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Diastereoselective Radical Mediated Hydrogen Atom Abstraction**

Philippe Renaud,* Florent Beaufils, Laurence Feray and Kurt Schenk

P. Renaud, F. Beaufils
University of Berne
Department of Chemisty and Biochemistry
Freiestrasse 3
CH-3000 Berne 9, Switzerland
Fax +41 31 631 34 26
philippe.renaud@ioc.unibe.ch

Dr. L. Feray
University of Fribourg
Department of Chemistry
Pérolles
CH-1700 Fribourg, Switzerland

Dr. K. Schenk
University of Lausanne
Institute of Crystallography, BP
CH-1015 Lausanne, Switzerland

General procedure 1 (GP1): halogenoacetalization. To a solution of alcohol (5.0 mmol) in dichloromethane (5 mL) was added at –20°C, under nitrogen atmosphere, methoxyallene (350 mg, 5.0 mmol); *N*-halogenosuccinimide (10 mmol) was then added portionwise. The solution was slowly warmed to 40°C and stirred until total disappearance of the starting alcohol (2-4 h, TLC monitoring). The reaction mixture was washed with an aqueous solution of KOH (5%), water, and brine. After drying ($MgSO_4$), the solution was concentrated under reduced pressure. FC (AcOEt/hexane) gave the desired halogenoacetal as colorless oil.

General procedure 2 (GP2): tin hydride mediated H-abstraction. To a solution of iodoacetal (1.00 mmol) in *t*BuOH (100 mL) were added tributyltin chloride (13 μL, 0.05 mmol) and sodium cyanoborohydride (126 mg, 2.00 mmol) at room temperature under nitrogen atmosphere. The reaction mixture was stirred at reflux during 5 h. After disappearance of the starting iodoacetal (TLC monitoring), the solution was colded and *t*BuOH evaporated under reduced pressure. The residue was filtrated through silica gel. The filtrate was then evaporated under reduce pressure and flash chromatography (AcOEt/hexane or Et_2O/pentane) of the residue afforded the desired cyclic compounds. The ratio of isomers was determined from ^1H NMR spectra of the crude product.

2-(2-Iodo-1-methoxyallyloxy)indan (1a). According to GP1, starting from 2-indanol (5.37 g, 40 mmol), NIS (18 g, 80 mmol) and methoxyallene (2.80 g, 40 mmol). FC (hexane/AcOEt 95:5) gave **1a** (10.03 g, 76%). ^1H NMR (300MHz, $CDCl_3$) δ: 7.22–7.13 (m, 4H, Harom), 6.58 (s, 1H), 6.06 (s, 1H), 4.63 (s, 1H), 4.59 (quint, 1H, *J* = 5.1), 3.34 (s, 3H), 3.27-3.00 (m, 4H). ^{13}C NMR (400 MHz, $CDCl_3$) δ: 39.2 (CH_2), 39.6 (CH_2), 52.7 (CH_3), 77.3 (CH), 103.4 (CH), 108.3 (C), 124.3 (CH), 124.4 (CH), 126.4 (2×CH), 128.3 (CH_2), 140.2 (2×C). IR (KBr): 3047, 2942, 2831, 2361, 1618, 1483, 1460, 1316, 1265, 1203, 1075, 1052, 922, 738, 668 cm^{-1}. EI (MS) m/z (%): 330 (M^+, 4), 203 (8), 197 (40), 182 (49), 127 (21), 117 (47), 116 (100), 115 (81), 105(11), 89 (12), 63 (14), 39 (13). HRMS for $C_{13}H_{15}O_2I$ [M^+]: calcd 330.0117, found 330.0102.

(2-Iodo-1-methoxyallyloxy)cyclopentane (1b). According to GP1, starting from cyclopentanol (344 mg, 4.0 mmol), NIS (1.8 g, 8.0 mmol) and methoxyallene (280

mg, 4.0 mmol). FC (hexane/AcOEt 95:5) gave **1b** (902 mg, 80%). ^{1}H NMR (300 MHz, $CDCl_3$) □: 6.55 (s, 1H), 6.04 (s, 1H), 4.48 (s, 1H), 4.16 (m, 1H), 3.31 (s, 3H), 1.76-1.72 (m, 6H), 1.60-1.54 (m, 2H). ^{13}C NMR (400 MHz, $CDCl_3$) □: 23.2 (CH_2), 23.3 (CH_2), 31.2 (CH_2), 32.7 (CH_2), 53.6 (CH_3), 78.7 (CH), 103.2 (CH), 109.3 (C), 127.8 (CH_2). IR (KBr): 2960, 2872, 2829, 1617, 1437, 1331, 1265, 1180, 1074, 990, 919, 738, 703 cm^{-1}. CI (MS) m/z (%): 282 (M^+, 3), 251 (5), 197 (100), 183 (25), 182 (87), 167 (23), 153 (15), 129 (44), 127 (45), 95 (31), 69 (31), 67 (87), 61 (47), 55 (25), 53 (27), 41 (70), 39 (59). HRMS for $C_9H_{15}O_2I$ [M^+]: calcd, 282.0117, found 282.0103.

(2-Iodo-1-methoxyallyloxy)tetrahydrothiopyrane (1c). According to GP1, starting from tetrahydro-4H-thiopyran-4-ol (351 mg, 3.0 mmol), NIS (1.35 g, 6.0 mmol) and methoxyallene (210 mg, 3.0 mmol). FC (hexane/AcOEt 8:1) gave **1c** (763 mg, 81%). ^{1}H NMR (300 MHz, $CDCl_3$) □: 6.58 (s, 1H), 6.07 (s, 1H), 4.55 (s, 1H), 3.67 (m, 1H), 3.29 (s, 3H), 2.91-2.83 (m, 2H), 2.57-2.46 (m, 2H), 2.15-2.02 (m, 2H), 1.98-1.82 (m, 2H). ^{13}C NMR (400 MHz, $CDCl_3$) □: 25.4 (CH_2), 25.5 (CH_2), 32.5 (CH_2), 33.3 (CH_2), 52.4 (CH_3), 72.5 (CH), 102.5 (CH), 108.7 (C), 127.9 (CH_2). IR (KBr): 3050, 2936, 2830, 1617, 1428, 1266, 1204, 1077, 1044, 920, 737, 704 cm^{-1}. CI (MS) m/z (%): 314 (M^+, 20), 283 (12), 282 (16), 214 (39), 197 (66), 187 (52), 182 (100), 155 (55), 127 (48), 117 (52), 101 (92), 100 (71), 85 (46), 67 (52), 55 (51), 45 (38), 39 (52). HRMS for $C_9H_{15}O_2IS$ [M^+]: calcd, 313.9838, found 313.9847.

(2-Iodo-1-methoxyallyloxy)cycloheptane (1d). According to GP1, starting from cycloheptanol (457 mg, 4.0 mmol), NIS (1.80 g, 8.0 mmol) and methoxyallene (280 mg, 4.0 mmol). FC (hexane/AcOEt 95:5) gave **1d** (943 mg, 76%). ^{1}H NMR (300 MHz, $CDCl_3$) □: 6.56 (dd, 1H, J=1.1, 1.48), 6.03 (dd, 1H, J= 1.48, 0.74), 4.49 (d, 1H, J= 0.74), 3.74 (m, 1H), 3.29 (s, 3H), 1.96-1.1.80 (m, 2H), 1.77-1.62 (m, 4H), 1.57-1.52 (m, 4H), 1.43-1.32 (m, 2H). ^{13}C NMR (400 MHz, $CDCl_3$) □: 22.7 (CH_2), 22.8 (CH_2), 28.2 (CH_2), 28.3 (CH_2), 33.7 (CH_2), 34.5 (CH_2), 52.3 (CH_3), 77.4 (CH), 102.7 (CH), 109.56 (C), 127.7 (CH_2). IR (KBr): 2928, 2857, 1618, 1460, 1343, 1343, 1269, 1179, 1046, 1078, 917, 738, 703 cm^{-1}. CI (MS) m/z (%): 310 (M^+, 6), 279 (17), 214 (19), 197 (90), 182 (100). 157 (35), 127 (49), 97 (82), 81 (82), 67 (73), 55 (93), 41 (55), 39 (61). HRMS for $C_{11}H_{19}O_2I$ [M^+]: calcd 310.0430, found 310.0443.

2-Bromo-3-(1-isopropyl-2-methylpropoxy)-3-methoxypropene (1e). According to GP1, starting from 2,4-dimethyl-3-pentanol (1.66 g, 14.3 mmol), NBS (5.10 g, 28.6 mmol) and methoxyallene (1.00 g, 14.3 mmol). FC (hexane/AcOEt 90:10) gave **1e** (2.65 g, 70%). ^{1}H NMR (360 MHz, $CDCl_3$) : 6.13 (s, 1H), 5.75 (s, 1H), 4.82 (s, 1H), 3.27 (s, 3H), 3.15 (t, 1H, *J* = 5.5). 1.88 (oct, 2H, *J* = 6.3), 0.96 (d, 3H, *J* = 5.5), 0.94 (d, 3H, *J* = 5.5), 0.92 (d, 3H, *J* = 7.2), 0.90 (d, 3H, *J* =7.2). ^{13}C NMR (90 MHz, $CDCl_3$) : 18.2 (CH_3), 18.6 (CH_3), 20.6 (CH_3), 20.7 (CH_3), 30.6 (CH), 31.2 (CH), 52.2 (CH_3), 89.6 (CH), 104.0 (CH), 120.2 (CH_2), 130.4 (C). IR (KBr): 2963, 2876, 2832, 1742, 1663, 1630, 1468, 1387, 1335, 1207, 1078, 1047, 986, 910 cm^{-1}. EI (MS) m/z (%): 266 (M^+, 5), 264 (M^+, 5), 235 (97), 233 (100), 194 (6), 192 (6), 151 (27), 149 (27), 99 (97), 70 (5), 58 (67). HRMS for $C_{11}H_{22}O_2Br$ [MH^+, ^{79}Br]: calcd 265.07976, found 265.07963; [MH^+, ^{81}Br]: calcd 267.0777, found 267.0781.

1-[(2-Bromo-1-methoxy-allyloxy)cyclohexylmethyl]cyclohexane (1f). According to GP1, starting from dicyclohexylmethanol (2.80 g, 14.3 mmol), NBS (5.10 g, 28.6 mmol) and methoxyallene (1.00 g, 14.3 mmol). FC (hexane/AcOEt 99:1) gave **lf** (3.25g, 66%). ^{1}H NMR (360 MHz, $CDCl_3$) : 6.11 (s, 1H), 5.73 (s, 1H), 4.77 (s, 1H), 3.26 (s, 3H), 3.14 (t, 1H, *J* = 4.5), 1.87 1.48 (m, 11H), 1.29 0.99 (m, 11H). ^{13}C NMR (90 MHz, $CDCl_3$) : 26.8 (CH_2), 26.9 (CH_2), 26.9 (CH_2), 27.0 (CH_2), 27.1 (CH_2), 27.1 (CH_2), 28.5 (CH_2), 28.8 (CH_2), 31.0 (CH_2), 31.2 (CH_2), 40.3 (CH), 41.1 (CH), 52.6 (CH_3), 88.4 (CH), 104.2 (CH), 120.1 (CH_2), 130.5 (C). IR (KBr): 2922, 2855, 2666, 1630, 1448, 1350, 1203, 1078, 979, 908 cm^{-1}. EI (MS) m/z (%): 315 (M^+ OMe, 3), 313 (M^+ OMe, 3), 263 (3), 261 (3), 232 (25), 179 (86), 151 (100), 121 (8), 98 (11), 83 (26), 56 (32). HRMS for $C_{17}H_{29}O_2Br$ [MH^+, ^{79}Br]: calcd 344.13454, found 344.13426; [MH^+, ^{81}Br]: calcd 346.1325, found 346.1323.

1-{2-[(2-Iodo-1-methoxy-2-propenyloxy)-3-phenylpropyl]}benzene (1g). According to GP1, starting from 1,3-diphenylpropan-2-ol (2.09 g, 11.3 mmol), NIS (2.55 g, 22.7 mmol) and methoxyallene (794 mg, 11.3 mmol). FC (hexane/AcOEt 95:5) gave **1g** (2.96 g, 64%). ^{1}H NMR (300 MHz, $CDCl_3$) : 7.07 7.22 (m, 10H, Harom), 6.28 (s, 1H), 5.91 (s, 1H), 4.13 (s, 1H), 3.98 (quint, 1H, *J* = 6.36), 2.84 (s, 3H), 2.75 (m, 4H). ^{13}C NMR (400 MHz, $CDCl_3$) : 41.0 (CH_2), 41.3 (CH_2), 51.9

(CH_3), 79.7 (CH), 103.7(CH), 109.0 (C), 126.6 (2□CH), 128.6 (2□CH), 128.7 (2□CH), 128.9 (CH_2), 129.9 (2□CH), 130.1 (2□CH), 138.8 (C), 138.9 (C). IR (KBr): 3028, 2929, 2830, 1617, 1496, 1453, 1265, 1080, 1040, 918, 739, 701 cm^{-1}. CI (MS) m/z (%): 408 (M^+, 5), 317 (29), 197 (54), 194 (100), 193 (67), 182 (66), 179 (41), 116 (42), 115 (67), 91 (85), 65 (21), 39 (19). HRMS for $C_{19}H_{21}O_2I$ [M^+]: calcd, 408.0586, found 408.0579.

Methyl 3-methyl-3,3a,8,8a-tetrahydro-2*H*-indeno[2,1-6]furan-2-yl ether (2a). According to GP2, from **1a** (283 mg, 1.00 mmol). FC (hexane/AcOEt 90:10) gave **2a** (137 mg, 67%) as a mixture of 4 diastereomers in a 57/23/11/9 ratio. Isolation of the two major diastereomers was performed by FC (hexane/AcOEt 99:1).

(*r*-2,*c*-3,*t*-4,*t*-5)-**2a**: (57% ds) ^{1}H NMR (500 MHz, $CDCl_3$) □: 7.13-7.25 (m, 4H), 5.01 (dt, 1H, *J* = 2.6, 7.4), 4.84 (d, 1H, *J* = 4.6), 3.43 (t, 1H, *J* = 7.1), 3.39 (s, 3H), 3.26 (dd, 1H, *J* = 7.5, 17.4), 3.06 (dd, 1H, *J* = 2.6, 17.4), 2.13 (dquint, 1H, *J* =4.6, 7.2), 1.25 (d, 3H, *J* = 7.2). ^{13}C NMR (125 MHz, $CDCl_3$) □: 13.4 (CH_3), 39.1 (CH_2), 46.4 (CH), 54.7 (CH_3), 55.8 (CH), 81.8 (CH), 107.5 (CH), 123.8 (CH), 125.2 (CH), 126.9 (CH), 127.2 (CH), 141.0 (C), 145.3 (C). NOE difference spectra (500 MHz): 4.99-5.03 ((CH_2)C*H*O)□ 3.41-3.46 (6.29%), 3.36-3.40 (1.25%), 3.22-3.29 (5.09%); 4.83-4.84 (C*H*OMe) □ 3.36-3.40 (5.67%), 2.11-2.19 (7.67%); 3.41-3.46 (C*H*Ph) □ 4.99-5.03 (4.84%), 2.11-2.19 (1.09%), 1.21-1.29 (2.24%); 3.22-3.29 (C*H*HPh) □ 4.99-5.03 (6.63%), 3.02-3.08 (19.7%); 3.02-3.08 (CH*H*Ph) □ 4.83-4.84 (0.26%), 3.22-3.29 (21.53%), 2.11-2.19 (0.41%); 2.11-2.19 (C*H*Me) □ 7.20-7.25 (2.34%), 4.83-4.84 (9.42%), 1.21-1.29 (4.10%).

(*r*-2,*t*-3,*t*-4,*t*-5)-**2a**: (23% ds) ^{1}H NMR (500 MHz, $CDCl_3$) □: 7.15-7.21 (m, 4H), 5.04 (dt, 1H, *J* = 2.7, 7.3), 4.64 (d, 1H, *J* = 1.9), 3.99 (t, 1H, *J* = 7.9), 3.37 (s, 3H), 3.23 (dd, 1H, *J* = 7.3, 17.4), 3.05 (dd, 1H, *J* = 2.7, 17.4), 2.55 (dquint, 1H, *J* =7.9, 7.4), 0.80 (d, 3H, *J* = 7.4). ^{13}C NMR (125 MHz, $CDCl_3$) □: 14.7 (CH_3), 39.6 (CH_2), 42.5 (CH), 52.2 (CH_3), 54.9 (CH), 82.7 (CH), 112.5 (CH), 125.1 (CH), 126.3 (CH), 126.4 (CH), 127.1 (CH), 140.9 (C), 142.8 (C). NOE difference spectra (500 MHz): 5.01-5.04 ((CH_2)C*H*O) □ 3.96-4.01 (4.37%), 3.36-3.38 (1.00%), 3.20-3.29 (4.51%); 4.63-4.65 (C*H*OMe) □ 3.36-3.38 (3.41%), 2.51-2.59 (1.26%) 0.80-0.83 (1.71%); 3.96-4.01 (C*H*Ph) □ 5.01-5.04 (5.54%), 2.51-2.59 (7.02%); 3.20-3.29 (C*H*HPh) □ 5.01-5.04 (5.12%), 3.02-3.08 (13.0%); 3.02-3.08 (CH*H*Ph) □ 3.20-3.29 (17.69%); 2.51-

2.59 (C*H*Me) → 3.96-4.01 (5.31%), 0.80-0.83 (2.57%); 0.80-0.83 (CHCH_3) → 4.63-4.65 (1.59%), 2.51-2.59 (2.23%). Mixture of isomers: IR (KBr): 2930,2831, 1480, 1459, 1379, 1271, 1191, 1078, 1027, 929, 738, 668 cm^{-1}. EI (MS) m/z (%): 204 (M^+, 3), 172 (36), 144 (31), 129 (100), 115 (71), 103 (13), 91 (15), 77 (15), 63 (10), 51 (9). HRMS for $C_{13}H_{17}O_2$ [MH^+]: calcd 205.1223, found 205.1225.

2-Methoxy-3-methylhexahydrocyclopenta[*b*]furan (2b). According to GP2, from **1b** (240 mg, 1.00 mmol). FC (hexane/AcOEt 90:10) gave **2b** (69 mg, 44%) as a mixture of 3 diastereomers in a 62/27/11 ratio. The major diastereomer (*r*-2,*c*-3,*t*-4,*t*-5)-**2b** was isolated by FC (hexane/AcOEt 99:1). ^{1}H NMR (400 MHz, $CDCl_3$) : 4.59-4.62 (m, 1H), 4.58 (d, 1H, *J* = 3.1), 3.35 (s, 3H), 2.58-2.65 (m, 1H), 2.30 (ddq, 1H, *J* = 3.1, 8.6, 7.4), 1.55-1.68 (m, 6H), 1.01 (d, 3H, *J* = 7.4). ^{13}C NMR (400 MHz, $CDCl_3$) : 13.4 (CH_3), 25.7 (CH_2), 26.0 (CH_2), 33.1 (CH_2), 42.7(CH), 45.8 (CH), 55.2 (CH_3), 85.1 (CH), 112.5 (CH). NOE difference spectra (400 MHz): 4.63-4.59 (CH_2C*H*O) → 3.36-3.28 (0.97%), 2.65-2.59 (1.21%), 1.05-0.94 (2.8%); 3.36-3.28 (CH_3OCHO) → 4.60-4.57 (0.87%); 1.05-0.94 (0.6%); 2.65-2.59 (CH_2C*H*CHO) → 4.63-4.59 (1.75%), 3.36-3.28 (0.8%), 2.34-2.26 (2.1%). IR (KBr): 2958, 2218, 1732, 1464, 1367, 1274, 1110, 1018, 938, 739 cm^{-1}. CI (MS) m/z (%): 156 (M^+, 3), 155 (16), 139 (46), 125 (100), 98 (19), 97 (24), 67 (23), 62 (5). HRMS for $C_8H_{13}O$ [M^+–OMe]: calcd 125.0961, found 125.0961.

2-Methoxy-3-methylhexahydrothiopyrano[4,3b]furan (2c). According to GP2, from **1c** (408 mg, 1.00 mmol). FC (hexane/AcOEt 90:10) gave **2c** (141 mg, 44%) as a mixture of four diastereomers in a 60/34/3/3 ratio. ^{1}H NMR (300 MHz, $CDCl_3$) for the mixture of the two major diastereoisomers *c*-3 (= *r*-2,*c*-3,*t*-4,*t*-5: 66% ds) and *t*-3 (*r*-2,*t*-3,*t*-4,*t*-5: 34% ds) : 4.85 (d, J= 4.78, *t*-3), 4.62 (d, J= 5.52, *c*-3), 4.2 (m, *t*-3 + *c*-3), 3.4 (s, *c*-3), 3.35 (s, *t*-3), 2.9-2.7 (m, *t*-3 + *c*-3), 2.55-1.9 (m, *t*-3 + *c*-3), 1.08 (d, J=6.99, *c*-3), 1.02 (d, J= 6.98, *t*-3). ^{13}C NMR (400 MHz, $CDCl_3$) : 11.4 (CH_3, *c*-3), 12.2 (CH_3, *t*-3), 22.4 (CH_2, *t*-3), 23.4 (CH_2, *c*-3), 25.8 (CH_2, *c*-3), 26.5 (CH_2, *t*-3), 28.5 (CH_2, *t*-3), 29.6 (CH_2, *c*-3), 41.5 (CH, *t*-3), 42.5 (CH, *c*-3), 43.6 (CH, *t*-3), 45.7 (CH, *c*-3), 55.3 (CH_3, *t*-3), 56.7 (CH_3, *c*-3), 74.8 (CH, *t*-3), 75.5 (CH, *c*-3), 105.5 (CH, *t*-3), 110.7 (CH, *c*-3). Mixture of isomers: IR (KBr): 3049, 2934, 2832, 1460, 1423,1382, 1265, 1191, 1118, 1096,1005, 950, 910, 737, 704 cm^{-1}. CI (MS) m/z (%): 188 (M^+,

67), 157 (57), 128 (28), 99 (100), 85 (43), 67 (44), 45 (29), 41 (39), 39 (25). HRMS for $C_9H_{16}O_2S$ [M^+]: calcd 188.0871, found 188.0871.

2-Methoxy-3-methyloctahydrocyclohepta[*b*]furan (2d). According to GP2, from **1d** (263 mg, 1.00 mmol). FC (hexane/AcOEt 90:10) gave **2d** (144 mg, 78%) as a mixture of 4 diastereomers in a 57/23/11/9 ratio. Isolation of the 2 major diastereomers was done by flash chromatography (hexane/AcOEt 99:1).

(*r*-2,*c*-3,*t*-4,*t*-5)-**2d**: (57% ds) ^{1}H NMR (500 MHz, $CDCl_3$) δ: 4.72 (d, 1H, *J* = 4.5), 4.22 (ddd, 1H, *J* = 3.1, 9.1, 11.9), 3.33 (s, 3H), 2.00-2.10 (m, 1H), 1.71-1.86 (m, 2H), 1.10-1.69 (m, 9H), 0.97 (d, 3H, *J* = 6.8). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 11.9 (CH_3), 25.6 (CH_2), 28.1 (CH_2), 29.6 (CH_2), 31.4 (CH_2), 31.9 (CH_2), 44.8 (CH), 47.2 (CH), 54.4 (CH_3), 82.4 (CH), 105.9 (CH). NOE difference spectra (500 MHz): 4.69-4.75 (C*H*OMe) → 3.31-3.35 (3.13%), 1.71-1.80 (4.14%); 4.18-4.24 (OC*H*CH_2) → 3.31-3.35 (0.79%), 2.00-2.10 (4.06%); 3.31-3.35 (OC*H*$_3$) → 4.69-4.75 (1.25%), 4.18-4.24 (0.24%); 2.00-2.10 (CHC*H*CH_2) → 4.18-4.24 (4.17%), 3.31-3.35 (0.52%), 0.94-1.00 (2.33%).

(*r*-2,*t*-3,*t*-4,*t*-5)-**2d**: (23% ds) ^{1}H NMR (500 MHz, $CDCl_3$) δ: 4.59 (d, 1H, *J* = 1.2), 4.19 (ddd, 1H, *J* = 3.7, 8.7, 11.9), 3.33 (s, 3H), 2.50-2.59 (m, 1H), 2.14-2.23 (m, 1H), 1.10-1.96 (m, 10H), 0.94 (d, 3H, *J* = 7.4). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 13.9 (CH_3), 25.3 (CH_2), 25.6 (CH_2), 28.6 (CH_2), 31.2 (CH_2), 32.6 (CH_2), 43.5 (CH), 43.8 (CH), 54.5 (CH_3), 82.7 (CH), 110.5 (CH). NOE difference spectra (500 MHz): 4.58-4.60 (C*H*OMe) → 3.31-3.35 (3.29%), 2.14-2.23 (1.41%), 0.91-0.96 (2.26%); 4.16-4.22 (OC*H*CH_2) → 3.31-3.35 (0.71%), 2.50-2.59 (3.96%), 2.14-2.22 (0.47%); 3.31-3.35 (OC*H*$_3$) → 4.58-4.60 (1.30%), 4.16-4.22 (0.24%), 2.14-2.23 (0.13%); 2.50-2.59 (CHC*H*CH_2) → 4.16-4.22 (4.22%), 2.14-2.23 (4%); 2.14-2.23 (C*H*Me) → 4.58-4.60 (1.36%), 4.16-4.22 (0.30%), 2.50-2.59 (2.53%), 0.91-0.96 (2.30%); 0.91-0.96 (CHC*H*$_3$) → 4.58-4.60 (1.41%), 2.14-2.23 (1.82%).

Mixture of isomers: IR (KBr): 2926, 2854, 1658, 1456, 1379, 1195, 1095, 1008, 947 cm^{-1}. CI (MS) m/z (%): 184 (M^+, 5), 183 (21), 67 (10), 153 (100), 125 (10), 96 (8), 84 (10), 61 (5). HRMS for $C_{10}H_{17}O$ [M^+–OMe]: calcd 153.1274, found 153.1271.

2-Isopropyl-5-methoxy-3,3,4-trimethyltetrahydrofuran (2e). According to GP2, from **1e** (265 mg, 1.00 mmol). FC (hexane/AcOEt 90:10) gave **2e** (149 mg, 80%) as a

mixture of 4 diastereomers in a 52/35/9/4 ratio. Isolation of the two major diastereomers was done by flash chromatography (hexane/AcOEt 99:1).

(*r*-2,*c*-3,*t*-5)-**2e** (52% ds): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 4.78 (d, 1H, *J* = 5.3), 3.32 (s, 3H), 3.28 (d, 1H, *J* = 8.1), 1.85 (dq, 1H *J* = 5.3, 7.3), 1.78 (dhept, 1H, *J* = 8.1, 6.6), 0.99 (d, 3H, *J* = 6.6), 0.97 (s, 3H), 0.96 (s, 3H), 0.93 (d, 3H, *J* = 6.6), 0.88 (d, 3H, *J* = 7.3). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 8.7 (CH_3), 20.3 (CH_3), 20.4 (CH_3), 24.2 (CH_3), 24.8 (CH_3), 29.3 (CH), 40.9 (C), 49.2 (CH), 54.6 (CH_3), 90.6 (CH), 104.9 (CH). NOE difference spectra (500 MHz): 4.77-4.79 (C*H*OMe) → 3.31-3.33 (3.46%), 1.82-1.89 (3.57%); 3.31-3.33 (OCH_3) → 4.77-4.79 (1.40%); 3.26-3.29 (OC*H*iPr) → 1.74-1.82 (0.52%); 1.82-1.89 (C*H*Me) → 4.77-4.79 (3.29%), 0.87-0.89 (4%); 1.74-1.82 (C*H*(Me)$_2$) → 4.77-4.79 (0.17%), 0.91-1.01 (9.8%); 0.87-0.89 (CHCH_3) → 3.31-3.33 (0.29%), 1.82-1.89 (1.17%), 3.26-3.29 (0.16%).

(*r*-2,*t*-3,*t*-5)-**2e** (35% ds): ^{1}H NMR (500 MHz, $CDCl_3$) δ: 4.49 (d, 1H, *J* = 5.8), 3.39 (s, 3H), 3.26 (d, 1H, *J* = 9.4), 1.78 (dq, 1H *J* = 5.8, 7.2), 1.73 (dhept, 1H, *J* = 6.6, 9.4), 1.05 (d, 3H, *J* = 6.6), 0.98 (s, 3H), 0.94 (d, 3H, *J* = 7.2), 0.91 (d, 3H, *J* = 6.6), 0.78 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 9.9 (CH_3), 15.5 (CH_3), 18.7 (CH_3), 21.7 (CH_3), 24.3 (CH_3), 29.5 (CH), 43.3 (C), 51.9 (CH), 55.8 (CH_3), 91.8 (CH), 109.2 (CH). NOE difference spectra (500 MHz): 4.48-4.50 (C*H*OMe) → 3.38-3.40 (3.54%), 1.73-1.79 (1.16%); 3.38-3.40 (OCH_3) → 4.48-4.50 (1.55%); 3.25-3.27 (OC*H*iPr) → 1.76-1.80 (3.96%); 0.97-0.99 (C(CH_3)$_2$) → 3.25-3.27 (0.98%), 1.76-1.80 (1.66%); 0.77-0.79 (C(CH_3)$_2$) → 4.48-4.50 (0.88%), 1.71-1.74 (2.13%), 0.93-0.95 (0.56%). Mixture of isomers: IR (KBr): 2963, 2876, 2835, 2247, 1697, 1470, 1450, 1380, 1204, 1101, 1020, 912, 735 cm^{-1}. EI (MS) m/z (%): 155 (M^+–OMe, 22), 143 (20), 114 (37), 99 (100), 97 (12), 83 (29), 70 (13), 56 (36). HRMS for $C_{11}H_{23}O_2$ [MH^+]: calcd 187.1693, found 187.1699.

1-Cyclohexyl-3-methoxy-4-methyl-2-oxaspiro[4.5]decane (2f). According to GP2, from **1f** (345 mg, 1.00 mmol). FC (hexane/AcOEt 90:10) gave **2f** (215 mg, 81%) as a mixture of 4 diastereomers in a 60/26/12/2 ratio. The major diastereomer was isalted by flash chromatography (hexane/AcOEt 99:1).

(*r*-2,*c*-3,*t*-5)-**2f** (60% ds)**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ: 4.86 (d, 1H, *J* = 5.7), 3.60 (d, 1H, *J* = 4.7), 3.36 (s, 3H), 2.12 (dq, 1H, *J* = 5.7, 7.2), 1.07-1.85 (m, 21H), 0.91 (d, 3H, *J* = 7.2). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 8.4 (CH_3), 23.4 (CH_2), 23.6 (CH_2), 26.0

(CH_2), 26.1 (CH_2), 26.3 (CH_2), 26.6 (CH_2), 29.5 (CH_2), 30.8 (CH_2), 30.9 (CH_2), 31.5 (CH_2), 38.9 (CH), 45.0 (CH), 45.8 (C), 55.6 (CH_3), 88.5 (CH), 105.9 (CH). NOE difference spectra (500 MHz): 4.84-4.88 (C*H*OMe) → 3.33-3.38 (4.95%), 2.08-2.16 (4.40%); 3.58-3.62 (OC*H*Cy) → 1.07-1.85 (11.21%), 0.88-0.93 (3.06%); 3.33-3.38 (OCH_3) → 4.84-4.88 (1.67%); 2.08-2.16 (C*H*Me) → 4.84-4.88 (4.96%), 0.88-0.93 (7.48%); 0.88-0.93 (CHCH_3) → 3.58-3.62 (0.48%), 3.33-3.38 (0.28%), 2.08-2.16 (1.65%). Mixture of isomers: IR (KBr): 2924, 2854, 1743, 1707, 1450, 1379, 1194, 1078, 1006, 900 cm^{-1}. EI (MS) m/z (%): 266 (M^+, 3), 265 (M^+–1, 15), 235 (66), 183 (92), 154 (100), 122 (53), 111 (30), 95 (16), 82 (40), 55 (77). HRMS for $C_{17}H_{31}O_2$ [MH^+]: calcd 267.2318, found 267.2318.

2-Benzyl-5-methoxy-4-methyl-3-phenyltetrahydrofuran (2g). According to GP2, from **1g** (408 mg, 1.0 mmol). FC (hexane/AcOEt 90:10) gave **2g** (208 mg, 74%) as a mixture of 4 diastereomers in a 65/20/8/7 ratio. The two major diastereomers were isolated by FC (hexane/AcOEt 99:1).

(*r*-2,*c*-3,*c*-4,*t*-5)-**2g** (65% ds): ^{1}H NMR (360 MHz, $CDCl_3$) □: 7.15 (m, 10H), 4.61 (d, 1H, J=3.18), 4.29 (m, 1H), 3.32 (s, 3H), 2.81 (m, 1H), 2.65 (m, 2H), 2.19 (m, 1H), 0.94 (d, 3H, J=7.27). ^{13}C NMR (360 MHz, $CDCl_3$) □: 16.8 (CH_3), 39.1 (CH_2), 49.8 (CH_3), 55.3 (CH), 58.9 (CH), 84.5 (CH), 110.8 (CH), 126.0 (CH), 126.8 (CH), 128.0 (2□CH), 128.2 (2□CH), 128.6 (2□CH), 129.3 (2□CH), 138.5 (C), 139.8 (C). NOE difference spectra (500 MHz): 4.51-4.72 ((CH$_2$)C*H*O) → 3.22-3.44 (4.65%), 2.29-2.51 (1.08%), 2.08-2.29 (2.04%), 0.86-1.07 (3.17%); 4.13-4.34 (C*H*OMe) → 4.51-4.72 (0.48%), 3.22-3.44 (0.73%), 2.75-2.96 (2.78%), 2.53-2.75 (1.72%), 2.29-2.51 (1.05%), 2.08-2.29 (2.6%), 0.86-1.07 (0.22%); 2.75-2.96 (C*H*HPh) → 4.51-4.72 (0.38%), 4.13-4.34 (4.12%), 3.22-3.44 (0.42%), 2.53-2.75 (5.65%), 2.29-2.51 (1.18%), 0.86-1.07 (0.38%); 2.53-2.75 (CH*H*Ph) → 4.51-4.72 (0.47%), 4.13-4.34 (1.6%), 3.22-3.44 (0.79%), 2.75-2.96 (4.17%), 2.29-2.51 (3.22%), 0.86-1.07 (0.71%); 2.29-2.51 (C*H*Ph) → 4.51-4.72 (1.6%), 4.13-4.34 (1.66%), 3.22-3.44 (0.72%), 2.75-2.96 (1.76%), 2.53-2.75 (3.63%), 0.86-1.07 (4.11%); 2.08-2.29 (C*H*Me) → 4.51-4.72 (2.35%), 4.13-4.34 (2.89%), 3.22-3.44 (0.96%), 2.29-2.51 (1.6%), 0.86-1.07 (4.3%)

(*r*-2,*t*-3,*c*-4,*t*-5)-**2g** (20% ds): ^{1}H NMR (360 MHz, $CDCl_3$) □: 7.14 (m, 10H), 4.88 (d, 1H, J=4.99), 4.41 (m, 1H), 3.33 (s, 3H), 3.1 (m, 1H), 2.87 (m, 2H), 2.43 (m, 1H), 0.54 (d, 3H, J=7.72). ^{13}C NMR (360 MHz, $CDCl_3$) □: 10.07 (CH_3), 40.4 (CH_2), 41.4 (CH),

52.0 (CH_3), 54.2 (CH), 84.3 (CH), 106.5 (CH), 126.1 (CH), 126.3 (CH), 127.9 (2□CH), 128.1 (2□CH), 129.4 (2□CH), 129.9 (2□CH), 138.8 (C), 140.3 (C). NOE difference spectra (500 MHz): 4.80-4.93 ((CH_2)C*H*O)□ 3.27-3.42 (5.46%), 2.34-2.47 (6.98%), 0.45-0.58 (0.88%); 4.32-4.45 (C*H*OMe) □ 4.80-4.93 (0.47%), 3.27-3.42 (1.11%), 2.92-3.07 (1.10%), 2.78-2.92 (5.25%); 2.92-3.07 (C*H*Ph) □4.32-4.45 (1.06%), 2.78-2.92 (3.32%), 2.34-2.47 (7.86%), 0.45-0.58 (0.35%); 2.78-2.92 (C*HH*Ph) □ 4.80-4.93 (0.47%), 4.32-4.45 (4.28%), 2.92-3.07 (2.90%), 2.34-2.47 (0.22%); 2.34-2.47 (C*H*Me) □ 4.80-4.93 (8.76%), 2.92-3.07 (8.71%), 0.45-0.58 (5.17%). Mixture of isomers: IR (KBr): 3029, 2931, 1603, 1454, 1265, 1105, 1032, 910, 739, 701 cm^{-1}. CI (MS) m/z (%): 282 (M^+, 16), 251 (59), 191 (55), 162 (47), 159 (52), 147 (41), 131(51), 115 (31), 91 (100). HRMS for $C_{19}H_{22}O_2$ [M^+]: calcd 282.1620, found 282.1621.

2-(1-Methoxyprop-2-ynyloxy)indan (3). To a solution of 2-(2-iodo-1-methoxy-allyloxy)indan **1a** (6.60 g, 20 mmol) in *t*BuOH (30 mL) was added portionwise *t*BuOK (2.46 g, 22 mmol) at room temperature under nitrogen atmosphere. The solution was slowly warmed up to 40°C and stirred until total disappearance of the starting iodoacetal (TLC monitoring). Then water was added to the reaction mixture; the aqueous phase was extracted with Et_2O. The combined organic phases were dried ($MgSO_4$) and concentrated under reduced pressure. FC (AcOEt/Hexane: 95/5) gave **3** (3.84 g, 95%) as a colorless oil. ^{1}H NMR (300MHz, $CDCl_3$) □: 7.14□7.18 (m, 4H, Harom), 5.34 (d, 1H, J= 1.84), 4.75 (m, 1H), 3.43 (s, 3H), 3.42-3.27 (m, 2H), 3.17-2.99 (m, 2H), 2.59 (d, 1H, J= 1.84). ^{13}C NMR (400 MHz, $CDCl_3$) □: 39.4 (CH_2), 39.9 (CH_2), 52.1 (CH_3), 73.9 (CH), 77.1 (CH), 78.5 (C), 91.3 (CH), 124.4 (2□CH), 126.4 (2□CH), 140.3 (C), 140.5 (C). IR (KBr): 3303, 3012, 2907, 2126, 1734, 1482, 1460, 1329, 1219, 1192, 1107, 1050, 980, 756, 667 cm^{-1}. EI (MS) m/z (%): 202 (M^+, 4), 184 (5), 171 (13), 159 (13), 159 (13), 117 (65), 116 (100), 115 (45), 105 (35), 91 (13), 77 (15), 69 (89), 39 (23). HRMS for $C_{13}H_{14}O_2$ [M^+]: calcd 202.0994, found 202.0994.

2-Methoxy-3-phenylsulfanylmethyl-3,3a,8,8a-tetrahydro-2H-1-oxa-cyclopenta[*a*]indene ((±)-4). A solution of AIBN (164 mg, 1.00 mmol) in benzene (2 mL) and a solution of thiophenol (110 mg, 1.00 mmol) in benzene (2 mL) were added with a syringe pump (needles were placed in the condensor) over 20 h to a solution of

dialkoxypropyne **3** (202 mg, 1.00 mmol) and AIBN (80 mg, 0.5 mmol) in refluxing *t*BuOH (100 mL). After disappearance of the starting acetal **3** (TLC monitoring), the solution was cooled down and concentrated under reduced pressure. The residue was filtrated through silica gel. The filtrate was concentrated under reduced pressure and FC (AcOEt/hexane) of the residue afforded **4** (209 mg, 67% yield). The ratio of the isomers was determined from ^{1}H NMR spectra of the crude product.

(*r*-2,*t*-3,*t*-4,*t*-5)-**4** (66% ds): ^{1}H NMR (400 MHz, $CDCl_3$) : 7.13-7.25 (m, 9H), 5.08 (td, 1H, *J* = 2.1, 7.28), 4.84 (m, 1H), 4.09 (1H), 3.46 (s, 3H), 3.22 (dd, 1H, *J* = 7.28, 17.57), 3.16 (dd, 1H, *J* = 1.51, 7.57), 2.90 (dd, 1H, *J* =5.27, 13.05), 2.66 (m, 1H), 2.38 (m, 1H). ^{13}C NMR (400 MHz, $CDCl_3$) : 33.1 (CH_2), 39.3 (CH_2), 47.5 (CH), 51.7 (CH), 54.9 (CH_3), 82.5 (CH), 109.6 (CH), 125.4 (CH), 126.1 (CH), 126.2 (CH), 126.6 (CH), 127.5 (CH), 128.9 (CH), 129.1 (CH), 129.4 (CH), 130.5 (C), 130.6 (CH),131.9 (C), 135.8 (C). NOE difference spectra (400 MHz): 5.03-5.18 ((CH$_2$)C*H*O) 4.06-4.21 (6.38%), 3.2-3.37 (4.65%), 2.66-2.81 (0.68%), 2.39-2.52 (0.2%); 4.95-5.03 (C*H*OMe) 3.35-3.5 (5.2%), 3.2-3.37 (0.92%), 2.66-2.81 (2.46%), 2.38-2.52 (2.6%); 4.06-4.21 (C*H*CHO) 5.03-5.18 (6.65%), 2.66-2.81 (9.23%); 3.35-3.5 (C*H*$_3$OCH) 5.03-5.18 (0.59%), 4.95-5.03 (2.4%), 4.06-4.21 (0.26%), 2.66-2.81 (0.33%); 3.04-3.19 (C*H*$_2$Ph) 5.03-5.18 (1.56%), 4.95-5.03 (0.6%), 4.06-4.21 (0.5%), 2.38-2.52 (0.99%); 2.88-3.03 (C*H*HSPh) 4.95-5.03 (2.2%), 2.66-2.81 (3.27%); 2.66-2.81 (C*H*CH(OMe) 5.03-5.18 (0.34%), 4.95-5.03 (2.9%), 4.06-4.21 (9.3%); 2.38-2.52 (CH*H*SPh) 4.95-5.03 (5.3%), 4.06-4.21 (0.44%), 3.22-3.37 (1.3%), 3.35-3.5 (3.12%).

(*r*-2,*c*-3,*t*-4,*t*-5)-**4** (34% ds): ^{1}H NMR (400 MHz, $CDCl_3$) : 7.5-7.0 (m, 9H), 4.92 (d, 1H, J=4.77), 4.89 (td, 1H, J=1.76, 6.77), 3.54 (m, 1H), 3.42 (dd, 1H, J= 7.53, 12.8), 3.29 (m, 1H), 3.28 (s, 3H), 3.16 (m, 1H), 2.98 (dd, 1H, J=7.78, 17.32), 2.55 (m, 1H). ^{13}C NMR (400 MHz, $CDCl_3$) : 39.9 (CH_2), 39.2 (CH_2), 52.5 (CH_3), 54.8 (CH), 55.2 (CH), 82.2 (CH), 106.2 (CH), 125.4 (CH), 125.7 (CH), 126.3 (CH), 127.7 (2CH), 127.8 (2CH), 129.5 (CH), 129.6 (CH), 137.9 (C), 141.8(C), 145.6 (C). NOE difference spectra (400 MHz): 4.90-5.02 (C*H*(OMe)) 2.9-3.05 (0.94%), 2.48-2.64 (6.9%); 4.78-4.90 (CH$_2$C*H*O) 3.47-3.62 (6.82%), 2.9-3.05 (1.9%); 3.47-3.62 (C*H*CHO) 2.9-3.05 (2.4%), 2.48-2.64 (2.02%); 3.35-3.47 (C*H*$_2$SPh) 4.78-4.9 (0.5%), 3.47-3.62 (1.33%), 2.48-2.64 (4.2%). Mixture of isomers: IR (KBr): 3008, 2930, 1683, 1580, 1481, 1438, 1100, 1059, 1033, 756, 735, 690 cm^{-1}. EI (MS) m/z

(%): 335 ([M+Na]$^+$, 49), 280 (51), 218 (20), 171 (17), 143 (26), 116 (52), 105 (19), 84 (100), 69 (46), 56 (19), 39 (15). HRMS for $C_{19}H_{20}O_2SNa$ [M+Na]$^+$: calcd 335.1068, found 335.1081.

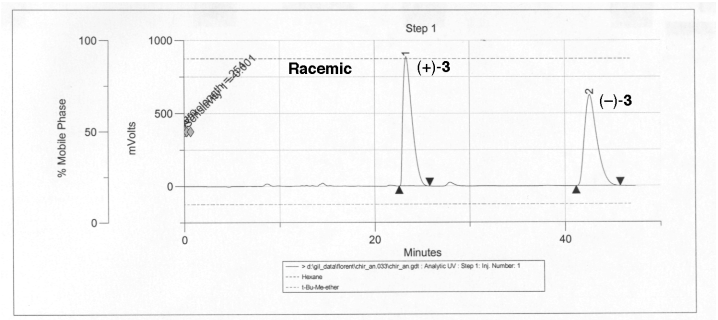
***cis*-3-Methylene-3,3a,8,8a-tetrahydro-1-oxacyclopenta[a]inden-2-one ((±)-5).** To a cooled solution (0°C) of **4** (423 mg, 1.35 mmol) in acetone (2 mL) was added the Jones reagent (16 mL prepared according to the procedure described below). The solution was allowed to warm up slowly to room temperature and stirred during 2 h, then chromium salts were removed by filtration. After evaporation of acetone, CH_2Cl_2 was added to the residue and the solution was washed with $NaHCO_3$ (3□10 mL). The organic phase was dried ($MgSO_4$) and concentrated under reduced pressure to give crude 3-benzenesulfonylmethyl-3,3a,8,8a-tetrahydro-1-oxa-cyclopenta[*a*]inden-2-one. DBU (193 mg, 1.27 mmol) was added to the crude sulfonyllactone in toluene (2 mL). The reaction mixture was heated at 100 °C for 4 h. The solvent was removed and FC (AcOEt/hexane 1:2) of the residue afforded the desired □-methylenelactone **5** (143 mg, 57 %). Spectroscopic data are in accordance with literature data.[1]

Preparation of Jones reagent: Concentrated H_2SO_4 (230 mL) was added under cooling with an ice bath to CrO_3 (267 g, 2.67 mol) in H_2O (400 mL). The cold solution is diluted with H_2O up to 1 L to form the 8 N Jones reagent ready for use.

2-Methoxy-3-(toluene-4-sulfonylmethyl)-3,3a,8,8a-tetrahydro-2*H*-1-oxacyclopenta[*a*]indene (6): To a solution of **3** (186 mg, 1.0 mmol) and AIBN (164 mg, 1.0 mmol) in refluxing *t*BuOH (100 mL) was added over 20 h a solution of AIBN (1.00 mmol, 164 mg) and thiocresol (1.00 mmol, 124 mg) in benzene (2 mL) through a syringe pump (the needles were placed in the condensor). After completion (TLC monitoring), the solution was cooled and *t*BuOH evaporated under reduced pressure. The residue was filtrated through silica gel and concentrated. FC (AcOEt/hexane 10:90) of the residue afforded **6** (147 mg, 45%) as a mixture of two diastereomers in a 2:1 ratio. The major isomer (*r*-2,*t*-3,*t*-4,*t*-5)-**6** (66% ds) was isolated by FC (AcOEt/hexane 1:99). A solution of (*r*-2,*t*-3,*t*-4,*t*-5)-**6** (33 mg, 0.1 mmol) in THF (5 mL) was oxidized with magnesium monoperphthalate (92 mg, 0. 20 mmol). The solution was stirred during 24 h at rt and then $CHCl_3$ (10 mL) was added. After filtration, the solution was washed with sat. Na_2CO_3, H_2O, NaCl, and dried over

$MgSO_4$. Recrystallization from dichloromethane/hexane gave crystals of **6** (33 mg, 92%) that were suitable for an X-ray diffraction analysis. M.p. 153–155 °C. ^{1}H NMR (400 MHz, $CDCl_3$) □: 7.72 (m, 2H), 7.5 (m, 2H), 7.19 (m, 3H), 7.05 (m, 1H) 4.98 (m, 1H), 4.91 (m, 1H), 4.15 (m, 1H), 3.32 (s, 3H), 3.22 (m, 1H), 2.90 (m, 3H), 2.53 (m, 1H), 2.45 (s, 3H). ^{13}C NMR (400 MHz, $CDCl_3$) □: 21.7 (CH_3), 39.1 (CH_2), 42.7 (CH), 51.7 (CH), 54.9 (CH_3), 55.0 (CH_2), 81.8 (CH), 109.1 (CH), 125.6 (CH), 126.2 (CH), 127.1 (CH), 127.9 (CH), 128.2 (2□CH), 130.0 (2□CH), 136.1 (C), 139.0(C), 142.7 (C), 144.8 (C). EI (MS) m/z (%): 381 ([M+Na]$^+$, 100), 353 (27), 171 (85), 149 (75), 143 (48). HRMS for $C_{20}H_{22}O_4SNa$ [M+Na]$^+$: calcd 381.1144, found 381.1136.

Enantiomerically enriched □-methylene lactone (+)-5: Separation of enantiomers of 2-(1-methoxy-prop-2-ynyloxy)indan **3** was performed by HPLC on chiral column *Daicel Chiralcel OJ* 250 □ 4.6 mm (hexane/EtOAc 90:10, 8 mg/run). (+)-**3**: t_{ret} = 23 min; [□]$^{23}_D$= +25.5° (c = 1.0, $CHCl_3$); 99% ee). (–)-**3**: t_{ret} = 43 min; [□]$^{23}_D$= –20.5° (c = 1.0, $CHCl_3$); 96% ee.



	Inj. Number	Peak Name	R. Time	Area	Sample Descrip.				
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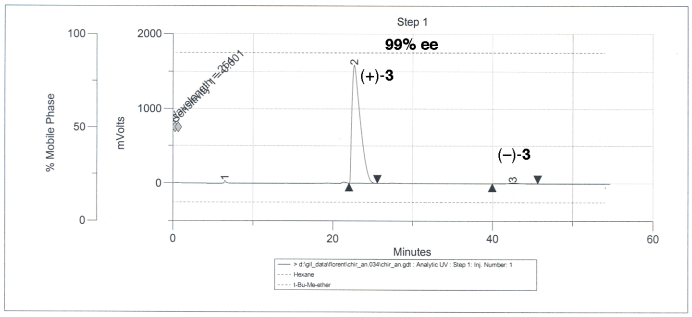
2	1.00	*2	42.58	94431416.00	Step 1				



	Inj. Number	Peak Name	R. Time	Area	Sample Descrip.				
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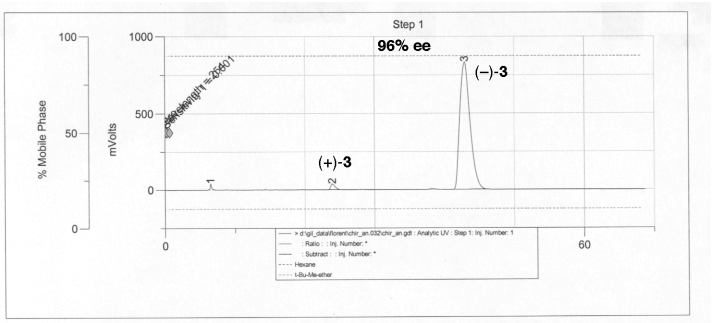
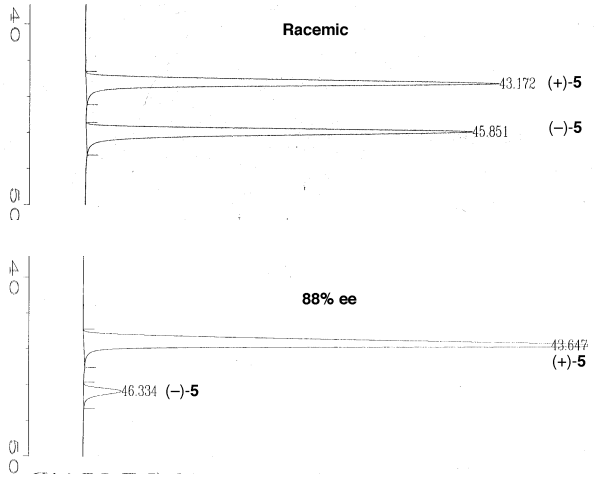
2	1.00	*3	42.53	2121033.50	Step 1				



	Inj. Number	Peak Name	R. Time	Area	Sample Descrip.				
1	1.00	2	23.93	2865413.75	Step 1				
2	1.00	3	42.95	141917168.00	Step 1				

According to procedure described above, from (–)-**3** (90 mg, 0.45 mmol, 96% ee). FC (hexane/AcOEt 90:10) gave **4** (90 mg, 64%) as a mixture of 2 diastereomers in a 2:1 ratio that was used as it stand for the next step. Compound **4** (mixture of diastereomers, 90 mg, 0.29 mmol) was converted into (+)-**5** according to the procedure reported above for the racemic compound (see above). FC (AcOEt/hexane 1:2) of the residue afforded (+)-**5** (17 mg, 31%). $[\square]^{23}_{D}$= +117° (c = 1.0, $CHCl_3$). The enantiomeric purity of (+)-**5** (88% ee) was determined by GC on a chiral column (15% DIMETTBSB in OV-1701, 25 m, 150 °C).



[1] G. Haaima, A. Routledge, R. Weavers *Tetrahedron Lett.* **1989**, *30*, 5159.