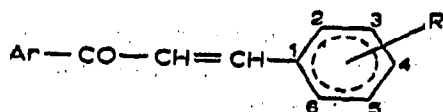


CHROM. 6038

Thin-layer chromatography of some α, β -unsaturated carbonyl compounds. III

Thin-layer chromatographic (TLC) separations on silica gel and polyamide plates of some α, β -unsaturated carbonyl compounds of the chalcone type and their derivatives have been reported in the literature¹⁻⁸. We have examined ninety-three

TABLE I

 hR_F VALUES OF SOME α, β -UNSATURATED CARBONYL COMPOUNDS OF FORMULA

Length of run 10 cm; chamber saturation at 20°.

No.	Compound	hR_F^a	
	Ar	R	
1	Diphenyl	—	64
2	Diphenyl	3-Hydroxy	27
3	Diphenyl	4-Hydroxy	60
4	Diphenyl	2-Chloro	69
5	Diphenyl	3-Chloro	62
6	Diphenyl	2,6-Dichloro	72
7	Diphenyl	2-Methyl	61
8	Diphenyl	3-Methyl	71
9	Diphenyl	4-Methyl	70
10	Diphenyl	3-Methoxy	61
11	Diphenyl	3-Hydroxy-4-methoxy	55
12	Diphenyl	4-Hydroxy-3-methoxy	17
13	Diphenyl	3,4-Dimethoxy	29
14	Diphenyl	4-Nitro	64
15	Diphenyl	3,4-Methylenedioxy	58
16	Diphenyl	5-Bromo-4-hydroxy-3-methoxy	60
17	Diphenyl	5-Bromo-3,4-dimethoxy	48
18	Diphenyl	6-Bromo-3,4-dimethoxy	38
19	Diphenyl	6-Nitro-3,4-dimethoxy	17
20	1-Naphthyl	—	60
21	1-Naphthyl	2-Bromo	57
22	1-Naphthyl	4-Bromo	52
23	1-Naphthyl	2,6-Dichloro	72
24	1-Naphthyl	3-Methyl	51
25	1-Naphthyl	4-Dimethylamino	46
26	1-Naphthyl	5-Bromo-2-hydroxy	31
27	1-Naphthyl	6-Bromo-3,4-methylenedioxy	56
28	1-Naphthyl	5-Bromo-3,4-dimethoxy	37
29	1-Naphthyl	6-Bromo-3,4-dimethoxy	5
30	4-Hydroxy-1-naphthyl	3-Methyl	30
31	4-Hydroxy-1-naphthyl	2-Chloro	24
32	4-Hydroxy-1-naphthyl	4-Chloro	32
33	4-Hydroxy-1-naphthyl	4-Dimethylamino	20
34	4-Hydroxy-1-naphthyl	3,4-Dimethoxy	6
35	4-Methoxy-1-naphthyl	—	58
36	4-Methoxy-1-naphthyl	4-Methyl	59
37	4-Methoxy-1-naphthyl	4-Methoxy	44
38	4-Methoxy-1-naphthyl	3-Hydroxy	19
39	4-Methoxy-1-naphthyl	4-Chloro	61
40	4-Methoxy-1-naphthyl	4-Nitro ^b	9
41	2-Methoxy-1-naphthyl	4-Methyl	34

TABLE I (continued)

No.	Compound		<i>hR_F</i> ^a
	<i>Ar</i>	<i>R</i>	
42	2-Naphthyl	4-Bromo	72
43	2-Naphthyl	4-Methoxy ^b	8
44	2-Naphthyl	2,6-Dichloro	68
45	2-Naphthyl	5-Bromo-2-hydroxy	29
46	2-Naphthyl	5-Bromo-3,4-dimethoxy	7
47	2-Naphthyl	6-Bromo-3,4-methylenedioxy	58
48	2-Naphthyl	6-Nitro-3,4-dimethoxy	26
49	1-Hydroxy-2-naphthyl	4-Hydroxy	66
50	1-Methoxy-2-naphthyl	4-Methyl	52
51	1-Methoxy-2-naphthyl	2-Methoxy	62
52	1-Methoxy-2-naphthyl	4-Methoxy	48
53	1-Methoxy-2-naphthyl	2,3-Dimethoxy	34
54	1-Methoxy-2-naphthyl	3,4-Dimethoxy	21
55	1-Methoxy-2-naphthyl	4-Dimethylamino	33
56	2-Phenanthryl	2-Bromo	61
57	2-Phenanthryl	3-Hydroxy	23
58	2-Phenanthryl	4-Hydroxy	56
59	2-Phenanthryl	2-Methoxy	59
60	2-Phenanthryl	2,3-Dimethoxy	65
61	2-Phenanthryl	3,4-Dimethoxy	28
62	2-Phenanthryl	4-Dimethylamino	55
63	2-Phenanthryl	6-Bromo-3,4-methylenedioxy	53
64	3-Phenanthryl	2-Bromo	53
65	9-Anthryl	2-Nitro	39
66	2-Thienyl	—	64
67	2-Thienyl	3-Hydroxy	23
68	2-Thienyl	4-Hydroxy	20
69	2-Thienyl	4-Methoxy	51
70	2-Thienyl	3-Chloro ^c	61
71	2-Thienyl	4-Chloro	61
72	2-Thienyl	2-Nitro	29
73	2-Thienyl	3-Nitro	37
74	2-Thienyl	4-Nitro ^b	10
75	2-Thienyl	4-Dimethylamino	42
76	2-Thienyl	3-Hydroxy-4-methoxy	17
77	2-Thienyl	3,4-Methylenedioxy	49
78	2,4-Dimethylphenyl	4-Dimethylamino	55
79	5-Nitro-2,4-dimethoxyphenyl	—	12
80	5-Nitro-2,4-dimethoxyphenyl	2-Methyl	24
81	5-Nitro-2,4-dimethoxyphenyl	4-Bromo	8
82	5-Nitro-2,4-dimethoxyphenyl	4-Hydroxy-3-methoxy	10
83	5-Nitro-2,4-dimethoxyphenyl	3,4-Dimethoxy	6
84	5-Nitro-2,4-dimethoxyphenyl	5-Bromo-2-hydroxy	8
85	5-Nitro-2,4-dimethoxyphenyl	5-Bromo-3,4-dimethoxy	37
86	5-Nitro-2,4-dimethoxyphenyl	6-Bromo-3,4-methylenedioxy	53
Ar-CO-CH = CH-Ar'			
87	Diphenyl	2-Furyl	65
88	Diphenyl	2-Hydroxy-1-naphthyl	62
89	5-Nitro-2,4-dimethoxyphenyl	1-Naphthyl	8
90	5-Nitro-2,4-dimethoxyphenyl	2-Furyl	9
91	1-Naphthyl	2-Furyl	59
92	2-Methoxy-1-naphthyl	2-Furyl	52
93	2-Naphthyl	1-Naphthyl	66

^a Average of the results of two separate measurements.

^b Dissolved in dioxan for its application as a spot on TLC plate.

^c Reference compound.

α,β -unsaturated carbonyl compounds by TLC on silica gel using petroleum ether (b.p. 40–60°)–ethyl acetate (5:1) as the mobile phase. The hR_F values ($hR_F = R_F \times 100$) of these compounds are reported in this communication.

Experimental and results

The hR_F values were determined by TLC on 20 × 20 cm glass plates, each of which was coated with a suspension of 6.6 g of Silica Gel G (Merck, G. F. R.) slurried with 25 ml of ethyl acetate. The compounds were applied, as spots, to the dried TLC plate in ethyl acetate or dioxan solutions as described in the literature⁹.

The chromatoplates were developed in equilibrated tanks lined with Whatman No. 3MM filter-paper saturated with freshly prepared solvent mixture. The spots on the thin-layer chromatogram were identifiable by their yellow colour in daylight, which became intensely coloured on brief exposure to iodine vapour, or by the characteristic halochromic colours developed after spraying the layer with concentrated sulphuric acid.

The compounds required for the study were prepared¹⁰ by the Claisen–Schmidt reaction, and were purified by several recrystallisations from appropriate solvent(s). The hR_F values are listed in Table I.

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