



SYNTHESIS OF 2-HYDROXY SUBSTITUTED CHALCONE DIBROMIDE

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ABSTRACT

2-Hydroxy substituted chalcone have been synthesized by using acetophenone with anisaldehyde and acetophenone with benzaldehyde by using ethanol and then synthesis 2-hydroxy substituted chalcone dibromides was synthesized by using bromine in acetic acid. The constitution of the synthesized compounds was supported by IR, UV, PMR and elemental analysis.

Key words: 2-Hydroxy substituted chalcone dibromide, Characterization.

INTRODUCTION

Acetophenone ($\text{CH}_3\text{COC}_6\text{H}_5$) is a ketone which contains methyl group ($-\text{CH}_3$) and a phenyl group ($-\text{C}_6\text{H}_5$). In view of noble application of acetophenone, for enhancing the pharmaceutical, industrial, medicinal and biological uses, various substituted acetophenones were synthesized. One of the most notable and considerable substituted acetophenone is 2-hydroxy-5-chloro acetophenone.

Synthesis and antimicrobial activity of some novel chalcones and their related compounds were studied by Kaithwal et al.¹ Shridhar et al.² have synthesized and studied the antibacterial and anti-inflammatory activity of chalcone 1-(2',5'-dimethyl-3-furyl)-3-(aryl)-2-propen-1-one.

The reaction between substituted benzaldehyde and acetophenones has been used to demonstrate green chemistry in under graduate chemistry education³. In a study investigating green chemistry synthesis, chalcones were also synthesized from the same starting material in high temperature water (200 to 350°C)⁴.

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Raut et al.⁵ reported the synthesis of 2-hydroxy-3-bromo-5-chloro substituted chalcone dibromide by using bromine (25% w/v) in acetic acid from 2-hydroxy-3-bromo-5-chloro substituted chalcone. Deshmukh et al.⁶ synthesized p-substituted chalcone dibromide from p-substituted chalcone by bromination in acetic acid (25% w/v). while Utale et al.⁷ reported chalcone dibromide by the reaction of bromine in 25% w/v acetic acid with 3-substituted-2-hydroxy-5-chloro chalcone. Khadsan and Doshi⁸ synthesized the α , β -acrylophenone dibromide from acrylophenones by bromination using acetic acid.

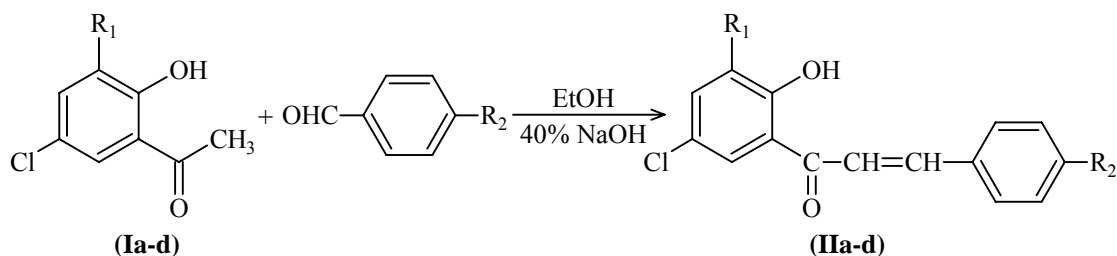
EXPERIMENTAL

Preparation of 2'-hydroxy substituted chalcone

2'-Hydroxy substituted acetophenone with anisaldehyde and benzaldehyde were dissolved in ethanol and the reaction mixture was brought to boiling in presence of aq. NaOH with constant stirring. The solution was kept overnight and next day, sodium salt obtained was decomposed by aq. HCl. A yellow coloured product was obtained.

Product:

- (i) 2'-Hydroxy-5'-chloro-4-methoxy chalcone (**IIa**). m.p. 117°C, yield 70%.
- (ii) 2-Hydroxy-5-chlorochalcone (**IIb**). m.p. 121°C, yield 74%.
- (iii) 2'-Hydroxy-3'-bromo-5'-chloro-4-methoxy chalcone (**IIc**). m.p. 172°C, yield 72%.
- (iv) 2-Hydroxy-3-bromo-5-chloro chalcone (**II d**). m.p. 124°C, yield 80%.



where, $\text{R}_1 = -\text{H}, -\text{Br}$

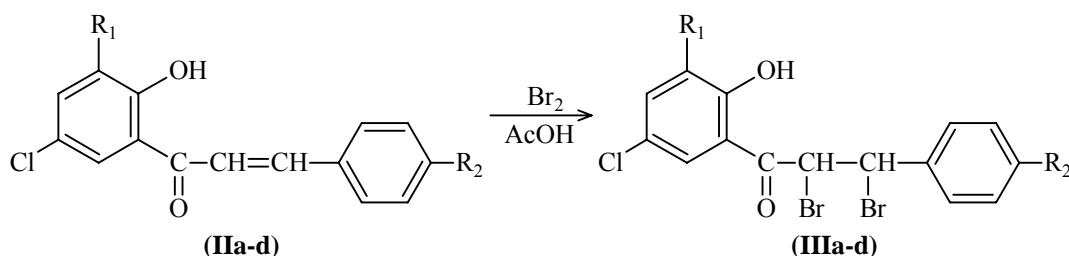
$\text{R}_2 = -\text{H}, -\text{OCH}_3$

Preparation of 2'-hydroxy substituted chalcone dibromide (**IIIa**)

2'-Hydroxy substituted chalcone was dissolved in glacial acetic acid by warming and then the solution was cooled. A solution of bromine in acetic acid was added to this solution

with stirring. A yellow coloured Product was obtained.

- (i) 2'-Hydroxy-5'-chloro-4-methoxy chalcone dibromide (**IIIa**), m.p. 148°C, yield 80%.
- (ii) 2'-Hydroxy-5'-chloro chalcone dibromide (**IIIb**), m.p. 182°C, yield 72%.
- (iii) 2'-Hydroxy-3'-bromo-5'-chloro-4-methoxy chalcone dibromide (**IIIc**), m.p. 138°C, yield 68%.
- (iv) 2'-Hydroxy-3'-bromo-5'-chloro chalcone dibromide (**III d**), m.p. 116°C, yield 75%.



where, $R_1 = -\text{H}, -\text{Br}$

$R_2 = -\text{H}, -\text{OCH}_3$

The structures of the compounds (**IIIa-d**) confirmed on the basis of chemical properties, analytical results and spectral data.

RESULTS AND DISCUSSION

2-Hydroxy-5-chloroacetophenone (**Ib**), 2-hydroxy-3-bromo-5-chloro-acetophenone (**Ic**), 2'-hydroxy-5'-chloro-4-methoxy chalcone (**IIa**), 2'-hydroxy-5'-chloro-4-methoxy chalcone dibromide (**IIIa**) and 2-hydroxy-3-bromo-5-chloro chalcone dibromide (**III d**) were prepared and their UV, IR and PMR are discussed here.

2'-Hydroxy-5'-chloro-4-methoxy chalcone dibromide (**IIIa**)

It is a yellow crystalline compound, m.p. 148°C. It gives green colouration with neutral FeCl_3 solution indicating the presence of free phenolic $-\text{OH}$ group. All the compounds (**IIIa-d**) have same properties.

IR (KBr) cm^{-1} : 3406 (Ar-OH), 3074 (Ar-C-H), 1691 (C=O), 1600-1500 (Ar-C-C), 1469 (-CH-C), 1257 and 1053 (Ar-O-CH₃), 1023 and 1128 (Ar-C-Cl), 831 (p-substitution), 813 (m-substitution), 690 (o-substitution), 590 (CH-Br); **$^1\text{H NMR}$ (CDCl_3 with TMS): δ**

2.35 (q, 1H, -CH-Br), 2.61 (d, 1H, HCBBr-C = O), 3.94 (s, 3H, O-CH₃), 6.75-7.51 (m, 7H, Ar-H), 15.93 (s, 1H, -OH); **Mass (m/z):** 448.3. Anal. Calcd. for C₁₆H₁₃O₃ClBr₂ (%) C, 42.83; H, 2.90; O, 10.71; Cl, 7.91; Br, 35.65; Found: C, 43.06; H, 2.89; O, 10.78; Cl, 7.85; Br, 35.52; **UV (nm):** 235 ($\pi \rightarrow \pi^*$) and 301 ($n \rightarrow \pi^*$).

2'-Hydroxy-5'-chloro chalcone dibromide (IIIb)

IR (KBr) cm⁻¹: 3480 (Ar-OH), 3084 (Ar-C-H), 1760 (C=O), 1640-1560 (Ar-C-C), 1416 (-CH-C), 1234 and 1168 (Ar-C-Cl), 875 (m-substitution), 721 (o-substitution); **¹H NMR (CDCl₃ with TMS):** δ 2.634 (d, 1H, -CH-Br), 2.764 (d, 1H, HCBBr-C = O), 6.88-8.13 (m, 7H, Ar-H), 12.79 (s, 1H, -OH); **Mass (m/z):** 418.3. Anal. Calcd. for C₅H₁₁O₂ClBr₂ (%) C, 43.03; H, 2.63; O, 7.65; Cl, 8.47; Br, 38.20; Found : C, 43.38; H, 2.61; O, 7.67; Cl, 8.42; Br, 38.82; **UV (nm):** 238 ($\pi \rightarrow \pi^*$) and 349 ($n \rightarrow \pi^*$).

2'-Hydroxy-3'-bromo-5'-chloro-4-methoxy chalcone dibromide (IIIc)

IR (KBr)⁹⁻¹³ cm⁻¹: 3500 (Ar-OH), 3050 (Ar-C-H), 1692 (C=O), 1600-1560 (Ar-C-C), 1470 (-CH-C), 1256 and 1055 (Ar-O-CH₃), 1221 and 1178 (Ar-C-Cl), 1220 and 1025 (Ar-Br), 880 (p-substitution), 817 (m-substitution), 720 (o-substitution), 660-580 (CH-Br)⁹⁻¹³; **¹H NMR (CDCl₃ with TMS):** δ 2.37 (d, 1H, -CH-Br), 2.62 (d, 1H, HCBBr-C = O), 3.98 (s, 3H, O-CH₃), 6.80-7.54 (m, 7H, Ar-H), 15.8 (s, 1H, -OH); **Mass (m/z):** 527.2. Anal. Calcd. for C₁₆H₁₂O₃ClBr₃ (%) C, 36.42; H, 2.28; O, 9.10; Cl, 6.72; Br, 45.47; Found: C, 36.76; H, 2.22; O, 10.01; Cl, 6.77; Br, 46.04; **UV (nm):** 231 ($\pi \rightarrow \pi^*$) and 334 ($n \rightarrow \pi^*$)¹⁴⁻¹⁵.

2'-Hydroxy-3'-bromo-5'-chloro chalcone dibromide (IIIId)

IR (KBr) cm⁻¹: 3479 (Ar-OH), 3089 (Ar-C-H), 1714 (C=O), 1681 and 1602 (Ar-C-C), 1429 (-CH-C), 1234 and 1166 (Ar-C-Cl), 1125 and 1080 (Ar-Br), 880 (m-substitution), 720 (o-substitution), 642 and 580 (CH-Br); **¹H NMR (CDCl₃ with TMS):** δ 2.37 (d, 1H, -CH-Br), 2.64 (d, 1H, HCBBr-C = O), 6.77-7.53 (m, 7H, Ar-H), 15.7 (s, 1H, -OH); **Mass (m/z):** 49.2. Anal. Calcd. for C₁₅H₁₀O₂ClBr₃ (%) C, 36.20; H, 2.01; O, 6.44; Cl, 7.13; Br, 48.21; Found: C, 36.82; H, 1.99; O, 6.52; Cl, 7.10; Br, 48.09; **UV (nm):** 254 ($\pi \rightarrow \pi^*$) and 350 ($n \rightarrow \pi^*$).

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