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IDENTIFICATION OF SOME CLOSELY RELATED N-CARBAMOYL-3,5-DIMETHYL-4-ARYLAZOPYRAZOLES BY TLC

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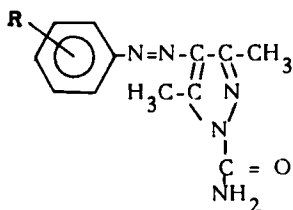
ABSTRACT

Due to the key role of arylazopyrazoles in biochemical sciences, interest has sprung up to achieve their resolution and identification by thin layer chromatographic technique. Present communication describes the resolution of some N-carbamoyl-3, 5-dimethyl-4-arylazopyrazoles on silica gel-G plates impregnated with various surfactants viz., 1% acetoacetanilide, 1% triton X-100 and 1% tetrabutylammounium bromide. A correlation of R_f value with Hammett substituent constant (σ) is also interpreted.

INTRODUCTION

Derivatives of pyrazole heterocyclic compound have attracted wide attention and are noteworthy for their participation in important biological processes.[1-4]. Keeping in view the chemotherapeutic significance associated with pyrazoles, linked with azo moiety at one of its end, it was considered worthwhile to achieve resolution of some substituted arylazopyrazoles by thin layer chromatography in benzene-ethylacetate solvent system. As these compounds contains the azo grouping -N=N-, they have found wide application in synthetic chemistry for the preparation of the compounds of most diverse structure and also in the detection of a large number of metal cations. [5]

The present paper reports the thin layer chromatographic resolution of some closely related N-carbamoyl-3, 5-dimethyl-4-arylazopyrazoles, on various surfactants impregnated silica gel-G plates.



Where R = -H; 4-CH₃; 3-No₂; 4-No₂; 2-No₂; 2-OH; 4-OH; 4-Br; 4-Cl.

EXPERIMENTAL

An ascending irrigation technique was employed commercially available Silica gel-G and chromatoplates of size 20x10 cm². A stahl type applicator was used to maintain layer thickness of 0.5 mm and plates were used after activation for 2 hours at 110° C. In quest of getting good resolution, slurry of Silica gel-G mixed with various adsorbents viz., 1% Triton X-100; 1% acetoacetanilide and 1% tetra butyl ammonium bromide was also used. All the compounds under investigation were synthesized in the laboratory and repeatedly recrystallized with ethanol before subjecting them to chromatographic separation.

All the compounds dissolved in acetone were applied by means of a micropipette and then put in the developer for 45 minutes.

RESULTS AND DISCUSSION

The TLC data for the separation of N-carbamoyl - 3, 5 - dimethyl - 4 - arylazopyrazoles are given in table-1.

TABLE - 1

R_f (X 100) Values of N-Carbamoyl - 3, 5-Dimethyl-4-Arylazopyrazoles.

| Sr. No. | R | M.P. °C | Silica gel-G | Silica gel-G + 1% Aceto-acetanilide | Silica gel-G + 1% Triton X-100 | Silica gel-G + 1% Tetrabutyl ammonium bromide |
|---------|-------------------|------------|--------------|-------------------------------------|--------------------------------|---|
| 1. | -H | 105 | 70 | 73 | 72 | 70 |
| 2. | 3-No ₂ | 130 | 75 | 79 | 77 | 74 |
| 3. | 4-Br | 95 | 77 | 82 | 81 | 79 |
| 4. | 4-CH ₃ | 191 | 22 | 18 | 14 | 15 |
| 5. | 4-Cl | 130 | 88 | 96 | 96 | 93 |
| 6. | 4-OH | 223 | 21 | 43 | 41 | 29 |
| 7. | 2-OH | 110 | 80 | 83 | 73 | 90 |
| 8. | 2-No ₂ | 97 | 74 | 77 | 76 | 89 |
| 9. | 4-No ₂ | 187 | 24 | 14 | 13 | 15 |

Solvent Composition - Benzene & Ethylacetate (90:10)

Average time for two identical runs : 15 cm. in 45 min.

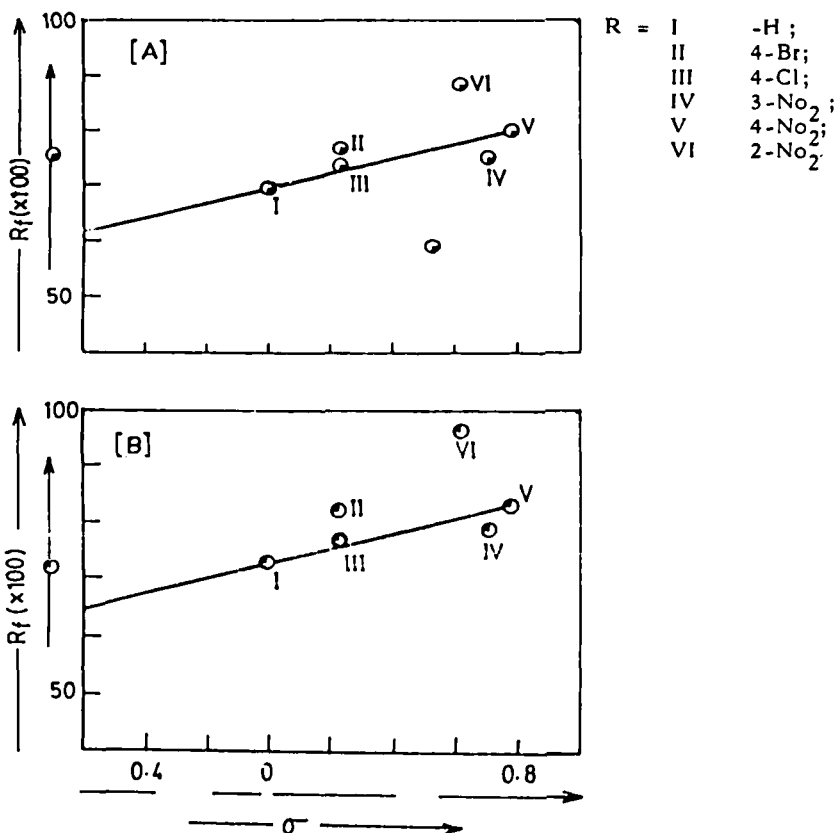


FIGURE 1. Plots for R_f (X-100) vs. σ for N-carbamoyl-3,5-dimethyl-4-arylazopyrazole using Silica Gel-G plates impregnated with surfactants, viz., (a) Silica Gel-G; (b) 1% acetoacetanilide.

Satisfactory resolution was achieved for all the compounds. They were visible on the chromatogram as light yellow to orange coloured spots. On impregnating the plates with 1% triton X-100, better separation could not be achieved. However, interestingly more satisfactory resolution was obtained in case of 1% tetrabutyl ammonium bromide as an adsorbent. Results are also fairly good with 1% acetoacetanilide.

Effect of Substituents and their Influence on R_f Values: A
Correlation with Hammett Substituent Constant : [6-7]

In order to view the effect of various substituents on R_f values, a correlation has been drawn between R_f vs. σ (Hammett substituent constant). It has been predicted that an

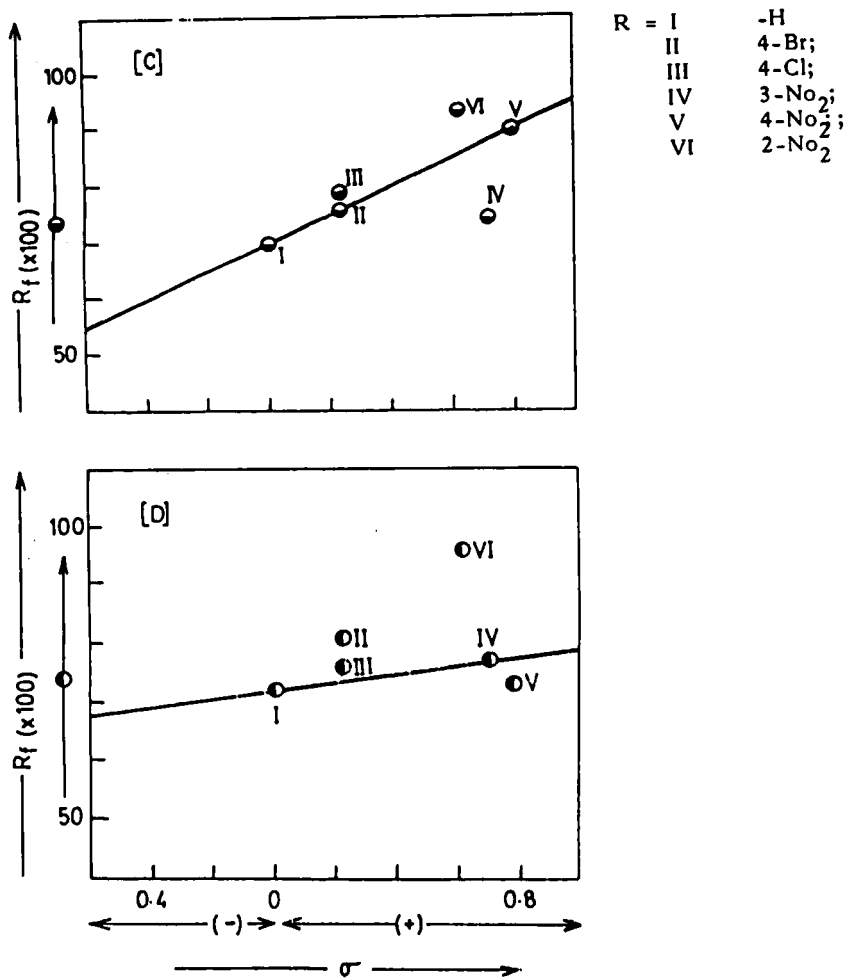


FIGURE 2. Plots of R_f (X-100) vs. σ for N-carbamoyl-3,5-dimethyl-4-arylazopyrazole using Silica Gel-G plates impregnated with surfactants, viz. (c) 1% tetrabutylammonium bromide; (d) 1% Triton X-100. Solvent Composition: Benzene and Ethyl Acetate (90:10).

increase in R_f value was observed in case of electron-withdrawing groups, whereas there is a decrease in electron-releasing substituents.

Furthermore, an ortho-shift was also observed in case of ortho substituted derivatives. A linear correlation as shown in fig. 1,2 indicate that result for m- and p- derivatives are pretty satisfactory, whereas ortho derivatives lie far off to either side of straight line.

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