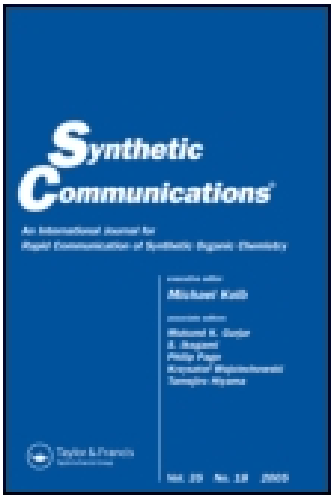

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3-(Substituted Aryl)-1-(benzofuran-2-yl)-2-propenones, Part 1: Synthesis and Characterization of Some Novel Chalcones

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3-(Substituted Aryl)-1-(benzofuran-2-yl)-2-propenones, Part 1: Synthesis and Characterization of Some Novel Chalcones

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Abstract: Synthesis and characterization of some novel chalcones by condensation of 2-acetylbenzofurane and various aromatic aldehydes is described.

Keywords: Aromatic aldehydes, benzofuranes, chalcones

INTRODUCTION

A chalcone group as a classical photosensitive unit has been well studied and used in photocrosslinkable polymers because it affords high sensitivity to UV radiation and chemical resistance of the resultant polymers.[1] Chalcone derivatives are materials noted for their excellent blue-light transmittance and good crystallizability, and chalcones provide a necessary configuration to show nonlinear optical property with planar rings connected through a conjugated double bond.[2,3] Many studies have been reported on the biological activity of chalcone derivatives.[4–7]

The most widely used of the many methods available for synthesis of chalcones is the base-catalyzed Claisen–Schmidt reaction in which the condensation of a ketone with an aldehyde is carried out in the presence of potassium hydroxide,[8] sodium hydroxide,[9–11] barium hydroxide,[12]

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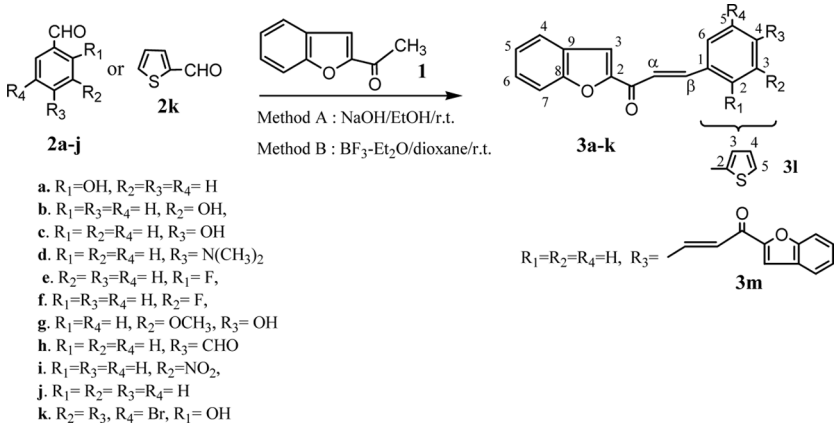


Scheme 1. Scheme of chalcones.

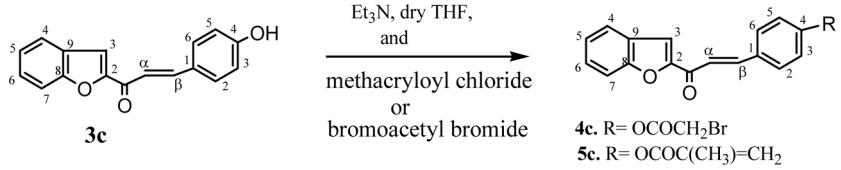
and LiHDMS.[13] Some methods for the synthesis of chalcones include the use of a Lewis acid such as BF_3-Et_2O,[14] $TiCl_4$,[15] or $RuCl_3$.[16]

We have synthesized some novel chalcones **3a–m** by condensation of 2-acetylbenzofurane and various aromatic aldehydes.

RESULTS AND DISCUSSION

This work is a part of the studies done on 3-(substituted aryl)-1-benzofuranyl-2-propenones and describes the synthesis and characterization of some new chalcones by condensation of 2-acetylbenzofurane and 13 aromatic aldehydes. These aldehydes are 2-hydroxybenzaldehyde **2a**, 3-hydroxybenzaldehyde **2b**, 4-hydroxybenzaldehyde **2c**, 4-(dimethylamino)benzaldehyde **2d**, 2-fluorobenzaldehyde **2e**, 3-fluorobenzaldehyde **2f**, 3-hydroxy-2-methoxybenzaldehyde **2g**, benzene-1,4-dicarboxaldehyde (terephthalaldehyde) **2h** (monofunctional), 2-nitrobenzaldehyde **2i**, benzaldehyde **2j**, 5-bromosalicylaldehyde **2k**, thiophene-2-carboxaldehyde **2l**, and benzene-1,4-dicarboxaldehyde **2m** (difunctional) (Scheme 1).

For the synthesis of chalcones, the most common route is the base-catalyzed Claisen–Schmidt reaction involving condensation of a benzaldehyde derivative with an acetophenone derivative in ethanol with sodium hydroxide catalyst[9–11] (method A). Another route is a new described methodology based on the Claisen–Schmidt reaction in the presence of BF_3-Et_2O[14] (method B). Although method B was used in preparing chalcones **3c**, **h**, and **g**, the synthesis of chalcone derivatives **3a**, **b**, **d–f**, and **i–m** was carried out according to procedures in method A.



Scheme 2. Acylation of **3c**.

All the chalcones, except **3h** and **i**, were purified by recrystallization from ethanol. Chalcones **3h** and **i** were crystallized from a mixture of N,N-dimethylformamide (DMF) and ethanol (1:3, v:v). The acylation of **3c** with bromoacetyl bromide and methacryloyl chloride gave **4c** and **5c**, respectively (Scheme 2). Chalcone derivative **4c** can be used as an initiator of atom transfer radical polymerization (ATRP)[17] and gives a polymer with a chalcone end group. Chalcone derivative **5c** is a methacrylate monomer with a chalcone side group, and it can be used in synthesis of polymers with a chalcone side group. Our research group continues to study the syntheses of the polymers involving chalcone.

All new compounds were characterized by ^{1}H NMR, Fourier transform infrared (FTIR), elemental analysis, and (for some compounds) ^{13}C NMR. Although the starting material 2-acetylbenzofurane showed a carbonyl stretching vibration at 1675 cm^{-1} in the FTIR spectrum, the chalcones showed the characteristic bands between 1639 and 1662 cm^{-1} (C=O stretching in chalcone) and between 1605 and 1615 cm^{-1} (C=C stretching in chalcone) in wave numbers changing according to chalcone structure. The most characteristic signals in ^{1}H NMR spectra of the chalcones were observed at 8.08–8.43 ppm (3b-H in benzofurane of chalcone) and at 7.60–8.00 ppm (α- and β-H of chalcone, resonance of β-H is at a lower field than that of α-H) with a coupling constant about 15 Hz, which characterizes the trans-configuration.

EXPERIMENTAL

Melting points were measured using a differential scanning calorimeter (Shimadzu DSC-50) and are uncorrected. Elemental analyses were performed on a Leco CHNS-932 apparatus. ^{1}H NMR spectra were determined on a Bruker AC 300 (300-MHz) spectrometer, with tetramethylsilane (TMS) as the internal standard in the solvents shown, and ^{13}C NMR spectra were recorded on a Bruker (75.47-MHz) spectrometer. Infrared (IR) spectra were recorded as KBr pellets on a Perkin-Elmer Spectrum One FTIR spectrometer.

Preparation of Chalcones

Method A

A solution of 2-acetylbenzofurane **1** (2.24 g, 14.0 mmol) and the aldehyde **2a**, **b**, **d–f**, and **i–k** (14.0 mmol) in methanol (25 mL) was cooled to 0–5 °C, and then 18 mL of aqueous NaOH (1 mol/L) was added to this solution and stirred at room temperature for 3 h. The solution was allowed to stand in the refrigerator overnight. The solid precipitated upon the addition of water, was filtered after neutralization with diluted HCl, and was washed several times with water. The solid was recrystallized from an appropriate solvent to provide the chalcone.

Method B

This method was adapted from the literature.[14] To a stirred solution of 2-acetylbenzofurane **1** (4.0 g, 25 mmol) and the aldehyde **2c**, **h**, and **g** (25 mmol) in a little dry dioxane, BF_3-Et_2O (1.5 mL, 12.5 mmol) was added gradually at room temperature. The solution was stirred for 5 h at 35 °C. The solid product was precipitated by addition to excess water of the solution, and the precipitate was filtered off. The product was washed a few times with ether and dried in a vacuum oven at 50 °C for 24 h.

Data

3-(2-Hydroxyphenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3a**)

Compound **3a** was synthesized according to method A and crystallized from ethanol. Yield: 2.03 g, 55%; mp193 °C; FTIR (KBr, cm^{-1}): 3236, 3000–3100, 1639, 1614, 1602, 1575, 1250, 1141, 947, 764, 750; ^1H NMR (300 MHz, $CDCl_3$): δ 9.03 (s, 1H, OH), 8.08 (d, $J = 8.37$ Hz, 1H, b4-H), 7.72 (d, $J = 7.20$ Hz, 1H, b7-H), 7.22–7.68 (m, 9H, all the others); ^{13}C NMR: δ 179.68 (C = O), 155.90 (b8-C), 155.62 (b2-C), 152.53 (2-C), 141.91 (β-C), 132.01 (b9-C), 110.48–128.11 (the other Cs). Anal. calcd. for $C_{17}H_{12}O_3$: C, 77.29; H, 4.54. Found: C, 77.43; H, 4.35.

3-(3-Hydroxyphenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3b**)

Compound **3b** was synthesized according to method A and crystallized from ethanol. Yield: 2.15 g, 58%; mp 176 °C; FTIR (KBr, cm^{-1}): 3226,

3000–3100, 1639, 1646, 1612, 1582, 1229, 1145, 973, 853, 785, 679; ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.70 (broad, OH), 8.31 (s, 1H, b3-H), 7.87 (d, $J = 7.76$ Hz, 1H, b4-H), 7.82 (d, $J = 15.69$ Hz, 1H, β-H), 7.76 (d, $J = 7.90$ Hz, 1H, b7-H), 7.75 (d, $J = 15.72$ Hz, 1H, α-H), 7.57 (dd, $J = 7.80$ Hz and 7.36 Hz, 1H, b6-H), 7.40 (dd, $J = 7.75$ Hz and 7.40 Hz, 1H, b5-H), 6.91 (d, $J = 7.20$ Hz, 1H, 4-H), 7.27–7.33 (m, 3H, 2,5, 6-H). Anal. calcd. for $C_{17}H_{12}O_3$: C, 77.29; H, 4.54. Found: C, 77.54; H, 4.54.

3-(4-Hydroxyphenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3c**)

Compound **3c** was synthesized according to method B and purified by washing with hot ethanol. Yield: 3.10 g, 47%; mp 254 °C; FTIR (KBr, cm^{-1}): 3246, 3000–3100, 1643, 1609, 1575, 1547, 1516, 1285, 1164, 979, 829, 759; ^{1}H NMR (300 MHz, DMSO-d_6): δ 10.17 (s,1H, OH), 8.21 (s, 1H, b3-H), 7.86 (d, $J = 7.70$ Hz, 1H, b4-H), 7.70–7.80 (m, 4H, 2, 6, b7, β-H), 7.67 (d, $J = 15.10$ Hz, 1H, α-H), 7.54 (dd, $J = 7.75$ Hz and 7.50 Hz, 1H, b6-H), 7.38 (dd, $J = 7.57$ and 7.47 Hz, 1H, b5-H), 6.89 (d, $J = 6.60$ H, 2H, 3, 5-H); ^{13}C NMR: δ 178.94 (C=O), 160.91 (b8-C), 155.66 (4-C), 154.00 (b2-C), 144.48 (β-C), 132.58 (b9-C), 131.67 (2, 6-C), 127.84 (1-C), 127.54 (b6-C), 125.97 (b5-C), 124.50 (α-C), 124.10 (b4-C), 118.76 (b3-C), 116.39 (3,5-C), 112.70 (b7-C). Anal. calcd. for $C_{17}H_{12}O_3$: C, 77.29; H, 4.54. Found: C, 77.28; H, 4.40.

3-(4-N,N-dimethylaminophenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3d**)

Compound **3d** was synthesized according to method A and crystallized from ethanol. Yield: 3.26 g, 80%; mp 150 °C; FTIR (KBr, cm^{-1}): 3000–3100, 1643, 1611, 1569, 1544, 1522, 1375, 1359, 1267, 1152, 804, 746; ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.16 (s, 1H, b3-H), 7.86 (d, $J = 7.74$ Hz, 1H, b4-C), 7.76 (d, $J = 15.50$ Hz, 1H, β-H), 7.75 (d, $J = 6.80$ Hz, 1H, b7-H), 7.73 (d, $J = 8.83$ Hz, 2H, 2, 6-H), 7.60 (d, $J = 15.51$ Hz, 1H, α-H), 7.54 (dd, $J = 7.80$ and 7.03 Hz, 1H, b6-H), 7.39 (dd, $J = 7.55$ and 7.20 Hz, 1H, b5-H), 6.78 (d, $J = 8.91$ Hz, 2H, 3, 5-H), 3.03 (s, 6H, N,N-dimethyl). Anal. calcd. for $C_{19}H_{17}O_2N$: C, 78.37; H, 5.84; N, 4.81. Found: C, 77.93; H, 5.56; N, 4.42.

3-(2-Fluorophenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3e**)

Compound **3e** was synthesized according to method A and crystallized from ethanol. Yield: 2.02 g, 54%; mp 100 °C; FTIR (KBr, cm^{-1}): 3120,

3067, 1659, 1611, 1576, 1550, 1292, 1160, 753; ^{1}H-NMR (300 MHz, DMSO-d_6): δ 8.30 (s, 1H, b3-H), 8.11 (dd, $J = 7.60$ and 7.20 Hz, 1H, b6-H), 7.93 (d, $J = 15.87$ Hz, 1H, β-H), 7.90 (dd, $J = 8.37$ and 7.55 Hz, 1H, b5-H), 7.89 (d, $J = 15.87$ Hz, 1H, α-H), 7.76 (d, $J = 8.41$ Hz, 1H, b4-H), 7.29–7.61 (m, 5 H, b7, 3, 4, 5, 6-H). Anal. calcd. for $C_{17}H_{11}O_2F$: C, 76.71; H, 4.13. Found: C, 76.45; H, 4.05.

3-(3-Fluorophenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3f**)

Compound **3f** was synthesized according to method A and crystallized from ethanol. Yield: 1.75 g, 47%; mp 99 °C; FTIR (KBr, cm^{-1}): 3118, 3073, 1662, 1611, 1582, 1545, 1278, 1156, 981, 850, 753; ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.39 (s, 1H, b3-H), 7.97 (d, $J = 15.60$ Hz, 1H, β-H), 7.70–7.90 (m, 3H, b4, b6, α-H), 7.35–7.63 (m, 6 H, b5, b6, 2, 4, 5, 6-H). Anal. calcd. for $C_{17}H_{11}O_2F$: C, 76.71; H, 4.13. Found: C, 76.57; H, 4.18.

3-(3-Methoxy-4-hydroxyphenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3g**)

Compound **3g** was synthesized according to method B and crystallized from ethanol. Yield: 3.67 g, 50%; mp177 °C; FTIR (KBr, cm^{-1}): 3250, 3000–3100, 1653, 1611, 1580, 1570, 1272, 1239, 1163, 112, 978, 807, 758; ^{1}H NMR (300 MHz, DMSO-d_6): δ 9.82 (broad, 1H, OH), 8.25 (s, 1H, b3-H), 7.88 (d, $J = 7.60$ Hz, 1H, b4-H), 7.78 (d, $J = 15.60$ Hz, 1H, β-H), 7.70 (d, $J = 15.80$ Hz, 1H, α-H), 7.60 (d, $J = 7.50$ Hz, 1H, b7-H), 7.54 (dd, $J = 7.57$ and 7.48, 1H, b6-H), 7.53 (s, 1H, 2-H), 7.39 (t, $J = 7.60$ Hz, 1H, b5-H), 7.33 (dd, $J = 8.10$ and 1.8 Hz, 1H, 6-H), 6.87 (d, $J = 8.14$ Hz, 1H, 5-H), 3.90 (s, 3H, CH_3). Anal. calcd. for $C_{18}H_{14}O_4$: C, 77.27; H, 4.55. Found: C, 76.95; H, 4.35.

3-(4-Methanoylphenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3h**)

Compound **3h** was synthesized according to method B and crystallized from a mixture of N,N-dimethylformamide (DMF) and ethanol (1:3, v:v). Yield: 2.76 g, 40%; mp164 °C; FTIR (KBr, cm^{-1}): 3000–3115, 2848, 2752, 1695, 1662, 1609, 1568, 1548, 1171, 1042, 979, 816, 744; ^{1}H NMR (300 MHz, DMSO-d_6): δ 10.07 (s, 1H, CHO), 8.40 (s, 1H, b3-H), 8.13 (d, $J = 8.22$ Hz, 2H, 3, 5-H), 8.04 (d, $J = 15.6$ Hz, 1H, β-H), 8.01 (d, $J = 8.27$ Hz, 2H, 2, 6-H), 7.92 (d, $J = 7.53$, 1H, b4-H), 7.89 (d, $J = 15.60$ Hz, 1H, α-H), 7.77 (d, $J = 8.41$ Hz, 1H, b7-H), 7.59 (dd,

$J = 8.30$ and 7.30 Hz, 1H, b6-H), 7.43 (dd, $J = 7.53$ and 7.50 Hz, 1H, b5-H). Anal. calcd. for $C_{18}H_{12}O_3$: C, 78.28; H, 4.35. Found: C, 79.03; H, 4.28.

3-(3-Nitrophenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3i**)

Compound **3i** was synthesized according to method A and crystallized from a mixture of DMF and ethanol (1:3, v:v). Yield: 2.13 g, 52%; mp 189 °C; FTIR (KBr, cm^{-1}): 3000–3120, 1669, 1615, 1549, 1521, 1358, 1282, 1165, 1040, 976, 812, 740; ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.79 (s, 1H, 2-H), 8.43 (s, 1H, b3-H), 8.33 (d, $J = 7.74$ Hz, 1H, 4-H), 8.27 (dd, $J = 7.41$ and 1.52 Hz, 1H, 6-H), 8.08 (d, $J = 15.77$ Hz, 1H, β-H), 7.92 (d, $J = 15.90$ Hz, 1H, α-H), 7.90 (d, $J = 7.50$ Hz, 1H, b4-H), 7.74–7.79 (m, 2H, 5, b7-H), 7.58 (dd, $J = 7.60$ and 7.62 Hz, 1H, b6-H), 7.40 (dd, $J = 7.51$ and 7.48 Hz, 1H, b5-H). Anal. calcd. for $C_{17}H_{11}O_4N$: C, 69.64; H, 3.75; N, 4.78. Found: C, 70.03; H, 3.59; N, 4.65.

3-Phenyl-1-(benzofurane-2-yl)-2-propen-1-one (**3j**)

Compound **3j** was synthesized according to method A and crystallized from ethanol. Yield: 2.60 g, 75%; mp 121 °C; FTIR (KBr, cm^{-1}): 3000–3100, 1657, 1605, 1595, 1575, 1553, 1159, 1138, 1044, 937, 752, 681; ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.34 (s, 1H, b3-H), 7.88–7.96 (m, 4H, 2, β, b4-H), 7.83 (d, $J = 15.6$ Hz, 1H, α-H), 7.77 (dd, $J = 7.40$ and 1.0 Hz, 1H, b7-H), 7.58 (dt, $J = 7.20$ and 1.2 Hz, 1H, b6-H), 7.46–7.52 (m, 3H, 3, b5-H), 7.40 (dt, $J = 7.50$ and 1.0 Hz, 1H, 4-H). Anal. calcd. for $C_{17}H_{12}O_2$: C, 82.26; H, 4.84. Found: C, 81.35; H, 5.58.

3-(2-Hydroxy-5-bromophenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**3k**)

Compound **3k** was synthesized according to method A and crystallized from ethanol. Yield: 1.92 g, 40%; mp 208 °C; FTIR (KBr, cm^{-1}): 3200, 3000–3100, 1646, 1615, 1594, 1546, 1294, 1163, 1050, 981, 819, 738; ^{1}H NMR (300 MHz, DMSO-d_6): δ 10.69 (s, broad, 1H, OH), 8.34 (s, 1H, b3-H), 8.16 (d, $J = 2.44$ Hz, 1H, 6-H), 8.03 (d, $J = 15.87$ Hz, 1H, β-H), 7.92 (d, $J = 15.94$ Hz, 1H, α-H), 7.89 (d, $J = 7.40$ Hz, 1H, b4-H), 7.76 (d, $J = 8.40$ Hz, 1H, b7-H), 7.58 (dd, $J = 8.32$ and 7.26 Hz, 1H, b6-H), 7.44 (dd, $J = 8.54$ and 2.45 Hz, 1H, b5-H), 7.41 (dd, $J = 7.50$ and 7.20 Hz), 6.92 (d, $J = 8.74$ Hz, 1H, 6-H). Anal. calcd. for $C_{17}H_{11}O_3Br$: C, 59.48; H, 3.21. Found: C, 61.25; H, 3.70.

3-(Thiophene-2-yl)-1-(benzofurane-2-yl)-2-propen-1-one (**3l**)

Compound **3l** was synthesized according to method A and crystallized from ethanol. Yield: 2.52 g, 71%; mp 140 °C; FTIR (KBr, cm^{-1}): 300–3100, 1656, 1592, 1557, 1292, 1157, 1139, 995, 937, 823, 744, 707, 603; ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.21 (s, 1H, b3-H), 8.01 (d, $J = 15.40$ Hz, 1H, β-H), 7.86 (d, $J = 7.51$ Hz, 1H, b4-H), 7.83 (d, $J = 5.14$ Hz, 1H, 3-H), 7.75 (dd, $J = 8.40$ and 0.74 Hz, 1H, b7-H), 7.72 (d, $J = 3.56$ Hz, 1H, 5-H), 7.56 (ddd, $J = 8.32$, 7.35, and 1.11 Hz, 1H, b6-H), 7.52 (d, $J = 15.42$ Hz, 1H, α-H), 7.39 (dt, $J = 7.52$ and 0.74 Hz), 7.22 (dd, $J = 5.01$ and 3.67 Hz, 1H, 4-H). Anal. calcd. for $C_{19}H_{17}O_2N$: C, 70.86; H, 3.93; S, 12.62. Found: C, 71.28; H, 4.06; S, 12.18.

Benzene-1,4-bis[1-(2-benzofuranyl)-3-propenyl-1-one] (**3m**)

Compound **3m** was synthesized according to method A using 28 mmol of 2-acetylbenzofurane and 36 mL of aqueous NaOH (1 mol/L) per 14 mmol of benzene-1,4-dicarboxaldehyde **2h** and purified by washing with water, a mixture of DMF and ethanol (3:1, v:v), and chlorform, respectively. Yield: 6.44 g, 55%; mp 286 °C; FTIR (KBr, cm^{-1}): 3106, 3055, 1661, 1608, 1544, 1182, 1048, 981, 821, 732; ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.40 (s, 2H, b3-H), 8.06 (s, 4H, 2, 3, 5, 6-H), 8.02 (d, $J = 15.23$ Hz, 2H, β-H), 7.93 (d, $J = 8.17$ Hz, 2H, b4-H), 7.88 (d, $J = 15.70$ Hz, 2H, α-H),7.79 (d, $J = 8.10$ Hz, 2H, b7-H), 7.59 (dd, $J = 7.65$ and 7.45 Hz, 2H, b6-H), 7.43 (dd, $J = 7.65$ and 7.32 Hz, 2H, b5-H). Anal. calcd. for $C_{28}H_{18}O_4$: C, 80.38; H, 4.31; Found: C, 81.28; H, 4.05.

3-[4-(α-Bromoacetoxy)phenyl]-2-(benzofurane-2-yl)-2-propen-1-one (**4c**)

Compound **3c** (0.70 g, 2.65 mmol) was dissolved in 15 mL of dry THF, and triethylamine (0.39 mL, 2.70 mmol) was added. α-Bromoacetyl bromide (0.55 g, 2.70 mmol) was added dropwise to the mixture and cooled to 0 °C. After stirring overnight at room temperature, water (50 mL) was added, and the precipitate was crystallized from ethanol. Yield: 0.36 g, 35%; mp 130 °C; FTIR (KBr, cm^{-1}): 3000–3114, 1763, 1663, 1609, 1584, 1550, 1505, 1166, 1135, 982, 833, 756; ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.33 (s, 1H, b3-H), 8.01 (d, $J = 8.66$ Hz, 2H, 2, 6-H), 7.90 (d, $J = 15.67$ Hz, 1H, β-H), 7.87 (d, $J = 7.70$ Hz, 1H, b4-H), 7.84 (d, $J = 15.90$ Hz, 1H, α-H), 7.76 (d, $J = 8.30$ Hz, 1H, b7-H), 7.58 (dd, $J = 8.10$ and 7.20 Hz, 1H, b6-H), 7.40 (dd, $J = 7.51$ and 7.49 Hz, 1H, b5-H), 7.31 (d, $J = 8.62$ Hz, 2H, 3,5-H), 4.49 (s, 2H, CH_2Br). Anal. calcd. for $C_{19}H_{13}O_4Br$: C, 59.22; H, 3.38; Found: C, 60.08; H, 3.55.

3-(4-Methacryloyloxyphenyl)-1-(benzofurane-2-yl)-2-propen-1-one (**5c**)

Compound **3c** (0.60 g, 2.27 mmol) was dissolved in 15 mL of dry THF, and triethylamine (0.4 mL, 2.79 mmol) was added. Methacryloyl chloride (0.29 g, 2.79 mmol) was added dropwise to the mixture cooled to 0 °C. After stirring overnight at room temperature water (50 mL) was added, and the precipitate was crystallized from ethanol. Yield: 0.38 g, 51%; mp 160 °C; FTIR (KBr, cm^{-1}): 3000–3119, 1725, 1659, 1607, 1597, 1548, 1341, 1212, 1167, 978, 880, 809, 745; ^1H NMR (300 MHz, DMSO-d_6): δ 8.32 (s, 1H, b3-H), 7.98 (d, $J = 8.61$ Hz, 2H, 2,6-H), 7.91 (d, $J = 15.72$ Hz, 1H, β-H), 7.88 (d, $J = 7.90$ Hz, 1H, b4-H), 7.83 (d, $J = 15.76$ Hz, 1H, α-H), 7.78 (d, $J = 8.55$ Hz, 1H, b7-H), 7.57 (dd, $J = 8.41$ and 7.18 Hz, 1H, b6-H), 7.40 (dd, $J = 7.54$ and 7.51 Hz, 1H, b5-H), 7.32 (d, $J = 8.57$ Hz, 2H, 3, 5-H), 6.31 (d, $J = 1.2$ Hz, 1H, =C-H, trans to methyl), 5.93 (d, $J = 1.2$ Hz, = C-H, cis to methyl), 2.02 (s, 3H, CH_3). Anal. calcd. for $C_{21}H_{16}O_4$: C, 75.90; H, 4.82. Found: C, 76.25; H, 4.75.

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