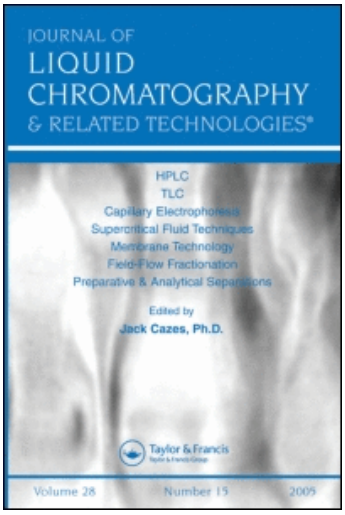

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Identification of some closely related Pyrazolin-5-Ones by TLC

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ABSTRACT

A rapid thin layer chromatographic procedure that utilizes neutral solvent system for the separation of 22 closely related pyrazolin-5-Ones on silica gel adsorbent is reported.

INTRODUCTION

Pyrazolin-5-Ones and its derivatives are biologically active compounds and have been used as analgesies[1], antimicrobial agents[2], fungicides[3], herbicides[4], antidiabetics[5], antidiuretics[6], antioxidants[7] and in influenza[8]. They also find many applications in photography as colour couplers[9], sensitizers[10], super sensitizers[11] and developers[12]. Another important commercial use of pyrazolin-5-Ones is as a dye for rayon[13], silk[14], leather[15], rubber[16], polyester[17] and plastics[18].

Recently, Garg and Prakash[19,20] have synthesised some 1-phenyl-3-methyl-4-arylhydrazono-2-pyrazolin-5-Ones (A) as potential antidiabetic agents. As these compounds contain the hydrazono grouping -NH-N=C- , they have found wide applications in synthetic chemistry for the preparation of compounds of most diverse structure and also for the detection of a large number of metal cations.

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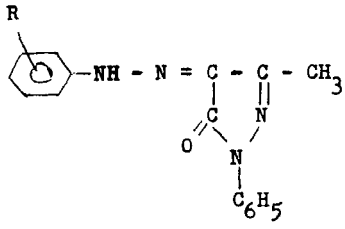

(A)

where, R represents different substituents.

Keeping in view also the medicinal properties of pyrazolin-5-Ones it was now considered worthwhile to study the separation of some closely related 1-phenyl-3-methyl-4-arylhydrazono-2-pyrazolin-5-Ones by TLC.

EXPERIMENTAL

Commercially available silica gel G, TLC plates of size 21.5 x 21.5 cm^2, layer thickness 0.4 mm were used after activation for 24 hrs. All the pyrazolin-5-Ones were synthesised[19] in the laboratory and repeatedly recrystallised with DMF:water mixture before subjecting them to chrometographic separation. All the compounds (I-XXII) in acetone (1 % V/V) were applied by means of a fine class capillary and they put in the developer for the development process. The resolved compounds were visualized by exposing to NO_2 for 40 sec. It is pertinent to note that no tailing was observed in any case except in 4-OC_2H_5 derivative where slight tailing was observed. The R_f values obtained were found reproducible in different identical runs.

RESULTS AND DISCUSSION

The TLC data obtained are given in Table 1. The development time for the solvent systems employed was about 45 min. Both the solvent systems used gave satisfactory separation of most of the compounds. The colour of all the spots (I-XXII) was light yellow, darkened on exposure to NO_2. The results show an interesting

Table - 1

No.	R	M.P. °C	R_f x 100		Detection limit (µg)
			A	B	
1	H	132	71	36	2.5
2	2-CH_3	175	51	26	3.0
3	3-CH_3	180	76	32	3.0
4	4-CH_3	137	85	43	3.5
5	2-Cl	185	35	57	2.5
6	3-Cl	135	55	63	2.5
7	4-Cl	140	62	72	3.0
8	2-Br	180	26	29	3.5
9	3-Br	150	30	39	3.5
10	4-Br	145	49	45	2.0
11	2-OCH_3	163	40	40	3.5
12	4-OCH_3	135	62	51	3.5
13	2-OC_2H_5	163	58	24	3.5
14	4-OC_2H_5	150	67	35	2.0
15	2-NO_2	212	38	18	3.5
16	3-NO_2	175	46	22	3.0
17	4-NO_2	170	80	48	3.5
18	2,3-$(CH_3)_2$	210	21	26	2.5
19	2,5-$(CH_3)_2$	188	17	18	2.5
20	3,5-$(CH_3)_2$	193	26	21	3.0
21	2,6-$(Cl)_2$	179	13	11	3.5
22	2,4,6-$(Cl)_3$	227	8	9	2.5

Solvent Composition for compounds I-XXII

(A) Xylene:Chloroform (60 % : 40 %).

(B) Xylene:Chloroform:Acetone (65 %:25 %:10 %).

trend in the R_f values. It is observed that in the case of ortho substituted derivative the rate of flow (R_f) of the spot is low whereas meta and para substituents increase the value of R_f in comparison with that of the ortho substituted derivative.

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