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# Synthesis and Antibacterial Efficacy of 1-(2', 4'-Dihydroxyphenyl)-3-Aryl-Propane-1, 3-Diones

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**Abstract**

*1-(2', 4'-Dihydroxyphenyl)-3-aryl-propane-1, 3-diones* ***3a-h*** *have been synthesized by employing Baker-Venkataraman transformation on 2-aroyloxy-4-hydroxyacetophenones* ***2a-h*** *with NaOH in dimethylsulfoxide regardless of pyridine. The structure of the synthesized compounds has been assigned on the basis of elemental and spectral analyses (IR, $^{1}$HNMR, $^{13}$C NMR and Mass). The synthesized compounds were evaluated for the antibacterial activity against gram negative and gram positive bacteria.*

**Keywords:** β-Diketones, dibenzoylmethanes, esterification, Baker-Venkataraman Transformation, antibacterial activity.

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## INTRODUCTION

β-Diketones, besides a key building block for the synthesis of core heterocycles such as pyrazole [1], isoxazole [2], flavone [3], benzodiazepine [4] and pyrimidine [5] have been shown to have varying degree of pharmacological activities like antibacterial [6], antiviral [7], insecticidal [8], antioxidant [9] and potential prophylactic antitumor activity [10]. It has also been used as anti-sunscreen agent [11]. Dibenzoylmethane (DBM) has antimutagenic activity, it has been demonstrated that DBM inhibits 7, 12-dimethylbenz[α]anthracene (DMBA)-induced breast tumorigenesis in mice [12]. Singletary et al. have shown that DBM has potent chemopreventive activity against DMBA-induced carcinogenesis in rat [13]. Also β-ketoenols are important pharmacophores of HIV-1 integrase (IN) inhibitors [14].

Since, the chemist would not stop at this stage of β-diketones having such varying pharmacological activities and in continuation of our earlier research work on β-diketones oriented our attention to open the library of new 1-(2', 4'-dihydroxyphenyl)-3-aryl-propane-1, 3-diones **3a-h**. Herein, we aim to report the synthesis and antibacterial activity of **3a-h** which

were obtained via simple and convenient method using Baker-Venkataraman rearrangement with NaOH in DMSO [15], despite the use of pyridine having unpleasant odour and not so easy to remove from the reaction mixture. The 2-aroyloxy-4-hydroxyacetophenones **2a-h**, the precursor of **3a-h** were previously obtained by conventional method.

## MATERIALS AND METHODS

### Experimental Section

Resacetophenone **1** was prepared by Nenki reaction from resorcinol and glacial acetic acid [16]. Melting points were determined in open glass capillaries and were uncorrected. Elemental analyses were determined using the Perkin Elmer 2400 CHN analyzer. FT-IR spectra were recorded using (KBr) disc on Perkin-Elmer spectrum Rx-I spectrometer. $^{1}$H NMR and $^{13}$C NMR were recorded on Brucker AC-300 F (300 MHz) NMR spectrometer by using DMSO-$d_6$ and $CDCl_3$ as solvent and tetramethylsilane as an internal standard. Mass spectra were recorded on 70-S Mass spectrometer using m-nitro benzyl alcohol (NBA) matrix.

**2-Benzoyloxy-4-hydroxyacetophenone 2a:** Resacetophenone **1** (0.01 mol) and benzoic acid (0.01 mol) were dissolved in 5 mL of redistilled pyridine and cooled, to that $POCl_3$ 1 mL was added dropwise with constant stirring maintaining the temperature below 20$^{O}$C. The reaction mixture was kept overnight at room temperature and poured with stirring on ice cold dil HCl (1 mol in 50 mL). A white granulated solid compound **2a** separated out which was washed with cold water, dil $NaHCO_3$ solution and again with cold water. The product was filtered off, dried and recrystallized from alcohol. It gave positive test for ester. Yield 70%, mp 99-100$^{O}$C, FT-IR (KBr): 3440 (OH), 1765 (ester C=O), 1690 (C=O), 1601 (aromatic C=C), 1145 (C-O); $^{1}$H NMR (DMSO-$d_6$, 300MHz) δ: 2.53 (s, 3H, $CH_3$), 4.79 (s, 1H, OH), 8.02 (d, 2H, $J_{2'\text{-}6'}$=7.8 Hz, 2'-H, 6'-H), 6.73-7.91 (m, 5H, Ar-H), 6.73 (s, 3-H, Ar-H); $^{13}$C NMR (DMSO-$d_6$, 300MHz,) δ: 198.9 (s, C-7, C=O), 153.7 (s, C-2, C-O), 165.1 (s, C-9, C=O), 130.1 (s, C-1'), 129.7 (s, C-2', C-6'), 128.2 (s, C-3', C-5'), 133.1 (s, C-4'), 28.8 (s, $CH_3$ of C-8), 120.3 (s, C-1), 108.5 (s, C-3), 164 (s, C-4), 113.7 (s, C-5), 131 (s, C-6); MS (EI, 70eV): m/z (%) 256 ($M^+$, 100), 135 (20), 105 (54), 51 (18). Elemental analysis: C, 70.65, H, 4.80. Similarly other 2-aroyloxy-4-hydroxyacetophenones **2b-h** were prepared by the same method.

**2-(2'-Chlorobenzoyloxy)-4-hydroxyacetophenone 2b**: Yield 68%, mp 105-106$^{O}$C, FT-IR (KBr): 3431 (OH), 1763 (ester C=O), 1685 (C=O), 1602 (aromatic C=C), 1131 (C-O); $^{1}$H NMR (DMSO-$d_6$, 300MHz) δ: 2.49 (s, 3H, $CH_3$), 4.85 (s, 1H, OH), 6.70-8.04 (m, 6H, Ar-H), 6.69 (s, 3-H, Ar-H); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 199.1 (s, C-7, C=O), 155.7 (s, C-2, C-O), 165.1 (s, C-9, C=O), 130.5 (s, C-1'), 134.7 (s, C-2'), 128.6 (s, C-3'), 135.1 (s, C-4'), 127 (s, C-5'), 131.1 (s, C-6'), 29.2 (s, $CH_3$ of C-8), 119 (s, C-1), 108.9 (s, C-3), 164.2 (s, C-4), 113.2 (s, C-5), 130.9 (s, C-6); MS (EI, 70eV): m/z (%) 290 ($M^+$, 100), 292 (21), 135 (10), 105 (47), 51 (11). Elemental analysis: C, 62.01, H, 3.92.

**2-(3'-Chlorobenzoyloxy)-4-hydroxyacetophenone 2c**: Yield 74%, mp 97-98$^{O}$C, FT-IR (KBr): 3435 (OH), 1763 (ester C=O), 1680 (C=O), 1599 (aromatic C=C), 1140 (C-O); $^{1}$H NMR (DMSO-$d_6$, 300MHz) δ: 2.57 (s, 3H, $CH_3$), 4.91 (s, 1H, OH), 6.73-8.11 (m, 6H, Ar-H), 6.67 (s, 3-H, Ar-H); $^{13}$C NMR (DMSO-$d_6$, 300MHz,) δ: 198.2 (s, C-7, C=O), 154.9 (s, C-2, C-O), 166.3 (s, C-9, C=O), 130.9 (s, C-1'), 134.2 (s, C-2'), 130.6 (s, C-3'), 135.2 (s, C-4'), 128 (s, C-5'), 130.9 (s, C-6'), 28.8 (s, $CH_3$ of C-8), 118.3 (s, C-1), 109.2 (s, C-3), 165.1 (s, C-4), 113.7

(s, C-5), 131.2 (s, C-6); MS (EI, 70eV): m/z (%) 290 ($M^+$, 100), 292 (19), 135 (18), 105 (43), 51 (11). Elemental analysis: C, 61.83, H, 3.99.

**2-(4'-Chlorobenzoyloxy)-4-hydroxyacetophenone 2d**: Yield 65%, mp 101-102$^O$C, FT-IR (KBr): 3441 (OH), 1762 (ester C=O), 1689 (C=O), 1602 (aromatic C=C), 1139 (C-O); $^1$H NMR (DMSO-$d_6$, 300MHz,) δ: 2.61 (s, 3H, $CH_3$), 4.81 (s, 1H, OH), 8.06 (d, 2H, $J_{2'-6'}$= 8.4 Hz, 2'-H, 6'-H), 7.47 (d, 2H, $J_{3'-5'}$= 8.4 Hz, 3'-H, 5'-H), 6.69-7.74 (m, 2H, Ar-H), 6.71 (s, 3-H, Ar-H); $^{13}$C NMR (DMSO-$d_6$, 300MHz): δ 199.1 (s, C-7, C=O), 154.7 (s, C-2, C-O), 165.4 (s, C-9, C=O), 129.4 (s, C-1'), 130.8 (s, C-2', C-6'), 128.7 (s, C-3', C-5'), 138.3 (s, C-4'), 29.8 (s, $CH_3$ of C-8), 120.1 (s, C-1), 109.5 (s, C-3), 164.1 (s, C-4), 112.7 (s, C-5), 130.2 (s, C-6); MS (EI, 70eV): m/z (%) 290 ($M^+$, 100), 292 (13), 135 (19), 105 (39), 51 (18). Elemental analysis: C, 61.90, H, 3.78.

**4-Hydroxy-2-(3'-methoxybenzoyloxy)acetophenone 2e**: Yield 71%, mp 89-90$^O$C, FT-IR (KBr): 3430 (OH), 1760 (ester C=O), 1681 (C=O), 1600 (aromatic C=C), 1143 (C-O); $^1$H NMR (DMSO-$d_6$, 300MHz) δ: 2.53 (s, 3H, $CH_3$), 4.99 (s, 1H, OH), 7.02-7.79 (m, 6H, Ar-H), 6.63 (s, 3-H, Ar-H), 3.70 (s, 3H, 3'-$OCH_3$); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 199.0 (s, C-7, C=O), 155.9 (s, C-2, C-O), 166.8 (s, C-9, C=O), 131.1 (s, C-1'), 114.2 (s, C-2'), 160.6 (s, C-3'), 119.2 (s, C-4'), 129.7 (s, C-5'), 122.9 (s, C-6'), 29.4 (s, $CH_3$ of C-8), 119.3 (s, C-1), 109.8 (s, C-3), 164.3 (s, C-4), 112.7 (s, C-5), 130.2 (s, C-6), 56.2 (s, $OCH_3$ of C-7'); MS (EI, 70eV): m/z (%) 286 ($M^+$, 100), 135 (20), 105 (43), 51 (11). Elemental analysis: C, 67.25, H, 4.99.

**4-Hydroxy-2-(4'-methoxybenzoyloxy)acetophenone 2f**: Yield 69%, mp 95-96$^O$C, FT-IR (KBr): 3448 (OH), 1766 (ester C=O), 1685 (C=O), 1603 (aromatic C=C), 1144 (C-O); $^1$H NMR (DMSO-$d_6$, 300MHz) δ: 2.59 (s, 3H, $CH_3$), 4.89 (s, 1H, OH), 8.03 (d, 2H, $J_{2'-4'}$=8.1 Hz, 2'-H, 4'-H), 6.99 (d, 2H, $J_{3'-5'}$=8.1 Hz, 3'-H, 5'-H), 6.68-7.76 (m, 2H, Ar-H), 6.79 (s, 3-H, Ar-H), 3.68 (s, 3H, 4'-$OCH_3$); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 199.7 (s, C-7, C=O), 155.7 (s, C-2, C-O), 164.4 (s, C-9, C=O), 122.4 (s, C-1'), 131.8 (s, C-2', C-6'), 114.7 (s, C-3', C-5'), 165.3 (s, C-4'), 29.1 (s, $CH_3$ of C-8), 120.4 (s, C-1), 108.7 (s, C-3), 164.1 (s, C-4), 112.1 (s, C-5), 130.9 (s, C-6), 56.1 (s, $OCH_3$ of C-7'); MS (EI, 70eV): m/z (%) 286 ($M^+$, 100), 135 (21), 105 (49), 51 (14). Elemental analysis: C, 67.22, H, 4.91.

**4-Hydroxy-2-(3'-methylbenzoyloxy)acetophenone 2g**: Yield 76%, mp 103-104$^O$C, FT-IR (KBr): 3433 (OH), 1767 (ester C=O), 1689 (C=O), 1603 (aromatic C=C), 1146 (C-O); $^1$H NMR (DMSO-$d_6$, 300MHz) δ: 2.58 (s, 3H, $CH_3$), 4.83 (s, 1H, OH), 6.79-7.95 (m, 6H, Ar-H), 6.75 (s, 3-H, Ar-H), 2.39 (s, 3H, 3'-$CH_3$); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 199.6 (s, C-7, C=O), 154.9 (s, C-2, C-O), 166.2(s, C-9, C=O), 130.1 (s, C-1'), 130.2 (s, C-2'), 138.6 (s, C-3'), 134.2 (s, C-4'), 128.7 (s, C-5'), 127.9 (s, C-6'), 29.1 (s, $CH_3$ of C-8), 120.3 (s, C-1), 109.6 (s, C-3), 164.7 (s, C-4), 112.9 (s, C-5), 130.8 (s, C-6), 24.8 (s, $CH_3$ of C-7'); MS (EI, 70eV): m/z (%) 270 ($M^+$, 100), 135 (14), 105 (66), 51 (21), 15 (11). Elemental analysis: C, 71.16, H, 5.30.

**4-hydroxy-2-(4'-methylbenzoyloxy)acetophenone 2h**: Yield 72%, mp 107-108$^O$C, FT-IR (KBr): 3442 (OH), 1767 (ester C=O), 1680 (C=O), 1601 (aromatic C=C), 1141(C-O); $^1$H NMR (DMSO-$d_6$, 300MHz) δ: 2.52 (s, 3H, $CH_3$), 4.80 (s, 1H, OH), 8.01 (d, 2H, $J_{2'-4'}$=8.6 Hz, 2'-H, 4'-H), 7.19 (d, 2H, $J_{3'-5'}$=8.6 Hz, 3'-H, 5'-H), 6.79-7.73 (m, 2H, Ar-H), 6.71 (s, 3-H, Ar-H), 2.43 (s, 3H, 4'-$CH_3$); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 199.1 (s, C-7, C=O), 155.2 (s, C-2, C-O), 166.4 (s, C-9, C=O), 122.3 (s, C-1'), 130.4 (s, C-2', C-6'), 129.7 (s, C-3', C-5'), 143.3 (s, C-4'), 29.5 (s, $CH_3$ of C-8), 120.2 (s, C-1), 108.8 (s, C-3), 164.4 (s, C-4), 113.1 (s, C-5),

131.9 (s, C-6), 24.1 (s, $CH_3$ of C-7'); MS (EI, 70eV): m/z (%) 270 ($M^+$, 100), 135 (19), 105 (53), 51 (18), 15 (14). Elemental analysis: C, 71.18, H, 5.35.

**1-(2', 4'-Dihydroxyphenyl)-3-phenyl-propane-1, 3-dione 3a:** The product **2a** (0.005 mol) was dissolved in 4mL of DMSO. To that solution powdered NaOH (1g) was added with vigorous stirring for about five min. The stirring was continued for about 5 min further. The reaction mixture was then cooled and poured on cold water. The pale yellow solid product obtained was washed with water and filtered off. It was crystallized from alcohol. Yield 67%, mp 158-159$^O$C, FT-IR (KBr): 3420 (OH), 1743 (C=O), 1598 (aromatic C=C), 1142 (C-O); $^1$H NMR (DMSO-$d_6$, 300MHz,) δ: 15.72 (s, 1H, enolic OH), 12.02 (s, 1H, 2'-OH), 4.76 (s, 1H, 4'-OH), 8.41 (s, 1H, -CH=), 7.39 (d, 2H, $J_{2''\text{-}6''}$= 8.4 Hz, 2''-H, 6''-H), 6.48-7.47 (m, 5H, Ar-H), 6.39 (s, 3'-H, Ar-H); $^{13}$C NMR (DMSO-$d_6$, 300MHz,) δ: 189.8 (s, C-1, C=O), 184.9 (s, C-3), 94 (s, C-2, -CH=), 115.4 (s, C-1'), 163.2 (s, C-2'), 104.5 (s, C-3'), 165.7 (s, C-4'), 109 (s, C-5'), 132.7 (s, C-6'), 130.4 (s, C-1''), 126.4 (s, C-2'', C-6''), 128.7 (s, C-3'', C-5''), 128 (s, C-4''); MS (EI, 70eV): m/z (%) 256 ($M^+$, 100), 224 (41), 105 (56), 51 (16); Elemental analysis: C, 70.55, H, 4.91. In a same way other 1-(2', 4'-Dihydroxyphenyl)-3-aryl-propane-1, 3-diones **3b-h** have been prepared.

**1-(2', 4'-Dihydroxyphenyl)-3-(2''-chlorophenyl)-propane-1, 3-dione 3b**: Yield 70%, mp 151-151$^O$C, FT-IR (KBr): 3417 (OH), 1738 (C=O), 1599 (aromatic C=C), 1144 (C-O); $^1$H NMR (DMSO-$d_6$, 300MHz) δ: 15.79 (s, 1H, enolic OH), 12.00 (s, 1H, 2'-OH), 4.79 (s, 1H, 4'-OH), 8.49 (s, 1H, -CH=), 6.41-7.39 (m, 6H, Ar-H), 6.33 (s, 3'-H, Ar-H); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 189.7 (s, C-1, C=O), 185.9 (s, C-3), 93 (s, C-2, -CH=), 115.3 (s, C-1'), 163.4 (s, C-2'), 104.1 (s, C-3'), 165.2 (s, C-4'), 109.4 (s, C-5'), 133.1 (s, C-6'), 131.7 (s, C-1''), 131.2 (s, C-2''), 128.8 (s, C-3''), 129.4 (s, C-4''), 126.8 (s, C-5''), 130.2 (s, C-6''); MS (EI, 70eV): m/z (%) 290 ($M^+$, 100), 292 (13), 224 (43), 105 (59), 51 (11). Elemental analysis: C, 62.09, H, 3.97.

**1-(2', 4'-Dihydroxyphenyl)-3-(3''-chlorophenyl)-propane-1, 3-dione 3c**: Yield 71%, mp 124-125$^O$C, FT-IR (KBr): 3423 (OH), 1739 (C=O), 1600 (aromatic C=C), 1143 (C-O); $^1$H NMR (DMSO-$d_6$, 300MHz) δ: 15.89 (s, 1H, enolic OH), 12.01 (s, 1H, 2'-OH), 4.86 (s, 1H, 4'-OH), 8.42 (s, 1H, -CH=), 6.47-7.23 (m, 6H, Ar-H), 6.33 (s, 3'-H, Ar-H); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 190.1 (s, C-1, C=O), 185.2 (s, C-3), 93.4 (s, C-2, -CH=), 115.9 (s, C-1'), 163.2 (s, C-2'), 104.3 (s, C-3'), 164.2 (s, C-4'), 109 (s, C-5'), 132.7 (s, C-6'), 131.8 (s, C-1''), 126.2 (s, C-2''), 134.4 (s, C-3''), 128.4 (s, C-4''), 130.8 (s, C-5''), 124.4 (s, C-6''); MS (EI, 70eV): m/z (%) 290 ($M^+$, 100), 292 (17), 224 (37), 105 (51), 51 (15). Elemental analysis: C, 61.97, H, 3.92.

**1-(2', 4'-Dihydroxyphenyl)-3-(4''-chlorophenyl)-propane-1, 3-dione 3d**: Yield 69%, mp 210-211$^O$C, FT-IR (KBr): 3422 (OH), 1733 (C=O), 1600 (aromatic C=C), 1143 (C-O); $^1$H NMR (DMSO-$d_6$, 300MHz) δ: 15.89 (s, 1H, enolic OH), 12.09 (s, 1H, 2'-OH), 4.72 (s, 1H, 4'-OH), 8.51 (s, 1H, -CH=), 7.30 (d, 2H, $J_{2''\text{-}4''}$= 7.9 Hz, 2''-H, 4''-H), 7.12 (d, 2H, $J_{3''\text{-}5''}$= 7.9 Hz, 3''-H, 5''-H ), 6.46-7.40 (m, 2H, Ar-H), 6.37 (s, 3'-H, Ar-H); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 189.7 (s, C-1, C=O), 185.3 (s, C-3), 94.5 (s, C-2, -CH=), 116.4 (s, C-1'), 163.6 (s, C-2'), 104.5 (s, C-3'), 164.7 (s, C-4'), 109.5 (s, C-5'), 132.1 (s, C-6'), 128.4 (s, C-1''), 127.4 (s, C-2'', C-6''), 128.8 (s, C-3'', C-5''), 133.5 (s, C-4''); MS (EI, 70eV): m/z (%) 290 ($M^+$, 100), 292 (12), 224 (39), 105 (57), 51 (17). Elemental analysis: C, 62.15, H, 4.01.

**1-(2', 4'-Dihydroxyphenyl)-3-(3"-methoxyphenyl)-propane-1, 3-dione 3e**: Yield 73%, mp 189-190$^{O}$C, FT-IR (KBr): 3428 (OH), 1743 (C=O), 1601 (aromatic C=C), 1145 (C-O); $^{1}$H NMR (DMSO-$d_6$, 300MHz) δ: 16.01 (s, 1H, enolic OH), 12.05 (s, 1H, 2'-OH), 4.89 (s, 1H, 4'-OH), 8.42 (s, 1H, -CH=), 6.43-7.49 (m, 6H, Ar-H), 6.39 (s, 3'-H, Ar-H), 3.76 (s, 3H, 3"-$OCH_3$); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 189.7 (s, C-1, C=O), 184.9 (s, C-3), 93.4 (s, C-2, -CH=), 115.6 (s, C-1'), 163.7 (s, C-2'), 104.1 (s, C-3'), 164.2 (s, C-4'), 109.8 (s, C-5'), 132.7 (s, C-6'), 131.4 (s, C-1"), 110.2 (s, C-2"), 160.4 (s, C-3"), 113.4 (s, C-4"), 129.8 (s, C-5"), 118.4 (s, C-6"), 55.9 (s, $OCH_3$ of C-7"); MS (EI, 70eV): m/z (%) 286 ($M^+$, 100), 224 (32), 105 (49), 51 (13). Elemental analysis: C, 67.11, H, 4.89.

**1-(2', 4'-Dihydroxyphenyl)-3-(4"-methoxyphenyl)-propane-1, 3-dione 3f**: Yield 64%, mp 164-165$^{O}$C, FT-IR (KBr): 3419 (OH), 1713 (C=O), 1600 (aromatic C=C), 1139 (C-O); $^{1}$H NMR (DMSO-$d_6$, 300MHz) δ: 15.99 (s, 1H, enolic OH), 12.29 (s, 1H, 2'-OH), 4.68 (s, 1H, 4'-OH), 8.41 (s, 1H, -CH=), 7.20 (d, 2H, $J_{2"-4"}$= 8.1 Hz, 2"-H, 4"-H), 7.19 (d, 2H, $J_{3"-5"}$= 8.1 Hz, 3"-H, 5"-H), 6.49-7.41 (m, 2H, Ar-H), 6.37 (s, 3'-H, Ar-H), 3.65 (s, 3H, 4"-$OCH_3$); $^{13}$C NMR (300MHz, DMSO-$d_6$): δ 189.3 (s, C-1, C=O), 184.9 (s, C-3), 94 (s, C-2, -CH=), 116.3 (s, C-1'), 163.9 (s, C-2'), 104.5 (s, C-3'), 165.7 (s, C-4'), 110.4 (s, C-5'), 131.2 (s, C-6'), 122.4 (s, C-1"), 127.8 (s, C-2", C-6"), 114.8 (s, C-3", C-5"), 159.5 (s, C-4"), 56 (s, $OCH_3$ of C-7"); MS (EI, 70eV): m/z (%) 286 ($M^+$, 100), 224 (21), 105 (66), 51 (20). Elemental analysis: C, 67.21, H, 4.95.

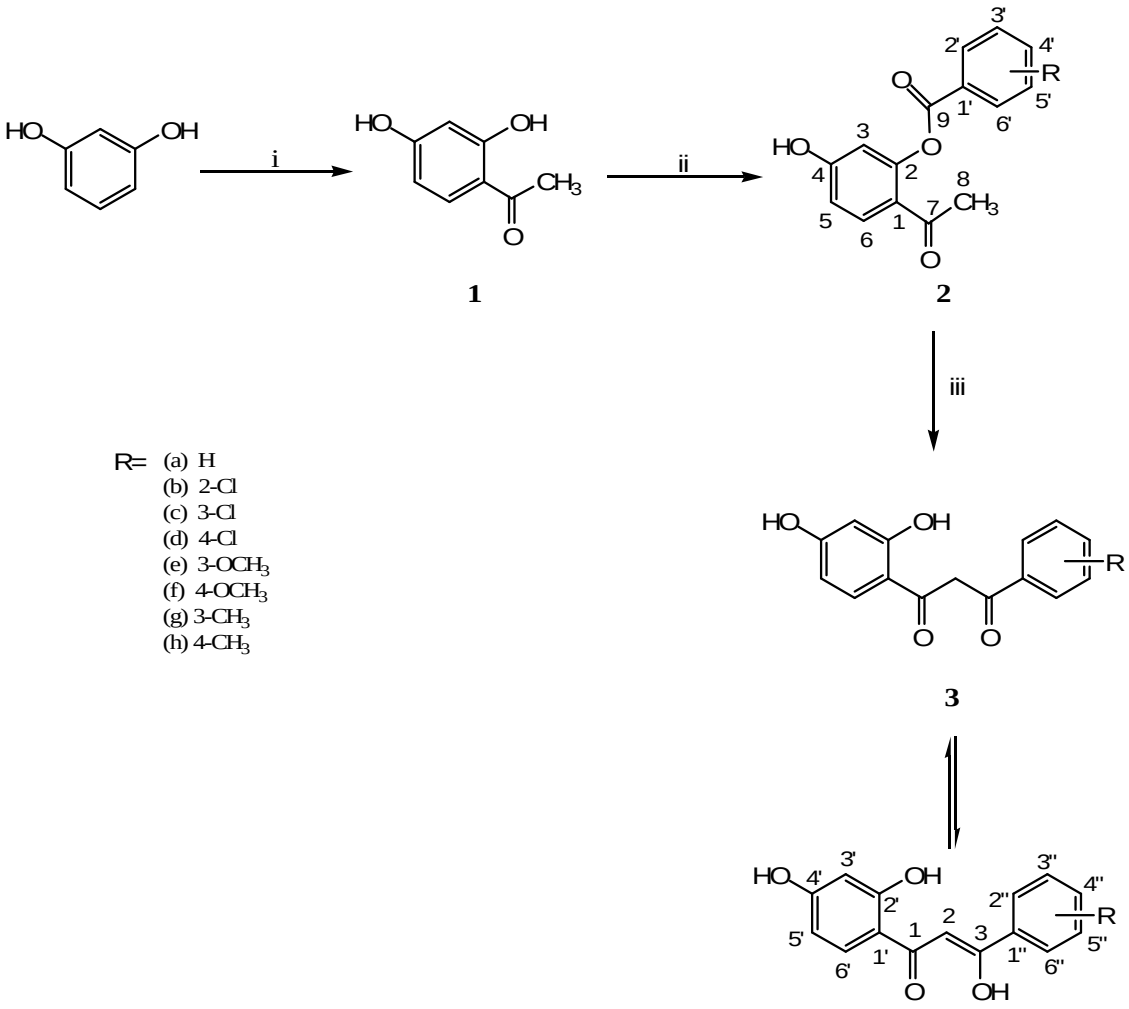
**1-(2', 4'-Dihydroxyphenyl)-3-(3"-methylphenyl)-propane-1, 3-dione 3g**: Yield 74%, mp 136-137$^{O}$C, FT-IR (KBr): 3439 (OH), 1744 (C=O), 1600 (aromatic C=C), 1148 (C-O); $^{1}$H NMR (DMSO-$d_6$, 300MHz) δ: 16.03 (s, 1H, enolic OH), 12.01 (s, 1H, 2'-OH), 4.78 (s, 1H, 4'-OH), 8.49 (s, 1H, -CH=), 6.46-7.47 (m, 6H, Ar-H), 6.39 (s, 3'-H, Ar-H), 2.46 (s, 3H, 3"-$CH_3$); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 189.9 (s, C-1, C=O), 184.7 (s, C-3), 93.8 (s, C-2, -CH=), 115.9 (s, C-1'), 164.7 (s, C-2'), 104.1 (s, C-3'), 164 (s, C-4'), 109.2 (s, C-5'), 131.9 (s, C-6'), 130.4 (s, C-1"), 126.2 (s, C-2"), 138.4 (s, C-3"), 128.4 (s, C-4"), 128.8 (s, C-5"), 123.4 (s, C-6"), 24.8 (s, $CH_3$ of C-7"); MS (EI, 70eV): m/z (%) 270 ($M^+$, 100), 224 (29), 105 (55), 51 (16). Elemental analysis: C, 71.19, H, 5.32.

**1-(2', 4'-Dihydroxyphenyl)-3-(4"-methylphenyl)-propane-1, 3-dione 3h**: Yield 72%, mp 170-171$^{O}$C, FT-IR (KBr): 3425 (OH), 1723 (C=O), 1602 (aromatic C=C), 1140 (C-O); $^{1}$H NMR (DMSO-$d_6$, 300MHz) δ: 15.97 (s, 1H, enolic OH), 12.12 (s, 1H, 2'-OH), 4.78 (s, 1H, 4'-OH), 8.43 (s, 1H, -CH=), 7.19 (d, 2H, $J_{2"-4"}$= 8.6 Hz, 2"-H, 4"-H), 7.11(d, 2H, $J_{3"-5"}$= 8.6 Hz, 3"-H, 5"-H), 6.42-7.39 (m, 2H, Ar-H), 6.41 (s, 3'-H, Ar-H); 2.45 (s, 3H, 4"-$CH_3$); $^{13}$C NMR (DMSO-$d_6$, 300MHz) δ: 189.6 (s, C-1, C=O), 184.5 (s, C-3), 94.2 (s, C-2, -CH=), 116.3 (s, C-1'), 164.3 (s, C-2'), 104.6 (s, C-3'), 165.7 (s, C-4'), 109.6 (s, C-5'), 131.6 (s, C-6'), 127.4 (s, C-1"), 126.8 (s, C-2", C-6"), 129.2 (s, C-3", C-5"), 137.5 (s, C-4"), 24.4 (s, $CH_3$ of C-7"); MS (EI, 70eV): m/z (%) 270 ($M^+$, 100), 224 (24), 105 (59), 51 (19). Elemental analysis: C, 71.21, H, 5.33.

## RESULTS AND DISCUSSION

The 2-aroyloxy-4-hydroxyacetophenones **2a-h** were prepared by the esterification of resacetophenone **1** with aromatic carboxylic acids in the presence of $POCl_3$ (**Scheme-1**). 1-(2', 4'-Dihydroxyphenyl)-3-aryl-propane-1, 3-diones **3a-h** were prepared by Baker-Venkataraman Transformation (intramolecular Claisen condensation) of **2** with NaOH in DMSO. The $^{1}$H

NMR spectrum of **3a** exhibited a singlet at δ 15.72 ppm due to enolic proton (since enol form in β-diketone is more stable), a singlet at δ 12.02 ppm due to phenolic proton adjacent to the carbonyl group and a singlet at δ 4.76 ppm corresponds to the phenolic proton away from carbonyl group. $^{13}$C NMR spectra gives singlet at δ 189.9 ppm due to ketonic carbon C-1 and at δ 184.9 ppm due to enolic carbon C-3 confirming the keto-enol tautomerism in β-diketone **3a**. The IR spectrum showed absorption bands at 3420 (-OH), 1743 (C=O) and 1142 cm$^{-1}$(C-O). The negative test for ester, the presence of characteristic $^{1}$H NMR peaks and $^{13}$C NMR peaks are consistent with the structure of 1-(2', 4'-dihydroxyphenyl)-3-phenyl-propane-1, 3-dione **3a**. The FAB-MS spectrum showed a molecular ion peak at 256 ($M^+$), confirms the molecular formula $C_{15}H_{12}O_4$.




**Scheme-1: Synthesis of 1-(2', 4'-Dihydroxyphenyl)-3-aryl-propane-1, 3-diones; (i) Glacial $CH_3COOH$/anhydrous $ZnCl_2$ (ii) ArCOOH/Pyridine/$POCl_3$, (iii) NaOH/DMSO.**

**Antibacterial activity**

The synthesized compounds **3a-h** were screened for antibacterial activities against bacteria such as *E. coli*, *P. vulgaris*, *S. aureus*, *B. substilis* and *P. aeruginosa* using well diffusion method at concentration of 100µg/mL in acetone. The pure gentamycin, erythromycin and nystatin were used as control. The cultures of above bacterial strains were inoculated in 10 mL nutrient broth and incubated at 37$^{O}$C for 24 hrs. The Petridishes and nutrient agar medium was sterilized by autoclaving. To this sterilized nutrient medium 1 mL of one day old bacterial culture was added

and stirred well; this medium was poured into petridishes. The well impregnated with 100µg/mL of newly synthesized compounds were introduced aseptically in the nutrient agar plate. All the nutrient agar plates were incubated at $37^{O}C$ for 24 hrs after which the plates were observed for clear zone of inhibition. The screening results indicate that the compounds **3a-h** showed moderate to excellent antibacterial activities (**Table-1**).

**Table 1 — Antibacterial activity of compounds 3a-h**

| Sr. No. | Minimum Inhibitory Concentration (MIC) µgmL$^{-1}$ Diameter of Inhibition zone (in mm) | | | | |
|---|---|---|---|---|---|
| | Gram-negative | | | Gram- positive | |
| | *E. coli* | *P.aeruginosa* | *P.vulgaris* | *B. substilis* | *S. aureus* |
| **3a** | 17 | 12 | 13.6 | 10 | 20 |
| **3b** | 14 | 03 | 10 | 08 | -- |
| **3c** | 07 | 22 | 01 | 16 | 13 |
| **3d** | 07 | 03 | -- | 12 | 06 |
| **3e** | 06 | -- | 08 | 06 | -- |
| **3f** | 14 | 10 | 12 | 15 | 24 |
| **3g** | 04 | 19 | 07 | -- | -- |
| **3h** | 17 | 01 | 09 | 08 | 16 |
| **Std 1** | 28 | 22 | 10 | 16 | 10 |
| **Std 2** | 31 | 19 | 08 | 21 | 07 |
| **Std 3** | 23 | 21 | 17 | 06 | 11 |

Minimum Inhibitory Concentration (MIC) 100 µgmL$^{-1}$
**Std 1** Gentamycin
**Std 2** Erythromycin
**Std 3** Nystatin
-- No zone of inhibition

## CONCLUSION

In the present work different 1-(2', 4'-dihydroxyphenyl)-3-aryl-propane-1, 3-diones **3a-h** were synthesized by Baker-Venkataraman Transformation with NaOH in DMSO and their structures elucidated on the basis of spectral analysis. The $^{1}H$ NMR and $^{13}C$ NMR spectra revealed that the prepared compounds **3a-h** possess characteristic peaks due to the presence of enolic proton (enol form of β-diketone) and phenolic proton adjacent to carbonyl group. These synthesized compounds were screened for in vitro antibacterial activity and found to be promising candidates as new antibacterial agents.

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