

Synthesis and Characterization of Novel Chalcones Derivatives having 1-(4-benzyloxy-2-hydroxy-5-iodophenyl) Ethanone Moiety

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ABSTRACT

Chalcones derivatives has wide applications in Pharmaceutical and medicinal chemistry. 1-(4-benzyloxy-2-hydroxy-5-iodophenyl)-3-(substitutedphenyl)prop-2-en-1-one Compounds (B₁₋₁₀) were synthesized by coupling with aromatic substituted aldehyde. All the synthesized compounds were characterized by IR, ¹H NMR.

Keywords: Chalcones, Phenone Derivatives.

I. INTRODUCTION

The presence of chalcones is one main structural components in Various naturally occurring biologically active compounds chalcones, analogs of 1,3-diaryl pro-2-ene-1-one form a wide class of compounds containing two aromatic rings bound with vinyl ketone fragment. It is well known that largely natural or synthetic chalcones are highly active with extensive pharmaceutical and medicinal application. Chalcones are found to be effective as analgesic [1], antimalarial [2], antiviral [3], antibacterial [4], antifungal [5], antimutagenic [6], cytotoxic [7], antifeedant [8], anti-inflammatory [9], antileishmanial [10], antitumor [11], anticancer [12], antimicrobial [13], antinociceptive [14], insecticidal [15] and antiinvasive [16] activities.

II. EXPERIMENTAL

Melting points were determined in open capillary tubes and are uncorrected. The IR spectra were recorded in KBr pellets on a Buker spectrometer and ¹H NMR spectra in CDCl₃ on Hitachi R-1500, 60 MHz spectrometer using TMS as an internal standard. All chemicals used were of laboratory grade. Preparation of 1-(4-benzyloxy-2-hydroxy-3-iodo phenyl) ethanone (BHIE) and chalcone is as given below.

Synthesis of 1-(4-benzyloxy-2-hydroxy phenyl) ethanone

General procedure:

1-(2, 4 dihydroxyphenyl) ethanone (0.10 mol), Benzyl bromide (0.1 mol) and Potassium carbonate (0.1 mol) were taken in 100ml of Acetone. Reaction mixture was shake for 7 hrs at reflux 50-60°C temperature. Reaction mixture was cooled to room temperature and quenched with 100ml cold water. The final product

1-(2-hydroxy-4-benzyloxyphenyl) ethanone was passed through a filter and rinsed with water. Prepared product was recrystallized by ethanol.

Synthesis of 1-(4-benzyloxy-2-hydroxy-5-iodo phenyl) ethanone: (A)

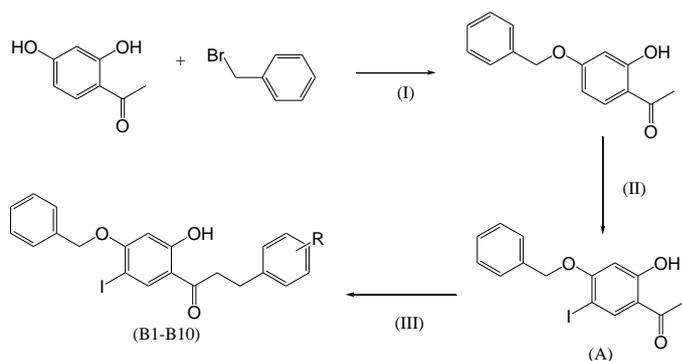
1-(2-hydroxy-4-benzyloxyphenyl) ethanone (0.1 mol) was taken in 100ml of ethanol. Iodination method⁷ has been used. Iodine granules (0.1 mol) and 300ml were taken in 250ml R.B.F and stirred them till 15 minutes. Iodic acid (0.1 mol) dilute in to 4ml of dist.water in a small beaker. Slowly add this iodic acid solution in to the reaction mixture and stirred them continuously for 30 minutes at 35 – 40 C, the reaction was monitored by TLC. Pour it in to ice. Excess iodine was removed by adding fresh saturated sodium bisulphite solution. Formed material 1-(2-hydroxy-4-benzyloxy-3-iodo phenyl) ethanone was passed through filter out and washes them two to three times with distilled water. Synthesized material was recrystallized in ethanol.

Synthesis of 1-(4-benzyloxy-2-hydroxy-5-iodo phenyl)-3-(substituted phenyl) prop-2-en-1-one from 1-(4-benzyloxy-2-hydroxy-3-iodo phenyl) ethanone : (B₁-B₁₀)

General procedure:

1-(4-benzyloxy-2-hydroxy-5-iodophenyl) ethanone (0.01 mol) and substituted aromatic aldehydes (0.01 mol) were dissolved in ethanol (25 ml) was added 10% sodium hydroxide solution, (25 ml) was added slowly and the mixture stirred for 4 hrs, the reaction monitored by TLC. Then it was poured into 400 ml of water with constant stirring and neutralized with 10% hydrochloric acid solution and left overnight in refrigerator. The precipitate obtained was filtrated, washed and recrystallized from ethanol.

REACTION SCHEME



R= H, 2-Cl, 3-Cl, 4-Cl, 2-OH, 3-OH, 4-OH, 2-OCH₃, 3-OCH₃, 4-OCH₃

1-(4-benzyloxy-2-hydroxy-5-iodophenyl) ethanone(A)

Mass;368.17 ; IR(KBr cm⁻¹): 2870(C-H str. vib.) 3032(-Aromatic C-H),1573,1489, (C=C str.Vib.),879(-C - H o.o.p multi sub. benzene),1280, 1080(C-O-C str.vib), 3634(O-H str.vib), 1620(-C=O str.vib),501(C-I str.vib);¹H NMR 6.44 – 7.77 (s,7H,of the Ar-H),13.5 (s,1H, Ar-OH), 5.16 (2H,s, -CH₂-O-), 2.5 (3H,s, O=CCH₃);Yield 64.30%;

1-(4-Benzyloxy-2-hydroxy-5-iodophenyl)-3-phenylprop-2-en-1-one [B₁]:

Mass;456.27 IR(KBr cm⁻¹): 3063(-Aromatic C-H),1573, 1489,(C=C str. Vib.),817(-C - H o.o.pmulti sub. benzene),1273, 1072(C-O-C str.vib), 3634(O-H str.vib), 1627(-C=O str.vib), 501(C-I str.vib) ,972(CH=CH bending);¹H NMR:8.0 -7.7 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH₂-O-), Yield 57.23%;

1-(4-benzyloxy-2-hydroxy-5-iodophenyl)-3-(4-chlorophenyl) prop-2-en-1-one [B₂]:

Mass; 490.72 IR(KBr cm⁻¹): 3032(Aromatic C-H),1573, 1489,(C=C str. Vib.),817(-C - H o.o.pmultisub. benzene),1280, 1041(C-O-C str.vib), 3201(O-H str.vib), 1627(-C=O str.vib), 786(C-Cl str.vib) 578(C-Istr.vib),972(CH=CH bending), ¹H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.49 (m, 2H, -CH=CH-), 5.16(d,2H, -CH₂-O-); Yield 59.91%;

1-(4-benzyloxy- 2-hydroxy-5-iodophenyl)-3-(3-hydroxyphenyl) prop-2-en-1-one [B3]:

Mass;472.27 IR(KBr cm-1): 3050(Aromatic C-H),1489, 1404,(C=C str. Vib.),817(-C - H o.o.pmultisub. benzene),1273, 1080(C-O-C str.vib), 3201(O-H str.vib), 1627(-C=O str.vib), 501(C-I str.vib),964(CH=CHbending), 1H NMR:8.0 -7.7 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH₂-O-); Yield 64.32%;

1-(4-benzyloxy- 2-hydroxy- 5-iodophenyl)-3-(4-methoxyphenyl) prop-2-en-1-one [B4]:

Mass:486.3; IR(KBr cm-1): 3063(Aromatic C-H),1558, 1404,(C=C str. Vib.),825(-C - H o.o.pmultisub. benzene),1280, 1072(C-O-C str.vib), 3194(O-H str.vib), 1627(-C=O str.vib), 540(C-I str.vib),972(CH=CH)bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH₂-O-); Yield 62.81%;

1-(4-benzyloxy -2-hydroxy-5-iodophenyl)-3-(2-chlorophenyl) prop-2-en-1-one [B5]:

Mass; 490.72 IR(KBr cm-1): 3063(Aromatic C-H),1573, 1450,(C=C str. Vib.),864(-C - H o.o.pmultisub. benzene),1226, 1049(C-O-C str.vib), 3649(O-H str.vib), 1627(-C=O str.vib), 732(C-Cl str.vib) 509(C-Istr.vib),972(CH=CH bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.49 (m, 2H, -CH=CH-), 5.16(d,2H, -CH₂-O-); Yield 58.50%;

1-(4-benzyloxy- 2-hydroxy-5-iodophenyl)-3-(3-hydroxyphenyl) prop-2-en-1-one [B6]:

Mass;472.27 IR(KBr cm-1): 3032(Aromatic C-H),1489, 1404,(C=C str. Vib.),817(-C - H o.o.pmultisub. benzene),1219, 1080(C-O-C str.vib), 3518(O-H str.vib), 1620(-C=O str.vib), 501(C-I str.vib),941(CH=CHbending), 1H NMR:8.0 -7.7

(m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH₂-O-); Yield 59.37%;

1-(4-benzyloxy- 2-hydroxy- 5-iodophenyl)-3-(4-methoxyphenyl) prop-2-en-1-one [B7]:

Mass:486.3; IR(KBr cm-1): 3063(Aromatic C-H),1573, 1489,(C=C str. Vib.),856(-C - H o.o.pmultisub. benzene),1280, 1072(C-O-C str.vib), 3194(O-H str.vib), 1627(-C=O str.vib), 509(C-I str.vib),972(CH=CH)bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH₂-O-); Yield 53.17%;

1-(4-benzyloxy -2-hydroxy-5-iodophenyl)-3-(4-chlorophenyl) prop-2-en-1-one [B8]:

Mass; 490.72 IR(KBr cm-1): 3032(Aromatic C-H),15723, 1492,(C=C str. Vib.),817(-C - H o.o.pmultisub. benzene),1273, 1041(C-O-C str.vib), 3439(O-H str.vib), 1620(-C=O str.vib), 786(C-Cl str.vib) 501(C-Istr.vib),972(CH=CH bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.49 (m, 2H, -CH=CH-), 5.16(d,2H, -CH₂-O-); Yield 58.23%;

1-(4-benzyloxy- 2-hydroxy-5-iodophenyl)-3-(3-hydroxyphenyl) prop-2-en-1-one [B9]:

Mass;472.27 IR(KBr cm-1): 3032(Aromatic C-H),1489, 1404,(C=C str. Vib.),879(-C - H o.o.pmultisub. benzene),1280, 1080(C-O-C str.vib), 3510(O-H str.vib), 1620(-C=O str.vib), 501(C-I str.vib),966(CH=CHbending), 1H NMR:8.0 -7.7 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH₂-O-); Yield 59.71%;

1-(4-benzyloxy- 2-hydroxy- 5-iodophenyl)-3-(4-methoxyphenyl) prop-2-en-1-one [B10]:

Mass:486.3; IR(KBr cm-1): 3032(Aromatic C-H),1489, 1404,(C=C str. Vib.),815(-C - H o.o.pmultisub. benzene),1280, 1041(C-O-C str.vib), 3510(O-H

str.vib), 1627(-C=O str.vib), 501(C-I) ,648 (C-I str.vib),966(CH=CH)bending), ¹H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH₂-O-); Yield 54.33%;

III. RESULTS AND DISCUSSION

In the present work, some novel chalcones of 1- (4-benzyloxyphenyl-2-hydroxy-5-iodophenyl) ethanone (BHIE) from ten aromatic substituted aldehydes have been prepared. During the preparation work, it was found that most of the chalcones using aromatic aldehydes could be easily prepared by most convenient claisen-schmidt condensation method. The chalcones could be easily prepared at room temperature after 72 hours. It was found that the chalcones derived from aromatic aldehydes were stable and can be easily converted to its heterocyclic compound. To establish a new synthetic process for chalcones, different reaction conditions were applied. From the results, it was found that the chalcones could be prepared from BHIE using aromatic substituted aldehyde by shaking the reaction mixture at normal temperature for 4 hours. Thus, a new developed synthetic process has been applied to prepare chalcones from BHIE using aromatic substituted aldehydes in the present work.

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