

Supporting Information

Palladium-Catalyzed Carbonylative Synthesis of Aryl Esters from *p*- Benzoquinones and Aryl Triflates

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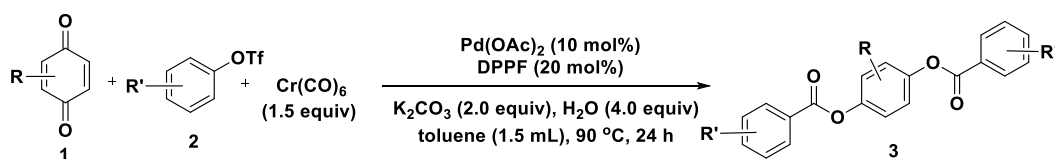
Table of Contents

| | |
|--|-----|
| 1. General experimental information | S1 |
| 2. General procedure for the carbonylative synthesis of aryl esters (3aa-at, 3bb-fb) | S1 |
| 3. Characterization data of products (3aa-at, 3bb-fb) | S2 |
| 4. Reference | S11 |
| 5. ¹ H, ¹³ C NMR spectra of products (3aa-at, 3bb-fb) | S12 |

1. General experimental information

Unless otherwise noted, all reactions were carried out under nitrogen atmosphere. All commercially available reagents were used without further purification. All of the solvents were treated according to known methods. Column chromatography was performed on silica gel (200-400 mesh). ^1H NMR (400 MHz) chemical shifts were reported in ppm (δ) relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard. ^{13}C NMR (100 MHz) chemical shifts were reported in ppm (δ) from tetramethylsilane (TMS) with the solvent resonance as the internal standard. Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, qd = quartet of doublets, m = multiplet), coupling constants (Hz) and integration. HRMS measurements were obtained on a TOF analyzer.

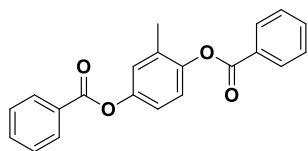
2. General procedure for the carbonylative synthesis of aryl esters (3aa-at, 3bb-fb)



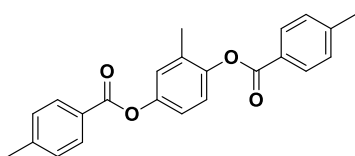
$\text{Pd}(\text{OAc})_2$ (4.5 mg, 10 mol%), DPPF (22.2 mg, 20 mol%), $\text{Cr}(\text{CO})_6$ (66.1 mg, 0.3 mmol, 1.5 equiv), a *p*-benzoquinone **1** (24.4 mg, 0.2 mmol), K_2CO_3 (55.3 mg, 0.4 mmol, 2.0 equiv) were added to an oven-dried tube (15.0 mL) which was then placed under vacuum and refilled with nitrogen three times. Then an aryl triflate **2** (0.2 mmol), H_2O (14.4 mg, 0.8 mmol, 4.0 equiv) and toluene (1.5 mL) were added into the tube via syringe. The tube was sealed and stirred at 90 °C for 24 h. Upon the reaction was completed, the resulting mixture was purified by silica gel column using chromatography (petroleum ether/ ethyl acetate = 10:1) to obtain a product **3**.

1 mmol scale: $\text{Pd}(\text{OAc})_2$ (10 mol%), DPPF (20 mol%), $\text{Cr}(\text{CO})_6$ (1.5 mmol, 1.5 equiv), a *p*-benzoquinone **1a** (1 mmol), K_2CO_3 (2 mmol, 2.0 equiv) were added to an oven-dried tube (15.0 mL) which was then placed under vacuum and refilled with nitrogen three times. Then an aryl triflate **2b** (1 mmol), H_2O (4 mmol, 4.0 equiv) and toluene (5 mL) were added into the tube via syringe. The tube was sealed and stirred at 90 °C for 24 h. Upon the reaction was completed, the resulting mixture was purified by silica gel column using chromatography (petroleum ether/ ethyl acetate = 10:1) to obtain a product **3ab** in 72% (129.6 mg).

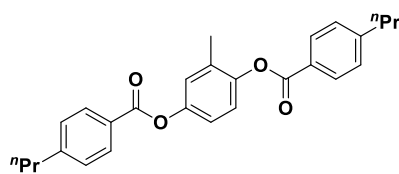
3. Characterization data of products (3aa-at, 3bb-fb)



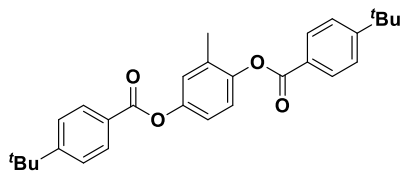
2-methyl-1,4-phenylene dibenzoate (3aa).¹ Yellow solid, 25.9 mg, 78% yield; mp.104.2-106.1 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.26 – 8.21 (m, 4H), 7.69-7.62 (m, 2H), 7.58-7.48 (m, 4H), 7.26-7.10 (m, 3H), 2.28 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 165.3, 164.9, 148.6, 147.2, 133.83, 133.78, 132.0, 130.3, 129.6, 129.5, 128.8, 128.7, 128.1, 124.3, 123.0, 120.2, 16.5.



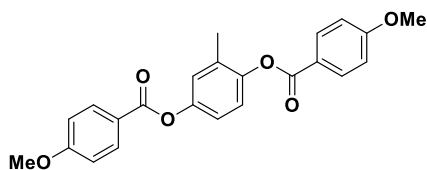
2-methyl-1,4-phenylene bis(4-methylbenzoate) (3ab).² Yellow solid, 29.1 mg, 81% yield; mp.137.0-138.8 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.15-8.06 (m, 4H), 7.36-7.29 (m, 4H), 7.21-7.08 (m, 3H), 2.47 (s, 3H), 2.46 (s, 3H), 2.26 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 165.4, 165.0, 148.6, 147.2, 144.7, 144.6, 132.0, 130.37, 130.36, 129.5, 129.4, 126.9, 126.7, 124.2, 123.0, 120.2, 22.0, 16.6.



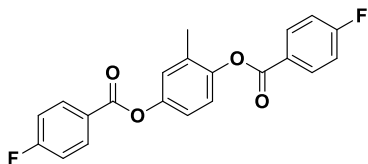
2-methyl-1,4-phenylene bis(4-propylbenzoate) (3ac).² Yellow solid, 26.6 mg, 64% yield; mp.95.7-97.2 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.21-8.10 (m, 4H), 7.39-7.31 (m, 4H), 7.19 (d, *J* = 8.6 Hz, 1H), 7.15 (d, *J* = 2.5 Hz, 1H), 7.10 (dd, *J* = 8.6, 2.7 Hz, 1H), 2.72 – 2.67 (m, 4H), 2.26 (s, 3H), 1.70 (ddd, *J* = 15.1, 7.5, 2.3 Hz, 4H), 0.98 (td, *J* = 7.3, 3.0 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 165.4, 165.0, 149.21, 149.25, 149.3, 148.6, 147.2, 132.0, 130.4, 128.90, 128.85, 127.1, 127.0, 124.2, 123.0, 120.2, 38.3, 24.4, 16.6, 13.9.



2-methyl-1,4-phenylene bis(4-(*tert*-butyl)benzoate) (3ad). Black solid, 30.2 mg, 68% yield; mp.150.7-152.1 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.20-8.05 (m, 4H), 7.59-7.51 (m, 4H), 7.21-7.09 (m, 3H), 2.26 (s, 3H), 1.39 (s, 9H), 1.38 (s, 9H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 165.3, 164.9, 157.63, 157.56, 148.6, 147.2, 132.0, 130.3, 130.2, 126.8, 126.7, 125.8, 125.7, 124.2, 123.0, 120.2, 35.4, 31.2, 16.5; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd. for $\text{C}_{29}\text{H}_{32}\text{O}_4\text{H}^+$ 445.2373; found: 445.2379.

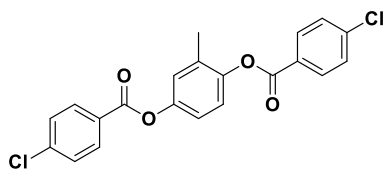


2-methyl-1,4-phenylene bis(4-methoxybenzoate) (3ae).² White solid, 21.6 mg, 55% yield; mp.166.0-166.8 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.19 – 8.17 (m, 2H), 8.17 – 8.14 (m, 2H), 7.20-7.06 (m, 3H), 7.01 – 7.00 (m, 2H), 7.00 – 6.98 (m, 2H), 3.91 (s, 3H), 3.90 (s, 3H), 2.25 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 165.1, 164.7, 164.11, 164.06, 148.6, 147.2, 132.5, 131.9, 124.3, 123.1, 121.9, 121.8, 120.2, 114.1, 114.0, 55.7, 16.6.

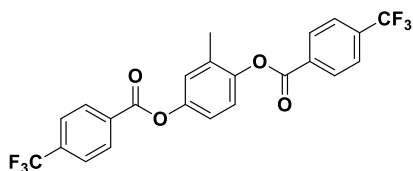


2-methyl-1,4-phenylene bis(4-fluorobenzoate) (3af). White solid, 30.5 mg, 83% yield; mp.134.8-135.7 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.27 – 8.21 (m, 4H), 7.23 – 7.07 (m, 7H), 2.26 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.39 (d, $J = 255.3$ Hz, 1C), 166.35 (d, $J = 255.2$ Hz, 1C), 164.3, 163.9, 148.5, 147.1, 133.0 (d, $J = 9.4$ Hz, 1C), 132.0, 125.8 (d, $J = 15.3$ Hz, 1C), 124.3, 123.0, 120.2,

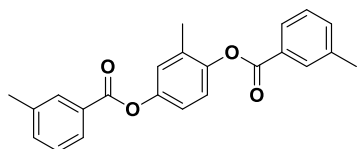
116.04 (d, $J = 22.1$ Hz, 1C), 115.97 (d, $J = 22.1$ Hz, 1C), 16.6; HRMS (ESI) m/z : $[M + Na]^+$ Calcd. for $C_{21}H_{14}F_2O_4H^+$ 391.0752; found: 391.0717.



2-methyl-1,4-phenylene bis(4-chlorobenzoate) (3ag). White solid, 26.8 mg, 67% yield; mp. 147.2-148.3 °C; 1H NMR (400 MHz, $CDCl_3$) δ 8.17 – 8.13 (m, 4H), 7.52-7.48 (m, 4H), 7.21-7.10 (m, 3H), 2.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4, 164.1, 148.5, 147.1, 140.5, 140.4, 132.0, 131.7, 130.5, 129.2, 129.1, 128.0, 127.8, 124.2, 123.0, 122.9, 120.2, 16.6; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{21}H_{14}Cl_2O_4H^+$ 401.0348; found: 401.0342.

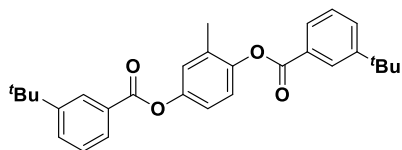


2-methyl-1,4-phenylene bis(4-(trifluoromethyl)benzoate) (3ah). Yellow solid, 38.8 mg, 83% yield; mp. 116.2-117.8 °C; 1H NMR (400 MHz, $CDCl_3$) δ 8.36 – 8.32 (m, 4H), 7.80 (t, $J = 7.0$ Hz, 4H), 7.24 (d, $J = 8.7$ Hz, 1H), 7.19 (s, 1H), 7.15 (d, $J = 8.6$ Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 163.7, 148.5, 147.1, 135.4 (q, $J = 32.6$ Hz), 135.3 (q, $J = 32.6$ Hz), 132.8, 132.6, 132.0, 130.74, 130.73, 125.8 (d, $J = 3.5$ Hz), 124.2, 123.7 (q, $J = 273.4$ Hz), 123.0, 120.2, 16.6; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{23}H_{14}F_6O_4H^+$ 469.0869; found: 469.0854.

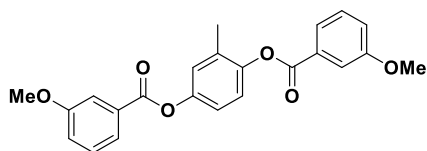


2-methyl-1,4-phenylene bis(3-methylbenzoate) (3ai). Black solid, 24.1 mg, 67% yield; mp. 122.5-123.3 °C; 1H NMR (400 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 4H), 7.48 – 7.39 (m, 4H), 7.22-7.10 (m, 3H), 2.47 (s, 3H), 2.46 (s, 3H), 2.27 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5, 165.1, 148.6,

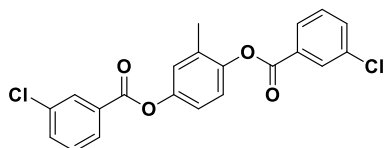
147.2, 138.64, 138.56, 134.6, 134.5, 131.9, 130.8, 129.5, 129.4, 128.7, 128.6, 127.5, 124.2, 123.0, 120.2, 21.4, 16.5; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{23}H_{20}O_4H^+$ 361.1434; found: 361.1438.



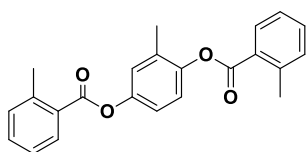
2-methyl-1,4-phenylene bis(3-(*tert*-butyl)benzoate) (3aj). Black oil, 39.9 mg, 90% yield; 1H NMR (400 MHz, $CDCl_3$) δ 8.27 (t, $J = 1.7$ Hz, 1H), 8.24 (t, $J = 1.7$ Hz, 1H), 8.08 – 8.03 (m, 2H), 7.72 – 7.68 (m, 2H), 7.49 – 7.47 (m, 1H), 7.46 – 7.44 (m, 1H), 7.22 (d, $J = 8.6$ Hz, 1H), 7.18 (d, $J = 2.6$ Hz, 1H), 7.13 (dd, $J = 8.6, 2.7$ Hz, 1H), 2.29 (s, 3H), 1.40 (s, 9H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 165.3, 151.98, 151.90, 148.6, 147.3, 132.0, 130.99, 130.91, 129.4, 129.2, 128.6, 128.5, 127.5, 127.23, 127.21, 124.3, 123.0, 120.2, 35.0, 31.4, 16.6; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{29}H_{32}O_4H^+$ 445.2373; found: 445.2383.



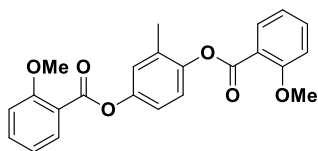
2-methyl-1,4-phenylene bis(3-methoxybenzoate) (3ak). Yellow solid, 24.7 mg, 63% yield; mp. 105.7-107.0 °C; 1H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.80 (m, 2H), 7.75-7.70 (m, 2H), 7.49-7.41 (m, 2H), 7.21 – 7.10 (m, 5H), 3.90 (s, 6H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 164.8, 159.90, 159.86, 148.6, 147.2, 132.0, 131.0, 130.7, 129.83, 129.77, 124.3, 123.0, 122.8, 120.4, 120.2, 114.7, 114.6, 55.7, 16.5; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{23}H_{20}O_6H^+$ 393.1333; found: 393.1357.



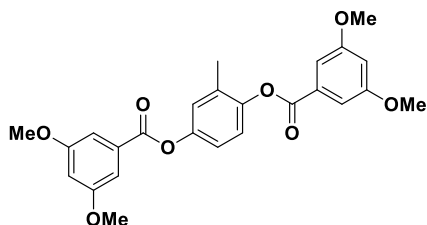
2-methyl-1,4-phenylene bis(3-chlorobenzoate) (3al). Yellow solid, 31.9 mg, 80% yield; mp.142.4-144.1 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.19 (d, *J* = 9.0 Hz, 2H), 8.10 (t, *J* = 8.6 Hz, 2H), 7.63 (s, 2H), 7.48 – 7.47 (m, 2H), 7.20 (d, *J* = 8.5 Hz, 1H), 7.16 (s, 1H), 7.12 (d, *J* = 8.2 Hz, 1H), 2.26 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 164.1, 163.7, 148.5, 147.1, 135.0, 134.9, 133.9, 133.8, 132.0, 131.3, 131.1, 130.3, 130.2, 130.14, 130.07, 128.4, 124.2, 123.0, 120.2, 16.6; HRMS (ESI) *m/z*: [M + H]⁺ Calcd. for C₂₁H₁₄Cl₂O₄H⁺ 401.0342; found: 401.0347.



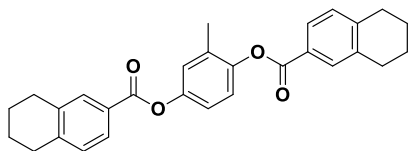
2-methyl-1,4-phenylene bis(2-methylbenzoate) (3am). Yellow solid, 29.8 mg, 83% yield; mp.93.1-94.2 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.10 – 8.05 (m, 4H), 7.53 – 7.44 (m, 4H), 7.25-7.11 (m, 3H), 2.52 (s, 3H), 2.51 (s, 3H), 2.32 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.0, 165.6, 148.5, 147.2, 141.6, 141.5, 133.0, 132.9, 132.2, 132.1, 132.0, 131.33, 131.30, 128.6, 128.5, 126.12, 126.08, 124.4, 123.1, 120.3, 22.1, 16.7; HRMS (ESI) *m/z*: [M + H]⁺ Calcd. for C₂₃H₂₀O₄H⁺ 361.1434; found: 361.1436.



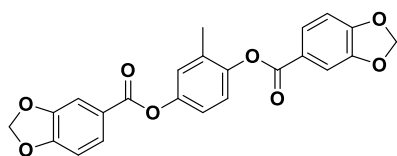
2-methyl-1,4-phenylene bis(2-methoxybenzoate) (3an). Black solid, 27.8 mg, 71% yield; mp.95.6-96.7 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.03 (td, *J* = 8.0, 1.8 Hz, 2H), 7.58 – 7.53 (m, 2H), 7.21-7.15 (m, 2H), 7.12 – 7.08 (m, 1H), 7.08 – 7.03 (m, 4H), 3.95 (s, 3H), 3.95 (s, 3H), 2.29 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 164.5, 164.4, 160.0, 156.0, 148.5, 147.1, 134.4, 132.34, 132.30, 131.8, 124.3, 123.1, 120.40, 120.36, 120.2, 119.2, 112.36, 112.35, 56.2, 56.1, 16.7; HRMS (ESI) *m/z*: [M + H]⁺ Calcd. for C₂₃H₂₀O₆H⁺ 393.1333; found: 445.1343.



2-methyl-1,4-phenylene bis(3,5-dimethoxybenzoate) (3ao). White solid, 37.1 mg, 82% yield; mp.138.0-139.8 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.37 (d, $J = 2.3$ Hz, 2H), 7.34 (d, $J = 2.3$ Hz, 2H), 7.20 (d, $J = 8.6$ Hz, 1H), 7.15 (d, $J = 2.5$ Hz, 1H), 7.10 (dd, $J = 8.6, 2.6$ Hz, 1H), 6.73 (dt, $J = 4.8, 2.3$ Hz, 2H), 3.87 (s, 6H), 3.87 (s, 6H), 2.26 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.1, 164.7, 160.97, 160.92, 148.6, 147.2, 132.0, 131.4, 131.2, 124.2, 123.0, 120.2, 107.9, 107.8, 106.6, 106.5, 55.8, 16.5; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd. for $\text{C}_{25}\text{H}_{24}\text{O}_8\text{H}^+$ 453.1544; found: 453.1554.

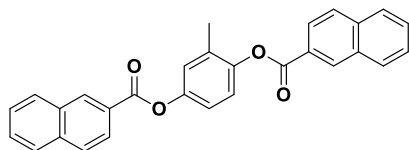


2-methyl-1,4-phenylene bis(5,6,7,8-tetrahydronaphthalene-2-carboxylate) (3ap). Yellow solid, 35.7 mg, 81% yield; mp.127.7-129.1 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.94 – 7.89 (m, 4H), 7.21-7.08 (m, 5H), 2.86 (s, 8H), 2.25 (s, 3H), 1.85 (s, 8H); ^{13}C NMR (100MHz, CDCl_3) δ 165.6, 165.2, 148.6, 147.2, 143.6, 143.9, 137.8, 137.7, 132.0, 131.2, 129.6, 129.5, 127.3, 126.7, 126.6, 124.3, 123.0, 120.2, 29.9, 29.5; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd. for $\text{C}_{29}\text{H}_{28}\text{O}_4\text{H}^+$ 441.2060; found: 441.2065.

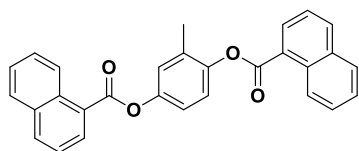


2-methyl-1,4-phenylene bis(benzo[d][1,3]dioxole-5-carboxylate) (3aq). Yellow solid, 35.7 mg, 85% yield; mp.149.6-151.7 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.83 (t, $J = 9.2$ Hz, 2H), 7.62 (d, $J = 9.5$ Hz, 2H), 7.20-7.05 (m, 3H), 6.93 – 6.90(m, 2H), 6.09 (s, 4H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 164.6, 164.3, 152.42, 152.37, 148.5, 148.10, 148.06, 147.1, 131.9, 126.4, 124.2, 123.5,

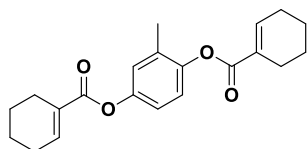
123.3, 123.0, 120.1, 110.1, 108.4, 108.3, 102.14, 102.13, 16.5; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{23}H_{16}O_8H^+$ 421.0918; found: 421.0945.



2-methyl-1,4-phenylene bis(2-naphthoate) (3ar). White solid, 32.8 mg, 76% yield; mp.180.8-181.7 °C; 1H NMR (400 MHz, $CDCl_3$) δ 9.08 (t, $J = 8.3$ Hz, 2H), 8.59-8.46 (m, 2H), 8.16-8.10 (m, 2H), 7.97-7.90 (m, 2H), 7.71-7.56 (m, 6H), 7.35-7.21 (m, 3H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5, 165.1, 148.7, 147.3, 136.03, 136.0, 132.67, 132.66, 132.13, 132.07, 129.7, 128.9, 128.8, 128.64, 128.58, 128.0, 127.1, 127.0, 126.8, 126.6, 125.6, 124.3, 123.1, 120.3, 16.8; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{29}H_{20}O_4H^+$ 433.1434; found: 433.1445.

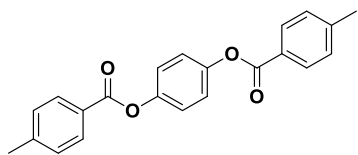


2-methyl-1,4-phenylene bis(1-naphthoate) (3as). White solid, 32.8 mg, 76% yield; mp.140.1-141.6 °C; 1H NMR (400 MHz, $CDCl_3$) δ 9.07 (t, $J = 8.3$ Hz, 2H), 8.57-8.46 (m, 2H), 8.16-8.09 (m, 2H), 7.97-7.92 (m, 2H), 7.71-7.56 (m, 6H), 7.33-7.21 (m, 3H), 7.2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 163.7, 148.5, 147.1, 132.8, 132.6, 132.0, 130.8, 130.7, 125.91, 125.87, 125.84, 125.80, 125.0, 124.3, 123.0, 122.33, 122.27, 120.2, 16.6; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{29}H_{20}O_4H^+$ 433.1434; found: 433.1433.

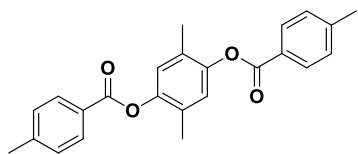


1,1,4-trimethyl-5-phenyl-7-(*o*-tolyl)-1,3-dihydroisobenzofuran (3at). Black oil, 15.5 mg, 57% yield; 1H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.23 (m, 1H), 7.23 – 7.21 (m, 1H), 7.03 (d, $J = 8.6$ Hz, 1H), 6.98 (d, $J = 2.6$ Hz, 1H), 6.94 (dd, $J = 8.6, 2.7$ Hz, 1H), 2.39 – 2.36 (m, 4H), 2.29 – 2.27 (m,

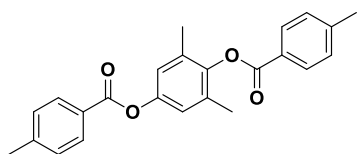
4H), 2.17 (s, 3H), 1.73 – 1.70 (m, 4H), 1.69 – 1.65 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.1, 165.8, 148.4, 147.0, 142.09, 142.08, 131.7, 130.0, 129.9, 124.1, 122.9, 120.0, 26.2, 24.39, 24.35, 22.2, 21.5, 16.5; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{24}\text{O}_4\text{H}^+$ 341.1747; found: 341.1750.



1,4-phenylene bis(4-methylbenzoate) (3bb).³ Black oil, 20.4 mg, 59% yield; ^1H NMR (400 MHz, CDCl_3) δ 8.11 (s, 2H), 8.09 (s, 2H), 7.33 (s, 2H), 7.31 (s, 2H), 7.27 (s, 4H), 2.46 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.3, 148.6, 144.7, 130.4, 129.5, 126.8, 122.8, 21.9.

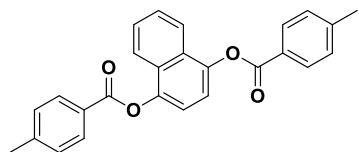


2,5-dimethyl-1,4-phenylene bis(4-methylbenzoate) (3cb). Yellow solid, 29.9 mg, 80% yield; mp.189.1-189.5 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.11 (d, $J = 8.2$ Hz, 4H), 7.32 (d, $J = 8.0$ Hz, 4H), 7.06 (s, 2H), 2.46 (s, 6H), 2.20 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.1, 147.1, 144.6, 130.4, 129.5, 129.1, 126.8, 124.4, 21.9, 16.2; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd. for $\text{C}_{24}\text{H}_{22}\text{O}_4\text{H}^+$ 375.1591; found: 375.1595.

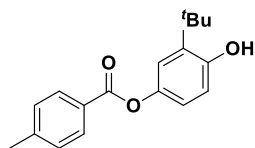


2,6-dimethyl-1,4-phenylene bis(4-methylbenzoate) (3db). Yellow solid, 26.1 mg, 70% yield; mp.148.7-149.5 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.14 (d, $J = 8.2$ Hz, 2H), 8.09 (d, $J = 8.2$ Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 6.97 (s, 2H), 2.47 (s, 3H), 2.46 (s, 3H), 2.21 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.5, 164.5, 148.3, 146.0, 144.7, 144.5, 132.0, 130.4, 130.3,

129.5, 129.4, 127.0, 126.5, 121.6, 22.0, 16.8; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{24}H_{22}O_4H^+$ 375.1591; found: 375.1595.



naphthalene-1,4-diyl bis(4-methylbenzoate) (3eb). Black solid, 20.9 mg, 53% yield; mp.166.6-167.3 °C; 1H NMR (400 MHz, $CDCl_3$) δ 8.26 (s, 2H), 8.24 (s, 2H), 7.98 (dd, $J = 6.5, 3.2$ Hz, 2H), 7.54 (dd, $J = 6.5, 3.2$ Hz, 2H), 7.44 (s, 2H), 7.40 (s, 2H), 7.38 (s, 2H), 2.50 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 144.7, 144.6, 130.4, 129.5, 127.9, 126.9, 126.5, 121.8, 118.0, 21.8; HRMS (ESI) m/z : $[M + H]^+$ Calcd. for $C_{26}H_{20}O_4H^+$ 397.1434; found: 397.1440.



3-(*tert*-butyl)-4-hydroxyphenyl 4-methylbenzoate (3fb).⁴ Black solid, 9.1 mg, 40% yield; mp.162.5-163.7 °C; 1H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, $J = 8.2$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 7.06 (d, $J = 2.8$ Hz, 1H), 6.89 (dd, $J = 8.5, 2.8$ Hz, 1H), 6.65 (d, $J = 8.5$ Hz, 1H), 5.17 (s, 1H), 2.45 (s, 3H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2, 152.2, 144.5, 144.2, 137.6, 130.3, 129.4, 127.1, 120.4, 119.8, 117.1, 34.9, 29.52, 29.48, 22.0.

4. References

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5. ¹H, ¹³C spectra of products (3aa–at,3bb–fb)

