Supporting information

Tandem Cross-Metathesis/ Hydrogenation/Cyclization Reactions by Using Compatible catalysts.

Janine Cossy*, Frédéric Bargiggia and Samir BouzBouz

Laboratoire de Chimie Organique associé au CNRS, ESPCI, 10 rue Vauquelin, 75231 Paris Cedex 05, France

Typical procedure for tandem cross-metathesis/hydrogenation reaction: In a 10 mL round bottom flask, a solution of α,β -unsaturated carbonyl compound (3 equiv., 1.2 mmol) and allylic or homoallylic alcohol (1 equiv., 0.4 mmol) in CH_2Cl_2 (0.2 M, 2.5 mL) was degased under vacuum, purged with H_2 and then catalyst **I** and PtO_2 were introduced. The reaction mixture was degased again under vacuum and then vigorously stirred under one atmosphere of hydrogen. After 15 h at rt, the solvent was evaporated and the residue was purified by flash chromatography to produce the corresponding lactol or lactone.

5-Hexyldihydrofuran-2-one (13).¹

IR: 1770; 1460 cm^{-1} .

NMR ^1H (300 MHz, CDCl_3): δ 4.47 (dddd, $J = 13.6$ Hz, $J = 7.4$ Hz, $J = 6.6$ Hz, $J = 5.5$ Hz, 1H); 2.51 (dd, $J = 9.2$ Hz, $J = 6.3$ Hz, 2H); 2.32 (d, $J = 12.9$ Hz, $J = 6.6$ Hz, 1H); 2.91-1.78 (m, 1H); 1.78-1.66 (m, 1H); 1.64-1.51 (m, 1H); 1.50-1.17 (m, 8H); 0.87 (t, $J = 7.0$ Hz, 3H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 177.2, 80.9, 35.5, 31.5, 28.9, 28.8, 27.9, 25.1, 22.4, 13.9.

GC-MS m/z (EI) (rel. int.): 169 ($\text{M}^+ - 1$, 1), 128 (12), 85 (100), 55 (8).

6-Hexyltetrahydropyran-2-one (14).²

IR: 1730; 1470 cm^{-1} .

NMR ^1H (300 MHz, CDCl_3): δ 4.26 (tdd, $J = 8.1$ Hz, $J = 5.2$ Hz, $J = 2.9$ Hz, 1H); 2.6 (m, 1H); 2.43 (m, 1H); 1.96-1.43 (m, 4H); 1.42-1.14 (m, 10H); 0.87 (t, $J = 7.0$ Hz, 3H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 171.9, 80.5, 35.7, 31.6, 29.4, 29.0, 27.7, 24.8, 22.4, 18.4, 13.9.

GC-MS m/z (EI) (rel. int.): 184 (M^+ , 0.5), 114 (13), 99 (100), 71 (10), 55 (22).

4-(2-phenylethyl)dihydrofuran-2-one (15).³

IR: 1770; 1450 cm^{-1} .

NMR ^1H (300 MHz, CDCl_3): δ 7.40-7.10 (m, 5H); 4.47 (dddd, $J = 8.8$ Hz, $J = 8.5$ Hz, $J = 6.6$ Hz, $J = 5.2$ Hz, 1H); 2.84 (m, 1H); 2.73 (m, 1H); 2.54 (dd, $J = 9.9$ Hz, $J = 6.3$ Hz, 1H); 2.53 (dd, $J = 9.2$ Hz, $J = 7.4$ Hz, 1H); 2.31 (m, 1H); 2.05 (m, 1H); 1.98-1.78 (m, 2H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 177.1, 140.6, 128.4 ($\times 2$), 128.3 ($\times 2$), 126.0, 79.8, 37.2, 31.5, 28.7, 27.8.

GC-MS m/z (EI) (rel. int.): 190 (M^+ , 54), 130 (100), 117 (34), 91 (70), 85 (44), 77 (12), 55 (10).

5-(2-phenylethyl)tetrahydropyran-2-one (16).⁴

IR: 1730; 1450 cm^{-1} .

NMR ^1H (300 MHz, CDCl_3): δ 7.33-7.25 (m, 2H); 7.23-7.15 (m, 3H); 4.26 (dddd, $J = 8.1$ Hz, $J = 7.4$ Hz, $J = 4.4$ Hz, $J = 2.6$ Hz, 1H); 2.87 (m, 1H); 2.74 (m, 1H); 2.58 (m, 1H); 2.45 (m, 1H); 2.11-1.73 (m, 5H); 1.68-1.46 (m, 1H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 171.8, 141.0, 128.4, 128.4, 128.3, 128.2, 125.7, 79.3, 37.4, 31.0, 29.4, 27.8, 18.4.

GC-MS m/z (EI) (rel. int.): 204 (M^+ , 54), 186 (18), 144 (18), 130 (18), 117 (100), 104 (59), 91 (87), 77 (10), 71(18), 55 (11).

5-Hexyltetrahydrofuran-2-ol (18).⁵

Anomers ratio: 60/40.

IR: 3420; 1460 cm^{-1} .

Major anomer:

NMR ^1H (300 MHz, CDCl_3): δ 5.54 (dd, $J = 5.1$ Hz, $J = 2.2$ Hz, 1H); 4.18 (p; $J = 6.3$ Hz, 1H); 3.58 (sl, 1H); 2.17-1.86 (m, 2H); 1.85-1.14 (m, 12 H); 0.86 (t, $J = 6.6$ Hz, 3H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 98.1, 78.3, 35.5, 32.8, 31.6, 29.2 ($\times 2$), 25.9, 22.4, 13.9.

Minor anomer:

NMR ^1H (300 MHz, CDCl_3): δ 5.45 (d, $J = 3.3$ Hz, 1H); 3.95 (p; $J = 6.3$ Hz, 1H); 3.47 (sl, 1H); 2.17-1.86 (m, 2H); 1.85-1.14 (m, 12 H); 0.86 (t, $J = 6.6$ Hz, 3H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 98.0, 81.0, 37.3, 33.8, 31.7, 29.3, 29.1, 26.0, 22.4, 13.9.

GC-MS m/z (EI) (rel. int.): 171 (2), 87 (100), 55 (22).

6-Hexyltetrahydropyran-2-ol (19).⁶

Anomers ratio: 64/36.

IR: 3380; 1460 cm^{-1} .

Major anomer:

NMR ^1H (300 MHz, CDCl_3): δ 4.68 (ddd, $J = 8.8$ Hz, $J = 6.2$ Hz, $J = 1.8$ Hz, 1H); 3.66 (bd, $J = 6.3$ Hz, 1H); 3.45-3.33 (m, 1H); 1.90-1.76 (m, 2H); 1.76-1.04 (m, 14 H); 0.86 (t, $J = 6.3$ Hz, 3H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 96.4, 76.4, 35.8, 32.8, 31.7, 30.3, 29.2, 25.4, 22.5, 22.1, 14.0.

Minor anomer:

NMR ^1H (300 MHz, CDCl_3): δ 5.29 (bs, 1H); 3.97-3.85 (m, 1H); 3.02 (bs, 1H); 1.90-1.76 (m, 2H); 1.76-1.04 (m, 14 H); 0.86 (t, $J = 6.3$ Hz, 3H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 91.8, 65.7, 36.1, 32.8, 31.0, 29.8, 29.3, 22.5, 22.1, 17.3, 14.0.

GC-MS m/z (EI) (rel. int.): 168 (M^+ -18, 80), 112 (32), 97 (33), 83 (95), 70 (90), 69 (75), 57 (100), 55 (87).

4-(2-phenylethyl)dihydrofuran-2-ol (20).⁷

Anomers ratio: 68/42.

IR: 3400; 1600 cm^{-1} .

Major anomer:

NMR ^1H (300 MHz, CDCl_3): δ 7.48-7.16 (m, 5H); 5.60 (dd, $J = 4.8$ Hz, $J = 1.8$ Hz, 1H); 4.25 (m, 1H); 3.69 (sl, 1H); 2.94-2.45 (m, 2H); 2.27-1.10 (m, 6H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 141.8, 128.4 ($\times 2$), 128.3 ($\times 2$), 125.7, 98.3, 77.6, 37.3, 32.8, 32.3, 29.3.

Minor anomer:

NMR ^1H (300 MHz, CDCl_3): δ 7.48-7.16 (m, 5H); 5.51 (d, $J = 3.3$ Hz, 1H); 4.02 (m, 1H); 3.65 (sl, 1H); 2.94-2.45 (m, 2H); 2.27-1.10 (m, 6H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 141.8, 128.3 ($\times 2$), 128.2 ($\times 2$), 125.6, 98.2, 80.1, 38.9, 33.8, 32.5, 29.1.

GC-MS m/z (EI) (rel. int.): 174 (32), 130 (100), 117 (44), 91 (80), 55 (7).

5-(2-phenylethyl)tetrahydropyran-2-ol (21).⁸

Anomers ratio: 64/36.

IR: 3350; 1450 cm^{-1} .

Major anomer:

NMR ¹H (300 MHz, $CDCl_3$): δ 7.25-7.17 (m, 2H); 7.15-7.06 (m, 3H); 4.63 (ddd, $J = 8.5$ Hz, $J = 5.9$ Hz, $J = 1.8$ Hz, 1H); 3.35 (m, 1H); 3.07 (bs, 1H); 2.79-2.48 (m, 2H); 1.90-1.33 (m, 6H); 1.32-1.05 (m, 2H).

NMR ¹³C (75.45 MHz, $CDCl_3$): δ 142.1, 128.3 ($\times 2$), 128.2 ($\times 2$), 125.7, 96.4, 75.4, 37.5, 32.8, 31.6, 30.4, 22.0.

Minor anomer:

NMR ¹H (300 MHz, $CDCl_3$): δ 7.25-7.17 (m, 2H); 7.15-7.06 (m, 3H); 4.63 (bs, 1H); 3.88 (dddd, $J = 11.0$ Hz, $J = 7.4$ Hz, $J = 5.2$ Hz, $J = 1.8$ Hz, 1H); 2.79-2.48 (m, 2H); 2.46 (bs, 1H); 1.90-1.33 (m, 6H); 1.32-1.05 (m, 2H).

NMR ¹³C (75.45 MHz, $CDCl_3$): δ 142.2, 128.4 ($\times 2$), 128.3 ($\times 2$), 125.7, 91.9, 68.0, 37.8, 32.8, 30.3, 29.8, 17.3.

GC-MS m/z (EI) (rel. int.): 188 ($M^+ - 18$, 74), 117 (24), 104 (25), 91 (100), 77 (8), 55 (12).

6-(4-Methoxyphenoxyethyl)tetrahydropyran-2-ol (22).

Anomers ratio: 53/47.

IR: 3480; 1640; 1500 cm^{-1} .

Major anomer:

NMR ¹H (300 MHz, $CDCl_3$): δ 6.85 (dd, $J = 6.9$ Hz, $J = 1.1$ Hz, 2H); 6.81 (dd, $J = 6.9$ Hz, $J = 1.1$ Hz, 2H); 4.82 (ddd, $J = 5.9$ Hz, $J = 2.9$ Hz, $J = 1.8$ Hz, 1H); 4.43-4.32 (m, 1H); 4.23 (bs, 1H); 4.05-3.95 (m, 1H); 3.93-3.79 (m, 1H); 3.75 (s, 3H); 1.80-2.05 (m, 2H); 1.79-1.24 (m, 4H).

NMR ¹³C (75.45 MHz, $CDCl_3$): δ 153.8, 152.7, 115.5 ($\times 2$), 114.4 ($\times 2$), 96.3, 74.5, 71.4, 55.6, 32.2, 26.9, 21.5.

Minor anomer:

NMR ^1H (300 MHz, CDCl_3): δ 6.85 (dd, $J = 6.85$ Hz, $J = 1.10$ Hz, 2H); 6.81 (dd, $J = 6.85$ Hz, $^3J = 1.10$ Hz, 2H); 5.38 (bs, 1H); 4.10-3.80 (m, 3H); 3.74 (s, 3H); 3.62 (bs, 1H); 1.80-2.05 (m, 2H); 1.79-1.24 (m, 4H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 153.8, 152.9, 115.6 ($\times 2$), 114.5 ($\times 2$), 91.6, 72.1, 67.2, 55.6, 29.5, 27.4, 16.8.

GC-MS m/z (EI) (rel. int.): 238 (M^+ , 28), 220 (19), 138 (15), 124 (100), 109 (33), 77 (9), 55 (15).

HRMS: Calcd for $\text{C}_{13}\text{H}_{20}\text{O}_3\text{N}$: 238.1443. Found: 238.1447.

4,4-dipropyldihydrofuran-2-one (23).⁹

IR: 2940; 2870; 1770 cm^{-1} .

NMR ^1H (300 MHz, CDCl_3): δ 2.57 (t, $J = 8.6$ Hz, 2H); 2.01 (t, $J = 8.6$ Hz, 2H) 1.62 (dd, $J = 13.2$ Hz, $J = 6.6$ Hz, 2H); 1.61 (dd, $J = 9.6$ Hz, $J = 4.04$ Hz, 2H); 1.36 (dq, $J = 9.6$ Hz, $J = 7.4$ Hz, $J = 6.6$ Hz, 2H); 1.35 (dq, $J = 13.2$ Hz, $J = 7.4$ Hz, $J = 4.0$ Hz, 2H); 0.93 (t, $J = 7.4$ Hz, 6H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 176.9, 88.9, 40.9, 30.7, 29.0 ($\times 2$), 16.7 ($\times 2$), 14.2 ($\times 2$).

GC-MS m/z (EI) (rel. int.): 127 (M^+ -53, 100), 115 (10), 71 (12), 55 (8).

4,4-dipropyltetrahydrofuran-2-one (24).¹⁰

IR: 1730; 1460 cm^{-1} .

NMR ^1H (300 MHz, CDCl_3): δ 2.46 (t, $J = 6.6$ Hz, 2H); 1.90-1.78 (m, 2H); 1.76-1.67 (m, 2H); 1.66-1.45 (m, 4H); 1.44-1.07 (m, 4H); 0.92 (t, $J = 7.0$ Hz, 6H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 171.5, 86.3, 41.0 ($\times 2$), 30.0, 29.4, 16.6 ($\times 2$), 16.4, 14.3 ($\times 2$).

GC-MS m/z (EI) (rel. int.): 184 (M^+ , 0.2), 141 (100), 113 (58), 71 (28), 55 (17).

1-Oxaspiro(4.5)decan-2-one (25).¹¹

IR: 1760; 1460 cm^{-1} .

NMR ^1H (300 MHz, CDCl_3): δ 2.56 (t, $J = 8.5$ Hz, 2H); 1.98 (t, $J = 8.5$ Hz, 2H); 1.83-1.60 (m, 4H); 1.60-1.29 (m, 6H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 181.4, 86.2, 36.7 ($\times 2$), 32.6, 28.4, 24.7, 22.3 ($\times 2$).

GC-MS m/z (EI) (rel. int.): 154 (M^+ , 22), 125 (11), 111 (100), 98 (24), 55 (15).

1-Oxaspiro[5.5]undecan-2-one (26).¹²

$C_{10}H_{16}O_2$

IR: 1740; 1440 cm^{-1} .

NMR 1H (300 MHz, $CDCl_3$): δ 2.47 (d, $J = 7.0$ Hz, 2H); 1.90-1.77 (m, 4H); 1.77-1.62 (m, 4H); 1.61-1.19 (m, 6H).

NMR ^{13}C (75.45 MHz, $CDCl_3$): δ 171.2, 82.8, 37.1 ($\times 2$), 32.3, 29.3, 25.1, 21.5 ($\times 2$), 15.9.

GC-MS m/z (EI) (rel. int.): 168 (M^+ , 51), 125 (82), 112 (77), 97 (100), 83 (68), 77 (5), 55 (68).

(6*S,7*S**,10*R**) 7-Isopropyl-10-methyl-1-oxaspiro(5.5)undecan-2-one (28).**

$C_{14}H_{24}O_2$

IR: 1710; 1460 cm^{-1} .

NMR 1H (300 MHz, $CDCl_3$): δ 2.36 (t, $J = 5.9$ Hz, 2H); 2.07 (q, $J = 7.0$ Hz, $J = 6.3$ Hz, $J = 1.5$ Hz, 1H); 1.86-1.26 (m, 9H); 1.19-1.03 (m, 2H); 0.96-0.94 (m, 1H); 0.90 (d, $J = 6.6$ Hz, 3H); 0.88 (d, $J = 7.0$ Hz, 3H); 0.87 (d, $J = 6.3$ Hz, 3H).

NMR ^{13}C (75.45 MHz, $CDCl_3$): δ 178.9, 75.1, 47.9, 46.4, 40.3, 34.9, 34.4, 27.9, 25.4, 23.5, 22.3, 20.4, 19.2, 18.5.

GC-MS m/z (EI) (rel. int.): 224 (M^+ , 4); 168 (18), 139 (100), 111 (40), 95 (15), 81 (18), 55 (22).

HRMS: Calcd for $C_{14}H_{25}O_2$: 225.1855. Found: 225.1856.

1-Oxa-spiro[4.5]decan-2-ol (29).¹³

$C_9H_{16}O_2$

Anomers ratio: > 90/10.

Major anomer:

IR: 3400; 1440 cm^{-1} .

NMR 1H (300 MHz, $CDCl_3$): δ 5.49 (dd, $J = 3.3$ Hz, $J = 1.5$ Hz, 1H); 4.09 (sl, 1H); 2.03-1.81 (m, 3H); 1.80-1.52 (m, 5H); 1.51-1.25 (m, 6H).

NMR ^{13}C (75.45 MHz, $CDCl_3$): δ 97.8, 84.6, 39.7, 32.7, 25.3 ($\times 2$), 23.8 ($\times 3$).

GC-MS m/z (EI) (rel. int.): 154 (4), 125 (45), 83 (100), 70 (45), 55 (78).

1-Oxaspiro[5.5]undecan-2-ol (30).¹⁴

$C_{10}H_{18}O_2$

Anomers ratio: 86/14.

IR: 3420; 1440 cm^{-1} .

Major anomer:

NMR ^1H (300 MHz, CDCl_3): δ 5.10 (dd, $J = 8.1$ Hz, $J = 2.6$ Hz, 1H); 1.90 (sl, 1H); 1.82-1.10 (m, 16H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 95.6, 77.1, 39.7, 34.8, 32.9, 31.5, 26.3, 22.0, 21.8, 18.0.

Minor anomer:

NMR ^1H (300 MHz, CDCl_3): δ 4.62 (dd, $J = 8.5$ Hz, $J = 2.2$ Hz, 1H); 2.00 (sl, 1H); 1.82-1.10 (m, 16H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 92.8, 73.8, 39.1, 34.8, 32.9, 31.3, 26.2, 22.0, 21.8, 17.5.

GC-MS m/z (EI) (rel. int.): 170 (4), 152 (2), 99 (100), 81 (84), 55 (24).

(6*S,7*S**,10*R**) 7-Isopropyl-10-methyl-1-oxa-spiro[5.5]undecan-2-ol (32).**

Anomers ratio: 64/26.

IR: 3480; 1440 cm^{-1} .

Major anomer:

NMR ^1H (300 MHz, CDCl_3): δ 4.76 (dd, $J = 4.0$ Hz, $J = 1.8$ Hz, 1H); 2.54 (dt, $J = 12.9$ Hz, $J = 2.9$ Hz, 1H); 2.21-0.93 (m, 15H); 0.93-0.82 (m, 9H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 97.7, 75.2, 51.5, 47.7, 46.8, 41.1, 35.1, 33.1, 31.5, 30.7, 28.0, 23.6, 22.7, 18.2.

Minor anomer:

NMR ^1H (300 MHz, CDCl_3): δ 4.56 (dd, $J = 9.9$ Hz, $J = 1.8$ Hz, 1H); 2.31 (dt, $J = 14.7$ Hz, $J = 2.6$ Hz, 1H); 2.21-0.93 (m, 15H); 0.93-0.82 (m, 9H).

NMR ^{13}C (75.45 MHz, CDCl_3): δ 95.8, 77.2, 53.5, 47.6, 46.7, 41.3, 33.6, 33.6, 30.7, 30.7, 28.2, 24.2, 22.5, 18.5.

GC-MS m/z (EI) (rel. int.): 226 (4), 208 (2), 155 (42), 141 (100), 95 (70), 81 (58), 55 (30).

HRMS: Calcd for $\text{C}_{13}\text{H}_{20}\text{O}_3\text{N}$: 226.2171. Found: 226.2163.

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