

NOTE

An Efficient Synthesis of 2'-Hydroxychalcones

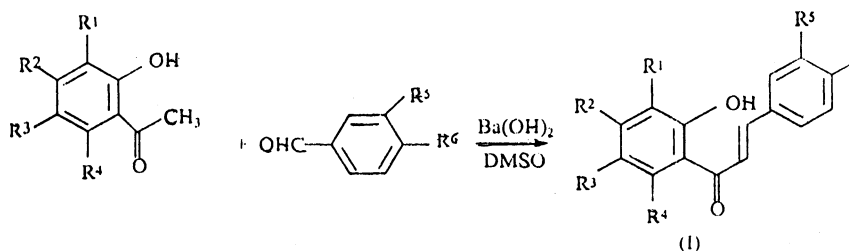
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2'-Hydroxychalcones have been synthesized by condensation of 2-hydroxyacetophenones with aromatic aldehydes in the presence of dehydrated barium hydroxide in dry dimethyl sulphoxide medium.

Key Words: Synthesis, 2'-Hydroxychalcones.

2'-Hydroxychalcones constitute an important class of naturally occurring compounds which occupy a central place in the biogenesis and also are used as intermediates for the synthesis of various other flavonoids.¹ These compounds are usually synthesized by Claisen-Schmidt condensation between 2-hydroxyacetophenone and an aromatic aldehyde in the presence of ethanol-aqueous alkali (10–30%) at room temperature for 24–48 h.² Various modifications in the reaction conditions have also been reported^{2,3} but longer reaction periods or increase in reaction temperature usually lead to the formation of side products, thus lowering the yields of the required compounds.

Now we report a simple and efficient procedure for the synthesis of 2'-hydroxychalcones that involves the condensation between a 2-hydroxyacetophenone and aromatic aldehyde in the presence of dehydrated barium hydroxide in dry dimethylsulphoxide medium. Various substituted 2'-hydroxychalcones were obtained in 65–90% yield and their structures were confirmed by comparison of their authentic samples (m.m.p.-IR) and ¹H NMR data.



Commercial Ba(OH)₂·8H₂O was heated in an oven at 200°C for 2 h. The mass was powdered and stored in a desiccator.

2'-Hydroxychalcone (General procedure)

A solution of 2-hydroxyacetophenone (2.8 mmol) and aromatic aldehyde (3.0

mmol) in dry dimethyl sulphoxide (20 mL) was heated with dehydrated barium hydroxide (1 g) on boiling water bath for 30 min. Completion of the reaction was checked by TLC. Ice-cold water was added to the reaction mixture after cooling and acidified with conc. hydrochloric acid. The solid that separated out was filtered, washed with water and recrystallised from ethanol to give 2'-hydroxychalcone. The physical data of the synthesized 2'-hydroxychalcones are given in Table-1.

TABLE-1
PHYSICAL DATA OF SYNTHESIZED 2'-HYDROXYCHALCONES

Compound	R ¹	R ²	R ³	R ⁴	R ⁵	R ⁶	Yields	m.p. (°C)	Lit. mp (°C)
1a	H	H	H	H	H	H	90	89	90 ⁴
1b	H	H	H	H	H	OCH ₃	85	95	95 ⁵
1c	H	H	H	H	OCH ₃	OCH ₃	75	113	115 ⁶
1d	H	H	CH ₃	H	H	H	85	105	106 ⁷
1e	H	H	CH ₃	H	H	OCH ₃	80	98	99 ⁷
1f	H	OCH ₃	H	H	H	H	80	105	105 ⁸
1g	H	OCH ₃	H	H	H	OCH ₃	80	113	113 ⁸
1h	H	OCH ₃	H	H	OCH ₃	OCH ₃	70	155	156 ⁹
1i	OCH ₃	OCH ₃	H	H	H	H	75	97	98 ¹⁰
1j	OCH ₃	OCH ₃	H	H	H	OCH ₃	70	131	132 ¹¹
1k	H	OCH ₃	H	OCH ₃	H	H	70	91	91 ¹²
1l	H	OCH ₃	H	OCH ₃	H	OCH ₃	70	132	132 ¹³
1m	H	OCH ₃	H	OCH ₃	OCH ₃	OCH ₃	65	150	151 ¹⁴

REFERENCES

1. J.B. Harborne, T.J. Mabry and H. Mabry, *The Flavonoids*, Chapman & Hall Ltd., pp. 127-213 (1975).
2. D.N. Dhar and J.B. Lal, *J. Org. Chem.*, **23**, 1159 (1958).
3. P.K. Jain, Pinkey and S.K. Grover, *Curr. Sci.*, **52**, 1185 (1983).
4. S.V. Kostanecki and W. Feuerstein, *Chem. Ber.*, **31**, 715 (1889).
5. G.V. Bhide, K.R. Chandarkar, K. Pendse and S.D. Limaye, *Rasayanam*, **2**, 135 (1956).
6. H. Ozawa, T. Okuda, M. Kawanishi and K. Fujii, *J. Pharm. Soc. Japan*, **71**, 1178 (1951).
7. C.H. Lin, C.T. Lin and C.I. Yeh, *J. Chinese Chem. Society*, **14**, 9 (1967); *Chem. Abstr.*, **68**, 12632 (1968).
8. R.N. Goel, A.C. Jain and T.R. Seshadri, *Proc. Ind. Acad. Sci.*, **48A**, 180 (1958).
9. N. Narasinhachari and T.R. Seshadri, *Proc. Ind. Acad. Sci.*, **38A**, 223 (1948).
10. S.V. Kostanecki, J. Tambor and E. Bonifzi, *Chem. Ber.*, **36**, 4238 (1903).
11. S.V. Kostanecki and B. Schreiber, *Chem. Ber.*, **38**, 2749 (1905).
12. Y. Kimura, *J. Pharm. Soc. (Japan)*, **60**, 151 (1940).
13. S.V. Kostanecki and J. Tambor, *Chem. Ber.*, **37**, 79 (1904).
14. T. Emilewicz and S.V. Kostanecki, *Chem. Ber.*, **31**, 696 (1898).