Green synthesis of *trans*-2-(4-chlorobenzoyl)-5-hydroxy-3-(aryl)-2,3-dihydrobenzofuran-4,7-diones

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A green and efficient synthesis of *trans*-2-(4-chlorobenzoyl)-5-hydroxy-3-(aryl)-2,3-dihydrobenzofuran-4,7-diones has been achieved in excellent yield *via* a three-component one-pot condensation of 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide with 2,5-dihydroxy-1,4-benzoquinone and aromatic aldehydes in the presence of catalytic amounts of choline hydroxide in water under reflux conditions. The salient features of this protocol are the short reaction time, the high yields and it does not involve any hazardous organic solvent.

Keywords: 2,5-dihydroxy-1,4-benzoquinone, green chemistry, dihydrobenzofuran-4,7-dione, aromatic aldehydes, 4-chlorophenacyl bromide, choline hydroxide

Dihydrofurans are important heterocycles which are commonly found in a variety of naturally-occurring substances.[1,2] The development of new and efficient methods for their synthesis remains an area of current interest and a series of new synthetic methods have appeared in literature.[3–16] Amongst these synthetic methodologies, both non-ionic and ionic procedures have been developed. Radical[2] or carbenoid[7–9] additions to olefins have been utilised as non-ionic procedures. Amongst the ionic reaction conditions which have been reported were syntheses *via* tandem nucleophilic reactions of 1,3-dicarbonyl compounds[10–12] or the reaction of ylides[13–16] with enones. The methods reported previously for the synthesis of dihydrofurans suffer from serious disadvantages such as hazardous solvents, longer reaction time, inadequate yields and use of expensive non-recoverable surfactant. Consequently we have developed a synthetic strategy using choline hydroxide as a green catalyst in a new rapid method to afford dihydrofurans in excellent yield.

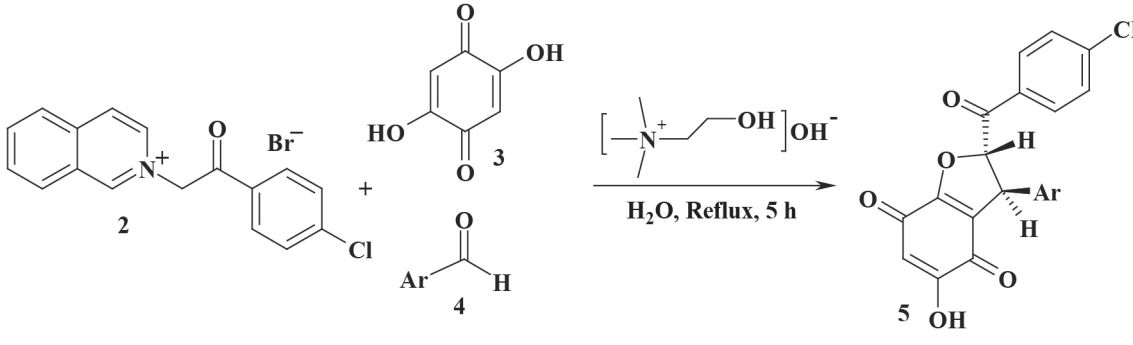
Recently, organic reactions in aqueous media have attracted a great deal of attention[17] as a result of increasing interest in the concepts of sustainability and green chemistry.[18] In continuation of our previous work for the synthesis of heterocyclic compounds,[19–21] we decided to investigate the reaction of 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** with 2,5-dihydroxy-1,4-benzoquinone **3** and an aromatic aldehyde **4** in presence of catalytic amounts of choline hydroxide in water under reflux conditions (Scheme 1).

Results and discussion

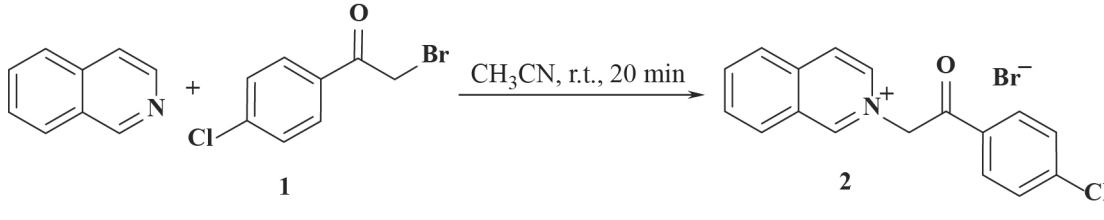
Treatment of isoquinoline with 4-chlorophenacyl bromide **1** in acetonitrile for 20 min yielded the 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** in nearly quantitative yields (Scheme 2).

One-pot three-component reaction of 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** with 2,5-dihydroxy-1,4-benzoquinone **3** and an aromatic aldehyde **4** in the presence of catalytic amounts of choline hydroxide in boiling water gave the corresponding products **5** in excellent yields (Scheme 3).

The structures of compounds **5a–l** were deduced from their elemental analyses and their IR, ^{1}H NMR and ^{13}C NMR spectra. The mass spectra of compounds **5a–l** are fairly similar and



Scheme 1



Scheme 2

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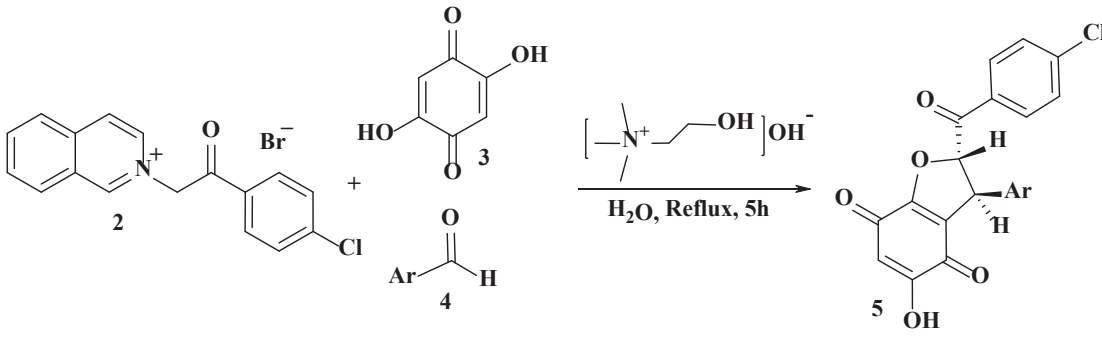
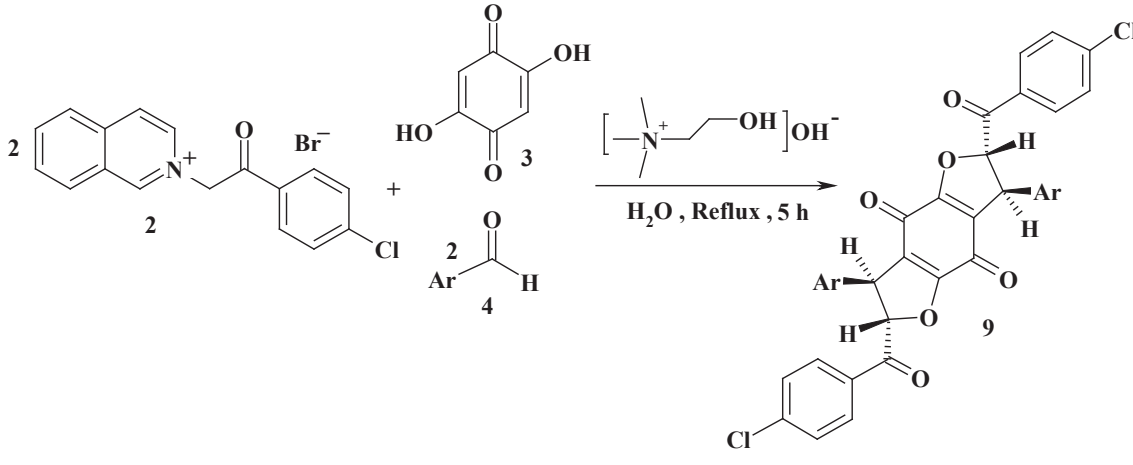


5	Ar	Yield (%)*	m.p. (°C)
a	C_6H_5	93	268–270
b	2-$O_2NC_6H_4$	96	272–274
c	4-$O_2NC_6H_4$	97	276–278
d	2-ClC_6H_4	88	286–288
e	4-ClC_6H_4	91	289–291
f	2-FC_6H_4	94	275–277
g	4-$CH_3OC_6H_4$	90	273–275
h	4-FC_6H_4	94	290–292
i	4-Cl-3-$O_2NC_6H_3$	91	283–285
j	4-NCC_6H_4	92	275–277
k	4-$CH_3C_6H_4$	89	292–294
l	4-BrC_6H_4	92	278–280

*Yields refer to the pure isolated products

Scheme 3



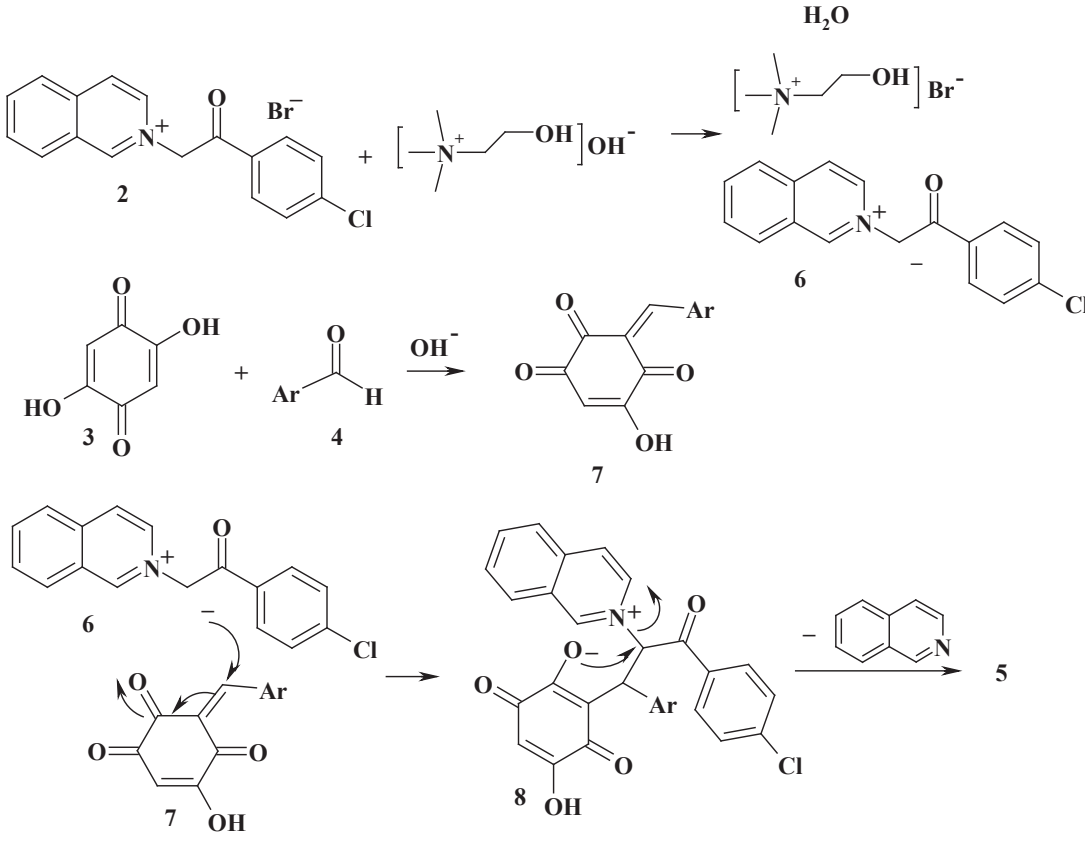
Scheme 4

display the anticipated molecular ion. In the ^{1}H NMR spectra, the two protons at 2,3-positions of the dihydrofuran ring gave two doublet signals at δ 4.36 and 6.08 ppm with the vicinal coupling constant J = 5.2 and 5.2 Hz, respectively. It has been reported that in *cis*-2,3-dihydrofuran the vicinal coupling constant of the two methine protons is J = 7–10 Hz, while in *trans*-2,3-dihydrofuran the vicinal coupling constant has a value of J = 2.8–6 Hz. Therefore we concluded that thermodynamically stable *trans* isomers of the 2,3-dihydrofuran derivatives were formed.[22]

Five-component reaction between 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** (2 equiv.) and aromatic aldehyde **4** (2 equiv.) and 2,5-dihydroxy-1,4-benzoquinone **3** in the presence of catalytic amounts of choline hydroxide in boiling water afforded the addition product **9** in 94% yield (Scheme 4).

A proposed mechanism for this reaction is shown in Scheme 5. The formation of the product can be explained as follows. The 2-[2-(4-chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** undergoes deprotonation in the presence of aqueous choline hydroxide to give the reactive isoquinolinium ylide **6** at room temperature. The 2,5-dihydroxy-1,4-benzoquinone **3** reacts with the aromatic aldehyde **4** in the presence of choline hydroxide to give the Knoevenagel product **7**. This reacts instantly with the isoquinolinium ylide **6** to form the zwitterionic intermediate **8**. The intermediate **8** undergoes cyclisation with the elimination of isoquinoline to give the desired product **5**.

In summary, we report that a simple and efficient one-pot condensation of 2-[2-(4-chlorophenyl)-2-oxoethyl)] isoquinolinium bromide with 2,5-dihydroxy-1,4-benzoquinone and an aromatic aldehyde in the presence of catalytic amounts of choline hydroxide in boiling water gives *trans*-2-(4-



Scheme 5

chlorobenzoyl)-5-hydroxy-3-(aryl)-2,3-dihydrobenzofuran-4,7-dione. The advantages of this method are readily available starting materials, short reaction times, easy and clean work-up and excellent yields.

Experimental

Melting points were determined with an Electrothermal 9100 apparatus. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyser. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionisation potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on Bruker DRX-400 Avance spectrometer in $CDCl_3$ using TMS as and internal standard. 2-[2-(4-Chlorophenyl)-2-oxoethyl)]isoquinolinium bromide **2** was prepared following literature method.[23] Others chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

Synthesis of trans*-2-(4-chlorobenzoyl)-5-hydroxy-3-(aryl)-2,3-dihydrobenzofuran-4,7-diones* (**5a–l**)*; general procedure*

A magnetically-stirred solution of 2-[2-(4-chlorophenyl)-2-oxoethyl)] isoquinolinium bromide (1 mmol), 2,5-dihydroxy-1,4-benzoquinone (1 mmol) and the appropriate aryl aldehyde (1 mmol) in H_2O (10 mL) was treated with choline hydroxide (0.0121 g, 0.1 mmol) in H_2O (4 mL). The mixture was then refluxed for 5 h. The solid product was filtered and recrystallised from ethanol to afford the pure product.

trans*-2-(4-Chlorobenzoyl)-5-hydroxy-3-phenyl-2,3-dihydrobenzofuran-4,7-dione* (**5a**): White powder; m.p. 268–270 °C; IR (ν_{max}, cm^{-1}) KBr: 3189, 2904, 1662, 1629, 1580, 1468, 1419, 1369, 1216, 1025, 818, 789, 667, 604, 503; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.34 (d, *J* = 5.2 Hz, 1H, CH), 6.08 (d, *J* = 5.2 Hz, 1H CH), 6.88 (s, 1H, CH), 7.27–8.06 (m, 9H, ArH), 10.68 (s, 1H, OH); ^{13}C NMR (100.6 MHz, $CDCl_3$: δ 39.1, 84.7, 103.8, 125.9, 127.7, 128.6, 128.7, 130.2, 132, 132.3, 138.7, 140.6, 157.8, 174.3, 181.2, 189.4; MS *m/z* (%): 380 (4); Anal. calcd for $C_{21}H_{13}ClO_5$: C, 66.24; H, 3.44; found: C, 66.38; H, 3.61%.

trans*-2-(4-Chlorobenzoyl)-5-hydroxy-3-(2-nitrophenyl)-2,3-dihydrobenzofuran-4,7-dione* (**5b**): White powder; m.p. 272–274 °C; IR (ν_{max}, cm^{-1}) KBr: 3425, 3380, 3035, 2900, 2220, 1667, 1628, 1570, 1513, 1421, 1370, 847, 823, 778, 742, 667, 576; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.35 (d, *J* = 5.3 Hz, CH, 1H,), 6.10 (d, *J* = 5.3Hz CH, 1H), 6.87 (s, 1H, CH), 7.60–8.09 (m, 8H, ArH), 10.68 (s, 1H, OH); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 37.6, 84.8, 103.2, 124.1, 126.1, 128.3, 128.6, 128.7, 130.2, 132.4, 134.1, 138.7, 148.2, 157.7, 179.5, 182.6, 188.6; MS *m/z* (%): 425 (7); Anal. calcd for $C_{21}H_{12}ClNO_7$: C, 59.24; H, 2.84; N, 3.29; found: C, 59.40; H, 2.72; N, 3.45%.

trans*-2-(4-Chlorobenzoyl)-5-hydroxy-3-(4-nitrophenyl)-2,3-dihydrobenzofuran-4,7-dione* (**5c**): White powder; m.p. 276–278 °C; IR (ν_{max}, cm^{-1}) KBr: 2905, 2630, 1667, 1634, 1572, 1504, 1418, 1331, 1267, 1124, 846, 817, 744, 664, 604; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.39 (d, *J* = 4.8 Hz, CH, 1H), 6.08 (d, *J* = 4.8 Hz, CH, 1H), 6.85 (s, CH, 1H), 7.59–8.17 (m, 8H, ArH), 10.63 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 39.7, 84.4, 103.4, 123.7, 128.6, 128.8, 130.4, 130.5, 132.3. 13.6, 138.5, 145.9, 146.7, 157.4, 179.5, 182.6, 188.4; MS *m/z* (%): 425 (5); Anal. calcd for $C_{21}H_{12}ClNO_7$: C, 59.24; H, 2.84; N, 3.29; found: C, 59.37; H, 2.70; N, 3.41%.

trans*-2-(4-Chlorobenzoyl)-3-(2-chlorophenyl)-5-hydroxy-2,3-dihydrobenzofuran-4,7-dione* (**5d**): White powder; m.p. 286–288 °C; IR (ν_{max}, cm^{-1}) KBr: 3140, 2900, 1668, 1636, 1570, 1420, 1371, 1117, 1013, 817, 755, 667, 604, 574; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.46 (d, *J* = 5.4 Hz, CH, 1H), 5.96 (d, *J* = 5.4 Hz, CH, 1H), 6.83 (s, CH, 1H), 6.85–8.06 (m, 8H, ArH), 10.61 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 35.7, 84.8, 103.7, 126.8, 127.3, 128.6, 129.1, 130.2, 132.3, 133.0, 138.2, 138.8, 157.6, 179.2, 182.6, 188.3; MS *m/z* (%): 414 (6); Anal. calcd for $C_{21}H_{12}Cl_2O_5$: C, 60.75; H, 2.91; found: C, 60.88; H, 2.75%.

trans*-2-(4-Chlorobenzoyl)-3-(4-chlorophenyl)-5-hydroxy-2,3-dihydrobenzofuran-4,7-dione* (**5e**): White powder; m.p. 289–291 °C; IR (ν_{max}, cm^{-1}) KBr: 3140, 2900, 1666, 1632, 1570, 1512, 1420, 1371, 1115, 1016, 817, 667, 604, 516; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.91 (d, *J* = 5.2 Hz, CH, 1H), 6.47 (d, *J* =5.2 Hz, CH, 1H), 6.85 (s, CH,

1H),7.29–8.01 (m, 8H, ArH), 10.62 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 38.7, 90.9, 103.4, 128.8, 129.1, 130.1, 131.5, 132.2, 132.4, 138.2, 157.4, 178.9, 182.4, 188.5; MS *m/z* (%): 414 (3); Anal. calcd for $C_{21}H_{12}Cl_2O_5$: C, 60.75; H, 2.91; found: C, 60.90; H, 2.72%.

trans-*2-(4-Chlorobenzoyl)-3-(2-fluorophenyl)-5-hydroxy-2,3-dihydrobenzofuran-4,7-dione* (**5f**): White powder; m.p. 275–277 °C; IR (ν_{max}, cm^{-1}) KBr: 3443, 3040, 2905, 1667, 1631, 1570, 1513, 1421, 1250, 121, 823, 755, 604; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.10 (d, *J* = 5.2 Hz, CH, 1H), 6.15 (d, *J* = 5.2 Hz, CH, 1H), 6.88 (s, CH, 1H), 7.05–8.12 (m, 8H, ArH), 10.65 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 30.3, 84.7, 103.5, 115.4, 124.2, 127.4, 127.5, 128.6, 129.3, 130.2, 132.3, 138.7, 157.4, 160.4, 179.4, 182.6, 188.3; MS *m/z* (%): 398 (5); Anal. calcd for $C_{21}H_{12}ClFO_5$: C, 63.25; H, 3.03; found: C, 63.41; H, 3.18%.

trans-*2-(4-Chlorobenzoyl)-5-hydroxy-3-(4-methoxyphenyl)-2,3-dihydrobenzofuran-4,7-dione* (**5g**): White powder; m.p. 273–275 °C; IR (ν_{max}, cm^{-1}) KBr: 3445, 2905, 2845, 1669, 1634, 1572, 1507, 1418, 1254, 1201, 1175; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.82 (s, 3H, OCH_3), 4.37 (d, *J* =4.8 Hz, 1H), 6.10 (d, *J* = 4.8 Hz, 1H) 6.83 (s, CH, 1H), 6.86–8.10 (m, 8H, ArH), 10.62 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 40.8, 55.8, 83.9, 103.4, 114.3, 128.6, 128.7, 130.2, 132.4, 132.9, 138.7, 157.8, 179.2, 182.5, 188.6; MS *m/z* (%): 410 (10); Anal. calcd for $C_{22}H_{15}ClO_6$: C, 64.32; H, 3.68; found: C, 64.50; H, 3.81%.

trans-*2-(4-Chlorobenzoyl)-3-(4-fluorophenyl)-5-hydroxy-2,3-dihydrobenzofuran-4,7-dione* (**5h**): White powder; m.p. 290–292 °C; IR (ν_{max}, cm^{-1}) KBr: 3452, 2904, 2851, 1667, 1634, 1585, 1523, 1425, 1272, 1017, 816, 667, 534; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.25 (d, *J* = 5.0 Hz, CH, 1H), 6.12 (d, *J* =5.0 Hz, CH, 1H,), 6.84 (s, CH, 1H), 7.16–8.06 (m, 16H, ArH), 10.68 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 41.9, 82.3, 103.4, 115.4, 128.7, 129.3, 130.2, 132.1, 132.3, 136.5, 138.7, 157.6, 160.3, 179.6, 182.5, 188.3; MS *m/z* (%): 398 (6); Anal. calcd for $C_{21}H_{12}ClFO_5$: C, 63.25; H, 3.03; found: C, 63.38; H, 3.16%.

trans-*3-(4-chloro-3-nitrophenyl)-2-(4-chlorobenzoyl)-5-hydroxy-2,3-dihydrobenzofuran-4,7-dione* (**5i**): White powder; m.p. 283–285 °C; IR (ν_{max}, cm^{-1}) KBr: 3442, 3130, 2910, 1665, 1628, 1571, 1523, 1445, 1422, 819, 771, 745, 665, 604; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.40 (d, *J* = 4.8 Hz, CH, 1H,), 6.13 (d, *J* = 4.8 Hz CH, 1H), 6.81 (s, CH, 1H), 7.58–8.33 (m, 7H, ArH), 10.34 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 40.4, 91.8, 103.1, 125.1, 125.5, 128.6, 130.3, 132.2, 132.7, 135.2, 138.7, 139.4, 147.3, 157.4, 178.9, 182.5, 188.8; MS *m/z* (%): 459 (7); Anal. calcd for $C_{21}H_{11}Cl_2NO_7$: C, 54.81; H, 2.41; N, 3.04; found: C, 54.65; H, 2.55; N, 3.17%.

trans-*2-(4-Chlorobenzoyl)-5-hydroxy-4,7-dioxo-2,3,4,7-tetrahydrobenzofuran-3-yl)benzonitrile* (**5j**): White powder; m.p. 275–277 °C; IR (ν_{max}, cm^{-1}) KBr: 3445, 3185, 2906, 1665, 1632, 1554, 1418, 1369, 1215, 817, 768, 565; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.15 (d, *J* = 4.8 Hz, CH, 1H), 6.08 (d, *J* = 4.8 Hz, CH, 1H), 6.84 (s, CH, 1H), 7.60–8.07 (m, 8H, ArH), 10.35 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 40.7, 82.3, 103.7, 109.8, 118.5, 127.5, 128.4, 130.2, 132.1, 132.3, 138.7, 144.9, 157.4, 179.2, 182.5, 188.3; MS *m/z* (%): 405 (4); Anal. calcd for $C_{22}H_{12}ClNO_5$: C, 65.12; H, 2.98; N, 3.45; found: C, 65.01; H, 3.15; N, 3.32%.

trans-*2-(4-chlorobenzoyl)-5-hydroxy-3-(*p*-tolyl)-2,3-dihydrobenzofuran-4,7-dione* (**5k**): White powder; m.p. 292–294 °C; IR (ν_{max}, cm^{-1}) KBr: 3185, 2902, 1667, 1629, 1572, 1502, 1468, 1419, 1367, 1214, 757; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.19 (s, 3H, CH_3), 4.42 (d, *J* = 5.4 Hz, CH, 1H), 6.04 (d, *J* = 5.4 Hz, CH, 1H), 6.82 (s, CH, 1H), 6.93–8.04 (m, 8H, ArH), 10.62 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 21.3, 41.8, 82.2, 103.6, 109.6, 127.6, 1287, 128.9, 130.2, 132.1, 132.3, 135.6, 137.5, 138.7, 157.4, 179.3, 182.5, 188.2; MS *m/z* (%): 394 (6); Anal. calcd for $C_{22}H_{15}ClO_5$: C, 66.93; H, 3.83; found: C, 67.10; H, 3.95%.

trans-*3-(4-bromophenyl)-2-(4-chlorobenzoyl)-5-hydroxy-2,3-dihydrobenzofuran-4,7-dione* (**5l**): White powder; m.p. 278–280 °C; IR (ν_{max}, cm^{-1}) KBr: 3440, 2905, 2874, 1668, 1633, 1580, 1529, 1468, 1419, 1369, 1216, 818, 789, 667, 603, 505; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.21 (*J* =5.2 Hz, CH, 1H), 6.32 (*J* = 5.2 Hz, CH, 1H), 6.84 (s, CH, 1H), 7.32–8.02 (m, 16H, ArH), 6.85 (s, OH, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 42.1, 82.1, 103.4, 121.2, 128.6, 129.8, 130.3, 131.5, 132.0, 132.3, 138.6, 139.2, 157.7, 179.5, 182.6, 188.7; MS *m/z* (%): 459 (8); Anal. calcd for $C_{21}H_{12}BrClO_5$: C, 54.87; H, 2.63; found: C, 54.98; H, 2.50%.

(2S,3S,6S,7S)-2,6-bis(4-chlorobenzoyl)-3,7-bis(4-nitrophenyl)-2,3,6,7-tetrahydrobenzo[1,2-b:4,5-b']difuran-4,8-dione (**9**): White powder; m.p. 316–318 °C; IR (ν_{max}, cm^{-1}) KBr: 3195, 2905, 2630, 1667, 1634, 1572, 1504, 1418, 1331, 1267, 1124, 846, 817, 744, 664, 604; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.39 (d, *J* = 4.8 Hz, CH, 2H), 6.08 (d, *J* = 4.8 Hz, CH, 2H), 7.59–8.17 (m, 16H, ArH); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 39.7, 84.4, 123.7, 128.6, 128.8, 130.4, 130.5, 132.3. 13.6, 138.5, 145.9, 146.7, 157.4, 182.6, 188.4; MS *m/z* (%): 710 (5); Anal. calcd for $C_{36}H_{20}Cl_2N_2O_{10}$: C, 60.78; H, 2.83; N, 3.94; found: C, 60.93; H, 2.70; N, 9.12%.

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