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Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

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Published online: 23 Sep 2006.

To cite this article: N. Hans & S. K. Grover (1993) An Efficient Convrsion of 2′-Hydroxychalcones to Flavones, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 23:7, 1021-1023, DOI: 10.1080/00397919308013299

To link to this article: http://dx.doi.org/10.1080/00397919308013299

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SYNTHETIC COMMUNICATIONS, 23(7), 1021-1023 (1993)

AN EFFICIENT CONVERSION OF 2'-HYDROXYCHALCONES TO FLAVONES

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Abstract: 2'-Hydroxychalcones have been found to undergo smooth conversion to flavones when heated with sodium periodate in dimethyl sulphoxide.

Flavones, an important and abundant group of flavonoids, have been synthesised, among various methods[1], by the oxidative cyclisation of 2'-hydroxychalcones. Some of the important reagents which have been used for this purpose are selenium dioxide in pentan-1-ol[2], Pd-C/vacuum[3], iodine in dimethyl sulphoxide[4], selenium dioxide in dimethyl sulphoxide[5], DDQ in dioxan[6] and nickel peroxide in dioxan[7].

The above methods are of limited application as in some of these procedures[2], the yields are not satisfactory while some procedures[6,7] give a mixture of products containing flavone, flavonone and aurone. The recent reports on the use of iodine in DMSO or selenium dioxide in DMSO suffer from the limitation that 2'-hydroxychalcones possessing a phloroglucinol oxygenation pattern in ring A do not give the desired product but a complex mixture.

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In our search for a reagent which would have wider and general applicability it has been found that 2'-hydroxychalcones (Ia-h) when heated at 100-120° for 24-48 hrs with sodium periodate in dimethyl sulphoxide undergo smooth conversion to the corresponding flavones (Table). The structures of the products were confirmed by their ^{1}H-NMR spectra and comparison with authentic samples (co-IR).

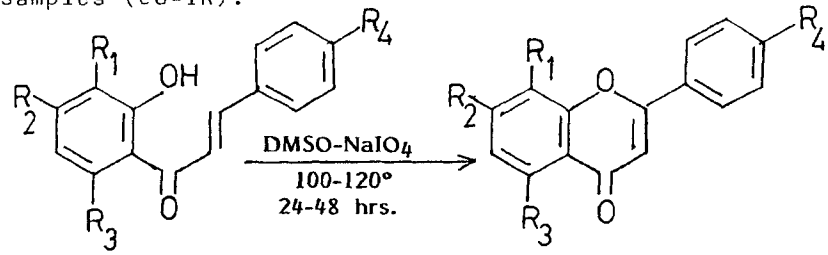

Table: Flavones

S.No.	R_1	R_2	R_3	R_4	Rxn time (hrs)	m.p. (°C)	Lit (°C)	Yield (%)
IIa	H	OCH_3	H	H	24	110°	110°[9]	60
IIb	H	OCH_3	OCH_3	H	48	145°	146-47°[9]	50
IIc	H	H	H	H	24	99°	99°[9]	60
IId	H	H	H	OCH_3	24	157°	158°[9]	55
IIe	H	OCH_3	H	OCH_3	48	141°	141°[9]	55
IIf	H	OCH_3	OCH_3	OCH_3	48	156°	156°[9]	25
IIg	OCH_3	OCH_3	H	H	48	150°	151°[5]	50
IIh	OCH_3	OCH_3	H	OCH_3	48	190°	190°[9]	50

Experimental

2'-Hydroxychalcones (Ia-h) were prepared by modified method[8] involving reaction of appropriate acetophenone with benzaldehyde in aqueous ethanolic alkali medium in the presence of benzyl triethyl

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ammonium chloride. Their m.ps were in agreement with those recorded in literature.

Flavones (General procedure)

2'-Hydroxychalcone (0.92 mmole) in dry dimethyl sulphoxide (4.0 ml) was heated at 100-120^{0} with sodium periodate (4.67 mmole) in an oil bath till the starting material had completely undergone conversion (TLC; 24-48 hrs). The reaction mixture was cooled and to it was added sodium thiosulphate solution (10%, 20 ml). The solid so obtained was filtered and dried in vacuo over phosphorous pentoxide. The dry solid on recrystallisation from ethyl acetate - light petroleum afforded flavone (II).

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(Received in UK 21 October 1992)

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