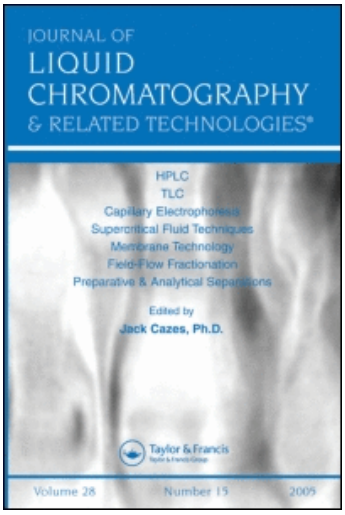

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THIN LAYER CHROMATOGRAPHIC STUDIES OF SOME SULPHA DRUGS SUBSTITUTED PYRAZOLES USING SILICA GEL-G PLATES IMPREGNATED WITH VARIOUS ADSORBENTS

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ABSTRACT

Present communication describes the resolution of some Sulpha drugs substituted synthesized pyrazole derivatives on Silica gel-G plates impregnated with various adsorbents viz; sodium lauryl sulphate, Tetra ethyl ammonium perchlorate, Tetrabutyl ammonium bromide, Tetrabutyl ammonium hydroxide and Triton X-100.

INTRODUCTION

Substituted pyrazole derivatives have been found to possess pharmacodynamic significance, some of which have been synthesized as potential antidiabetic and potential antineoplastic agents.[1-3] Some pyrazole derivatives have

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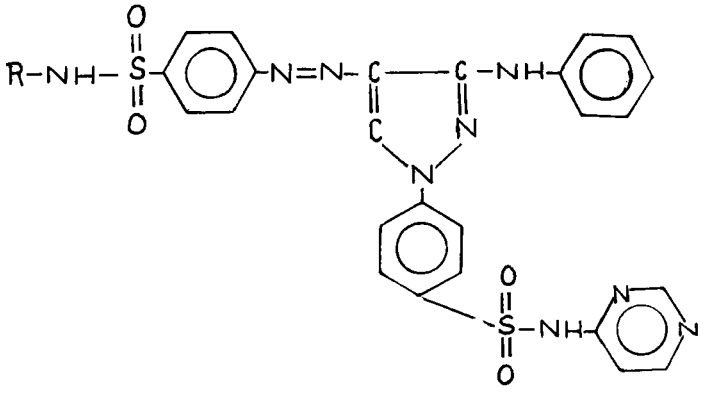
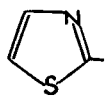
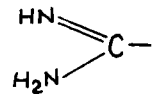
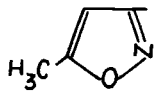
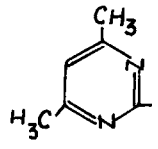
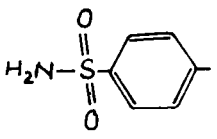
been reported as bacteriostatics, bacteriocidals[4] and fungicides[5].

Keeping in view the immense importance of Sulpha drugs, azo compounds, hydrazono compounds and pyrazoles it was thought worthwhile to synthesize a series of Sulpha drugs substituted pyrazoles which have chemotherapeutic significance, followed by their resolution on thin layer chromatoplates using Silica gel-G plates impregnated with various surfactants.

The present communication reports the thin layer chromatographic resolution of some closely related Sulpha drugs substituted pyrazoles, on various surfactants impregnated Silica gel-G plates.



R =  ;  ; 

 ;  ; 

EXPERIMENTAL

The glass plates (20x10 cm^2) were impregnated with following different adsorbents, thickness 0.75 mm, using Stahl's applicator.

(A) Silica gel-G (E.Merck).

(B) Silica gel-G (E.Merck) + 1% Sodium lauryl sulphate

(C) Silica gel-G (E.Merck) + 1% Tetraethyl ammonium perchlorate.

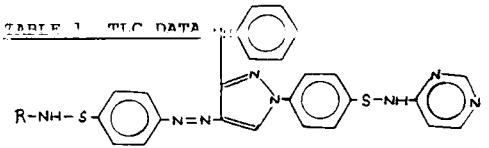
(D) Silica gel-G (E.Merck) + 1% Tetrabutyl ammonium bromide.

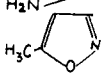
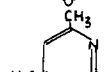
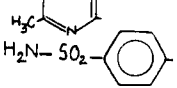
(E) Silica gel-G (E.Merck) + 1% Tetrabutyl ammonium hydroxide.

(F) Silica gel-G (E.Merck) + 1% Triton X-100.

All the Sulpha drugs substituted pyrazoles were synthesized in the laboratory and repeatedly recrystallised with ethanol before subjecting them to chromatographic separation. These Sulpha drugs substituted pyrazoles (Table 1,2) were dissolved in acetone and spotted on the activated chromatoplate by use of a glass capillary. The spots were allowed to air dry and then put to an ascending irrigation process. The spots were observed as yellow or orange spots. In cases when they were not visible, the locations were determined by keeping the chromatoplates in Iodine chamber. Yellow or orange spots appeared on violet background after sometime. The R_F values obtained were found reproducible in different identical runs.

TABLE 1. TLC DATA



S.No.	R	M.P. (°C)	R_f (X 100)*								
			Silica Gel-G			Silica Gel-G + 1% Sodium Lauryl Sulphate			Silica Gel-G + 1% Tetraethyl Ammonium Perchlorate		
			1	2	3	1	2	3	1	2	3
1.		138	97.43	44.32	94.18	94.44	55.00	87.75	96.05	35.52	96.05
2.		62	93.38	58.60	91.86	78.88	+	79.59	+	45.89	76.31
3.		58	+	+	92.00	80.00	+	84.69	86.66	+	86.84
4.		85	94.05	69.69	96.59	95.34	60.82	92.00	94.66	63.15	95.00
5.		100	96.25	65.65	94.31	91.86	79.38	89.00	96.00	70.51	92.50
6.		110	98.75	71.35	89.77	93.02	77.31	91.00	94.66	66.66	93.75

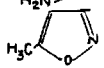
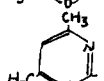
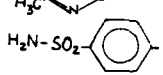
* Solvent Composition

1. Methanol/ Chloroform (80:20)
2. Ethylacetate / Xylene (20:80)
3. Methanol/ Toluene (70:30)

Average time for two identical runs - 45 cm in 15 minutes.

\+ Spots could not be located.

TABLE 2

S.No.	R	R_f (X 100)*								
		Silica Gel-G + 1% Tetrabutyl Ammonium Bromide			Silica Gel-G + 1% Tetrabutyl Ammonium Hydroxide			Silica Gel-G + 1% Triton X-100		
		1	2	3	1	2	3	1	2	3
1.		94.62	27.27	90.62	93.00	15.30	90.62	92.00	32.91	91.20
2.		92.32	35.63	88.54	+	20.26	+	+	61.28	88.68
3.		+	+	95.83	55.00	+	91.68	96.00	+	77.52
4.		90.90	58.13	87.75	96.00	39.58	93.87	91.78	63.15	85.39
5.		97.72	55.81	+	95.00	47.91	89.79	94.52	64.47	+
6.		93.18	60.46	86.48	97.00	50.96	97.95	89.04	65.82	96.62

* Solvent Composition

1. Methanol/ Chloroform (80:20)
2. Ethylacetate/ Xylene (20:80)
3. Methanol/ Toluene (70:30)

Average time for two identical runs - 45 cm in 15 minutes.

RESULTS AND DISCUSSION

A mixture of Methanol: Chloroform (80% : 20%), Ethylacetate : Xylene (20% : 80%) and Methanol : Toluene (70% : 30%) have been found to give good separation of all the pyrazoles studied as shown by the R_f values in Table 1 and 2.

The mobility of compounds in Silica gel-G alone was better as compared to when Silica gel-G was impregnated with different adsorbents. The reduced mobility by the addition of different adsorbents gives support in order to get distinct separation. With most of the adsorbents separation was fairly good in Methanol : Toluene and was excellent when Ethylacetate : Xylene was used as the Solvent System. However, with 1% Triton X-100 poor separation was found to occur in all the Solvent Systems, whereas the results are fairly good in Methanol : Toluene solvent system.

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