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

Twenty α,β -unsaturated carbonyl compounds have been separated by thinlayer chromatography (TLC) on silica gel and their R_F values are collected in Table I.

TABLE I

TLC R_F values (\times 100) on silica gel of some α, β -unsaturated carbonyl compounds

$$R_1$$
— C — CH = CH — R_2

Compounds I-III, VI-IX and XI-XX were applied to the TLC plate in ether solution, while the rest of the compounds were used in THF solution.

Compound	R_1	R_2	R_F (\times 100)	Colour with H ₂ SO ₄
I	4-Bromophenyl	4-N,N-Dimethylaminophenyl	15	Yellow
II	4-Bromophenyl	4-Diphenyl	40	Dull brick red
IIIa	4-Bromophenyl	3,4-Methylenedioxyphenyl	29	
IV	4-Bromophenyl	r-Naphthyl	25	Pink to bright.
Vp	4-Bromophenyl	2-Naphthyl	40	Dirty orange
VI	4-Bromophenyl	9-Anthryl	53	Green to blue
VII	4-Bromophenyl	2-N-Methylpyrryl	17	Dark red
VIII	4-Bromophenyl	2-Furyl	43	Deep yellow
IX	4-Bromophenyl	2-Thienyl	38	Saffron-yellow
Xb	2-Thienyl	4-Diphenyl	27	Dull brick red
XI	2-Thienyl	i-Naphthyl	32	Dark violet
XIIb	2-Thienyl	2-Naphthyl	26	Dark red
XIII	2-Thienyl	g-Anthryl	47	Red
XIV	2-Thienyl	2-N-Methylpyrryl	9	Dark red
XV	2-Hydroxyphenyl	9-Anthryl	57	Yellowish green
XVI	2,4-Dihydroxy-5-nitrophenyl	2-N-Methylpyrryl	11	Orange yellow
XVII	2-Hydroxy-4-methoxyphenyl	9-Anthryl	44	Greenish yellow magenta
XVIII	2-Hydroxy-4-methoxyphenyl	2-N-Methylpyrryl	18	Red
XIX	2-Hydroxy-4-methoxyphenyl	2-Thienyl	42	Pink
XX°	Phenyl	Phenyl	44	

a UV detection.

Experimental

The R_F values were determined by TLC on 20 \times 20 cm glass plates, each of which was coated with a suspension of 3.3 g of silica gel slurried with 12 ml of ethyl acetate. The compounds were applied to the dried TLC plate, in ether/tetrahydrofuran (THF) solutions, in the usual manner². The chromatoplates were developed using benzene-light petroleum ether (b.p. 40°-60°) (2:3) in equilibrated tanks lined with Whatman No. 3MM filter paper, saturated with the solvent mixture just before chromatography. The spots on the developed chromatogram could be located either by their yellow colour or by the characteristic halochromic colours developed after spraying the plate with concentrated sulphuric acid. In some cases exposure of the

^b Streak.

[·] Iodine detection.

chromatogram to UV light of wave length 365 nm, or to iodine vapour was found to be necessary.

The α,β -unsaturated carbonyl compounds were prepared³ by the Claisen–Schmidt reaction and were purified by several crystallizations.

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Department of Chemistry, Indian Institute of Technology, Durga Nath Dhar Kanpur (India)

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