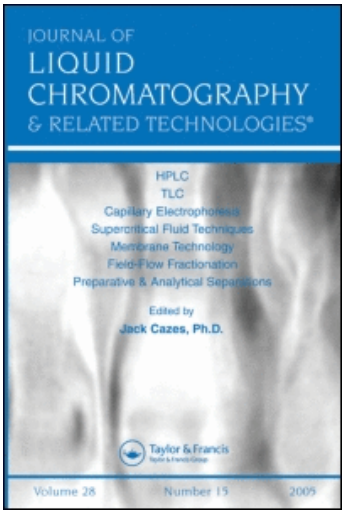

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Rapid Tlc Separation of Some Closely Related Potential Antineoplastic Arylazothiazoles

Rajeev Jain; D. D. Agarwal[a]; R. N. Goyal[a]
[a] Department of Chemistry, University of Roorkee, Roorkee, India

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RAPID TLC SEPARATION OF SOME CLOSELY RELATED POTENTIAL ANTINEOPLASTIC ARYLAZOTHIAZOLES

*Rajeev Jain, D.D. Agarwal, and R.N. Goyal

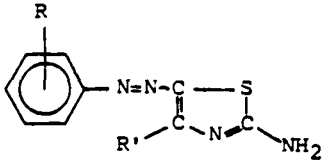
Department of Chemistry
University of Roorkee
Roorkee, India

ABSTRACT

A rapid thin layer chromatographic procedure that utilizes neutral solvent system for the separation of 24 closely related arylazothiazoles on silica gel adsorbent is reported.

INTRODUCTION

The importance of organic sulphur compounds is mainly due to their remarkable pharmacological activity. A large number of sulphur compounds have been used as analgesics[1], local anasthetics[2], fungicides[3], insecticides[4], antituberculoucis[5], antipsychotics[6], antidiabetic[7], and antineoplastics[8]. Keeping in view the pharmaceutical importance of 4-phenyl-5-phenylazo-2-aminothiazoles and 4-mentyl-5-phenylazo-2-aminothiazoles as potential antineoplastic compounds it was considered worthwhile to study the separation of these compounds by T.L.C. as this information may provide their better identifications during the evaluation of drugs. The general structure of the thiazoles is:



where, R and R' represent different substitutes.

*Address to which correspondence should be addressed: 224, Khandaq Street, Meerut-250002 (India).

TABLE 1

No.	R	R'	R_f x 100 A	R_f x 100 B	Detection Limit µg
I	2-NO_2	CH_3	18	30	2.5
II	2-CH_3	CH_3	67	50	2.0
III	2-Br	CH_3	50	61	2.0
IV	2-Cl	CH_3	62	56	2.5
V	2,4-diCl	CH_3	20	15	1.5
VI	2,5-diBr	CH_3	26	8	2.5
VII	2,6-diCH_3	CH_3	29	12	2.5
VIII	2-Cl	C_6H_5	43	57	2.0
IX	2-CH_3	C_6H_5	24	26	1.0
X	2-Br	C_6H_5	56	46	2.5
XI	4-CH_3	C_6H_5	30	40	2.5
XII	4-Cl	C_6H_5	49	36	2.0
XIII	4-OCH_3	C_6H_5	40	32	1.5
XIV	4-Br	C_6H_5	35	49	1.5
XV	4-OC_2H_5	C_6H_5	46	38	1.0
XVI	4-NO_2	C_6H_5	13	11	1.0
XVII	H	2-ClC_6H_4	84	73	1.5
XVIII	H	2-$CH_3C_6H_4$	72	78	1.5
XIX	H	4-ClC_6H_4	30	69	2.0
XX	H	4-$CH_3C_6H_4$	89	56	1.0
XXI	H	4-BrC_6H_4	30	51	2.5
XXII	H	4-OHC_6H_4	91	88	2.5
XXIII	H	4-$OCH_3C_6H_4$	76	82	2.0
XXIV	H	4-$OC_2H_5C_6H_4$	80	60	1.5
XXV	H	4-$NO_2C_6H_4$	21	27	1.0

Solvent Composition:

For compounds I-XV (A) = Benzene:Chloroform (50%:50%)
(B) = Benzene:Chloroform:Dioxane (50%:40%:10%)

For compounds XVI-XXV (A) = Benzene:Methanol (80%:20%)
(B) = Benzene:Methanol:Dioxane (75%:20%:5%)

EXPERIMENTAL

Commercially available silica gel G, T.L.C. plates of size 21.5 x 21.5 cm^2, layer thickness 0.20 mm were used after activation. TLC plates were developed in glass troughs saturated with the vapours of the solvent system. All the arylazothiazoles were synthesised in the laboratory[8] and repeatedly recrystallised with ethanol before subjecting them to chromatographic separation. A 0.2% methanolic solution was applied to the plates with the help of fine glass capillary. The composition of the developers used for compounds I-XV was (A) Benzene:Chloroform, (50%:50%), (B) Benzene:Chloroform:Dioxane (50%:40%:10%), and for compounds XVI-XXIV was (A) Benzene:Methanol (80%:20%), (B) Benzene: Methanol:Dioxane (75%:20%:5%). After development, the colour of the spots in the first series of compounds (I-XV) was light yellow which was being darkened by exposure to NO_2 for about one minute. In compounds (XVI-XXV) colour of the spots was dark yellow. It is pertinent to note that no tailing was observed in any of the compounds of the two series studied. The R_f-values obtained were found reproducible in the different identical runs and are compiled in Table 1.

RESULTS AND DISCUSSION

The T.L.C. data on the separation of arylazothiazoles (I-XXV) are given in Table 1. The chromatographic development time for solvent systems (A-B) employed was about 30 minutes. Both the solvent systems used gave satisfactory separation of most of the compounds. The results for their series of compounds show an interesting trend in the R_f-values. It is observed that in the case of electron donating substituents, the rate of flow (R_f) of the spots in most of the cases, is increased whereas electron withdrawing groups decrease the value of R_f.

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