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Note

Dehydrogenation of terpenoids on chromatoplates

J. C. KOHLI*, RANJANA GUPTA, A. K. ARORA and KHUSHMINDER

Chemistry Department, P.A.U., Ludhiana (India)

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A number of organic reactions in a solid matrix are known to occur¹⁻⁴. For example, El-Maghraby⁵ carried out chemical reactions on chromatoplates in order to solve several problems, particularly involving natural products. Materials obtainable from natural sources are often too small for preparing derivatives for comparison with known products or for carrying out reactions in a flask. We describe here the dehydrogenation of terpenoids with *p*-chloranil and 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) on chromatoplates.

EXPERIMENTAL AND RESULTS

Glass plates (5 × 20 cm) were coated with a silica gel G slurry, which was prepared by mixing thoroughly one part by mass of silica gel G with two parts by volume of water. After drying in air the plates were stored and heated in an oven for 30 min at 110°C before use.

TABLE I

R_F VALUES OF REACTION PRODUCTS OF TERPENOIDS WITH *p*-CHLORANIL ON SILICA GEL G PLATES

Solvent: benzene-ethyl acetate (9:1 or 8:2).

Compound	Original compound		Reaction product	
	9:1	8:2	9:1	8:2
Carvone	0.23	0.24	0.23	0.24
Linalool	0.22	0.23	0.27	0.30
Eugenol	0.64	0.65	0.57	0.60
Khusinodiol	0.20	0.24	0.13	0.14
Santonin	0.34	0.35	0.34	0.35
Abietic acid	0.37	0.39	0.46	0.50
Camphene	0.36	0.44	0.81	0.83
Menthol	0.50	0.53	0.72	0.79
Khusol	0.59	0.60	0.59	0.60
Khusilic acid	0.45	0.49	0.32	0.33
Khusilol	0.56	0.58	0.73	0.76
Daucol tosylate	0.78	0.79	0.83	0.87
Podocarpic acid	0.27	0.52	0.66	0.71
Δ^3 -Carene	0.55	0.56	0.77	0.84
Khusinol	0.52	0.63	0.78	0.87

TABLE II

R_F VALUES OF REACTION PRODUCTS OF TERPENOIDS WITH DDQ ON SILICA GEL G PLATES

Solvent: benzene-ethyl acetate (8:2 or 7:3)

Compound	Original compound		Reaction product	
	8:2	7:3	8:2	7:3
Equol	0.28	0.51	0.09	0.12
Khusinol	0.37	0.62	0.08	0.08
Khusol	0.35	0.56	0.07	0.10
Citral	0.44	0.68	0.06	0.08
Camphene	0.61	0.75	0.07	0.07
Khusinodiol	0.14	0.27	0.05	0.06
Santonin	0.21	0.36	0.03	0.06
Khusilic acid	0.06	0.10	0.03	0.05
Khusilol	0.38	0.53	0.06	0.06
Daucol tosylate	0.56	0.59	0.06	0.08
Podocarpic acid	0.30	0.37	0.10	0.12
Δ^3 -Carene	0.50	0.51	0.08	0.09
Carotol	0.37	0.62	0.11	0.13
Daucol	0.51	0.54	0.06	0.07

General method for carrying out the reaction on chromatoplates

Terpenoids in the appropriate solvent were applied to a plate at the starting line together with *p*-chloranil or DDQ dissolved in benzene. The plates were heated in an electric oven at 100°C for 12 h in order to simulate, as far as possible, the normal reaction conditions in a flask. The adsorbed layer was then treated in the normal manner as a chromatographic medium for the resolution of the components of the reaction mixture. Thus, after development with a suitable solvent system, the plates were sprayed with concentrated sulphuric acid-methanol (1:1) and heated in an electric oven at 100°C in order to reveal the spots. The results are given in Tables I and II.

It is interesting that *p*-chloranil or DDQ dehydrogenated khusinol and khusol on the plate. The product was isolated and identified as 1,6-dimethyl-4-isopropyl-naphthalene by comparison with an authentic sample identified using thin-layer chromatography and IR spectroscopy.

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