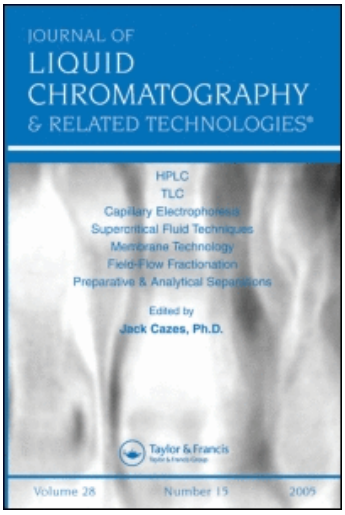

Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Characterization of Derivatives of Some Closely Related 1-Picolinoyl-3-methyl-4-(substituted)-pyrazoline-5-ones by Thin-Layer Chromatography

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To cite this Article Gupta, D. R. and Arora, R. K.(1983) 'Characterization of Derivatives of Some Closely Related 1-Picolinoyl-3-methyl-4-(substituted)-pyrazoline-5-ones by Thin-Layer Chromatography', Journal of Liquid Chromatography & Related Technologies, 6: 14, 2695 — 2697
To link to this Article: DOI: 10.1080/01483918308064939
URL: http://dx.doi.org/10.1080/01483918308064939

JOURNAL OF LIQUID CHROMATOGRAPHY, 6(14), 2695-2697 (1983)

CHARACTERIZATION OF DERIVATIVES OF SOME CLOSELY RELATED 1-PICOLINOYL-3-METHYL-4-(SUBSTITUTED) PYRAZOLINE-5-ONES BY THIN LAYER CHROMATOGRAPHY

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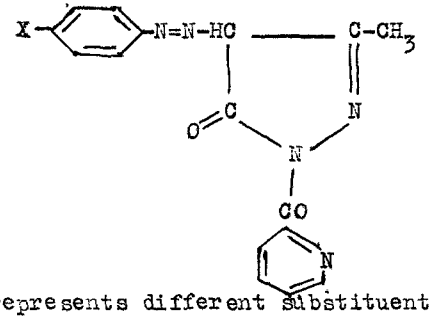
INTRODUCTION

The antitubercular activity associated with pyrazoline-5-ones (1) , picolinic acid hydrazide (2) and the antibacterial activity of sulphonamides (3) prompted us to undertake the synthesis of 1-picolinoyl-3-methyl-4-(substituted)-pyrazoline-5-ones (see formula) as possible new biological active agents. In view of their medicinal importance, it is thought worthwhile to carry out a TLC separation of these compounds.



where X-represents different substituents (Table 1)

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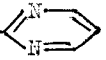
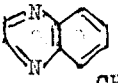
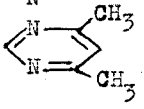
Table - 1

Sl. No.	X	R_f x100	
		A	B
1	$-So_2NH_2$	37	36
2	$-So_2NH.C(=NH)NH_2$	49	-
3	$-So_2NH$ 	59	-
4	$-So_2NH$ 	74	83
5	$-So_2NH.Co.CH_3$	76	77
6	$-So_2NH$ 	68	60
7	$-So_2NH$ 	71	73
8	$-So_2NH$ 	40	71
9	2-Cl	12	14
10	3-Cl	24	23
11	4-Cl	27	32
12	$2-OCH_3$	20	17
13	$3-OCH_3$	61	60
14	$4-OCH_3$	13	25
15	4-Br	43	77
	Rate of development for 15 cms in min	45	45

A = Chlorofarm:methanol (90:10)

B = Benzene :ethyl acetate (60:40)

EXPERIMENTAL

All the derivatives of pyrazoline-5-one were synthesized in this laboratory by usual methods (4). TLC plates (0.5 m.m. thick) were prepared from silica gel G (Sisco) by means of Stahl's applicator. The plates were activated at 60°C for 24 hrs. All the compounds were recrystallized prior to their use. The pure organic compounds were dissolved in hot glacial acetic acid and were applied to the chromatoplates with the help of a glass capillary and the spots were allowed to air dry. The chromatograms were developed at constant temperature (30 ± 2^{o}) with different solvent systems. The chamber used for the development was saturated with vapours of the solvent mixture before carrying out the separation. A mixture of chloroform:methanol (90:10) and benzene:ethyl acetate (60:40) was found to give the best results. As these compounds gave coloured spots, no visualiser was therefore employed. The R_f values of the compounds were determined and recorded in table 1. The results were found fairly reproducible within the limits of experimental error.

ACKNOWLEDGEMENT

One of us (R.K.A.) is grateful to U.G.C., India for financial assistance.

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