Synthesis of Biarylketones and Phthalides from Organoboronic Acids and Aldehydes Catalyzed by Cobalt Complexes

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Supporting Information

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**General.** All reactions were conducted under nitrogen atmosphere on a dual-manifold Schlenk line unless otherwise mentioned and in oven-dried glass wares. All solvents were dried according to known methods and distilled prior to use.¹ Starting materials were commercially available and used as purchased.

**General procedure for the cobalt-catalyzed synthesis of biarylketones.** A seal tube (20 mL) containing CoCl₂ (0.10 mmol, 10 mol%), tmphen (0.015 mmol, 15 mol%) 4-methoxycarbonylbenzaldehyde (1.00 mmol) phenylboronic acid (1.50 mmol) and Cs₂CO₃ (2.00 mmol) were evacuated and purged with nitrogen gas three times. Freshly distilled acetonitrile (3.0 mL) and toluene (1.0 mL) were added and stirred well until the solution became deep blue. Then the reaction mixture was evacuated and filled with oxygen. The reaction mixture was heated with stirring at 80 °C for 14 h and then cooled, diluted with 30% n-hexane-ethyl acetate and stirred in air for 10 min. The mixture was filtered through a Celite and silica gel pad and washed with 30% n-hexane-ethyl acetate. The filtrate was concentrated and the residue was purified on a silica gel column using n-hexane-ethyl acetate as eluent to afford the desired product 3a. Similar procedures were employed to prepare compounds 3b-t. Similar procedures were also used for the preparation of 3u-w, except that the reactions were carried out under nitrogen atmosphere.
Table 1. Optimization studies for the cobalt-catalyzed addition of organoboronic acids with aldehydes to give diarylketones.\(^a\)

\[
\begin{align*}
\text{CHO} & \quad + \quad \text{B(OH)}_2 \\
& \quad \xrightarrow{\text{CoCl}_2, \text{ligand}, \text{additive, solvent}} \\
& \quad \quad 80 ^\circ \text{C, 12 h} \\
& \quad \quad \quad \quad \text{MeO}_2\text{C} \\
\end{align*}
\]

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<th>Additive</th>
<th>Solvent</th>
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\(^a\) Unless otherwise mentioned, all reactions were carried out using aldehyde \(1\) (1.00 mmol), arylboronic acid \(2\) (1.50 mmol), CoCl\(_2\) (0.10 mmol) tmphen (0.15 mmol), Cs\(_2\)CO\(_3\) (1.50 mmol) and CH\(_3\)CN/toluene (3:1, 4 mL) at 80 °C for 12 h under O\(_2\) (1 atm at room temperature). \(^b\) Isolated yields. \(^c\) The reaction was carried out under N\(_2\).
General procedure for the cobalt-catalyzed synthesis of 3-arylphthalides. A seal tube (20 mL) containing CoI₂ (0.050 mmol, 5 mol%), dppe (0.050 mmol, 5 mol%), K₂CO₃ (1.50 mmol) phthalaldehyde (1.00 mmol) and phenylboronic acid (1.50 mmol) was evacuated and purged with nitrogen gas three times. Freshly distilled THF (3.0 mL) was added and the mixture was stirred until the solution became deep brown. The reaction mixture was heated with stirring at 80 °C for 14 h and was then cooled, diluted with 30% n-hexane-ethyl acetate and stirred in the air for 10 min. The mixture was filtered through a Celite and silica gel pad and washed with 30% n-hexane-ethyl acetate. The filtrate was concentrated and the residue was purified on a silica gel column using n-hexane-ethyl acetate as eluent to afford the desired product 4a. Similar procedures were employed to prepare compounds 4b-4k.
Table 2. Optimization studies for the cobalt-catalyzed addition reaction of
organoboronic acids with phthalaldehydes to give 3-arylphthalides.\textsuperscript{a}

\[
\begin{array}{cccccc}
\text{Entry} & \text{Catalyst} & \text{Ligand} & \text{Additive} & \text{Solvent} & \text{Yield of} \\[-0.5ex]
& & & & \text{4a (\%)} \text{b} & \text{Yield of} \\[-0.5ex]
& & & & \text{5a (\%)} \text{b} \\
\hline
1 & \text{CoCl}_2 & \text{tmphen} & \text{Cs}_2\text{CO}_3 & \text{CH}_3\text{CN:} \text{Tol (3:1)} & 62 & 28 \\
2 & \text{CoCl}_2 & 1,10-\text{Phen} & \text{K}_2\text{CO}_3 & \text{CH}_3\text{CN} & 36 & 65 \\
3 & \text{CoI}_2 & \text{dppe} & \text{K}_2\text{CO}_3 & \text{CH}_3\text{CN} & 56 & 30 \\
4 & \text{CoI}_2 & \text{dppe} & \text{K}_2\text{CO}_3 & \text{THF} & \textbf{89} & \textbf{10} \\
5 & \text{CoI}_2 & \text{dppe} & \text{K}_2\text{CO}_3 & \text{THF} & 87\text{c} & 10\text{c} \\
6 & \text{CoI}_2 & \text{dppe} & \text{K}_2\text{CO}_3 & \text{Toluene} & 48 & 41 \\
7 & \text{CoI}_2 & \text{dppe} & \text{K}_2\text{CO}_3 & 1,2-\text{DCE} & 35 & 60 \\
8 & \text{CoI}_2 & \text{dppe} & \text{K}_2\text{CO}_3 & 1,4-\text{Dioxane} & - & - \\
9 & \text{CoI}_2 & \text{dppe} & \text{CsF} & \text{THF} & 67 & 32 \\
10 & \text{CoI}_2 & \text{dppe} & \text{Cs}_2\text{CO}_3 & \text{THF} & 78 & 18 \\
11 & \text{CoI}_2 & \text{dppe} & \text{Na}_2\text{CO}_3 & \text{THF} & 80 & 14 \\
12 & \text{CoI}_2 & \text{dppe} & \text{K}_3\text{PO}_4 & \text{THF} & 82 & 15 \\
\end{array}
\]

\textsuperscript{a} Unless otherwise mentioned, all reactions were carried out using phthalaldehyde 1 (1.00 mmol),
arylboronic acid 2 (1.50 mmol), \text{CoI}_2 (0.050 mmol), \text{dppe} (0.050 mmol), \text{K}_2\text{CO}_3 (1.50 mmol) and \text{THF}
(4 mL) at 80 °C for 12 h under N\textsubscript{2}. \textsuperscript{b} Isolated yields. \textsuperscript{c} The reaction was carried out under air.
The spectral data and a copy of the $^1$H and $^{13}$C NMR spectra of compounds 3 are listed below.

**4-Methoxycarbonylphenyl(phenyl)methanone (3a)**

![Structure of 4-Methoxycarbonylphenyl(phenyl)methanone (3a)](image)

White solid m.p. 65-67 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.13 (d, $J$ = 8.0 Hz, 2 H), 7.82 (d, $J$ = 8.0 Hz, 2 H), 7.78 (d, $J$ = 7.6 Hz, 2 H), 7.60 (t, $J$ = 7.2 Hz, 1 H), 7.48 (t, $J$ = 7.6 Hz, 2 H), 3.94 (s, 3 H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 196.0 (CO), 166.3 (CO-Ester), 141.3 (C), 136.9 (C), 133.2 (C), 132.9 (CH), 130.1 (2 CH), 129.7 (2 CH), 129.5 (2 CH), 128.5 (2 CH), 52.4 (CH$_3$); IR (neat) 1720 (ν$_{CO}$), 1655, 1438 and 1282 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{15}$H$_{12}$O$_3$ 240.0786, found 240.0780.

**4-Nitrophenyl(phenyl)methanone (3b)**

![Structure of 4-Nitrophenyl(phenyl)methanone (3b)](image)

Light yellow solid m.p. 76-77 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.32 (d, $J$ = 8.4 Hz, 2 H), 7.92 (d, $J$ = 8.8 Hz, 2 H), 7.80 (d, $J$ = 7.6 Hz, 2 H), 7.63 (t, $J$ = 7.6 Hz, 1 H), 7.50 (t, $J$ = 7.6 Hz, 2 H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.8 (CO), 149.8 (C), 142.8 (C), 136.3 (C), 133.5 (CH), 130.7 (2 CH), 130.1 (2 CH), 128.7 (2 CH), 123.5 (2 CH); IR (neat) 1650 (ν$_{CO}$), 1594, and 1513 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{13}$H$_9$NO$_3$ 227.0582, found 227.0590.

**3-Cyanophenyl(phenyl)methanone (3c)**
Pale yellow oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.04 (s, 1 H), 8.02 (d, $J$ = 8.4 Hz, 1 H), 7.85 (d, $J$ = 7.6 Hz, 2 H), 7.75 (d, $J$ = 7.6 Hz, 2 H), 7.64-7.59 (m, 2 H), 7.50 (t, $J$ = 7.6 Hz, 2 H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.4 (CO), 138.6 (C), 136.3 (C), 135.3 (CH), 133.8 (CH), 133.4 (CH), 133.2 (CH), 129.9 (2 CH), 129.4 (CH), 128.7 (CH), 117.9 (CN), 112.8 (C); IR (neat) 2231, 1664 ($\nu_{CO}$), 1597 and 1283 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{14}$H$_9$NO 207.0684, found 207.0681.

4-Cyanophenyl(phenyl)methanone (3d)

Pale yellow oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.83 (d, $J$ = 8.0 Hz, 2 H), 7.74 (d, $J$ = 8.4 Hz, 4 H), 7.60 (t, $J$ = 7.2 Hz, 1 H), 7.47 (t, $J$ = 7.6 Hz, 2 H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.9 (CO), 141.1 (C), 136.2 (C), 133.2 (CH), 132.1 (2 CH), 130.1 (2 CH), 129.9 (2 CH), 128.5 (2 CH), 117.9 (CN), 115.5 (C); IR (neat) 2228, 1649 ($\nu_{CO}$), 1596, and 1447 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{14}$H$_9$NO 207.0684, found 207.0681.

Phenyl(2-tolyl)methanone (3e)

Light yellow solid m.p. 114-116 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.84 (dd, $J$ = 7.6, $J$ = 1.4 Hz, 2 H), 7.56 (tt, $J$ = 7.2 Hz, $J$ = 1.2 Hz, 1 H), 7.37 (t, $J$ = 7.6 Hz, 2 H), 7.37 (dt, $J$ = 7.4 Hz, $J$ = 1.2 Hz, 1 H), 7.30-7.21 (m, 3 H), 2.31 (s, 3 H). $^{13}$C NMR (100
MHz, CDCl$_3$): $\delta$ 198.6 (CO), 138.6 (C), 137.7 (C), 136.7 (C), 133.1 (CH), 130.9 (CH), 130.2 (CH), 130.1 (2 CH), 128.5 (CH), 128.4 (2 CH), 125.2 (CH), 19.9 (CH$_3$); IR (neat) 1654 ($\nu_{\text{CO}}$), 1598, and 840 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{14}$H$_{12}$O 196.0888, found 196.0884.

**Phenyl(4-tolyl)methanone (3f)**

![Phenyl(4-tolyl)methanone](image)

Pale yellow oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.85 (d, $J = 8.0$ Hz, 2 H), 7.72 (d, $J = 7.6$ Hz, 2 H), 7.57 (t, $J = 7.2$ Hz, 1 H), 7.47 (t, $J = 7.6$ Hz, 2 H), 7.28 (d, $J = 7.6$ Hz, 2 H), 2.43 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 196.3 (CO), 143.0 (C), 137.7 (C), 134.7 (C), 131.9 (CH), 130.1 (2 CH), 129.7 (2 CH), 128.8 (2 CH), 128.0 (2 CH), 21.5 (CH$_3$); IR (neat) 1657 ($\nu_{\text{CO}}$), 1605 and 833 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{14}$H$_{12}$O 196.0884, found 196.0884.

**4-Methoxyphenyl(phenyl)methanone (3g)**

![4-Methoxyphenyl(phenyl)methanone](image)

Pale yellow oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.80 (dd, $J = 7.6$ Hz, $J = 1.2$ Hz, 2 H), 7.72 (dd, $J = 7.6$ Hz, $J = 1.2$ Hz, 2 H), 7.51 (t, $J = 7.6$ Hz, 1 H), 7.43 (t, $J = 7.2$ Hz, 2 H), 6.90 (dd, $J = 8.4$ Hz, $J = 1.2$ Hz, 2 H), 3.84 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 195.0 (CO), 162.0 (C), 137.9 (C), 132.1 (2 CH), 131.5 (CH), 129.7 (C), 129.3 (2 CH), 127.8 (2 CH), 113.2 (2 CH), 55.1 (CH$_3$); IR (neat) 2839, 1651 ($\nu_{\text{CO}}$), 1508 and 1318 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{14}$H$_{12}$O$_2$ 212.0837, found 212.0836.

**4-Bromophenyl(phenyl)methanone (3h)**

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Pale pink solid m.p. 81-83 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta 7.76-7.70\) (m, 2 H) 7.67-7.50 (m, 5 H), 7.48-7.44 (m, 2 H). \(^{13}\)C NMR (125 MHz, CDCl\(_3\)): \(\delta 195.6\) (CO), 137.1 (C), 136.2(C), 132.6 (CH), 131.6 (2 CH), 131.5 (2 CH), 129.8 (2 CH), 128.3 (2 CH), 127.4 (C); IR (neat) 1648 (\(\nu_{CO}\)), 1586 and 1279 cm\(^{-1}\); HRMS (EI\(^+\)) calcd for C\(_{13}\)H\(_9\)BrO 259.9837, found 259.9836.

4-Chlorophenyl(phenyl)methanone (3i)

Pale yellow solid m.p. 72-75 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta 7.74\) (t, \(J = 8.0\) Hz, 4 H), 7.57 (t, \(J = 7.6\) Hz, 1 H), 7.48-7.42 (m, 4 H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta 195.4\) (CO), 138.8 (C), 137.1(C), 135.8(C), 132.5 (CH), 131.4 (2 CH), 129.8 (2 CH), 128.5 (2 CH), 128.3 (2 CH); IR (neat) 1649 (\(\nu_{CO}\)), 1586, 1444 and 863 cm\(^{-1}\); HRMS (EI\(^+\)) calcd for C\(_{13}\)H\(_9\)ClO 216.0342, found 216.0338.

Benzophenone (3j)

White solid m.p. 48-50 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta 7.78\) (d, \(J = 7.6\) Hz, 4 H),7.50 (t, \(J = 7.2\) Hz, 2 H), 7.45 (t, \(J = 8.0\) Hz, 4 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta 196.4\) (CO), 137.4 (2C), 132.2 (2 CH), 129.8 (4 CH), 128.0 (4 CH); IR (neat) 1651
(ν<sub>CO</sub>), 1598 and 1320 cm<sup>-1</sup>; **HRMS** (EI<sup>+</sup>) calcd for C<sub>13</sub>H<sub>10</sub>O 182.0732, found 182.0736.

**2-Furyl(phenyl)methanone (3k)**

![Structure of 2-Furyl(phenyl)methanone (3k)](image)

Yellow oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.94 (d, J = 7.2 Hz, 2 H), 7.68 (s, 1 H), 7.56 (t, J = 7.2 Hz, 1 H), 7.46 (t, J = 7.6 Hz, 2 H), 7.20 (d, J = 2.8 Hz, 1 H), 6.57 (dd, J = 3.4 Hz, J = 1.2 Hz, 1 H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 182.5 (CO), 152.2 (C), 147.0 (CH), 137.2 (C), 132.5 (CH), 129.2 (2 CH), 128.3 (2 CH), 120.5 (CH), 112.1 (CH); IR (neat) 2839, 1651 (ν<sub>CO</sub>), 1508 and 1318 cm<sup>-1</sup>; **HRMS** (EI<sup>+</sup>) calcd for C<sub>13</sub>H<sub>10</sub>O 172.0524, found 172.0521.

**Phenyl(2-thienyl)methanone (3l)**

![Structure of Phenyl(2-thienyl)methanone (3l)](image)

Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.84 (d, J = 7.2 Hz, 2 H), 7.70 (d, J = 5.2 Hz 1 H), 7.62 (d, J = 3.6 Hz, 1 H), 7.57 (t, J = 7.2 Hz, 1 H), 7.47 (t, J = 7.6 Hz, 2 H), 7.14 (t, J = 4.4 Hz, 1 H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 188.1 (CO), 143.6 (C), 138.0(C), 134.8 (CH), 134.1 (CH), 132.2 (CH), 129.1 (2 CH), 128.3 (2 CH) 127.9 (CH); IR (neat) 1636 (ν<sub>CO</sub>), 1598 and 1412 cm<sup>-1</sup>; **HRMS** (EI<sup>+</sup>) calcd for C<sub>11</sub>H<sub>8</sub>OS 188.0296, found 188.0302.

**Phenyl(3-pyridinyl)methanone (3m)**

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Colourless oil; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.96 (s, 1 H), 8.79 (d, $J = 4.8$ Hz, 1 H), 8.15 (d, $J = 7.6$ Hz, 1 H), 7.79 (d, $J = 7.6$ Hz, 2 H), 7.62 (t, $J = 7.6$ Hz, 1 H), 7.52-7.41 (m, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.4 (CO), 152.5 (CH), 150.5 (CH), 150.5 (C), 136.8(CH), 136.3 (C), 132.8 (CH), 132.7 (C), 129.7 (2 CH), 128.3 (2 CH), 123.0 (CH); IR (neat) 2231, 1664 ($\nu$ CO), 1597 and 1283 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{13}$H$_9$NO 183.0684, found 183.0677.

**Phenyl(4-Pyridinyl)methanone (3n)**

White solid m.p. 66-69 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.75 (d, $J = 6.0$ Hz, 2 H), 7.76 (d, $J = 7.2$ Hz, 2 H), 7.58 (t, $J = 7.60$ Hz, 2 H), 7.52 (d, $J = 6.0$ Hz, 2 H), 7.45 (t, $J = 7.6$ Hz, 2 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 195.0 (CO), 150.2 (2 CH), 144.2(C), 135.7(C), 133.4 (CH), 130.0 (2 CH), 128.5 (2 CH), 122.7 (2 CH); IR (neat) 1652 ($\nu$ CO), 1598, 1550 and 1408 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{13}$H$_9$NO 183.0684, found 183.0683.

**4-Cyanophenyl(4-tolyl)methanone (3o)**
Yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.85 (d, $J = 8.4$ Hz, 2 H), 7.78 (d, $J = 8.4$ Hz, 2 H), 7.69 (d, $J = 8.0$ Hz, 2 H), 7.31 (d, $J = 8.0$ Hz, 2 H), 2.45 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.8 (CO), 144.4 (C), 141.6 (C), 133.6 (C), 132.1 (2 CH), 130.3 (2 CH), 130.1 (2 CH), 129.3 (2 CH), 118.1(CN), 115.4(C), 21.7 (CH$_3$); IR (neat) 2230, 1649 (ν$_{CO}$), 1603 and 1140 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{15}$H$_{11}$NO 221.0841, found 221.0843.

4-Cyanophenyl(2-methoxyphenyl)methanone (3p)

Yellow solid m.p. 128-130 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.82 (d, $J = 8.4$ Hz, 2 H), 7.69 (d, $J = 8.4$ Hz, 2 H), 7.50 (t, $J = 8.0$ Hz, 1 H), 7.40 (dd, $J = 7.6$ Hz, $J = 1.6$ Hz, 1 H), 7.04 (t, $J = 7.6$ Hz, 1 H), 6.96 (d, $J = 8.0$ Hz, 1 H), 3.65 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.9 (CO), 157.5 (C), 141.4 (C), 133.0 (CH), 131.9 (2 CH), 130.0 (CH), 129.7 (2 CH), 127.3 (C), 130.8 (CH), 118.1(CN), 115.6(C), 111.4 (CH), 55.4 (CH$_3$); IR (neat) 2847, 2230, 1666 (ν$_{CO}$) and 1598 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{15}$H$_{11}$NO$_2$ 237.0790, found 237.0793.

4-Cyanophenyl(4-methoxyphenyl)methanone (3r)

Yellow solid m.p. 131-134 °C; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.81-7.52 (m, 6 H), 6.97 (dd, $J = 7.5$ Hz, $J = 2.0$ Hz, 2 H), 3.88 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 193.7 (CO), 163.8 (C), 142.0 (C), 132.6 (2 CH), 132.0 (2 CH), 129.9 (2 CH), 128.9
(C), 118.1(CN), 115.1(C), 113.9 (2 CH), 55.6 (CH₃); IR (neat) 2851, 2230, 1655 (νCO) and 1598 cm⁻¹; HRMS (EI⁺) calcd for C₁₅H₁₁NO₂ 237.0790, found 237.0793.

4-Chlorophenyl(4-cyanophenyl)methanone (3s)

Yellow solid m.p. 137-140 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.83 (d, J = 8.4 Hz, 2 H), 7.78 (d, J = 8.0 Hz, 2 H), 7.71 (d, J = 8.4 Hz, 2 H), 7.47 (d, J = 8.4 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ 193.8 (CO), 140.8 (C), 139.9 (C), 134.6 (C), 132.2 (2 CH), 131.4 (C), 130.1 (2 CH), 129.0 (2 CH), 117.8(CN), 115.9(C); IR (neat) 2228, 1645 (νCO) and 1588 cm⁻¹; HRMS (EI⁺) calcd for C₁₄H₈ClNO 241.0294, found 241.0298.

4-Cyanophenyl(4-flourophenyl)methanone (3t)

Yellow solid m.p. 92-95 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.84- 7.76 (m, 6 H), 7.17 (t, J = 8.6 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ 193.5 (CO), 165.8 (d, J_CF = 254.0 Hz, C), 141.1 (C), 133.3 (C), 132.7 (2 CH), 132.5 (2 CH), 130.0 (2 CH), 117.8(CN), 116.0 (CH), 115.8 (CH), 115.7(C); IR (neat) 2231, 1648 (νCO), 1598 and 860 cm⁻¹; HRMS (EI⁺) calcd for C₁₄H₉FNO 225.0590, found 225.0588.

4-Methoxycarbonylphenyl(Phenyl)proponone (3u)
White solid; m.p. 78-82 °C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.11 (dd, $J = 6.8$ Hz, $J = 2.0$ Hz, 2 H), 8.00 (dd, $J = 6.8$ Hz, $J = 2.0$ Hz, 2 H), 7.32 - 7.21 (m, 5 H), 3.94 (s, 3H), 3.33 (t, $J = 7.6$ Hz, 2 H), 3.07 (t, $J = 7.6$ Hz, 2 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 198.6 (CO-ketone), 166.1 (CO-Ester), 140.8 (C), 139.9 (C), 133.7 (C), 129.7 (2 CH), 128.5 (2 CH), 128.3 (2 CH), 127.8 (2 CH), 126.1 (CH), 52.4 (CH$_3$), 40.7 (CH$_2$), 29.8 (CH$_2$); IR (neat) 1712, 1684 (v$_{CO}$), 1626 and 1576 cm$^{-1}$; HRMS (El$^+$) calcd for C$_{17}$H$_{16}$O$_3$ 268.1099, found 268.1102.

**4-Methoxycarbonylphenyl(4-trifluoromethylPhenyl)proponone (3v)**

White solid m.p. 117-120 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.10 (d, $J = 8.0$ Hz, 2 H), 7.97 (d, $J = 8.4$ Hz, 2 H), 7.52 (d, $J = 7.6$ Hz, 2 H), 7.35 (d, $J = 8.0$ Hz, 2 H), 3.91 (s, 3H), 3.32 (t, $J = 7.6$ Hz, 2 H), 3.11 (t, $J = 7.6$ Hz, 2 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 198.0 (CO-ketone), 166.1 (CO-Ester), 145.1 (C), 139.7 (C), 133.9 (C), 129.8 (2 CH), 128.7 (2 CH), 128.7 (2 CH), 127.8 (2 CH), 125.4 (q, C$_{C-F}$ J = 3.6Hz, C), 52.4 (CH$_3$), 40.1 (CH$_2$), 29.5 (CH$_2$); IR (neat) 2924, 1725, 1671 (v$_{CO}$) and 1571 cm$^{-1}$; HRMS (El$^+$) calcd for C$_{18}$H$_{15}$F$_3$O$_3$ 336.0973, found 336.0977.

**4-Methoxycarbonylphenyl(4-chloroPhenyl)proponone (3w)**
White solid m.p. 104-106 °C; $^1$H NMR (400 MHz, CDCl$_3$): δ 8.28 (d, $J$ = 8.4 Hz, 2 H), 8.17 (d, $J$ = 8.4 Hz, 2 H), 7.43 (d, $J$ = 8.4 Hz, 2 H), 7.35 (d, $J$ = 8.4 Hz, 2 H), 4.11 (s, 3H), 3.48 (t, $J$ = 7.6 Hz, 2 H), 3.21 (t, $J$ = 7.6 Hz, 2 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 198.6 (CO-ketone), 166.1 (CO-Ester), 140.8 (C), 139.9 (C), 133.7 (C), 129.7 (2 CH), 128.5 (2 CH), 128.3 (2 CH), 127.8 (2 CH), 126.1 (CH), 52.4 (CH$_3$), 40.7 (CH$_2$), 29.8 (CH$_2$); IR (neat) 1717 (ν$_{CO}$), 1681(ν$_{CO}$), 1618 and 1572 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{17}$H$_{15}$ClO$_3$ 302.0710, found 302.0712.
The spectral data and a copy of the $^1$H and $^{13}$C NMR spectra of compounds 4 are listed below.

**3-Phenylisobenzofuran-1(3$H$)-one (4a)**

![Chemical Structure](image)

White solid m.p. 112-113 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.93 (d, $J = 7.6$ Hz, 1 H), 7.62 (t, $J = 7.6$ Hz, 1 H), 7.52 (t, $J = 7.6$ Hz, 1 H), 7.36-7.29 (m, 4 H), 7.26-7.24 (m, 2 H), 6.38 (s, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 170.4 (CO-lactone), 149.6 (C), 136.3 (C), 134.3 (C), 129.3 (CH), 129.2 (CH), 128.9 (2 CH), 126.9 (2 CH), 125.5 (CH), 125.4(C), 122.8 (CH), 82.6 (CH); IR (neat) 1764 ($\nu$CO), 1495, and 1285 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{14}$H$_{10}$O$_2$ 210.0681, found 210.0681.

**3-p-Tolylisobenzofuran-1(3$H$)-one (4b)**

![Chemical Structure](image)

White solid m.p. 107-110 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.92 (d, $J = 8.0$ Hz, 1 H), 7.62 (dt, $J = 7.6$ Hz, $J = 1.2$ Hz, 1 H), 7.52 (t, $J = 7.6$ Hz, 1 H), 7.29 (dd, $J = 7.2$ Hz, $J = 1.2$ Hz, 1 H), 7.17-7.11 (m, 4 H), 6.35 (s, 1 H), 2.32 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 170.5 (CO-lactone), 149.7 (C), 136.2 (C), 134.2 (CH), 133.3 (C), 129.5 (2 CH), 129.2 (CH), 126.9 (2 CH), 125.6 (C), 125.4(CH), 122.8 (CH), 82.7
(CH), 21.1 (CH$_3$); IR (neat) 1754 (ν$_{CO}$), 1513 and 1463 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{15}$H$_{12}$O$_2$ 224.0837, found 224.0835.

3-(3-Methoxyphenyl)isobenzofuran-1(3H)-one (4c)

White solid m.p. 118-120 °C; $^1$H NMR (400 MHz, CDCl$_3$): δ 7.92 (td, $J = 7.6$ Hz, $J = 0.8$ Hz, 1 H), 7.61 (dt, $J = 7.6$ Hz, $J = 1.2$ Hz, 1 H), 7.52 (t, $J = 7.6$ Hz, 1 H), 7.32 (qd, $J = 8.0$ Hz, $J = 0.2$ Hz, 1 H), 7.32 (t, $J = 8.0$ Hz, 1 H), 6.88-6.75 (m, 3 H), 6.38 (s, 1 H), 3.74 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 170.4 (CO-lactone), 159.9 (C), 149.5 (C), 137.9 (C), 134.3 (CH), 129.9 (CH), 129.3 (CH), 125.5 (CH), 125.3 (C), 122.8 (CH), 118.9 (CH), 114.5 (CH), 112.3 (CH), 82.4 (CH), 55.2 (CH$_3$); IR (neat) 2837, 1766 (ν$_{CO}$), and 1600 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{15}$H$_{12}$O$_3$ 240.0786, found 240.0785.

3-(4-Methoxyphenyl)isobenzofuran-1(3H)-one (4d)

White solid m.p. 110-112 °C; $^1$H NMR (400 MHz, CDCl$_3$): δ 7.92 (dd, $J = 7.6$ Hz, $J = 1.2$ Hz, 1 H), 7.62 (dt, $J = 7.6$ Hz, $J = 1.2$ Hz, 1 H), 7.52 (dt, $J = 7.6$ Hz, $J = 0.8$ Hz, 1 H), 7.28 (dd, $J = 7.6$ Hz, $J = 1.2$ Hz, 1 H), 7.14 (d, $J = 8.8$ Hz, 2H), 6.86 (dd, $J = 8.8$ Hz, $J = 2.4$ Hz, 2H), 6.34 (s, 1 H), 3.77 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ
170.5 (CO-lactone), 160.4 (C), 149.7 (C), 134.2 (CH), 129.2 (CH), 129.2 (CH), 128.7 (2 CH), 128.2 (C), 125.8(C), 125.5 (CH), 122.9 (CH), 114.3 (2 CH), 82.7 (CH), 55.3 (CH₃); IR (neat) 2840, 1753 (νCO), 1612 and 1286 cm⁻¹; HRMS (EI⁺) calcd for C₁₅H₁₂O₃ 240.0786, found 240.0782.

(E)-3-Styrylisobenzofuran-1(3H)-one (4e)

White solid m.p. 98-100 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.92 (d, J = 7.5 Hz, 1 H), 7.68 (t, J = 7.5 Hz, 1 H), 7.54 (t, J = 7.5 Hz, 1 H), 7.45 (d, J = 7.0 Hz, 2H), 7.39 (d, J = 7.0 Hz, 2 H), 7.31-7.25 (m, 3 H), 6.90 (d, J = 15.0 Hz, 1 H), 6.12 (dd, J = 15.0 Hz, J = 7.0 Hz, 1 H), 5.99 (d, J = 7.5 Hz, 1 H); ¹³C NMR (125 MHz, CDCl₃): δ 170.3 (CO-lactone), 148.8 (C), 136.3 (C), 135.4 (C), 135.2 (CH), 134.2 (CH), 129.4 (CH), 128.7 (2 CH), 128.6 (CH), 126.9 (2 CH), 125.7 (CH), 123.8 (C), 122.6 (CH), 82.1 (CH); IR (neat) 1760 (νCO), 1598 and 1284 cm⁻¹; HRMS (EI⁺) calcd for C₁₆H₁₂O₂ 236.0837, found 236.0836.

3-(4-Bromophenyl)isobenzofuran-1(3H)-one (4f)

White solid m.p. 157-159 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.96 (d, J = 7.6 Hz, 1 H), 7.66 (dt, J = 7.6 Hz, J = 1.2 Hz, 1 H), 7.56 (dt, J = 7.2 Hz, J = 1.2 Hz, 1 H), 7.50
(d, \( J = 6.8 \) Hz, 2 H), 7.31 (dd, \( J = 7.6 \) Hz, \( J = 1.2 \) Hz, 1 H), 7.16 (d, \( J = 8.8 \) Hz, 1 H), 6.36 (s, 1 H); \(^{13}\text{C} \text{ NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) 170.2 (CO-lactone), 149.1 (C), 135.4 (C), 134.4 (CH), 132.1 (2 CH), 129.5 (CH), 128.6 (2 CH), 125.7 (CH), 123.4 (C), 122.7 (CH), 81.8 (CH); IR (neat) 1768 (\( \nu_{\text{CO}} \)), 1598 and 1284 cm\(^{-1}\); \text{HRMS} \) (EI\(^+\)) calcd for \( \text{C}_{14}\text{H}_9\text{BrO}_2 \) 287.9786, found 287.9786.

3-(3-Chlorophenyl)isobenzofuran-1(3\( H \))-one (4g)

![3-Chlorophenyl isobenzofuran structure](image)

White solid m.p. 132-134 °C; \(^1\text{H} \text{ NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.95 (d, \( J = 7.6 \) Hz, 1 H), 7.62 (dt, \( J = 7.6 \) Hz, \( J = 1.2 \) Hz, 1 H), 7.56 (dt, \( J = 7.6 \) Hz, \( J = 0.8 \) Hz, 1 H), 7.35-7.29 (m, 3 H), 7.25 (s, 1 H), 7.20-7.17 (m, 1 H), 6.39 (s, 1 H); \(^{13}\text{C} \text{ NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) 170.1 (CO-lactone), 148.9 (C), 138.4 (C), 134.8 (C), 134.5 (CH), 130.3 (CH), 129.6 (CH), 129.4 (CH), 126.9 (CH), 125.7 (CH), 125.3 (C), 125.0 (CH), 122.7 (CH) 81.6 (CH); IR (neat) 1764 (\( \nu_{\text{CO}} \)), 1597 and 1284 cm\(^{-1}\); \text{HRMS} \) (EI\(^+\)) calcd for \( \text{C}_{14}\text{H}_9\text{ClO}_2 \) 244.0291, found 244.0287.

3-(3-Fluorophenyl)isobenzofuran-1(3\( H \))-one (4h)

![3-Fluorophenyl isobenzofuran structure](image)

White solid m.p. 85-87 °C; \(^1\text{H} \text{ NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.93 (d, \( J = 7.6 \) Hz, 1 H), 7.64 (t, \( J = 7.6 \) Hz, 1 H), 7.54 (t, \( J = 7.6 \) Hz, 1 H), 7.36-7.31 (m, 2 H), 7.09 (d, \( J = 7.6 \) Hz, 1 H), 7.03 (td, \( J = 8.4 \) Hz, \( J = 2.8 \) Hz, 1 H), 6.94 (td, \( J = 8.8 \) Hz, \( J = 2.0 \) Hz,
1 H), 6.36 (s, 1 H); \(^{13}\text{C} \text{NMR} \) (100 MHz, CDCl\(_3\)):  \(\delta\) 170.1 (CO-lactone), 163.1 (d,  \(J_{C-F} =986.8\) Hz, C), 149.0 (C), 138.8 (d,  \(J_{C-F} =7.4\) Hz, C), 134.4 (CH), 130.6 (d,  \(J_{C-F} =8.1\) Hz, CH), 129.5 (CH), 125.7 (CH), 125.3 (C), 122.7 (CH), 122.5 (d,  \(J_{C-F} =3.7\) Hz, CH), 116.2 (d,  \(J_{C-F} =21.2\) Hz, CH), 113.7 (d,  \(J_{C-F} =22.6\) Hz, CH), 81.6 (CH); IR (neat) 1772, 1762 (\(\nu_{\text{CO}}\)) and 1466 cm\(^{-1}\); \text{HRMS} \ (\text{EI}^+) \text{ calcd for } C_{14}H_{10}FO_2 \text{ 228.0587, found 228.0583.}

\textbf{3-(4-Biphenyl)isobenzofuran-1(3H)-one (4i)}

![3-(4-Biphenyl)isobenzofuran-1(3H)-one (4i)](image)

White solid m.p. 211-213 °C; \(^{1}\text{H} \text{NMR} \) (400 MHz, CDCl\(_3\)):  \(\delta\) 7.97 (d,  \(J = 7.6\) Hz, 1 H), 7.66 (t,  \(J = 7.6\) Hz, 1 H), 7.59- 7.54 (m, 5 H), 7.42 (m, 4 H), 7.42 (t,  \(J = 7.6\) Hz, 1 H), 7.35- 7.25 (m, 4 H), 6.38 (s, 1 H); \(^{13}\text{C} \text{NMR} \) (100 MHz, CDCl\(_3\)):  \(\delta\) 170.6 (CO-lactone), 149.6 (C), 142.3 (C), 140.2 (C), 135.2 (C), 134.4 (CH), 129.4 (CH), 128.8 (2 CH), 128.3 (2 CH), 127.7 (2 CH), 127.4(2 CH), 126.7 (CH), 125.6 (C), 122.7 (CH), 115.7 (CH), 82.6 (CH); IR (neat) 1750 (\(\nu_{\text{CO}}\)), 1598 and 1464 cm\(^{-1}\); \text{HRMS} \ (\text{EI}^+) \text{ calcd for } C_{20}H_{14}O_2 \text{ 286.0994, found 286.0996.}

\textbf{3-(4-Vinylphenyl)isobenzofuran-1(3H)-one (4j)}

![3-(4-Vinylphenyl)isobenzofuran-1(3H)-one (4j)](image)
White solid m.p. 116-118 °C; $^1$H NMR (400 MHz, CDCl$_3$): δ 7.96 (d, $J = 7.6$ Hz, 1 H), 7.64 (t, $J = 7.6$ Hz, 1 H), 7.55 (t, $J = 7.6$ Hz, 1 H), 7.41 (d, $J = 7.6$ Hz, 2 H), 7.32 (dd, $J = 7.6$ Hz, $J = 1.2$ Hz, 1 H), 7.23 (d, $J = 8.4$ Hz, 2 H), 6.70 (dd, $J = 17.6$ Hz, $J = 10.8$ Hz, 1 H), 6.40 (s, 1 H), 5.76 (d, $J = 17.6$ Hz, 1 H), 5.26 (d, $J = 11.2$ Hz, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 170.5 (CO-lactone), 149.6 (C), 138.3 (C), 135.9 (CH), 135.7 (C), 134.3 (CH), 129.4 (CH), 127.2 (2 CH), 126.7 (2 CH), 125.6 (CH), 125.5 (C), 122.8 (CH), 114.9 (CH$_2$), 82.5 (CH); IR (neat) 1756 ($\nu$CO), 1598 and 1284 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{16}$H$_{12}$O$_2$ 236.0837, found 236.0835.

3-(Naphthalen-2-yl)isobenzofuran-1(3H)-one (4k)

White solid m.p. 148-150 °C; $^1$H NMR (400 MHz, CDCl$_3$): δ 7.98 (d, $J = 7.6$ Hz, 1 H), 7.83- 7.78 (m, 4 H), 7.61 (t, $J = 7.6$ Hz, 1 H), 7.55- 7.47 (m, 3 H), 7.31 (d, $J = 7.6$ Hz, 1 H), 7.21 (d, $J = 8.4$ Hz, 1 H), 6.53 (s, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 170.5 (CO-lactone), 149.6 (C), 134.3 (CH), 133.6 (C), 133.5 (C), 132.9 (C), 129.4 (CH), 128.9 (CH), 128.0 (CH), 127.7 (CH), 126.8 (CH), 126.7 (CH), 126.6 (CH), 125.6 (CH), 125.5(C), 123.7 (CH), 122.9 (CH), 82.8 (CH); IR (neat) 1764 ($\nu$CO), 1599, 1465 and 1285 cm$^{-1}$; HRMS (EI$^+$) calcd for C$_{18}$H$_{12}$O$_2$ 260.0837, found 260.0836.

References:
$^1$H and $^{13}$C NMR spectra of compound 3a
$^1$H and $^{13}$C NMR spectra of compound 3b
$^{1}$H and $^{13}$C NMR spectra of compound 3c
$^1$H and $^{13}$C NMR spectra of compound 3d
$^1$H and $^{13}$C NMR spectra of compound 3e
\(^1\)H and \(^{13}\)C NMR spectra of compound 3f
$^1$H and $^{13}$C NMR spectra of compound 3g
$^1$H and $^{13}$C NMR spectra of compound 3h
\(^1\)H and \(^{13}\)C NMR spectra of compound \(3i\)
\( ^1 \)H and \( ^{13} \)C NMR spectra of compound 3j
\(^{1}\)H and \(^{13}\)C NMR spectra of compound \(3k\)
$^1$H and $^{13}$C NMR spectra of compound 31
$^1$H and $^{13}$C NMR spectra of compound 3m
$^1$H and $^{13}$C NMR spectra of compound 3n
$^1$H and $^{13}$C NMR spectra of compound 3o
$^1$H and $^{13}$C NMR spectra of compound $3p$
$^{1}$H and $^{13}$C NMR spectra of compound 3q
$^1$H and $^{13}$C NMR spectra of compound 3r
$^1$H and $^{13}$C NMR spectra of compound 3s
$^1$H and $^{13}$C NMR spectra of compound 3t
$^1$H and $^{13}$C NMR spectra of compound 3u
$^1$H and $^{13}$C NMR spectra of compound 3v
$^1$H and $^{13}$C NMR spectra of compound 3w
$^1$H and $^{13}$C NMR spectra of compound 4a
$^{1}H$ and $^{13}C$ NMR spectra of compound 4b
$^1$H and $^{13}$C NMR spectra of compound 4c
$^{1}$H and $^{13}$C NMR spectra of compound 4d

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$^1$H and $^{13}$C NMR spectra of compound 4e
$^1$H and $^{13}$C NMR spectra of compound 4f
$^1$H and $^{13}$C NMR spectra of compound 4g
$^1$H and $^{13}$C NMR spectra of compound 4i
$^1$H and $^{13}$C NMR spectra of compound 4j
$^1$H and $^{13}$C NMR spectra of compound 4k
$^1$H and $^{13}$C NMR spectra of compound 41