Supporting Information

Highly Effective Synthetic Methods for Substituted 2-Arylbenzofurans Using [3,3]-Sigmatropic Rearrangement: Short Syntheses of Stemofuran A and Eupomatenoid 6

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General Procedure for Preparation of the Oxime Ethers 4.

To a solution of *O*-phenylhydroxylamine **7** (1.94 mmol) in EtOH (3.6 ml) were added an appropriate acetophenone or propiophenone (1.94 mmol) and c. HCl (0.10 ml) under a nitrogen atmosphere at room temperature. After being stirred at the same temperature for 2 h, the reaction mixture was concentrated at reduced pressure. Purification of the residue by medium-pressure column chromatography (*n*-hexane/AcOEt 10:1) afforded the oxime ethers **4** as shown in Table 1.

(E)-1-(4-Bromophenyl)ethanone O-Phenyloxime (4b): a colorless oil:

¹H-NMR (CDCl₃, 300 MHz) δ 7.65 (2H, br d, *J*=8.5 Hz), 7.54 (2H, br d, *J*=8.5 Hz), 7.36-7.25 (4H, m), 7.04 (1H, tt, *J*=8, 1.5 Hz), 2.42 (3H, s);

¹³C-NMR (CDCl₃, 50 MHz) δ 159.4, 156.6, 134.8, 131.6, 129.3, 127.9, 124.0, 122.3, 114.7, 13.0;

HRMS (EI) *m/z* calcd for C₁₄H₁₂⁷⁹BrNO (M⁺) 289.0102, found 289.0099.

(E)-1-(4-Nitrophenyl)ethanone O-Phenyloxime (4c): colorless crystals: mp 78-79°C (*n*-hexane/AcOEt); ¹H-NMR (CDCl₃, 300 MHz)

δ 8.25 (2H, br d, *J*=8.5 Hz), 7.94 (2H, br d, *J*=8.5 Hz), 7.39-7.27 (4H, m),

7.08 (1H, br t, *J*=8 Hz), 2.48 (3H, s); ¹³C-NMR (CDCl₃, 50 MHz) δ 159.2, 155.7, 148.4, 141.9, 129.4, 127.2, 123.7, 122.8, 114.8, 13.2; IR (CHCl₃) 1523, 1348 cm⁻¹; HRMS (EI) *m/z* calcd for

C₁₄H₁₂N₂O₃ (M⁺) 256.0847, found 256.0850; *Anal.* calcd for C₁₄H₁₂N₂O₃: C, 65.62; H, 4.72; N, 10.93, found C, 65.64; H, 4.88; N, 10.89.

(E)-1-(4-Hydroxyphenyl)ethanone O-Phenyloxime (4d): colorless crystals: mp 85-86°C (*n*-hexane/AcOEt); $^1\text{H-NMR}$ (CDCl_3 , 300 MHz) δ 7.69 (2H, br d, $J=8.5$ Hz), 7.35-7.25 (4H, m), 7.02 (1H, tt, $J=8, 1.5$ Hz), 6.86 (2H, br d, $J=8.5$ Hz), 5.04 (1H, br s), 2.41 (3H, s); $^{13}\text{C-NMR}$ (CDCl_3 , 50 MHz) δ 159.5, 157.9, 157.0, 129.2, 128.3, 128.1, 122.1, 115.4, 114.8, 13.3; IR (CHCl_3) 3293 cm^{-1} ; HRMS (EI) m/z calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_2$ (M^+) 227.0946, found 227.0944; *Anal.* calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_2$: C, 73.99; H, 5.77; N, 6.16, found C, 73.97; H, 5.85; N, 6.13.

(E)-1-(4-Methoxyphenyl)ethanone O-Phenyloxime (4e): colorless crystals: mp 32-34°C (*n*-hexane/AcOEt); $^1\text{H-NMR}$ (CDCl_3 , 300 MHz) δ 7.73 (2H, br d, $J=8.5$ Hz), 7.35-7.24 (4H, m), 7.01 (1H, tt, $J=8, 1.5$ Hz), 6.92 (2H, br d, $J=8.5$ Hz), 3.83 (3H, s), 2.41 (3H, s); $^{13}\text{C-NMR}$ (CDCl_3 , 50 MHz) δ 160.9, 159.6, 157.2, 129.2, 128.4, 127.8, 121.9, 114.7, 113.8, 55.3, 13.1; HRMS (EI) m/z calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_2$ (M^+) 241.1102, found 241.1080; *Anal.* calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_2$: C, 74.67; H, 6.27; N, 5.81, found C, 74.76; H, 6.33; N, 5.79.

(*E:Z*=10:1)

(E/Z)-1-(4-Bromophenyl)-1-propanone O-Phenyloxime (4g)
(*E:Z*=10:1): a colorless oil: $^1\text{H-NMR}$ (CDCl_3 , 300 MHz) δ 7.64 (2H, br d, $J=8.5$ Hz), 7.54 (2H, br d, $J=8.5$ Hz), 7.33 (2H, br t, $J=8$ Hz), 7.27 (2H, br d, $J=8$ Hz), 7.04 (1H, br tt, $J=8, 1.5$ Hz), 2.92 (20/11H, q, $J=7.5$ Hz), 2.67 (2/11H, q, $J=7.5$ Hz), 1.23 (30/11H, t, $J=7.5$ Hz), 1.16 (3/11H, t, $J=7.5$ Hz); $^{13}\text{C-NMR}$ (CDCl_3 , 75 MHz) δ 161.7, 159.5, 133.8, 131.7, 129.3, 128.2, 124.0, 122.3, 114.8, 20.4, 11.3; HRMS (EI) m/z calcd for $\text{C}_{15}\text{H}_{14}^{79}\text{BrNO}$ (M^+) 303.0258, found 302.0262.

(*E:Z*=10:1)

(E/Z)-1-(4-Hydroxyphenyl)-1-propanone O-Phenyloxime (4h)
(*E:Z*=10:1): a colorless oil: $^1\text{H-NMR}$ (CDCl_3 , 300 MHz) δ 7.66 (2H, br d, $J=8.5$ Hz), 7.36-7.24 (4H, m), 7.02 (1H, br tt, $J=8, 1.5$ Hz), 6.84 (2H, br d, $J=8.5$ Hz), 5.36 (1H, br s), 2.91 (20/11H, q, $J=7.5$ Hz), 2.69 (2/11H, q, $J=7.5$ Hz), 1.23 (30/11H, t, $J=7.5$ Hz), 1.15 (3/11H, t, $J=7.5$ Hz); $^{13}\text{C-NMR}$ (CDCl_3 , 75 MHz) δ 163.0, 159.6, 157.1, 129.2, 128.3, 127.2, 122.0, 115.5, 114.8, 20.7, 11.4; HRMS (EI) m/z calcd for

C₁₅H₁₅NO₂ (M⁺) 241.1102, found 241.1108.

4i
(*E:Z*=10:1)

(*E/Z*)-1-(4-Methoxyphenyl)-1-propanone O-Phenyloxime (4i)

(*E:Z*=10:1): a colorless oil; ¹H-NMR (CDCl₃, 300 MHz) δ 7.72 (2H, br d, *J*=8.5 Hz), 7.35-7.26 (4H, m), 7.01 (1H, br tt, *J*=8, 1.5 Hz), 6.93 (2H, br d, *J*=8.5 Hz), 3.83 (3H, s), 2.92 (20/11H, q, *J*=7.5 Hz), 2.70 (2/11H, q, *J*=7.5 Hz), 1.23 (30/11H, t, *J*=7.5 Hz), 1.16 (3/11H, t, *J*=7.5 Hz); ¹³C-NMR (CDCl₃, 75 MHz) δ 162.3, 160.8, 159.6, 129.2, 128.0, 127.2, 121.9, 114.7, 113.9, 55.2, 20.5, 11.4; HRMS (EI) *m/z* calcd for C₁₆H₁₇NO₂ (M⁺) 255.1259, found 255.1257.

4j

(*E*)-1-(3-Bromophenyl)ethanone O-Phenyloxime (4j): a colorless oil:

¹H-NMR (CDCl₃, 300 MHz) δ 7.93 (1H, t, *J*=2 Hz), 7.69 (1H, ddd, *J*=8, 2, 1 Hz), 7.54 (1H, ddd, *J*=8, 2, 1 Hz), 7.37-7.25 (5H, m), 7.05 (1H, tt, *J*=8, 1.5 Hz), 2.42 (3H, s); ¹³C-NMR (CDCl₃, 75 MHz) δ 159.8, 156.7, 138.3, 133.0, 130.4, 129.8, 129.7, 125.4, 123.1, 122.8, 115.2, 13.6; HRMS (EI) *m/z* calcd for C₁₄H₁₂⁷⁹BrNO (M⁺) 289.0102, found 289.0108.

4k

(*E*)-1-(3-Nitrophenyl)ethanone O-Phenyloxime (4k): colorless

crystals: mp 64-65°C (*n*-hexane/AcOEt); ¹H-NMR (CDCl₃, 300 MHz) δ 8.59 (1H, t, *J*=2 Hz), 8.25 (1H, ddd, *J*=8, 2, 1 Hz), 8.13 (1H, ddd, *J*=8, 2, 1 Hz), 7.58 (1H, t, *J*=8 Hz), 7.35 (2H, br t, *J*=8 Hz), 7.29 (2H, br d, *J*=8 Hz), 7.07 (1H, br tt, *J*=8, 1.5 Hz), 2.49 (3H, s); ¹³C-NMR (CDCl₃, 50 MHz) δ 159.2, 155.5, 148.4, 137.6, 132.1, 129.5, 129.4, 124.2, 122.7, 121.3, 114.8, 13.2; IR (CHCl₃) 1533, 1353 cm⁻¹; HRMS (EI) *m/z* calcd for C₁₄H₁₂N₂O₃ (M⁺) 256.0847, found 256.0845; *Anal.* calcd for C₁₄H₁₂N₂O₃: C, 65.62; H, 4.72; N, 10.93, found C, 65.62; H, 4.86; N, 10.88.

4l

(*E*)-1-(3-Hydroxyphenyl)ethanone O-Phenyloxime (4l): a colorless oil:

¹H-NMR (CDCl₃, 300 MHz) δ 7.36-7.25 (7H, m), 7.03 (1H, tt, *J*=8, 1.5 Hz), 6.89 (1H, dm, *J*=8 Hz), 5.06 (1H, br s), 2.42 (3H, s); ¹³C-NMR (CDCl₃, 75 MHz) δ 159.3, 155.5, 137.2, 129.8, 129.3, 122.3, 119.1, 117.0, 114.9, 113.3, 13.4; IR (CHCl₃) 3317 cm⁻¹; HRMS (EI) *m/z* calcd for C₁₄H₁₃NO₂ (M⁺) 227.0946, found 227.0960.

(E)-1-(3-Methoxyphenyl)ethanone O-Phenyloxime (4m): a colorless

oil: $^1\text{H-NMR}$ (CDCl_3 , 300 MHz) δ 7.36-7.27 (7H, m), 7.03 (1H, tt, $J=8$, 1.5 Hz), 6.98-6.94 (1H, m), 3.85 (3H, s), 2.44 (3H, s); $^{13}\text{C-NMR}$ (CDCl_3 ,

50 MHz) δ 159.6, 159.5, 157.6, 137.3, 129.5, 129.2, 122.1, 119.0, 115.3, 114.8, 111.9, 55.3, 13.4; HRMS (EI) m/z calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_2$ (M^+) 241.1102, found 241.1098.

General Procedure for Preparation of the Benzofurans 5.

To a solution of oxime ether **4** (0.13 mmol) in CH_2Cl_2 (2.0 ml) were added DMAP (0.39 mmol) and TFAT (0.65 mmol) at room temperature under a nitrogen atmosphere. After being stirred at the same temperature for 2 h, the reaction mixture was diluted with H_2O and extracted with CHCl_3 . The organic phase was dried over MgSO_4 and concentrated at reduced pressure. Purification of the residue by medium-pressure column chromatography (*n*-hexane/AcOEt 10:1) afforded the benzofurans **5** as shown in Table 1.

2-(4-Bromophenyl)-3-methylbenzofuran (5g): colorless crystals: mp

80-81°C (*n*-hexane/AcOEt); $^1\text{H-NMR}$ (CDCl_3 , 300 MHz) δ 7.66 (2H, br d, $J=8.5$ Hz), 7.58 (2H, br d, $J=8.5$ Hz), 7.54-7.50 (1H, dm, $J=8$ Hz),

7.48-7.44 (1H, dm, $J=8$ Hz), 7.30 (1H, td, $J=8$, 1.5 Hz), 7.24 (1H, td, $J=8$, 1.5 Hz), 2.44 (3H, s);

$^{13}\text{C-NMR}$ (CDCl_3 , 50 MHz) δ 153.8, 150.0, 131.8, 131.0, 130.3, 128.0, 124.6, 122.5, 121.9, 119.4,

111.9, 111.0, 9.5; HRMS (EI) m/z calcd for $\text{C}_{15}\text{H}_{11}^{79}\text{BrO}$ (M^+) 285.9993, found 285.9998; *Anal.*

calcd for $\text{C}_{15}\text{H}_{11}\text{BrO}$: C, 62.74; H, 3.86, found C, 62.65; H, 3.70.

4-(2-Benzofuranyl)phenyl Trifluoroacetate (5n): colorless

crystals: mp 165-167°C (*n*-hexane/AcOEt); $^1\text{H-NMR}$ (CDCl_3 , 300

MHz) δ 7.94 (2H, br d, $J=8.5$ Hz), 7.62-7.59 (1H, dm, $J=8$ Hz), 7.54-7.52 (1H, dm, $J=8$ Hz),

7.34-7.22 (4H, m), 7.05 (1H, d, $J=1$ Hz); $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ 155.8 (q, COCF_3), 155.0,

154.3, 149.2, 129.8, 129.0, 126.3, 124.7, 123.2, 121.1, 121.0, 115.0 (q, CF_3), 111.3, 102.2; IR

(CHCl_3) 1799 cm^{-1} ; HRMS (EI) m/z calcd for $\text{C}_{16}\text{H}_9\text{F}_3\text{O}_3$ (M^+) 306.0503, found 306.0512; *Anal.*

calcd for $\text{C}_{16}\text{H}_9\text{F}_3\text{O}_3 \cdot 1/2\text{H}_2\text{O}$: C, 60.96; H, 3.20, found C, 60.96; H, 3.09.

(E)-1-(3,5-Dihydroxyphenyl)ethanone O-Phenyloxime (8): According to the procedure given for **4**, reaction of *O*-phenylhydroxylamine **7** (206mg, 1.9 mmol) with 3,5-dihydroxyacetophenone (288 mg, 1.9 mmol) and c. HCl (0.10 mL) in EtOH (3.6 mL) gave the oxime ether **8** (460 mg,

92%) as colorless crystals: mp 104-106°C (CH₂Cl₂); ¹H-NMR (CD₃OD, 300 MHz) δ 7.32 (2H, br t, *J*=8 Hz), 7.25 (2H, br d, *J*=8 Hz), 7.02 (1H, br t, *J*=8 Hz), 6.77 (2H, d, *J*=2 Hz), 6.41 (1H, t, *J*=2 Hz), 3.89 (2H, br s), 2.39 (3H, s); ¹³C-NMR (CD₃OD, 50 MHz) δ 161.1, 159.7, 159.5, 139.2, 130.4, 123.3, 115.9, 106.3, 105.3, 13.8; IR (nujol) 3242 cm⁻¹; HRMS (EI) *m/z* calcd for C₁₄H₁₃NO₃ (M⁺) 243.0894, found 243.0897; *Anal.* calcd for C₁₄H₁₃NO₃·1/30 CH₂Cl₂: C, 68.49; H, 5.35; N, 5.69, found C, 68.61; H, 5.48; N, 5.39.

Stemofuran A (2): According to the procedure given for **5**, reaction of oxime ether **8** (32.7 mg, 0.13 mmol) with DMAP (127 mg, 1.04 mmol) and TFAT (0.35 mL, 2.35 mmol) in CH₂Cl₂ (4 mL) gave Stemofuran A **2** (28.7

mg, 95%) as colorless crystals: mp 182-183°C (CHCl₃); ¹H-NMR (CD₃OD, 500 MHz) δ 7.56 (1H, br dd, *J*=8, 1.5 Hz), 7.48 (1H, br dd, *J*=8, 1.5 Hz), 7.25 (1H, td, *J*=8, 1.5 Hz), 7.20 (1H, td, *J*=8, 1.5 Hz), 7.02 (1H, dd, *J*=1 Hz), 6.84 (2H, d, *J*=2 Hz), 6.30 (1H, t, *J*=2 Hz); ¹³C-NMR (CD₃OD, 125 MHz) δ 160.0, 157.4, 156.0, 133.4, 130.5, 125.3, 123.9, 121.9, 111.8, 104.4, 104.1, 102.2; IR (nujol) 3294 cm⁻¹; HRMS (EI) *m/z* calcd for C₁₄H₁₀O₃ (M⁺) 226.0629, found 226.0645; *Anal.* calcd for C₁₄H₁₀O₃: C, 74.33; H, 4.46, found C, 74.31; H, 4.33.

(E/Z)-1-(4-Hydroxyphenyl)-1-propanone O-(4-Bromophenyl)-

oxime (11) (*E:Z*=10:1): According to the procedure given for **4**, reaction of *O*-(4-bromophenyl)hydroxylamine **10** (101.1 mg, 0.54 mmol) with 4-hydroxypropiophenone (80.7 mg, 0.54 mmol) and c.

HCl (0.10 mL) in EtOH (2 mL) gave the oxime ether **11** (162.1 mg, 94%) as a colorless oil: ¹H-NMR (CDCl₃, 300 MHz) δ 7.65 (2H, br d, *J*=8.5 Hz), 7.41 (2H, br d, *J*=8.5 Hz), 7.16 (2H, br d, *J*=8.5 Hz), 6.86 (2H, br d, *J*=8.5 Hz), 5.08 (1H, br s), 2.90 (20/11H, q, *J*=7.5 Hz), 2.68 (2/11H, q, *J*=7.5 Hz), 1.22 (30/11H, t, *J*=7.5 Hz), 1.15 (3/11H, t, *J*=7.5 Hz); ¹³C-NMR (CDCl₃, 50 MHz) δ

163.3, 158.6, 157.3, 132.0, 128.3, 126.9, 116.5, 115.5, 114.0, 20.7, 11.4; IR (CHCl₃) 3295 cm⁻¹; HRMS (EI) *m/z* calcd for C₁₅H₁₄⁷⁹BrNO₂ (M⁺) 319.0207, found 319.0210.

4-(5-Bromo-3-methyl-2-benzofuranyl)phenol (12): According to the procedure given for **5**, reaction of oxime ether **11** (37.4 mg, 0.12 mmol) with DMAP (42.8 mg, 0.36 mmol) and TFAT (0.09 mL, 0.6

mmol) in CH₂Cl₂ (3.7 mL) gave the bromobenzofuran **12** (32.7 mg, 92%) as colorless crystals: mp 157-159°C (*n*-hexane/Et₂O); ¹H-NMR (CDCl₃, 300 MHz) δ 7.67 (2H, br d, *J*=8.5 Hz), 7.60 (1H, dd, *J*=2, 0.5 Hz), 7.32 (1H, d, *J*=2 Hz), 7.31 (1H, d, *J*=0.5 Hz), 6.94 (2H, br d, *J*=8.5 Hz), 5.12 (1H, br s), 2.38 (3H, s); ¹³C-NMR (CDCl₃, 50 MHz) δ 155.6, 152.3, 152.1, 133.3, 128.5, 126.6, 123.8, 121.8, 115.7, 115.4, 112.2, 109.3, 9.3; IR (CHCl₃) 3295 cm⁻¹; HRMS (EI) *m/z* calcd for C₁₅H₁₁⁷⁹BrO₂ (M⁺) 301.9941, found 301.9939; *Anal.* calcd for C₁₅H₁₁BrO₂: C, 59.43; H, 3.66, found C, 59.37; H, 3.48.

Eupomatenoid 6 (3): To a stirred mixture of bromobenzofuran **12** (46.2 mg, 0.15 mmol), powdered CsF (93 mg, 0.60 mmol) and *trans*-propenylboronic acid (39 mg, 0.45 mmol) in DME

(0.54 mL) was added Pd(PPh₃)₄ (0.005 mg, 3 mol%) at room temperature. The reaction mixture was flushed with argon gas and maintained under an argon atmosphere while being heated at reflux for 7 h. The reaction mixture was then cooled, diluted with AcOEt, and washed with brine. The organic phase was dried over MgSO₄ and concentrated at reduced pressure. Purification of the residue by medium-pressure column chromatography (*n*-hexane/AcOEt 5:1) afforded Eupomatenoid 6 **3** (39 mg, 97%) as colorless crystals: mp 147.5-148 °C (benzene/petroleum ether) (lit.¹ 147.5-149°C); ¹H-NMR (CDCl₃, 500 MHz) δ 7.68 (2H, br d, *J*=8.5 Hz), 7.43 (1H, br d, *J*=2 Hz), 7.36 (1H, br d, *J*=8.5 Hz), 7.27 (1H, br dd, *J*=8.5, 2 Hz), 6.93 (2H, br d, *J*=8.5 Hz), 6.52 (1H, br dd, *J*=15.5, 2 Hz), 6.33 (1H, dq, *J*=15.5, 6.5 Hz), 4.90 (1H, br s), 2.42 (3H, s), 1.91 (3H, dd, *J*=6.5, 2 Hz); ¹³C-NMR (CDCl₃, 125 MHz) δ 155.3, 152.9, 151.1, 132.6, 131.5, 131.3, 128.3, 124.4, 124.2, 122.2, 116.1, 115.6, 110.6, 109.8, 18.5, 9.4; IR (CHCl₃) 3305 cm⁻¹; HRMS (EI) *m/z* calcd for C₁₈H₁₆O₂ (M⁺) 264.1149, found 264.1145.

Reference

1. McKittrick, B. A.; Stevenson, R. J. *Chem. Soc., Perkin Trans. 1* **1983**, 475-482.

100%

810201 810201

SAMPLE
Date Oct 8 2003 dfrg 380.023
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AT 56
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[illegible]

11
(E:Z=10:1)

[illegible]