Supporting information

for

Facile Dearomatizing Radical Arylation of Furan and Thiophene

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Experimental part

General Procedure:

A solution of aryl iodide (1.0 mmol), diphenyl diselenide (62.0 mg, 0.2 mmol) and freshly distilled furan (thiophene or pyrrole) (20 mL) is sparged with argon for 45 min, followed by heating at 50 °C (bath temperature) with stirring. A solution of 2,2'-azobis(4-methoxy-2,4-dimethylvaleronitrile) (V-70) (62.0 mg, 0.2 mmol) and Bu₃SnH (0.47 mL, 1.75 mmol) in degassed furan (thiophene or pyrrole) (10 mL) is added via syringe pump over 16 h, after which heating is continued for 1 h, before the reaction mixture is cooled to room temperature and concentrated under vacuum. The residue is taken up in acetonitrile (75 mL) and washed with (4 × 25 mL) of hexane. The acetonitrile phase is concentrated and purified by silica gel chromatography (eluent: EtOAc/hexane).

2,3,4,5-Tetrahydro-2,5-epoxy-1-benzoxepin (2)¹

¹H NMR (CDCl₃, 500 MHz) δ: 2.19-2.28 (m, 4H), 5.12 (d, *J* = 4.5 Hz, 1H), 5.94 (m, 1H), 6.75 (d, *J* = 8.5 Hz, 1H), 6.84 (dt, *J* = 7.5, 1.5 Hz, 1H), 6.94 (dd, *J* = 7.0, 2.0 Hz, 1H), 7.15 (dt, *J* = 7.5, 2.0 Hz, 1H); ¹³C NMR (CDCl₃, 125 MHz) δ: 33.4, 35.7, 100.4, 116.1, 120.1, 124.4, 127.2, 128.7, 150.0.

2-(2,5-Dihydrofuran-2-yl)phenol (3)

¹H NMR (CDCl₃, 500 MHz) δ: 4.79 (m, 1H), 4.86 (m, 1H), 6.07 (m, 3H), 6.86 (m, 2H), 7.02 (ddd, *J* = 7.5, 2.0, 1.0 Hz, 1H), 7.17 (dt, *J* = 7.5, 1.5 Hz, 1H), 7.98 (s, 1H); ¹³C NMR (CDCl₃, 125 MHz) δ: 75.5, 87.2, 117.1, 119.9, 124.8, 126.5, 126.6, 128.8, 129.0, 155.2; *m/z* (ESI) (*m* + Na): Calcd 185.0579, Found 185.0570.

9-Methoxy-2,3,4,5-tetrahydro-2,5-epoxy-1-benzoxepin-7-carbaldehyde (5)

Recrystallize from EtOAc/hexane; m.p 100-102° C; ¹H NMR (CDCl₃, 500 MHz) δ: 2.25-2.35 (m, 4H), 3.90 (s, 3H), 5.20 (d, *J* = 5.5 Hz, 1H), 6.09 (d, *J* = 4.0 Hz, 1H), 7.14 (d, *J* = 1.0 Hz, 1H), 7.30 (d, *J* = 1.5 Hz, 1H), 9.79 (s, 1H); ¹³C NMR (CDCl₃, 125 MHz) δ: 34.0, 35.3, 56.1, 77.0, 101.5, 109.7, 121.1, 127.8, 129.1, 145.2, 148.5, 190.6; Anal. Calcd for C₁₂H₁₂O₄: C, 65.45; H, 5.49 Found C, 65.36; H, 5.57.

3-(2,5-Dihydrofuran-2-yl)-4-hydroxy-5-methoxybenzaldehyde (6)

HPLC Eluent: (EtoAc/hexane); ^1H NMR (CDCl_3 , 500 MHz) δ : 3.95 (s, 3H), 4.83 (dd, J = 12.0, 3.5 Hz, 1H), 4.92 (dd, J = 14.0, 6.5 Hz, 1H), 6.03 (s, 2H), 6.16 (q, J = 4.5 Hz, 1H), 6.70 (s, 1H), 7.35 (d, J = 2.0 Hz, 1H), 7.47 (d, J = 1.5 Hz, 1H), 9.82 (s, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 56.3, 75.8, 82.6, 107.7, 124.7, 126.7, 128.0, 128.4, 129.3, 147.1, 148.4, 191.1; m/z (ESI) ($\text{M}+\text{Na}$): Calcd 243.0634, Found 243.0627.

2-(2,3-Dihydrofuran-2-yl)benzonitrile (8)

^1H NMR (CDCl_3 , 500 MHz) δ : 2.53 (m, 1H), 3.30 (m, 1H), 4.99 (q, J = 2.0, 1H), 5.83 (dd, J = 11.0, 7.5 Hz, 1H), 6.48 (q, J = 2.5, 1H), 7.38 (dt, J = 7.0, 2.0 Hz, 1H), 7.59 (m, 2H), 7.65 (dd, J = 7.5, 1.0 Hz, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 37.8, 79.8, 99.2, 109.6, 117.3, 125.8, 127.9, 132.9, 133.1, 145.2, 147.3; m/z (ESI) ($\text{M}+\text{Na}$): Calcd 194.0582, Found 194.0590.

2-(2,5-Dihydrofuran-2-yl)benzonitrile (9)

^1H NMR (CDCl_3 , 500 MHz) δ : 4.84 (m, 1H), 4.98 (m, 1H), 5.92 (m, 1H), 6.10 (m, 2H), 7.37 (dt, J = 7.5, 1.5 Hz, 1H), 7.50 (dd, J = 8.0, 1.0 Hz, 1H), 7.58 (dt, J = 7.5, 1.5 Hz, 1H), 7.66 (dd, J = 7.5, 1.0 Hz, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 76.5, 86.1, 110.1, 117.4, 127.1, 127.8, 128.1, 133.0, 133.3, 146.2; m/z (ESI) ($\text{M}+\text{Na}$): Calcd 194.0582, Found 194.0577.

Methyl 2-(2,3-Dihydrofuran-2-yl)benzoate (11)

Eluent: 1:1(1 % EtoAc/hexane : Benzene); ^1H NMR (CDCl_3 , 500 MHz) δ : 2.37 (m, 1H), 3.34 (m, 1H), 3.90 (s, 3H), 4.90 (q, J = 2.5, 1H), 6.26 (dd, J = 11.5, 7.5 Hz, 1H), 6.51 (q, J = 3.0 Hz, 1H), 7.33 (dt, J = 8.0, 1.5 Hz, 1H), 7.54 (dt, J = 7.5, 1.0 Hz, 1H), 7.63 (dd, J = 7.5, 1.0 Hz, 1H), 7.98 (dd, J = 7.5, 1.0 Hz, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 38.6, 52.1, 79.7, 99.0, 125.5, 126.8, 126.9, 130.7, 132.7, 145.1, 145.8, 167.2; m/z (ESI) ($\text{M}+\text{Na}$): Calcd 227.0684, Found 227.0691.

Methyl 2-(2,5-Dihydrofuran-2-yl)benzoate (12)

Eluent: 1:1(3 % EtoAc/hexane : Benzene); ^1H NMR (CDCl_3 , 500 MHz) δ : 3.90 (s, 3H), 4.85 (m, 1H), 4.95 (m, 1H), 5.92 (m, 1H), 6.04 (m, 1H), 6.50 (m, 1H), 7.31 (dt, J = 7.5, 1.0 Hz, 1H), 7.53 (dt, J = 7.5, 1.5 Hz, 1H), 7.63 (dd, J = 7.0, 1.0 Hz, 1H), 7.92 (dd, J = 8.0, 1.0 Hz, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 52.1, 76.2, 85.4, 125.5, 126.6, 127.0, 127.3, 130.2, 130.9, 132.8, 145.1, 167.5; m/z (ESI) ($\text{M}+\text{Na}$): Calcd 227.0684, Found 227.0686.

Methyl [2-(2,3-Dihydrofuran-2-yl)phenyl]carbamate (14)

Eluent: 1.5:1(4 % EtoAc/hexane : Chloroform); ^1H NMR (CDCl_3 , 500 MHz) δ : 2.78 (m, 1H), 2.92 (m, 1H), 3.77 (s, 3H), 5.15 (q, J = 2.5 Hz, 1H), 5.53 (t, J = 10.5 Hz, 1H), 6.45 (q, J = 2.5 Hz, 1H), 7.08 (t, J = 7.5 Hz, 1H), 7.23 (d, J = 7.0 Hz, 1H), 7.34 (dt, J = 7.5, 1.5 Hz, 1H), 7.38 (br s, 1H), 7.95 (br s, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 35.0, 52.4, 82.8, 101.4, 121.9, 123.7, 127.7, 129.1, 136.6, 144.6, 154.3; m/z (ESI) ($\text{M}+\text{Na}$): Calcd 242.0793, Found 242.0794.

Methyl [2-(2,5-Dihydrofuran-2-yl)phenyl]carbamate (15)

^1H NMR (CDCl_3 , 500 MHz) δ : 3.77 (s, 3H), 4.79 (m, 2H), 5.89 (m, 1H), 6.04 (m, 1H), 6.13 (m, 1H), 7.04 (t, J = 8.0 Hz, 1H), 7.12 (dd, J = 7.5, 1.5 Hz, 1H), 7.31 (dt, J = 9.0, 1.5 Hz, 1H),

7.82 (br s, 1H), 7.93 (br s, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 52.3, 75.3, 85.9, 121.4, 123.4, 127.0, 127.7, 128.1, 128.9, 137.1, 154.3; m/z (ESI) (M+H): Calcd 220.0974, Found 220.0967.

2,3,4,5-Tetrahydro-2,5-epithio-1-benzoxepin (16)

Eluent: 1:1:1(Benzene : Chloroform : Hexane); ^1H NMR (CDCl_3 , 500 MHz) δ : 2.26-2.53 (m, 4H), 4.36 (d, $J = 5.0$ Hz, 1H), 6.19 (d, $J = 4.5$ Hz, 1H), 6.81 (m, 2H), 6.99 (dd, $J = 7.5$, 2.0 Hz, 1H), 7.13 (dt, $J = 7.5$, 1.5 Hz, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 38.0, 40.4, 49.9, 86.9, 117.6, 120.1, 126.0, 128.6, 129.5, 151.3; m/z (ESI) (M+H): Calcd 179.0531, Found 179.0536.

2-(2,5-Dihydrothiophen-2-yl)phenol (17)

Recrystallize from EtoAc/Hexane; m.p 68°C ; ^1H NMR (CDCl_3 , 500 MHz) δ : 3.95 (m, 2H), 5.57 (m, 1H), 5.85 (m, 1H), 6.03 (m, 1H), 6.40 (br s, 1H), 6.85 (d, $J = 8.5$ Hz, 1H), 6.88 (dt, $J = 7.5$, 1.0 Hz, 1H), 7.15 (dd, $J = 7.5$, 2.0 Hz, 1H), 7.19 (dt, $J = 7.0$, 1.5 Hz, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 39.6, 56.0, 117.2, 120.7, 126.1, 129.2, 129.2, 129.4, 132.5, 155.0; Anal. Calcd for $\text{C}_{10}\text{H}_{10}\text{OS}$: C, 67.38; H, 5.65 Found C, 67.16; H, 5.44.

2-(2,3-Dihydrothiophen-2-yl)benzonitrile (18)

^1H NMR (CDCl_3 , 500 MHz) δ : 2.89 (m, 1H), 3.35(m, 1H), 5.27 (dd, $J = 10.0$, 5.5 Hz, 1H), 5.58 (m, 1H), 6.25 (m, 1H), 7.36 (m, 1H), 7.62 (dd, $J = 7.5$, 1.0 Hz, 1H), 7.75 (d, $J = 8.0$ Hz, 1H), 7.26- 7.66 (1H).

2-(2,3-Dihydrothiophen-3-yl)benzonitrile (19)

^1H NMR (CDCl_3 , 500 MHz) δ : 3.13 (ddd, $J = 11.0$, 6.5, 2.0 Hz, 1H), 3.83 (dt, $J = 11.5$, 2.0 Hz, 1H), 4.73 (m, 1H), 5.65 (m, 1H), 6.47 (m, 1H), 7.35 (t, $J = 7.0$ Hz, 1H), 7.53 (d, $J = 8.0$ Hz, 1H), 7.57 (t, $J = 8.0$ Hz, 1H), 7.66 (d, $J = 7.5$ Hz, 1H).

2-(2,3-Dihydrothiophen-2-yl)benzonitrile (18) & 2-(2,3-Dihydrothiophen-3-yl)benzonitrile (19)

^{13}C NMR (CDCl_3 , 125 MHz) δ : 39.5, 43.6, 49.3, 51.0, 111.1, 111.7, 117.5, 117.8, 120.1, 123.9, 125.4, 127.6, 127.6, 127.7, 129.3, 132.7, 133.0, 133.32, 133.36, 146.6, 147.5; m/z (ESI) (M+Na): Calcd 210.0354, Found 210.0352.

2-(2,5-Dihydrothiophen-2-yl)benzonitrile (20)

Recrystallize from EtoAc/hexane; m.p 80°C ^1H NMR (CDCl_3 , 500 MHz) δ : 3.93 (m, 1H), 4.03 (m, 1H), 5.70 (m, 1H), 5.78 (m, 1H), 6.08 (m, 1H), 7.32 (dt, $J = 8.0$, 1.5 Hz, 1H), 7.48 (dd, $J = 7.5$, 1.0 Hz, 1H), 7.55 (dt, $J = 7.5$, 1.5 Hz, 1H), 7.62 (dd, $J = 7.5$, 1.5 Hz, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 40.3, 56.5, 111.5, 117.4, 127.7, 128.9, 130.6, 130.9, 133.1, 133.1, 147.3; Anal. Calcd for $\text{C}_{11}\text{H}_9\text{NS}$: C, 70.55; H, 4.84 Found C, 70.47; H, 4.88.

Methyl 2-(2,3-Dihydrothiophen-2-yl)benzoate (21)

Eluent: 1:1(Benzene : Hexane); ^1H NMR (CDCl_3 , 500 MHz) δ : 2.89 (m, 1H), 3.31 (m, 1H), 3.91 (s, 3H), 5.58 (m, 1H), 5.69 (dd, $J = 10.0$, 4.5 Hz, 1H), 6.23 (m, 1H), 7.29 (dt, $J = 7.5$, 1.5

Hz, 1H), 7.49 (dt, $J = 7.5, 1.5$ Hz, 1H), 7.76 (dd, $J = 8.0, 1.0$ Hz, 1H), 7.85 (dd, $J = 8.0, 1.5$ Hz, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 43.3, 47.9, 52.2, 120.4, 125.5, 126.9, 127.8, 128.2, 130.2, 132.6, 145.3, 167.9; m/z (ESI) (M+H): Calcd 221.0636, Found 221.0635.

Methyl 2-(2,5-Dihydrothiophen-2-yl)benzoate (22)

Eluent: 1:1(Benzene : Hexane); ^1H NMR (CDCl_3 , 500 MHz) δ : 3.81-3.93 (m, 2H), 3.90 (s, 3H), 5.85 (m, 1H), 6.05 (m, 1H), 6.13 (m, 1H), 7.27 (dt, $J = 6.5, 1.5$ Hz, 1H), 7.48 (m, 2H), 7.85 (d, $J = 9.0$ Hz, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 39.1, 52.2, 54.8, 126.9, 128.7, 128.7, 129.5, 130.2, 132.5, 132.5, 144.9, 167.8; m/z (ESI) (M+H): Calcd 221.0636, Found 221.0636.

Methyl [2-(2,3-Dihydrothiophen-2-yl)phenyl]carbamate (23)

Eluent: 1:1(4 % EtOAc/hexane : Benzene); ^1H NMR (CDCl_3 , 500 MHz) δ : 2.97 (m, 1H), 3.17 (m, 1H), 3.77 (s, 3H), 5.09 (dd, $J = 10.5, 8.0$ Hz, 1H), 5.68 (m, 1H), 6.23 (m, 1H), 7.06 (t, $J = 7.0$ Hz, 1H), 7.15 (br s, 1H), 7.29 (m, 2H), 7.81 (br s, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 41.8, 50.2, 52.5, 122.3, 123.3, 124.4, 124.9, 128.5, 129.0, 136.0, 154.5; m/z (ESI) (M+Na): Calcd 258.0565, Found 258.0558.

Methyl [2-(2,5-Dihydrothiophen-2-yl)phenyl]carbamate (24)

^1H NMR (CDCl_3 , 500 MHz) δ : 3.75 (s, 3H), 3.94 (br m, 2H), 5.50 (br s, 1H), 5.81 (m, 1H), 6.03 (m, 1H), 7.08 (t, $J = 7.0$ Hz, 1H), 7.20 (d, $J = 7.5$ Hz, 1H), 7.22 (br s, 1H), 7.27 (dt, $J = 9.5, 2.0$ Hz, 1H), 7.73 (br s, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 39.8, 52.4, 56.7, 123.5, 124.6, 128.6, 129.4, 129.5, 132.2, 136.2, 154.5; m/z (ESI) (M+Na): Calcd 258.0565, Found 258.0568.

2-(2-Hydroxyphenyl)-1H-pyrrole (25)²

^1H NMR (CDCl_3 , 500 MHz) δ : 5.52 (s, 1H), 6.33 (d, $J = 3.0$ Hz, 1H), 6.58 (s, 1H), 6.85 (d, $J = 8.0$ Hz, 1H), 6.89 (s, 1H), 6.97 (t, $J = 7.0$ Hz, 1H), 7.09 (t, $J = 7.5$ Hz, 1H), 7.55 (d, $J = 8.0$ Hz, 1H), 9.42 (br s, 1H); ^{13}C NMR (CDCl_3 , 125 MHz) δ : 106.2, 109.2, 116.3, 118.6, 119.6, 121.5, 127.0, 127.2, 128.8, 151.0.

References

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- 2) Xu, Z.; Lu, X. *J. Org. Chem.* **1998**, 63, 5031.

2,3,4,5-Tetrahydro-2,5-epoxy-1-benzoxepin (2)

2,3,4,5-Tetrahydro-2,5-epoxy-1-benzoxepin (2)

2-(2,5-Dihydrofuran-2-yl)phenol (3)

2-(2,5-Dihydrofuran-2-yl)phenol (3)

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9-Methoxy-2,3,4,5-tetrahydro-2,5-epoxy-1-benzoxepin-7-carbaldehyde (5)

9-Methoxy-2,3,4,5-tetrahydro-2,5-epoxy-1-benzoxepin-7-carbaldehyde (5)

3-(2,5-Dihydrofuran-2-yl)-4-hydroxy-5-methoxybenzaldehyde (6)

3-(2,5-Dihydrofuran-2-yl)-4-hydroxy-5-methoxybenzaldehyde (6)

2-(2,3-Dihydrofuran-2-yl)benzonitrile (8)

2-(2,3-Dihydrofuran-2-yl)benzonitrile (8)

2-(2,5-Dihydrofuran-2-yl)benzonitrile (9)

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Methyl 2-(2,3-Dihydrofuran-2-yl)benzoate (11)

Methyl 2-(2,3-Dihydrofuran-2-yl)benzoate (11)

Methyl 2-(2,5-Dihydrofuran-2-yl)benzoate (12)

Methyl 2-(2,5-Dihydrofuran-2-yl)benzoate (12)

Methyl [2-(2,3-Dihydrofuran-2-yl)phenyl]carbamate (14)

Methyl [2-(2,3-Dihydrofuran-2-yl)phenyl]carbamate (14)

Methyl [2-(2,5-Dihydrofuran-2-yl)phenyl]carbamate (15)

Methyl [2-(2,5-Dihydrofuran-2-yl)phenyl]carbamate (15)

2,3,4,5-Tetrahydro-2,5-epithio-1-benzoxepin (16)

2,3,4,5-Tetrahydro-2,5-epithio-1-benzoxepin (16)

2-(2,5-Dihydrothiophen-2-yl)phenol (17)

2-(2,5-Dihydrothiophen-2-yl)phenol (17)

2-(2,3-Dihydrothiophen-2-yl)benzonitrile (18) & 2-(2,3-Dihydrothiophen-3-yl)benzonitrile (19)

2-(2,3-Dihydrothiophen-2-yl)benzonitrile (18) & 2-(2,3-Dihydrothiophen-3-yl)benzonitrile (19)

2-(2,3-Dihydrothiophen-3-yl)benzonitrile (19)

2-(2,5-Dihydrothiophen-2-yl)benzonitrile (20)

2-(2,5-Dihydrothiophen-2-yl)benzonitrile (20)

Methyl 2-(2,3-Dihydrothiophen-2-yl)benzoate (21)

Methyl 2-(2,3-Dihydrothiophen-2-yl)benzoate (21)

Methyl 2-(2,5-Dihydrothiophen-2-yl)benzoate (22)

Methyl 2-(2,5-Dihydrothiophen-2-yl)benzoate (22)

Methyl [2-(2,3-Dihydrothiophen-2-yl)phenyl]carbamate (23)

Methyl [2-(2,3-Dihydrothiophen-2-yl)phenyl]carbamate (23)

Methyl [2-(2,5-Dihydrothiophen-2-yl)phenyl]carbamate (24)

Methyl [2-(2,5-Dihydrothiophen-2-yl)phenyl]carbamate (24)

2-(2-Hydroxyphenyl)-1*H*-pyrrole (25)

2-(2-Hydroxyphenyl)-1*H*-pyrrole (25)