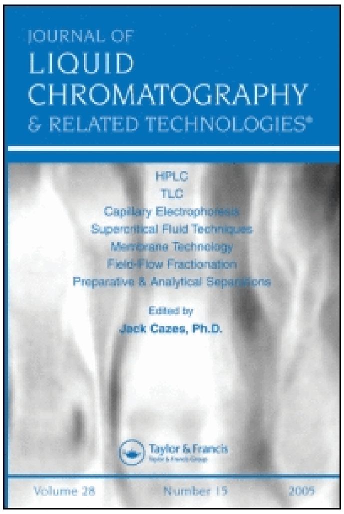

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Rapid TLC Separation of Some Closely Related Benzothiazoles

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RAPID TLC SEPARATION OF SOME CLOSELY RELATED BENZOTHIAZOLES

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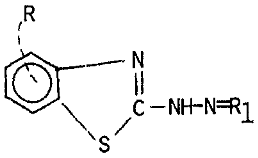
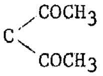
The thiazole heterocylces have found several uses in biology, medicine and industry. In 1935, William and Coworker[1] found the existence of thiazole moeity in vitamin B_1 (Thiamine) and several of its derivatives are well know for their chemotherapeutic properties for combating bacterial infections[2]. Amongst its other medicinal applications are the use of 6-aminobenzothiazoles as antitubercular reagents and local anaesthetics. The other uses to which this class of compounds have been put are as a rubber vulcanization accelerator[3], photographic sensitizer[4] and fungicidal[5] etc.

The introduction of arylhydrazono group results in the formation of compounds which may be expected to show high potential antineoplastic[6] and antidiabetic activities. In the light of the importance of 2-aminobenzothiazole nucleus and hydrazono group, it was considered worthwhile to combine both of them so as to enhance the overall activity and effectiveness of the resulting molecules. Keeping in view the pharmaceutical importance of 3-benzothiazolylhydrazono pentane-2, 4-diones (Ia), 2-benzothiazolylhydrazono ethyk 2-cyanoethanoates (Ib) and 2-benzothiazolylhydrazono-5, 5'-dimethylcyclohexane-1, 3-diones (Ic), it was considered worthwhile to study the separation of these compounds by T.L.C. as this information may provide their better identification during the evaluation of drug. The general structure of the benzothiazoles is



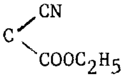
where, R represents different substituents.

(1a) R_1=



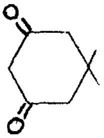
$C\begin{matrix}\diagup COCH_3\\ \diagdown COCH_3\end{matrix}$

(1b) R_1=



$C\begin{matrix}\diagup CN\\ \diagdown COOC_2H_5\end{matrix}$

(1c) R_1=



EXPERIMENTAL

Glass plates of the size 21.5 x 21.5 cm were coated with silica gel G (thickness 0.05 mm) with the help of Stahl type applicator and were developed in glass troughs. All the compounds were synthesized by the method developed in this laboratory[7] and repeatedly recrystallized with DMF before subjecting them to the separation. A 0.2% solution of the compound in acetone was applied to the plates with the help of fine glass capillaries. The composition of the developers used is given in the Table-1. After development the colour of the spots (compounds I-XXXIV) was yellow to brown. So there was no difficulty in detection of spots. However, when experiments were carried with very low concentration of the compounds, spots were visualized by exposure to NO_2 for about 1 minute. It is pertinent to note that no tailing was observed in any of the compounds studied. The R_f-values obtained were found reproducible in the different identical runs and are compiled in Table-1.

RESUTLS AND DISCUSSION

The TLC data on the separation of benzothiazoles are given in Table-1. The chromatographic development time of solvent systems

TABLE 1. TLC DATA

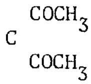
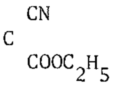
S.No.	R	R_1	M.P.°C	R_f x 100		Detection limit (µg)
				A	B	
1	H	 $C(COCH_3)_2$	187	63	42	4.0
2	4-CH_3	"	182	41	48	3.5
3	5-CH_3	"	165	47	55	3.5
4	6-CH_3	"	175	74	62	3.0
5	4-OCH_3	"	235	32	45	3.5
6	6-OCH_3	"	247	39	57	3.5
7	4-OC_2H_5	"	125	26	25	3.0
8	6-OC_2H_5	"	160	30	31	3.5
9	4-Cl	"	210	36	37	4.0
10	5-Cl	"	146	45	50	4.0
11	6-Cl	"	180	50	67	2.5
12	4,7-$(Cl)_2$	"	100	16	23	2.0
13	4,5-$(CH_3)_2$	"	159	19	27	2.0
14	6-NO_2	"	238	21	30	3.5
15	H	 $C(CN)(COOC_2H_5)$	190	53	32	3.5
16	4-CH_3	"	132	41	36	3.5
17	5-CH_3	"	210	45	40	3.0
18	6-CH_3	"	225	74	46	3.0
19	4-OCH_3	"	135	80	52	3.0
20	6-OCH_3	"	205	33	22	4.0
21	6-OC_2H_5	"	130	37	27	2.5
22	5-Cl	"	118	27	19	2.5

TABLE 1. (Continued)

S.No.	R	R_1	M.P.°C	R_f x 100		Detection limit (μg)
				A	B	
23	6-Cl	"	110	39	38	2.5
24	4,5-$(CH_3)_2$	"	125	10	14	3.0
25	6-NO_2	"	120	22	20	2.0
26	H		177	33	24	3.5
27	4-CH_3	"	122	39	28	3.0
28	5-CH_3	"	161	42	32	3.5
29	6-CH_3	"	193	48	39	3.5
30	4-OCH_3	"	200	30	17	3.5
31	6-OCH_3	"	210	37	21	3.0
32	4-Cl	"	135	26	45	3.0
33	5-Cl	"	108	30	53	4.0
34	4-OC_2H_5	"	177	20	14	3.5
35	6-OC_2H_5	"	140	25	21	3.5
36	4,5-$(CH_3)_2$	"	167	15	12	2.5

SOLVENT COMPOSITION

For compounds (I-XIV)

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

(B) Toluene: Chloroform: Ethylacetate (65% : 30% : 5%).

For compounds (XV-XXV)

(A) Xylene: Chloroform (50% : 50%).

(B) Xylene: Chloroform: Acetone (50% : 45% : 5%).

For compounds (XXVI-XXXVI)

(A) Xylene: Chloroform: Ethylacetate (45% : 45% : 10%).

(B) Xylene: Chloroform: Dioxane (50% : 45% : 5%).

(A and B) employed was about 50 minutes. Both the solvent systems used gave satisfactory separation of most of the compounds. The results show an interesting trend in the R_f-values. It is observed that in the case of ortho substituted derivatives the rate of flow (R_f) of the spots is low whereas meta and para substitutes increase the value of R_f in comparison with that of the parent unsubstituted compound.

REFERENCES

1. William, R.R.: J.Am.Chem.Soc. 57, 229,536 (1935)
2. Fosbinder, R.J., Walter, L.A.: J.Am.Chem.Soc. 61,2032 (1939)
3. Bedford, Sebrell: J.And.Eng.Chem. 15, 1009 (1923)
4. Mills: J.Chem.Soc. 121,455, 2004 (1922)
5. Allen, R.E., Shelton, R.S., Vancampen, M.G.: J.Am.Chem.Soc. 76, 1128 (1954)
6. Shrivastava, P.N., Ph.D. Thesis, University of Roorkee, India (1970)
7. Goyal, R.N., Jain, R., Tyagi, S.: J.Indian Chem.Soc., 58, 504,(1981)