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Note

Separation and identification of some hydroxychalcones and their derivatives

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Biogenetically, chalcones form an important class of organic natural products which are known to be precursors of various classes of flavonoids in plants[1]. In our investigations on the sensitized photooxygenations of chalcones with singlet oxygen[2], it was observed that 2′,4′,6′-trihydroxychalcone could be converted into the corresponding flavonol, galangin. To study this chalcone–flavonol reaction from a mechanistic point of view, chalcones with different oxygenation patterns were synthesized and purified. The known methods of purification by chromatography[3] were found to be inadequate owing to the presence of unreacted materials, side reactions and polymerization during their synthesis. However, the application of our recent findings[4–6] on thin-layer chromatography with nitrobenzene-treated plates gave satisfactory results and a mixture of 18 chalcones could be resolved and detected on a single chromatogram. In this paper, we report the procedure for purification, separation and identification of various chalcones and their derivatives given in Table I.

EXPERIMENTAL

Materials and methods

Glass plates (15 × 20 × 0.3 cm) were coated with silica gel G (E. Merck, Darmstadt, G.F.R.) ana allowed to stand in a chromatographic tank containing a solution of 2% nitrobenzene in benzene for 6 h. The plates were then removed and allowed to stand in a desiccating cabinet for drying for 2 days.

Developing solvents and detection reagents

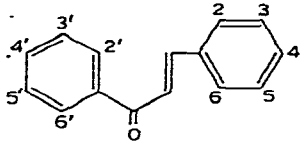
Benzene–ethyl acetate (48:2) was used as the developing solvent and the spots were detected by spraying with sulphuric acid (15%) followed by heating the plates at 110°, alcoholic iron(III) chloride solution (2%) and also by observing them under UV light using a short wavelength (180–300 nm) (UVSL-25). For preparative thin-layer separation the bands were observed under UV light, eluted with methanol, the methanol solution was evaporated to dryness and the compounds were crystallized from a suitable solvent.

RESULTS AND DISCUSSION

The chromatographic results are presented in Table I. Each R_F value reported represents the mean of three determinations carried out under identical conditions. Several other solvent systems were tried but the system reported gave the best results in terms of the separation of all of the components of the mixture of 18 chalcones on a single chromatographic plate. The chromatograms were also developed without

TABLE I

R_F VALUES OF CHALCONES AND SPOT COLOURS ON SILICA GEL G PLATES WITH BENZENE–ETHYL ACETATE (48:2) AS DEVELOPING SOLVENT AT 25°



Compound	$R_F \times 100$	*Spot colours**		
		15% Sulphuric acid	*Alcoholic iron(III) chloride (2%)*	*UV light*
Chalcone	65	DY	–	R
2′,4-Dihydroxychalcone	22	O	G	Y
2′-Hydroxy-4′-methoxychalcone	11	B	GY	RB
2′-Hydroxy-4-methoxychalcone	81	LY	Gr	YB
4′-Methoxy-6′-benzyloxychalcone	58	Y	G	RY
4′,6′-Dibenzyloxychalcone	68	Y	G	RY
2′-Hydroxy-4,4′-dimethoxychalcone	77	DY	YB	GrB
2′-Hydroxy-4′,6′-dimethoxychalcone	55	Y	B	Gr
2′,3,4-Trimethoxychalcone	20	O	GrB	YB
2′-Benzyloxy-4′,3,4-trimethoxychalcone	36	V	PO	GY
2′-Hydroxy-4′,5′-dimethoxychalcone	48	Y	Gr	Y
2′-Hydroxy-3′,4′,6′,3,4-pentamethoxychalcone	29	OR	Gr	Y
2′-Hydroxy-4′,6′,4-trimethoxychalcone	40	OR	OR	BY
2′-Hydroxy-3′-methyl-4′,6′,4-trimethoxychalcone	51	OR	O	Y
3′,5′-Dihydroxy-2′,4′,6′-trimethoxychalcone	32	YB	G	YB
2′,4′,5′,6′-Tetramethoxychalcone	60	LY	–	DY
2′-Hydroxy-3′,4′,6′,3,4-pentamethoxychalcone	23	PO	G	Y
2′,3′,6′-Trihydroxy-4′,5′-dimethoxychalcone	46	Y	GV	YD

* Abbreviations for colours: DY = dull yellow; YD = dark yellow; Y = yellow; R = red; O = orange; B = brown; LY = light yellow; GY = greenish yellow; RB = reddish brown; Gr = grey; YB = yellow-brown; GrB = greyish blue; V = violet; PO = pinkish orange; OR = orange-red; BY = bluish yellow; GV = greenish violet.

prior impregnation with nitrobenzene, which resulted in an unsatisfactory separation of one or more of the components and overlapping of spots. The higher degree of separation with this method is probably due to the formation of a loose complex between the adsorbed nitrobenzene and the substrates, which have a high electron density on the aromatic rings. We have earlier successfully separated a mixture of xanthones[4] and analgesic ingredients[6] of complex pharmaceutical formulations by using the same technique of impregnation with organic substances. Further work on the quantitative aspects of such complexation is being continued.

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