



SYNTHESIS OF IODO-CHALCONES

A. S. DIGHADE* and S. R. DIGHADE

Department of Chemistry, Bar. R.D.I.K. & N.K.D College, BADNERA (M.S.) INDIA

ABSTRACT

Six different chalcones **I(a)-I(f)** were synthesized by condensing 2-hydroxy-3-iodo-5-methyl acetophenone with six different aromatic aldehydes in ethanol using NaOH. The synthesized compounds were characterized by IR and NMR spectral analysis.

Key words: Iodo - chalcones.

INTRODUCTION

Claisen synthesized chalcones by condensation of 2-hydroxyacetophenone with aromatic aldehydes in presence of acidic¹ or basic² media. Thirunarayanan and Vanangamudi³ synthesized some 4-bromo-1-naphthyl chalcone using silica-sulfuric acid reagent under solvent free condition. Xu et al.⁴ synthesized the chalcones catalysed by a novel solid sulfonic acid from Bamboo.

Severi et al.⁵ synthesized and studied activities of a new series of chalcones as aldose reductase inhibitors. Palleros⁶ studied solvent free synthesis of chalcones. Srivastava⁷ synthesized ecofriendly microwave assisted synthesis of chalcones. Rohrmann et al.⁸ used the chalcone in the synthesis of medicinal intermediates. Chalcones show the amoebicidal activity⁹.

EXPERIMENTAL

Melting points of all synthesized compounds were determined in open capillary tube and are uncorrected. The purity of compounds was checked by TLC using silica G. IR. Spectra were recorded on Perkin-Elmer-841 spectrometer (cm^{-1}) in KBr disc and NMR (Bruker Avance II 400 NMR) using CDCl_3 as solvent.

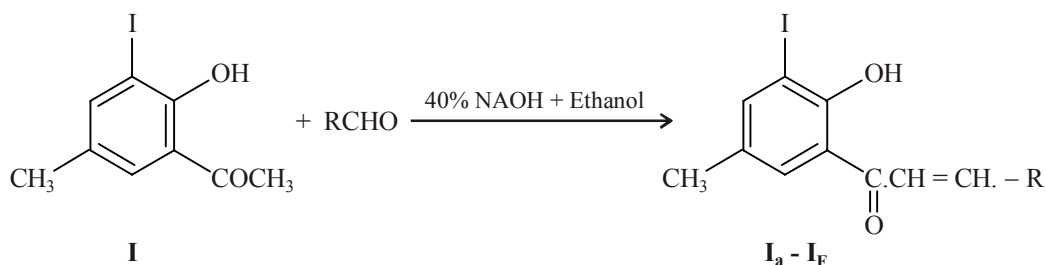
* Author for correspondence; E-mail: aprana_dighade@rediffmail.com

Synthesis of 2-hydroxy-3-iodo-5-methyl-acetophenone (Compound-1)

By known method p-cresyl acetate was prepared from p-cresol to and then by Fries migration-2-hydroxy-5-methyl acetophenone, which on iodination gives 2-hydroxy-3-iodo-5-methyl acetophenone (Comp. 1).

Synthesis of substituted 2-hydroxy-3-iodo-5-methyl chalcones (I_a – I_f)

Compound I_a to I_f were synthesized from 2-hydroxy-3-iodo-5-methyl acetophenone by reacting with six different aromatic aldehydes by known method in solvent ethanol using 40% NaOH. The physical data of compound I_a to I_f is given in Table 1.




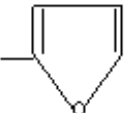
Reaction Scheme

The groups R are shown in Table 1.

Table 1

S. No.	Compd. No.	R	Mol. formula	M.P. (°C)	Yield (%)
1.	I _a		C ₁₆ H ₁₃ O ₂ I	110°C	72
2.	I _b		C ₁₇ H ₁₅ O ₃ I	142°C	68
3.	I _c		C ₁₆ H ₁₂ O ₂ ICl	160°C	70
4.	I _d		C ₁₆ H ₁₃ O ₃ I	80°C	66

Cont...

S. No.	Compd. No.	R	Mol. formula	M.P. (°C)	Yield (%)
5.	I _e	-HC = HC 	C ₁₈ H ₁₅ O ₂ I	130°C	63
6.	I _f		C ₁₄ H ₁₁ O ₃ I	80°C	65

Characterization data of compound (I)

2-Hydroxy-3-iodo-5-methyl acetophenone (I)

IR (KBr) ν_{\max} (cm⁻¹): 3200 cm⁻¹ (s) – phenolic OH, 2919 cm⁻¹ (s) – Aromatic C-H stretching, 1635 cm⁻¹ C = O stretching, 1082 cm⁻¹ (S) Ar-CH₃ - stretching, 1020 cm⁻¹ (S) CH₃ stretching and 550 cm⁻¹ C-I stretching .

¹H NMR: (δ CDCl₃): 2.3 δ (s, 3H, Ar- CH₃), 2.6 δ (s, 3H, COCH₃), 7.5 δ (s, 1H, Ar-H), 7.7 δ (s, 1H, Ar-H) and 12.9 δ (s, 1H, Ar-OH).

2-Hydroxy-3-iodo-5-methyl- chalcone (I)

IR (KBr) ν_{\max} (cm⁻¹): 3412 cm⁻¹ (br) – phenolic OH, 2917 cm⁻¹ (s) – Ar- C-H stretching, 1746 cm⁻¹ C = O of O = C-CH = CH stretching, 1358 cm⁻¹ C-O stretching in phenol, 1264-1230 cm⁻¹ Ar-O stretching in ether and 563-548 cm⁻¹ C-I stretching .

¹H NMR: (δ CDCl₃): 2.3-2.5 δ (s, 3H, Ar- CH₃), 7.5 δ (s, 2H, -CH = CH), 7.5-7.9 δ (m, 6H, Ar-H) and 13.5 δ (s, 1H, Ar-OH).

2-Hydroxy-3-iodo-5-methyl-4-methoxy chalcone (I)

IR (KBr) ν_{\max} (cm⁻¹): 3412 cm⁻¹ (br) – phenolic OH, 2912 cm⁻¹ (s) – Ar- C-H stretching , 1743 cm⁻¹ (S) C = O stretching, 1637-1604 cm⁻¹ O = C-CH = CH stretching, 1352 cm⁻¹ C-O stretching in phenol, 1266-1237 cm⁻¹ Ar-O stretching in ether and 536 cm⁻¹ C-I stretching .

¹H NMR: (δ CDCl₃): 2.31 δ (s, 3H, Ar- CH₃), 3.85 δ (s, 3H, OCH₃), 6.9 δ (d, 2H, -CH = CH), 7.2-8.1 δ (m, 6H, Ar-H) and 13.7 δ (s, 1H, Ar-OH).

RESULTS AND DISCUSSION

Compound no. 1, I_a and I_b were synthesized through the route as shown in general reactions R as shown in Table 1. Similarly, physical data are shown in Table 1. The synthesized compounds 1, I_a and I_b were confirmed on the basis of IR and NMR spectral analysis.

ACKNOWLEDGEMENT

Authors are thankful to Principal, Br. R. D. I. K. & N. K. D. College, Badnera and Head, Deptt. of Chemistry, Br. R. D. I. K. & N. K. D. College, Badnera and RC SAIF, Punjab University, Chandigarh for spectral analysis I. R. and N. M. R.

REFERENCES

1. L. Claisen and A. Chaparede, Ber., **14**, 2463 (1881).
2. St. Kostanencki, Voy and G. Rossbach, Ber., **29**, 1492 (1896).
3. G. Thirunaratyanan and G. Vanangamudi, ARKIVOC (XII), 58-64 (2006).
4. Q. Xu, Z. Yang, D. Yin and F. Zhang, J. Catal., **233(2)**, 266-275 (2005).
5. F. Severi, S. Bbenvenuti, L. Costantino and L. Antolini, European J. Med. Chem., **33(11)**, 859-866 (1998).
6. D. R. Palleros, J. Chem. Educ., **81(9)**, 1345 (2004).
7. Y. K. Srivastava, Rasayan, J. Chem., **1(4)**, 884-886 (2008).
8. E. Rohrmann, R. G. Jones and H. A. Shonle, J. Am. Chem. Soc., **66(11)**, 1856-1857 (1944).
9. U. K. Sanfi, S. Datta, and C. P. Datta, Natl. Acad. Sci. Lett., (India) **6**, 271 (1984).

Accepted : 17.02.2012