crack or peel and could be stored easily. Both sides of the glass are independent of each other and chromatography can be performed simultaneously on both sides. After visualization, the P–K layer[2] can be easily removed from the glass plate after a brief immersion in water followed by careful peeling of the layer using a spatula. The separated layer can be filed in a notebook for record purposes.

CHROMATOGRAPHY DATA OF SULFA DRUGS

Table I*

No. of samples	Solvent I[a]				Solvent II[b]			
	P–S[c]	S[d]	K[e]	P[f]	P–S	S	P	K
1. Sulfadimethoxine	0.71	0.64	1.00	0.58	0.69	0.88	0.84	0.97
2. Sulfamethazine	0.61	0.57	1.00	0.57	0.54	0.65	0.76	0.96
3. Sulfamerazine	0.54	0.55	0.99	0.54	0.48	0.55	0.70	0.96
4. Sulfamethoxypyridazine	0.50	0.52	0.99	0.37	0.49	0.58	0.65	0.96
5. Sulfadiazine	0.44	0.48	0.95	0.41	0.41	0.29	0.60	0.92
6. Sulfisoxazol	0.34	0.59	1.00	0.11	0.28	0.46	0.29	0.95
7. Sulfanilamide	0.27	0.53	0.98	0.10	0.11	0.16	0.11	0.89
8. Sulfisomidine	0.23	0.22	0.98	0.38	0.21	0.18	0.79	0.93
9. Sulfathiazol	0.06	0.26	0.75	0.03	0.18	0.22	0.27	0.94
10. Sulfaguanidine	0.01	0.08	0.14	0.00	0.05	0.15	0.09	0.87
Time required (min.)	90	20	20	90	85	45	90	20

2 Polyamide–Kieselguhr mixed layer

Table II**

No. of samples	Solvent III[g]				Solvent IV[e]			
	P-K[i]	S	K	P	P-K	S	K	P
1. Sulfadimethoxine	0.81	0.55	0.97	0.65	0.77	0.42	0.97	0.58
2. Sulfamethazine	0.74	0.41	0.96	0.54	0.67	0.25	0.97	0.48
3. Sulfisomidine	0.69	0.13	0.86	0.55	0.59	0.18	0.83	0.50
4. Sulfamerazine	0.59	0.35	0.95	0.48	0.59	0.24	0.94	0.40
5. Sulfamethoxypyridazine	0.54	0.36	0.96	0.30	0.62	0.26	0.97	0.29
6. Sulfadiazine	0.50	0.29	0.87	0.31	0.47	0.20	0.80	0.31
7. Sulfisoxazol	0.40	0.38	0.96	0.17	0.33	0.28	0.95	0.15
8. Sulfathiazol	0.17	0.18	0.90	0.06	0.21	0.24	0.92	0.07
9. Sulfanilamide	0.13	0.20	0.88	0.05	0.13	0.26	0.83	0.04
10. Sulfaguanidine	0.07	0.13	0.90	0.04	0.09	0.22	0.60	0.04
Tine required (min.)	80	35	20	180	133	40	20	125

* [a] Solvent I: Dioxane: Ethyl acetate: Benzene: Petroleum ether (30: 20: 10: 15).

[b] Solvent II: Glacial acetic acid: Trichloroethylene: Petroleum ether: Chloroform (19: 10: 10: 25).

[c] P-S, Rf value on polyamide-silica gel mixed layer.

[d] S, on silica gel layer.

[e] K, on kieselguhr layer.

[f] P, on polyamide layer.

** [g] Solvent III: Glacial acetic acid: Dioxane: Trichloroethylene: n-Hexane (15: 5: 25: 15).

[h] Solvent IV: Glacial acetic acid: Trichloroethylene: Petroleum ether (16: 22: 10).

[i] P-K: Rf value on polyamide-kieselguhr layer.

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ACKNOWLEDGMENTS AND ADDRESSES

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中 文 摘 要

磺胺劑之多醯胺混合薄層分析

姜宏哲 林 于 劉詔洋

臺北醫學院藥學系

10種磺胺類藥品應用 Polyamide–Silica gel 混合薄層，Polyamide–Kieselguhr 混合薄層，Silica gel 薄層，Kieselguhr 薄層，Polyamide 薄層等五種薄層分析法分別進行鑑別並檢討所得之結果。