Synthesis of Substituted 2,3-Dihydro-10b*H*- oxazolo[2,3-*a*]isoquinolines from *ortho*-Lithiated Phenyloxazolinyloxiranes[†]

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SUPPORTING INFORMATION

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[†] Dedicated to Prof. Tisler on the occasion of his 80th birthday.

General. Tetrahydrofuran (THF) was freshly distilled under a nitrogen atmosphere over sodium/benzophenone ketyl. Petroleum ether refers to the 40-60 °C boiling fraction. For the ^1H and ^{13}C NMR spectra (^1H NMR 400, 500 MHz; ^{13}C NMR 100 MHz, 125 MHz), CDCl_3 was used as the solvent. GC-MS spectrometry analyses were performed on a gas chromatograph (dimethylsilicon capillary column, 30 m, 0.25 mm i.d.) equipped with a mass selective detector operating at 70 eV (EI). Microanalyses were carried out on an EuroEA 3000 C, H, N, S analyzer. Melting points (uncorrected) were carried out on an Stuart SMP3 apparatus.. Analytical thin layer chromatography (TLC) was carried out on precoated 0.25 mm thick plates of Kieselgel 60 F254; visualization was accomplished by UV light (254 nm) or by spraying with a solution of 5 % (w/v) ammonium molybdate and 0.2 % (w/v) cerium(III) sulfate in 100 ml 17.6 % (w/v) aq. sulphuric acid and heating to 200 °C for some time until blue spots appear. All reactions involving air-sensitive reagents were performed under nitrogen in oven-dried glassware using syringe-septum cap technique. Oxiranes **1a-e** were prepared according to the reported procedure.¹

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(1*R,2*R**,3*R**)-2,4',4'-Trimethyl-2,3-dihydrospiro[2,3-epoxyindene-1,2'-[1,3]oxazolidine] (2c):** white solid, mp 115-116 °C (hexane/Et₂O), 83 %; ¹H NMR (500 MHz) δ 1.42 (s, 3 H), 1.49 (s, 3 H), 1.68 (s, 3 H), 2.22 (br s, exchanges with D₂O, 1 H), 3.85 and 3.92 (2 × d, AB system, *J* = 8.1 Hz, 2 H), 4.06 (s, 1 H), 7.27 (dt, *J* = 1.0, 7.4 Hz, 1 H), 7.33 (dt, *J* = 1.1, 7.4 Hz, 1 H) 7.37–7.41 (m, 2 H); ¹³C NMR (125 MHz) δ 12.3, 28.3, 28.4, 58.1, 62.8, 67.9, 78.9, 101.1, 124.1, 124.6, 128.6, 129.2, 140.4, 147.6; GC-MS (70 eV) *m/z* (%) 231 (*M*⁺, 1), 214 (100), 174 (23), 145 (20), 116 (31), 89 (27); FT-IR (KBr, cm⁻¹) 3333, 3031, 2969, 2871, 1652, 1465, 1254, 1125, 1056, 854, 757, 702. Anal. Calcd for C₁₄H₁₇NO₂: C, 72.70; H, 7.41; N, 6.06. Found: C, 73.02; H, 7.39; N, 5.88.

(1*R,2*R**,3*R**)-2-Ethyl-4',4'-Dimethyl-2,3-dihydrospiro[2,3-epoxyindene-1,2'-[1,3]oxazolidine] (2d):** white solid, mp 120-121 °C (hexane/Et₂O), 78%; ¹H NMR (500 MHz) δ 0.98 (t, *J* = 7.4 Hz, 3 H), 1.40 (s, 3 H), 1.48 (s, 3 H), 2.05–2.11 (m, 1 H), 2.32 (br s, exchanges with D₂O, 1 H), 2.34–2.40 (m, 1 H), 3.83 and 3.92 (2 × d, AB system, *J* = 8.1 Hz, 2 H), 4.11 (s, 1 H), 7.25 (t, *J* = 7.4 Hz, 1 H), 7.32 (t, *J* = 7.4 Hz, 1 H), 7.38 (d, *J* = 7.4 Hz, 1 H), 7.41 (d, *J* = 7.4 Hz, 1 H); ¹³C NMR (125 MHz) δ 8.11, 17.0, 28.3, 28.4, 57.9, 59.4, 71.6, 78.9, 101.4, 124.0, 124.4, 128.4, 129.0, 140.2, 147.9; GC-MS (70 eV) *m/z* (%) 245 (*M*⁺, 1), 228 (50), 216 (100), 174 (47), 116 (36), 89 (25); FT-IR (film, cm⁻¹) 3357, 2964, 2867, 1612, 1369, 1109, 854, 702, 698. Anal. Calcd for C₁₅H₁₉NO₂: C, 73.44; H, 7.81; N, 5.71. Found: C, 73.66; H, 7.84; N, 5.47.

3-[2-(4,5-Dihydro-4,4-dimethyloxazol-2-yl)phenyl]butan-2-one (4b): colorless oil, 52 %; ^1H -NMR (500 MHz) δ 1.29 (s, 6 H), 1.30 (d, J = 6.9 Hz, 3 H), 1.98 (s, 3 H), 3.98 (s, 2 H), 4.86 (q, J = 6.9 Hz, 1 H), 7.11 (d, J = 7.5 Hz, 1 H), 7.19 (t, J = 7.5 Hz, 1 H), 7.32 (t, J = 7.5 Hz, 1 H), 7.71 (d, J = 7.5 Hz, 1 H); ^{13}C -NMR (125 MHz) δ 17.0, 28.3, 28.4, 28.7, 49.5, 68.2, 78.4, 126.8, 127.4, 128.0, 130.3, 131.0, 141.0, 161.8, 209.4; GC-MS (70 eV) m/z (%): 245 (M^+ , 3), 230 (81), 228 (100), 203 (11), 148 (47), 130 (36), 103 (30); FT-IR (film, cm^{-1}) 3064, 2973, 2930, 1711, 1645, 1449, 1354, 910.

3,3,5,6,10b-Pentamethyl-2,3-dihydro-10bH-[1,3]oxazolo[2,3-a]isoquinoline (7b): yellow oil, 67%; ^1H NMR (500 MHz) δ 1.29 (s, 3 H), 1.35 (s, 3 H), 1.43 (s, 3 H), 2.45 (s, 3 H), 2.53 (s, 3 H), 3.89 (d, J = 7.9 Hz, 1 H), 3.95 (d, J = 7.9 Hz, 1 H), 7.29–7.31 (m, 1 H), 7.40–7.49 (m, 2 H), 7.56 (d, J = 7.3 Hz, 1 H); ^{13}C NMR (125 MHz) δ 20.8, 25.0, 25.5, 27.6, 27.7, 61.7, 79.2, 95.3, 110.0, 120.6, 123.7, 127.0, 128.3, 132.4, 134.9, 140.6; GC-MS (70 eV) m/z (%) 243 (M^+ , (2), 229 (9), 214 (100), 189 (12), 157 (31), 115 (21); FT-IR (film, cm^{-1}) 3063, 2969, 2866, 1624, 1597, 1480, 1250, 1067, 750, 696.

3,3,5-Trimethyl-10b-phenyl-2,3-dihydro-10bH-[1,3]oxazolo[2,3-a]isoquinoline (7c): red solid, mp 55-56 C° (hexane), 78%; ¹H NMR (500 MHz) δ 1.25 (s, 3 H), 1.55 (s, 3 H), 2.21 (s, 3 H), 3.73 (d, *J* = 8.4 Hz, 1 H), 3.84 (d, *J* = 8.4 Hz, 1 H), 5.70 (s, 1 H), 6.96–6.98 (m, 1 H), 7.20–7.30 (m, 5 H), 7.44–7.46 (m, 2 H), 7.61–7.63 (m, 1 H) ¹³C NMR (125 MHz) δ 21.6, 25.1, 28.2, 61.9, 78.2, 96.5, 106.1, 122.6, 123.2, 125.3, 125.6, 127.4, 127.7, 127.9, 130.6, 132.5, 139.0, 144.5; GC-MS (70 eV) *m/z* (%) 291 (M⁺, 9), 219 (43), 218 (100), 214 (62), 203 (12), 159 (10), 109 (11); FT-IR (film, cm⁻¹) 3063, 2970, 2863, 1621, 1567, 1398, 1251, 1067, 750, 695. Anal. Calcd for C₂₀H₂₁NO: C, 82.44; H, 7.26; N, 4.81. Found: C, 82.78; H, 7.15; N, 4.69.

10b-Butyl-3,3,5-trimethyl-2,3-dihydro-10bH-[1,3]oxazolo[2,3-a]isoquinoline (7d): yellow oil, 70%; ¹H NMR (500 MHz) δ 0.82 (t, *J* = 7.1 Hz, 3 H), 1.04 (s, 3 H), 1.19–1.35 (m, 4 H), 1.44 (s, 3 H), 1.49–1.62 (m, 2 H), 2.04 (s, 3 H), 3.48 (d, *J* = 8.0 Hz, 1 H), 3.86 (d, *J* = 8.0 Hz, 1 H), 5.73 (s, 1 H), 6.94 (dd, *J* = 7.3, 1.8 Hz, 1 H), 7.11–7.17 (m, 2 H), 7.25 (dd, *J* = 7.3, 1.8 Hz, 1 H); ¹³C NMR (125 MHz) δ 14.1, 21.2, 22.8, 24.1, 24.8, 29.1, 39.1, 61.2, 78.7, 97.0, 106.8, 122.3₀, 122.3₃, 125.1, 126.9, 128.5, 131.6, 139.3; GC-MS (70 eV) *m/z* (%) 271 (M⁺, 5), 214 (100), 159 (16), 157 (27), 115 (11); FT-IR (film, cm⁻¹) 3051, 2955, 2867, 1613, 1459, 1381, 1259, 1181, 1069, 800, 750.

3,3,5,10b-Tetramethyl-2,3-dihydro-10bH-[1,3]oxazolo[2,3-a]isoquinoline (7e): yellow oil, 65 %; ^1H NMR (500 MHz) δ 1.08 (s, 3 H), 1.46 (s, 3 H), 2.06 (s, 3 H), 2.44 (s, 3 H), 3.52 (d, $J = 8.1$ Hz, 1 H), 3.88 (d, $J = 8.1$ Hz, 1 H), 5.64 (s, 1 H), 6.93–6.95 (m, 1 H), 7.16–7.31 (m, 2 H), 7.39 (d, $J = 8.3$ Hz, 1 H); ^{13}C NMR (125 MHz) δ 21.7, 24.1, 27.7, 29.1, 61.4, 78.3, 95.1, 105.3, 125.2, 126.4, 127.4, 127.6, 132.5, 135.8, 139.0; GC-MS (70 eV) m/z (%) 229 (M^+ , 11), 214 (100), 159 (36), 157 (37), 115 (22); FT-IR (film, cm^{-1}) 3063, 2966, 2864, 1641, 1588, 1360, 1067, 799.

3,3,5-Trimethyl-10b-thiophen-2-yl-2,3-dihydro-10bH-[1,3]oxazolo[2,3-a]isoquinoline (7f): yellow oil, 65 %; ^1H NMR (500 MHz) δ 1.15 (s, 3 H), 1.60 (s, 3 H), 2.10 (s, 3 H), 3.68 (d, $J = 8.0$ Hz, 1 H), 3.99 (d, $J = 8.0$ Hz, 1 H), 5.71 (s, 1 H), 6.65–6.67 (m, 1 H), 6.82–6.84 (m, 1 H), 6.96–6.98 (m, 1 H), 7.19–7.26 (m, 3 H), 7.59–7.61 (m, 1 H); ^{13}C NMR (125 MHz) δ 21.5, 24.5, 28.2, 62.0, 78.4, 94.3, 107.2, 122.5, 122.8, 125.5, 126.6, 127.7, 127.8, 130.6, 132.5, 138.5, 145.6; GC-MS (70 eV) m/z (%) 297 (M^+ , 23), 224 (38), 214 (100), 159 (14), 142 (12), 115 (11); FT-IR (film, cm^{-1}) 3010, 2969, 2927, 1602, 1466, 1399, 1109, 1092, 800.

10b-Isopropyl-3,3,5-trimethyl-2,3-dihydro-10bH-[1,3]oxazolo[2,3-a]isoquinoline (7g): yellow oil, 44%; ^1H NMR (500 MHz) δ 0.74 (d, $J = 6.9$ Hz, 3 H), 0.92 (d, $J = 6.9$ Hz, 3 H), 0.99 (s, 3 H), 1.44 (s, 3 H), 1.78 (ep, $J = 6.9$ Hz, 1 H), 2.04 (s, 3 H), 3.44 (d, $J = 7.9$ Hz, 1 H), 3.85 (d, $J = 7.9$ Hz, 1 H), 5.80 (s, 1 H), 6.95 (d, $J = 7.4$ Hz, 1 H), 7.11–7.18 (m, 2 H), 7.24 (d, $J = 7.4$ Hz, 1 H); ^{13}C NMR (125 MHz) δ 15.0, 15.2, 24.2, 26.7, 27.9, 42.3, 63.2, 78.7, 98.1, 104.8, 122.1, 126.5, 128.2, 129.1, 133.9, 138.6, 139.8; GC-MS (70 eV) m/z (%) 257 (M^+ , 3), 214 (100), 162 (54), 132 (17), 115 (19), 43 (9); FT-IR (film, cm^{-1}) 2961, 2925, 2854, 1615, 1459, 1381, 1253, 1071, 800.

(*R)-10b-[(*R**)-sec-Butyl]- and (*R**)-10b-[(*S**)-sec-Butyl]-3,3,5-trimethyl-2,3-dihydro-10bH-[1,3]oxazolo[2,3-*a*]isoquinoline (7h):** yellow oil, inseparable mixture of two diastereoisomers (dr 1/1) 42%; ¹H NMR (500 MHz) δ 0.67 (t, *J* = 7.4 Hz, 3 H), 0.69 (d, *J* = 7.4 Hz, 3 H), 0.80 (t, *J* = 7.4 Hz, 3 H), 0.89 (d, *J* = 7.3 Hz, 3 H), 0.94 (s, 3 H), 0.95 (s, 3 H), 1.13–1.24 (m, 4 H), 1.40 (s, 6 H), 1.53–1.60 (m, 4 H), 1.80–1.89 (m, 2 H), 2.00 (s, 3 H), 2.01 (s, 3 H), 3.41 (d, *J* = 7.9 Hz, 1 H), 3.42 (d, *J* = 7.9 Hz, 1 H), 3.80 (d, *J* = 7.9 Hz, 1 H), 3.82 (d, *J* = 7.9 Hz, 1 H), 5.76 (s, 1 H), 5.77 (s, 1 H), 6.91–6.94 (m, 2 H), 7.08–7.15 (m, 4 H), 7.19–7.23 (m, 2 H); ¹³C NMR (125 MHz) δ 14.9₇, 14.9₈, 15.6₈, 15.6₉, 21.6, 21.7, 24.1, 25.1, 27.7, 28.2, 29.1, 29.7, 41.6, 41.8, 61.4, 61.9, 78.3, 78.5, 98.5, 98.8, 105.3, 106.0, 125.5, 126.4, 126.9, 127.1, 127.4, 127.6, 127.8, 132.5, 132.6, 134.8, 135.8, 138.9, 139.0; GC-MS (70 eV); *m/z* (%) 271 (*M*⁺, 1), 232 (36), 214 (99), 176(54), 160 (100), 132 (17), 115 (19); FT-IR (film, cm⁻¹) 2964, 2927, 2873, 1615, 1463, 1382, 1189, 1070, 800.

5-Ethyl-3,3-dimethyl-10b-phenyl-2,3-dihydro-10bH-[1,3]oxazolo[2,3-*a*]isoquinoline (7j): yellow solid, mp 66-67 °C (hexane), 81 %; ¹H NMR (500 MHz) δ 1.17 (s, 3 H), 1.20 (t, *J* = 7.3 Hz, 3 H), 1.61 (s, 3 H), 2.40–2.49 (m, 2 H), 3.69 (d, *J* = 8.2 Hz, 1 H), 3.96 (d, *J* = 8.2 Hz, 1 H), 5.86 (s, 1 H), 7.04–7.07 (m, 1 H), 7.25–7.28 (m, 5 H), 7.39–7.42 (m, 3 H); ¹³C NMR (125 MHz) δ 12.7, 24.5, 27.7, 29.4, 61.9, 79.3, 96.6, 106.4, 123.1, 123.3, 125.6, 126.2, 127.5, 127.6, 127.8, 133.2, 139.1, 144.2; GC-MS (70 eV) *m/z* (%) 305 (*M*⁺, 14), 232 (62), 228 (100), 204 (10), 173 (18); FT-IR (film, cm⁻¹) 3062, 2964, 2869, 1627, 1591, 1339, 1249, 752, 700. Anal. Calcd for C₂₁H₂₃NO: C, 82.58; H, 7.59; N, 4.59;. Found: C, 82.84; H, 7.55; N, 4.33.

3,3,5-Trimethyl-10b-phenyl-6-(2-thienyl)-2,3-dihydro-10bH-[1,3]oxazolo[2,3-a]isoquinoline (7k): yellow oil, 37 %; ^1H NMR (500 MHz) δ 1.06 (s, 3 H), 1.68 (s, 3 H), 2.03 (s, 3 H), 3.53 (d, J = 8.0 Hz, 1 H), 3.66 (d, J = 8.0 Hz, 1 H), 6.62–6.68 (m, 1 H), 6.80–6.83 (m, 1 H), 6.99–7.02 (m, 1 H), 7.17–7.34 (m, 8 H), 7.59–7.61 (m, 1 H); ^{13}C NMR (125 MHz) δ 18.9, 29.5, 29.7, 63.0, 79.9, 106.0, 115.3, 125.8, 126.5, 126.9, 127.2, 128.0, 128.1, 128.6, 130.0, 130.6, 131.2, 137.2, 137.8, 138.3, 139.5, 141.9; GC-MS (70 eV) m/z (%) 373 (M^+ , 5), 296 (100), 293 (76), 159 (6), 77 (12); FT-IR (film, cm^{-1}) 3028, 3017, 2973, 2879, 1612, 1519, 1274, 1109, 1092, 752, 699.

(1R*,1'R*)-1-(3,3-Dimethyl-1,3-dihydro-isobenzofuran-1-yl)-1-(4,4-dimethyl-4,5-dihydro-oxazol-2-yl)-ethanol (8a): Colorless oil, 70%. H^1 -NMR (500 MHz) δ : 1.23 (s, 3 H), 1.32 (s, 3 H), 1.45 (s, 3 H), 1.55 (s, 3 H), 1.59 (s, 3 H), 2.29 (br. s, exchanges with D_2O , 1 H), 4.00 and 4.03 (2 $\times$ d, AB system, J = 8.0 Hz, 2 H), 5.33 (s, 1 H) 7.08 (d, J = 7.5 Hz, 1 H), 7.18–7.25 (m, 2H), 7.48 (d, J = 7.5 Hz, 1 H); ^{13}C -NMR (125 MHz) δ : 22.8, 28.1, 28.9, 29.6, 29.9, 66.9, 73.2, 80.2, 85.70, 86.0, 120.5, 122.7, 124.8, 128.1, 136.4, 148.2, 168.1. GC-MS (70 eV) m/z (%) 289 (M^+ , 2), 147 (100), 129 (27), 91 (5). FT-IR (film, cm^{-1}) 3415, 2979, 1717, 1459, 1122, 813.

(1*R,1'*R**,3'*R**)- and (1*R**,1'*R**,3'*S**)-1-(3-phenyl-3-propyl-1,3-dihydro-isobenzofuran-1-yl)-1-(4,4-dimethyl-4,5-dihydro-oxazol-2-yl)-ethanol (8c):** yellow oil, 86%, inseparable mixture of two diastereoisomers (d.r. 1/1). ¹H-NMR (500 MHz) δ 0.83 (t, $J = 7.5$ Hz, 3 H), 0.88 (t, $J = 7.5$ Hz, 3 H), 1.25–1.30 (m, 4 H), 1.28 (s, 3 H), 1.29 (s, 3 H), 1.32 (s, 3 H), 1.37 (s, 3 H), 1.45 (s, 3 H), 1.59 (s, 3 H), 1.92–1.98 (m, 1 H), 2.06–2.18 (m, 2 H), 2.22–2.28 (m, 1 H), 2.65 (br s, exchanges with D₂O, 1 H), 3.03 (br s, exchanges with D₂O, 1 H), 4.00–4.09 (m, 4 H), 5.40 (s, 1 H), 5.46 (s, 1 H), 7.16–7.36 (m, 14 H), 7.48 (m, 2 H), 7.66 (m, 2 H); ¹³C-NMR (125 MHz) δ 14.1, 14.4, 17.3, 17.4, 22.0, 22.7, 28.0, 28.3, 28.4, 44.1, 45.8, 67.1₀, 67.1₁, 73.2, 73.5, 80.0, 80.1, 86.0, 87.2, 90.2, 91.3, 121.5, 122.4, 122.8, 122.9, 125.0, 125.5, 126.6, 126.8, 127.2, 127.4, 128.0, 128.0₁, 128.1₀, 128.1₁, 137.1, 137.8, 145.0, 145.2, 145.3, 146.6, 168.0, 168.2; GC-MS (70 eV) m/z (%) 379 (1.18), 318 (3.53), 237 (100), 219 (8.24), 194 (18.82), 165 (11.76), 115 (4.71), 91 (11.80), 77 (2.35), 43 (7.06); FT-IR (film, cm⁻¹) 3306, 3062, 2966, 2870, 1657, 1458, 1369, 1248, 1045, 908, 731.

(1*R,1'*R**,3'*R**)- and (1*R**,1'*R**,3'*S**)-1-[[3-(4-chloro)-phenyl-1,3-dihydro-isobenzofuran-1-yl]-1-(4,4-dimethyl-4,5-dihydro-oxazol-2-yl)]-ethanol (8d)**: yellow oil; 80%, inseparable mixture of two diastereoisomers (d.r. 1/1). ¹H-NMR (500 MHz) δ 1.30 (s, 6 H), 1.31 (s, 3 H), 1.37 (s, 3 H), 1.55 (s, 3 H), 1.62 (s, 3 H), 4.02 and 4.05 (2 × d, AB system, *J* = 8.3 Hz, 2 H), 4.10 and 4.13 (2 × d, AB system, *J* = 8.2 Hz, 2 H), 5.46 (s, 1 H), 5.52 (d, *J* = 2.6 Hz, 1 H), 5.65 (br s, exchanges with D₂O, 1 H), 6.08 (s, 1 H), 6.15 (br s, exchanges with D₂O, 1 H), 6.28 (d, *J* = 2.6 Hz, 1 H), 7.22–7.36 (m, 14 H), 7.41–7.46 (m, 2 H). ¹³C-NMR (125 MHz) δ 19.0, 19.2, 26.1, 27.4, 27.8, 27.9, 65.6, 66.7, 74.0, 74.6, 80.2₉, 80.3₁, 85.48, 86.2, 86.8, 87.9, 127.2, 127.3, 127.4, 127.5, 128.3₂, 128.3₆, 128.4, 128.5, 128.7, 128.8, 129.1, 129.3, 137.8, 138.2, 138.4, 139.0, 140.4, 140.7, 141.5, 141.7, 163.6, 163.7; GC-MS (70 eV) *m/z* (%): 371 (*M*⁺, 5), 358 (6), 229 (100), 194 (65), 160 (92), 88 (71); FT-IR (film, cm⁻¹) 3396, 3012, 2927, 1661, 1655, 1353, 1109, 801.

Figure S1. Ortep view of **2b**

Figure S2. Ortep view of 7a

CDCl₃, 125 MHz

CDCl₃, 100 MHz

CDCl₃, 125 MHz

CDCl₃, 100 MHz

CDCl₃, 100 MHz

CDCl₃, 125 MHz

CDCl₃, 125 MHz

CDCl₃, 125 MHz

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