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Note

Identification and separation of closely related sulfa drugs by thin-layer chromatography on cadmium acetate-impregnated silica gel plates

S. P. SRIVASTAVA, V. K. DUA, R. N. MEHROTRA and R. C. SAXENA
Chemical Laboratories, University of Roorkee, Roorkee (India)
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Recently the impregnation of thin-layer plates with different substances has been tried in order to improve the separation of various classes of substances. Yasuda[1–4] reported the use of different metal salts for the separation of aromatic amines, while Srivastava and Dua, in this laboratory, have developed suitable impregnants for the separation of phenols[5], aromatic amines[6], aliphatic amines[7], diols[8] and amino acids[9] by thin-layer chromatography (TLC). The loading of papers with strong acid exchange resin has been employed by Pietrzyk and Chan-Santos[10] for the separation of some sulfa drugs. However, no work seems to have been done, on the TLC separation of sulfa drugs on impregnated plates. The present paper reports our studies on the use of impregnated silica gel plates for the separation and identification of ten sulfa drugs.

EXPERIMENTAL

The plates (0.5 mm thick) were coated with a mixture of silica gel (50 g) and 1% metal salt solution (100 ml), and then activated at 60 ± 1° for 24 h. Solutions of sulfa drugs (0.05% w/v) in ethanol were used for spotting. After development the chromatoplates were sprayed with DAB (50 mg of *p*-dimethylaminobenzaldehyde dissolved in 1 ml concentrated sulphuric acid and made up to 100 ml with 95% ethanol)[11]. Yellow spots appeared on a white background.

The various impregnants tried were cadmium sulphate, cadmium acetate, zinc sulphate, zinc acetate, manganese sulphate and manganese acetate. In all cases, a 1% solution of the impregnant gave the best separation. On cadmium acetate all ten sulfa drugs were separated when present in the mixture. The most suiteble solvent system was found to be toluene–benzene–chloroform–ethyl methyl ketone–methanol (100:20:25:25:25). Some representative separations of sulfa drugs on metal salt-impregnated plates are given in Table I.

RESULTS

Comparison of the data in Table I shows that by using a metallic salt as impregnant the separation of different sulfa drugs is quite satisfactory and that

TABLE I

REPRESENTATIVE SEPARATIONS OF SULFA DRUGS ON TLC PLATES IMPREGNATED WITH METAL SALTS

The $R_F \cdot 100$ values reported are means from two or more identical runs. Rate of development: 12 cm in 30 min.

Sulfa drug	*Impregnant*						*Detection limit (ng)*
	Cadmium sulphate	*Cadmium acetate*	*Zinc sulphate*	*Zinc acetate*	*Manganese sulphate*	*Manganese acetate*	
Sulfaphenazole	—	68	70	60	68	72	23
Sulfamethizole	22	4	24	3	—	—	13
Sulfasomidine	27	10	43	36	45	42	37
Sulfadimidine	55	38	56	52	—	65	22
Sulfaguanidine	33	23	30	18	23	22	35
Sulfadiazine	46	18	—	—	54	51	10
Sulfathiazole	42	14	—	—	—	37	16
Sulphanilamide	—	45	—	—	—	—	35
Sulfamerazine	50	29	—	42	60	56	37
Sulfapyridine	—	34	52	—	—	—	44

there is no tailing. On cadmium acetate-impregnated plates the size of the spots is minimal and all the sulfa drugs are separated. The R_F values of the sulfa drugs do not change when present in a mixture. Manganese acetate as an impregnant results in equally good separations but only seven sulfa drugs can be separated on this adsorbent system, while only five or six sulfa drugs can be separated using cadmium sulphate, zind sulphate, zinc acetate or manganese sulphate as impregnant.

The behaviour of the sulfa drugs on impregnated plates depends on the following two factors:

(i) The formation of a metal–sulfa drug complex involving the metal ion and the amino group of the sulfa drug, as was suggested by Yasuda[1] in the case of aromatic amines. This is supported by the observation of Narang and Gupta[12] who reported the formation of a complex between copper(II) and a sulfa drug.

(ii) The formation of hydrogen bonding due to the hydrogen atoms of the NH_2 group and oxygen atoms of the acetate or sulphate anion, as reported by Srivastava and Dua[9] in the case of TLC separation of amino acids on calcium oxalate-impregnated plates.

Thus we see that the movement of sulfa drugs is governed by the nature of both the metal ion and the anion. Besides the above two factors, steric effects and solvation factors should also influence the R_F values. Further work in this direction is in progress.

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